Supplementary Information for

# Intermolecular Oxyarylation of Olefins with Aryl Halides and TEMPOH Catalyzed by Phenolate Anion under Visible Light

Kangjiang Liang, Qian Liu, Lei Shen, Xipan Li, Delian Wei, Liyan Zhang and Chengfeng Xia*

Key Laboratory of Medicinal Chemistry for Natural Resource (Ministry of Education and Yunnan Province), State Key Laboratory for Conservation and Utilization of Bio-Resources in Yunnan, and School of Chemical Science and Technology, Yunnan University, 2 North Cuihu Road, Kunming 650091, China.

*Correspondence to: xiacf@ynu.edu.cn

## Table of Contents

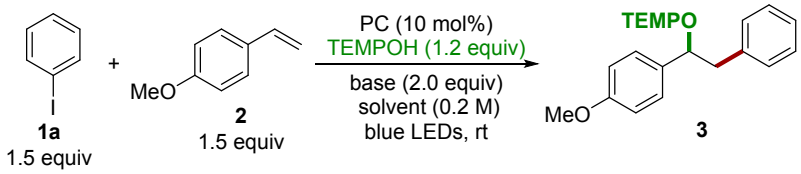
## General Information

All reactions were performed under argon atmosphere using flame-dried glassware unless otherwise noted. DMSO was distilled over $CaH_2$ and rigorously degassed by freeze/pump/thaw. All reagents were commercially available and used without further purification unless indicated otherwise. Thin layer chromatographies were carried out on GF254 plates (0.25 mm layer thickness). Flash chromatography was performed with 200-300 mesh silica gels. Visualization of the developed chromatogram was performed by fluorescence quenching or by ceric ammonium molybdate, or $KMnO_4$ stain. Yields reported were for isolated, spectroscopically pure compounds.

$^1H$ and $^{13}C$ NMR spectra were recorded on Bruker Avance 400 and 600 MHz spectrophotometers. Chemical shifts (δ) are expressed in ppm., and *J*-values are given in Hz. The residual solvent protons ($^1H$) or the solvent carbons ($^{13}C$) were used as internal standards. ESIMS and HRESIMS were taken on AB QSTAR Pulsar mass spectrometer or Aglient LC/MSD TOF mass spectrometer. UV-Vis measurements were carried out on a Hitachi UV-1900 UV-Visible spectrophotometer. Cyclic voltammetry studies was carried out on a CHI 760E electrochemical workstation (Shanghai CH Instruments Co., China). The emission spectra were recorded in a Hitachi F-7000 fluorescence spectrometer. Fluorescent lifetimes were determined on HORIBA Fluorolog-3 with a light-emitting diode lamp and analyzed by the use of a program for exponential fits.

## Optimization of the Conditions

**Supplementary Table 1.** Additional bases and solvents screening



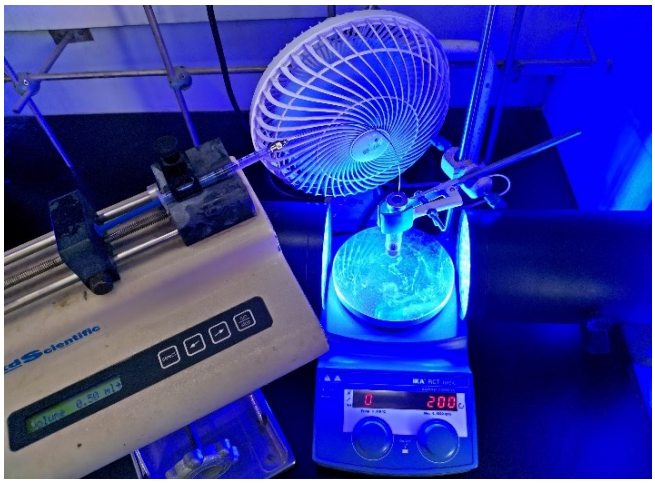

| entry[a] | base | solvent | yield (%)[b] |
|---|---|---|---|
| 1 | $Cs_2CO_3$ | DMSO | 87 |
| 2 | $KHCO_3$ | DMSO | 20 |

| | | | |
|---|---|---|---|
| 3 | $Na_2CO_3$ | DMSO | 18 |
| 4 | $K_2CO_3$ | DMSO | 72 |
| 5 | $K_3PO_4$ | DMSO | 50 |
| 6 | DBU | DMSO | 53 |
| 7 | TMG | DMSO | 65 |
| 8 | $Cs_2CO_3$ | DMF | 66 |
| 9 | $Cs_2CO_3$ | DMA | 61 |
| 10 | $Cs_2CO_3$ | acetone | 33 |
| 11 | $Cs_2CO_3$ | $CH_3CN$ | 38 |

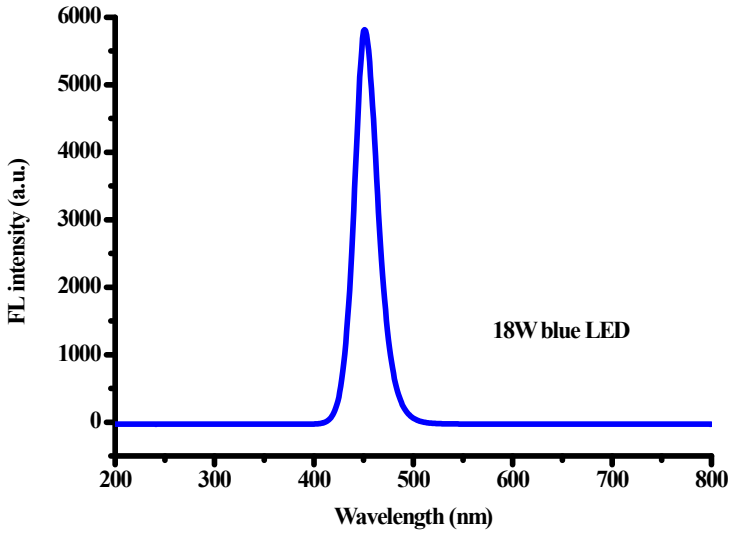
[a]General conditions: **1a** (0.30 mmol, 1.0 equiv), 4-methoxystyrene **2** (0.45 mmol, 1.5 equiv), TEMPOH (0.36 mmol, 1.2 equiv), base (0.60 mmol, 2.0 equiv), **PC3** (0.03 mmol, 10 mol%), and solvent (1.5 mL, rigorously degassed by freeze/pump/thaw). **1a** and TEMPOH was dissolved in solvent and added dropwise over 10 h. [b]Isolated yields by chromatography.

**General Procedure for Photocatalytic Intermolecular Oxyarylation of Olefins**

To an oven dried 10 mL glass tube with a magnetic stirring bar was added **PC3** (0.03 mmol, 10 mol%), and $Cs_2CO_3$ (0.60 mmol, 2.0 equiv). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (1.0 mL), and olefin (0.45 mmol, 1.5 equiv) were carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation at room temperature and a solution of (hetero)aryl halide (0.30 mmol, 1.0 equiv) and TEMPOH (0.36 mmol, 1.2 equiv) in DMSO (0.5 mL) was added dropwise by syringe over 10 hours. After irradiating for the indicated time, the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether) on silica gel to afford the product.



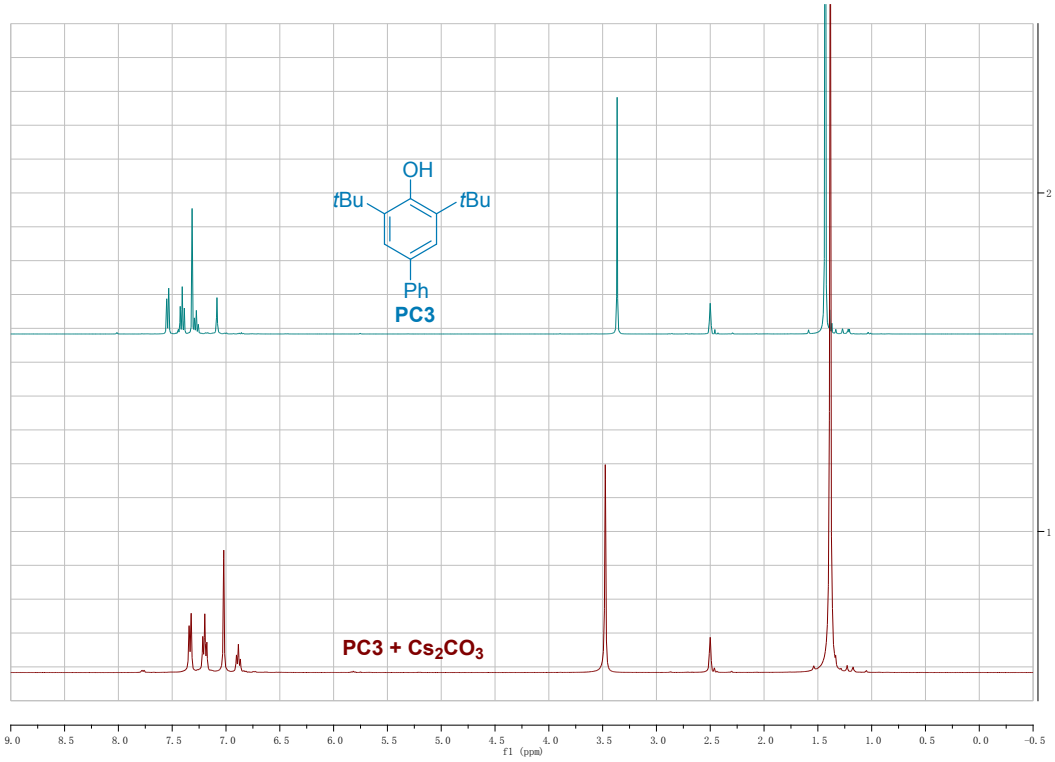
**Supplementary Figure 1.** Experimental setup for photocatalytic oxyarylation of olefins.




**Supplementary Figure 2.** Emission spectra of the 18W blue LED lamp.

**Supplementary Note 1. $^1$H NMR Spectroscopic Studies**

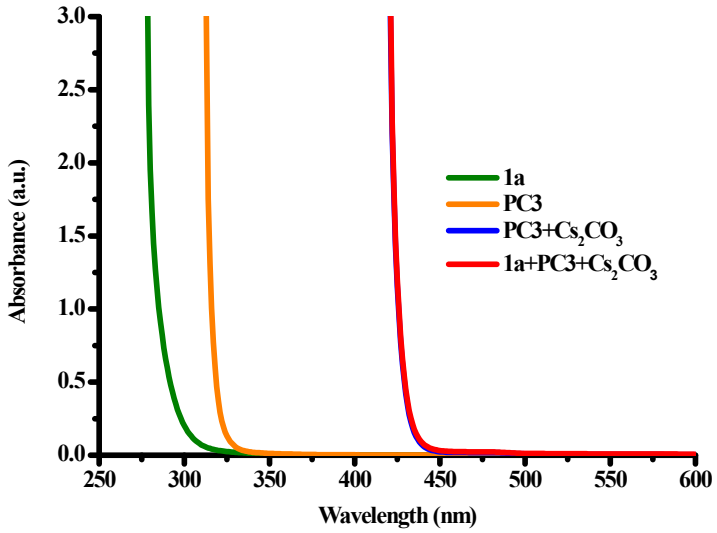
The $^1$H NMR analysis was made on a solution containing **PC3** (14.1 mg, 0.05 mmol) and $Cs_2CO_3$ (17.9 mg, 0.055 mmol) in 0.5 mL of DMSO-$d_6$ (rigorously degassed by freeze/pump/thaw). Under these conditions, **PC3** was completely deprotonated by $Cs_2CO_3$ and significant upfield peak shifting of hydrogens were observed.




**Supplementary Figure 3.** Comparison of $^1$H NMR spectra of **PC3** and the phenolate anion of **PC3** in DMSO-$d_6$.

**Supplementary Note 2. UV-Vis Spectroscopic Measurements**

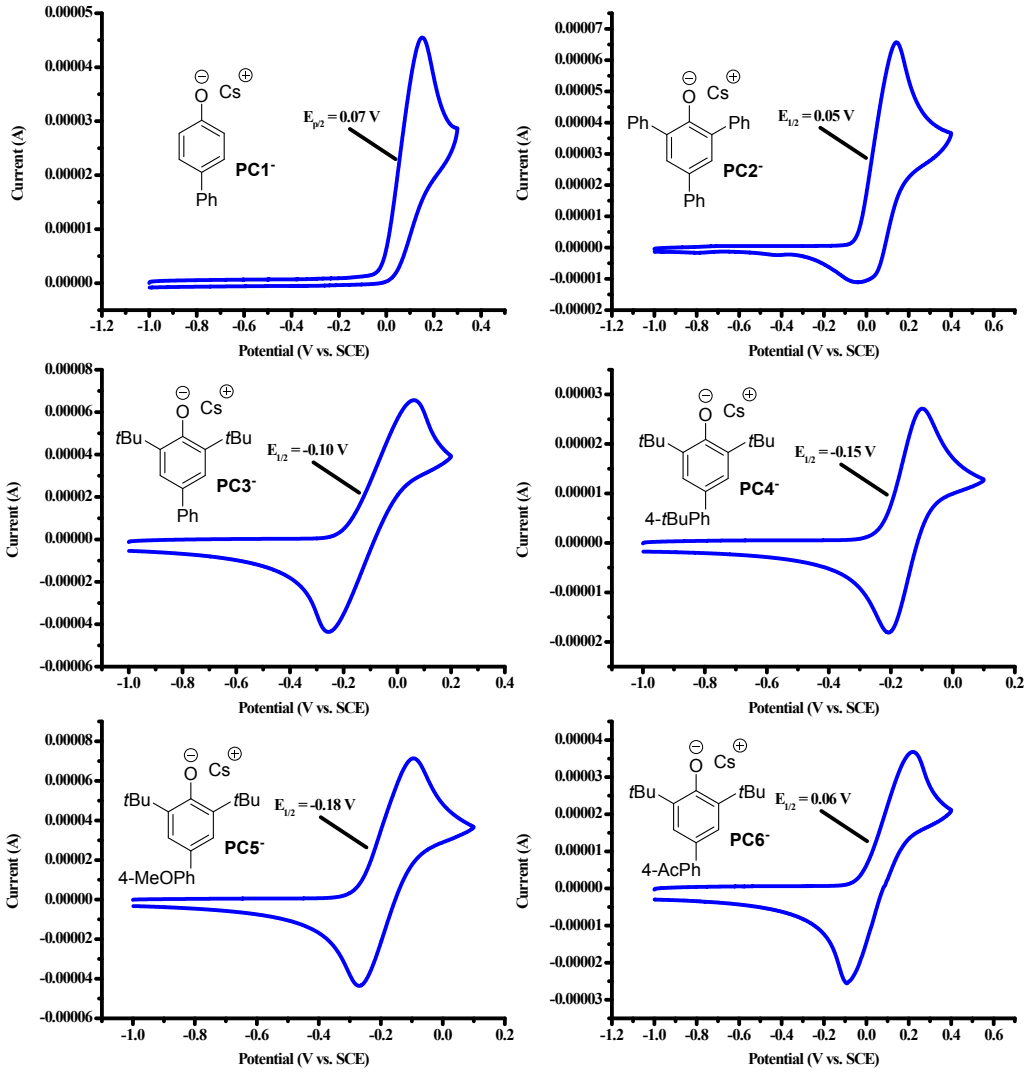
The UV-Vis absorption spectra of DMSO solutions (0.1 M) of **1a**, **PC3**, mixtures of **PC3** and $Cs_2CO_3$, and mixtures of **1a**, **PC3** and $Cs_2CO_3$ were recorded on Hitachi UV-1900 UV-Visible spectrophotometer (1 mm short light path cuvettes have been employed in order to avoid fast signal saturation). The colorless solution of **PC3** (orange line) was immediately turned to a primrose yellow color upon addition of $Cs_2CO_3$ (blue line) and no new color change after the iodobenzene **1a** was added to the solution of the phenolate anion of **PC3** (red line) indicating that no EDA ground state association occurred and the photon-absorbing ability of the phenolate anion in the visible spectral region was responsible for triggering the aryl radical from its halide.




**Supplementary Figure 4.** UV-Vis absorption spectra of mixtures of **1a**, **PC3**, and $Cs_2CO_3$ in DMSO at concentrations of 0.1M.

**Supplementary Note 3. Electrochemical Measurements**

Tetrabutylammonium hexafluorophosphate (387 mg, 1.0 mmol) was added to a 0.01 M solution of the phenolate anion of the phenol catalyst (generated *in situ* by the deprotonation of the phenol catalyst with 1.1 equiv $Cs_2CO_3$) in 10 mL of dry DMSO and the solution was vigorously bubbled with $N_2$ for 5 minutes prior to the measurement. The oxidation/reduction potential was measured using a glassy carbon working electrode, a platinum wire counter electrode, and a saturated calomel electrode (SCE) at 0.1 V/s scan rate.




**Supplementary Figure 5.** The cyclic voltammogram of the phenolate anions vs SCE in DMSO at 0.1V/s.

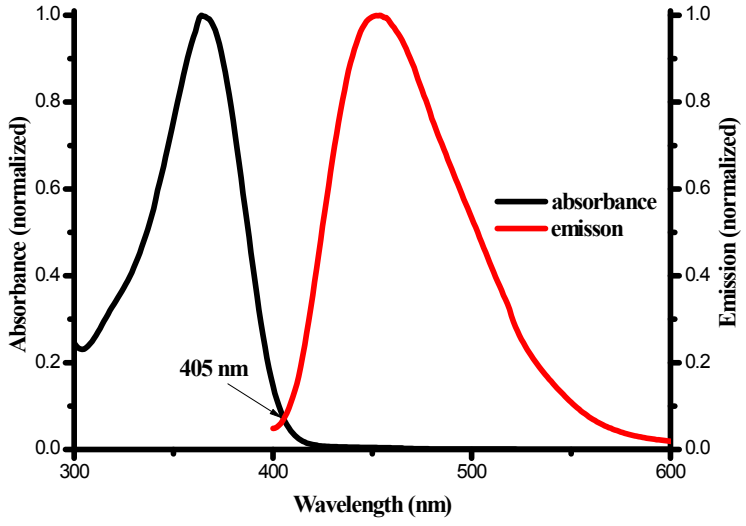
In order to probe whether the steric hindrance of the bis-*t*Bu system could improve the kinetic stability of the corresponding phenoxyl radicals, electrochemical analysis were carried out on the phenolate anions of **PC1** - **PC6**. The redox reversibility of the phenolate anions of **PC3** - **PC6** suggested that the introduction of bis-tBu remarkably prolongs the lifetime of corresponding phenoxyl radicals.

With this data in hand we calculated the redox potential of the excited phenolate anion of **PC3** employing the following equation: [1]

$$E_{1/2}(\mathbf{PC3}^{\bullet}/\ \mathbf{PC3}^{-*}) = E_{1/2}\ (\mathbf{PC3}^{\bullet}/\ \mathbf{PC3}^{-}) - E_{0\text{-}0}(\mathbf{PC3}^{-*}/\ \mathbf{PC3}^{-})$$

$E_{1/2}(\mathbf{PC3}^{\bullet}/\ \mathbf{PC3}^{-})$ = -0.10 V vs. SCE. $E_{0\text{-}0}(\mathbf{PC3}^{-*}/\ \mathbf{PC3}^{-})$, the excited state energy of the phenolate anion of **PC3**, was estimated from the intersection of the normalized absorbance and emission spectra.[2] This corresponds to 405 nm, which translates into an $E_{0\text{-}0}(\mathbf{PC3}^{-*}/\ \mathbf{PC3}^{-})$ of 3.06 eV for the phenolate anion of **PC3**.

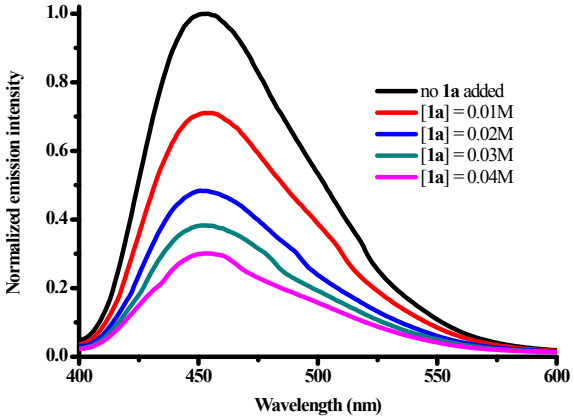
$E_{1/2}(\mathbf{PC3}^{\bullet}/\ \mathbf{PC3}^{-*}) = E_{1/2}\ (\mathbf{PC3}^{\bullet}/\ \mathbf{PC3}^{-}) - E_{0\text{-}0}(\mathbf{PC3}^{-*}/\ \mathbf{PC3}^{-})$ = -0.10 – 3.06 = -3.16 V vs. SCE




**Supplementary Figure 6.** Normalized absorption and emission spectra of the phenolate anion of **PC3** in dry DMSO ($5 \times 10^{-5}$ M), the intersect wavelength was considered to be 405 nm.

**Supplementary Note 4. Steady-State Luminescence Quenching Studies**

The samples were prepared mixing the phenolate anion of **PC3** ($5 \times 10^{-5}$ M, freshly prepared *in situ* by the deprotonation of **PC3** with 1.1 equiv $Cs_2CO_3$) with the required amount of **1a** in a total volume of 1 mL of dry DMSO (rigorously degassed by freeze/pump/thaw) in a 10 × 10 mm light path quartz fluorescence cuvette under an argon atmosphere. The samples were vigorously bubbled with dry argon for 5 minutes prior to the measurement. The excitation wavelength was fixed at 380 nm, the emission light was acquired from 400 nm to 600 nm.

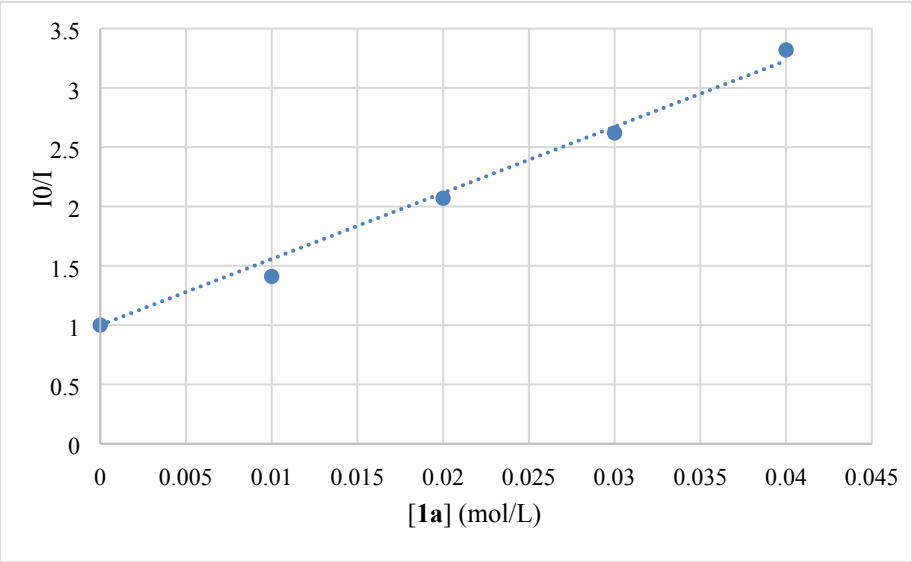



**Supplementary Figure 7.** Quenching of the phenolate anion of **PC3** emission ($5 \times 10^{-5}$ M in DMSO) in the presence of increasing amounts of **1a**.

The Stern-Volmer plot shows a linear correlation between the amounts of **1a** and the ratio $I_0/I$. The quenching of excited-state photosensitizer with externally added quencher (Q) follows Stern-Volmer relationship: [3]

$$I_0/I = 1 + k_q\tau_0[Q] = K_{SV}[Q] \qquad \text{Eq. S1}$$

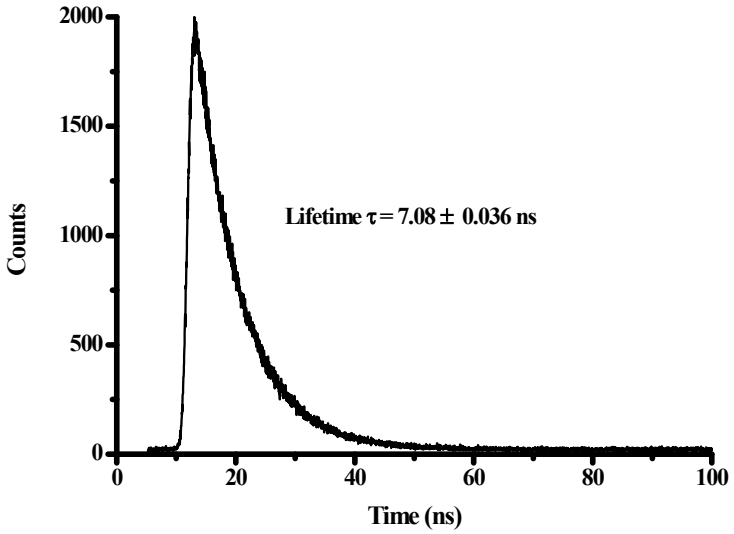
Where $I_0$ is the intensity at emission maximum in the absence of quencher; I is the intensity at the emission maximum in the presence of quencher at a concentration of [Q]; $\tau_0$ is the lifetime of the photosensitizer in the absence of quencher; $k_q$ is the rate constant for the quenching process by the quencher; $K_{SV}$ (= $k_q\tau_0$) is directly obtained from Stern-Volmer plots as 55.6 $M^{-1}$. Based on the fluorescence lifetime of the phenolate anion of **PC3** ($\tau_0$ = 7.08 ns), the quenching rate constant $k_q = K_{SV}/\tau_0 = 7.9 \times 10^9$ $M^{-1}$ $s^{-1}$ can be calculated for iodobenzene **1a**.



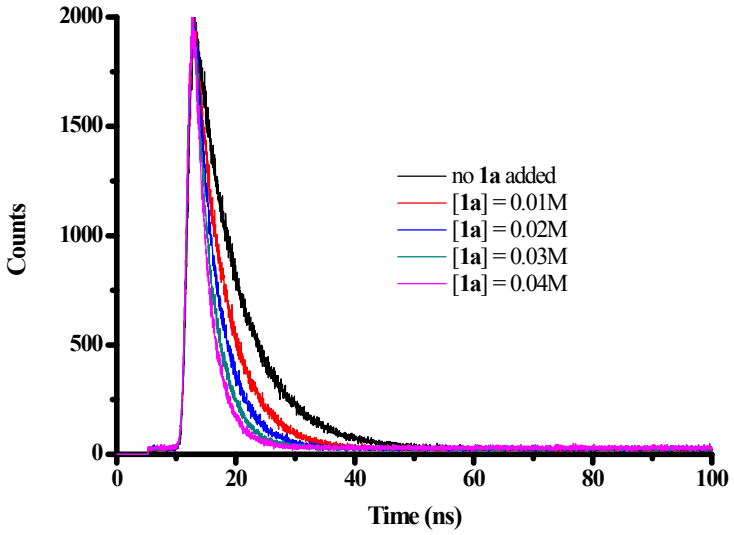

**Supplementary Figure 8.** Stern-Volmer quenching plot.

## Supplementary Note 5. Lifetime Measurements and Time-Resolved Quenching Studies

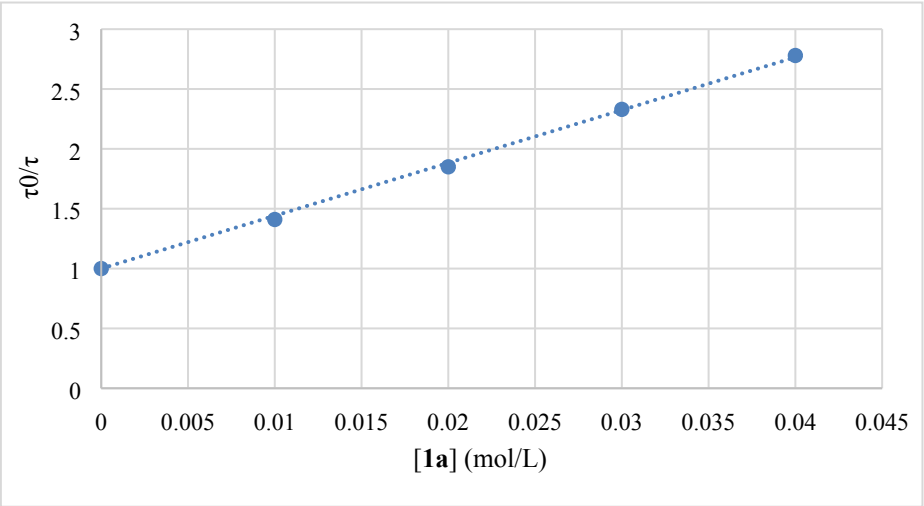
Fluorescence lifetime of the phenolate anion of **PC3** ($5 \times 10^{-5}$ M, freshly prepared *in situ* by the deprotonation of **PC3** with 1.1 equiv $Cs_2CO_3$) in DMSO were determined on HORIBA Fluorolog-3 with a light-emitting diode lamp and analyzed by the use of a program for exponential fits. The decay data was collected at 450 nm upon 380 nm excitation.




**Supplementary Figure 9.** Fluorescence lifetime of the phenolate anion of **PC3** ($5 \times 10^{-5}$ M) in DMSO.

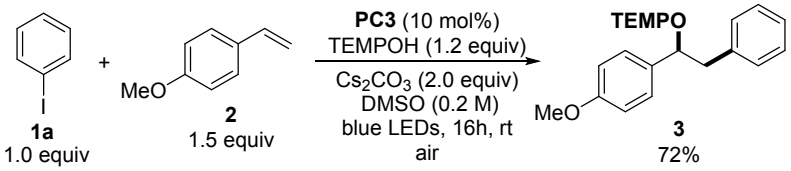


**Supplementary Figure 10.** Time-resolved luminescence quenching experiments of the phenolate anion of **PC3** ($5 \times 10^{-5}$ M) in DMSO with **1a** as quencher.



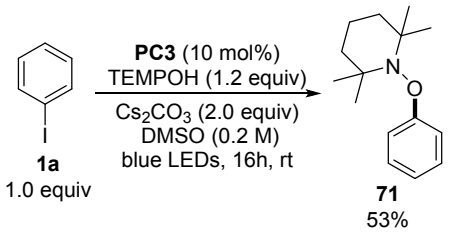

**Supplementary Figure 11.** Stern-Volmer plot of the time-resolved quenching experiments ($K_{SV}'$ = 44.1 $M^{-1}$).

## Supplementary Note 6. Oxygen Tolerance Experiments




To a 10 mL glass tube with a magnetic stirring bar was added **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol). and DMSO (1.0 mL). The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation at room temperature and a solution of iodobenzene **1a** (61.2 mg, 0.30 mmol) and TEMPOH (56.6 mg, 0.36 mmol) in DMSO (0.5 mL) was added dropwise by syringe over 10 hours. After irradiating for 16 hours under air, the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:200) on silica gel to afford **3** a colorless oil (79.4 mg, 72% yield).

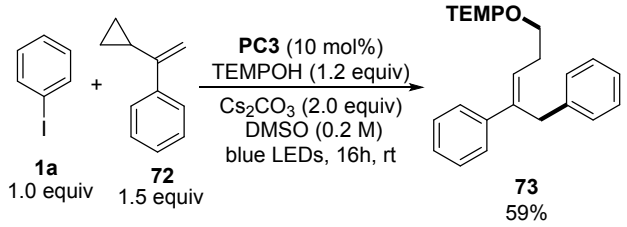
**Supplementary Note 7. Controlled Experiments without 4-Methoxystyrene.**




To an oven dried 10 mL glass tube with a magnetic stirring bar was added **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (1.0 mL) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation at room temperature and a solution of iodobenzene **1a** (61.2 mg, 0.30 mmol) and TEMPOH (56.6 mg, 0.36 mmol) in DMSO (0.5 mL) was added dropwise by syringe over 10 hours. After irradiating for 16 hours, the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:400) on silica gel to afford **71** a colorless oil (37.1 mg, 53% yield).

**2,2,6,6-tetramethyl-1-phenoxypiperidine (71)**: $^1$H NMR (600 MHz, $CDCl_3$): δ 7.23 – 7.14 (m, 4H), 6.86 – 6.81 (m, 1H), 1.70 – 1.38 (m, 6H), 1.23 (s, 6H), 1.01 (s, 6H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 163.6, 128.6, 119.9, 113.9, 60.3, 39.8, 32.6, 20.5, 17.1; HR-ESI-MS (*m/z*): calcd. for $C_{15}H_{24}ON$ $[M + H]^+$, 234.1852, found 234.1854.

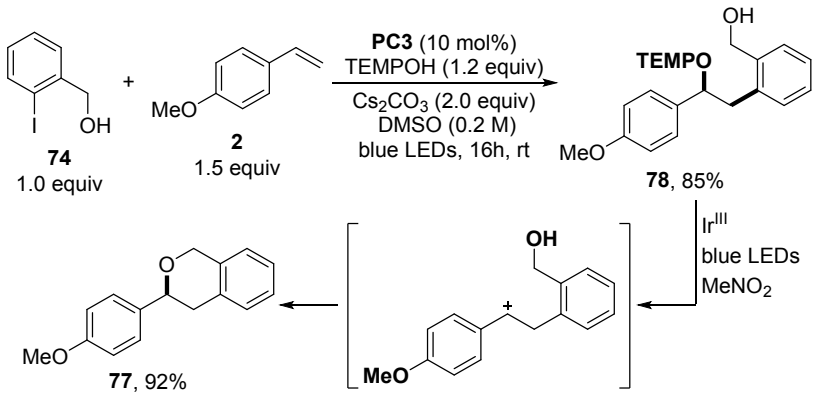
**Supplementary Note 8. Radical Clock Experiments**




To an oven dried 10 mL glass tube with a magnetic stirring bar was added **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (1.0 mL) and α-cyclopropylstyrene **72** (64.8 mg, 0.45 mmol) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation at room temperature and a solution of iodobenzene **1a** (61.2 mg, 0.30 mmol) and TEMPOH (56.6 mg, 0.36 mmol) in DMSO (0.5 mL) was added dropwise by syringe over 10 hours. After irradiating for 16 hours, the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:150) on silica gel to afford **73** a colorless oil (66.8 mg, 59% yield).

**(*E*)-1-((4,5-diphenylpent-3-en-1-yl)oxy)-2,2,6,6-tetramethylpiperidine (73)**: $^1$H NMR (600 MHz, $CDCl_3$): δ 7.28 – 7.22 (m, 2H), 7.20 – 7.12 (m, 5H), 7.12 – 7.03 (m, 3H), 5.96 (t, *J* = 7.3, 1H), 3.84 (s, 2H), 3.80 (t, *J* = 6.6, 2H), 2.45 – 2.40 (m, 2H), 1.41 – 1.23 (m, 6H), 1.10 (s, 6H), 1.04 (s, 6H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 143.1, 139.8, 138.7, 128.4, 128.2, 127.6, 126.6, 126.3, 125.8, 75.9, 59.8, 39.6, 35.9, 33.1, 28.7, 20.2, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{26}H_{36}ON$ $[M + H]^+$, 378.2791, found 378.2794.

**Supplementary Note 9. Intramolecular Carbocation Trapping Experiments**



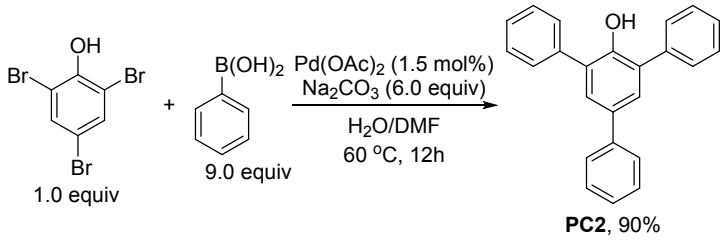

To an oven dried 10 mL glass tube with a magnetic stirring bar was added **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (1.0 mL) and 4-methoxystyrene **2** (60.3 mg, 0.45 mmol) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation at room temperature and a solution of **74** (70.2 mg, 0.30 mmol) and TEMPOH (56.6 mg, 0.36 mmol) in DMSO (0.5 mL) was added dropwise by syringe over 10 hours. After irradiating for 16 hours, the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:20) on silica gel to afford **78** a colorless oil (101.3 mg, 85% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.19 – 7.16 (m, 1H), 7.13 – 7.05 (m, 2H), 7.07 – 7.00 (m, 2H), 6.99 – 6.94 (m, 1H), 6.69 (d, $J$ = 8.3, 2H), 4.81 (t, $J$ = 7.1, 1H), 4.54 (d, $J$ = 12.4, 1H), 4.34 (d, $J$ = 12.4, 1H), 3.69 (s, 3H), 3.61 (dd, $J$ = 13.4, 6.2, 1H), 2.85 (dd, $J$ = 13.4, 8.1, 1H), 2.30 (s, 1H), 1.45 – 0.75 (m, 15H), 0.53 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 158.9, 139.4, 137.1, 134.7, 130.9, 129.3, 128.8, 127.6, 126.4, 113.01, 86.9, 63.3, 60.1, 55.2, 40.6, 38.8, 34.1, 20.6, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{36}O_3N$ $[M + H]^+$, 398.2690, found 398.2688.

To an oven dried 10 mL glass tube with a magnetic stirring bar was added **78** (39.7 mg, 0.10 mmol) and $[Ir(dF\text{-}CF_3\text{-}ppy)_2(d(CF_3)\text{-}bpy)]PF_6$ (1.6 mg, 0.002 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times.

$MeNO_2$ (1.0 mL) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 12h at room temperature. Solvent was removed under vacuum, and the crude product was subjected to column chromatography (acetone/petroleum ether = 1:30) on silica gel to afford **77** a white foam (22.1 mg, 92% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, *J* = 8.4, 2H), 7.35 – 7.24 (m, 2H), 7.21 – 7.18 (m, 1H), 7.17 – 7.09 (m, 1H), 7.00 (d, *J* = 8.4, 2H), 5.06 (s, 2H), 4.75 (dd, *J* = 10.8, 2.9, 1H), 3.88 (s, 3H), 3.16 (dd, *J* = 16.1, 10.8, 1H), 3.01 (dd, *J* = 16.1, 2.9, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 159.2, 134.7, 134.4, 133.7, 128.9, 127.3, 126.5, 126.2, 124.3, 113.9, 76.5, 68.8, 55.4, 36.0; HR-ESI-MS (*m/z*): calcd. for $C_{16}H_{16}O_2Na$ $[M + Na]^+$, 263.1043, found 263.1042.

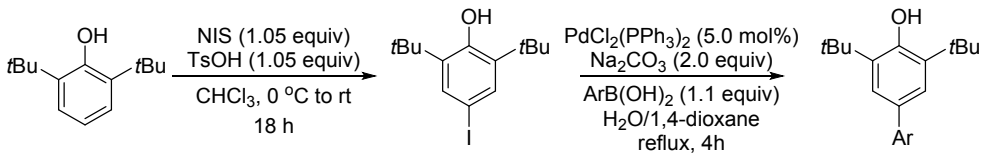
**Identification of Compounds**

**Synthesis of PC2**



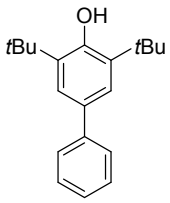

A mixture of $Na_2CO_3$ (636.0 mg, 6.00 mmol), $Pd(OAc)_2$ (3.0 mg, 1.5 mol%), 2,4,6-tribromophenol (330.8 mg, 1.00 mmol), phenylboronic acid (1098.0 mg, 9.0 mmol), distilled water (3.5 mL) and DMF (3 mL) was stirred at 60 °C for 12 h. Afterward, the reaction solution was extracted four times with diethyl ether (4 × 10mL). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:100) on silica gel to afford **PC2** as a white foam (290.0 mg, 90% yield). [4] $^1$H NMR (400 MHz, $CDCl_3$): δ 7.66 – 7.60 (m, 6H), 7.58 (s, 2H), 7.56 – 7.52 (m, 4H), 7.49 – 7.43 (m, 4H), 7.40 – 7.33 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 149.0, 140.6, 137.6, 133.9, 129.4, 129.2, 128.9, 128.8, 128.7, 127.8, 126.9, 126.8; HR-ESI-MS (*m/z*): calcd. for $C_{24}H_{17}O$ $[M - H]^-$, 321.1285, found 321.1283.

**Synthesis of PC3, PC4, PC5, PC6**



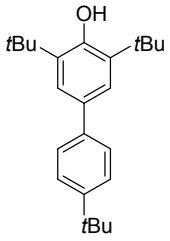

To a stirring mixture of 2,6-dibutylphenol (1.03 g, 5.00 mmol) and *p*-toluenesulfonic acid monohydrate (1.00 g, 5.30 mmol) in $CHCl_3$ (40.0 mL) was added NIS (1.20 g, 5.30 mmol) at 0 °C. The resulting mixture was allowed to room temperature. After stirring for 18 h, to the resulting mixture was added aqueous $NaHCO_3$, and aqueous layers were extracted with $CHCl_3$ (twice). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:150) on silica gel to give 4-iodo-2,6-dibutylphenol (1.53 g, 92% yield). [5]

A stirred mixture of 4-iodo-2,6-dibutylphenol (830.2 mg, 2.50 mmol), arylboronic acid (2.74 mmol), $Na_2CO_3$ (529 mg, 4.99 mmol), $PdCl_2(PPh_3)_2$ (58 mg, 0.14 mmol), water (5 mL), and 1,4-dioxane (15 mL) was heated at reflux under argon for 4 h. The mixture was cooled to room temperature, diluted with ethyl acetate (100 mL), and washed with $H_2O$, and then dried over anhydrous $MgSO_4$. Volatiles were removed by evaporation under reduced pressure, the residue was then purified by column chromatography on silica-gel (petroleum ether/acetone = 150 : 1) to afford the product. [6]



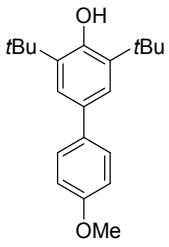

**PC3**

White foam in 585.6 mg, 83% yield. $^1$H NMR (400 MHz, $CDCl_3$): δ 7.61 (d, *J* = 7.7, 2H), 7.53 – 7.43 (m, 4H), 7.36 (t, *J* = 7.3, 1H), 5.32 (s, 1H), 1.56 (s, 18H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 153.5, 142.3, 136.2, 132.6, 128.6, 127.0, 126.4, 124.1, 34.5, 30.4; HR-ESI-MS (*m/z*): calcd. for $C_{20}H_{25}O$ [M - H]$^-$, 281.1911, found 281.1912.



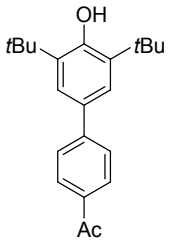

**PC4**

White foam in 676.5 mg, 80% yield. $^1$H NMR (600 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.5, 2H), 7.43 (d, *J* = 8.5, 2H), 7.37 (s, 2H), 4.72 (s, 1H), 1.49 (s, 18H), 1.36 (s, 9H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 153.3, 149.2, 139.5, 136.1, 126.7, 125.5, 123.9, 34.5, 31.4, 30.4; HR-ESI-MS (*m/z*): calcd. for $C_{24}H_{33}O$ [M - H]$^-$, 337.2537, found 337.2536.



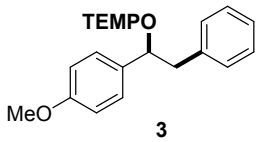

**PC5**

White foam in 601.0 mg, 77% yield. $^1$H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, *J* = 8.5, 2H), 7.27 (s, 2H), 6.88 (d, *J* = 8.5, 2H), 5.14 (s, 1H), 3.78 (s, 3H), 1.42 (s, 18H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 158.5, 153.0, 136.1, 134.9, 132.2, 128.0, 123.7, 114.1, 55.3, 34.5, 30.4; HR-ESI-MS (*m/z*): calcd. for $C_{21}H_{27}O$ $[M - H]^-$, 311.2017, found 311.2019.




**PC6**

White foam in 583.6 mg, 72% yield. $^1$H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 8.2, 2H), 7.56 (d, *J* = 8.2, 2H), 7.37 (s, 2H), 5.29 (s, 1H), 2.56 (s, 3H), 1.43 (s, 18H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 197.8, 154.4, 146.8, 136.5, 135.1, 131.1, 128.9, 126.8, 124.2, 34.5, 30.3, 26.6; HR-ESI-MS (*m/z*): calcd. for $C_{22}H_{27}O_2$ $[M - H]^-$, 323.2017, found 323.2015.

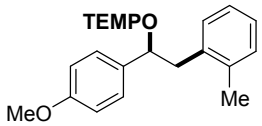



**3**

**1-(1-(4-methoxyphenyl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidin (3)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 16 hours; X = Br, 24 hours; X = Cl, 36 hours). The crude mixture

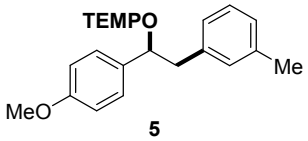
was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **3** as a colorless oil (X = I, 95.9 mg, 87% yield; X = Br, 88.2 mg, 80% yield; X = Cl, 46.3 mg, 42% yield). $^1$H NMR (600 MHz, $CDCl_3$) δ = 7.15 – 7.05 (m, 3H), 7.02 (d, *J* = 8.6, 2H), 6.89 (d, *J* = 7.0, 2H), 6.73 (d, *J* = 8.6, 2H), 4.75 (dd, *J* = 10.0, 4.6, 1H), 3.75 (s, 3H), 3.59 (dd, *J* = 12.8, 4.6, 1H), 2.87 (dd, *J* = 12.8, 10.0, 1H), 1.54 – 1.22 (m, 9H), 1.16 (s, 3H), 1.02 (s, 3H), 0.60 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.7, 138.7, 135.0, 129.7, 129.2, 127.8, 125.7, 112.9, 87.9, 60.1, 55.1, 42.9, 40.5, 34.4, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{24}H_{34}O_2N$ [M + H]$^+$, 368.2584, found 368.2585.




**4**

**1-(1-(4-methoxyphenyl)-2-(o-tolyl)ethoxy)-2,2,6,6-tetramethylpiperidine (4)**

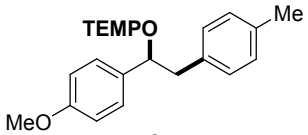
Prepared according to the general procedure using 1-iodo-2-methylbenzene (65.4 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 16 hours; X = Br, 24 hours; X = Cl, 36 hours). The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **4** as a colorless oil (X = I, 98.3 mg, 86% yield; X = Br, 80.1 mg, 70% yield; X = Cl, 48.0 mg, 42% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.06 – 6.94 (m, 5H), 6.91 (d, *J* = 7.3, 1H), 6.73 (d, *J* = 8.6, 2H), 4.80 (dd, *J* = 9.0, 5.7, 1H), 3.76 (s, 3H), 3.58 (dd, *J* = 13.1, 5.7, 1H), 2.85 (dd, *J* = 13.1, 9.1, 1H), 2.09 (s, 3H), 1.56 – 1.32 (m, 6H), 1.26 (s, 3H), 1.10 (s, 3H), 1.03 (s, 3H), 0.61 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.7, 137.1, 136.7, 135.3, 130.7, 129.8, 129.0, 125.9, 125.4, 112.9, 86.7, 59.6, 55.1, 40.6, 39.9, 34.3, 20.4, 19.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{36}O_2N$ [M + H]$^+$, 382.2741, found 382.2740.




**5**

**1-(1-(4-methoxyphenyl)-2-(m-tolyl)ethoxy)-2,2,6,6-tetramethylpiperidine (5)**

Prepared according to the general procedure using 1-iodo-3-methylbenzene (65.4 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 16 hours; X = Br, 24 hours; X = Cl, 36 hours). The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **5** as a colorless oil (X = I, 93.8 mg, 82% yield; X = Br, 86.9 mg, 76% yield; X = Cl, 45.8 mg, 40% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.06 – 6.95 (m, 3H), 6.89 (d, *J* = 7.5, 1H), 6.74 (d, *J* = 8.0, 3H), 6.67 (d, *J* = 7.5, 1H), 4.74 (dd, *J* = 9.9, 4.6, 1H), 3.76 (s, 3H), 3.54 (dd, *J* = 12.8, 4.6, 1H), 2.83 (dd, *J* = 12.8, 9.9, 1H), 2.21 (s, 3H), 1.53 – 1.20 (m, 9H), 1.16 (s, 3H), 1.02 (s, 3H), 0.61 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.7, 138.6, 137.2, 135.1, 129.2, 129.0, 127.7, 126.8, 126.4, 112.9, 87.9, 60.1, 55.1, 42.8, 40.5, 34.4, 21.3, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{36}O_2N$ $[M + H]^+$, 382.2741, found 382.2743.

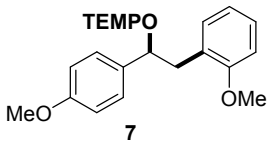



**6**

**1-(1-(4-methoxyphenyl)-2-(p-tolyl)ethoxy)-2,2,6,6-tetramethylpiperidine (6)**

Prepared according to the general procedure using 1-iodo-4-methylbenzene (65.4 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 16 hours; X = Br, 24 hours; X = Cl, 36 hours). The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **6** as a colorless oil (X = I, 99.5 mg, 87% yield; X = Br, 93.8 mg, 82% yield; X = Cl, 43.5 mg, 38% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.03 (d, *J* = 8.5, 2H), 6.92 (d, *J* = 7.8, 2H), 6.77 (d, *J* = 7.8, 2H), 6.73 (d, *J* = 8.5, 2H), 4.72 (dd, *J* = 10.2, 4.4, 1H), 3.76 (s, 3H), 3.54 (dd, *J* = 12.8, 4.4, 1H), 2.84 (dd, *J* = 12.8, 10.2, 1H), 2.24 (s, 3H), 1.56 – 1.23 (m, 9H), 1.16 (s, 3H), 1.02 (s, 3H), 0.62 (s, 3H);

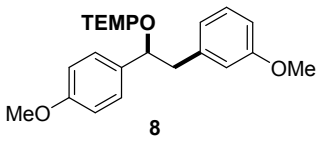
$^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.6, 135.5, 135.1, 135.0, 129.6, 129.3, 128.6, 112.9, 88.0, 60.1, 55.1, 42.3, 40.5, 34.4, 21.0, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{36}O_2N$ $[M + H]^+$, 382.2741, found 382.2745.




**7**

**1-(2-(2-methoxyphenyl)-1-(4-methoxyphenyl)ethoxy)-2,2,6,6-tetramethylpiperidine (7)**

Prepared according to the general procedure using 2-iodoanisole (70.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 16 hours; X = Br, 24 hours; X = Cl, 36 hours). The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **7** as a colorless oil (X = I, 98.9 mg, 83% yield; X = Br, 97.7 mg, 82% yield; X = Cl, 57.2 mg, 48% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.09 – 6.99 (m, 3H), 6.74 – 6.67 (m, 4H), 6.65 (t, *J* = 7.3, 1H), 4.85 (dd, *J* = 10.0, 5.0, 1H), 3.74 (s, 3H), 3.68 (s, 3H), 3.63 (dd, *J* = 12.6, 5.0, 1H), 2.84 (dd, *J* = 12.6, 10.0, 1H), 1.61 – 1.21 (m, 9H), 1.14 (s, 3H), 1.02 (s, 3H), 0.58 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.5, 157.7, 135.7, 131.6, 129.2, 127.1, 127.0, 120.0, 112.7, 110.0, 85.9, 59.3, 55.1, 55.0, 40.6, 37.3, 34.1, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{36}O_3N$ $[M + H]^+$, 398.2690, found 398.2689.

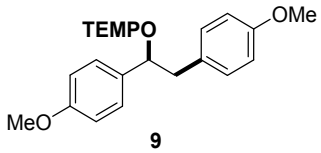



**8**

**1-(2-(3-methoxyphenyl)-1-(4-methoxyphenyl)ethoxy)-2,2,6,6-tetramethylpiperidine (8)**

Prepared according to the general procedure using 3-iodoanisole (70.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5

mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 16 hours; X = Br, 24 hours; X = Cl, 36 hours). The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **8** as a colorless oil (X = I, 103.7 mg, 87% yield; X = Br, 101.3 mg, 85% yield; X = Cl, 53.6 mg, 45% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.05 – 7.01 (m, 3H), 6.74 (d, *J* = 8.5, 2H), 6.63 (dd, *J* = 8.2, 2.4, 1H), 6.51 (d, *J* = 7.5, 1H), 6.42 (s, 1H), 4.75 (dd, *J* = 10.0, 4.5, 1H), 3.74 (s, 3H), 3.65 (s, 3H), 3.56 (dd, *J* = 12.8, 4.5, 1H), 2.86 (dd, *J* = 12.8, 10.0, 1H), 1.55 – 1.20 (m, 9H), 1.16 (s, 3H), 1.02 (s, 3H), 0.62 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 159.2, 158.7, 140.3, 135.0, 129.2, 128.8, 122.2, 115.3, 113.0, 111.5, 87.8, 60.1, 59.6, 55.1, 55.0, 42.9, 40.5, 34.4, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{36}O_3N$ $[M + H]^+$, 398.2690, found 398.2691.

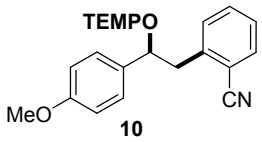



**9**

**1-(2-(3-methoxyphenyl)-1-(4-methoxyphenyl)ethoxy)-2,2,6,6-tetramethylpiperidine (9)**

Prepared according to the general procedure using 4-iodoanisole (70.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 16 hours; X = Br, 24 hours; X = Cl, 36 hours). The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **9** as a colorless oil (X = I, 102.5 mg, 86% yield; X = Br, 97.7 mg, 82% yield; X = Cl, 65.5 mg, 55% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.01 (d, *J* = 8.3, 2H), 6.79 (d, *J* = 8.3, 2H), 6.73 (d, *J* = 8.3, 2H), 6.66 (d, *J* = 8.3, 2H), 4.70 (dd, *J* = 10.0, 4.4, 1H), 3.75 (s, 3H), 3.72 (s, 3H), 3.51 (dd, *J* = 12.8, 4.4, 1H), 2.82 (dd, *J* = 12.8, 10.0, 1H), 1.54 – 1.20 (m, 9H), 1.15 (s, 3H), 1.02 (s, 3H), 0.64 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.6, 157.7, 135.1, 130.8, 130.6, 129.2, 113.3, 112.9, 88.1, 60.1, 59.5, 55.1, 41.9, 40.5, 34.4, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{36}O_3N$ $[M + H]^+$,

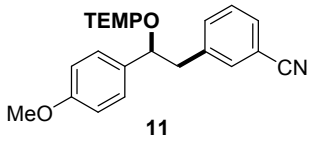
398.2690, found 398.2693.




**10**

**2-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)benzonitrile (10)**

Prepared according to the general procedure using 2-iodobenzontirlle (68.7 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 12 hours; X = Br, 16 hours; X = Cl, 24 hours). The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:120) to afford the product **10** as a colorless oil (X = I, 102.4 mg, 87% yield; X = Br, 98.8 mg, 84% yield; X = Cl, 90.6 mg, 77% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.48 (d, *J* = 7.7, 1H), 7.35 (t, *J* = 7.7, 1H), 7.18 (t, *J* = 7.6, 1H), 7.10 –7.06 (m, 3H), 6.74 (d, *J* = 8.0, 2H), 5.00 – 4.82 (m, 1H), 3.82 (dd, *J* = 13.2, 6.1, 1H), 3.76 (s, 3H), 3.14 (dd, *J* = 13.2, 9.0, 1H), 1.60 – 1.20 (m, 9H), 1.09 (s, 3H), 1.03 (s, 3H), 0.61 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 159.0, 142.9, 132.6, 132.1, 131.0, 129.3, 126.4, 118.1, 113.4, 113.1, 86.2, 60.1, 59.7, 55.1, 40.9, 40.5, 34.6, 34.2, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{33}O_2N_2$ $[M + H]^+$, 393.2537, found 393.2539.

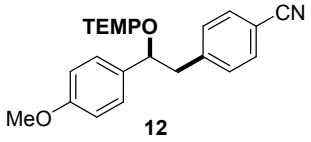



**11**

**3-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)benzonitrile (11)**

Prepared according to the general procedure using 3-iodobenzontirlle (68.7 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 12 hours; X = Br, 16 hours; X = Cl, 24 hours). The crude mixture

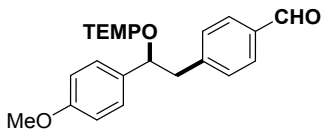
was purified by silica gel chromatography (acetone/petroleum ether 1:120) to afford the product **11** as a colorless oil (X = I, 96.5 mg, 82% yield; X = Br, 100.0 mg, 85% yield; X = Cl, 88.3 mg, 75% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.39 (d, *J* = 7.7, 1H), 7.24 – 7.16 (m, 2H), 7.10 (d, *J* = 7.8, 1H), 6.98 (d, *J* = 8.6, 2H), 6.75 (d, *J* = 8.6, 2H), 4.73 (dd, *J* = 9.7, 4.8, 1H), 3.76 (s, 3H), 3.59 (dd, *J* = 12.9, 4.8, 1H), 2.92 (dd, *J* = 12.9, 9.7, 1H), 1.60 – 1.22 (m, 9H), 1.15 (s, 3H), 1.02 (s, 3H), 0.63 (s, 3H); $^{13}$C NMR (151 MHz, $CDCl_3$): δ 159.0, 140.3, 134.3, 133.9, 133.2, 129.6, 129.0, 128.6, 119.0, 113.2, 111.9, 87.3, 59.8, 55.1, 42.4, 40.5, 34.4, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{33}O_2N_2$ $[M + H]^+$, 393.2537, found 393.2537.



**12**

**4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)benzonitrile (12)**

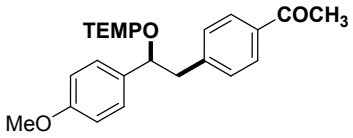
Prepared according to the general procedure using 4-iodobenzontirlle (68.7 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: (X = I, 12 hours; X = Br, 16 hours; X = Cl, 24 hours). The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:120) to afford the product **12** as a colorless oil (X = I, 100.0 mg, 85% yield; X = Br, 97.7 mg, 83% yield; X = Cl, 88.2 mg, 75% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.41 (d, *J* = 8.2, 2H), 7.01 – 6.92 (m, 4H), 6.74 (t, *J* = 5.7, 2H), 4.74 (dd, *J* = 9.8, 4.7, 1H), 3.77 (s, 3H), 3.63 (dd, *J* = 12.7, 4.7, 1H), 2.95 (dd, *J* = 12.7, 9.8, 1H), 1.56 – 1.20 (m, 9H), 1.15 (s, 3H), 1.02 (s, 3H), 0.64 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.9, 144.5, 134.0, 131.7, 130.5, 129.0, 119.1, 113.2, 109.7, 87.3, 59.8, 55.1, 43.1, 40.5, 34.5, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{33}O_2N_2$ $[M + H]^+$, 393.2537, found 393.2539.




13

**4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)benzaldehyde (13)**

Prepared according to the general procedure using 4-chlorobenzaldehyde (42.0 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **13** as a colorless oil (97.2 mg, 82% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 9.90 (s, 1H), 7.64 (d, *J* = 7.6, 2H), 7.06 (d, *J* = 7.6, 2H), 7.00 (d, *J* = 7.6, 2H), 6.73 (d, *J* = 7.6, 2H), 4.78 (dd, *J* = 9.7, 4.5, 1H), 3.75 (s, 3H), 3.67 (dd, *J* = 12.6, 4.5, 1H), 3.01 – 2.92 (m, 1H), 1.65 – 1.23 (m, 9H), 1.16 (s, 3H), 1.03 (s, 3H), 0.65 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 192.0, 158.9, 146.3, 134.4, 134.3, 130.4, 129.4, 129.0, 113.1, 87.4, 60.1, 59.8, 55.1, 43.2, 40.5, 34.4, 20.5, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{34}O_3N$ $[M + H]^+$, 396.2533, found 396.2532.

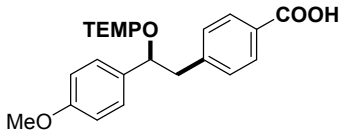



14

**1-(4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)phenyl)ethan-1-one (14)**

Prepared according to the general procedure using 1-(4-chlorophenyl)ethan-1-one (46.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **14** as a colorless oil (108.1 mg, 88% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.72 (d, *J* = 8.2, 2H), 7.02 –

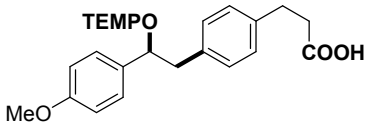
6.97(m, 4H), 6.73 (d, *J* = 8.6, 2H), 4.77 (dd, *J* = 9.9, 4.6, 1H), 3.76 (s, 3H), 3.64 (dd, *J* = 12.7, 4.6, 1H), 2.95 (dd, *J* = 12.7, 9.9, 1H), 2.52 (s, 3H), 1.51 – 1.20 (m, 9H), 1.16 (s, 3H), 1.03 (s, 3H), 0.64 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 197.8, 158.8, 144.6, 135.0, 134.4, 129.9, 129.1, 128.0, 113.1, 87.5, 60.1, 59.7, 55.1, 42.9, 40.5, 34.4, 26.4, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{26}H_{36}O_3N$ [M + H]$^+$, 410.2690, found 410.2693.




**15**

**4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)benzoic acid (15)**

Prepared according to the general procedure using 4-iodobenzoic acid (74.4 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (293.2 mg, 0.90 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:5) to afford the product **15** as a colorless oil (98.7 mg, 80% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.87 (d, *J* = 8.2, 2H), 7.06 – 6.91 (m, 4H), 6.73 (d, *J* = 8.6, 2H), 4.79 (dd, *J* = 9.9, 4.6, 1H), 3.76 (s, 3H), 3.66 (dd, *J* = 12.7, 4.6, 1H), 2.96 (dd, *J* = 12.7, 9.9, 1H), 1.62 – 1.23 (m, 9H), 1.15 (s, 3H), 1.00 (s, 3H), 0.64 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 172.0, 158.8, 145.4, 134.3, 129.9, 129.0, 126.9, 113.1, 87.5, 59.8, 55.1, 43.1, 40.5, 34.5, 20.5, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{34}O_4N$ [M + H]$^+$, 412.2482, found 412.2482.

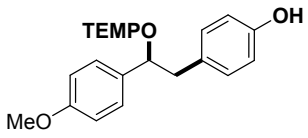



**16**

**3-(4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-**

**yl)oxy)ethyl)phenyl)propanoic acid (16)**

Prepared according to the general procedure using 3-(4-iodophenyl)propanoic acid (77.4 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (293.2 mg, 0.90 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:5) to afford the product **16** as a colorless oil (108.0 mg, 82% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.02 (d, *J* = 8.6, 2H), 6.96 (d, *J* = 7.9, 2H), 6.82 (d, *J* = 7.9, 2H), 6.73 (d, *J* = 8.6, 2H), 4.73 (dd, *J* = 9.9, 4.6, 1H), 3.76 (s, 3H), 3.54 (dd, *J* = 12.9, 4.6, 1H), 2.95 – 2.75 (m, 3H), 2.60 (t, *J* = 7.9, 2H), 1.60 – 1.22 (m, 9H), 1.15 (s, 3H), 1.01 (s, 3H), 0.61 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 178.5, 158.6, 137.5, 136.7, 135.0, 129.9, 129.2, 127.7, 112.9, 87.8, 60.1, 55.1, 42.4, 40.5, 35.6, 34.3, 30.2, 20.5, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{27}H_{38}O_4N$ $[M + H]^+$, 440.2795, found 440.2798.

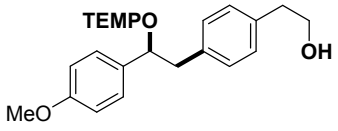



**17**

**4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)phenol (17)**

Prepared according to the general procedure using 4-iodophenol (66.0 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (293.2 mg, 0.90 mmol), and DMSO (1.5 mL). Time of irradiation: 20 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:30) to afford the product **17** as a colorless oil (88.5 mg, 77% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.01 (d, *J* = 8.6, 2H), 6.75 – 6.69 (m, 4H), 6.58 (d, *J* = 8.1, 2H), 4.69 (dd, *J* = 10.0, 4.5, 1H), 3.75 (s, 3H), 3.50 (dd, *J* = 13.0, 4.5, 1H), 2.80 (dd, *J* = 13.0, 10.0, 1H), 1.63 – 1.23 (m, 9H), 1.16 (s, 3H), 1.02 (s, 3H), 0.61 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.6, 153.6, 135.2, 130.9, 130.8, 129.2, 114.9, 113.0, 88.1, 60.1, 55.1, 42.0, 40.5, 34.4, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for

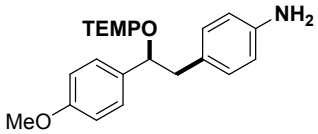
$C_{24}H_{34}O_3N$ $[M + H]^+$, 384.2533, found 384.2537.




**18**

**2-(4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)phenyl)ethan-1-ol (18)**

Prepared according to the general procedure using 2-(4-iodophenyl)ethan-1-ol (74.4 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:30) to afford the product **18** as a colorless oil (103.6 mg, 84% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.03 (d, *J* = 8.6, 2H), 6.98 (d, *J* = 7.9, 2H), 6.85 (d, *J* = 7.9, 2H), 6.73 (d, *J* = 8.6, 2H), 4.73 (dd, *J* = 9.9, 4.6, 1H), 3.82 – 3.67 (m, 5H), 3.55 (dd, *J* = 12.9, 4.6, 1H), 2.87 (dd, *J* = 12.9, 9.9, 1H), 2.77 (t, *J* = 6.6, 2H), 1.65 – 1.22 (m, 9H), 1.17 (s, 3H), 1.03 (s, 3H), 0.60 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.7, 136.9, 135.6, 135.0, 130.0, 129.5, 128.5, 112.9, 87.8, 63.7, 59.5, 55.1, 42.3, 40.5, 38.8, 34.4, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{26}H_{38}O_3N$ $[M + H]^+$, 412.2846, found 412.2846.

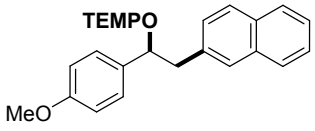



**19**

**4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)aniline (19)**

Prepared according to the general procedure using 4-iodoaniline (65.7 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of

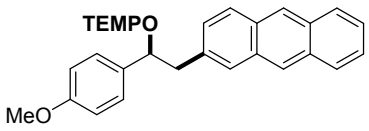
irradiation: 20 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:30) to afford the product **19** as a colorless oil (74.5 mg, 65% yield). $^{1}$H NMR (600 MHz, $CDCl_3$): δ 7.02 (d, *J* = 8.5, 2H), 6.73 (d, *J* = 8.5, 2H), 6.66 (d, *J* = 8.1, 2H), 6.45 (d, *J* = 8.1, 2H), 4.67 (dd, *J* = 10.0, 4.3, 1H), 3.74 (s, 3H), 3.49 – 3.40 (m, 3H), 2.76 (dd, *J* = 12.9, 10.0, 1H), 1.57 – 1.20 (m, 9H), 1.17 (s, 3H), 1.02 (s, 3H), 0.60 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.6, 144.1, 135.3, 130.5, 129.3, 128.8, 114.9, 112.9, 88.3, 60.1, 59.5, 55.1, 42.0, 40.6, 34.4, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{24}H_{35}O_2N_2$ $[M + H]^+$, 383.2693, found 383.2693.




**20**

**1-(1-(4-methoxyphenyl)-2-(naphthalen-2-yl)ethoxy)-2,2,6,6-tetramethylpiperidine (20)**

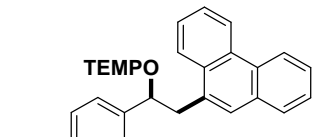
Prepared according to the general procedure using 2-bromonaphthalene (61.8 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 24 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **20** as a colorless oil (102.6 mg, 82% yield). $^{1}$H NMR (600 MHz, $CDCl_3$): δ 7.72 (d, *J* = 7.4, 1H), 7.64 (d, *J* = 7.7, 1H), 7.59 (d, *J* = 8.4, 1H), 7.40 – 7.29 (m, 3H), 7.06 – 6.97 (m, 3H), 6.70 (d, *J* = 8.4, 2H), 4.85 (dd, *J* = 10.0, 4.4, 1H), 3.82 – 3.66 (m, 4H), 3.12 – 2.96 (m, 1H), 1.63 – 1.24 (m, 9H), 1.18 (s, 3H), 1.05 (s, 3H), 0.65 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.7, 136.4, 134.9, 133.4, 132.0, 128.4, 128.1, 127.5, 127.3, 125.6, 125.0, 113.0, 87.9, 60.1, 59.7, 55.1, 43.0, 40.6, 34.6, 20.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{28}H_{36}O_2N$ $[M + H]^+$, 418.2741, found 418.2744.




21

**1-(2-(anthracen-2-yl)-1-(4-methoxyphenyl)ethoxy)-2,2,6,6-tetramethylpiperidine (21)**

Prepared according to the general procedure using 2-bromoanthracene (76.8 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 24 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **21** as a colorless oil (91.1 mg, 65% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.31 (s, 1H), 8.22 (s, 1H), 7.97 – 7.93 (m, 2H), 7.77 (d, $J$ = 8.7, 1H), 7.49 (s, 1H), 7.44 – 7.38 (m, 2H), 7.06 – 7.03 (m, 3H), 6.71 (d, $J$ = 8.5, 2H), 4.90 (dd, $J$ = 9.9, 4.4, 1H), 3.83 – 3.72 (m, 4H), 3.07 (dd, $J$ = 12.6, 9.9, 1H), 1.67 – 1.22 (m, 9H), 1.19 (s, 3H), 1.06 (s, 3H), 0.67 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.7, 135.8, 135.0, 131.8, 131.7, 131.3, 130.5, 129.2, 128.4, 128.1, 128.0, 127.8, 127.5, 125.7, 125.5, 125.1, 124.9, 113.0, 87.7, 59.7, 55.1, 43.2, 40.5, 34.5, 20.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{32}H_{38}O_2N$ $[M + H]^+$, 468.2897, found 468.2899.

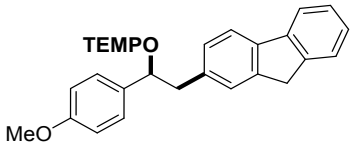



22

**1-(1-(4-methoxyphenyl)-2-(phenanthren-9-yl)ethoxy)-2,2,6,6-tetramethylpiperidine (22)**

Prepared according to the general procedure using 9-iodophenanthrene (91.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **22** as a colorless oil (123.4 mg,

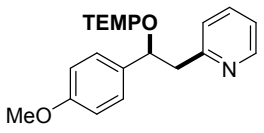
88% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.72 – 8.65 (m, 1H), 8.60 (d, *J* = 8.2, 1H), 8.20 (d, *J* = 7.7, 1H), 7.65 – 7.58 (m, 3H), 7.54 (t, *J* = 7.5, 1H), 7.48 (t, *J* = 7.3, 1H), 7.22 (s, 1H), 7.04 (d, *J* = 8.5, 2H), 6.68 (d, *J* = 8.5, 2H), 5.10 (dd, *J* = 8.8, 5.6, 1H), 4.18 (dd, *J* = 13.5, 5.6, 1H), 3.69 (s, 3H), 3.25 (dd, *J* = 13.5, 8.8, 1H), 1.59 – 1.30 (m, 6H), 1.27 (s, 3H), 1.08 (s, 3H), 1.06 (s, 3H), 0.70 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.7, 135.2, 133.2, 131.7, 130.5, 129.6, 129.0, 128.7, 128.1, 126.3, 125.9, 125.8, 124.8, 123.0, 122.3, 113.0, 85.7, 59.9, 55.1, 40.6, 40.3, 34.6, 34.3, 20.6, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{32}H_{38}O_2N$ $[M + H]^+$, 468.2897, found 468.2898.




**23**

**1-(2-(9H-fluoren-2-yl)-1-(4-methoxyphenyl)ethoxy)-2,2,6,6-tetramethylpiperidine (23)**

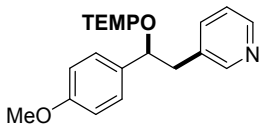
Prepared according to the general procedure using 2-bromo-9*H*-fluorene (73.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 36 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **23** as a colorless oil (75.1 mg, 55% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.69 (d, *J* = 7.5, 1H), 7.53 (d, *J* = 7.8, 1H), 7.48 (d, *J* = 7.4, 1H), 7.32 (t, *J* = 7.4, 1H), 7.25 (s, 1H), 7.07 (s, 1H), 7.02 (d, *J* = 8.6, 2H), 6.89 (d, *J* = 7.7, 1H), 6.72 (d, *J* = 8.6, 2H), 4.79 (dd, *J* = 9.9, 4.7, 1H), 3.85 – 3.60 (m, 6H), 2.93 (dd, *J* = 12.8, 9.9, 1H), 1.61 – 1.27 (m, 9H), 1.18 (s, 3H), 1.04 (s, 3H), 0.64 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.6, 143.2, 143.0, 141.8, 139.4, 137.5, 135.1, 129.2, 128.3, 126.6, 126.4, 126.2, 124.9, 119.6, 119.3, 112.9, 88.1, 60.4, 55.1, 43.1, 40.5, 36.8, 34.5, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{31}H_{38}O_2N$ $[M + H]^+$, 456.2897, found 456.2897.




**24**

**2-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)pyridine (24)**

Prepared according to the general procedure using 2-iodopyridine (61.5 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 12 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **24** as a colorless oil (81.8 mg, 74% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.46 (dd, *J* = 4.8, 0.8, 1H), 7.39 (td, *J* = 7.6, 1.8, 1H), 7.07 (d, *J* = 8.6, 2H), 7.03 – 6.97 (m, 1H), 6.78 (d, *J* = 7.8, 1H), 6.76 – 6.65 (m, 2H), 5.04 (dd, *J* = 9.4, 5.5, 1H), 3.86 – 3.69 (m, 4H), 3.03 (dd, *J* = 12.6, 9.4, 1H), 1.59 – 1.23 (m, 9H), 1.09 (s, 3H), 1.01 (s, 3H), 0.68 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 159.2, 158.7, 149.0, 135.6, 134.8, 129.1, 124.4, 120.8, 113.0, 86.0, 60.0, 55.1, 45.2, 40.4, 34.2, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{23}H_{33}O_2N_2$ $[M + H]^+$, 369.2537, found 369.2539.

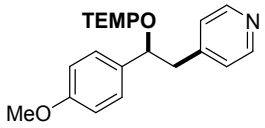



**25**

**3-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)pyridine (25)**

Prepared according to the general procedure using 3-iodopyridine (61.5 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 12 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **25** as a colorless oil (91.7 mg, 83% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.34 (dd, *J* = 4.8, 1.5, 1H), 8.18 (d, *J* = 1.8, 1H),

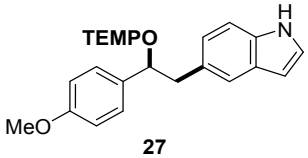
7.14 (dd, *J* = 7.8, 1.7, 1H), 7.08 – 6.91 (m, 3H), 6.74 (d, *J* = 8.6, 2H), 4.75 (dd, *J* = 9.7, 4.7, 1H), 3.76 (s, 3H), 3.55 (dd, *J* = 13.0, 4.7, 1H), 2.91 (dd, *J* = 13.0, 9.7, 1H), 1.64 – 1.23 (m, 9H), 1.16 (s, 3H), 1.02 (s, 3H), 0.63 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.9, 151.0, 147.3, 137.1, 134.1, 129.1, 122.8, 113.2, 87.4, 60.1, 59.7, 55.1, 40.5, 40.0, 34.4, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{23}H_{33}O_2N_2$ $[M + H]^+$, 369.2537, found 369.2536.




**26**

**4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)pyridine (26)**

Prepared according to the general procedure using 4-iodopyridine (61.5 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 12 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **26** as a colorless oil (60.8 mg, 55% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.34 (d, *J* = 5.9, 2H), 7.01 (d, *J* = 8.6, 2H), 6.82 (d, *J* = 5.9, 2H), 6.74 (d, *J* = 8.6, 2H), 4.76 (dd, *J* = 9.9, 4.6, 1H), 3.78 (s, 3H), 3.57 (dd, *J* = 12.6, 4.6, 1H), 2.89 (dd, *J* = 12.6, 9.9, 1H), 1.64 – 1.23 (m, 9H), 1.16 (s, 3H), 1.02 (s, 3H), 0.65 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.9, 149.3, 147.8, 134.0, 129.0, 125.1, 113.1, 87.0, 59.8, 55.1, 42.3, 40.5, 34.4, 20.5, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{23}H_{33}O_2N_2$ $[M + H]^+$, 369.2537, found 369.2540.

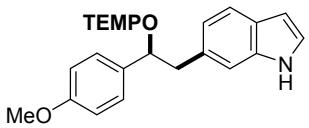



**27**

**5-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-1H-indole (27)**

Prepared according to the general procedure using 5-iodo-1H-indole (72.9 mg, 0.30

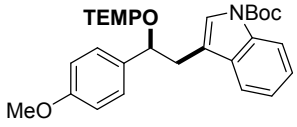
mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 20 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:100) to afford the product **27** as a colorless oil (98.7 mg, 81% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.91 (s, 1H), 7.18 (s, 1H), 7.09 (d, *J* = 8.3, 1H), 7.08 – 6.95 (m, 3H), 6.71 – 6.69 (m, 3H), 6.38 (s, 1H), 4.80 (dd, *J* = 10.3, 4.3, 1H), 3.75 – 3.64 (m, 4H), 2.96 (dd, *J* = 12.8, 10.3, 1H), 1.62 – 1.24 (m, 9H), 1.21 (s, 3H), 1.07 (s, 3H), 0.63 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.5, 135.5, 134.4, 130.0, 129.3, 127.9, 124.3, 123.9, 121.4, 112.9, 110.3, 102.3, 88.6, 60.1, 59.5, 55.1, 42.9, 40.6, 34.5, 20.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{26}H_{35}O_2N_2$ $[M + H]^+$, 407.2693, found 407.2691.




**28**

**6-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-1H-indole (28)**

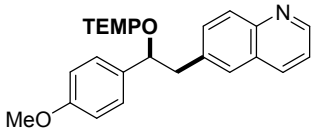
Prepared according to the general procedure using 6-iodo-1H-indole (72.9 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:100) to afford the product **28** as a colorless oil (88.9 mg, 73% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.85 (s, 1H), 7.40 (d, *J* = 8.1, 1H), 7.06 – 6.98 (m, 3H), 6.79 (s, 1H), 6.74 – 6.70 (m, 3H), 6.42 (s, 1H), 4.80 (dd, *J* = 10.3, 4.2, 1H), 3.75 – 3.67 (m, 4H), 2.96 (dd, *J* = 12.7, 10.3, 1H), 1.54 – 1.23 (m, 9H), 1.19 (s, 3H), 1.04 (s, 3H), 0.64 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.6, 136.0, 135.4, 132.6, 129.3, 125.9, 123.5, 122.1, 120.0, 112.9, 112.0, 102.2, 88.4, 59.6, 55.1, 43.2, 40.6, 34.5, 20.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{26}H_{35}O_2N_2$ $[M + H]^+$, 407.2693, found 407.2696.




**29**

***Tert*-butyl3-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-1H-indole-1-carboxylate (29)**

Prepared according to the general procedure using *tert*-butyl 3-iodo-1H-indole-1-carboxylate (102.9 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:100) to afford the product **29** as a colorless oil (60.8 mg, 40% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.03 (br, 1H), 7.36 (d, *J* = 7.7, 1H), 7.25 – 7.22 (m, 1H), 7.19 – 7.08 (m, 3H), 6.96 (s, 1H), 6.74 (d, *J* = 8.2, 2H), 4.90 (dd, *J* = 9.9, 3.8, 1H), 3.73 (s, 3H), 3.61 (dd, *J* = 13.8, 3.8, 1H), 3.03 (dd, *J* = 13.8, 9.9, 1H), 1.60 (s, 9H), 1.57 – 1.25 (m, 9H), 1.19 (s, 3H), 1.04 (s, 3H), 0.64 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.9, 149.7, 135.4, 131.2, 129.1, 124.1, 124.0, 122.2, 119.0, 117.2, 115.0, 113.2, 86.0, 83.1, 59.7, 55.2, 40.6, 34.5, 31.7, 28.2, 20.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{31}H_{43}O_4N_2$ $[M + H]^+$, 507.3217, found 507.3222.

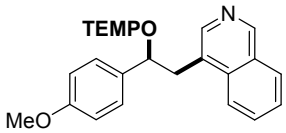



**30**

**6-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)quinoline (30)**

Prepared according to the general procedure using 6-iodoquinoline (76.5 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **30** as a colorless oil (82.8 mg, 66%

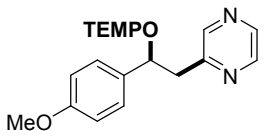
yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.81 (dd, *J* = 4.1, 1.4, 1H), 7.95 (d, *J* = 8.0, 1H), 7.91 – 7.83 (m, 1H), 7.31 – 7.27 (m, 3H), 7.01 (d, *J* = 8.6, 2H), 6.71 (d, *J* = 8.6, 2H), 4.86 (dd, *J* = 9.9, 4.6, 1H), 3.83 – 3.66 (m, 4H), 3.07 (dd, *J* = 12.8, 9.6, 1H), 1.67 – 1.22 (m, 9H), 1.18 (s, 3H), 1.05 (s, 3H), 0.66 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.8, 149.7, 147.1, 137.3, 135.6, 134.6, 132.0, 129.1, 128.8, 128.1, 127.9, 120.9, 113.1, 87.7, 59.7, 55.1, 42.9, 40.5, 34.5, 20.5, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{27}H_{35}O_2N_2$ $[M + H]^+$, 419.2693, found 419.2696.




**31**

**4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)isoquinoline (31)**

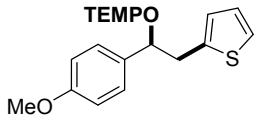
Prepared according to the general procedure using 4-bromoisoquinoline (62.1 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **31** as a colorless oil (114.2 mg, 91% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 9.03 (s, 1H), 8.15 – 7.95 (m, 2H), 7.91 (d, *J* = 8.1, 1H), 7.69 – 7.60 (m, 1H), 7.60 – 7.49 (m, 1H), 6.99 (d, *J* = 8.6, 2H), 6.69 (t, *J* = 8.6, 2H), 4.99 (dd, *J* = 9.0, 5.8, 1H), 4.06 (dd, *J* = 13.4, 5.8, 1H), 3.72 (s, 3H), 3.18 (dd, *J* = 13.4, 9.0, 1H), 1.65 – 1.33 (m, 6H), 1.27 (s, 3H), 1.06 (s, 3H), 1.03 (s, 3H), 0.66 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 159.0, 151.0, 144.6, 135.2, 134.4, 129.9, 128.8, 128.2, 128.1, 126.6, 123.2, 113.1, 86.0, 60.1, 59.8, 55.1, 40.5, 36.8, 34.6, 34.3, 20.5, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{27}H_{35}O_2N_2$ $[M + H]^+$, 419.2693, found 419.2693.




32

**2-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)pyrazine (32)**

Prepared according to the general procedure using 2-iodopyrazine (61.8 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **32** as a colorless oil (59.8 mg, 54% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.42 (d, *J* = 2.2, 1H), 8.28 (d, *J* = 2.2, 1H), 8.07 (s, 1H), 7.06 (d, *J* = 8.5, 2H), 6.74 (d, *J* = 8.5, 2H), 5.04 (dd, *J* = 9.2, 5.5, 1H), 3.83 – 3.64 (m, 4H), 3.10 (dd, *J* = 12.8, 9.2, 1H), 1.59 – 1.19 (m, 9H), 1.09 (s, 3H), 1.02 (s, 3H), 0.65 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.9, 155.0, 145.8, 143.8, 141.9, 134.0, 129.0, 113.3, 85.6, 60.0, 55.1, 42.4, 40.4, 34.2, 20.3, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{22}H_{32}O_2N_3$ $[M + H]^+$, 370.2489, found 370.2493.

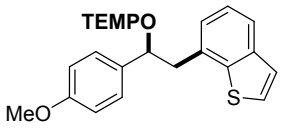



33

**1-(1-(4-methoxyphenyl)-2-(thiophen-2-yl)ethoxy)-2,2,6,6-tetramethylpiperidine (33)**

Prepared according to the general procedure using 2-iodothiophene (63.0 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **33** as a colorless oil (69.4 mg, 62% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.09 (d, *J* = 8.6, 2H), 7.01 (dd, *J* = 5.1, 1.0,

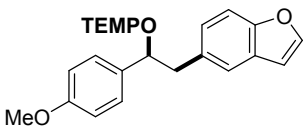
1H), 6.79 – 6.73 (m, 3H), 6.48 (d, $J$ =3.3, 1H), 4.81 (dd, $J$ = 9.8, 4.0, 1H), 3.77 (s, 3H), 3.70 (dd, $J$ = 14.2, 4.0, 1H), 3.22 (dd, $J$ = 14.2, 9.8, 1H), 1.53 – 1.12 (m, 12H), 1.03 (s, 3H), 0.66 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.8, 140.7, 134.7, 129.1, 126.3, 125.9, 123.5, 113.1, 87.3, 60.1, 55.1, 40.5, 36.7, 34.4, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{22}H_{32}O_2NS$ $[M + H]^+$, 374.2148, found 374.2149.




**34**

**1-(2-(benzo[b]thiophen-7-yl)-1-(4-methoxyphenyl)ethoxy)-2,2,6,6-tetramethylpiperidine (34)**

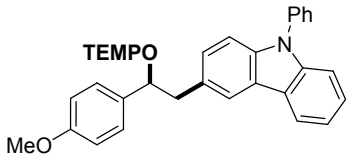
Prepared according to the general procedure using 7-bromobenzo[b]thiophene (63.6 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 36 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **34** as a colorless oil (82.6 mg, 65% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.58 (d, $J$ = 7.9, 1H), 7.36 (d, $J$ = 5.4, 1H), 7.28 (d, $J$ = 5.4, 1H), 7.12 (t, $J$ = 7.6, 1H), 7.05 (d, $J$ = 8.5, 2H), 6.79 (d, $J$ = 7.2, 1H), 6.70 (d, $J$ = 8.5, 2H), 5.07 (dd, $J$ = 9.6, 5.4, 1H), 3.88 (dd, $J$ = 13.3, 5.4, 1H), 3.73 (s, 3H), 3.14 (dd, $J$ = 13.3, 9.6, 1H), 1.53 – 1.18 (m, 9H), 1.10 (s, 3H), 1.04 (s, 3H), 0.63 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.8, 140.4, 139.5, 135.1, 133.0, 129.1, 125.6, 125.5, 124.3, 124.1, 121.4, 113.0, 85.2, 59.6, 55.1, 42.0, 40.5, 34.6, 34.4, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{26}H_{34}O_2NS$ $[M + H]^+$, 424.2305, found 424.2308.




**35**

**1-(2-(benzofuran-5-yl)-1-(4-methoxyphenyl)ethoxy)-2,2,6,6-tetramethylpiperidine (35)**

Prepared according to the general procedure using 5-bromobenzofuran (63.6 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 36 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **35** as a colorless oil (78.2 mg, 64% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.52 (d, *J* = 2.0, 1H), 7.24 (d, *J* = 8.4, 1H), 7.11 (s, 1H), 7.01 (d, *J* = 8.5, 2H), 6.81 (d, *J* = 8.4, 1H), 6.72 (d, *J* = 8.5, 2H), 6.61 (d, *J* = 1.2, 1H), 4.78 (dd, *J* = 10.0, 4.5, 1H), 3.75 (s, 3H), 3.68 (dd, *J* = 13.0, 4.5, 1H), 2.95 (dd, *J* = 13.0, 10.0, 1H), 1.69 – 1.22 (m, 9H), 1.17 (s, 3H), 1.03 (s, 3H), 0.63 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.6, 153.6, 144.7, 135.1, 133.1, 129.2, 127.1, 126.2, 121.9, 112.9, 110.5, 106.4, 88.3, 60.1, 55.1, 42.7, 40.5, 34.5, 20.4, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{26}H_{34}O_3N$ $[M + H]^+$, 408.2533, found 408.2536.

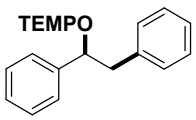



**36**

**3-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-9-phenyl-9H-carbazole (36)**

Prepared according to the general procedure using 3-iodo-9-phenyl-9H-carbazole (110.7 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 36 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:100) to afford the product **36** as a colorless oil (131.0 mg, 83% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.00 (d, *J* = 7.7, 1H), 7.67 (s, 1H), 7.58 – 7.45 (m, 4H), 7.38 – 7.28 (m, 3H), 7.24 – 7.18 (m, 1H), 7.15 (d, *J* = 8.4, 1H), 7.08 – 7.02 (m, 2H), 6.89 (d, *J* = 8.4, 1H), 6.71 (d, *J* = 8.4, 2H), 4.86 (dd, *J* = 9.9, 2.6, 1H), 3.77 (dd, *J* = 13.0, 2.6, 1H), 3.69 (s, 3H), 3.07 (t, *J* = 13.0, 9.9,

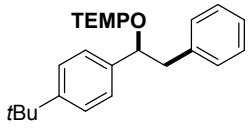
1H), 1.63 – 1.27 (m, 9H), 1.22 (s, 3H), 1.08 (s, 3H), 0.66 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.7, 141.0, 139.5, 138.0, 135.3, 130.3, 129.8, 129.4, 128.1, 127.2, 127.0, 125.7, 123.4, 123.2, 121.1, 120.2, 119.7, 113.0, 109.7, 109.1, 88.5, 60.1, 59.6, 55.1, 42.9, 40.6, 34.6, 20.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{36}H_{41}O_2N_2$ $[M + H]^+$, 533.3163, found 533.3167.




37

**1-(1,2-diphenylethoxy)-2,2,6,6-tetramethylpiperidine (37)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), styrene (46.8 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **37** as a colorless oil (83.9 mg, 83% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.20 – 7.13 (m, 3H), 7.13 – 7.03 (m, 5H), 6.87 (d, *J* = 7.4, 2H), 4.79 (dd, *J* = 10.0, 4.5, 1H), 3.62 (dd, *J* = 12.8, 4.5, 1H), 2.89 (dd, *J* = 12.8, 10.0, 1H), 1.60 – 1.25 (m, 9H), 1.20 (s, 3H), 1.06 (s, 3H), 0.61 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$); δ 142.7, 138.5, 129.7, 128.7, 128.1, 127.9, 127.5, 127.0, 125.8, 88.6, 60.1, 43.0, 40.6, 34.5, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{23}H_{32}ON$ $[M + H]^+$, 338.2478, found 338.2481.

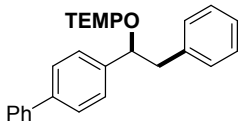



38

**1-(1-(4-(tert-butyl)phenyl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidine (38)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 1-(*tert*-butyl)-4-vinylbenzene (72.1 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel

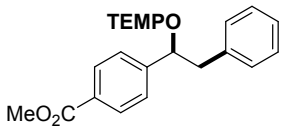
chromatography (acetone/petroleum ether 1:200) to afford the product **38** as a colorless oil (102.7 mg, 87% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.20 (d, *J* = 8.2, 2H), 7.15 – 7.05 (m, 3H), 7.03 (d, *J* = 8.3, 2H), 6.90 (d, *J* = 6.8, 2H), 4.79 (dd, *J* = 9.6, 4.8, 1H), 3.61 (dd, *J* = 12.8, 4.8, 1H), 2.87 (dd, *J* = 12.8, 9.6, 1H), 1.56 – 1.18 (m, 19H), 1.13 (s, 3H), 1.02 (s, 3H), 0.63 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 149.9, 139.5, 138.9, 129.8, 127.8, 127.7, 125.6, 124.3, 87.9, 59.7, 42.7, 40.5, 34.4, 31.4, 20.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{27}H_{40}ON$ $[M + H]^+$, 394.3104, found 394.3107.




**39**

**1-(1-([1,1'-biphenyl]-4-yl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidine (39)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-vinyl-1,1'-biphenyl (81.0 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **39** as a colorless oil (112.8 mg, 91% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.58 (d, *J* = 7.5, 2H), 7.44 (d, *J* = 8.1, 2H), 7.40 (t, *J* = 7.7, 2H), 7.30 (t, *J* = 7.4, 1H), 7.19 – 7.04 (m, 5H), 6.92 (d, *J* = 6.9, 2H), 4.85 (dd, *J* = 10.0, 4.6, 1H), 3.65 (dd, *J* = 12.8, 4.6, 1H), 2.92 (dd, *J* = 12.8, 10.0, 1H), 1.67 – 1.24 (m, 9H), 1.15 (s, 3H), 1.05 (s, sH), 0.65 (s, 1H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 141.8, 141.0, 139.6, 138.5, 129.8, 128.7, 128.5, 127.9, 127.1, 127.0, 126.2, 125.8, 88.3, 59.7, 43.0, 40.6, 34.5, 20.5, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{29}H_{36}ON$ $[M + H]^+$, 414.2791, found 414.2789.

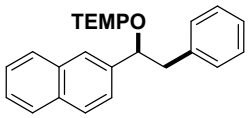



**40**

**Methyl 4-(2-phenyl-1-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)benzoate (40)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30

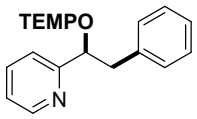
mmol), TEMPOH (56.6 mg, 0.36 mmol), methyl 4-vinylbenzoate (72.9 mg, 1.50 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **40** as a colorless oil (84.2 mg, 71% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.2, 2H), 7.18 – 6.99 (m, 5H), 6.92 – 6.76 (m, 2H), 4.85 (dd, *J* = 10.2, 4.5, 1H), 3.88 (s, 3H), 3.63 (dd, *J* = 12.9, 4.5, 1H), 2.88 (dd, *J* = 12.9, 10.2, 1H), 1.58 – 1.23 (m, 9H), 1.20 (s, 3H), 1.02 (s, 3H), 0.57 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 167.2, 148.1, 137.7, 129.6, 129.0, 128.8, 128.0, 127.9, 126.0, 88.5, 59.7, 51.9, 43.1, 40.5, 34.4, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{34}O_3N$ $[M + H]^+$, 396.2533, found 396.2533.




**41**

**2,2,6,6-tetramethyl-1-(1-(naphthalen-2-yl)-2-phenylethoxy)piperidine (41)**

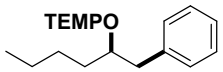
Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), methyl 2-vinylnaphthalene (69.3 mg, 1.50 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **41** as a colorless oil (105.7 mg, 91% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.74 – 7.67 (m, 1H), 7.65 (d, *J* = 8.4, 1H), 7.61 (d, *J* = 8.2, 1H), 7.37 – 7.28 (m, 4H), 7.05 – 6.92 (m, 3H), 6.80 (d, *J* = 7.3, 2H), 4.89 (dd, *J* = 10.1, 4.3, 1H), 3.60 (dd, *J* = 12.9, 4.3, 1H), 3.01 – 2.90 (m, 1H), 1.60 – 1.10 (m, 12H), 0.96 (s, 3H), 0.48 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 140.1, 138.4, 132.9, 132.8, 129.7, 128.0, 127.9, 127.6, 127.3, 127.1, 126.1, 125.8, 125.6, 125.4, 88.9, 59.6, 43.0, 40.5, 34.5, 30.4, 20.5, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{27}H_{34}ON$ $[M + H]^+$, 388.2635, found 388.2635.




**42**

**2-(2-phenyl-1-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)pyridine (42)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 2-vinylpyridine (47.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **42** as a colorless oil (87.2 mg, 86% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.55 (dd, *J* = 4.8, 0.7, 1H), 7.47 (td, *J* = 7.6, 1.8, 1H), 7.17 – 6.99 (m, 5H), 6.94 (d, *J* = 6.9, 2H), 4.97 (dd, *J* = 9.4, 5.1, 1H), 3.49 (dd, *J* = 13.2, 5.1, 1H), 3.25 (dd, *J* = 13.2, 9.4, 1H), 1.67 – 1.12 (m, 12H), 1.03 (s, 3H), 0.36 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 161.6, 148.8, 138.2, 135.3, 129.6, 127.9, 125.8, 124.2, 122.2, 89.0, 59.3, 41.0, 40.5, 34.2, 33.5, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{22}H_{31}ON_2$ $[M + H]^+$, 339.2431, found 339.2431.

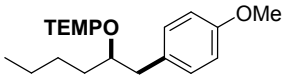



**43**

**2,2,6,6-tetramethyl-1-((1-phenylhexan-2-yl)oxy)piperidine (43)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), hex-1-ene (75.6 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **43** as a colorless oil (51.4 mg, 54% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.29 – 7.25 (m, 2H), 7.23 – 718 (m, 3H), 4.05 – 3.96 (m, 1H), 3.35 (dd, *J* = 13.0, 4.6, 1H), 2.53 (dd, *J* = 13.0, 8.7, 1H), 1.68 – 1.25 (m, 9H), 1.24 – 1.10 (m, 12H), 1.07 (s, 3H), 0.89 (t, *J* = 7.1, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 140.4, 129.6, 128.1, 125.6, 83.2, 60.1, 59.5, 40.5, 40.3, 39.9, 34.6, 34.3, 32.4, 27.9, 23.0, 20.7, 20.5, 17.4, 14.1; HR-ESI-MS (*m/z*): calcd. for $C_{21}H_{36}ON$

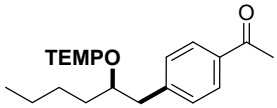
$[M + H]^+$, 318.2791, found 318.2788.




**44**

**1-((1-(4-methoxyphenyl)hexan-2-yl)oxy)-2,2,6,6-tetramethylpiperidine (44)**

Prepared according to the general procedure using 4-iodoanisole (70.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), hex-1-ene (75.6 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **44** as a colorless oil (54.2 mg, 52% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.10 (d, $J$ = 8.5, 2H), 6.81 (d, $J$ = 8.5, 2H), 3.99 – 3.84 (m, 1H), 3.78 (s, 3H), 3.25 (dd, $J$ = 13.2, 4.5, 1H), 2.45 (dd, $J$ = 13.2, 8.6, 1H), 1.71 – 1.22 (m, 12H), 1.18 – 0.90 (m, 12H), 0.86 (t, $J$ = 7.1, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 157.7, 132.4, 130.4, 113.5, 83.3, 60.0, 59.5, 55.2, 40.5, 40.3, 38.8, 34.6, 34.3, 32.3, 27.9, 23.0, 20.7, 20.5, 17.4, 14.1; HR-ESI-MS (*m/z*): calcd. for $C_{22}H_{38}O_2N$ $[M + H]^+$, 348.2897, found 348.2896.

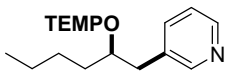



**45**

**1-(4-(2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)hexyl)phenyl)ethan-1-one (45)**

Prepared according to the general procedure using 1-(4-chlorophenyl)ethan-1-one (46.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), hex-1-ene (75.6 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **45** as a colorless oil (60.3 mg, 56% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.87 (d, $J$ = 7.6, 2H), 7.28 (d, $J$ = 7.6, 2H), 4.07 – 3.91 (m, 1H), 3.34 (dd, $J$ = 13.0, 4.8, 1H), 2.68 – 2.54 (m, 4H), 1.71 – 1.23 (m, 12H), 1.18 (s, 3H), 1.13 (s, 3H), 1.08 (s, 3H), 1.00 (s, 3H), 0.86 (t, $J$ = 6.8, 3H); $^{13}$C

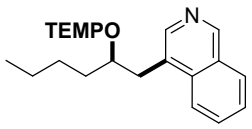
NMR (150 MHz, $CDCl_3$): δ 197.9, 146.4, 135.0, 129.7, 128.3, 82.9, 60.1, 59.6, 40.5, 40.3, 40.0, 34.6, 34.3, 32.5, 30.2, 29.5, 28.0, 26.5, 22.9, 20.7, 20.5, 17.4, 14.1; HR-ESI-MS (*m/z*): calcd. for $C_{23}H_{38}O_2N$ $[M + H]^+$, 360.2897, found 360.2896.




**46**

**3-(2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)hexyl)pyridine (46)**

Prepared according to the general procedure using 3-iodopyridine (61.5 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), hex-1-ene (75.6 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **46** as a colorless oil (58.2 mg, 61% yield). $^1H$ NMR (600 MHz, $CDCl_3$): δ 8.50 – 8.37 (m, 2H), 7.52 (d, *J* = 7.7, 1H), 7.19 (dd, *J* = 7.7, 4.8, 1H), 3.96 (dq, *J* = 11.3, 5.6, 1H), 3.22 (dd, *J* = 13.4, 5.6, 1H), 2.58 (dd, *J* = 13.4, 11.3, 1H), 1.78 – 1.20 (m, 12H), 1.20 – 0.94 (m, 12H), 0.92 – 0.82 (m, 3H); $^{13}C$ NMR (150 MHz, $CDCl_3$): δ 150.9, 147.2, 136.9, 135.6, 123.0, 82.7, 59.4, 40.3, 36.96, 34.3, 32.3, 28.0, 22.9, 20.7, 17.3, 14.0; HR-ESI-MS (*m/z*): calcd. for $C_{20}H_{35}ON_2$ $[M + H]^+$, 319.2744, found 319.2744.

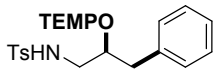



**47**

**4-(2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)hexyl)isoquinoline (47)**

Prepared according to the general procedure using 4-bromoisoquinoline (62.1 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), hex-1-ene (75.6 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **47** as a colorless oil (74.0 mg, 67% yield). $^1H$ NMR (600 MHz, $CDCl_3$): δ 9.11 (s, 1H), 8.40 (s, 1H), 8.20 (d, *J* = 8.5, 1H),

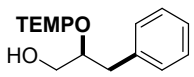
7.96 (d, $J$ = 8.1, 1H), 7.72 (t, $J$ = 7.6, 1H), 7.58 (t, $J$ = 7.4, 1H), 4.19 – 4.06 (m, 1H), 3.68 (dd, $J$ = 13.4, 5.1, 1H), 2.92 (dd, $J$ = 13.4, 8.1, 1H), 1.63 – 1.24 (m, 12H), 1.20 (s, 3H), 1.13 (s, 3H), 1.05 (s, 3H), 0.90 (s, 3H), 0.86 (t, $J$ = 7.0, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 151.0, 144.2, 135.2, 129.9, 129.6, 128.4, 128.1, 126.7, 123.7, 81.9, 60.3, 59.7, 40.5, 40.4, 34.6, 34.4, 33.3, 28.2, 23.1, 20.8, 20.5, 17.4, 14.1; HR-ESI-MS (*m/z*): calcd. for $C_{24}H_{37}ON_2$ $[M + H]^+$, 369.2900, found 369.2898.




**48**

***N*-(3-phenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)propyl)benzenesulfonamide (48)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), *N*-allyl-4-methylbenzenesulfonamide (190.0 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:30) to afford the product **48** as a colorless oil (56.0 mg, 42% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.66 (d, $J$ = 8.2, 2H), 7.33 – 7.15 (m, 5H), 7.11 (d, $J$ = 7.2, 2H), 6.30 – 6.12 (m, 1H), 4.29 – 4.15 (m, 1H), 3.22 – 3.03 (m, 2H), 2.96 (dd, $J$ = 13.5, 5.9, 1H), 2.59 (dd, $J$=13.5, 7.3, 1H), 2.42 (s, 3H), 1.64 – 1.20 (m, 6H), 1.14 – 0.82 (m, 12H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 143.1, 137.9, 137.3, 129.6, 129.4, 128.3, 127.0, 126.3, 100.0, 80.7, 60.9, 47.0, 40.3, 38.4, 34.1, 32.8, 21.4, 20.7, 20.4, 17.1; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{37}O_3N_2S$ $[M + H]^+$, 445.2519, found 445.2523.

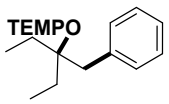



**49**

**3-phenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)propan-1-ol (49)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30

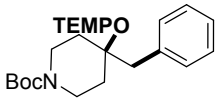
mmol), TEMPOH (56.6 mg, 0.36 mmol), prop-2-en-1-ol (104.4 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **49** as a colorless oil (53.3 mg, 61% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.37 – 7.10 (m, 5H), 5.62 (s, 1H), 4.54 – 4.37 (m, 1H), 3.97 (dd, *J* = 11.9, 9.5, 1H), 3.65 (d, *J* = 12.0, 1H), 2.73 (dd, *J* = 13.7, 7.1, 1H), 2.59 (dd, *J* = 13.7, 5.5, 1H), 1.66 – 1.32 (m, 6H), 1.24 (s, 3H), 1.21 (s, 3H), 1.12 (s, 3H), 0.98 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 138.4, 129.4, 128.1, 126.1, 81.2, 67.7, 61.5, 60.0, 40.3, 40.0, 37.7, 34.5, 32.4, 20.6, 20.2, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{18}H_{30}O_2N$ $[M + H]^+$, 292.2271, found 292.2275.




**50**

**1-((3-benzylpentan-3-yl)oxy)-2,2,6,6-tetramethylpiperidine (50)**

Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 3-methylenepentane (151.4 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:200) to afford the product **50** as a colorless oil (64.7 mg, 68% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.30 – 7.08 (m, 5H), 3.01 (s, 2H), 1.78 (dq, *J* = 14.7, 7.6, 2H), 1.63 (dq, *J* = 14.7, 7.6, 2H), 1.59 – 1.32 (m, 6H), 1.12 (s, 6H), 1.08 (s, 6H), 0.67 (t, *J* = 7.6, 6H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 139.9, 130.8, 127.7, 125.7, 82.9, 59.6, 44.4, 41.2, 34.6, 30.4, 21.1, 17.2, 9.2; HR-ESI-MS (*m/z*): calcd. for $C_{21}H_{36}ON$ $[M + H]^+$, 318.2791, found 318.2788.

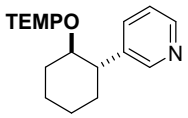



**51**

***Tert*-butyl 4-benzyl-4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)piperidine-1-**

**carboxylate (51)**

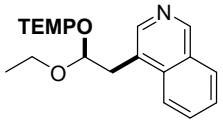
Prepared according to the general procedure using iodobenzene **1a** (61.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), *tert*-butyl 4-methylenepiperidine-1-carboxylate (177.4 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:150) to afford the product **51** as a colorless oil (77.4 mg, 60% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.24 – 6.97 (m, 5H), 3.90 – 3.57 (m, 2H), 3.33 – 3.03 (m, 4H), 1.98 – 1.78 (m, 2H), 1.67 – 1.38 (m, 6H), 1.39 – 1.29 (m, 11H), 1.25 – 1.05 m, 12H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 155.1, 130.7, 128.0, 126.1, 79.1, 59.7, 42.8, 40.9, 35.1, 28.4, 21.6, 17.1; HR-ESI-MS (*m/z*): calcd. for $C_{26}H_{43}O_3N_2$ $[M + H]^+$, 431.3268, found 431.3268.




52

**3-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)cyclohexyl)pyridine (52)**

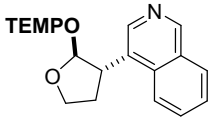
Prepared according to the general procedure using 3-iodopyridine (61.5 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), cyclohexene (73.8 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **52** as a colorless oil (48.4 mg, 51% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.52 (d, $J$ = 1.9, 1H), 8.42 (d, $J$ = 3.9, 1H), 7.56 (d, $J$ = 7.8, 1H), 7.21 (dd, $J$ = 7.8, 3.9, 1H), 3.92 – 3.79 (m, 1H), 2.59 – 2.43 (m, 2H), 1.91 – 1.53 (m, 3H), 1.54 – 1.17 (m, 10H), 1.11 (s, 3H), 0.93 (s, 3H), 0.89 (s, 3H), 0.46 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 150.1, 147.2, 140.3, 134.9, 122.8, 83.6, 60.6, 58.7, 48.1, 40.5, 40.1, 34.6, 34.3, 32.9, 32.4, 26.3, 25.0, 20.4, 19.8, 17.3; HR-ESI-MS (*m/z*): calcd. for $C_{20}H_{33}ON_2$ $[M + H]^+$, 317.2587, found 317.2589.




53

**4-(2-ethoxy-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)isoquinoline (53)**

Prepared according to the general procedure using 4-bromoisoquinoline (62.1 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), ethoxyethene (64.8 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **53** as a colorless oil (86.6 mg, 81% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 9.14 (s, 1H), 8.48 (s, 1H), 8.14 (d, *J* = 8.5, 1H), 7.95 (d, *J* = 8.2, 1H), 7.72 (t, *J* = 7.6, 1H), 7.58 (t, *J* = 7.5, 1H), 5.03 (t, *J* = 5.8, 1H), 3.90 (dq, *J* = 14.2, 7.1, 1H), 3.43 – 3.30 (m, 2H), 3.24 (dd, *J* = 14.2, 6.0, 1H), 1.64 – 1.18 (m, 9H), 1.06 (s, 3H), 0.93 – 0.89 (s, 3H), 0.82 (s, 3H), 0.79 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 151.4, 144.6, 135.5, 129.9, 128.3, 128.0, 127.2, 126.7, 123.7, 107.0, 66.6, 60.5, 59.3, 40.4, 40.2, 35.1, 33.6, 33.4, 20.6, 19.8, 17.2, 15.1; HR-ESI-MS (*m/z*): calcd. for $C_{22}H_{33}O_2N_2$ $[M + H]^+$, 357.2537, found 357.2538.




54

**4-((3S)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)tetrahydrofuran-3-yl)isoquinoline (54)**

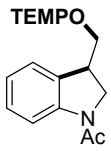
Prepared according to the general procedure using 4-bromoisoquinoline (62.1 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 2,3-dihydrofuran (63.0 mg, 0.90 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 16 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **54** as a colorless oil (79.7 mg, 75 % yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 9.15 (s, 1H), 8.48 (s, 1H), 8.17 (d, *J* = 8.5, 1H), 7.99 (t, *J* = 6.8, 1H), 7.75 (t, *J* = 7.7, 1H), 7.62 (t, *J* = 7.5, 1H), 5.74 (d, *J* = 1.8, 1H), 4.30 – 4.06 (m, 2H), 4.00 (t, *J* = 6.9, 1H), 2.65 – 2.42 (m, 1H), 2.07 (dt, *J* = 14.2, 7.6,

1H), 1.65 – 1.17 (m, 9H), 1.15 (s, 3H), 1.07 (s, 3H), 0.75 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 151.9, 141.7, 133.8, 131.5, 130.4, 128.6, 126.9, 122.9, 113.1, 67.0, 60.5, 58.9, 46.1, 40.2, 39.7, 34.0, 33.6, 33.0, 20.6, 20.1, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{22}H_{31}O_2N_2$ $[M + H]^+$, 355.2380, found 355.2377.


**55**

**1-((2,3-dihydrobenzofuran-3-yl)methoxy)-2,2,6,6-tetramethylpiperidine (55)**

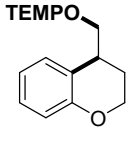
To an oven dried 10 mL glass tube with a magnetic stirring bar was added 1-(allyloxy)-2-iodobenzene (78.0 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (3.0 mL) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 16h at room temperature. Irradiation was stopped and the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:200) on silica gel to afford **55** a colorless oil (71.1 mg, 82% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.24 (d, *J* = 7.4, 1H), 7.13 (t, *J* = 7.7, 1H), 6.84 (t, *J* = 7.2, 1H), 6.79 (d, *J* = 8.0, 1H), 4.63 (t, *J* = 9.0, 1H), 4.39 (dd, *J* = 9.0, 6.2, 1H), 3.98 (dd, *J* = 8.5, 6.5, 1H), 3.88 (t, *J* = 8.3, 1H), 3.78 – 3.64 (m, 1H), 1.59 – 1.22 (m, 6H), 1.19 – 1.0.3 (s, 12H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 160.2, 128.5, 128.0, 125.1, 120.2, 109.5, 78.8, 74.5, 59.9, 41.8, 39.6, 33.2, 33.0, 20.2, 20.1, 17.1; HR-ESI-MS (*m/z*): calcd. for $C_{18}H_{28}O_2N$ $[M + H]^+$, 290.2115, found 290.2112.




56

**1-(3-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)indolin-1-yl)ethan-1-one (56)**

To an oven dried 10 mL glass tube with a magnetic stirring bar was added *N*-allyl-*N*-(2-iodophenyl)acetamide (90.3 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (3.0 mL) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 16h at room temperature. Irradiation was stopped and the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:100) on silica gel to afford **56** a colorless oil (78.3 mg, 79% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 8.21 (d, *J* = 8.0, 1H), 7.27 – 7.19 (m, 2H), 7.01 (t, *J* = 7.4, 1H), 4.13 (t, *J* = 10.0, 1H), 4.03 – 3.81 (m, 3H), 3.66 – 3.56 (m, 1H), 2.23 (s, 3H), 1.63 – 1.21 (m, 6H), 1.19 – 0.95 (m, 12H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 168.6, 143.0, 132.0, 128.2, 124.5, 123.5, 117.0, 78.9, 60.0, 52.7, 40.1, 39.6, 33.2, 33.1, 24.2, 20.2, 20.1, 17.0; HR-ESI-MS (*m/z*): calcd. for $C_{20}H_{31}O_2N_2$ $[M + H]^+$, 331.2380, found 331.2380.

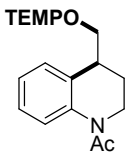



57

**1-(chroman-4-ylmethoxy)-2,2,6,6-tetramethylpiperidine (57)**

To an oven dried 10 mL glass tube with a magnetic stirring bar was added 1-(but-3-en-1-yloxy)-2-iodobenzene (82.2 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube

was allowed to be vacuumed and purged with Argon for three times. DMSO (3.0 mL) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 16h at room temperature. Irradiation was stopped and the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:200) on silica gel to afford **57** a colorless oil (77.3 mg, 85% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.17 (d, *J* = 7.6, 1H), 7.10 (t, *J* = 8.2, 1H), 6.84 (t, *J* = 7.4, 1H), 6.80 (d, *J* = 8.2, 1H), 4.21 (dd, *J* = 7.1, 3.7, 2H), 4.00 (dd, *J* = 8.9, 5.4, 1H), 3.91 (t, *J* = 8.8, 1H), 3.09 (dd, *J* = 8.6, 5.1, 1H), 2.19 – 1.97 (m, 2H), 1.62 – 1.23 (m, 6H), 1.19 – 1.05 (m, 12H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 155.0, 129.7, 127.6, 122.9, 120.0, 116.8, 80.2, 63.6, 59.9, 59.8, 39.6, 33.6, 33.2, 33.0, 25.3, 20.3, 17.1; HR-ESI-MS (*m/z*): calcd. for $C_{19}H_{30}O_2N$ $[M + H]^+$, 304.2271, found 304.2265.

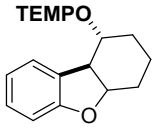



**58**

**1-(4-(((2,2,6,6-tetramethylpiperidin-1-yl)oxy)methyl)-3,4-dihydroquinolin-1(2H)-yl)ethan-1-one (58)**

To an oven dried 10 mL glass tube with a magnetic stirring bar was added *N*-(but-3-en-1-yl)-*N*-(2-iodophenyl)acetamide (94.5 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (3.0 mL) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 16h at room temperature. Irradiation was stopped and the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The

crude product was subjected to column chromatography (acetone/petroleum ether = 1:100) on silica gel to afford **58** a colorless oil (82.6 mg, 80% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.25 – 6.99 (m, 4H), 4.05 – 3.86 (m, 2H), 3.84 – 3.76 (m, 1H), 3.63 – 3.48 (m, 1H), 3.09 – 2.90 (m, 1H), 2.15 (s, 3H), 2.07 (td, *J* = 12.5, 6.1, 1H), 1.90 – 1.80 (m, 1H), 1.45 – 1.20 (m, 6H), 1.09 – 0.85 (m, 12H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 169.8, 128.5, 126.3, 125.1, 124.6, 79.3, 59.9, 39.6, 36.5, 33.1, 32.9, 27.4, 23.0, 20.2, 17.1; HR-ESI-MS (*m/z*): calcd. for $C_{21}H_{33}O_2N_2$ $[M + H]^+$, 345.2537, found 345.2540.

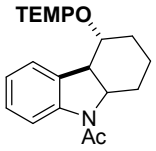



**59**

**1-((-1,2,3,4,4a,9b-hexahydrodibenzo[b,d]furan-1-yl)oxy)-2,2,6,6-tetramethylpiperidine (59)**

To an oven dried 10 mL glass tube with a magnetic stirring bar was added 1-(cyclohex-2-en-1-yloxy)-2-iodobenzene (90.0mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (3.0 mL) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 16h at room temperature. Irradiation was stopped and the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:200) on silica gel to afford **59** a colorless oil (88.9 mg, 90% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.27 (d, *J* = 7.3, 1H), 7.05 (d, *J* = 7.9, 1H), 6.78 (t, *J* = 7.2, 1H), 6.72 (d, *J* = 7.9, 1H), 4.78 – 4.63 (m, 1H), 3.76 (td, *J* = 9.1, 3.5, 1H), 3.09 (t, *J* =7.2, 1H), 2.09 – 1.94 (m, 2H), 1.76 – 1.17 (m, 10H), 1.13 – 0.96 (m, 9H), 0.77 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 159.4, 131.9, 128.0, 125.8, 120.1, 109.7, 83.7, 82.3, 60.6, 59.1, 46.5, 40.5, 40.3, 34.6, 33.9, 30.4, 27.9, 27.6, 20.5, 18.5, 17.3; HR-ESI-MS (*m/z*): calcd.

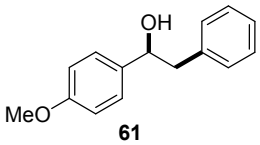
for $C_{21}H_{32}O_2N$ [M + H]$^+$, 330.2428, found 330.2425.




**60**

**1-(-4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)-1,2,3,4,4a,9a-hexahydro-9H-carbazol-9-yl)ethan-1-one (60)**

To an oven dried 10 mL glass tube with a magnetic stirring bar was added *N*-(cyclohex-2-en-1-yl)-*N*-(2-iodophenyl)acetamide (102.3 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), **PC3** (8.5 mg, 0.03 mmol), and $Cs_2CO_3$ (195.5 mg, 0.60 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. DMSO (3.0 mL) was carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 16h at room temperature. Irradiation was stopped and the reaction was diluted with water. The aqueous phase was extracted with ethyl acetate (15 mL × 3). The combined organic layers were washed with brine, dried over anhydrous $Na_2SO_4$ and concentrated. The crude product was subjected to column chromatography (acetone/petroleum ether = 1:100) on silica gel to afford **60** a colorless oil (96.6 mg, 87% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.13 (br, 1H), 7.21 (t, *J* = 7.5, 1H), 7.15 – 7.00 (m, 2H), 4.55 (br, 1H), 4.41 (br, 1H), 3.87 (br, 1H), 2.32 (s, 3H), 2.16 – 1.91 (m, 2H), 1.74 – 1.01 (m, 22H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 168.0, 141.6, 127.7, 123.9, 122.6, 118.2, 78.4, 60.6, 45.1, 40.5, 34.8, 28.0, 25.9, 23.3, 20.4, 17.1, 16.7; HR-ESI-MS (*m/z*): calcd. for $C_{23}H_{35}O_2N_2$ [M + H]$^+$, 371.2693, found 371.2693.

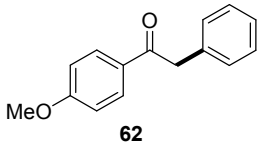



**61**

**1-(4-methoxyphenyl)-2-phenylethan-1-ol (61)**

1-(1-(4-methoxyphenyl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidin (**3**) (110.2 mg,

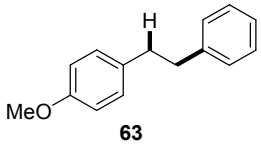
0.30 mmol) was dissolved in HOAc/$H_2O$ (3.0 mL : 9.0 mL) under argon. Then activated zinc powder (117.0 mg, 1.80 mmol) was added to the mixture in one portion and the solution was stirred over night at room temperature. NaOH (30 mL, 0.5 M) was added to quench the reaction. The mixture was extracted with DCM. The combined organic layers were washed with brine and dried over $Na_2SO_4$. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:5) to afford the product **61** as a colorless oil (65.7 mg, 96% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.38 – 7.18 (m, 7H), 6.91 (d, *J* = 8.6, 2H), 4.93 – 4.83 (m, 1H), 3.84 (s, 3H), 3.09 – 2.97 (m, 2H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 159.1, 138.2, 136.0, 129.5, 128.5, 127.2, 126.6, 113.8, 75.0, 55.3, 46.0; HR-ESI-MS (*m/z*): calcd. for $C_{15}H_{16}O_2Na$ [M + Na]$^+$, 251.1043, found 251.1044.




**62**

**1-(4-methoxyphenyl)-2-phenylethan-1-one (62)**

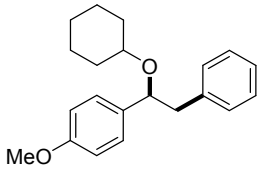
1-(1-(4-methoxyphenyl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidin (**3**) (110.2 mg, 0.30 mmol) and *m*CPBA (87.0 mg, 0.39 mmol, 70-75% in H2O) were dissolved in DCM (10 mL) and the reaction mixture was stirred over night at room temperature. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:10) to afford the product **62** as a colorless oil (64.4 mg, 95% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.8, 2H), 7.31 – 7.06 (m, 5H), 6.85 (d, *J* = 8.8, 2H), 4.16 (s, 2H), 3.78 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 196.2, 163.5, 135.0, 131.0, 129.7, 129.4, 128.7, 126.8, 113.8, 55.5, 45.3; HR-ESI-MS (*m/z*): calcd. for $C_{15}H_{14}O_2Na$ [M + Na]$^+$, 249.0886, found 249.0881.




**63**

**1-methoxy-4-phenethylbenzene (63)**

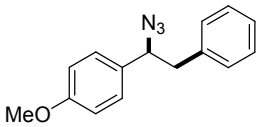
1-(1-(4-methoxyphenyl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidin (**3**) (110.2 mg, 0.30 mmol) and thiophenol (68.0 mg, 0.66 mmol) were dissolved in *tert*-butyl benzene (4 mL). The reaction mixture was heated and stirred at 120 °C overnight. After remove of the solvent, the crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **63** as a colorless oil (57.2 mg, 90% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.33 – 7.28 (m, 2H), 7.24 – 7.20 (m, 3H), 7.13 (d, $J$ = 8.5, 2H), 6.86 (d, $J$ = 8.5, 2H), 3.82 (s, 3H), 2.95 – 2.87 (m, 4H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 167.8, 151.9, 143.9, 139.3, 138.5, 138.3, 135.9, 123.7, 65.3, 48.2, 47.0; HR-ESI-MS (*m/z*): calcd. for $C_{15}H_{16}ONa$ $[M + Na]^+$, 235.1093, found 235.1096.




**64**

**1-(1-(cyclohexyloxy)-2-phenylethyl)-4-methoxybenzene (64)**

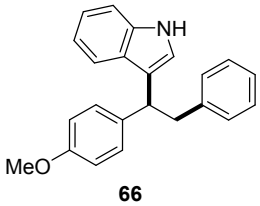
To an oven dried 10 mL glass tube with a magnetic stirring bar was added 1-(1-(4-methoxyphenyl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidin (**3**) (110.2 mg, 0.30 mmol) and $[Ir(dF\text{-}CF_3\text{-}ppy)_2(d(CF_3)\text{-}bpy)]PF_6$ (4.8 mg, 0.006 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. $MeNO_2$ (3.0 mL) and cyclohexanol (90.0 mg, 0.90 mmol) were carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 12h at room temperature. Solvent was removed under vacuum, and the crude product was subjected to column chromatography (acetone/petroleum ether = 1:80) on silica gel to afford **64** a colorless oil (88.4 mg, 95% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.19 – 6.99 (m, 7H), 6.76 (d, $J$ = 8.5, 2H), 4.49 – 4.37 (m, 1H), 3.70 (s, 3H), 3.06 – 2.90 (m, 2H), 2.74 (dd, $J$ = 13.5, 5.6, 1H), 1.69 – 0.85 (m, 10H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 158.8, 139.1, 135.5, 129.7, 127.9, 127.8, 126.0, 113.5, 79.8, 74.8, 55.2, 45.7, 33.5, 31.2, 25.8, 24.2, 23.8; HR-ESI-MS (*m/z*): calcd. for $C_{21}H_{26}O_2Na$ $[M + Na]^+$, 333.1825, found 333.1829.




**65**

**1-(1-azido-2-phenylethyl)-4-methoxybenzene (65)**

To an oven dried 10 mL glass tube with a magnetic stirring bar was added 1-(1-(4-methoxyphenyl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidin (**3**) (110.2 mg, 0.30 mmol) and $[Ir(dF\text{-}CF_3\text{-}ppy)_2(d(CF_3)\text{-}bpy)]PF_6$ (4.8 mg, 0.006 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. $MeNO_2$ (3.0 mL) and $TMSN_3$ (103.7 mg, 0.90 mmol) were carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm) irradiation for 12h at room temperature. Solvent was removed under vacuum, and the crude product was subjected to column chromatography (acetone/petroleum ether = 1:80) on silica gel to afford **65** a colorless oil (66.1 mg, 87% yield). $^1H$ NMR (600 MHz, $CDCl_3$): δ 7.42 – 7.34 (m, 5H), 7.27 (d, $J$ = 7.1, 2H), 7.03 (d, $J$ = 7.8, 2H), 4.76 (dd, $J$ = 8.2, 6.4, 1H), 3.93 (s, 3H), 3.21 (dd, $J$ = 13.8, 8.2, 1H), 3.14 (dd, $J$ = 13.8, 6.4, 1H); $^{13}C$ NMR (150 MHz, $CDCl_3$): δ 159.6, 137.6, 131.3, 129.4, 128.4, 128.3, 126.7, 114.1, 67.2, 55.3, 42.9; HR-ESI-MS (*m/z*): calcd. for $C_{15}H_{15}ON_3Na$ $[M + Na]^+$, 276.1107, found 276.1109.

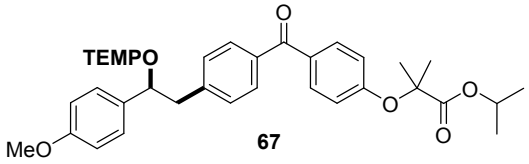



**66**

**3-(1-(4-methoxyphenyl)-2-phenylethyl)-1H-indole (66)**

To an oven dried 10 mL glass tube with a magnetic stirring bar was added 1-(1-(4-methoxyphenyl)-2-phenylethoxy)-2,2,6,6-tetramethylpiperidin (**3**) (110.2 mg, 0.30 mmol) and $[Ir(dF\text{-}CF_3\text{-}ppy)_2(d(CF_3)\text{-}bpy)]PF_6$ (4.8 mg, 0.006 mmol). Then the reaction tube was allowed to be vacuumed and purged with Argon for three times. $MeNO_2$ (3.0 mL) and indole (105.0 mg, 0.90 mmol) were carefully added under Argon. The reaction mixture was stirred under two 18 W blue LED lamps (the distance was about 7.5 cm)

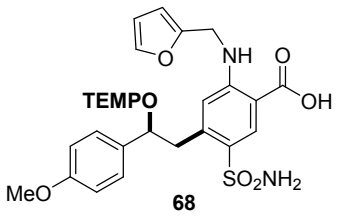
irradiation for 12h at room temperature. Solvent was removed under vacuum, and the crude product was subjected to column chromatography (acetone/petroleum ether = 1:10) on silica gel to afford **66** a colorless oil (91.3 mg, 93% yield). $^{1}$H NMR (600 MHz, $CDCl_3$): δ 7.91 (s, 1H), 7.47 (d, *J* = 7.9, 1H), 7.36 (d, *J* = 8.1, 1H), 7.29 – 7.16 (m, 6H), 7.14 – 7.03 (m, 4H), 6.82 (d, *J* = 7.9, 2H), 4.52 (dd, *J* = 8.9, 6.4, 1H), 3.80 (s, 3H), 3.59 (dd, *J* = 13.5, 6.4, 1H), 3.32 (dd, *J* = 13.5, 8.9, 1H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 157.8, 140.8, 136.6, 136.6, 129.2, 129.1, 128.1, 127.0, 125.9, 122.0, 121.4, 120.1, 119.7, 119.3, 113.6, 111.1, 55.2, 44.1, 42.7; HR-ESI-MS (*m/z*): calcd. for $C_{23}H_{21}ONNa$ $[M + Na]^+$, 350.1515, found 350.1516.




67

**Isopropyl 2-(4-(4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy) ethyl)benzoyl)phenoxy)-2-methylpropanoate (67)**

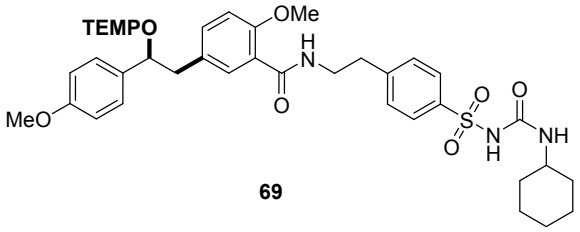
Prepared according to the general procedure using fenofibrate (108.0 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 20 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:50) to afford the product **67** as a colorless oil (125.6 mg, 68% yield). $^{1}$H NMR (600 MHz, $CDCl_3$): δ 7.68 (d, *J* = 8.8, 2H), 7.54 (d, *J* = 8.1, 2H), 7.11 – 6.98 (m, 4H), 6.84 (d, *J* = 8.8, 2H), 6.74 (d, *J* = 8.6, 2H), 5.08 (dt, *J* = 12.5, 6.2, 1H), 4.80 (dd, *J* = 9.8, 4.7, 1H), 3.77 (s, 1H), 3.66 (dd, *J* = 12.6, 4.7, 1H), 2.97 (dd, *J* = 12.6, 9.8, 1H), 1.65 (s, 6H), 1.53 – 1.23 (m, 11H), 1.20 (s, 3H), 1.19 (s, 3H), 1.17 (s, 3H), 1.03 (s, 3H), 0.64 (s, 3H); $^{13}$C NMR (151 MHz, $CDCl_3$): δ 195.3, 173.2, 159.4, 158.8, 143.5, 135.6, 134.5, 131.8, 129.6, 129.5, 129.1, 117.2, 113.1, 87.5, 79.4, 69.2, 60.1, 55.1, 42.9, 40.5, 34.2, 25.4, 21.5, 20.3, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{38}H_{50}O_6N$ $[M + H]^+$, 616.3633, found 616.3631.




**68**

**2-((furan-2-ylmethyl)amino)-4-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-5-sulfamoylbenzoic acid (68)**

Prepared according to the general procedure using furosemide (99.0 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (293.3 mg, 0.90 mmol), and DMSO (1.5 mL). Time of irradiation: 20 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:5) to afford the product **68** as a colorless oil (134.7 mg, 77% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 8.56 (s, 1H), 8.19 (s, 1H), 7.33 (s, 1H), 7.12 (d, *J* = 8.3, 2H), 6.84 (d, *J* = 8.3, 2H), 6.29 (s, 1H), 6.08 (d, *J* = 2.8, 1H), 6.02 (s, 1H), 5.63 (s, 2H), 4.94 (d, *J* = 9.4, 1H), 4.28 (dd, *J* = 13.6, 3.0, 1H), 4.07 (d, *J* = 14.9, 1H), 4.00 (d, *J* = 14.9, 1H), 3.77 (s, 3H), 3.09 (dd, *J* = 13.3, 10.2, 1H), 1.56 – 1.20 (m, 9H), 1.18 (s, 3H), 1.01 (s, 3H), 0.90 (s, 3H); $^{13}$C NMR (151 MHz, $CDCl_3$): δ 171.9, 159.3, 152.1, 150.9, 143.4, 142.3, 133.8, 133.6, 129.2, 127.9, 115.4, 113.6, 110.4, 107.3, 85.7, 61.3, 55.3, 40.0, 39.9, 39.7, 34.2, 27.8, 21.2, 20.2, 17.1; HR-ESI-MS (*m/z*): calcd. for $C_{30}H_{40}O_7N_3S$ $[M + H]^+$, 586.2581, found 586.2578.

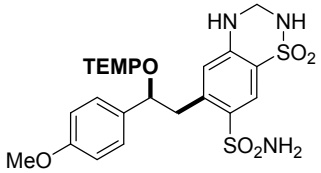



**69**

***N*-(4-(*N*-(cyclohexylcarbamoyl)sulfamoyl)phenethyl)-2-methoxy-5-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)benzamide (69)**

Prepared according to the general procedure using glibenclamide (147.9 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time

of irradiation: 20 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:10) to afford the product **69** as a colorless oil (137.0 mg, 61% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.95 – 7.80 (m, 4H), 7.39 (d, *J* = 8.3, 2H), 7.02 (d, *J* = 8.6, 2H), 6.80 (dd, *J* = 8.4, 2.2, 1H), 6.73 (d, *J* = 8.6, 2H), 6.65 (d, *J* = 8.5, 1H), 6.45 (d, *J* = 7.4, 1H), 4.76 (dd, *J* = 10.0, 4.7, 1H), 3.80 – 3.65 (m, 8H), 3.58 – 3.45 (m, 2H), 3.00 (t, *J* =7.0, 2H), 2.87 (dd, *J* = 13.0, 10.0, 1H), 1.89 – 1.76 (m, 2H), 1.65 (dd, *J* = 9.5, 4.0, 2H), 1.61 – 1.09 (m, 19H), 0.99 (s, 3H), 0.61 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$): δ 165.7, 158.7, 155.7, 150.9, 146.0, 138.0, 134.8, 134.2, 133.0, 131.8, 129.7, 129.2, 127.3, 120.5, 113.0, 111.0, 87.5, 60.0, 55.8, 55.1, 49.1, 41.6, 40.5, 35.6, 34.4, 32.9, 25.4, 24.5, 20.4, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{41}H_{57}O_7N_4S$ $[M + H]^+$, 749.3942, found 749.3945.




**70**

**6-(2-(4-methoxyphenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-3,4-dihydro-2H-benzo[e][1,2,4]thiadiazine-7-sulfonamide 1,1-dioxide (70)**

Prepared according to the general procedure using hydrochlorothhiazide (89.1 mg, 0.30 mmol), TEMPOH (56.6 mg, 0.36 mmol), 4-methoxystyrene **2** (60.3 mg, 0.45 mmol), **PC3** (8.5 mg, 0.03 mmol), $Cs_2CO_3$ (195.5 mg, 0.60 mmol), and DMSO (1.5 mL). Time of irradiation: 20 hours. The crude mixture was purified by silica gel chromatography (acetone/petroleum ether 1:10) to afford the product **70** as a colorless oil (117.6 mg, 71% yield). $^1$H NMR (600 MHz, DMSO-$d_6$): δ 7.90 (s, 1H), 7.75 (t, *J* = 8.1, 1H), 7.64 (s, 1H), 7.37 (s, 2H), 7.26 (d, *J* = 8.5, 2H), 6.83 (d, *J* = 8.5, 2H), 6.68 (s, 1H), 5.08 (t, *J* = 7.0, 1H), 4.73 – 4.55 (m, 2H), 3.70 (s, 3H), 3.58 – 3.43 (m, 2H), 1.59 – 1.13 (m, 6H), 1.09 (s, 3H), 1.05 (s, 3H), 0.98 (s, 3H), 0.38 (s, 3H); $^{13}$C NMR (150 MHz, DMSO-$d_6$): δ 159.0, 145.8, 141.6, 134.5, 130.5, 130.2, 124.4, 119.3, 118.1, 113.5, 84.4, 60.3, 59.2, 55.4, 54.7, 37.9, 34.3, 34.0, 20.8, 17.2; HR-ESI-MS (*m/z*): calcd. for $C_{25}H_{37}O_6N_4S_2$ $[M + H]^+$, 553.2149, found 553.2150.

## References

[1] a) Silvi, M., Arceo, E., Jurberg, I.D., Cassani, C., Melchiorre, P. *J. Am. Chem. Soc.* **2015**, *137*, 6120-6123. b) Bahamonde, A., Melchiorre, P. *J. Am. Chem. Soc.* **2016**, *138*, 8019-8030.

[2] Liang, M., Wang, Z.-Y., Zhang, L., Han, H.-Y., Sun Z., Xue, S. *Renewable Energy* **2011**, *36*, 2711-2716.

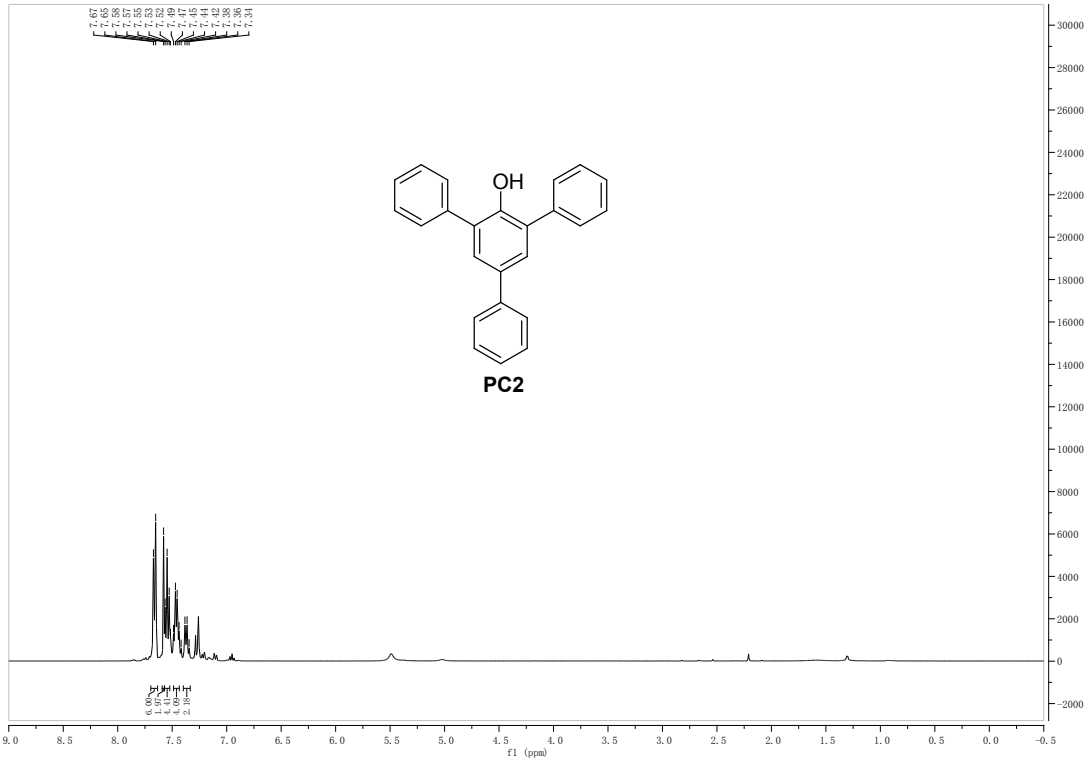
[3] Yin, H., Jin, Y., Hertzog, J. E., Mullane, K. C., Carroll, P. J., Manor, B. C., Anna, J. M., Schelter, E. J. *J. Am. Chem. Soc.*, 2016, **138**, 16266-16273.

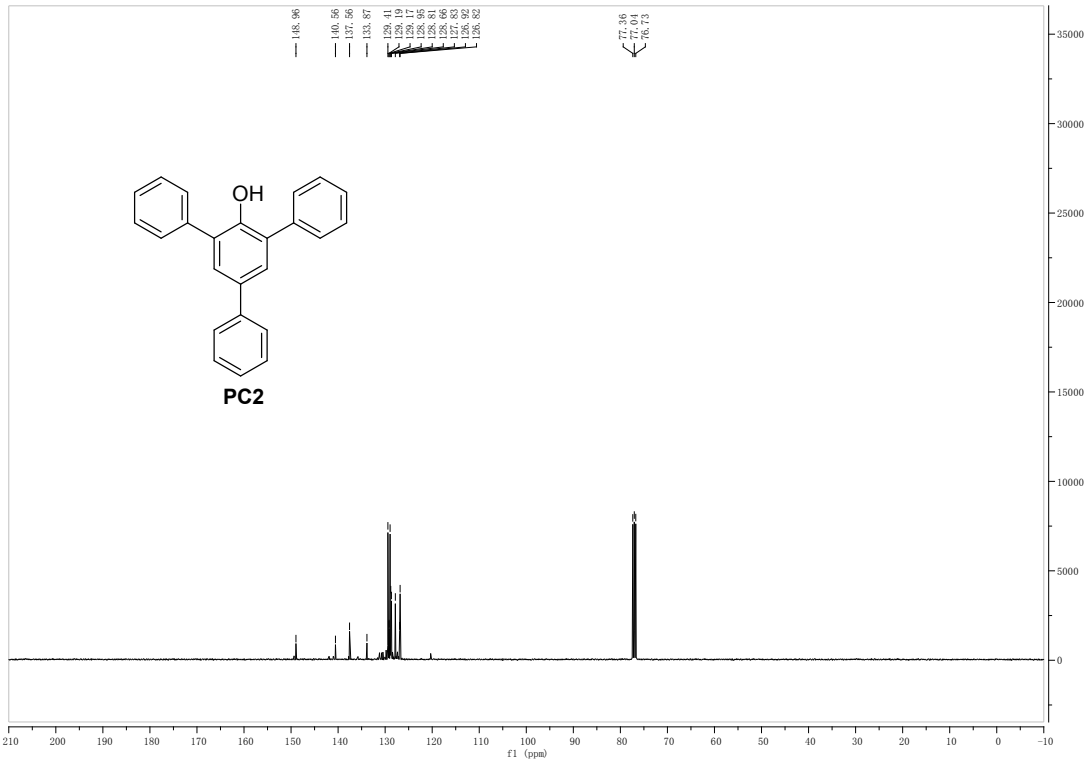
[4] Liu, L., Zhang, Y., Xin, B. *J. Org. Chem.* **2006**, *71*, 3994-3997.

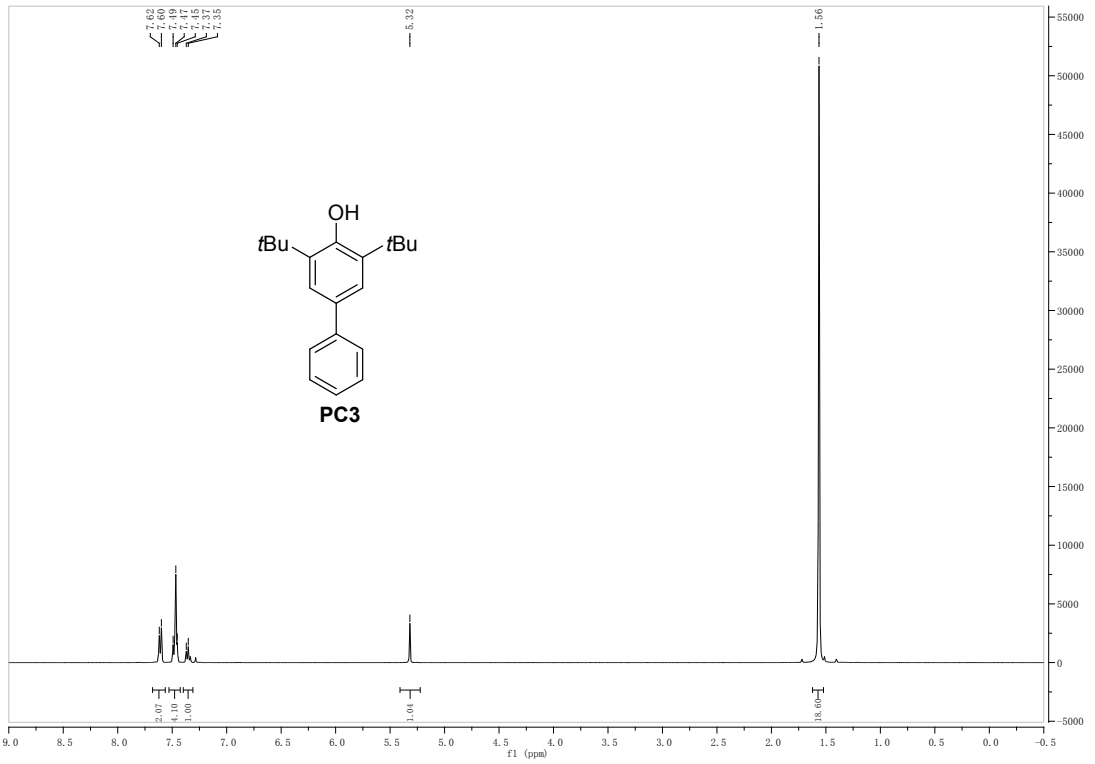
[5] Uyanik, M., Mutsuga, T., Ishihara, K. *Angew. Chem. Int. Ed.* **2017**, *56*, 3956-3960.

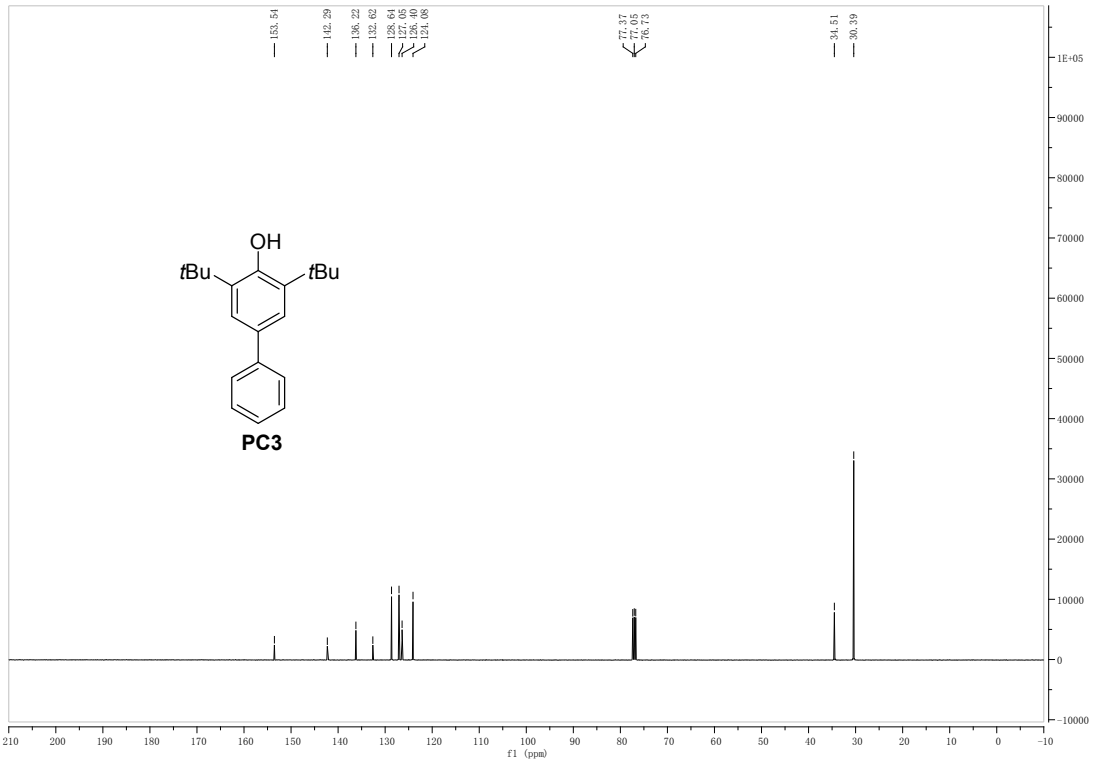
[6] Kurahashi, T., Fujii, H. *J. Am. Chem. Soc.* **2011**, *133*, 8307-8316.

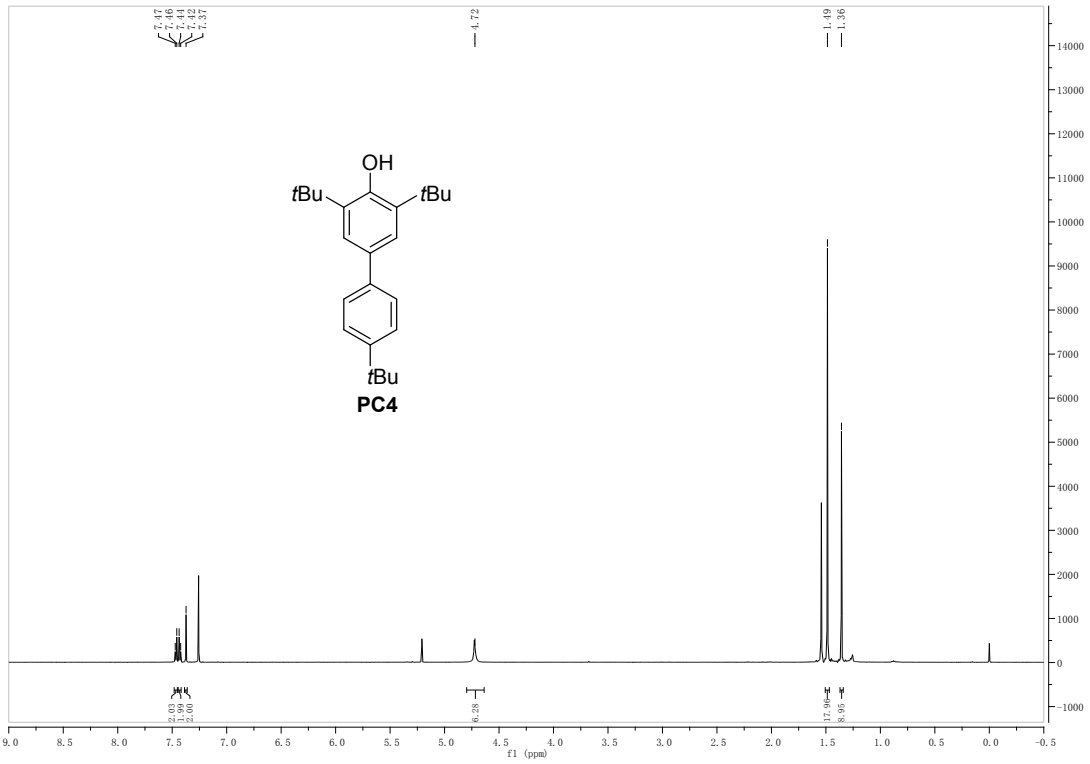
## NMR Spectral Data

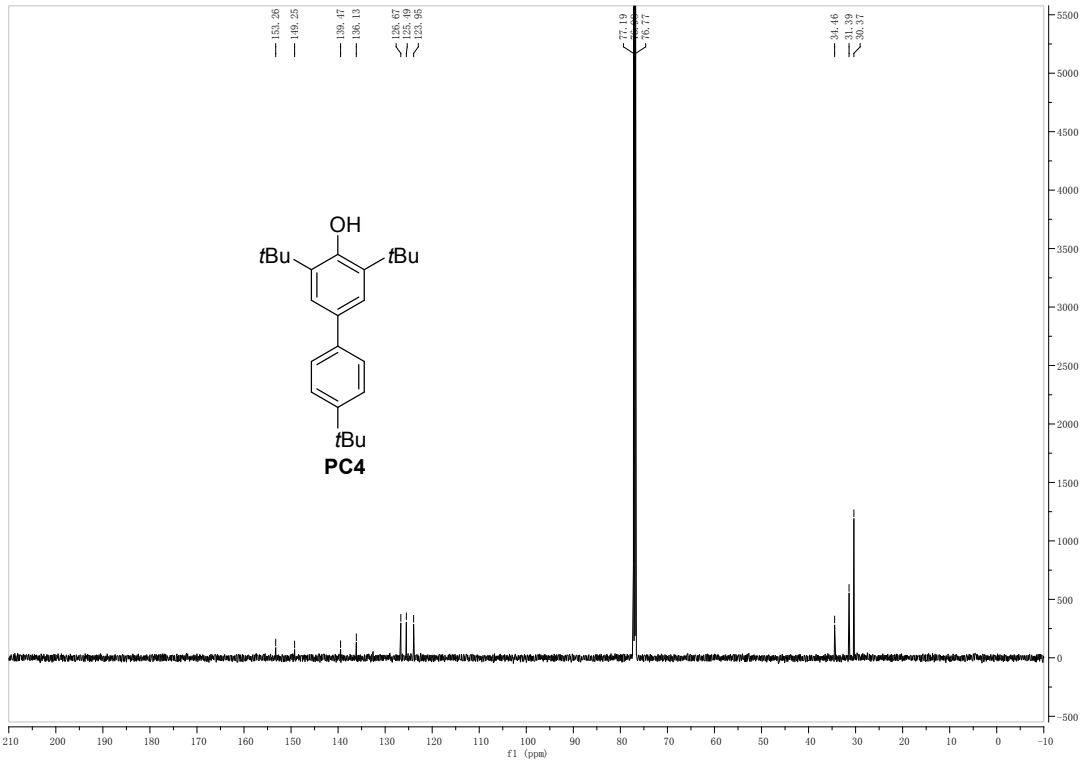


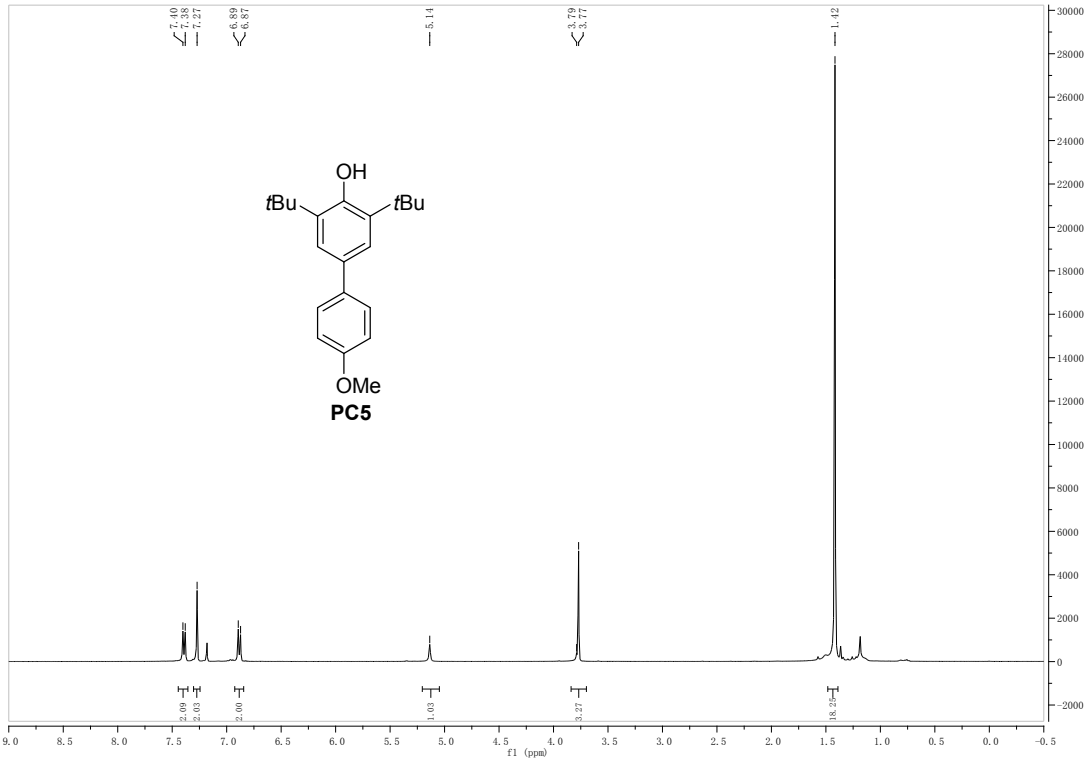



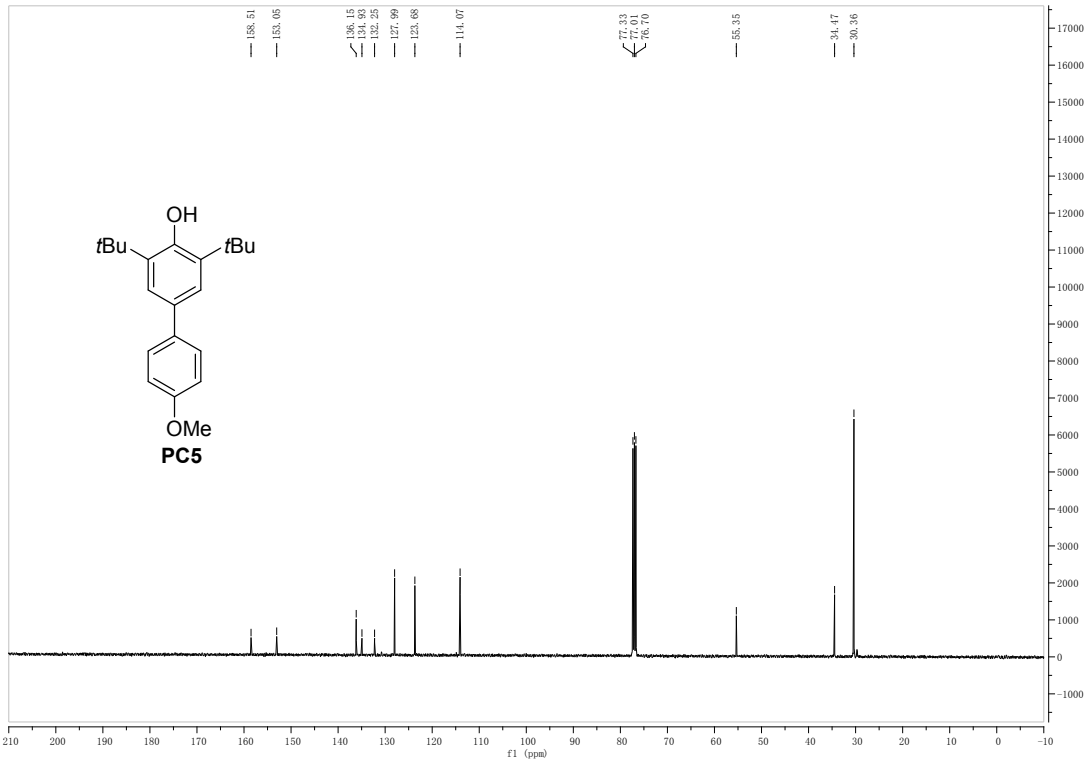

7.62
7.60
7.49
7.47
7.45
7.37
7.35
5.32
1.56
OH
tBu
tBu
PC3
2.07
4.10
1.00
1.04
18.60
f1 (ppm)

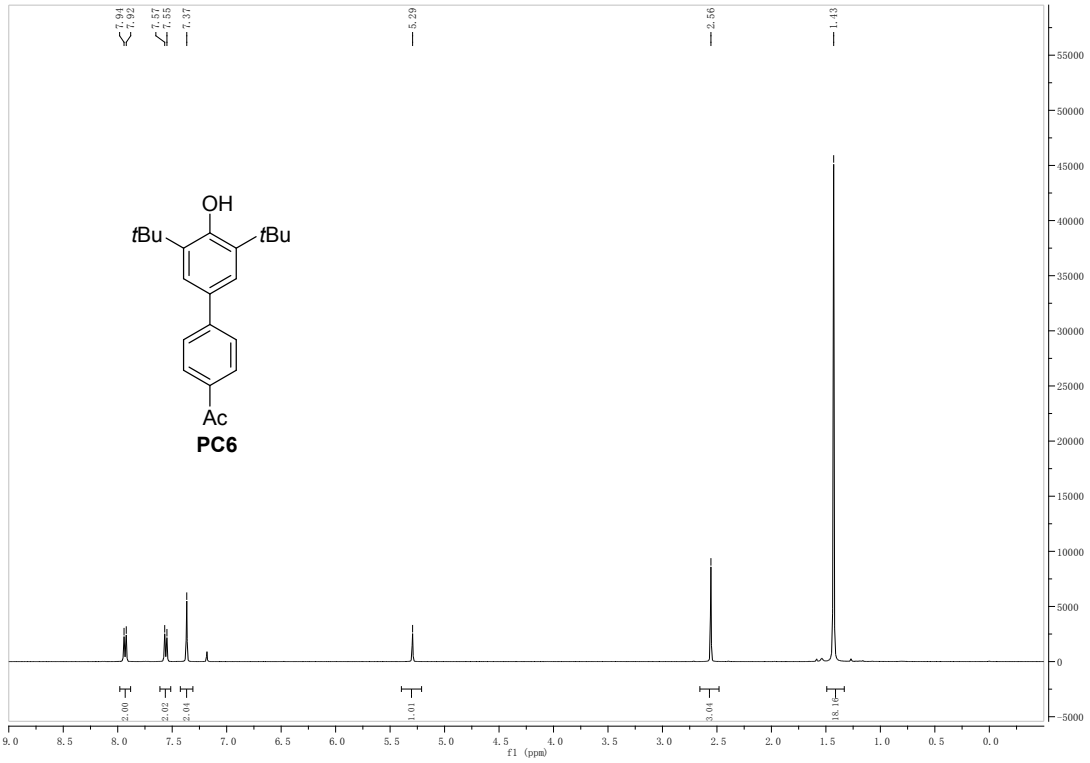

153.54
142.29
136.22
132.62
128.64
127.45
126.40
124.08
77.37
77.05
76.73
34.51
30.39
OH
tBu
tBu
PC3
f1 (ppm)

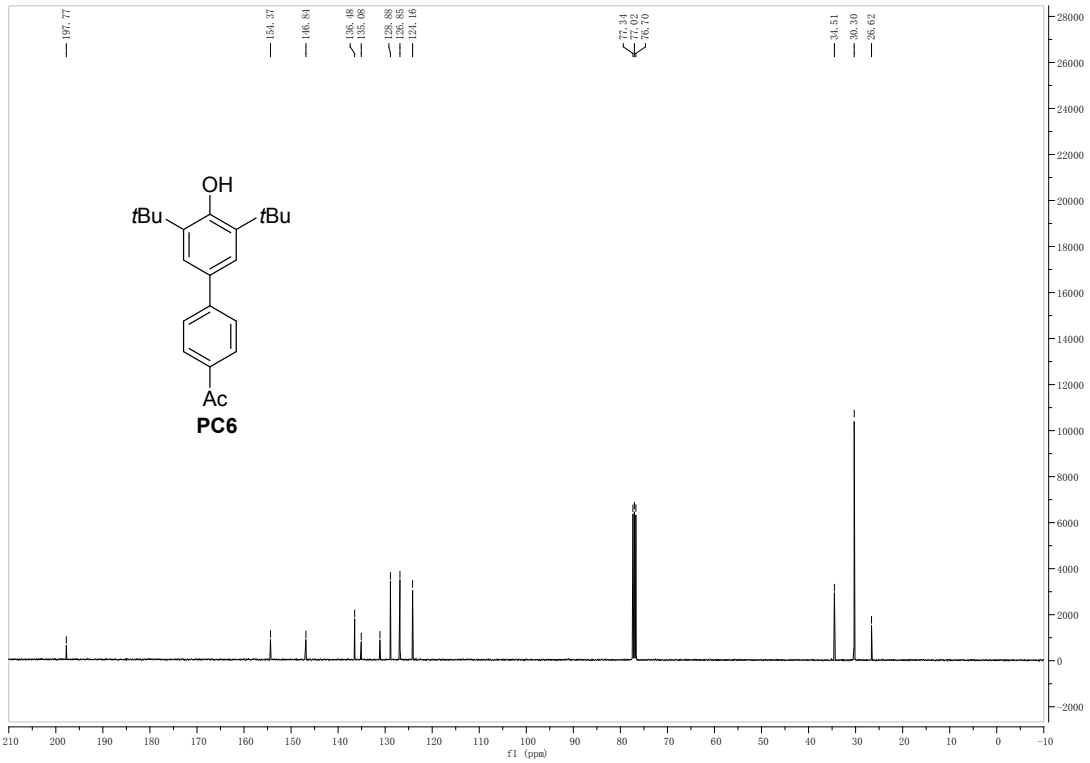

7.47
7.46
7.44
7.43
7.37
4.72
1.49
1.36
OH
tBu
tBu
tBu
PC4
2.03
1.99
1.00
6.28
17.96
8.95
f1 (ppm)

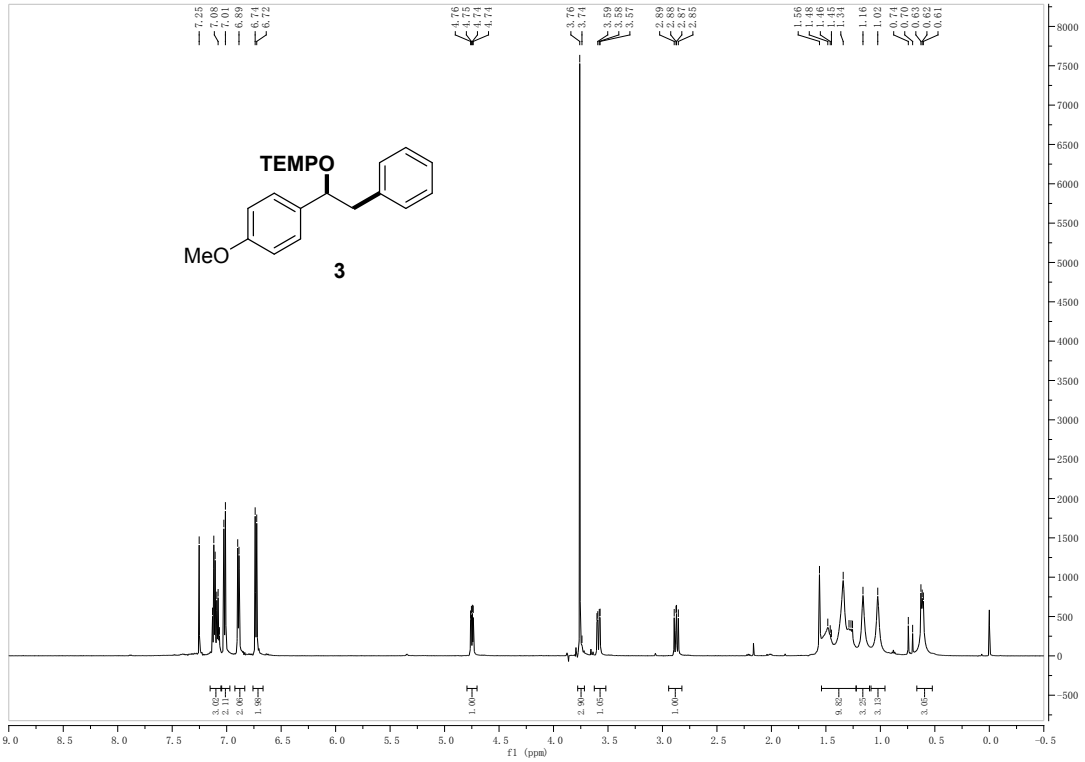

153.26
149.25
139.47
136.13
126.67
125.49
123.95
77.19
76.77
34.46
31.39
30.37
OH
tBu
tBu
tBu
PC4
f1 (ppm)

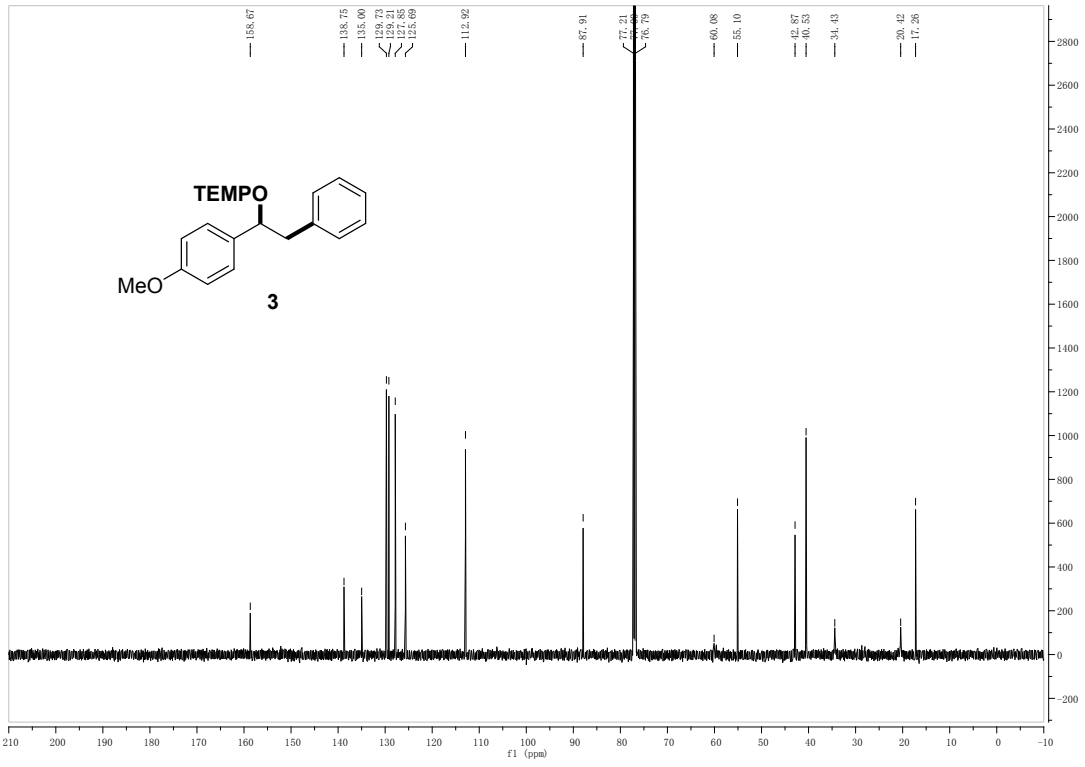

7.40
7.38
7.27
6.89
6.87
5.14
3.79
3.77
1.42
OH
tBu
tBu
OMe
PC5
2.09
2.03
2.00
1.03
3.27
18.25
f1 (ppm)

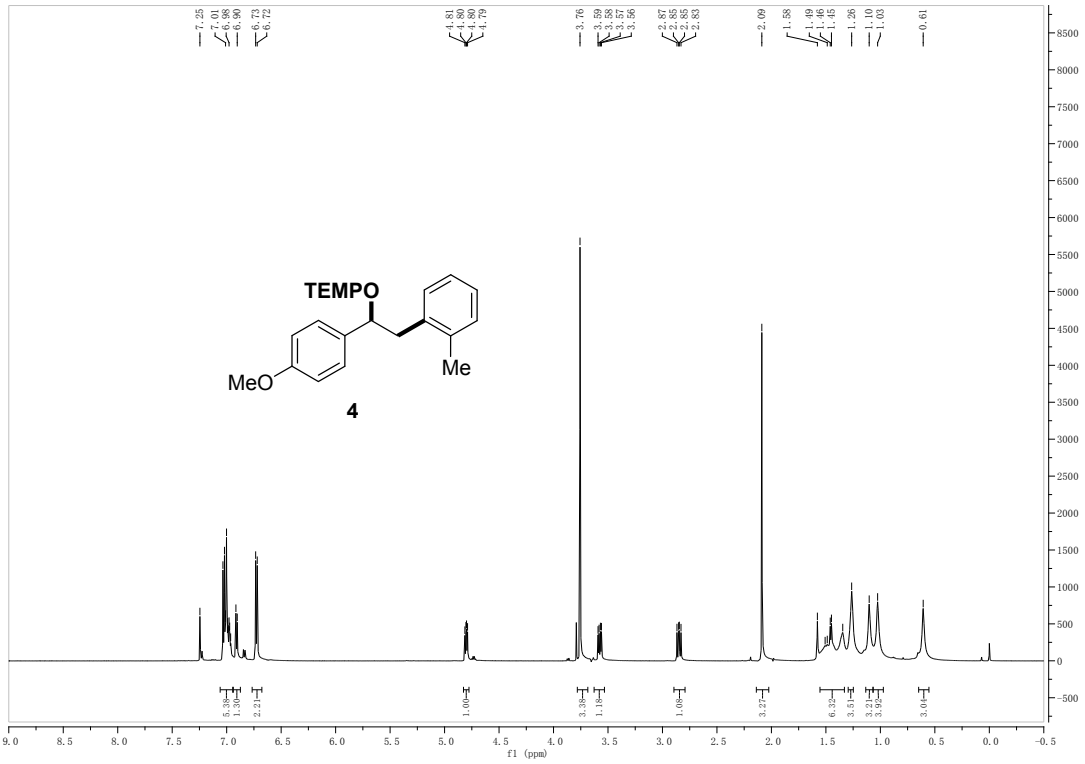

158.51
153.05
136.15
134.93
132.25
127.99
123.68
114.07
77.33
77.01
76.70
55.35
34.47
30.36
OH
tBu
tBu
OMe
PC5
f1 (ppm)

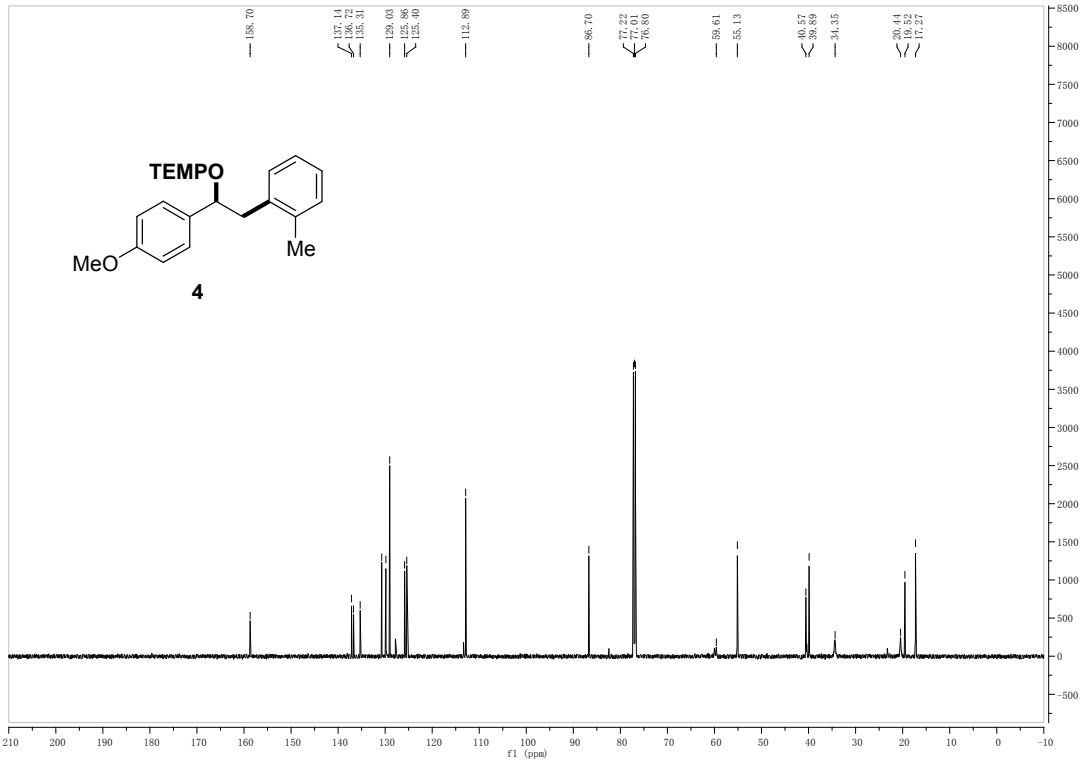

7.94
7.92
7.57
7.55
7.37
5.29
2.56
1.43
OH
tBu
tBu
Ac
PC6
2.00
2.02
2.04
1.01
3.04
18.16
f1 (ppm)

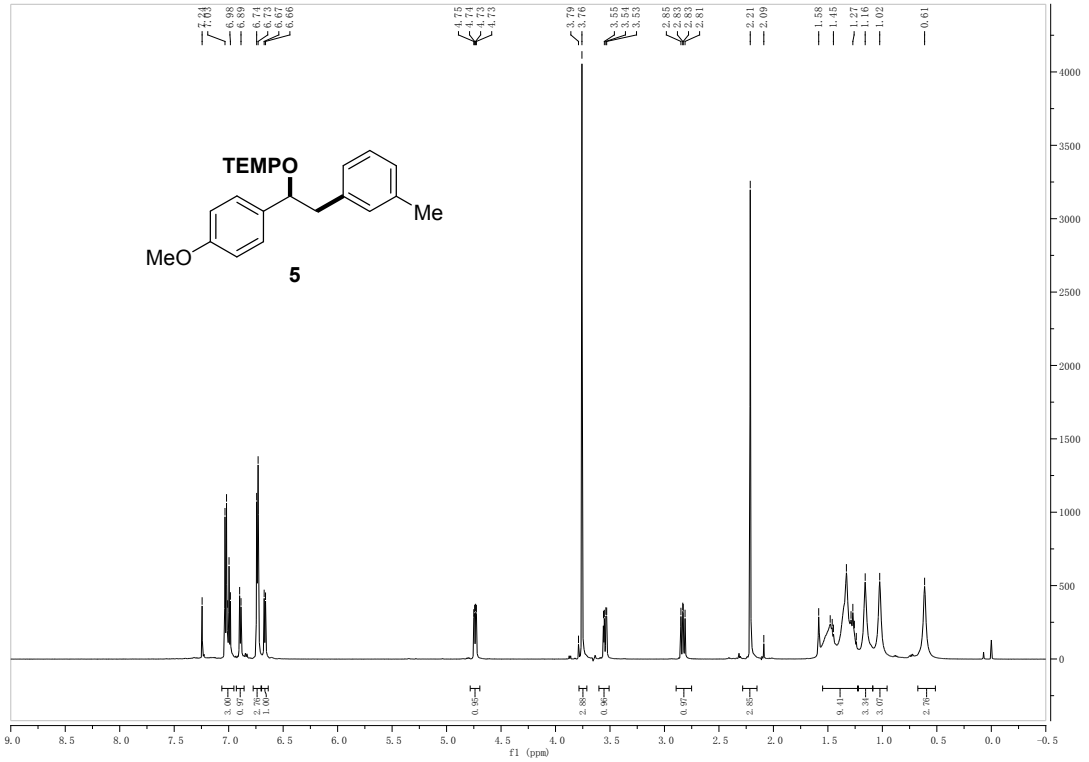

197.77
154.37
146.84
136.48
135.08
128.88
126.85
124.16
77.34
77.02
76.70
34.51
30.30
26.62
OH
tBu
tBu
Ac
PC6
f1 (ppm)

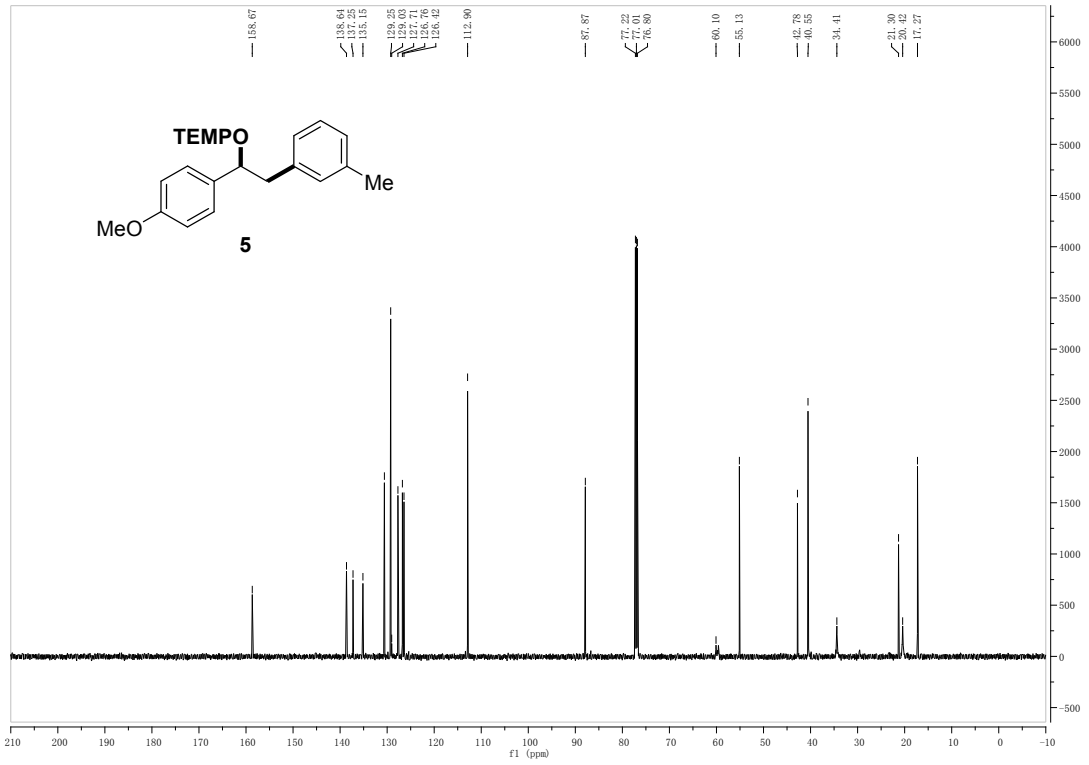

7.25
7.08
7.01
6.89
6.74
6.72
4.76
4.75
4.74
4.74
3.76
3.74
3.59
3.58
3.57
2.89
2.88
2.87
2.85
1.56
1.48
1.46
1.45
1.34
1.16
1.02
0.74
0.70
0.63
0.62
0.61
TEMPO
MeO
3
f1 (ppm)

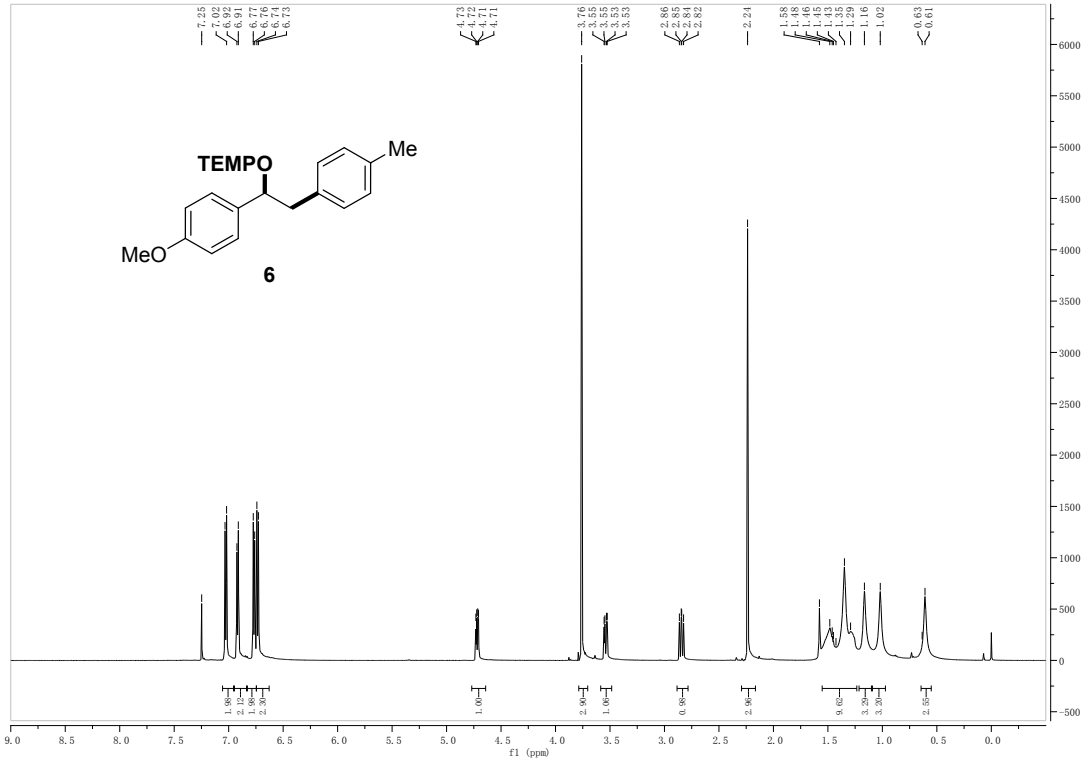

158.67
138.75
135.00
129.73
129.31
127.85
125.69
112.92
87.91
77.21
77.00
76.79
60.08
55.10
42.87
40.53
34.43
20.42
17.26
TEMPO
MeO
3
f1 (ppm)

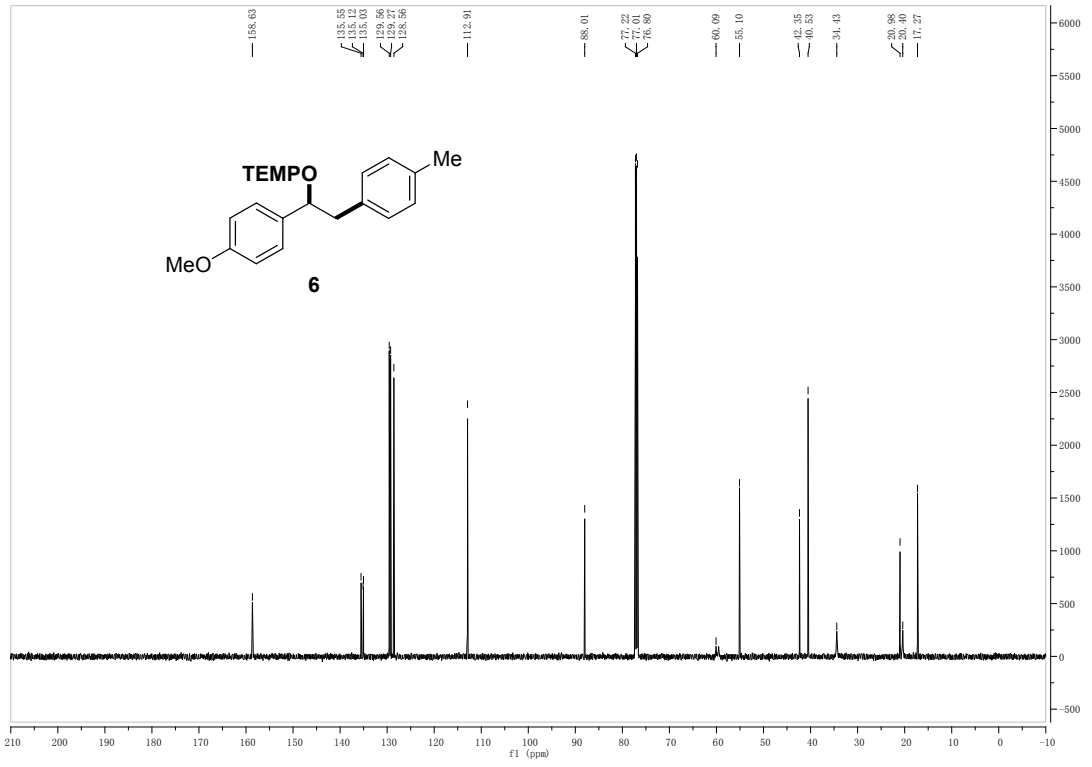

TEMPO
MeO
Me
4
f1 (ppm)

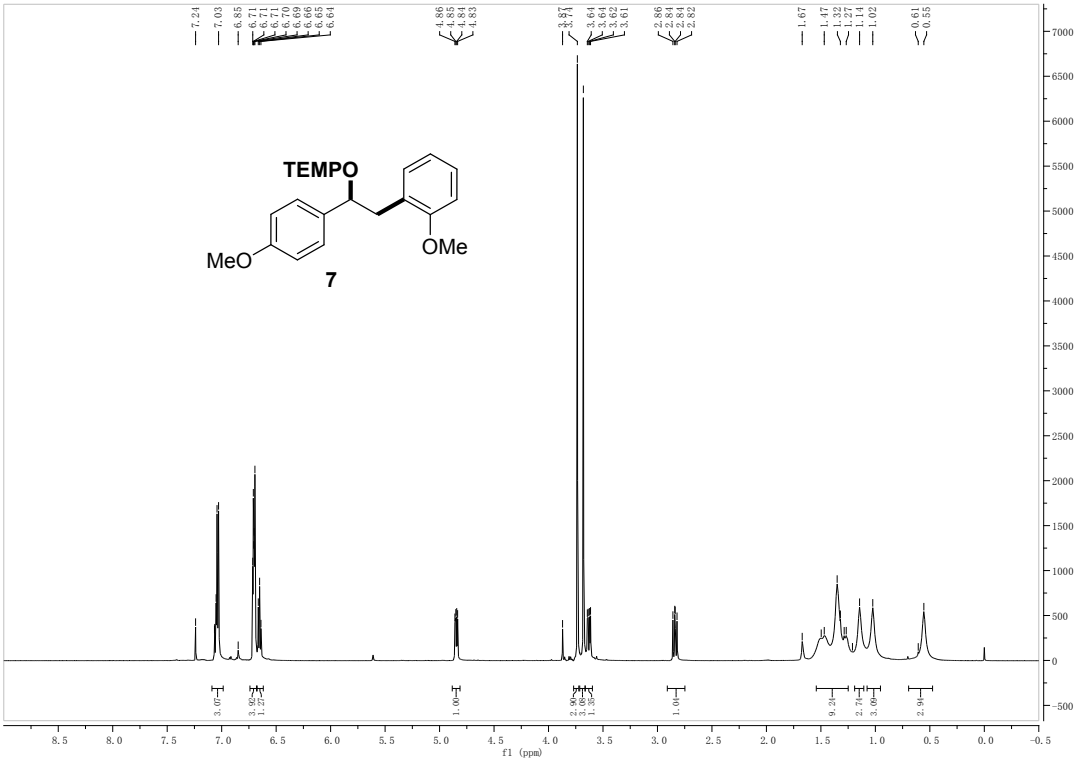

TEMPO
MeO
Me
4
f1 (ppm)

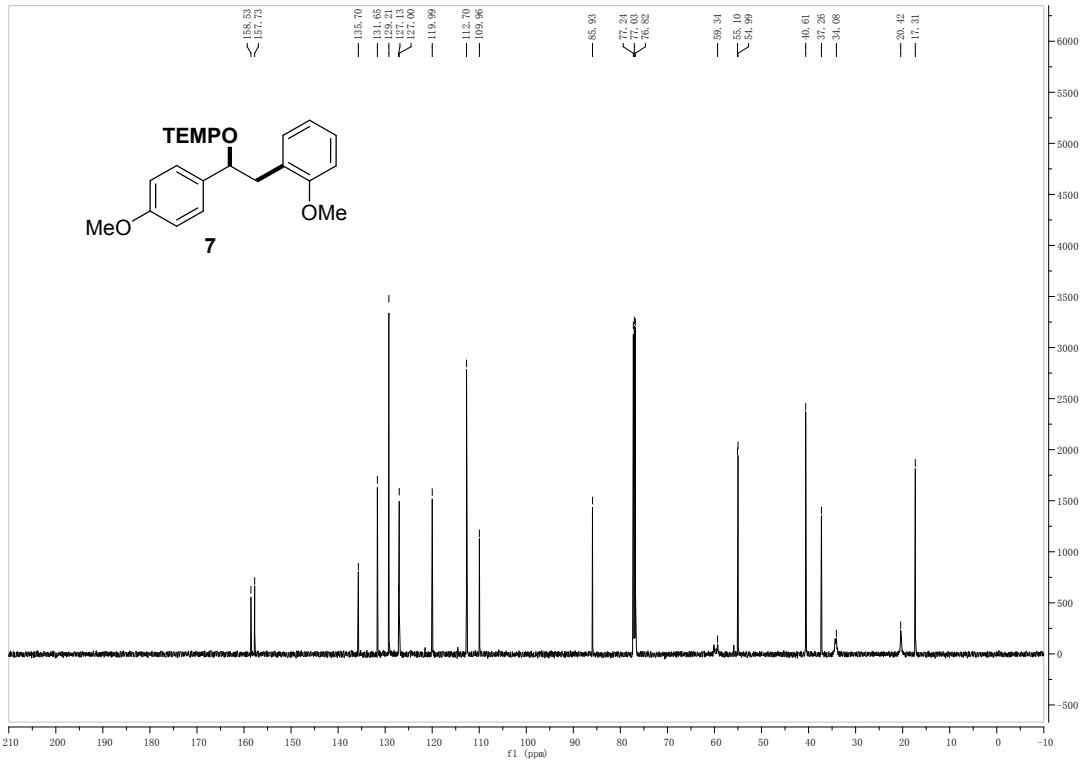

TEMPO
Me
MeO
5
f1 (ppm)

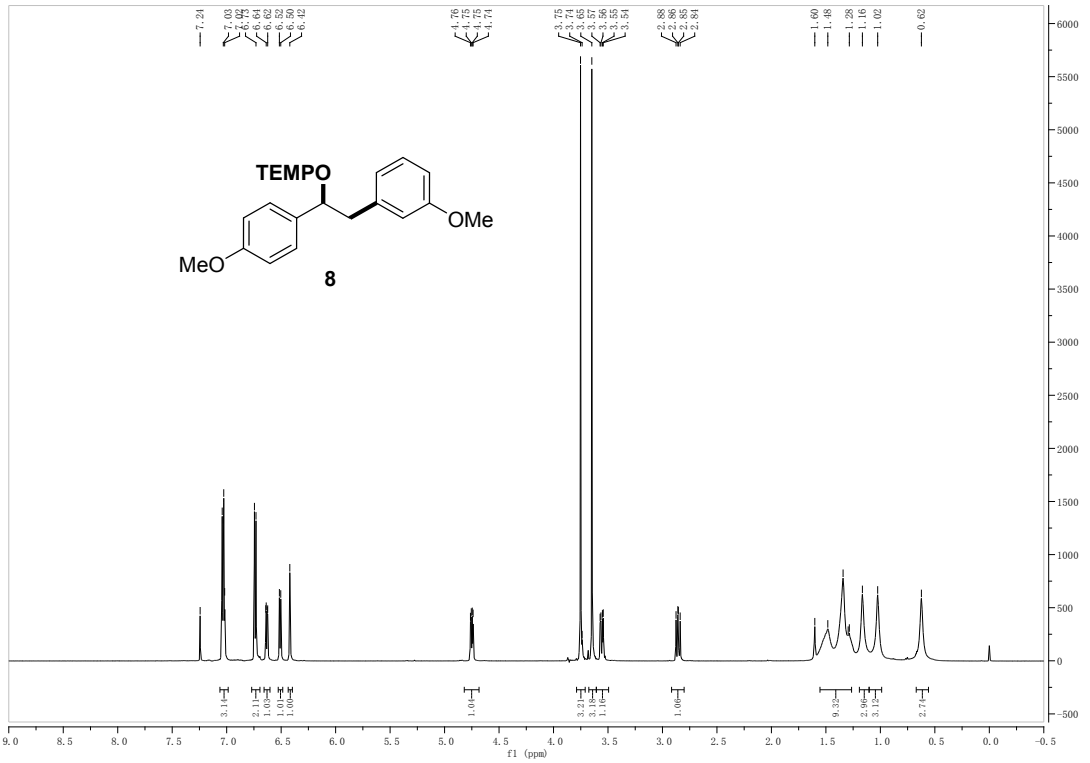

TEMPO
Me
MeO
5
f1 (ppm)

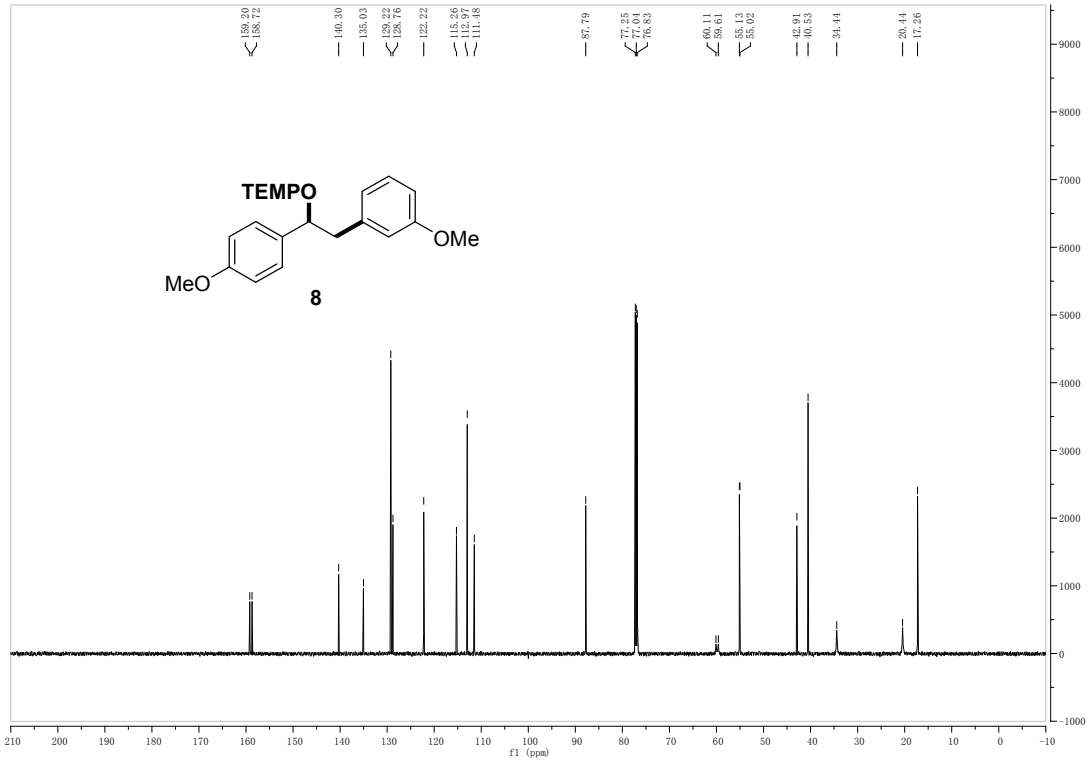

7.25
7.02
6.92
6.91
6.77
6.76
6.74
6.73
4.73
4.72
4.71
4.71
3.76
3.55
3.55
3.53
3.53
2.86
2.85
2.84
2.82
2.24
1.58
1.48
1.46
1.45
1.43
1.35
1.29
1.16
1.02
0.63
0.61
TEMPO
Me
MeO
6
f1 (ppm)

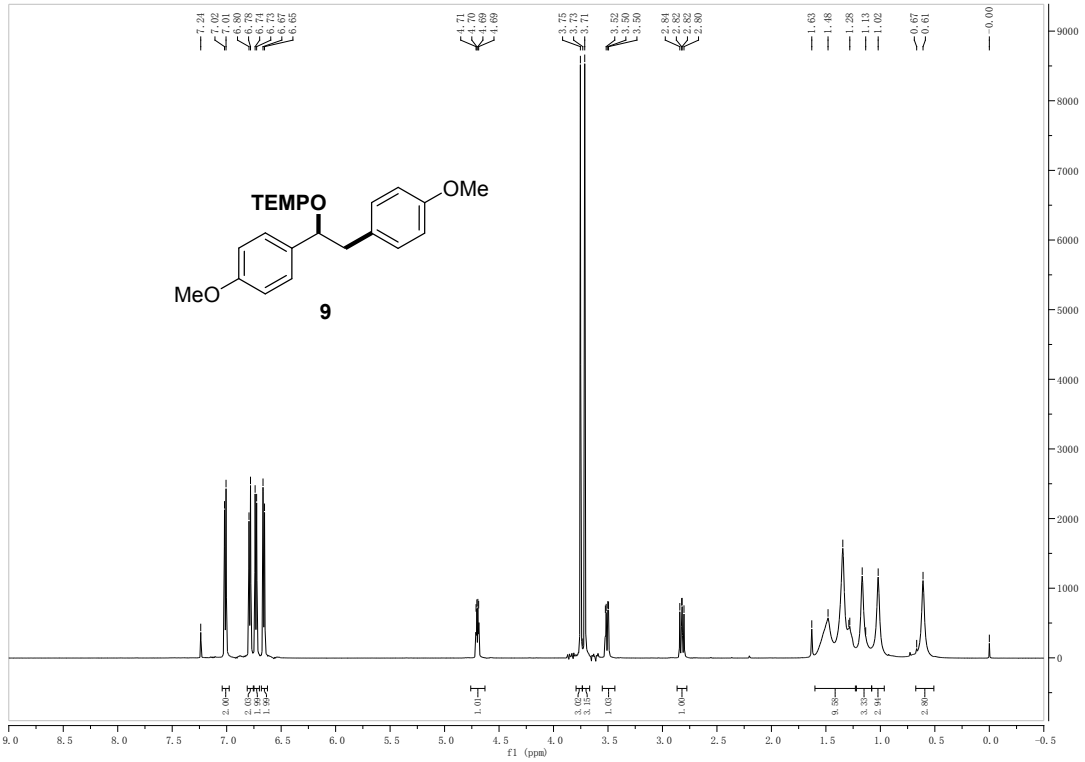

158.63
135.55
135.12
135.03
129.56
129.27
128.56
112.91
88.01
77.22
77.01
76.80
60.09
55.10
42.25
40.53
34.43
20.98
20.40
17.27
TEMPO
Me
MeO
6
f1 (ppm)

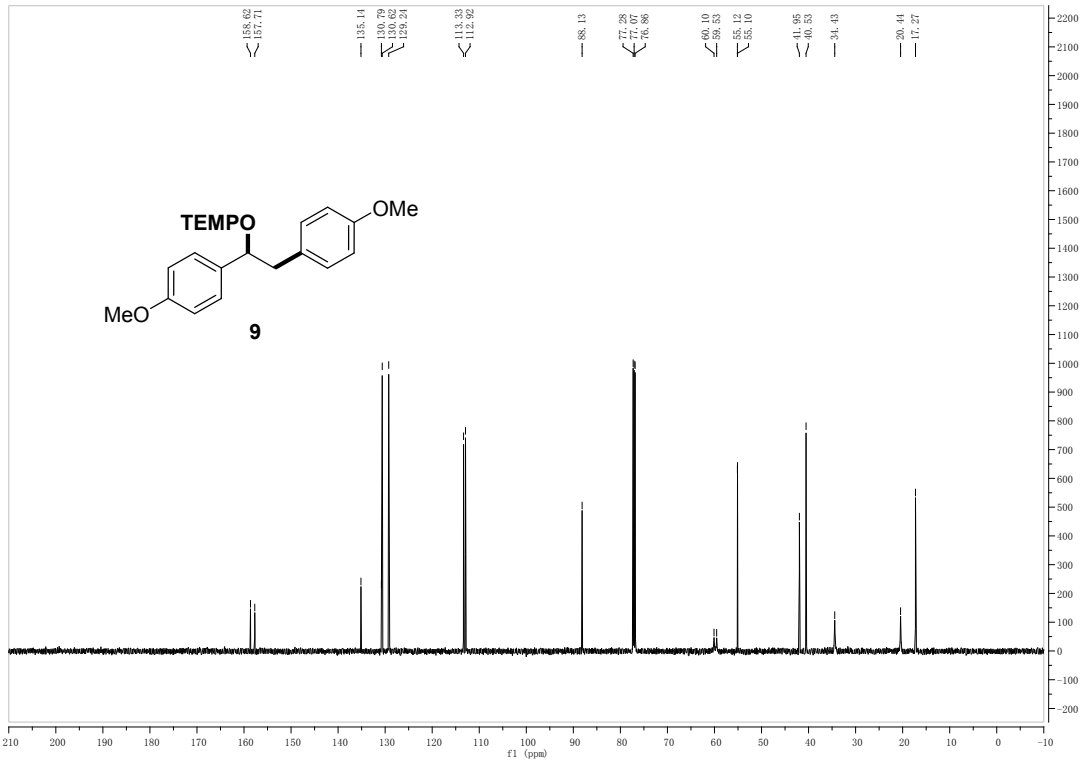

TEMPO
MeO
OMe
7
f1 (ppm)

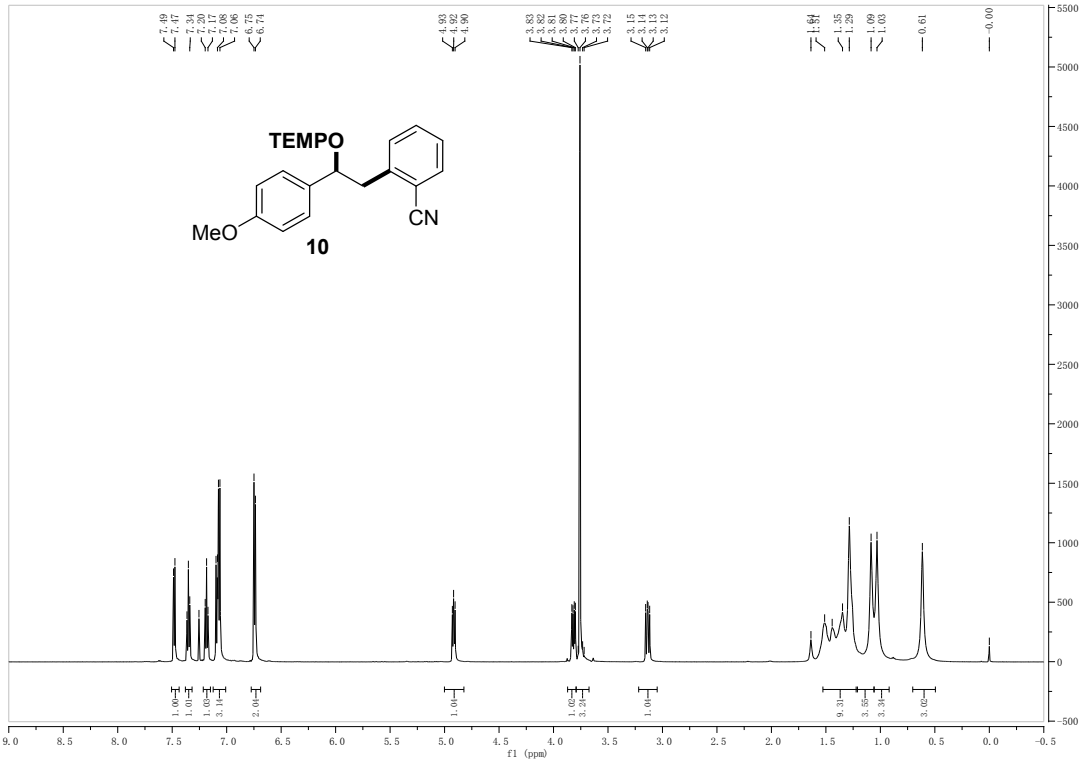

TEMPO
MeO
OMe
7
f1 (ppm)

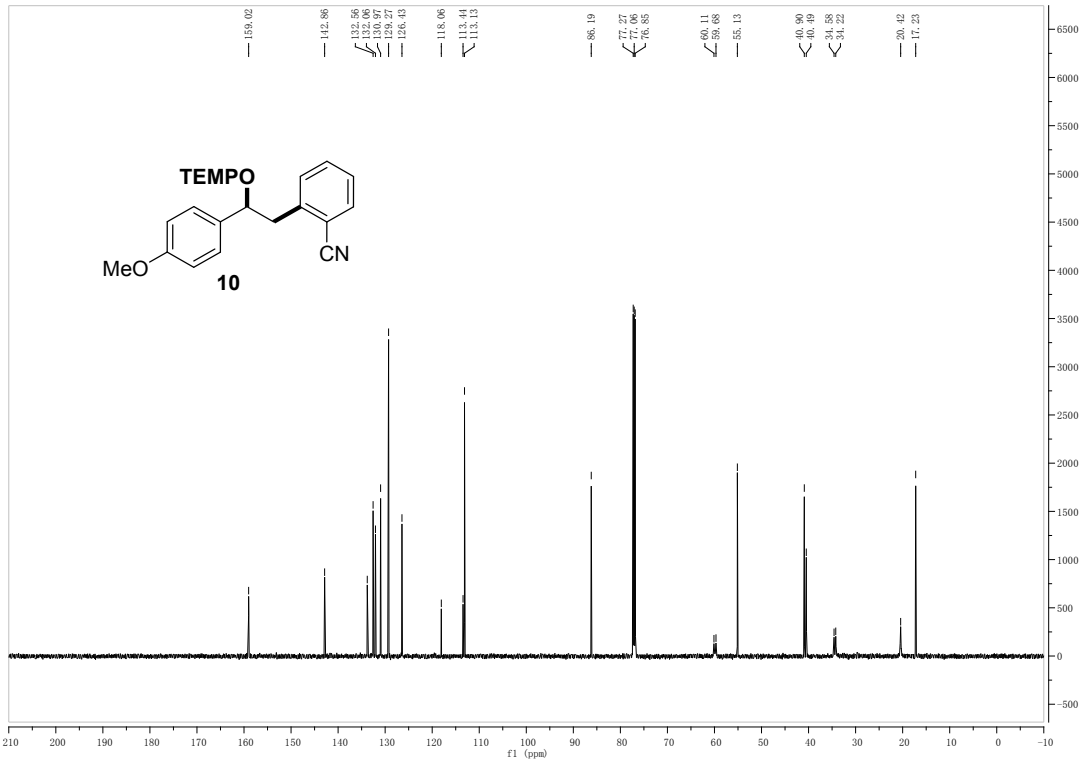

TEMPO
OMe
MeO
8
f1 (ppm)

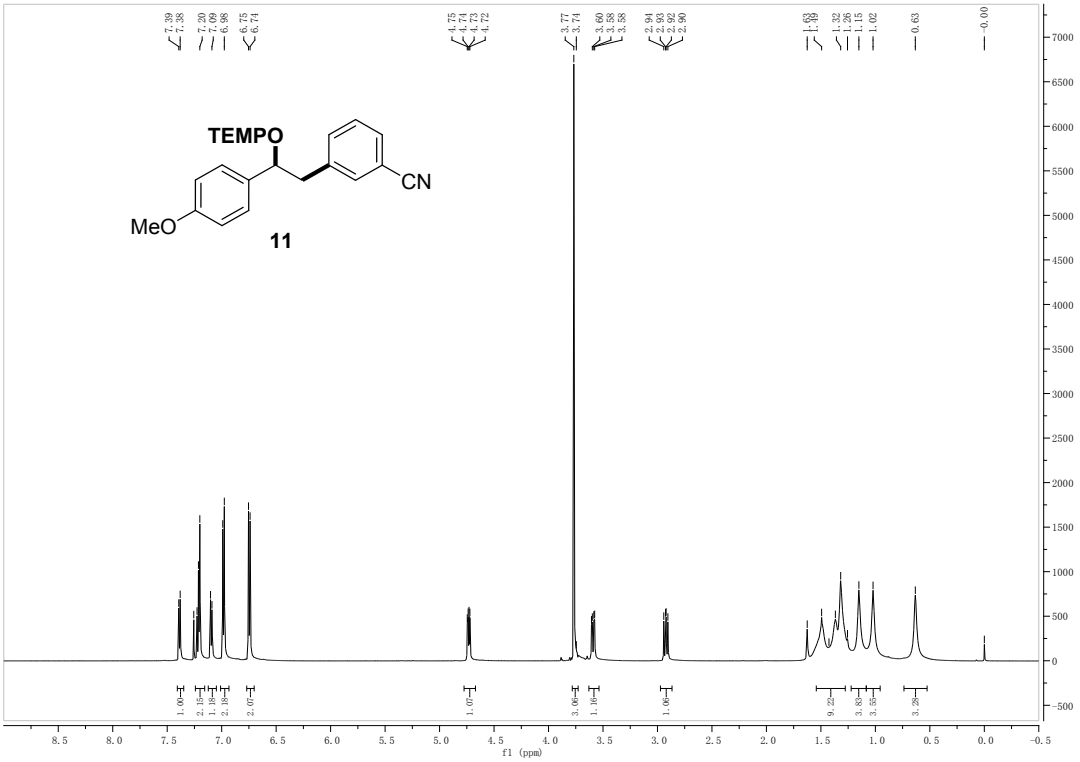

TEMPO
OMe
MeO
8
f1 (ppm)

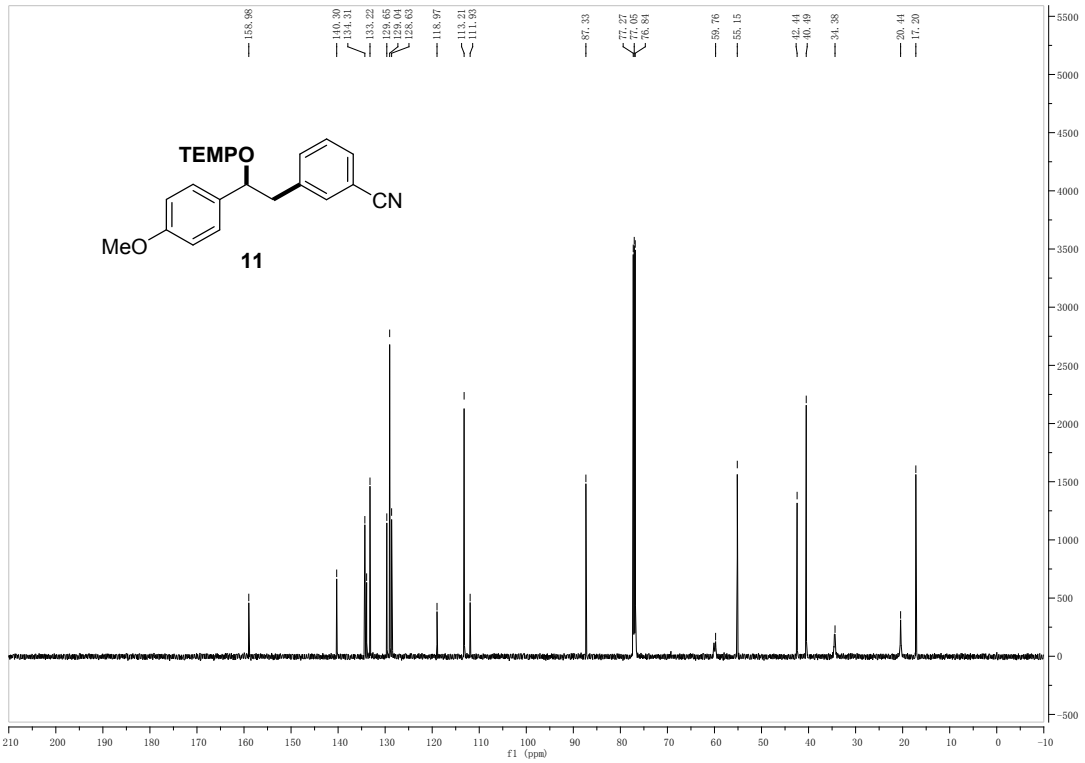

TEMPO
OMe
MeO
9
f1 (ppm)

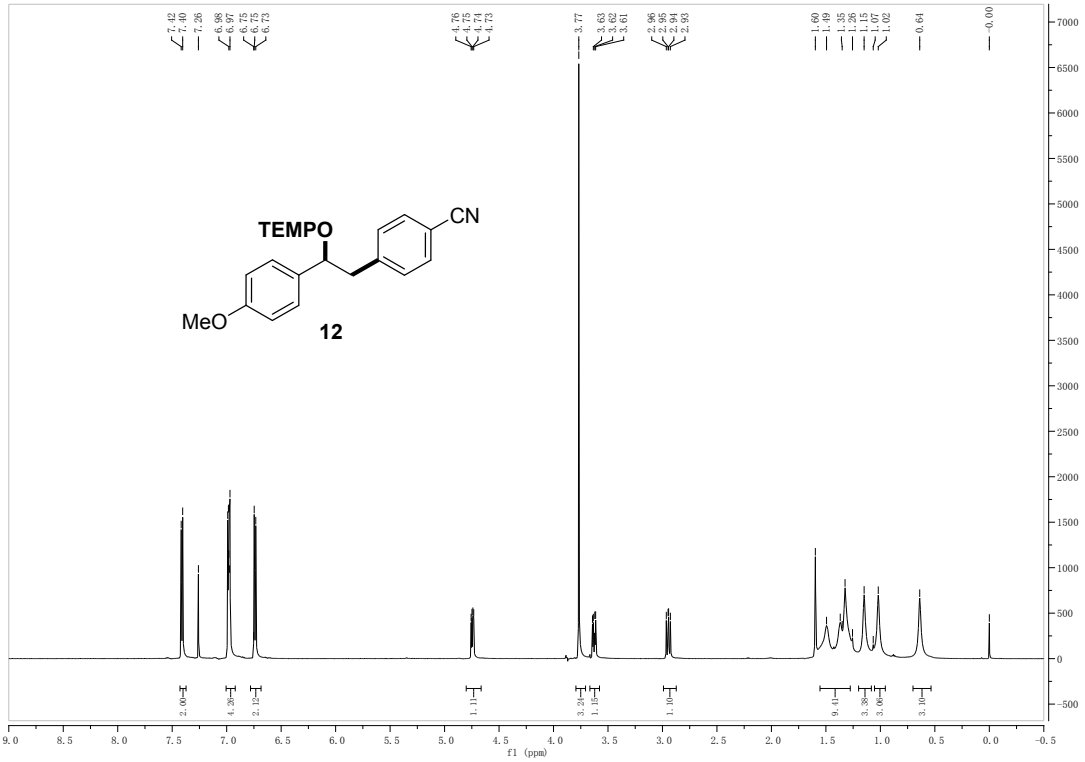

TEMPO
OMe
MeO
9
f1 (ppm)

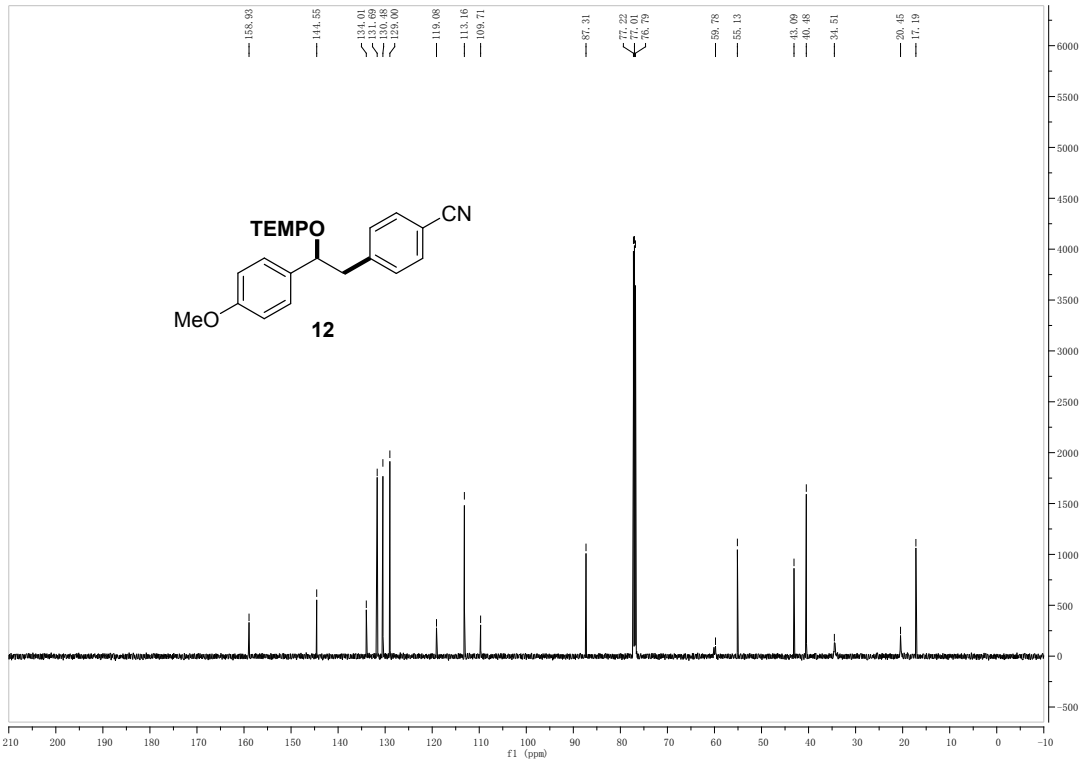

TEMPO
MeO
CN
10
f1 (ppm)

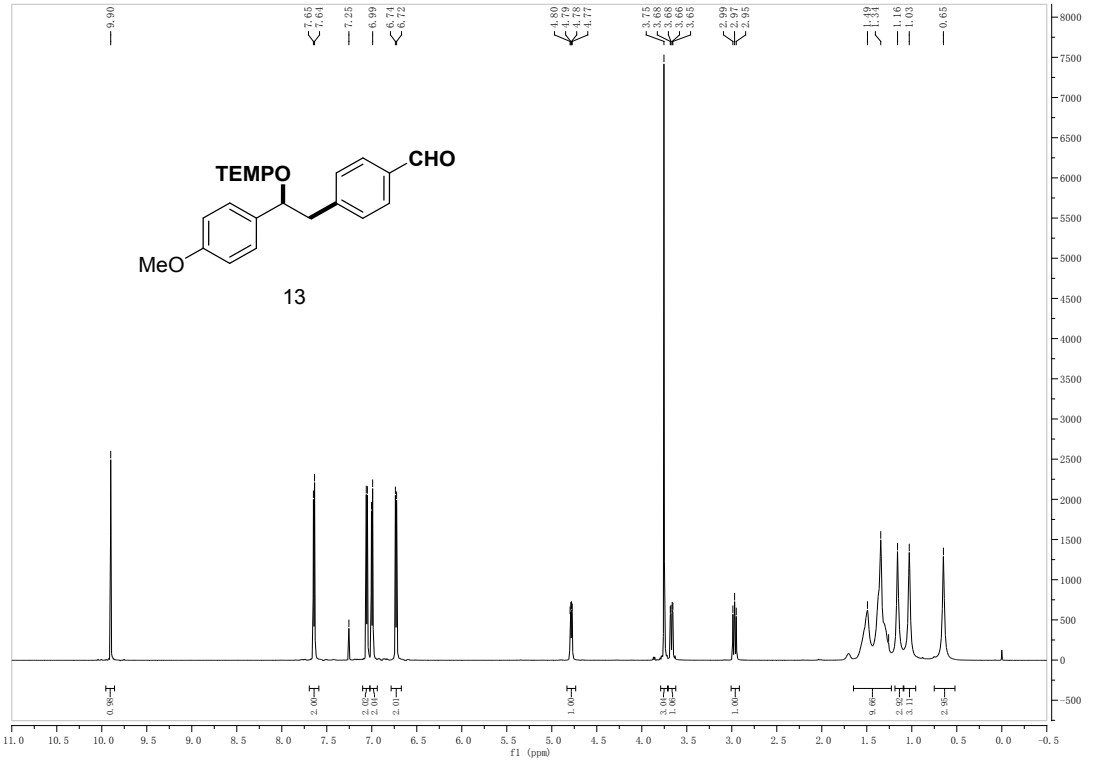

TEMPO
MeO
CN
10
f1 (ppm)

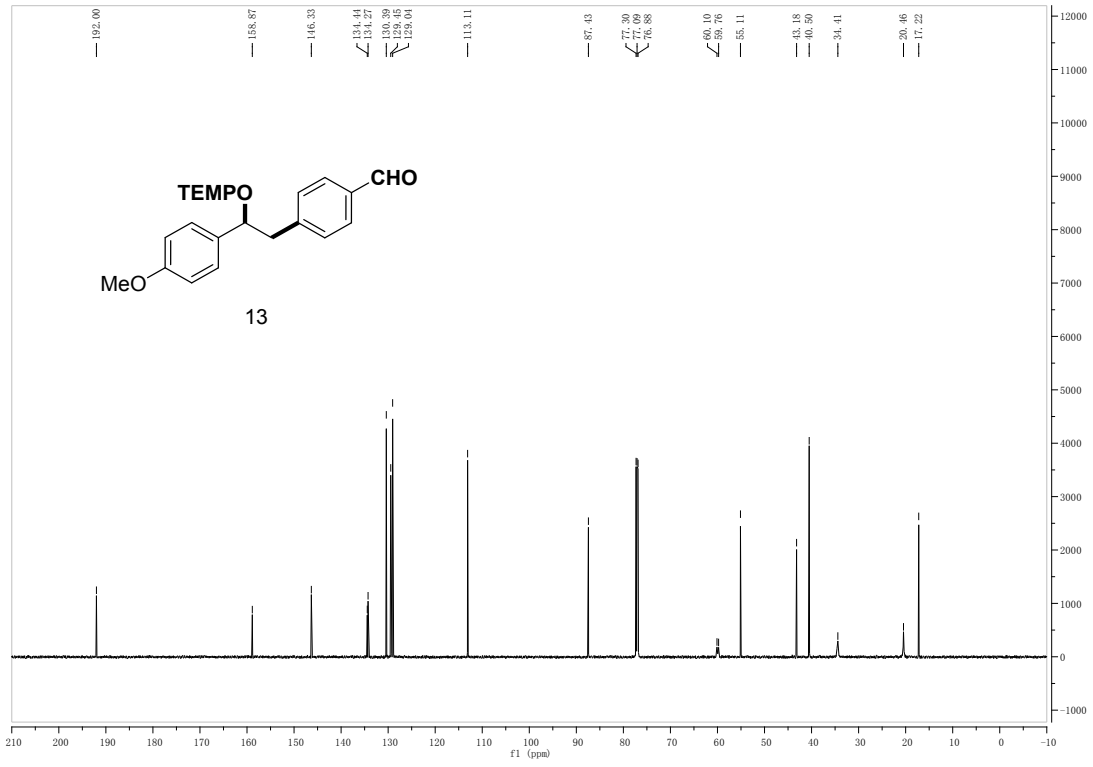

TEMPO
CN
MeO
11
f1 (ppm)

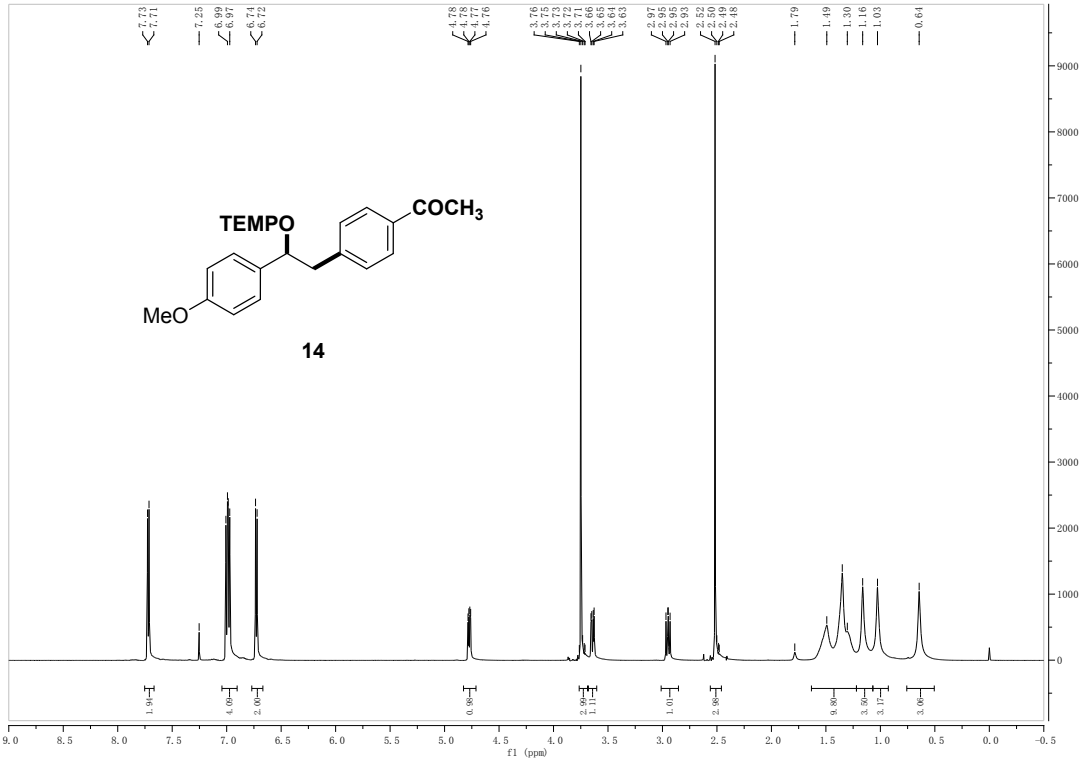

TEMPO
CN
MeO
11
f1 (ppm)

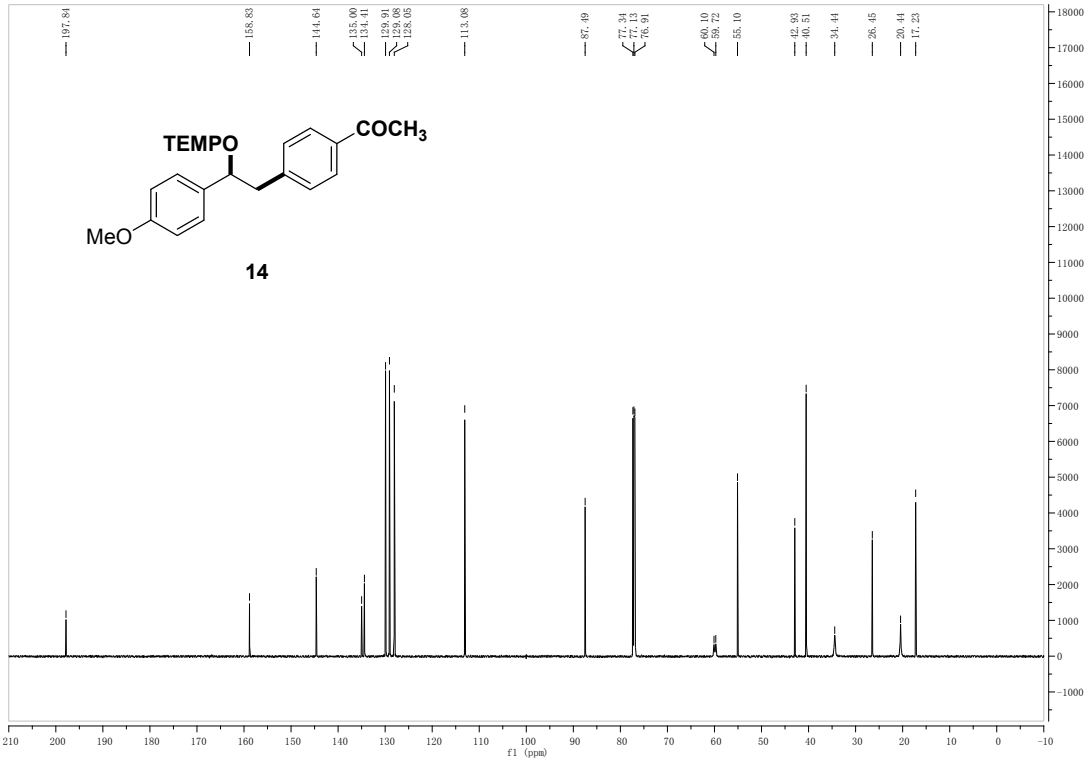

TEMPO
CN
MeO
12
f1 (ppm)

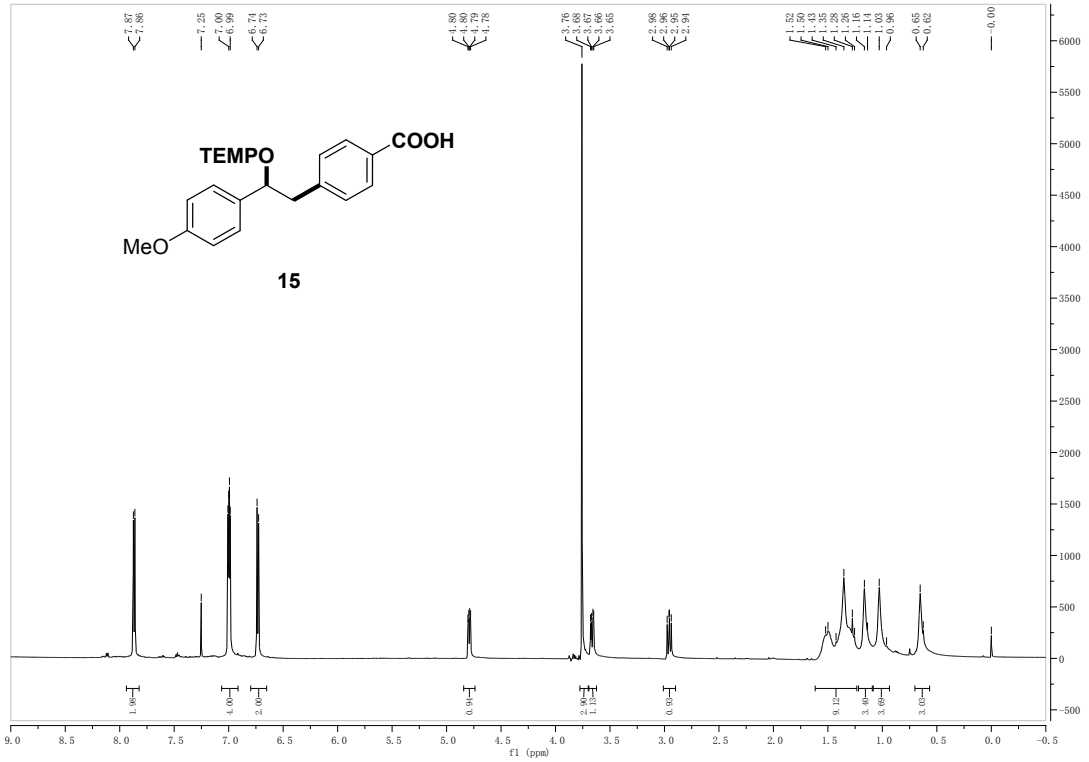

TEMPO
CN
MeO
12
f1 (ppm)

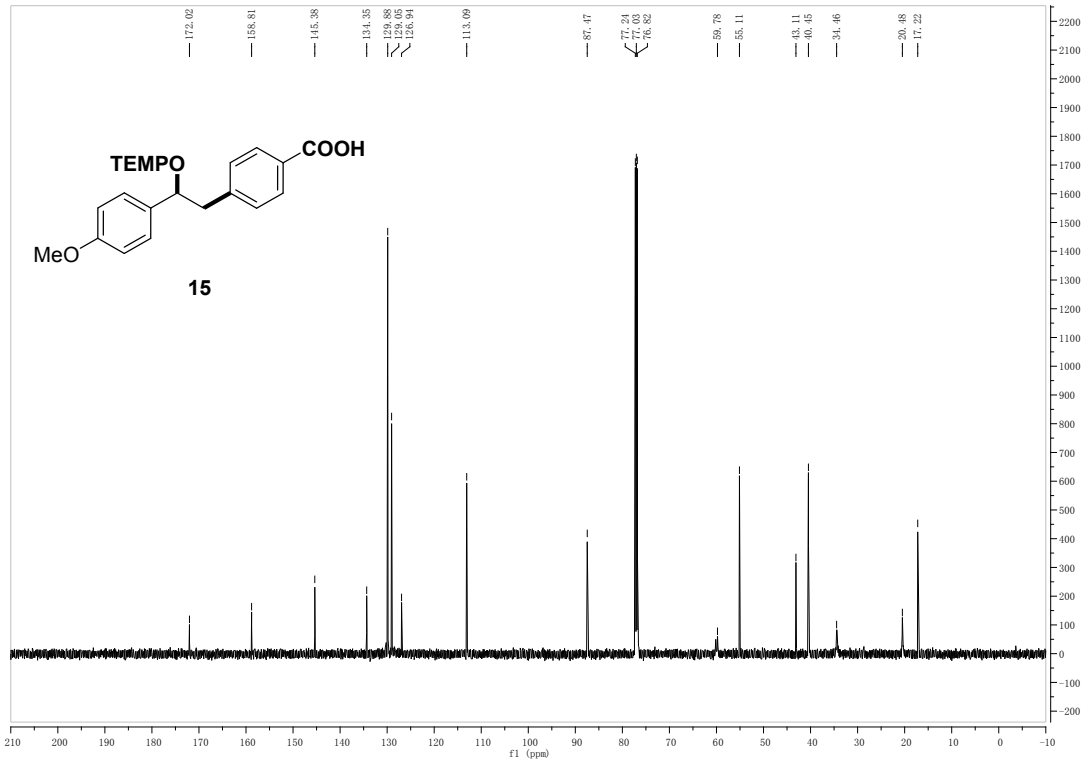

9.90
7.65
7.64
7.25
6.99
6.74
6.72
4.80
4.79
4.78
4.77
3.75
3.68
3.68
3.66
3.65
2.99
2.97
2.95
1.49
1.34
1.16
1.03
0.65
TEMPO
CHO
MeO
13
f1 (ppm)

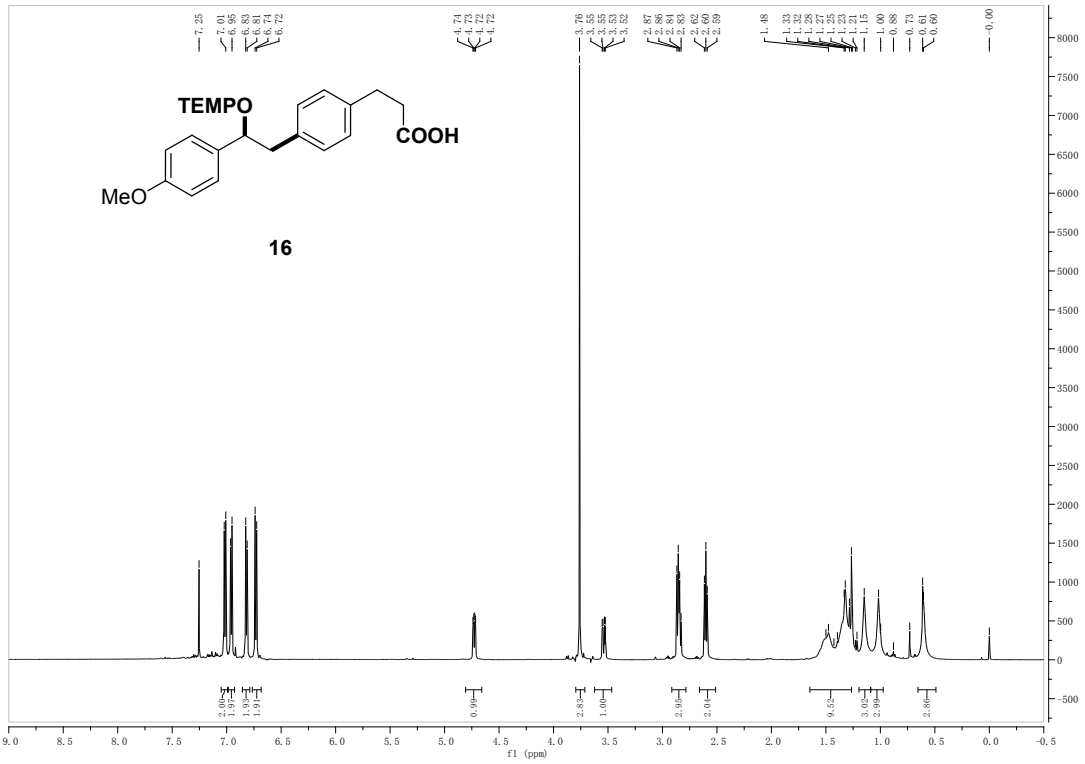

192.00
158.87
146.33
134.44
134.27
130.39
129.45
129.04
113.11
87.43
77.30
77.09
76.88
60.10
59.76
55.11
43.18
40.59
34.41
20.46
17.22
TEMPO
CHO
MeO
13
f1 (ppm)

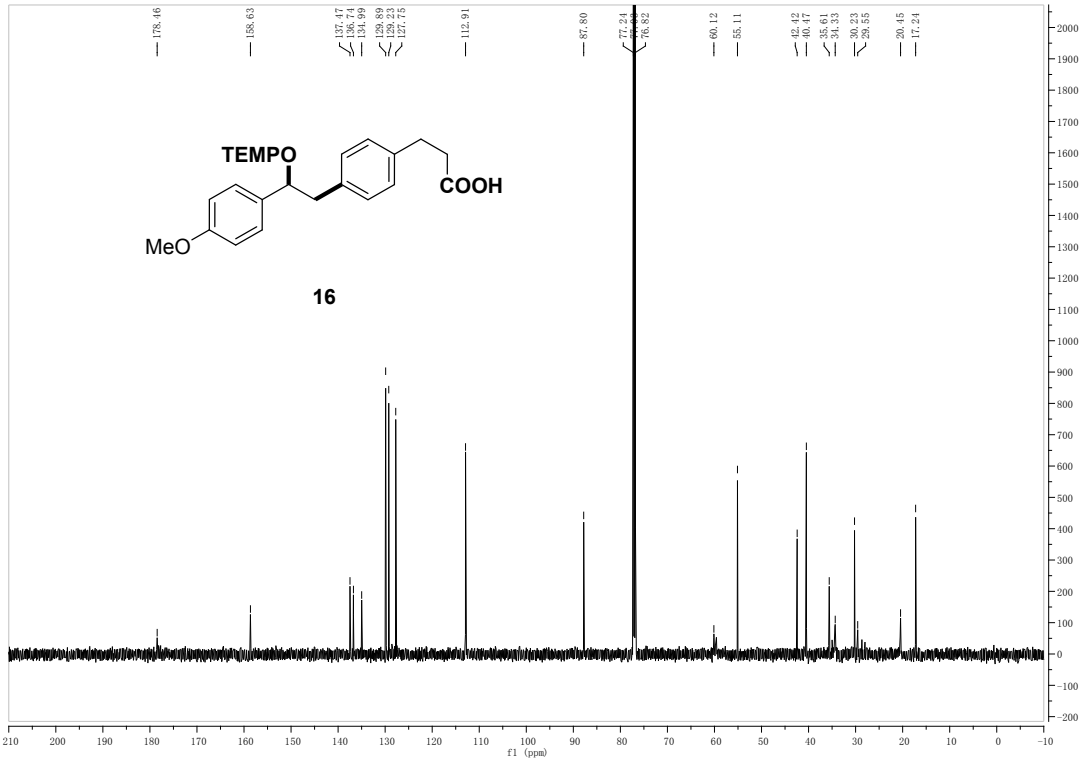

TEMPO
COCH3
MeO
14
f1 (ppm)

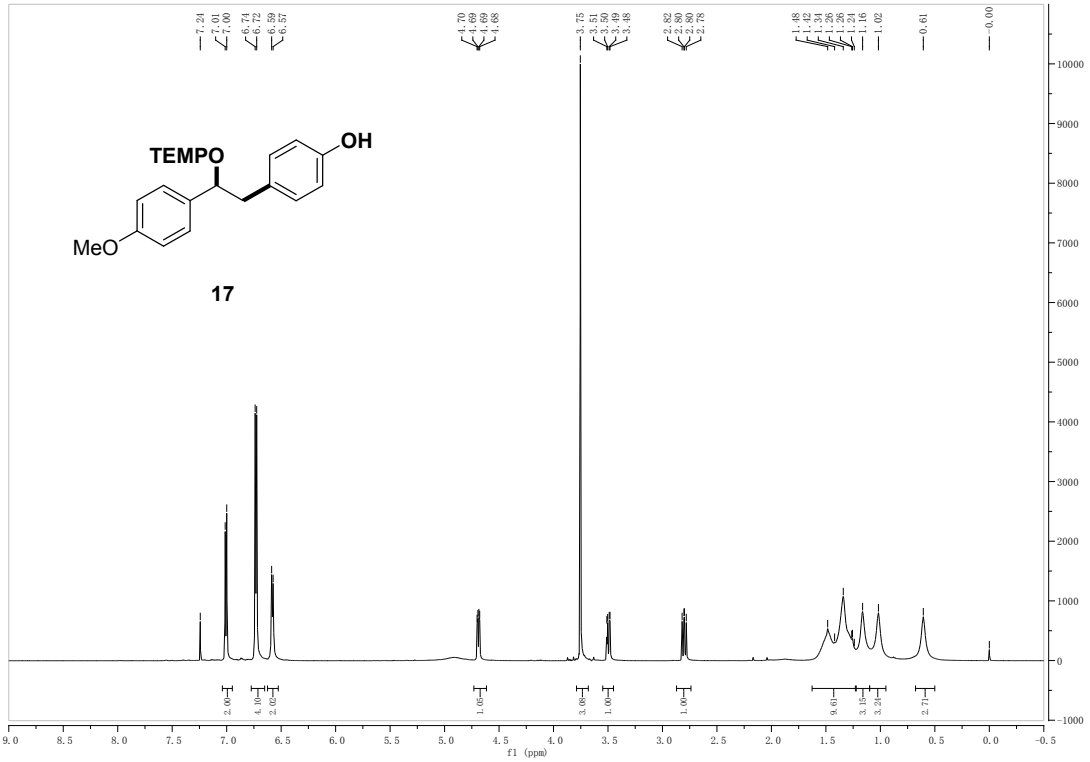

TEMPO
COCH3
MeO
14
f1 (ppm)

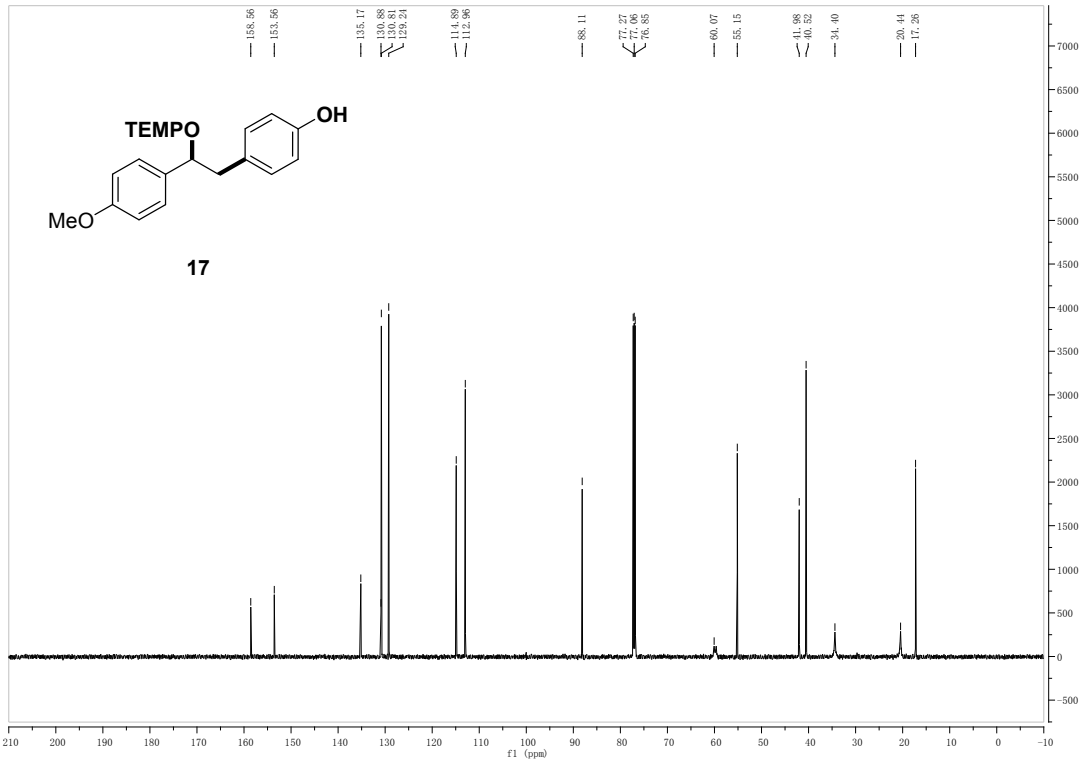

TEMPO
COOH
MeO
15
f1 (ppm)

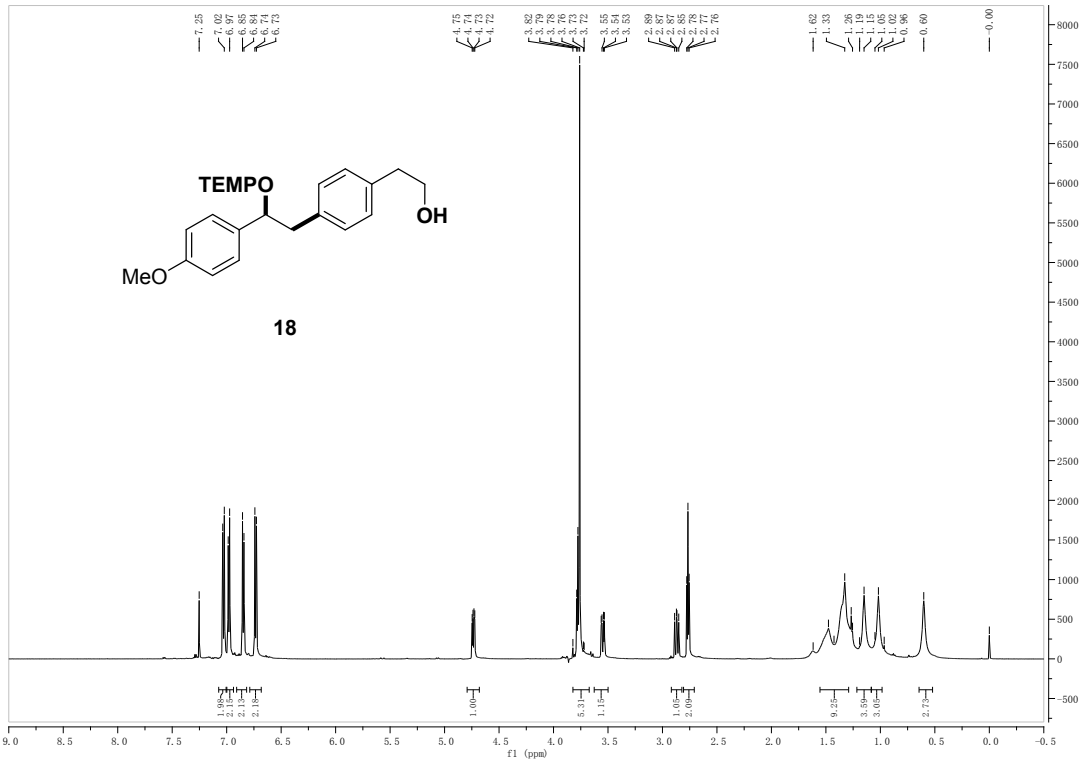

TEMPO
COOH
MeO
15
f1 (ppm)

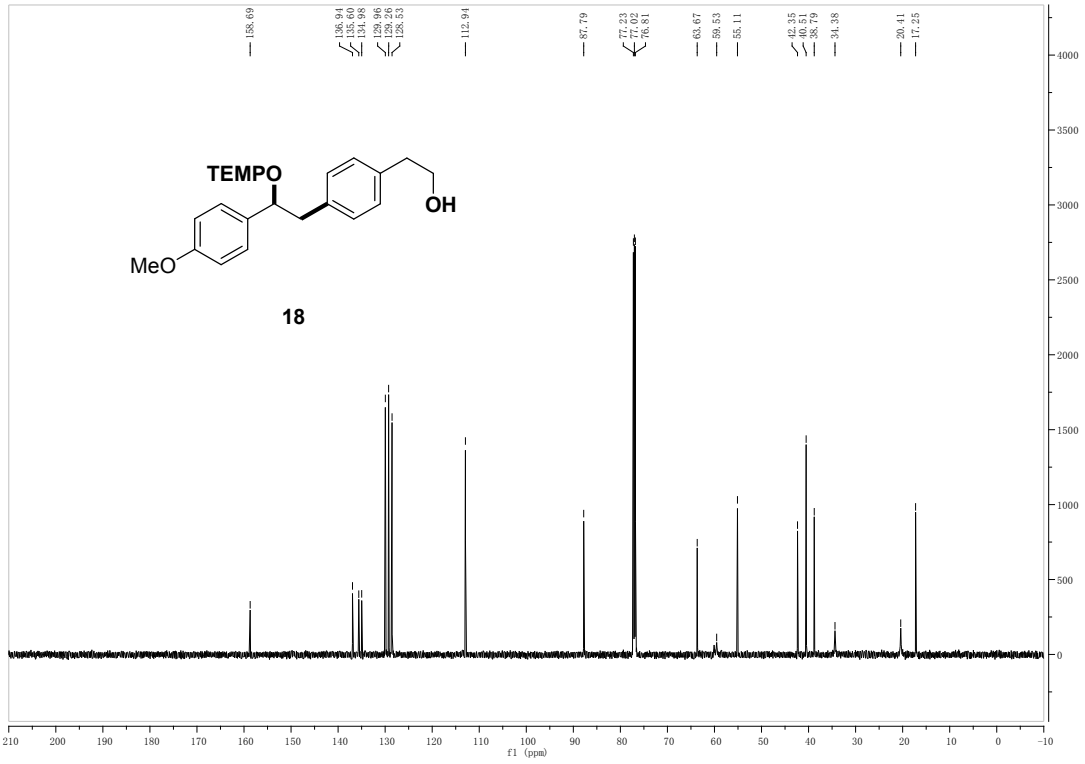

TEMPO
COOH
MeO
16
f1 (ppm)

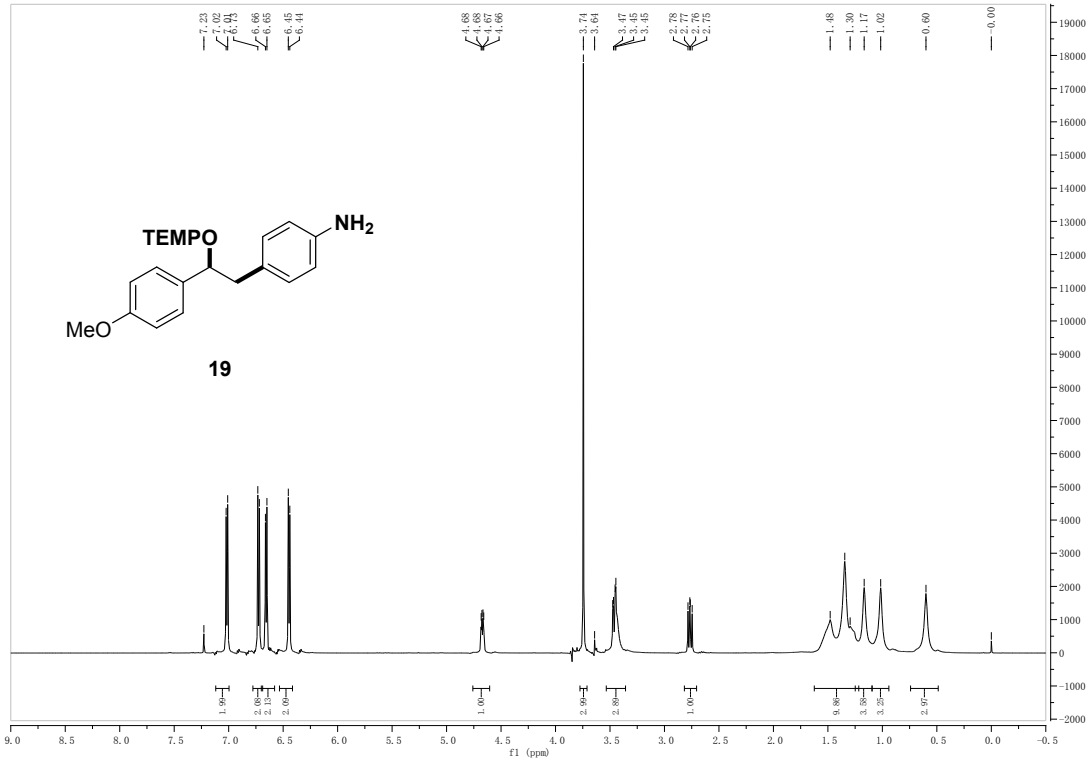

TEMPO
COOH
MeO
16
f1 (ppm)

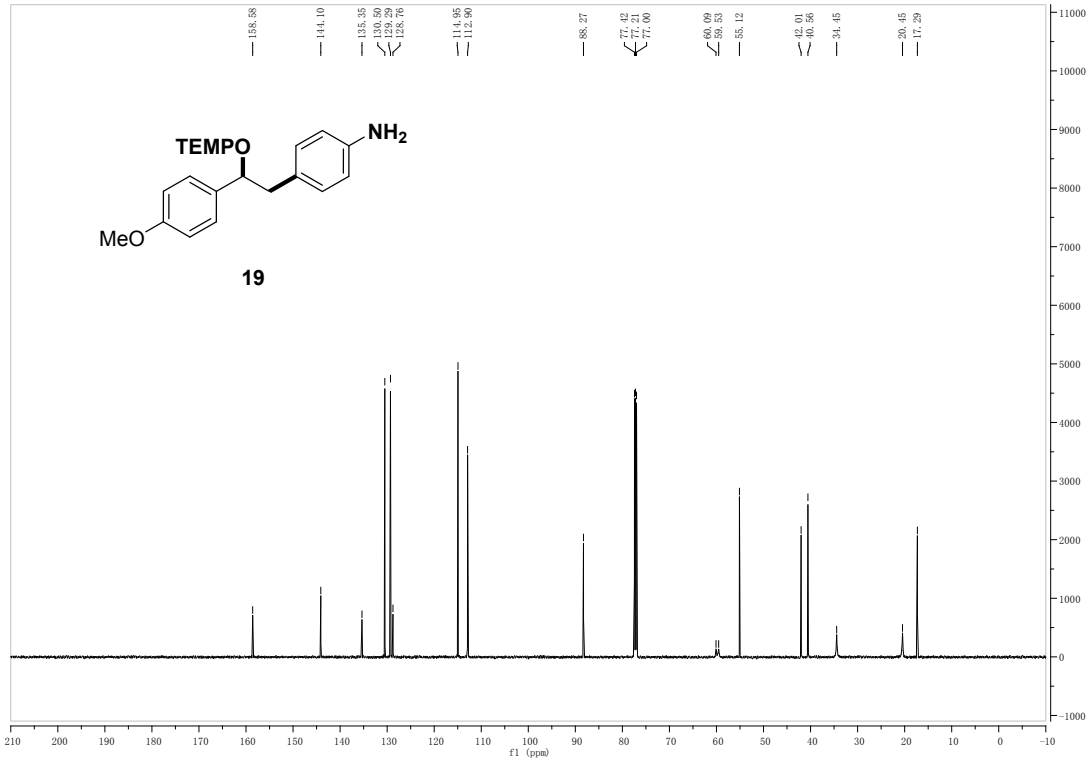

TEMPO
OH
MeO
17
f1 (ppm)

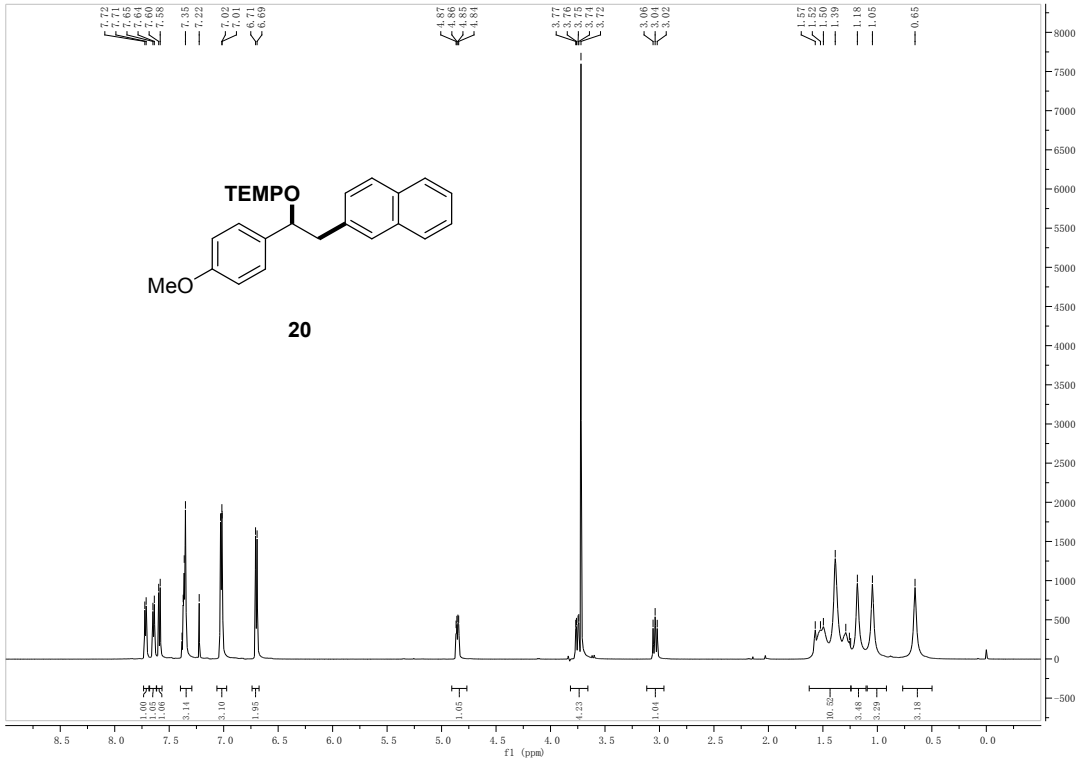

TEMPO
OH
MeO
17
f1 (ppm)

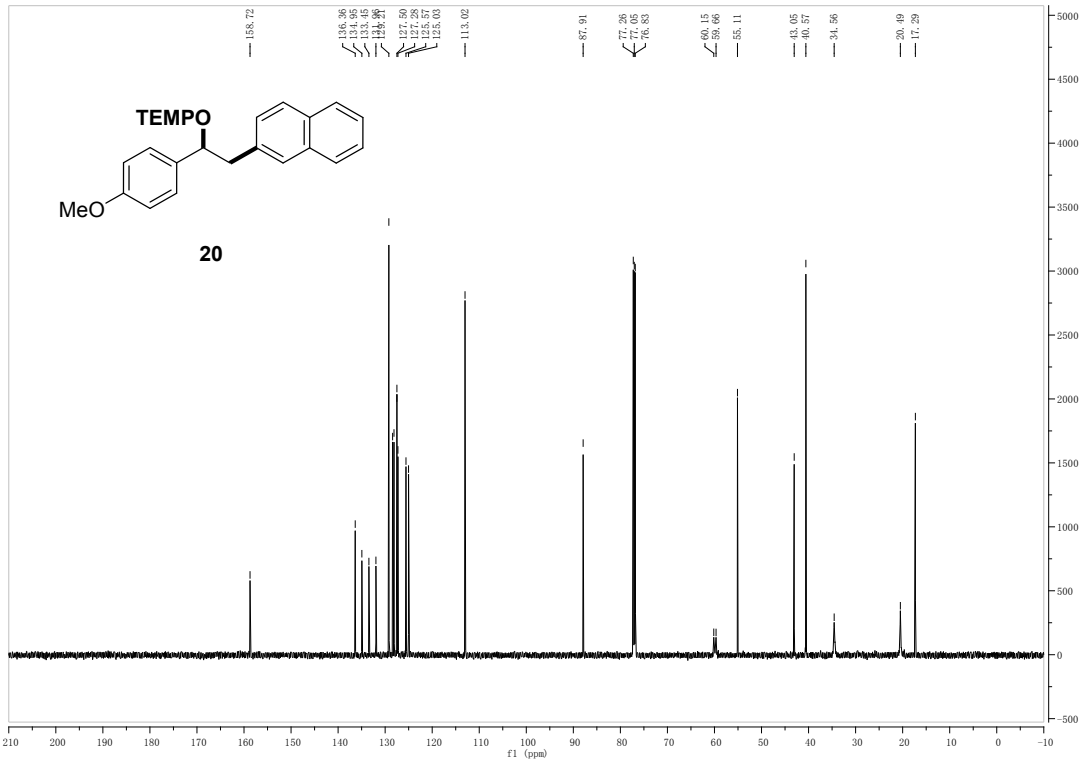

TEMPO
OH
MeO
18
f1 (ppm)

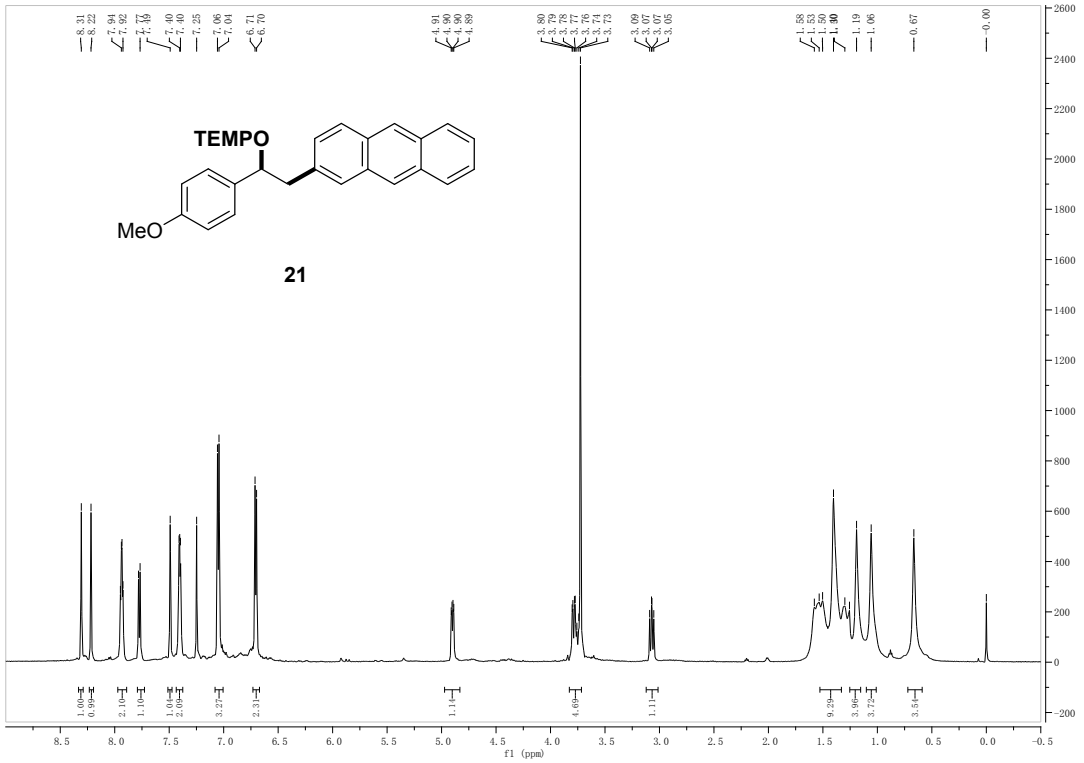

TEMPO
OH
MeO
18
f1 (ppm)

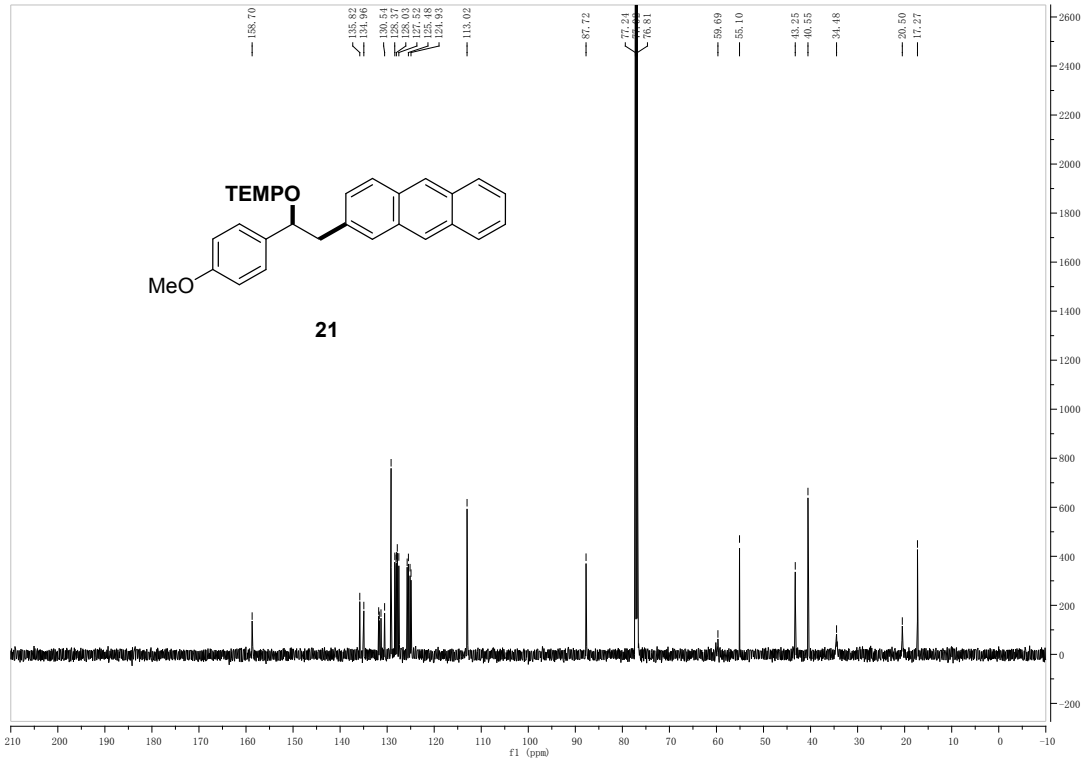

TEMPO
NH2
MeO
19
f1 (ppm)

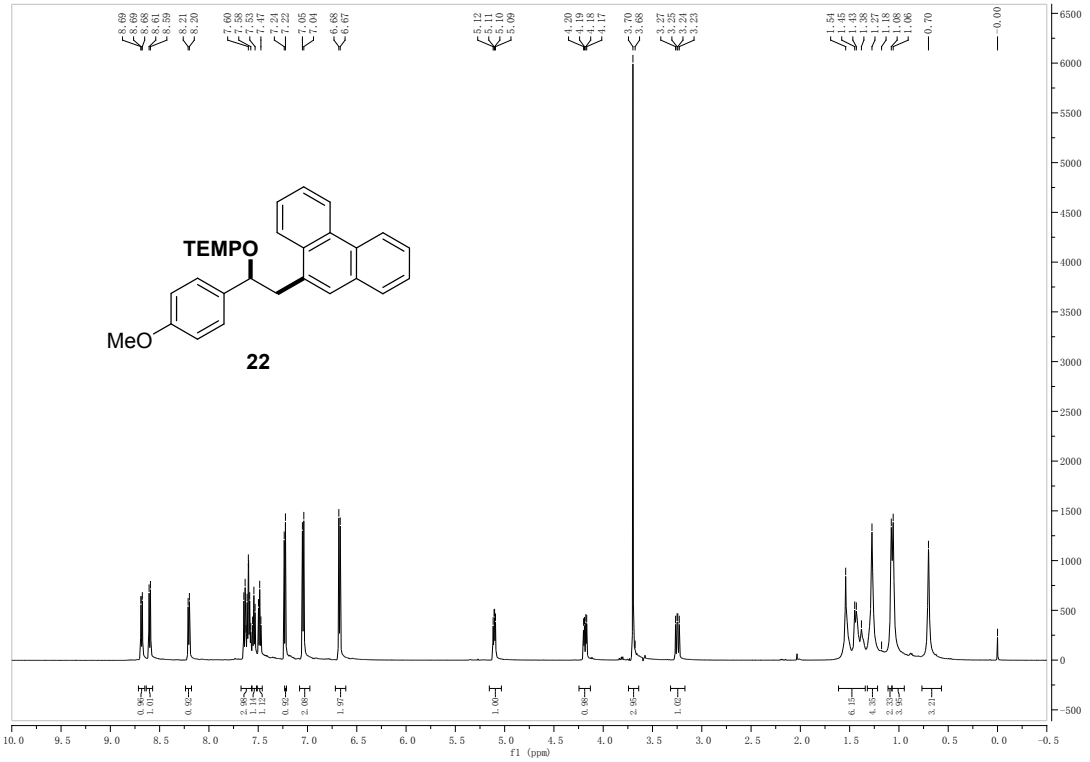

TEMPO
NH2
MeO
19
f1 (ppm)

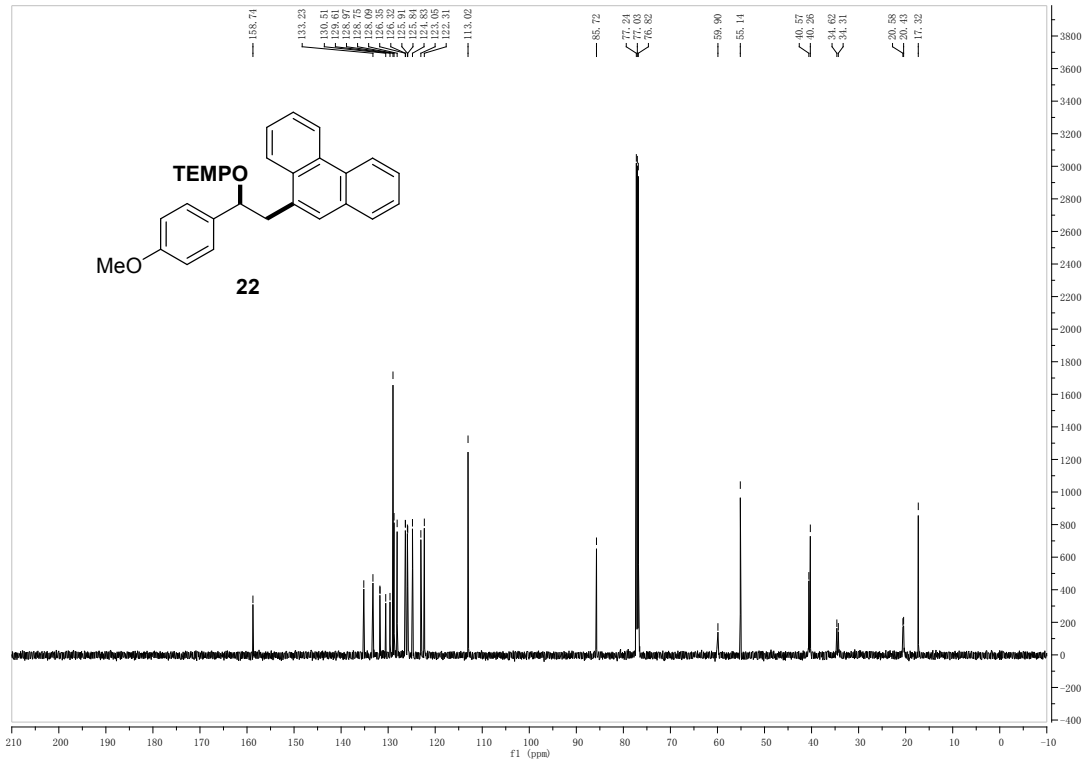

TEMPO
MeO
20

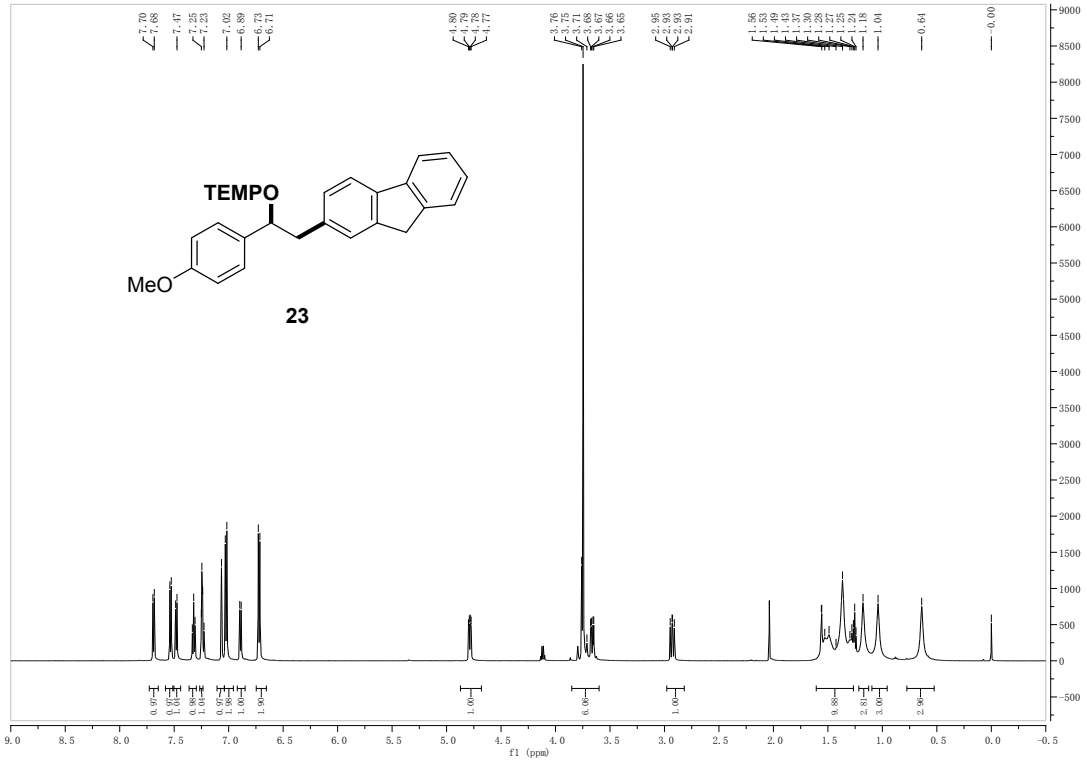

TEMPO
MeO
20

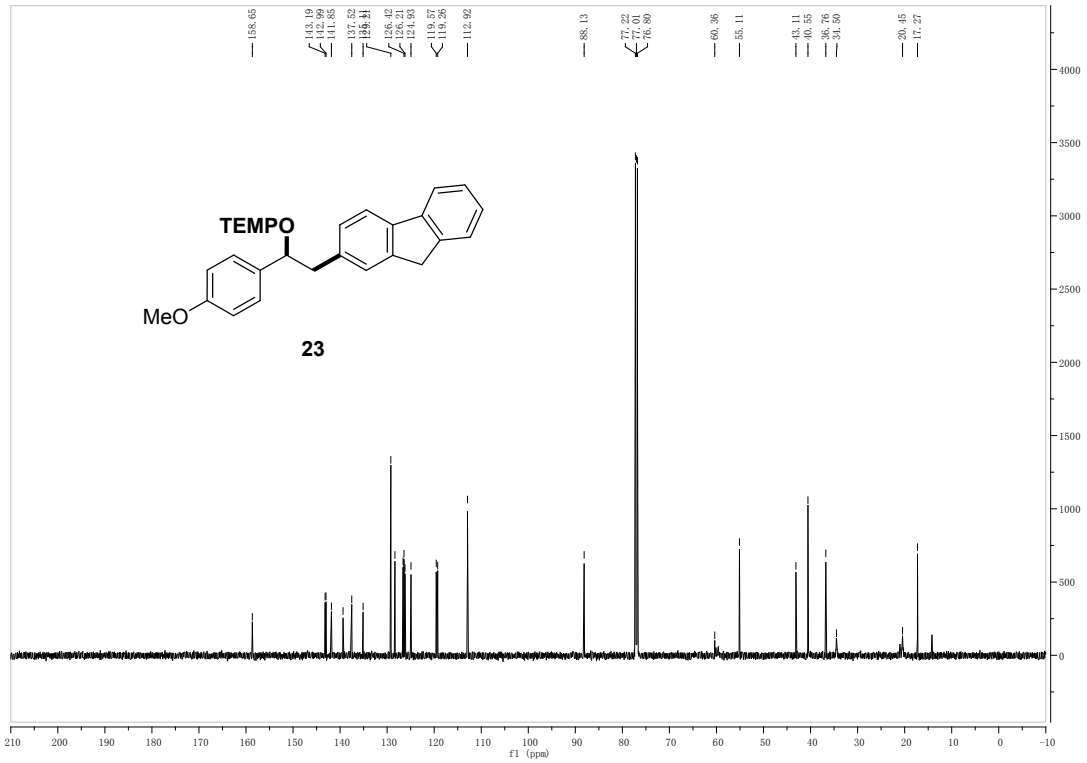

TEMPO
MeO
21
f1 (ppm)

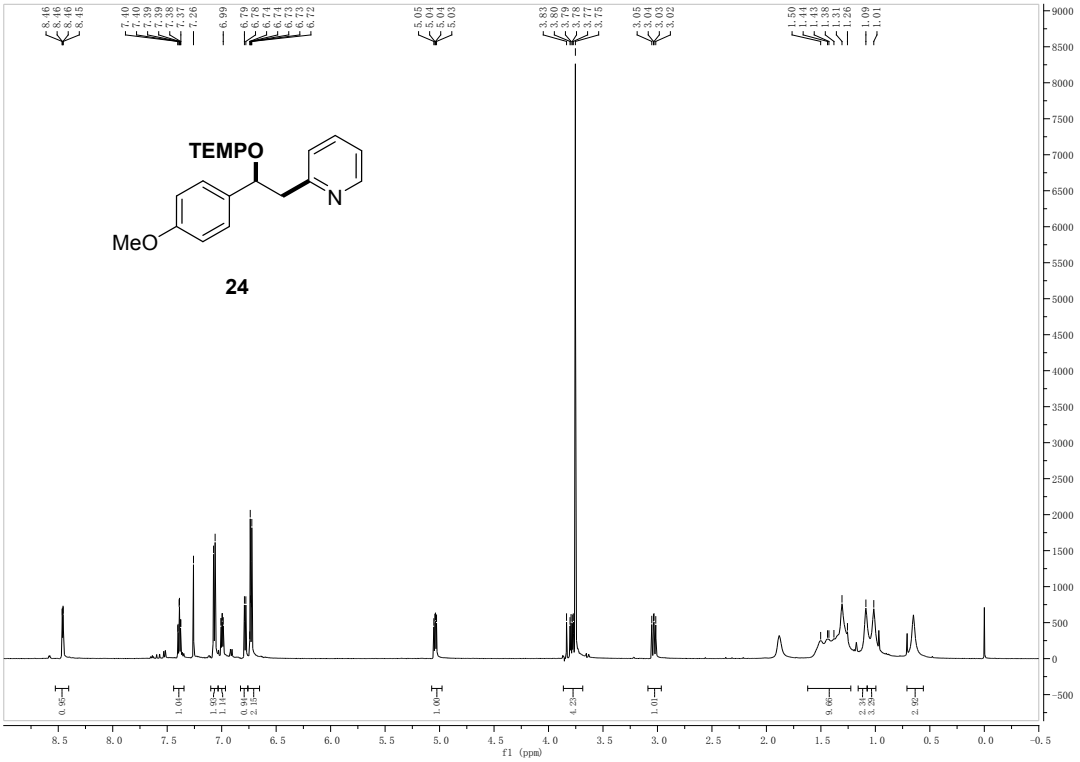

TEMPO
MeO
21
f1 (ppm)

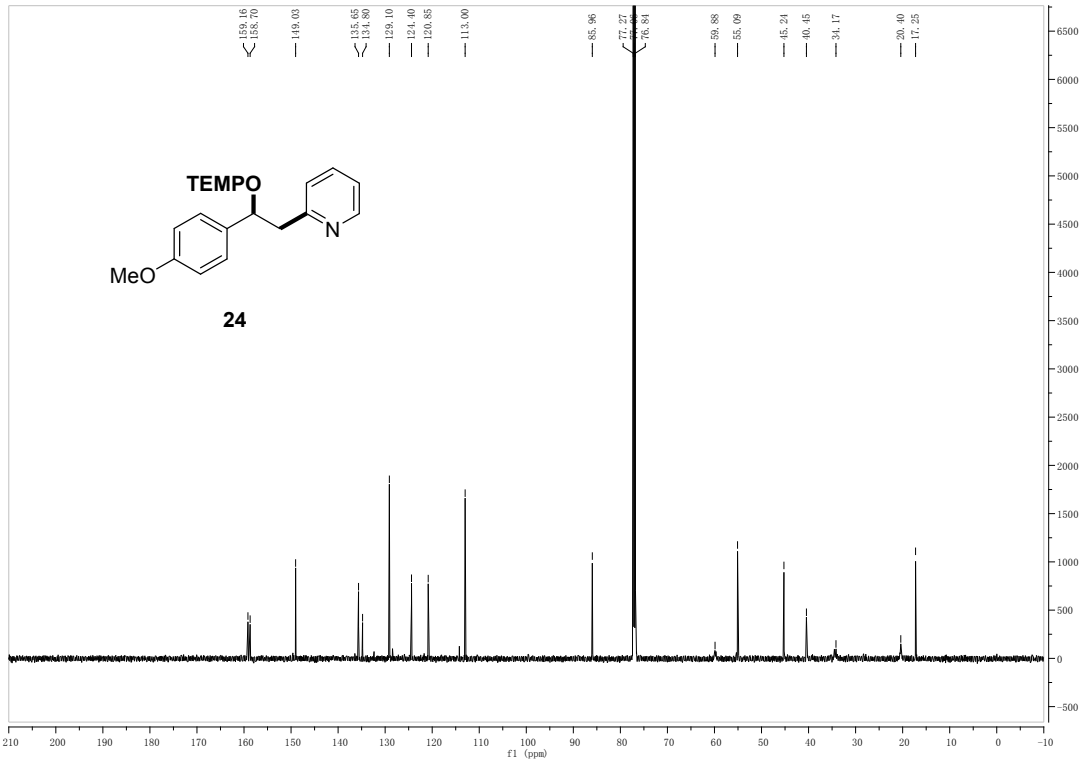

TEMPO
MeO
22
f1 (ppm)

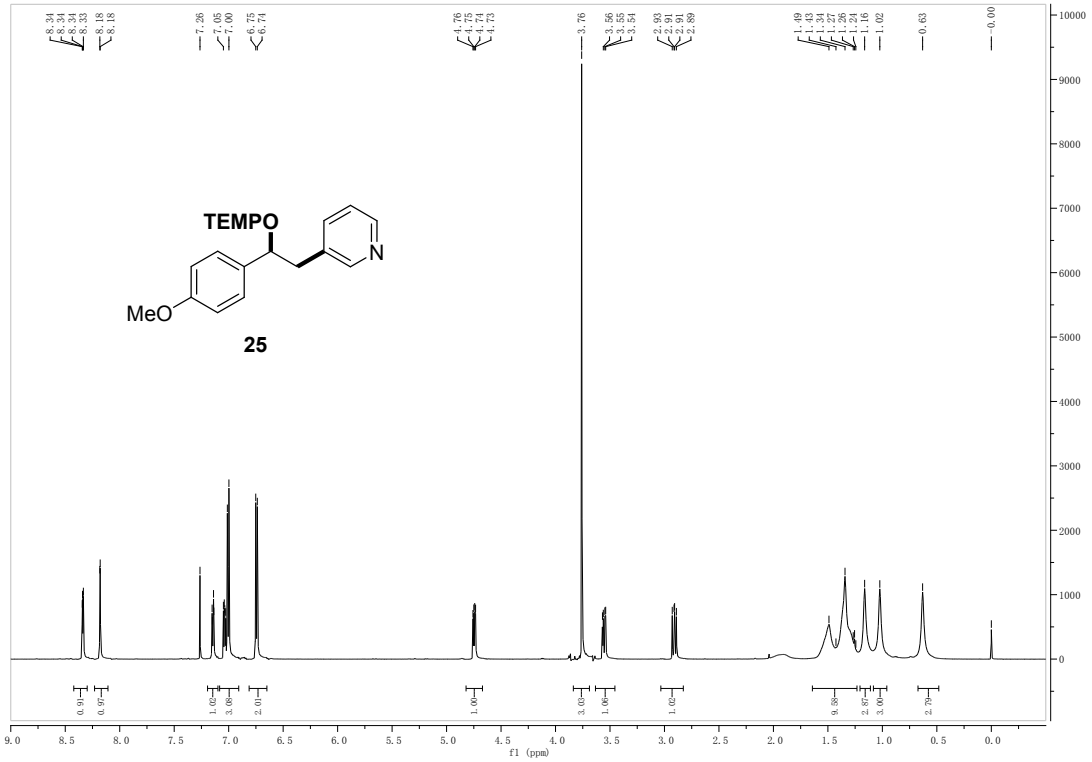

TEMPO
MeO
22
f1 (ppm)

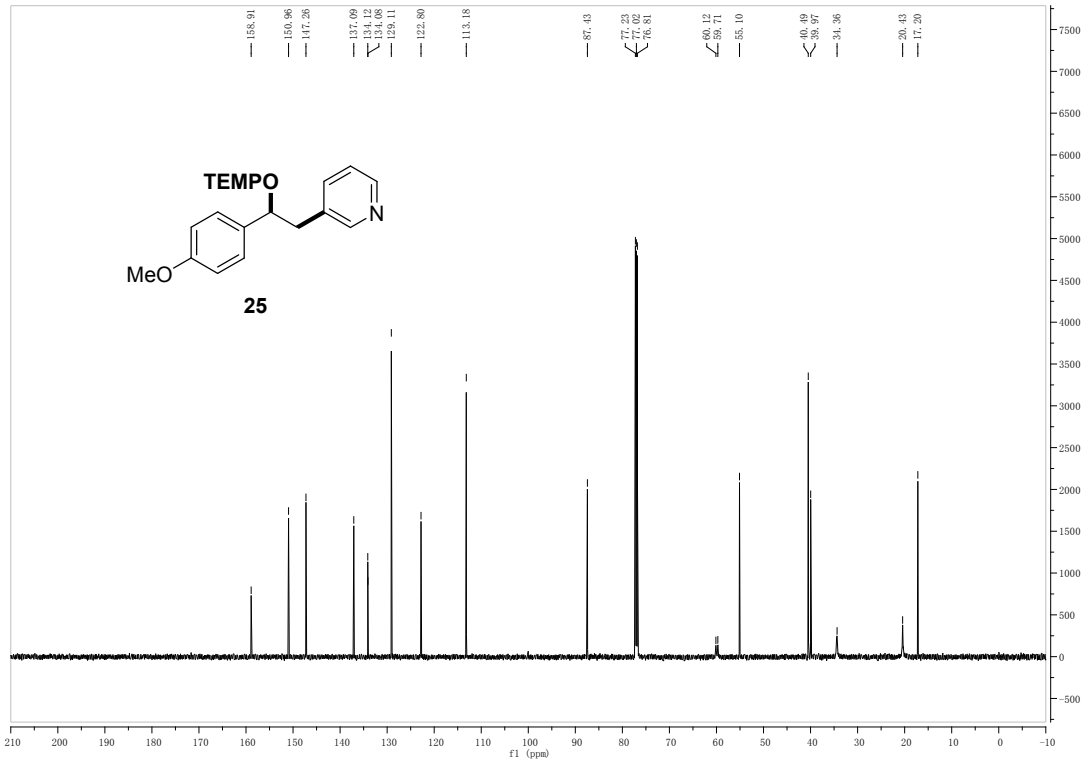

TEMPO
MeO
23
f1 (ppm)

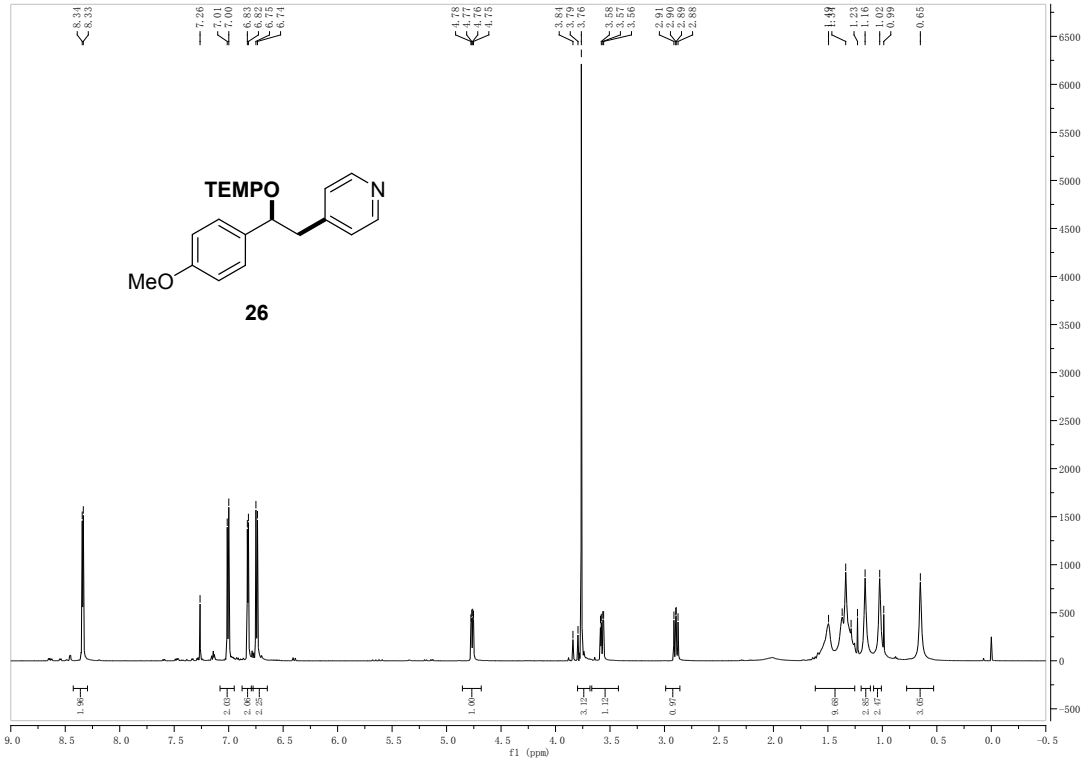

TEMPO
MeO
23
f1 (ppm)

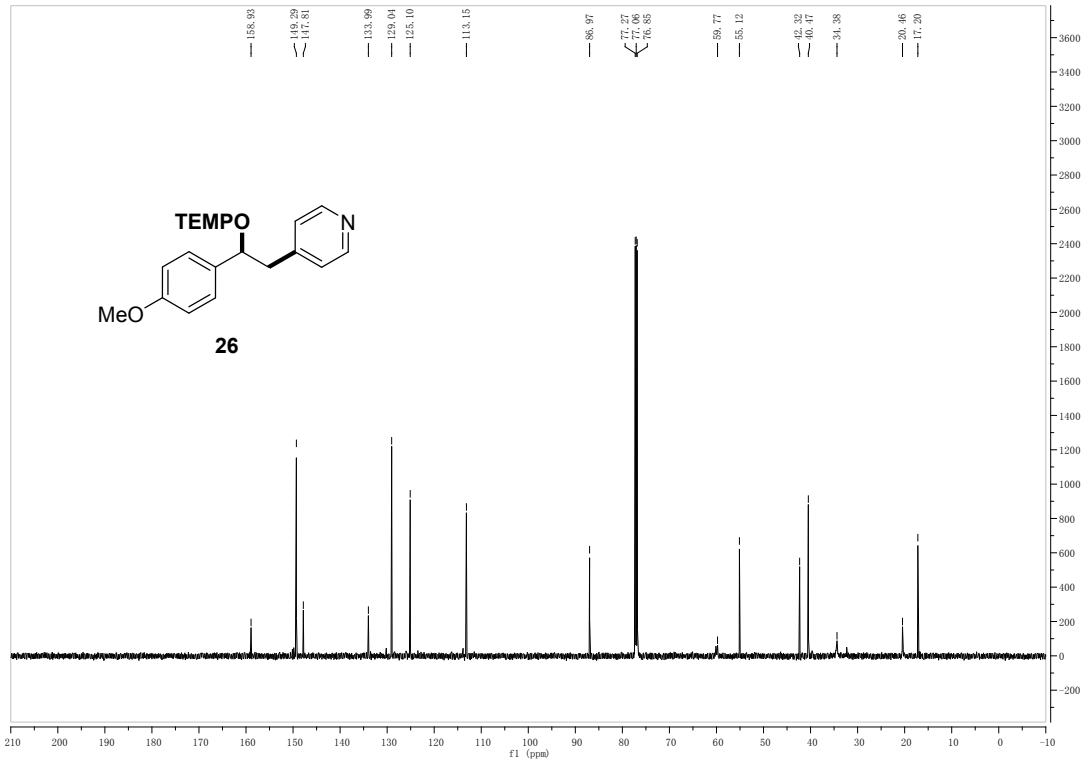

TEMPO
N
MeO
24
f1 (ppm)

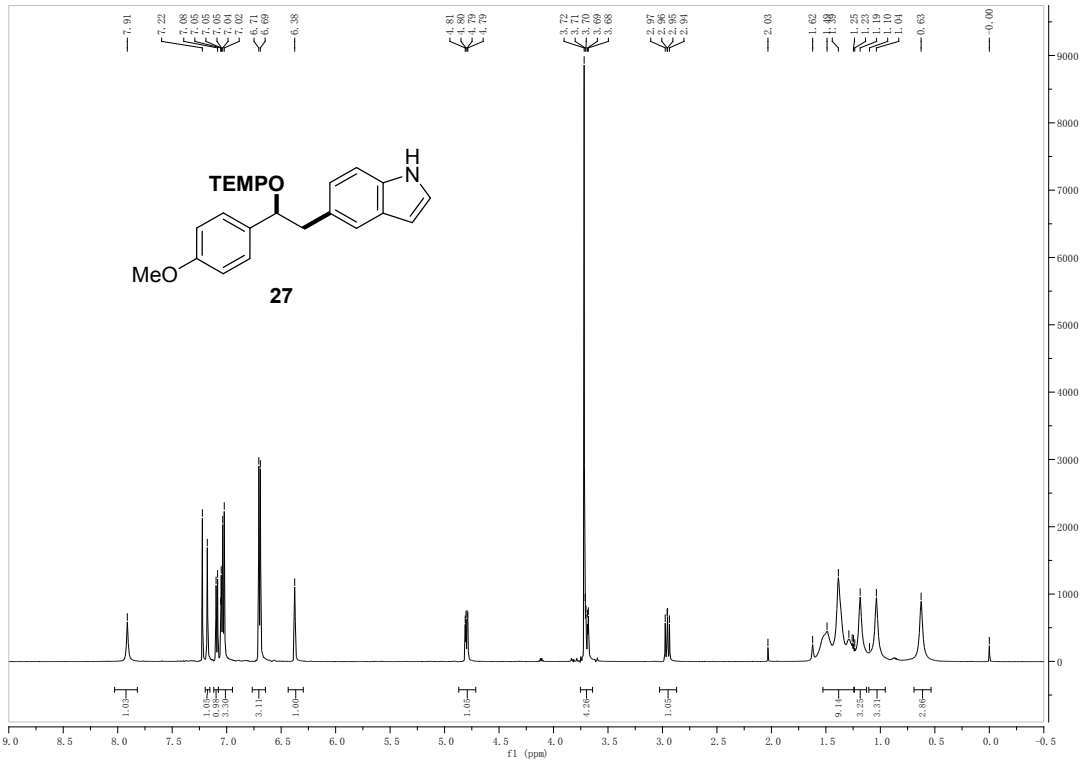

TEMPO
N
MeO
24
f1 (ppm)

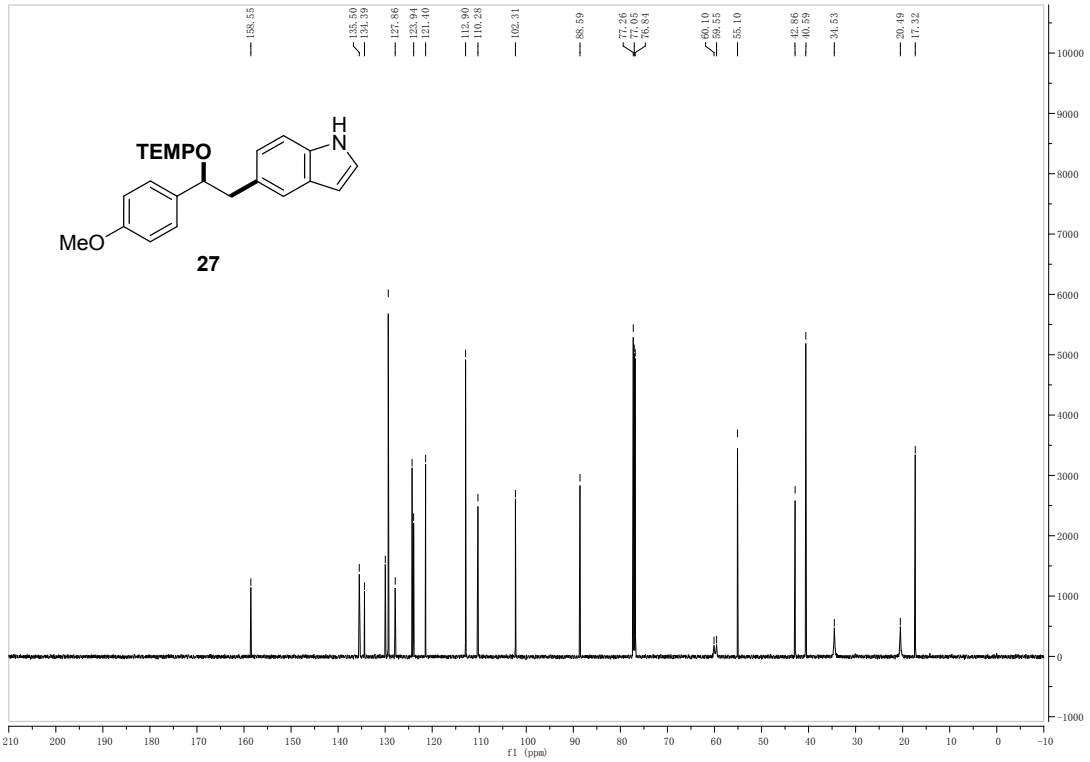

TEMPO
N
MeO
25
f1 (ppm)

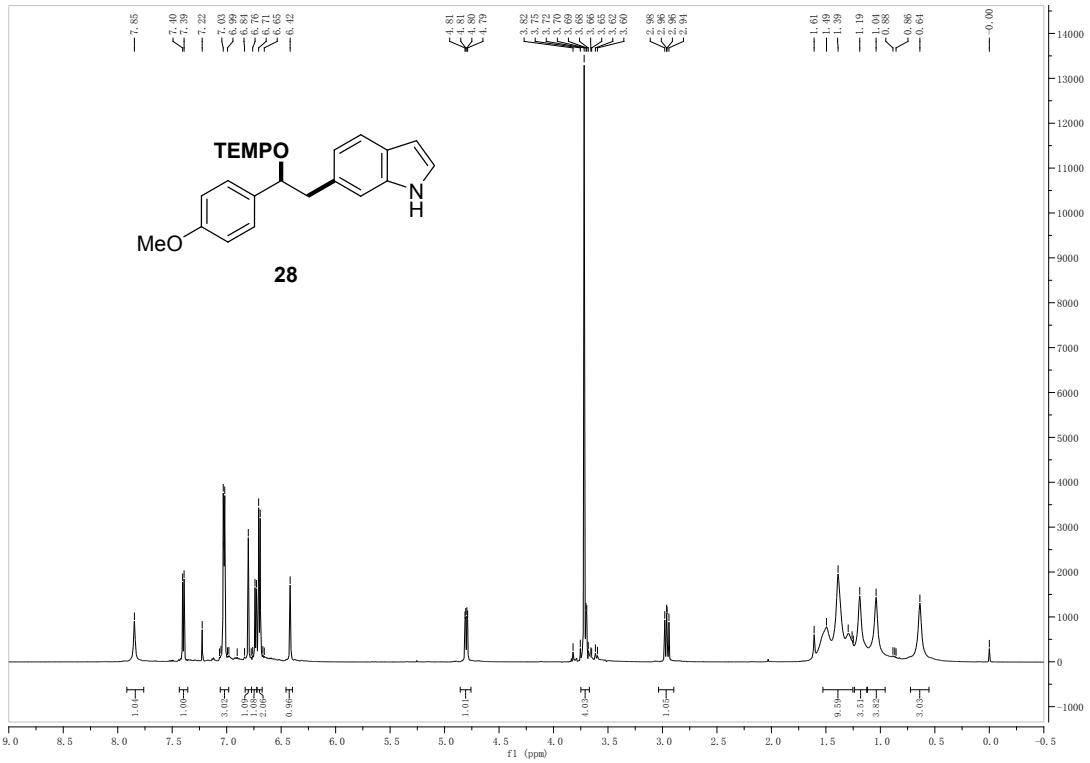

TEMPO
N
MeO
25
f1 (ppm)

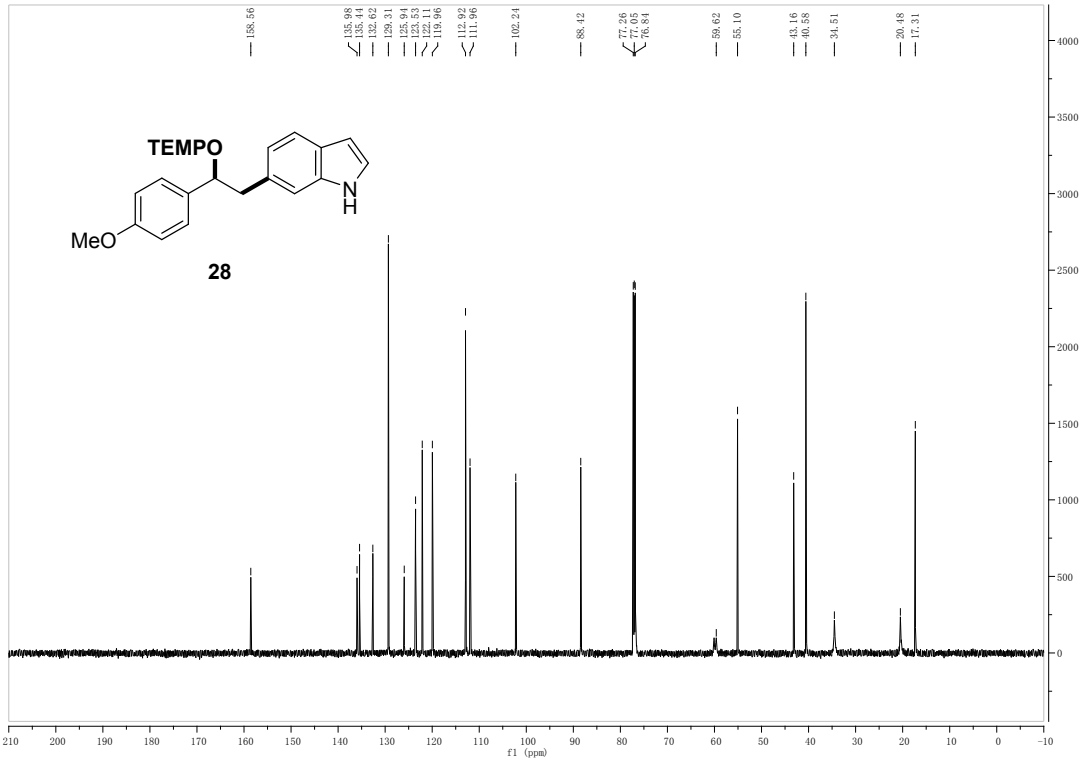

TEMPO
N
MeO
26
f1 (ppm)

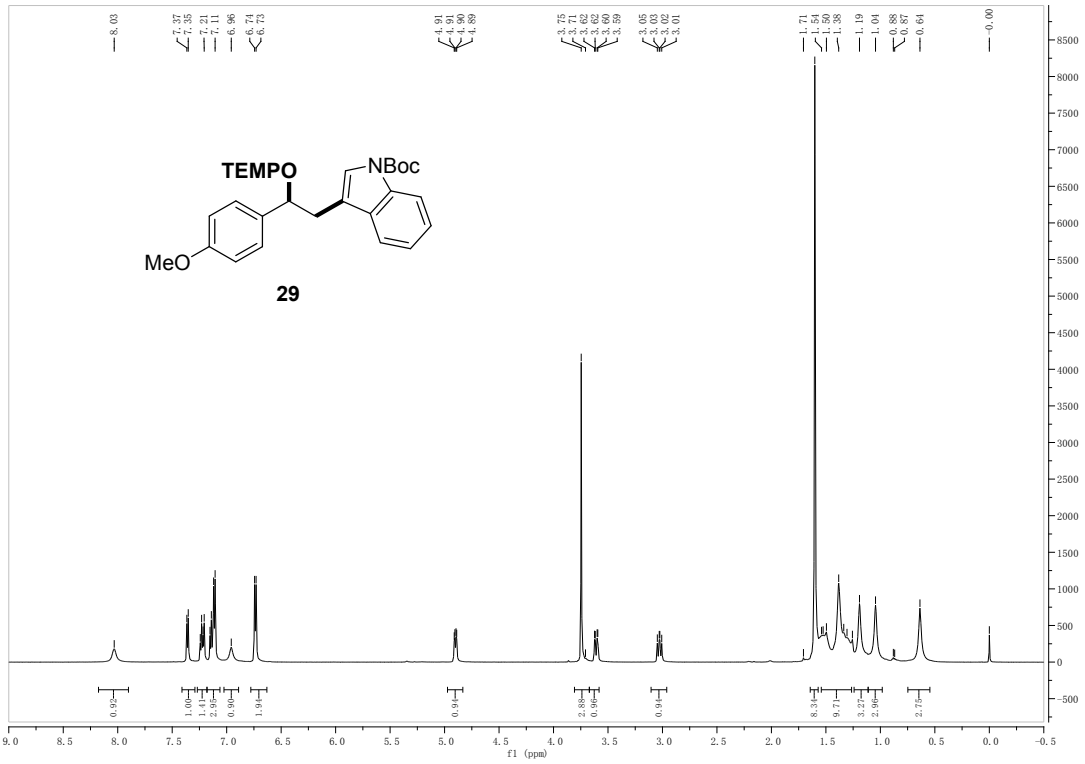

TEMPO
N
MeO
26
f1 (ppm)

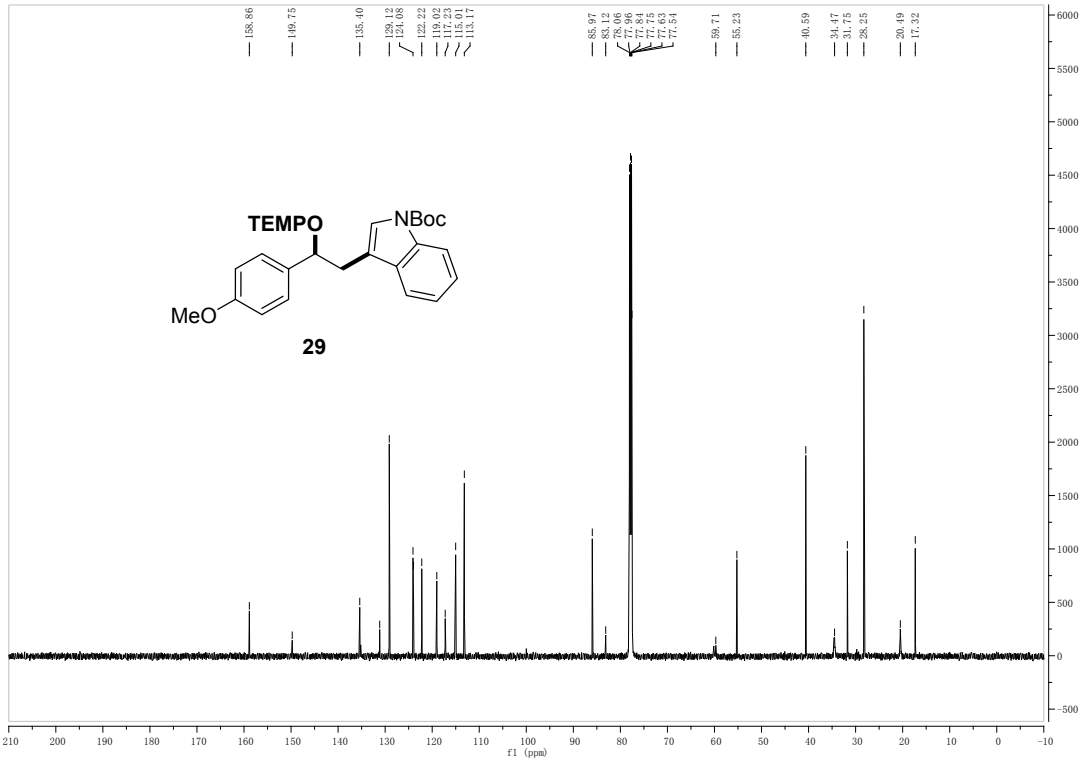

TEMPO
MeO
27
f1 (ppm)

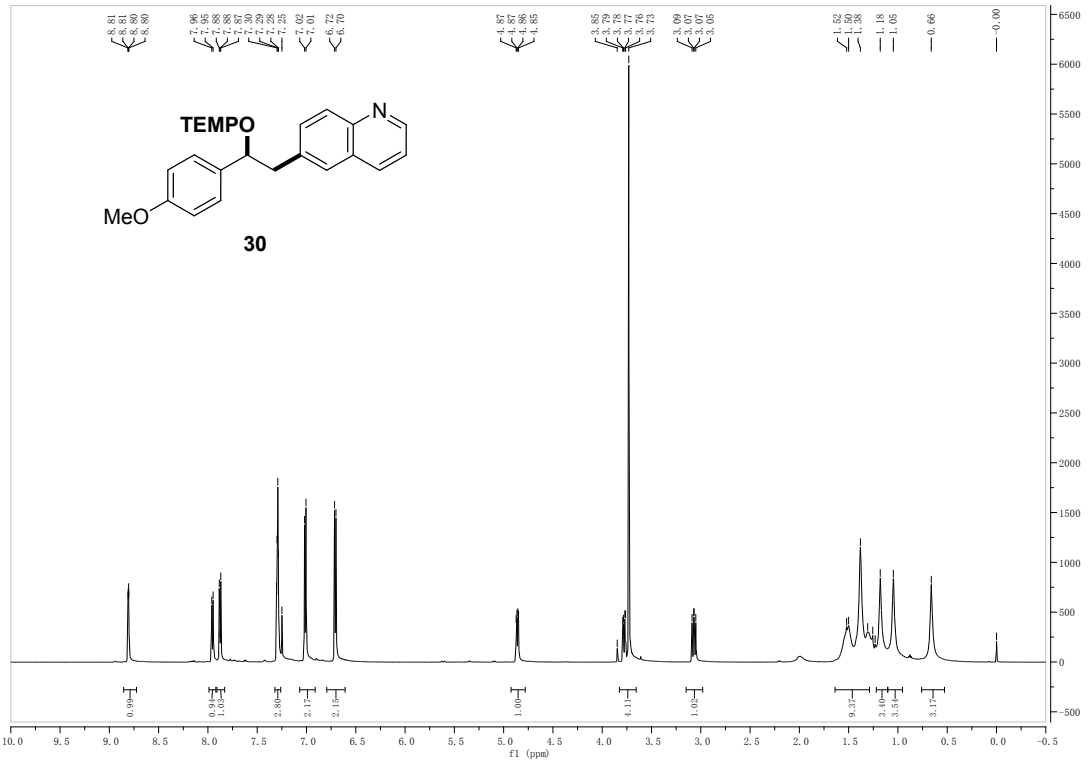

TEMPO
MeO
27
f1 (ppm)

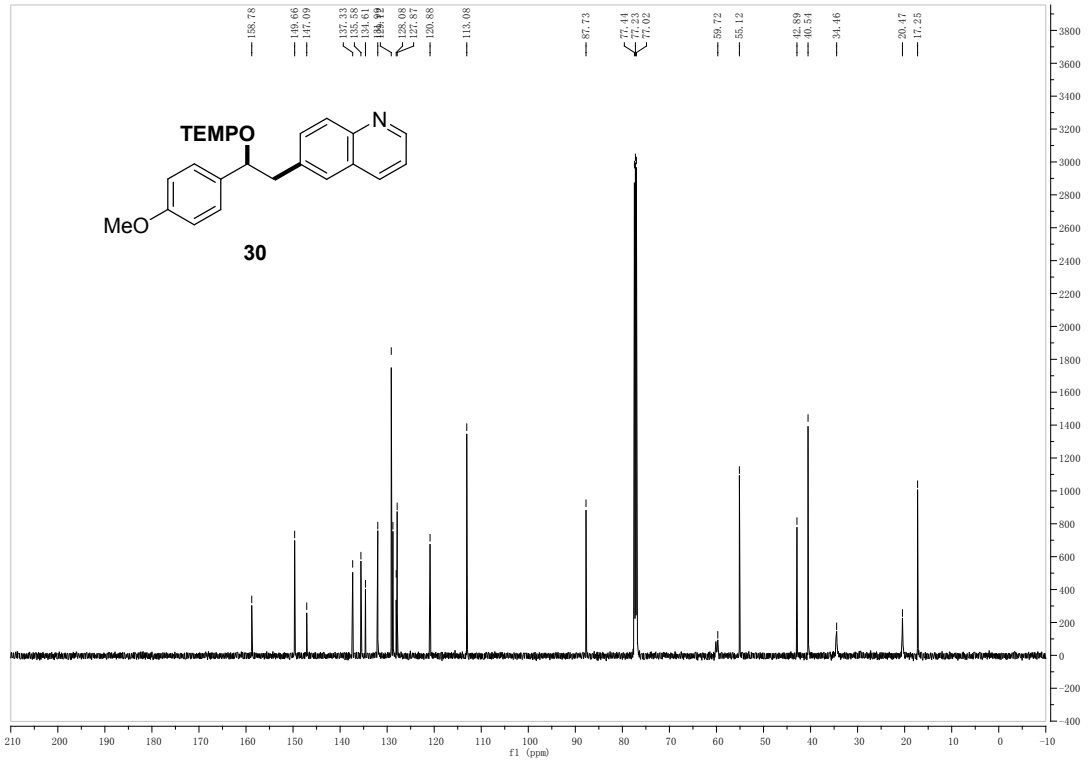

TEMPO
MeO
N
H
28
f1 (ppm)

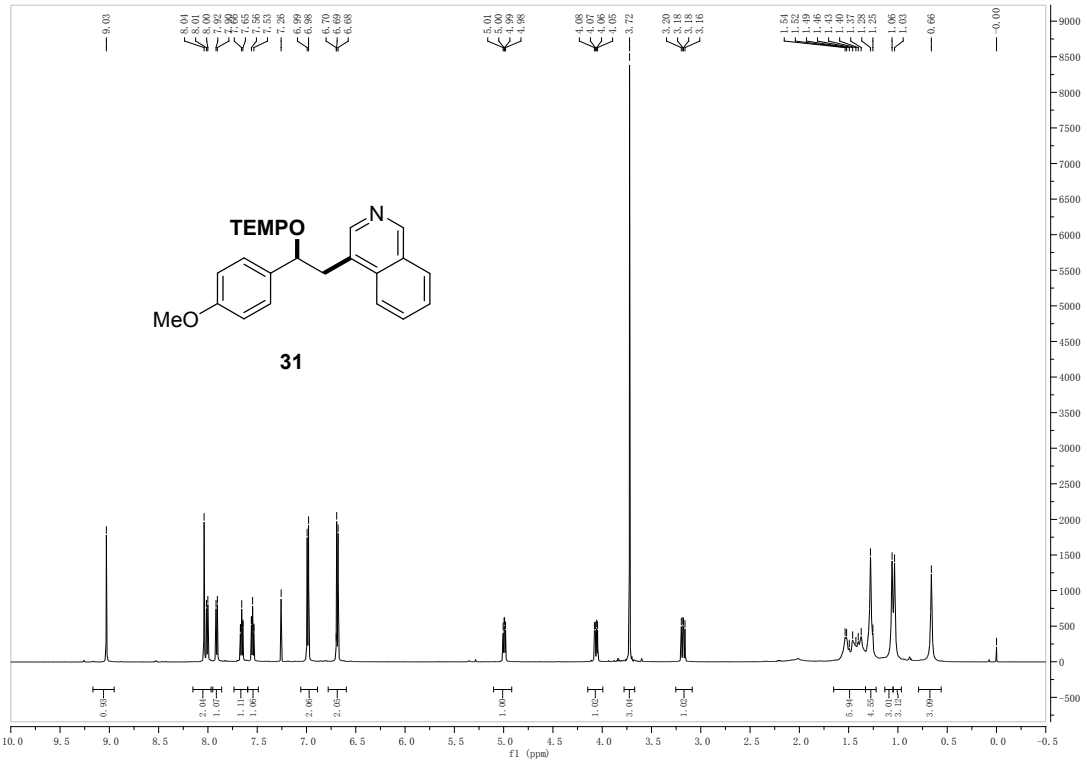

TEMPO
MeO
N
H
28
f1 (ppm)

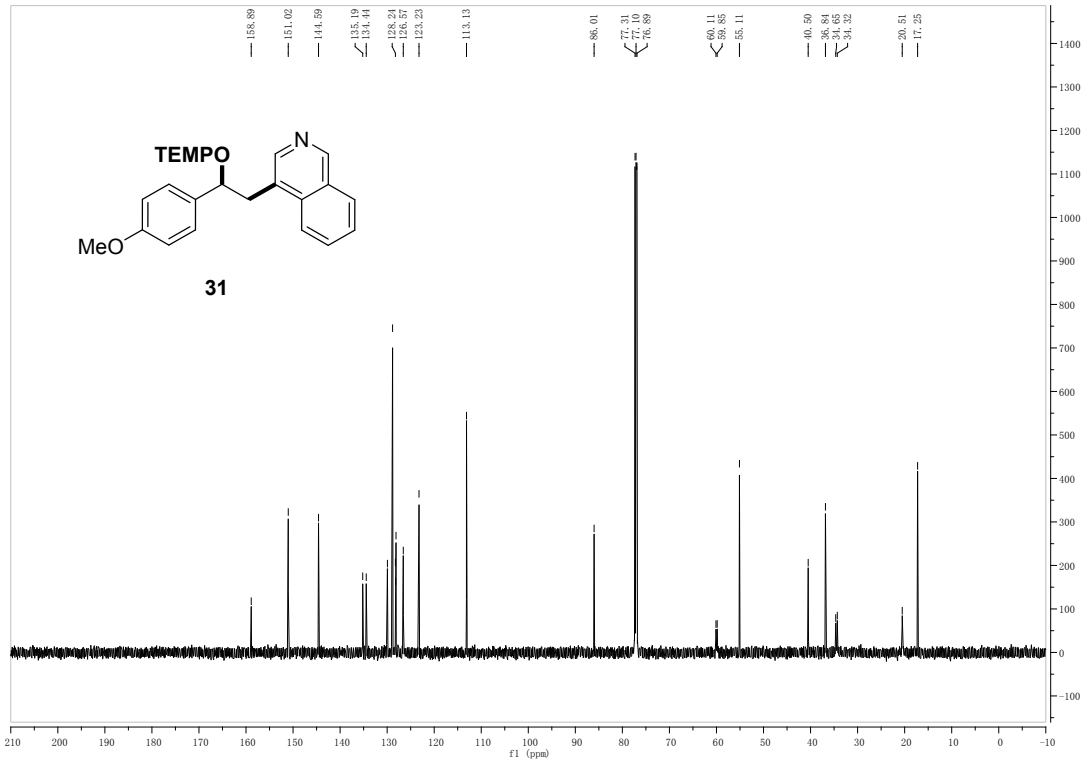

TEMPO
NBoc
MeO
29
f1 (ppm)

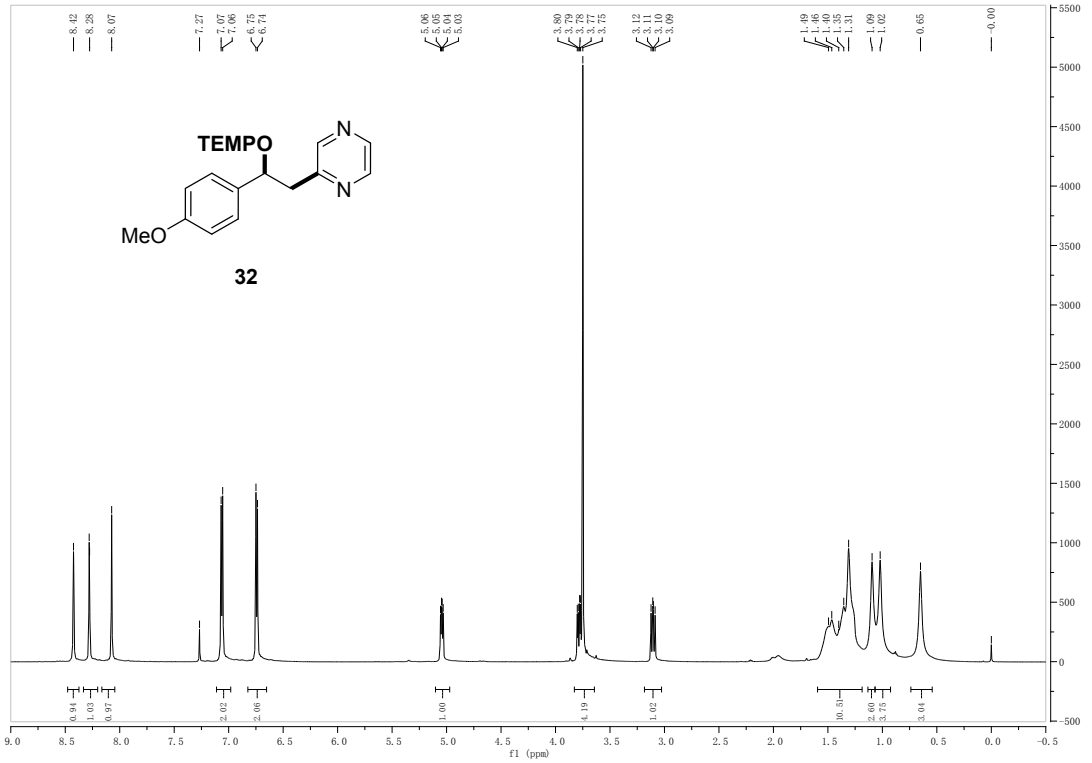

TEMPO
NBoc
MeO
29
f1 (ppm)

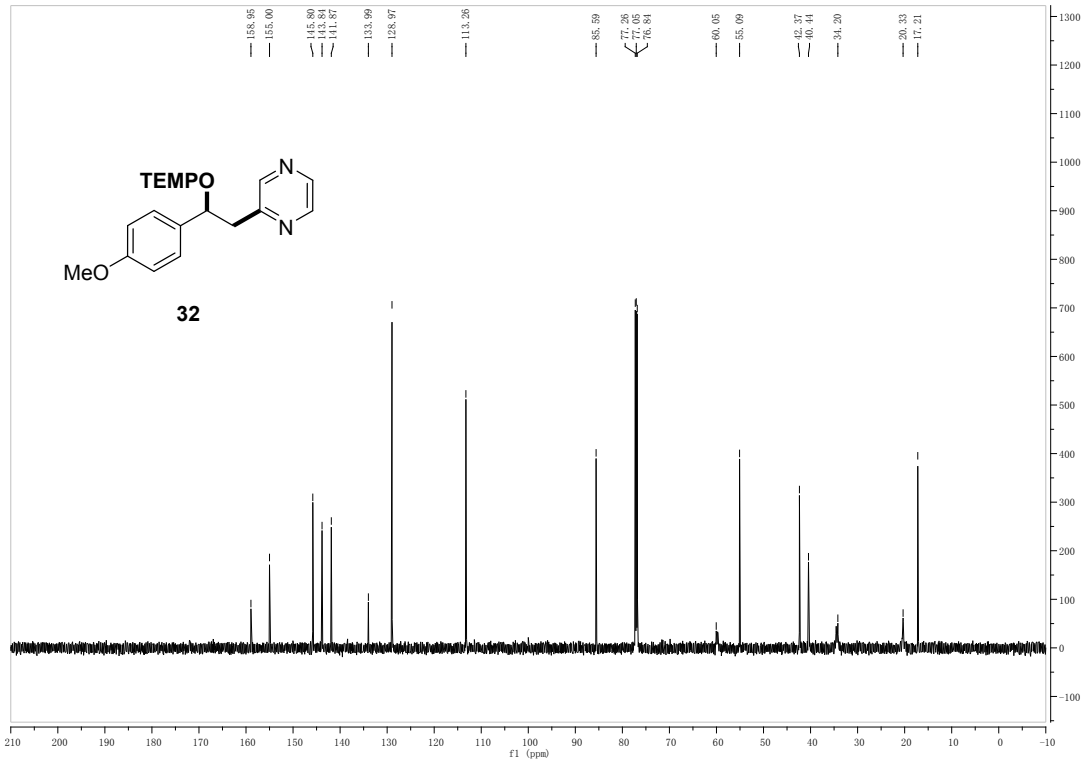

TEMPO
MeO
N
30
f1 (ppm)

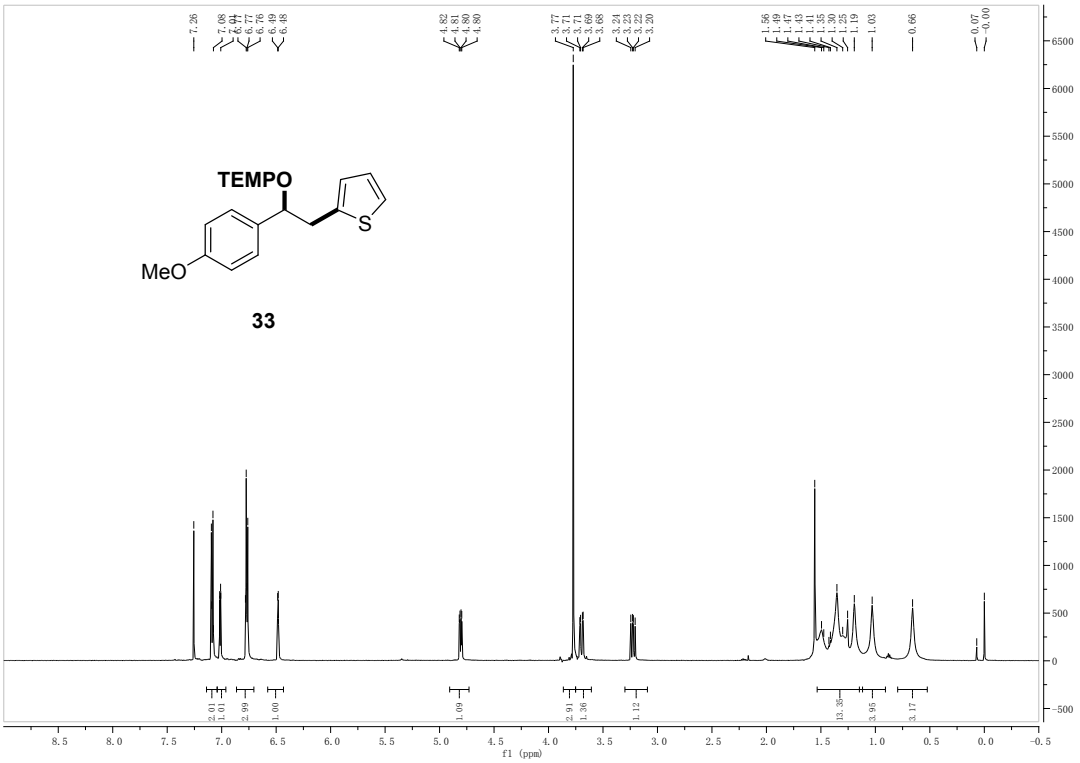

TEMPO
MeO
N
30
f1 (ppm)

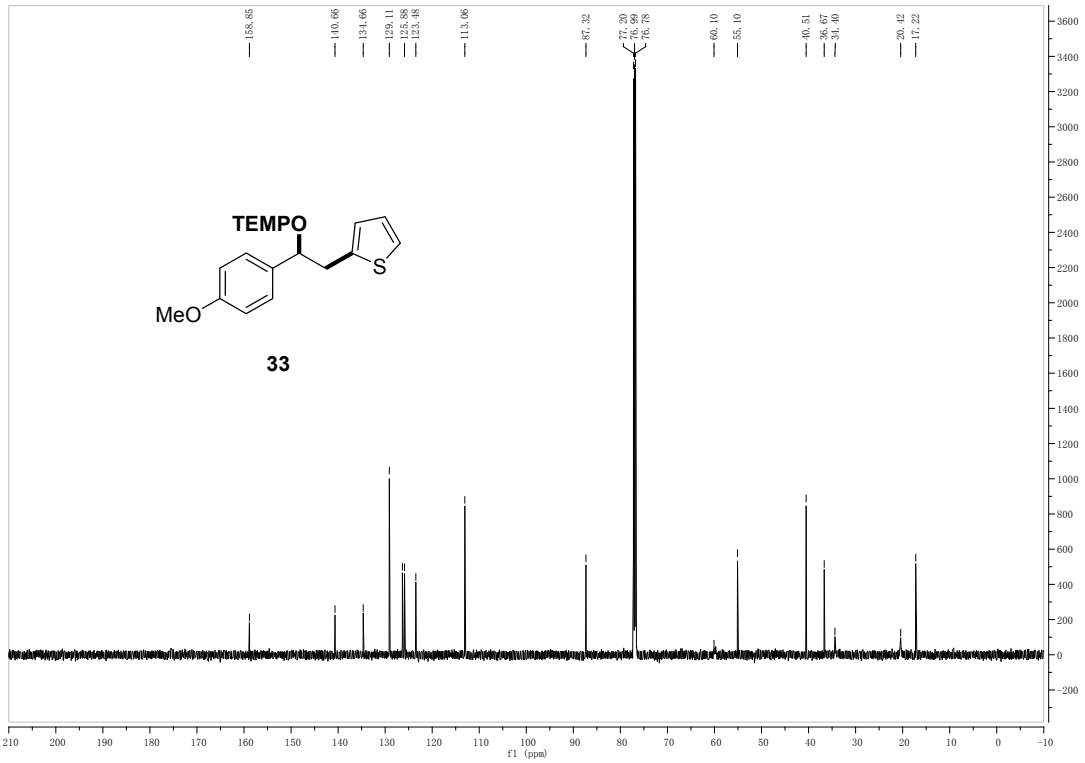

TEMPO
N
MeO
31
f1 (ppm)

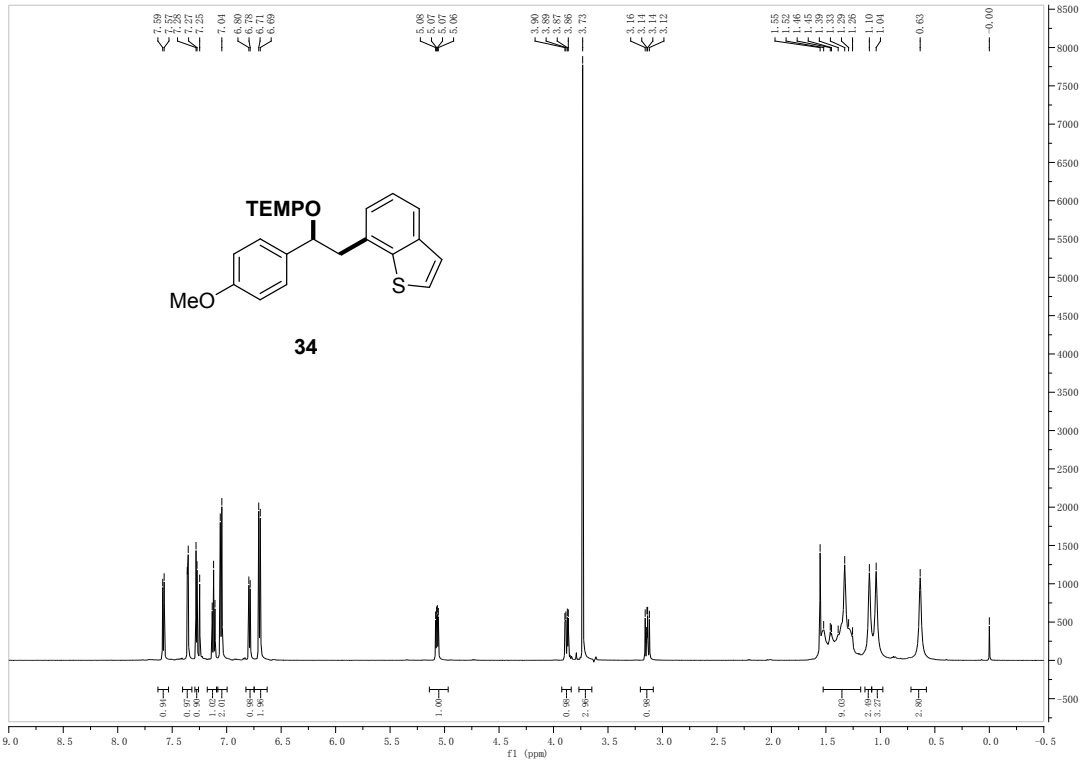

TEMPO
N
MeO
31
f1 (ppm)

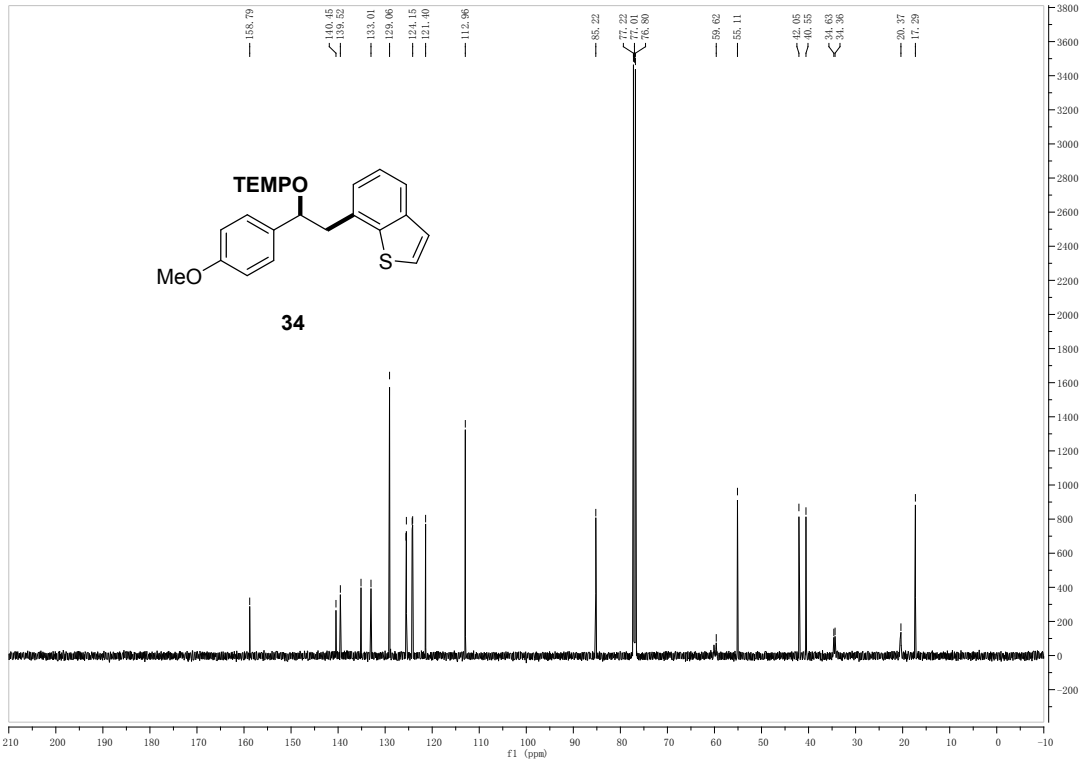

TEMPO
MeO
32
f1 (ppm)

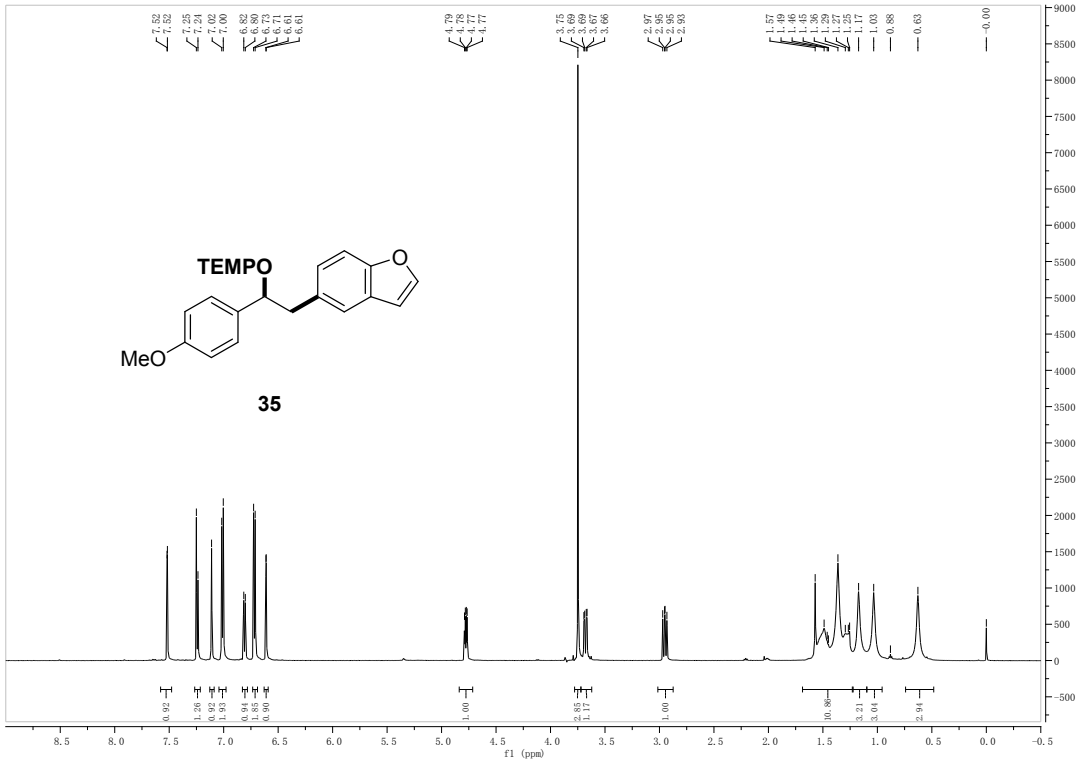

TEMPO
MeO
32
f1 (ppm)

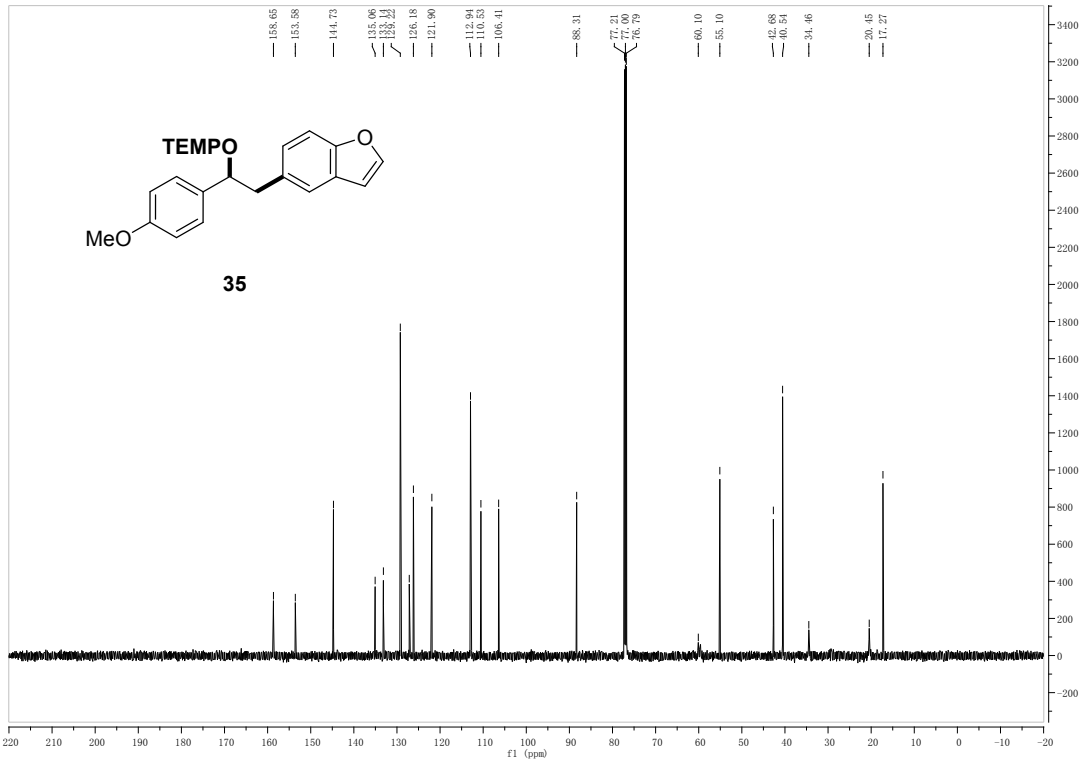

TEMPO
MeO
S
33
f1 (ppm)

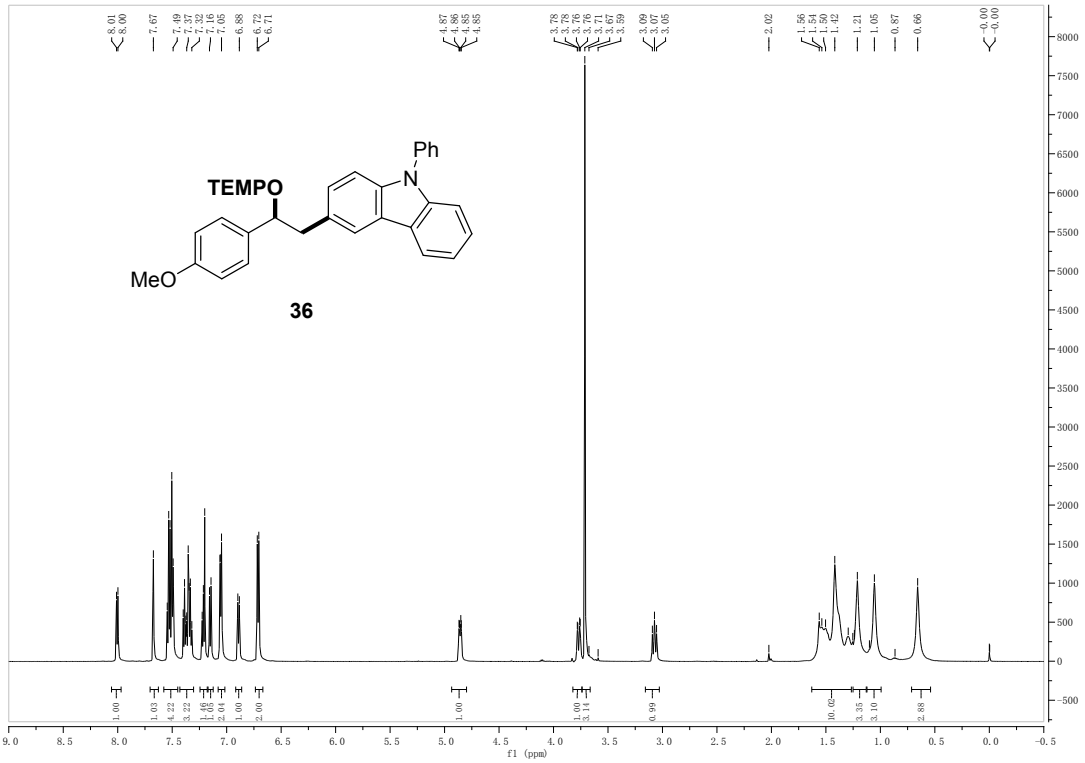

TEMPO
MeO
S
33
f1 (ppm)

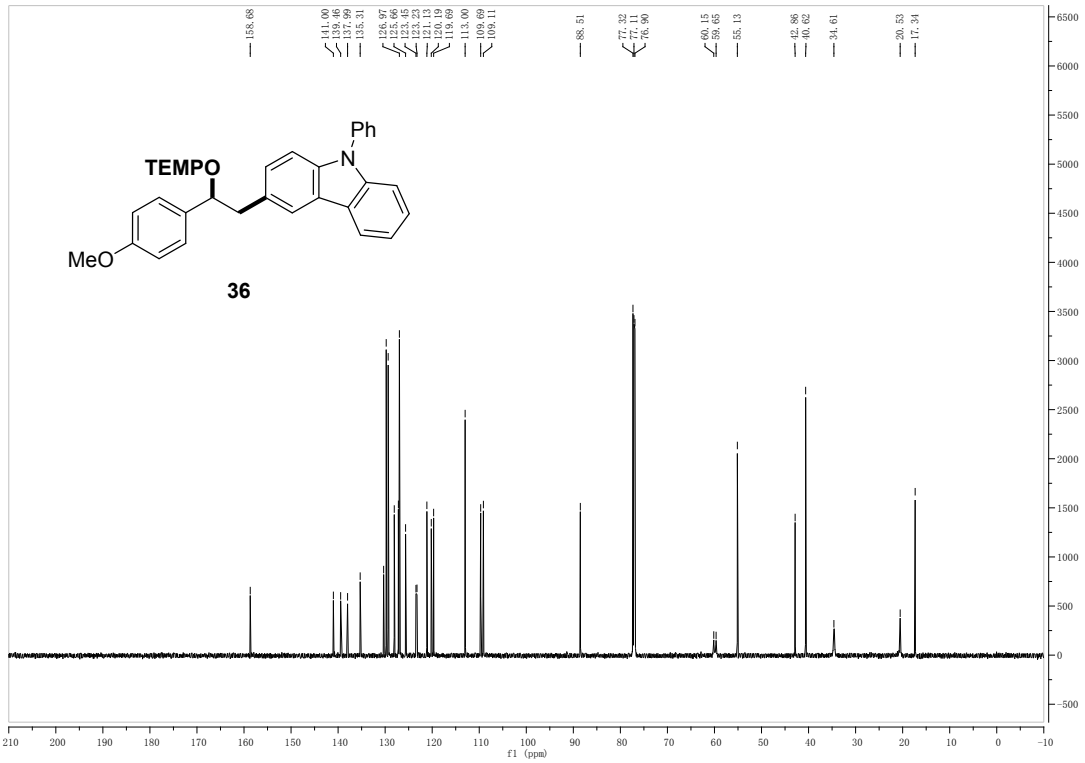

TEMPO
MeO
S
34
f1 (ppm)

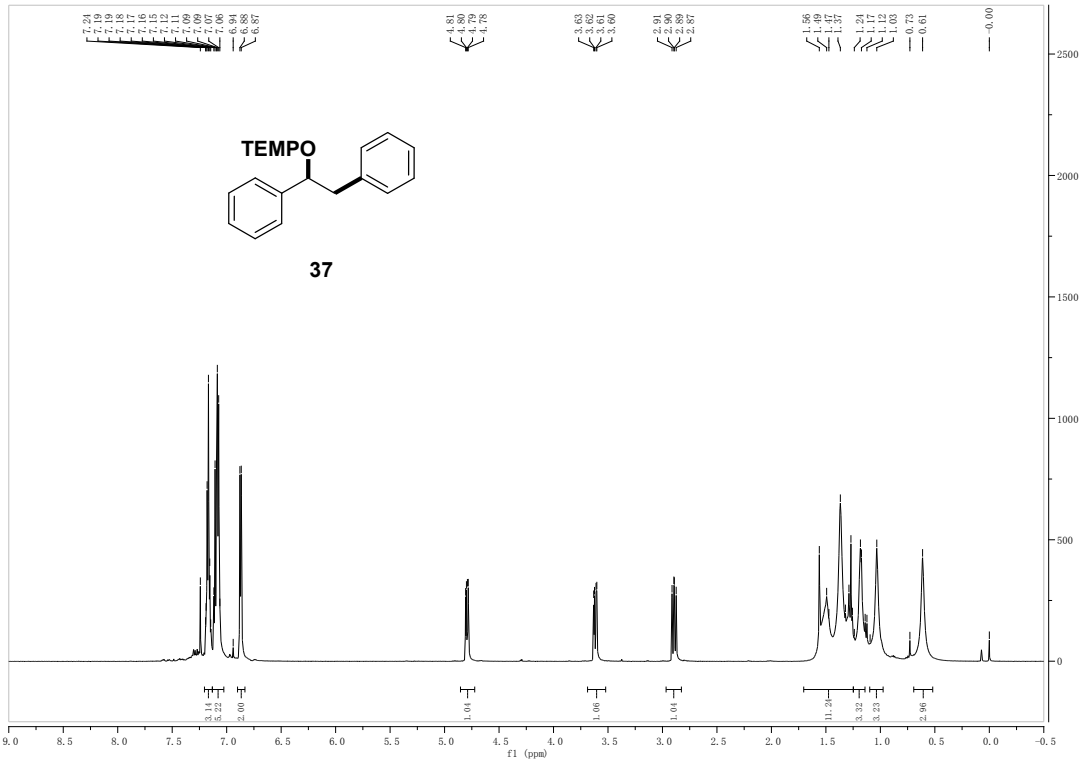

TEMPO
MeO
S
34
f1 (ppm)

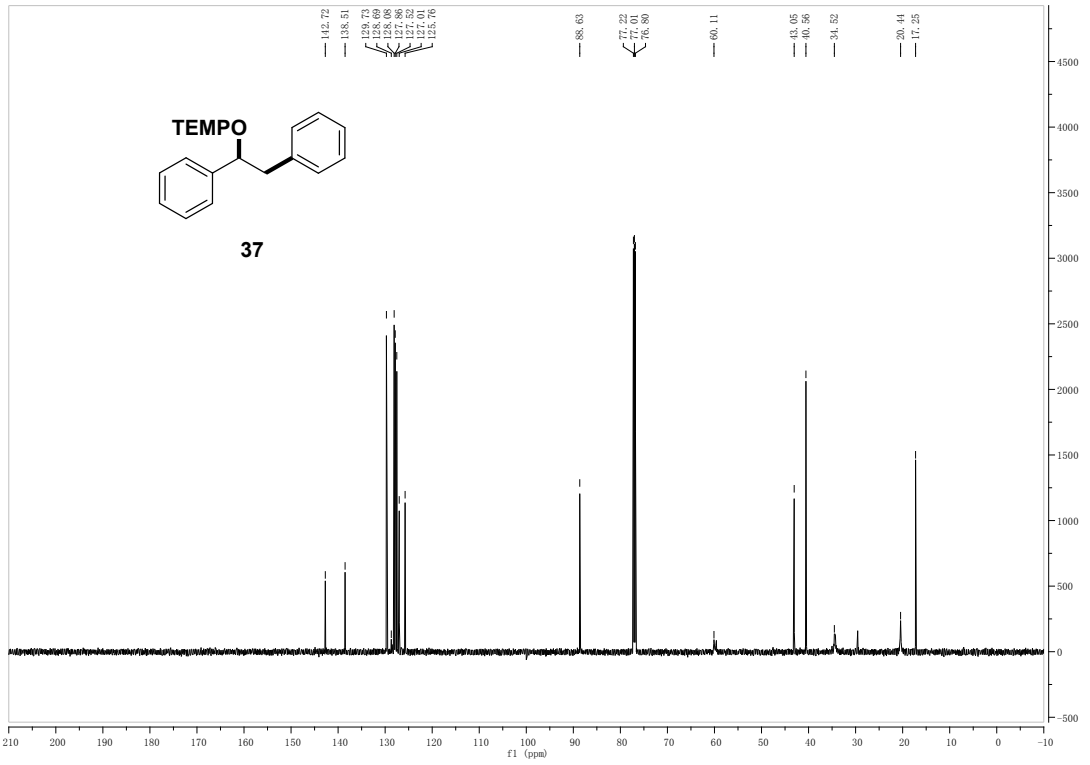

TEMPO
MeO
35
f1 (ppm)

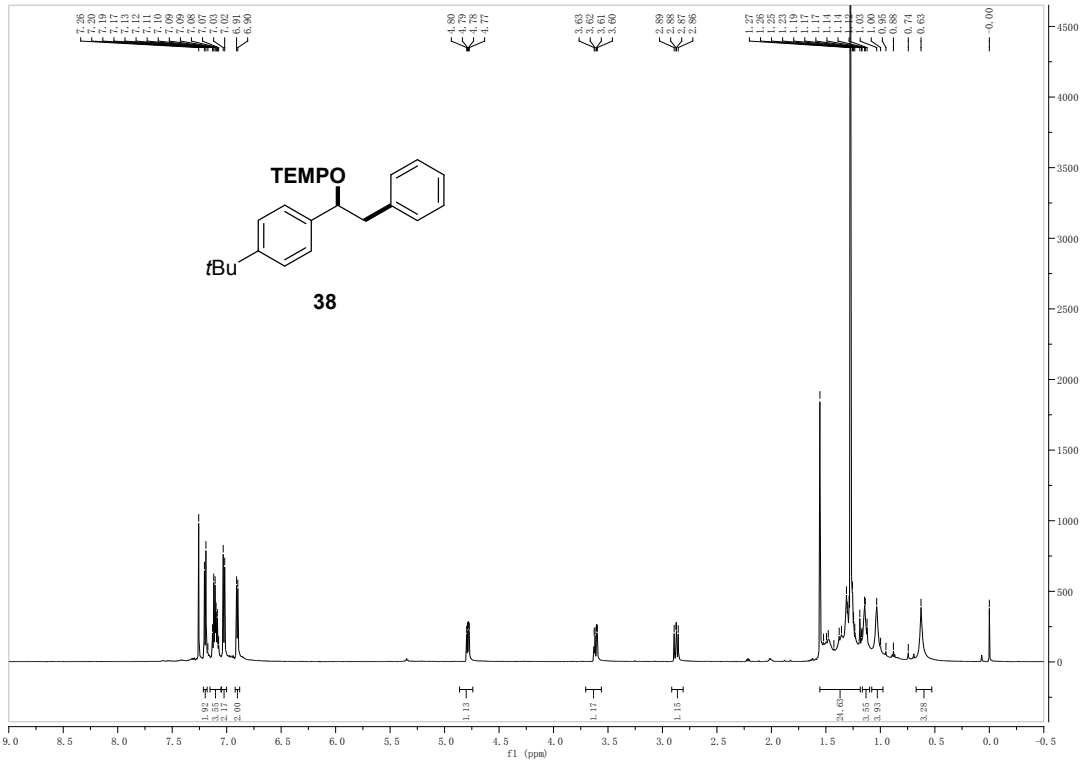

TEMPO
MeO
35
f1 (ppm)

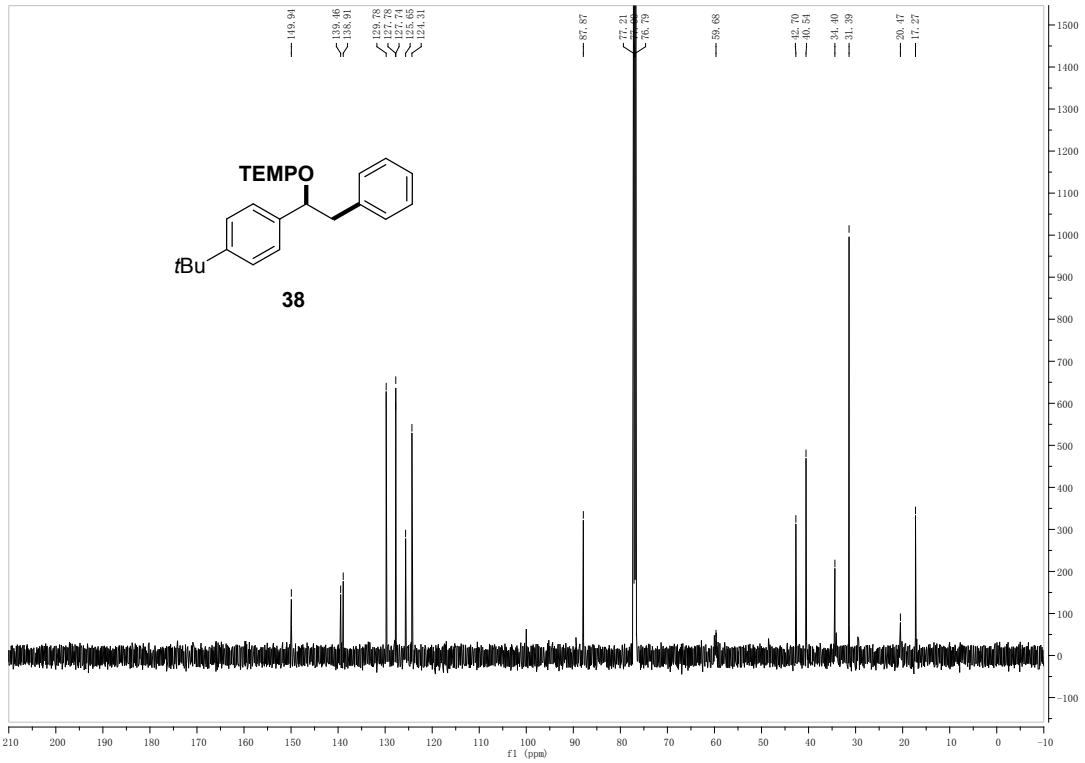

TEMPO
Ph
N
MeO
36
f1 (ppm)

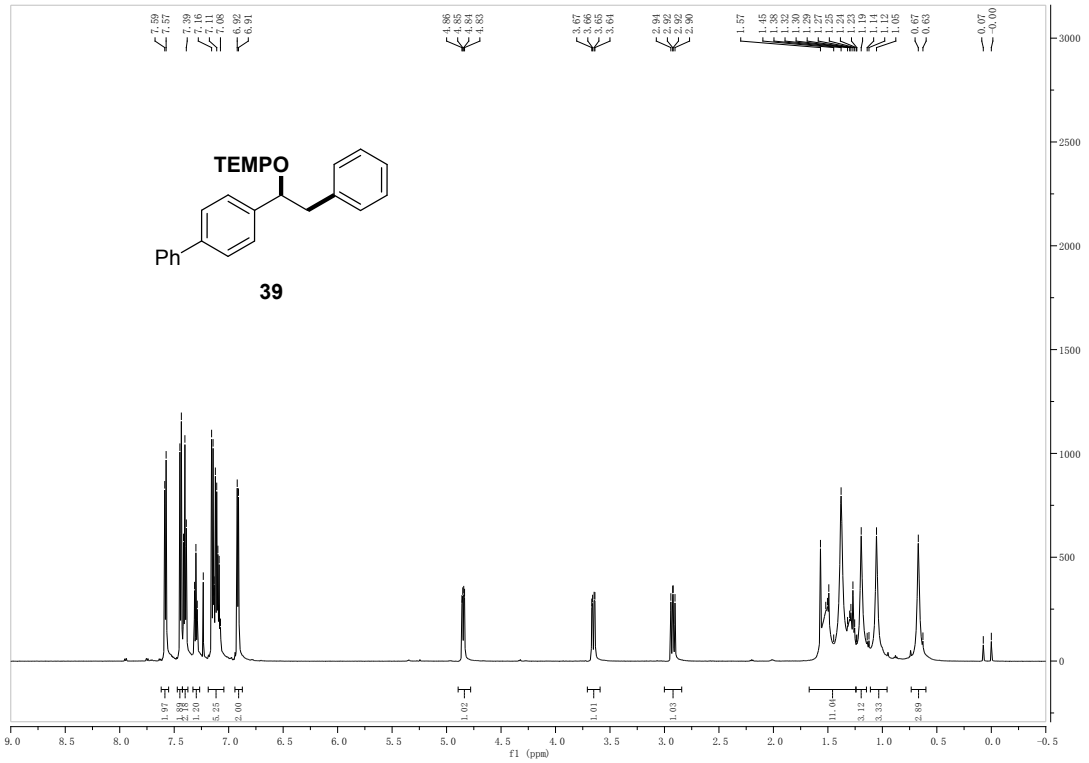

TEMPO
Ph
N
MeO
36
f1 (ppm)


TEMPO
37
f1 (ppm)

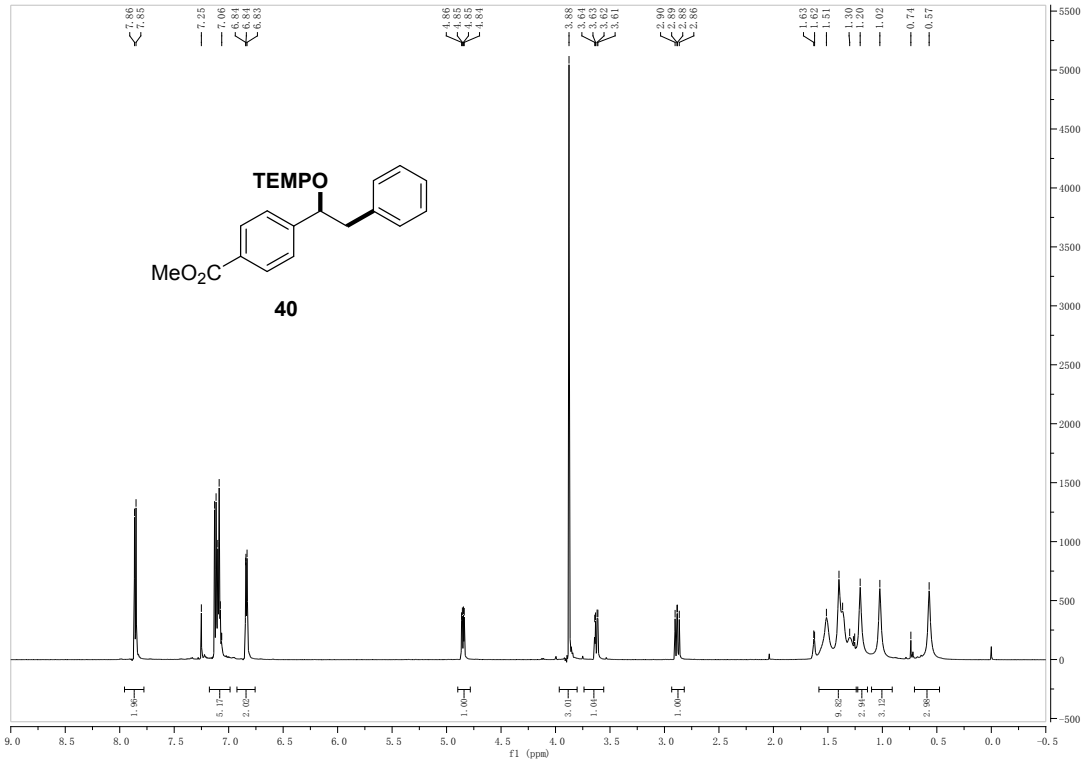

TEMPO
37
f1 (ppm)

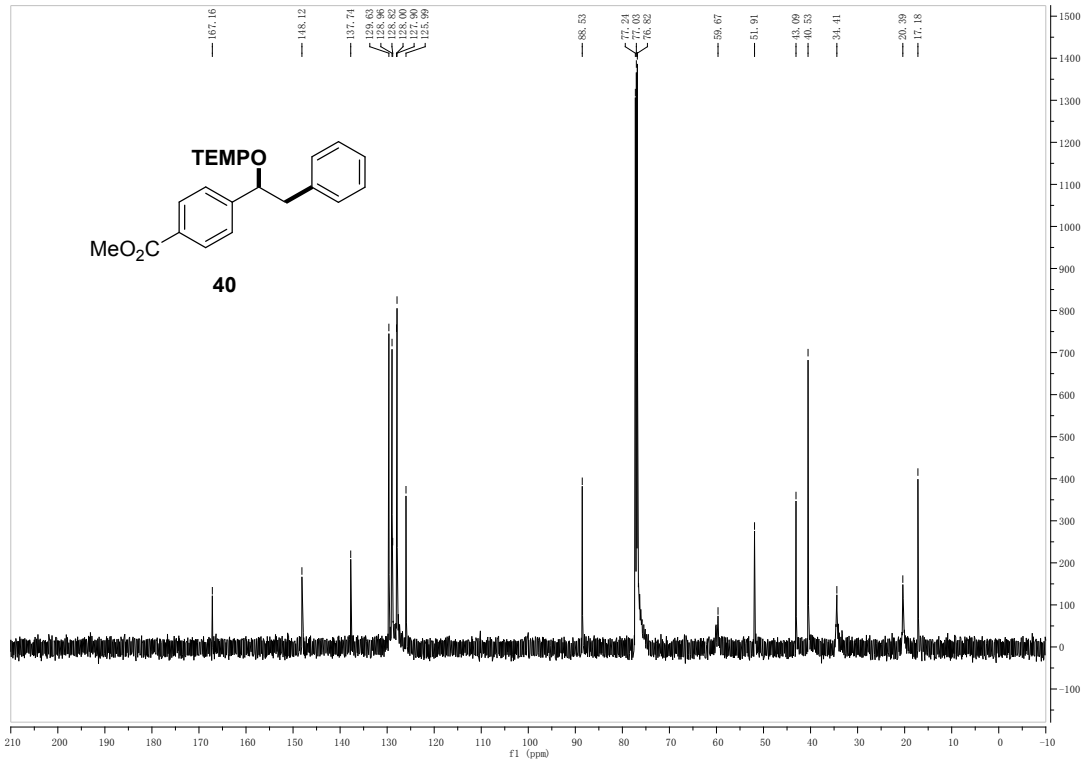

TEMPO
tBu
38
f1 (ppm)

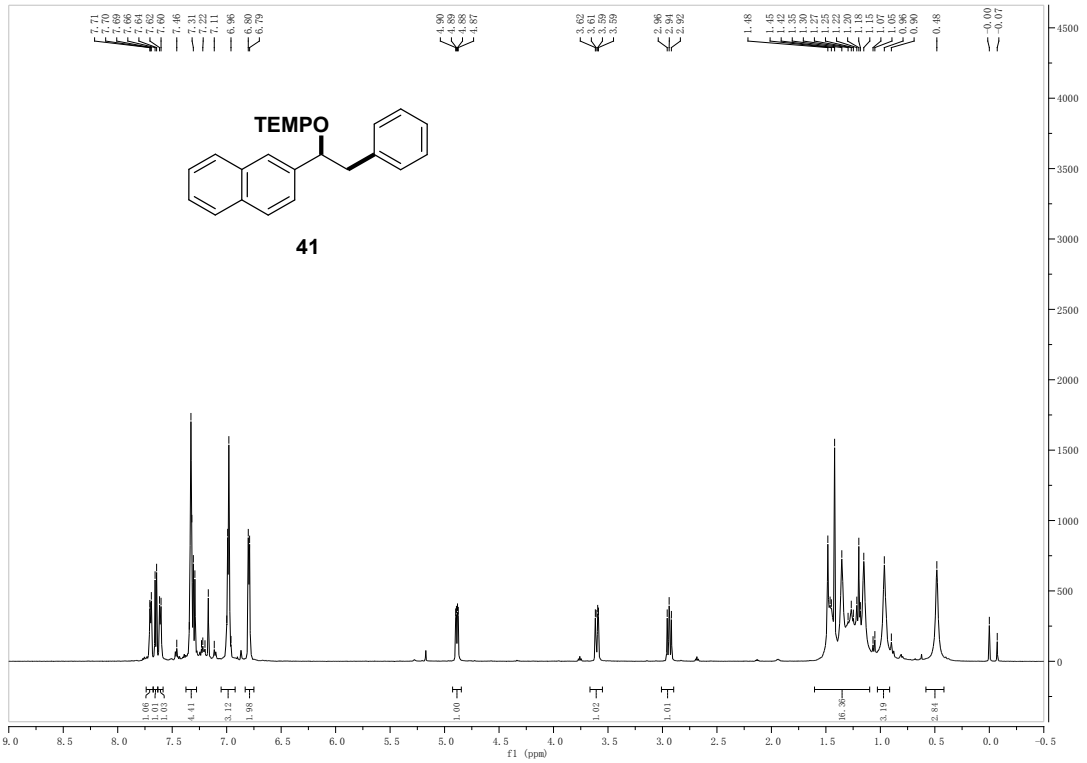

TEMPO
tBu
38
f1 (ppm)

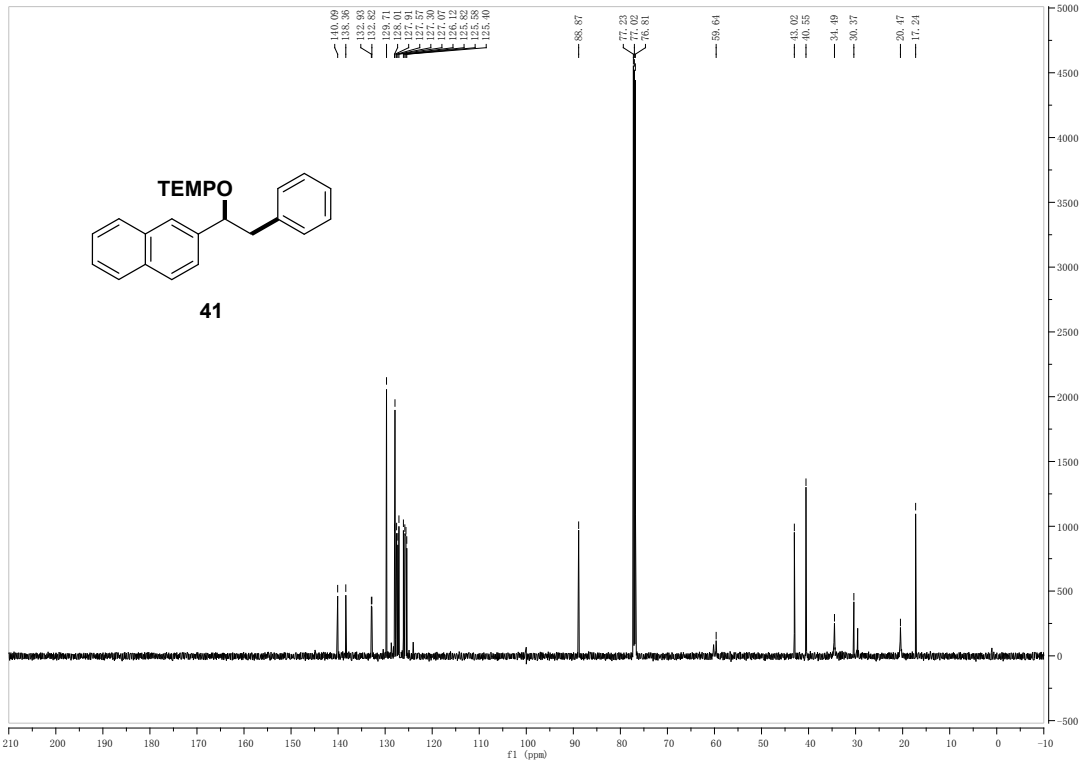

TEMPO
Ph
39
f1 (ppm)

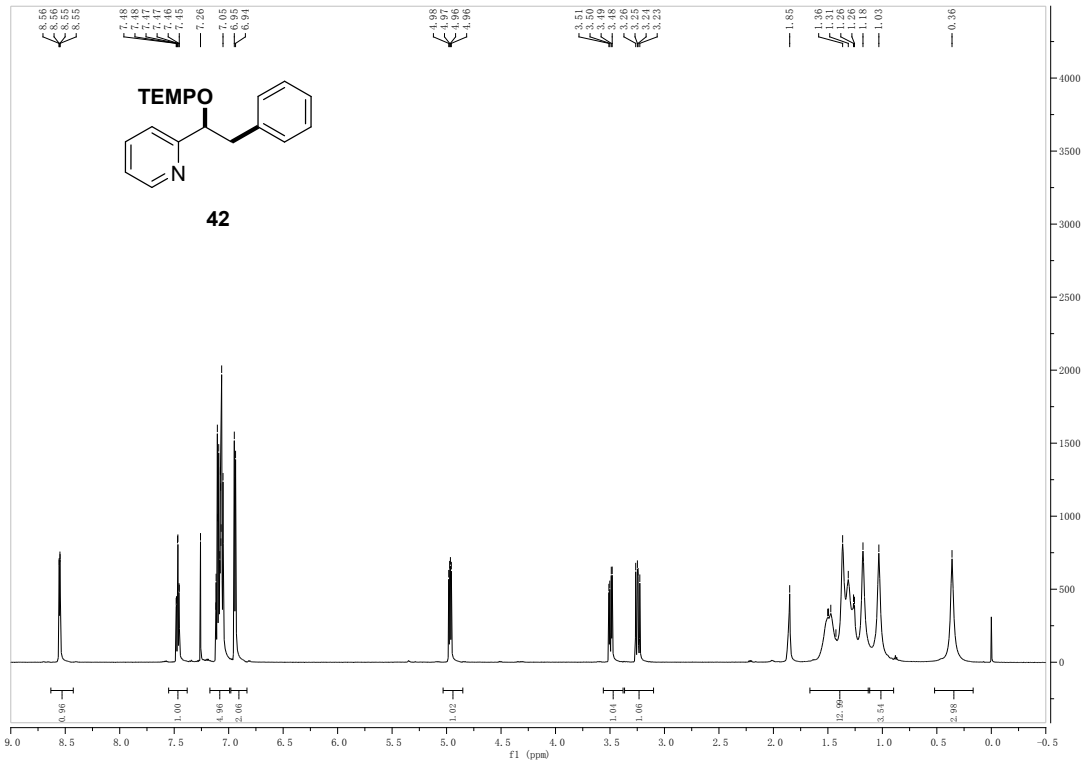

TEMPO
Ph
39
f1 (ppm)

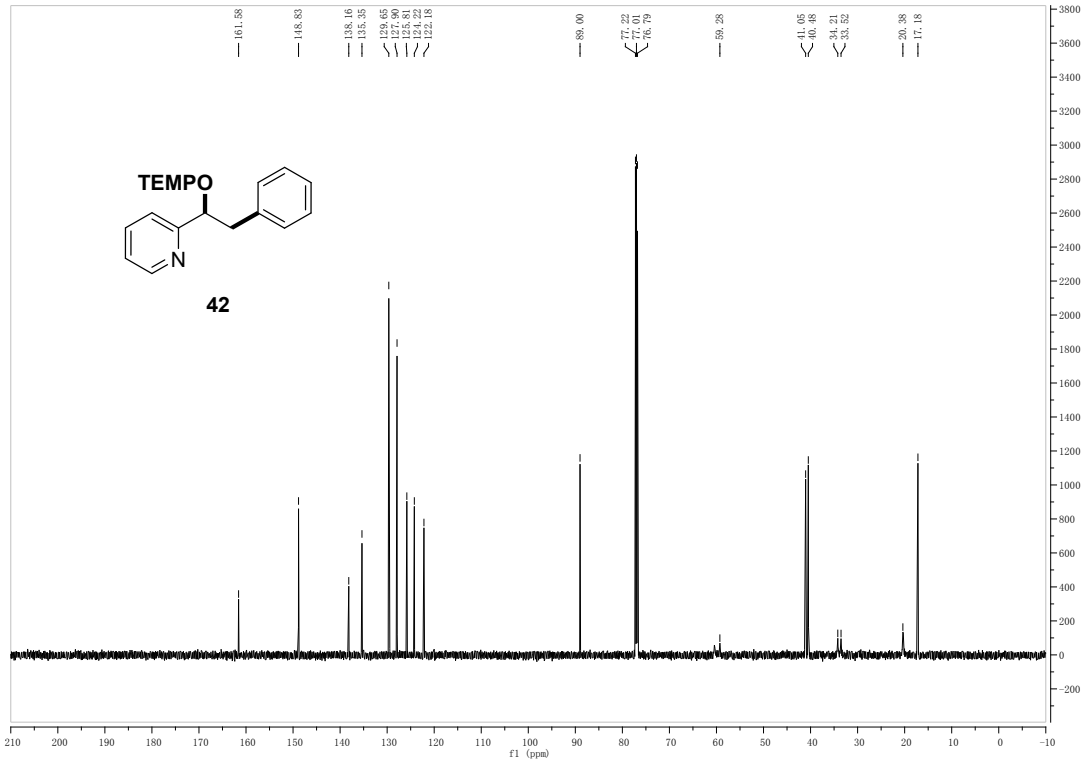

TEMPO
MeO2C
40
f1 (ppm)

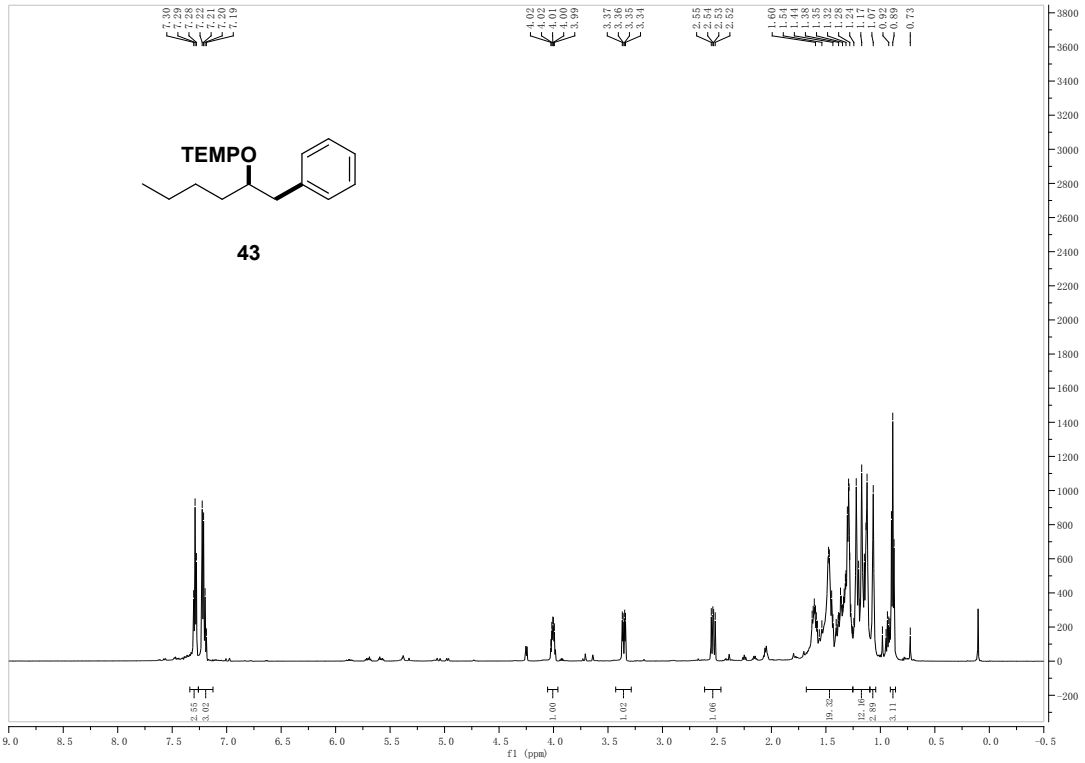

TEMPO
MeO2C
40
f1 (ppm)

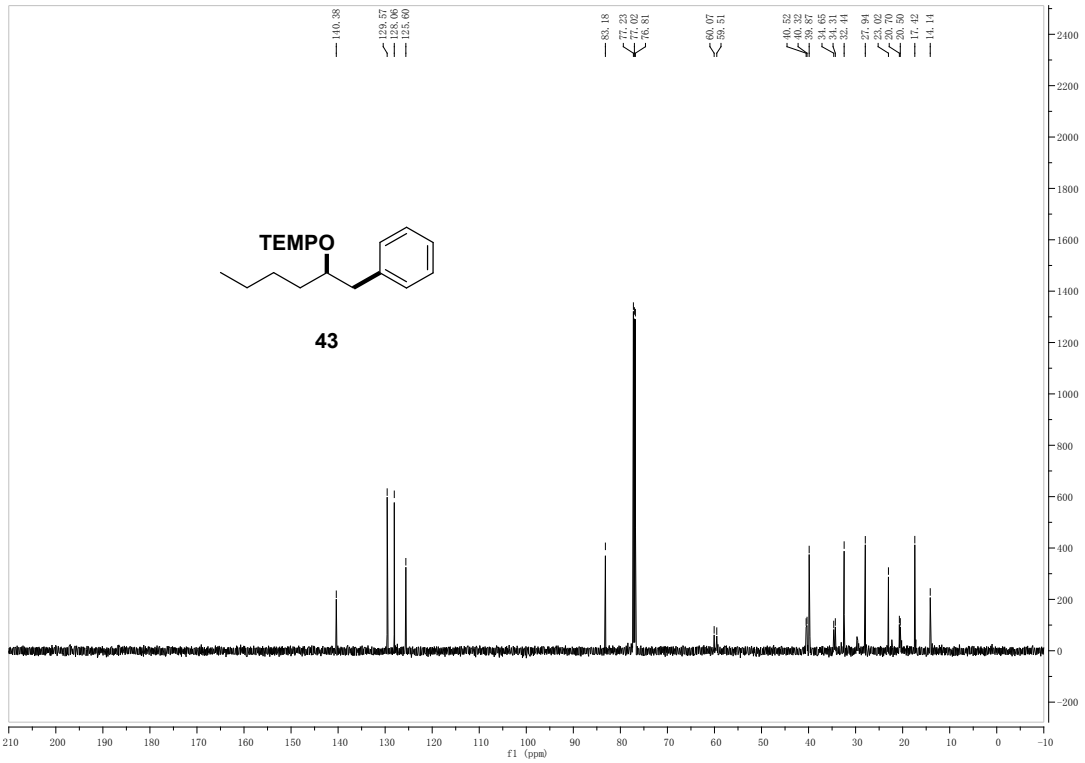

TEMPO
41
f1 (ppm)

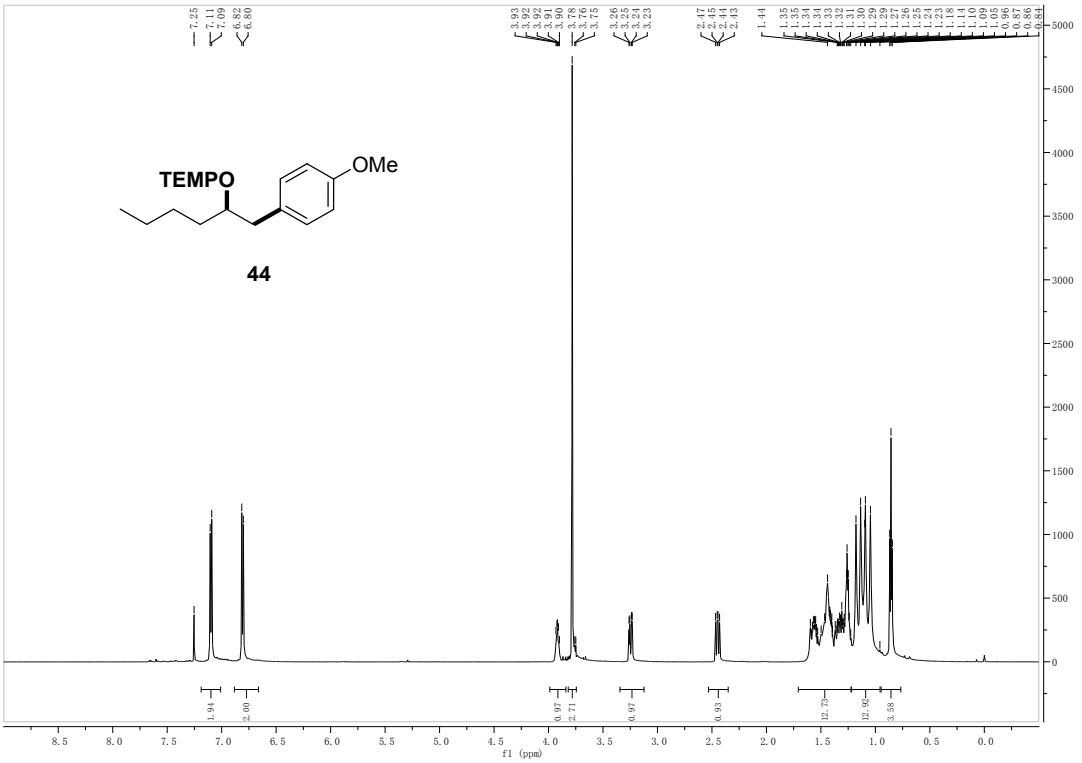

TEMPO
41
f1 (ppm)

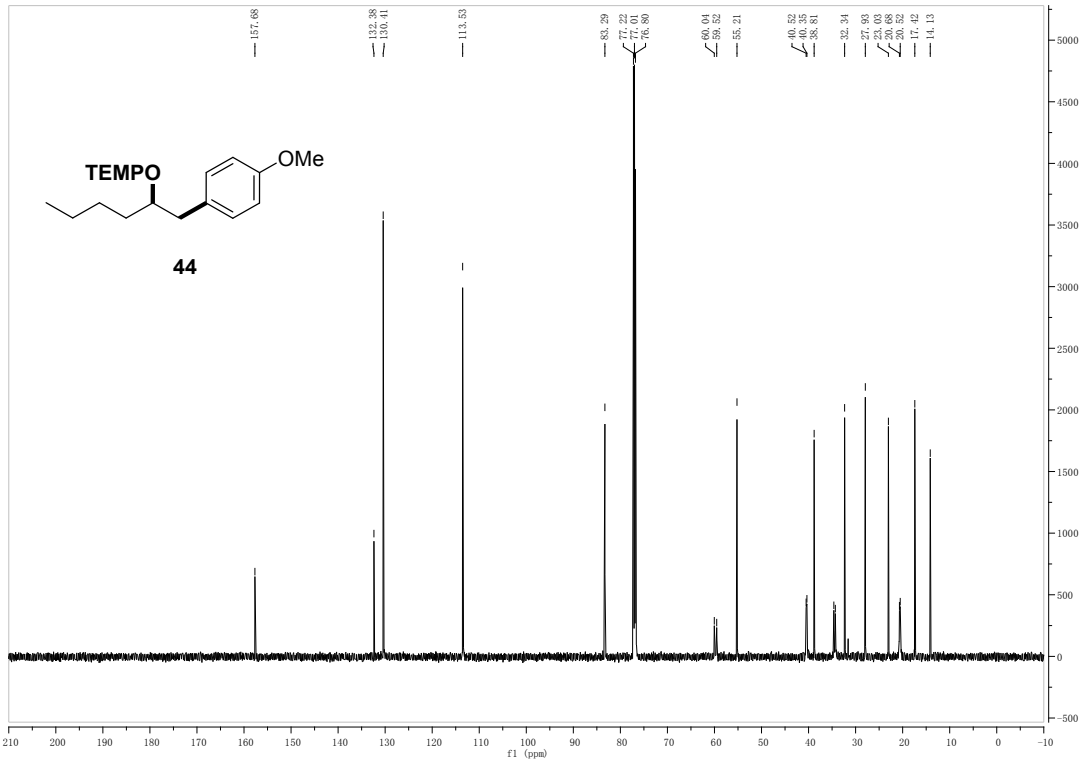

8.56
8.56
8.55
8.55
7.48
7.48
7.47
7.47
7.46
7.46
7.45
7.26
7.05
6.95
6.94
4.98
4.97
4.96
4.96
3.51
3.50
3.49
3.48
3.26
3.25
3.24
3.23
1.85
1.36
1.31
1.26
1.26
1.18
1.03
0.36
TEMPO
N
42
0.96
1.00
4.96
2.06
1.02
1.04
1.06
12.99
3.54
2.98
f1 (ppm)

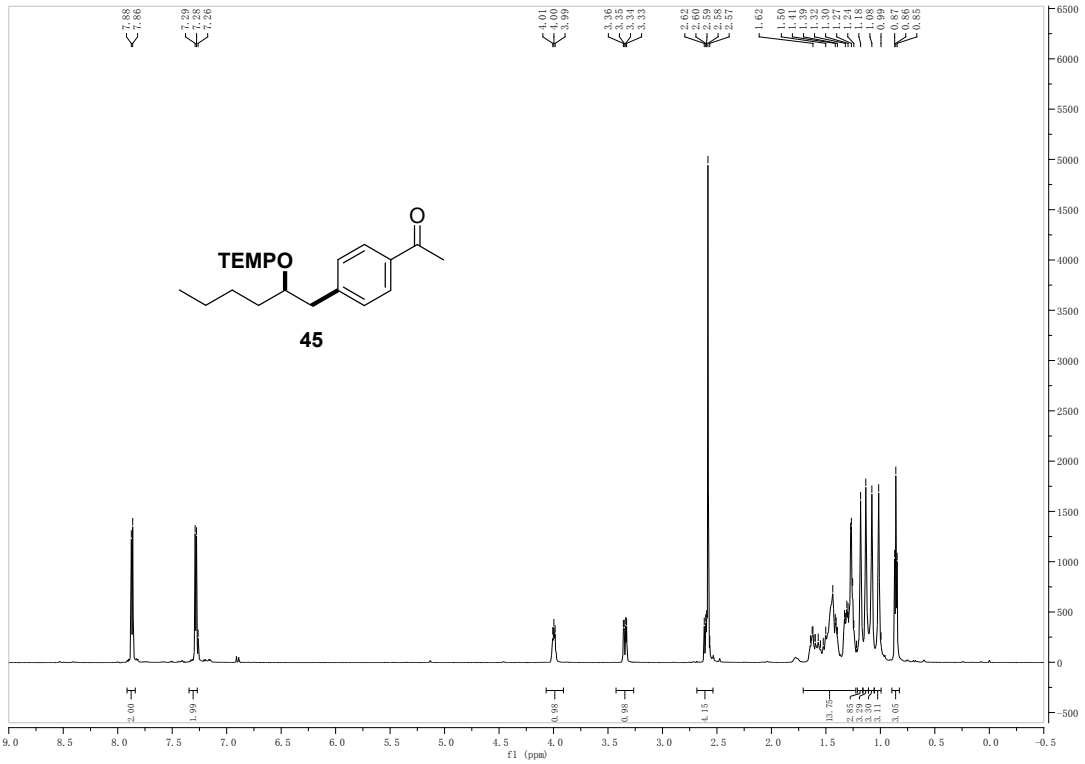

161.58
148.83
138.16
135.35
129.65
127.90
125.91
124.22
122.18
89.00
77.22
77.01
76.79
59.28
41.05
40.48
34.21
33.52
20.38
17.18
TEMPO
N
42
f1 (ppm)

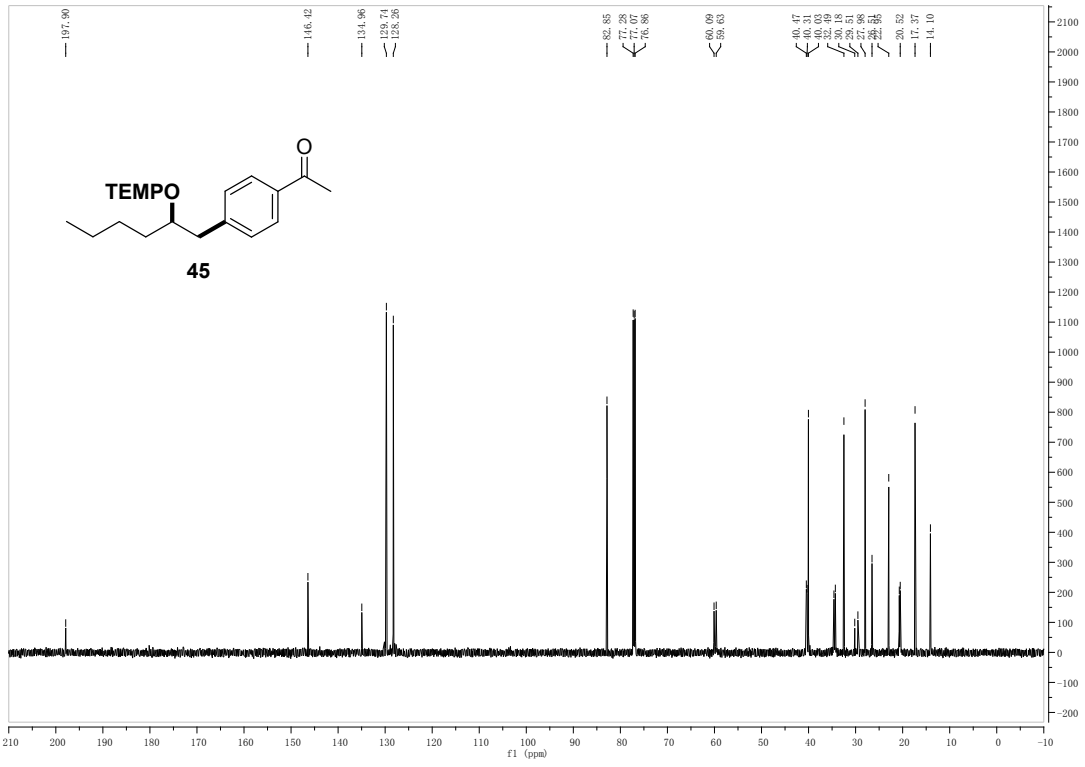

TEMPO
43
f1 (ppm)

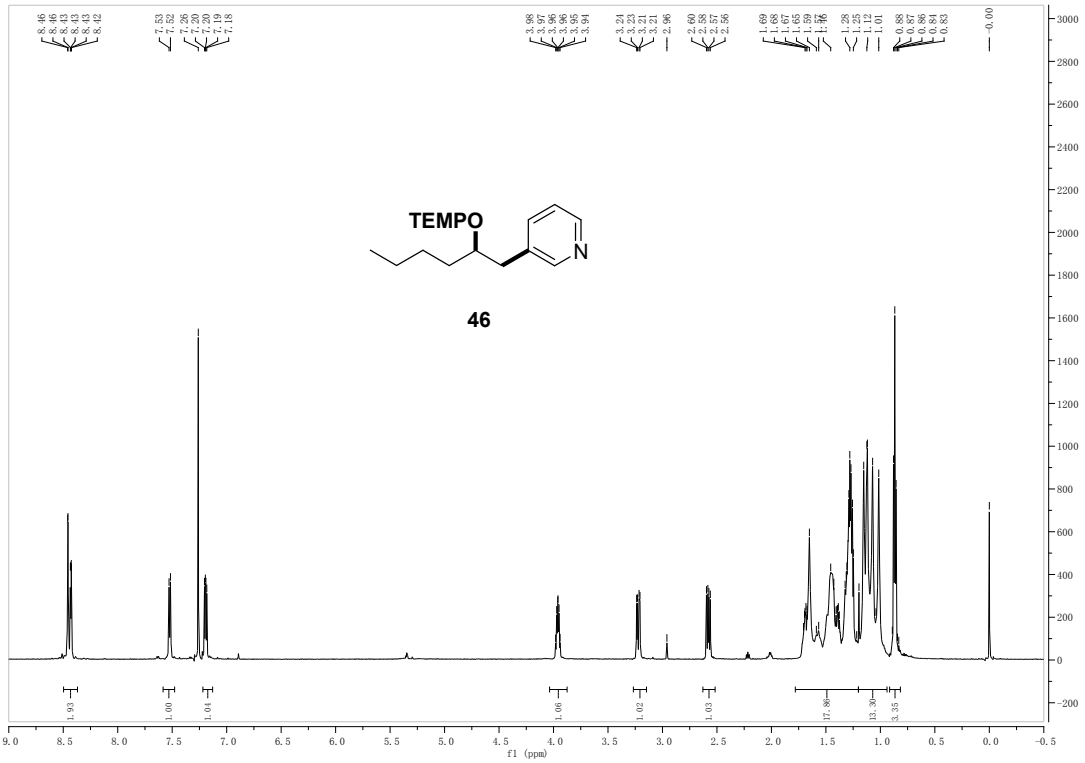

TEMPO
43
f1 (ppm)

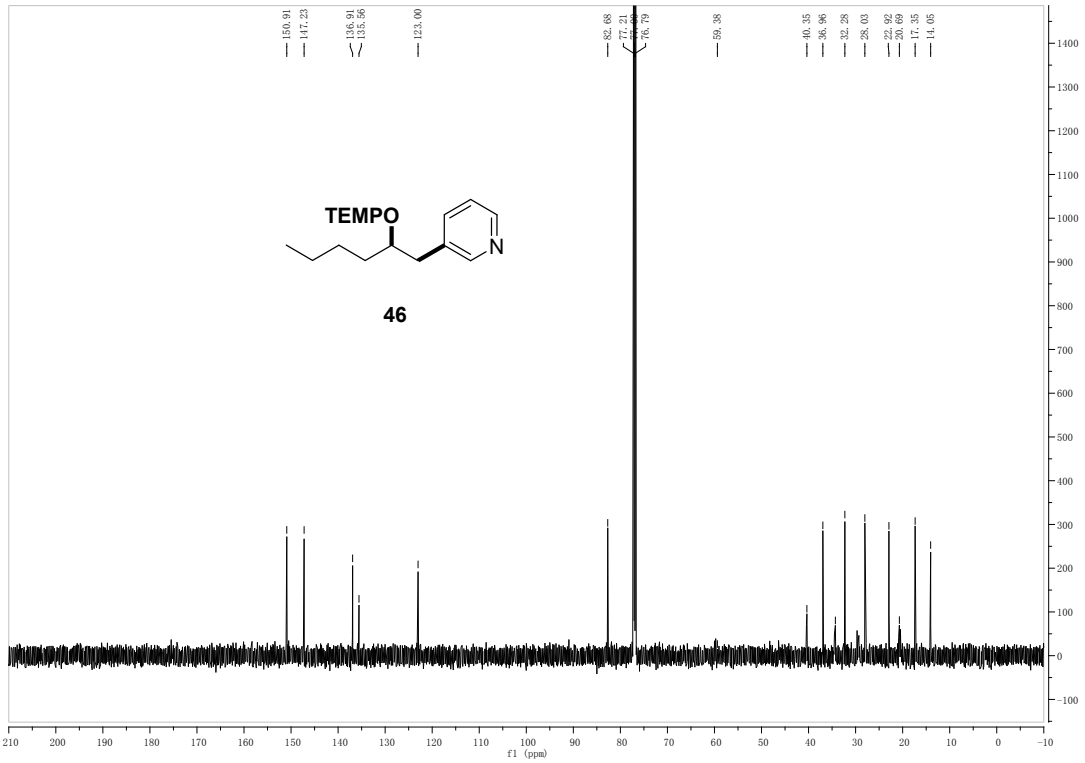

TEMPO
OMe
44
f1 (ppm)

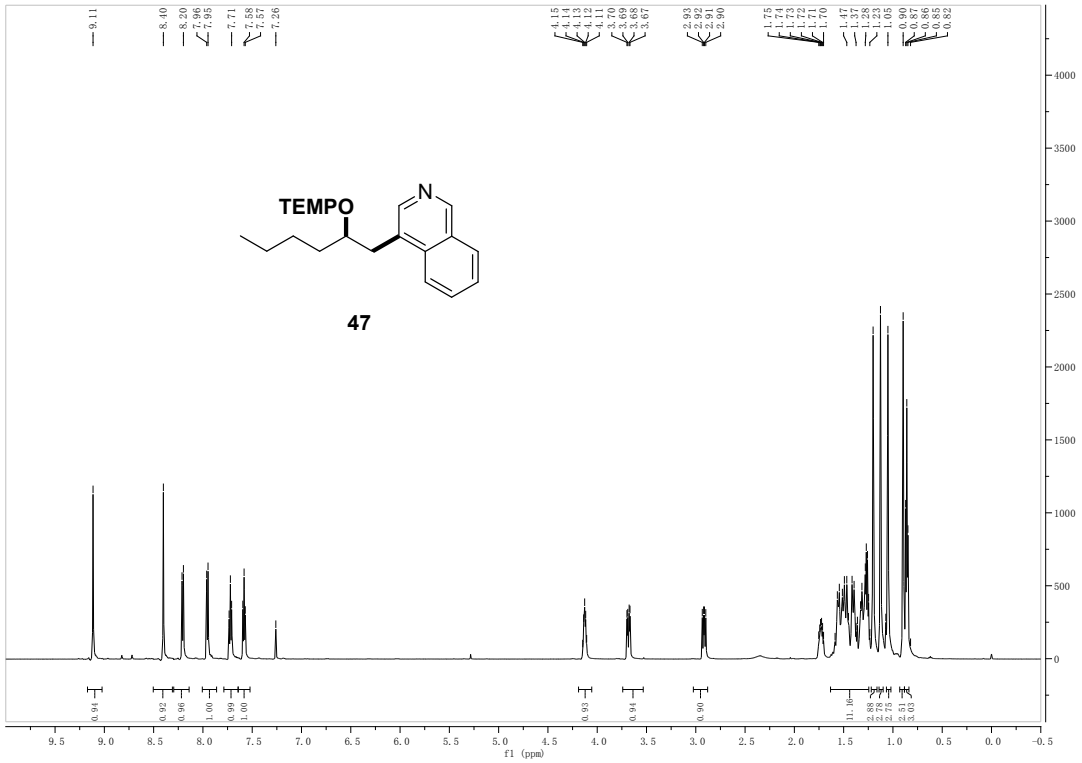

TEMPO
OMe
44
f1 (ppm)

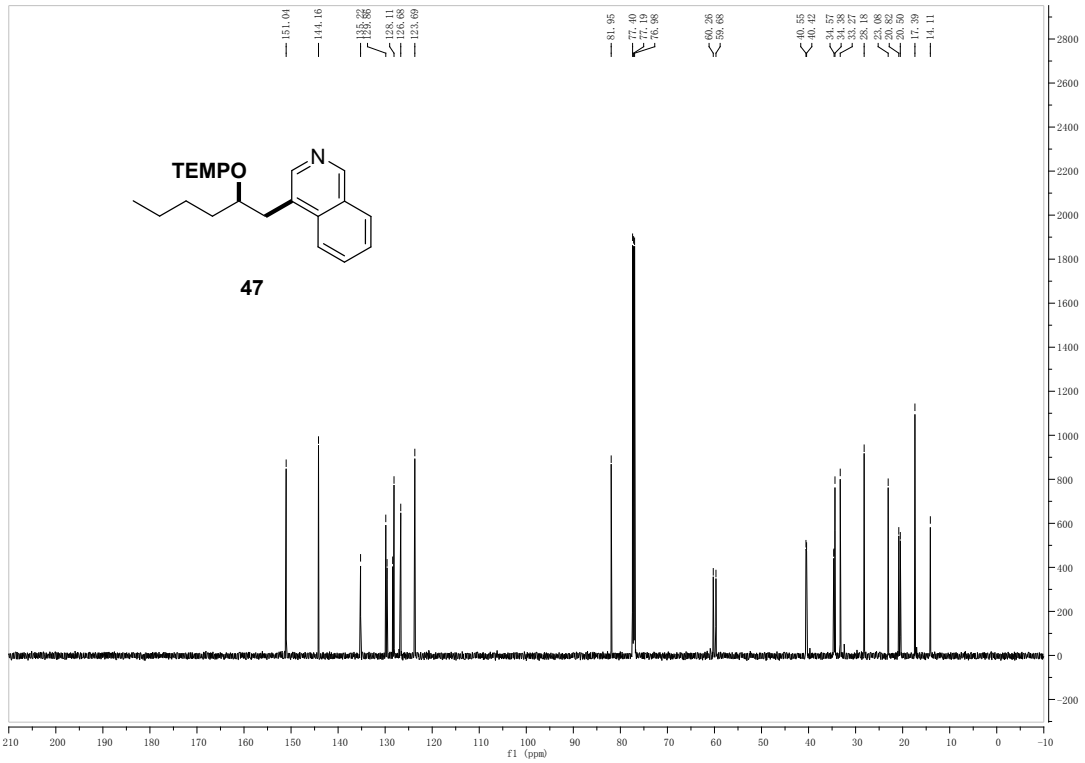

TEMPO
O
45
f1 (ppm)

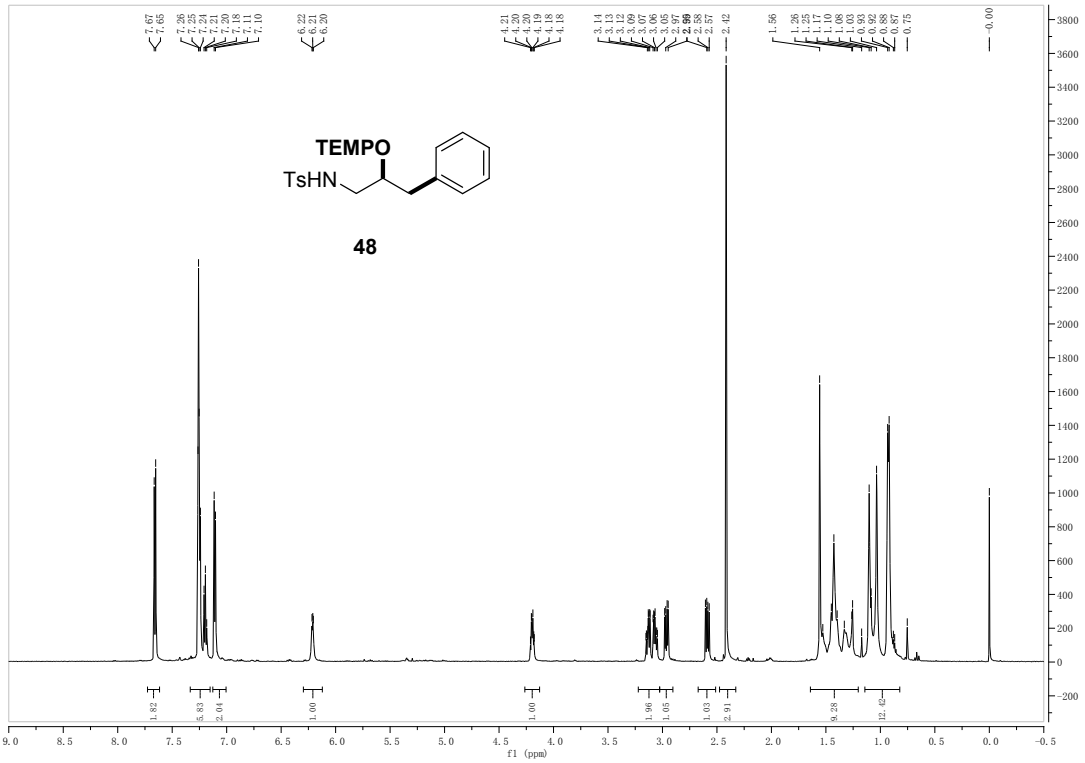

TEMPO
O
45
f1 (ppm)

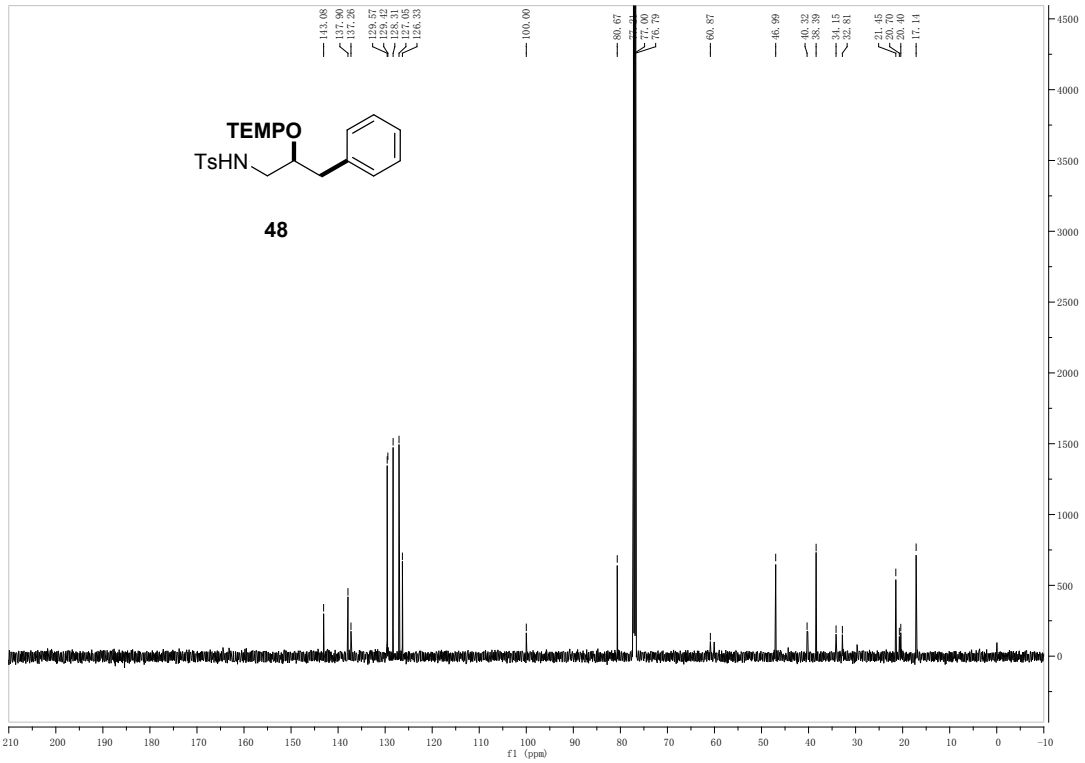

TEMPO
N
46
f1 (ppm)

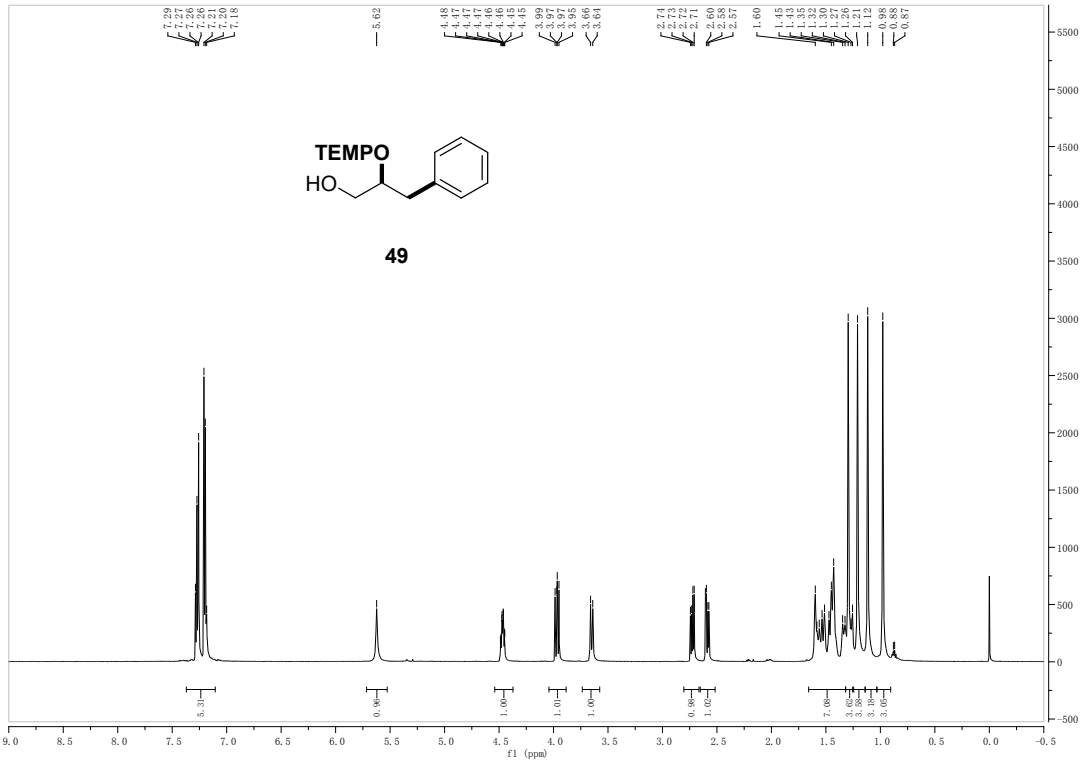

TEMPO
N
46
f1 (ppm)

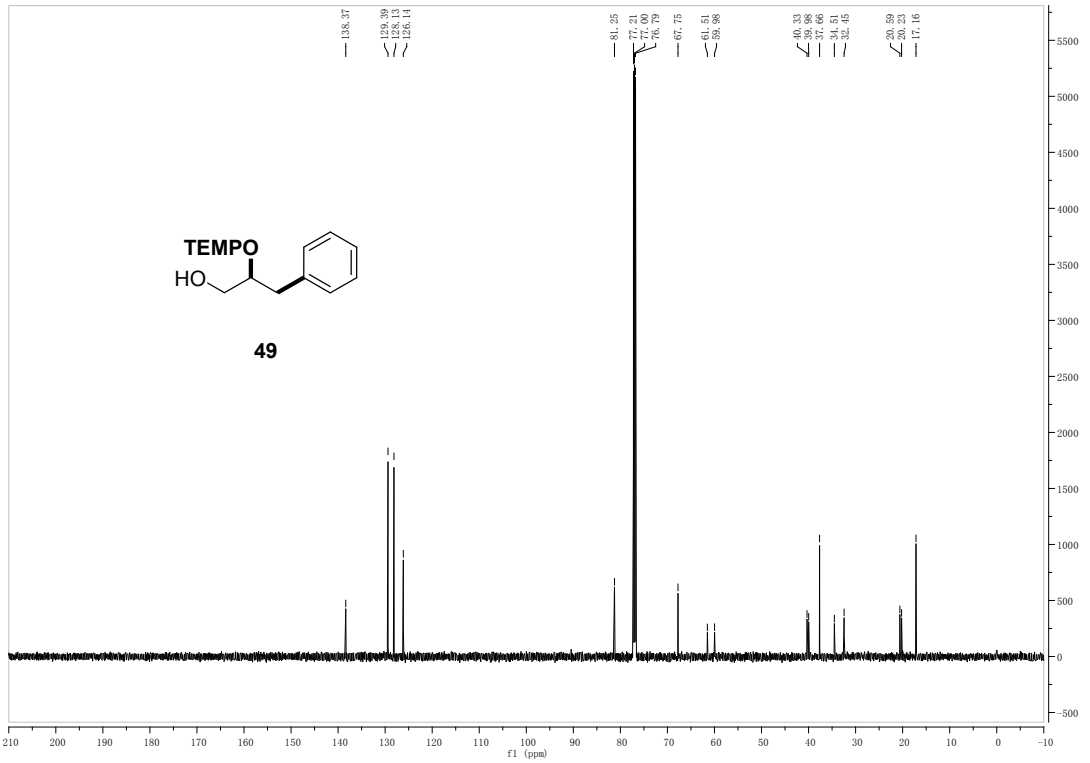

9.11
8.40
8.20
7.96
7.95
7.71
7.58
7.57
7.26
4.15
4.14
4.13
4.12
4.11
3.70
3.69
3.68
3.67
2.93
2.92
2.91
2.90
1.75
1.74
1.73
1.72
1.71
1.70
1.47
1.37
1.28
1.23
1.05
0.99
0.87
0.86
0.85
0.82
TEMPO
N
47
0.94
0.92
0.96
1.00
0.99
1.00
0.93
0.94
0.90
11.16
2.88
2.78
2.75
2.51
3.03
f1 (ppm)

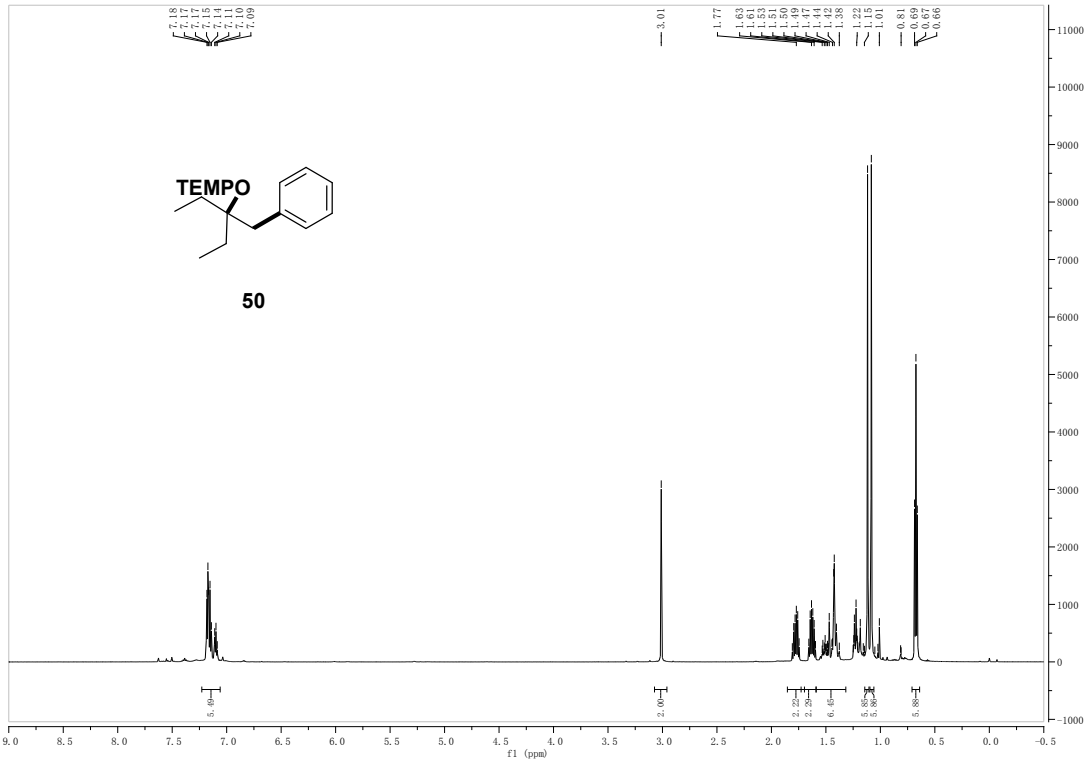

151.04
144.16
135.22
129.86
128.11
126.68
123.69
81.95
77.40
77.19
76.98
60.26
59.68
40.55
40.42
34.57
34.38
33.47
28.15
26.48
20.82
20.50
17.39
14.11
TEMPO
N
47
f1 (ppm)

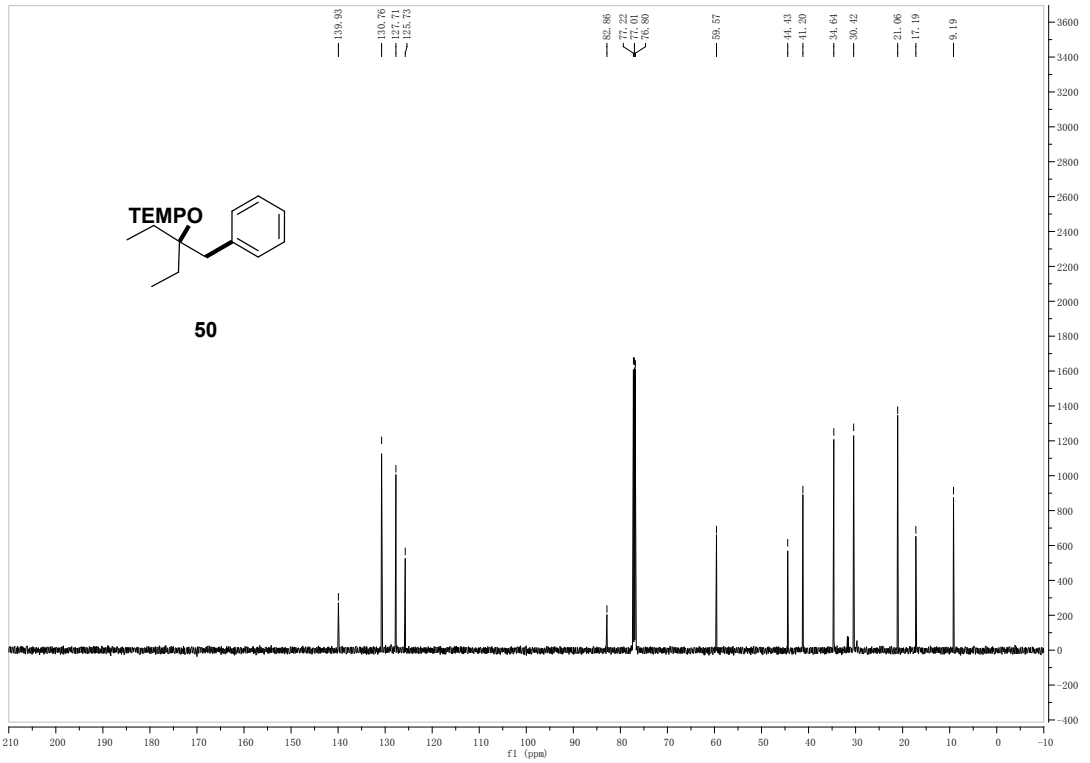

TEMPO
TsHN
48

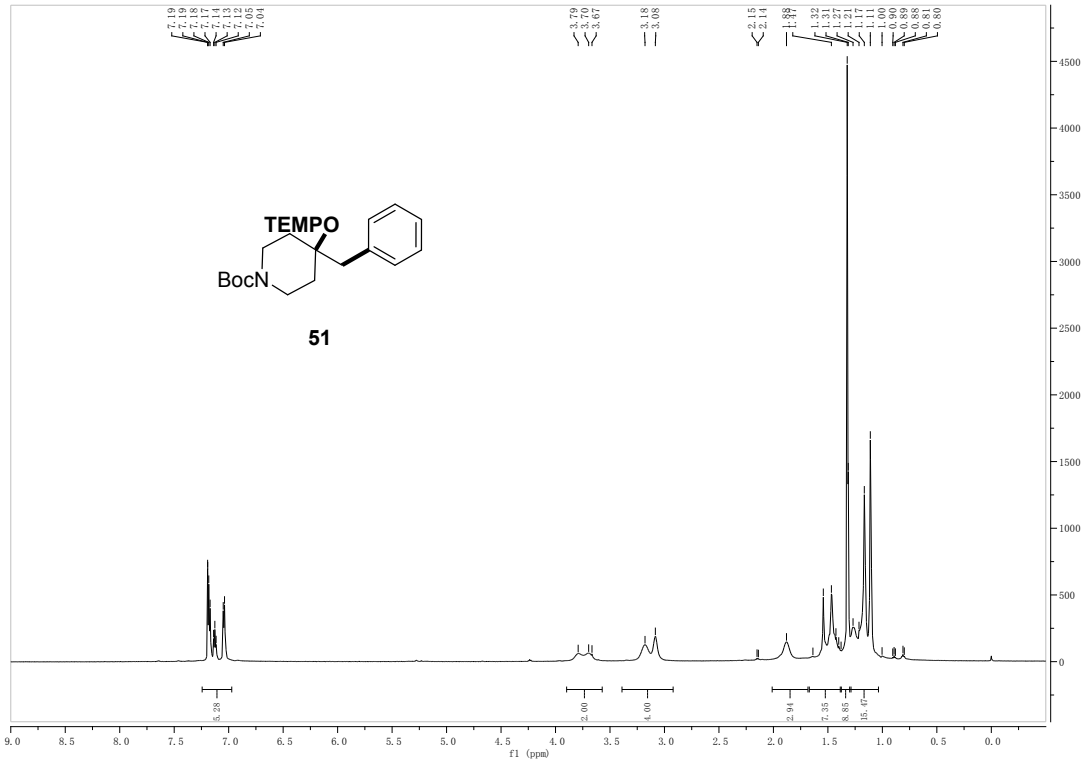

TEMPO
TsHN
48

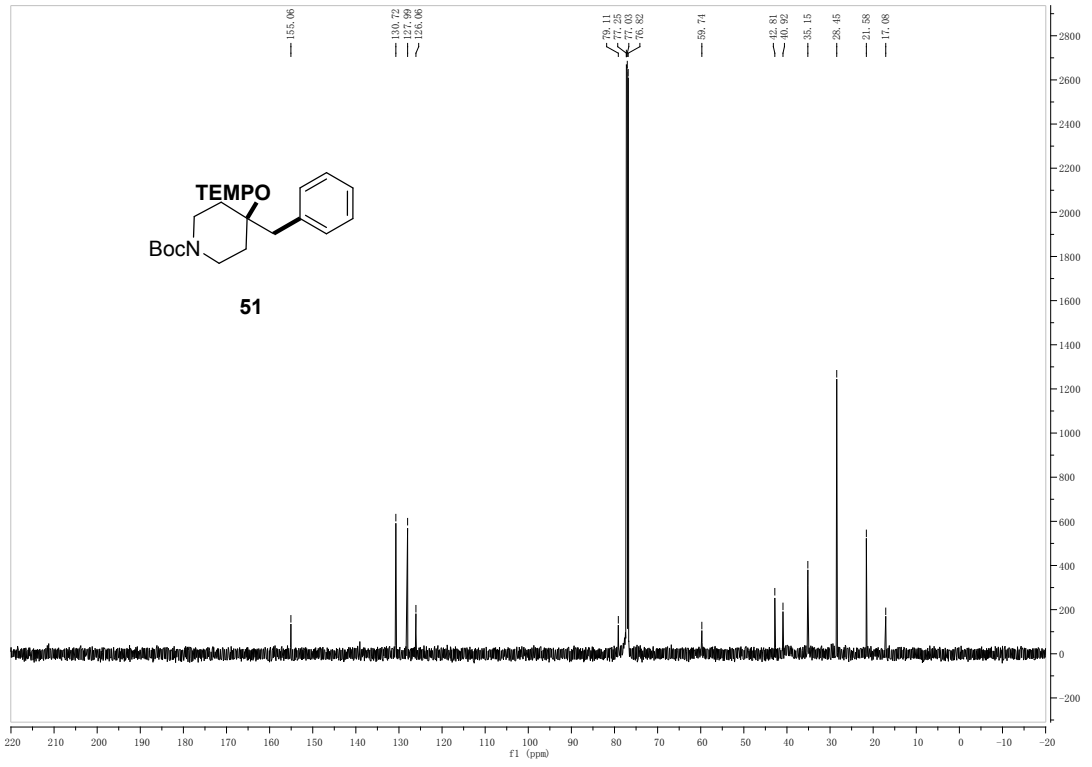

TEMPO
HO
49
f1 (ppm)

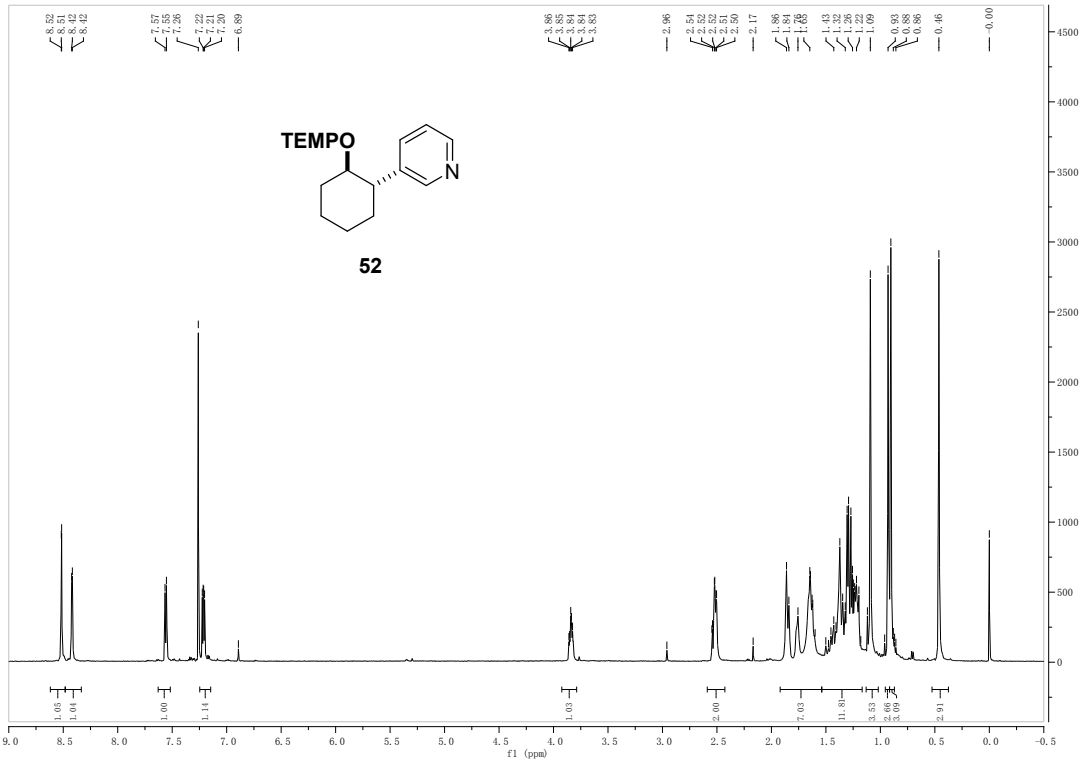

TEMPO
HO
49
f1 (ppm)

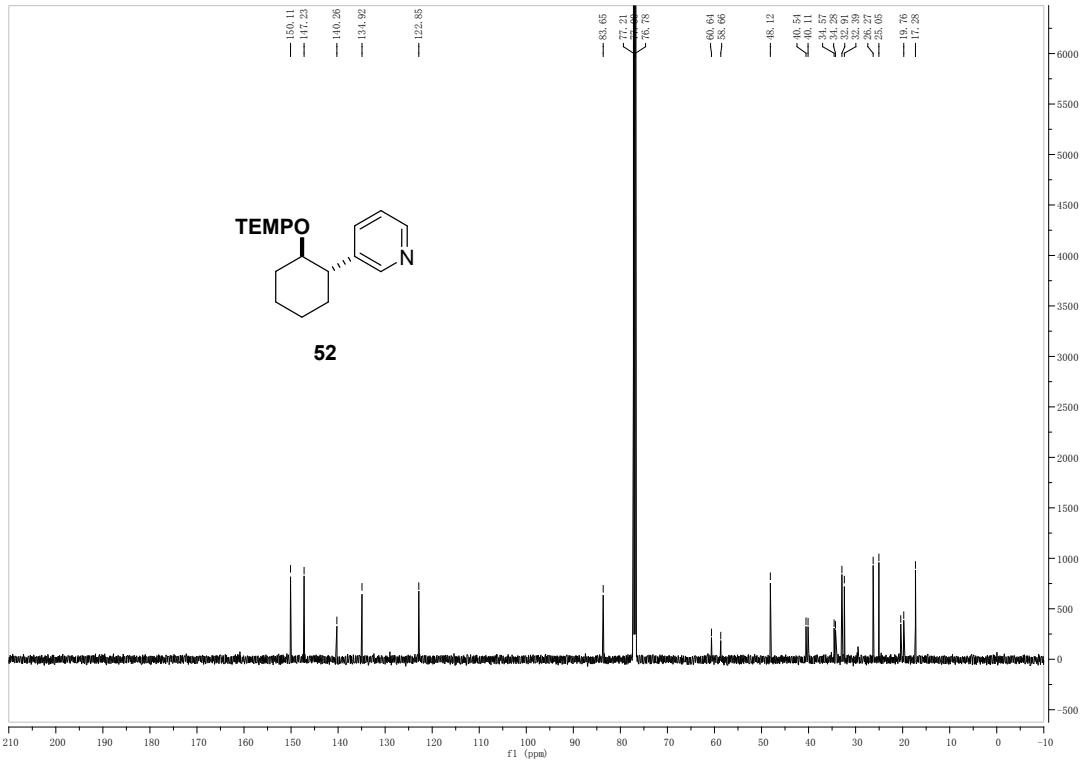

TEMPO
50
f1 (ppm)

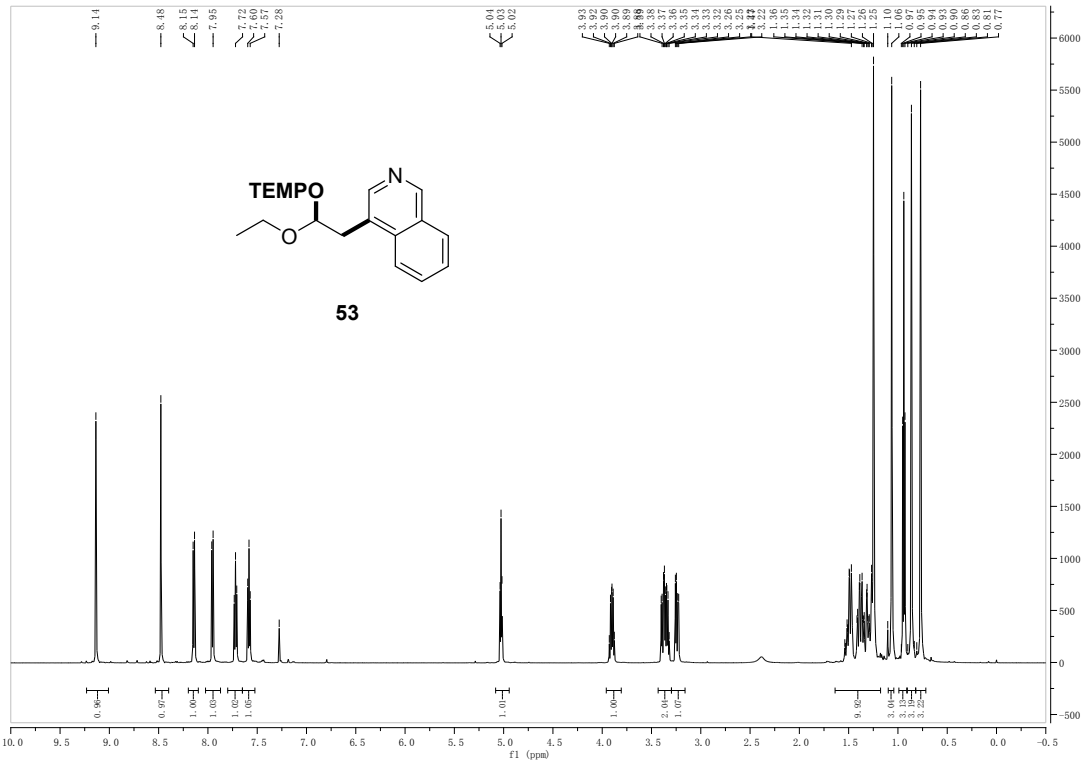

TEMPO
50
f1 (ppm)

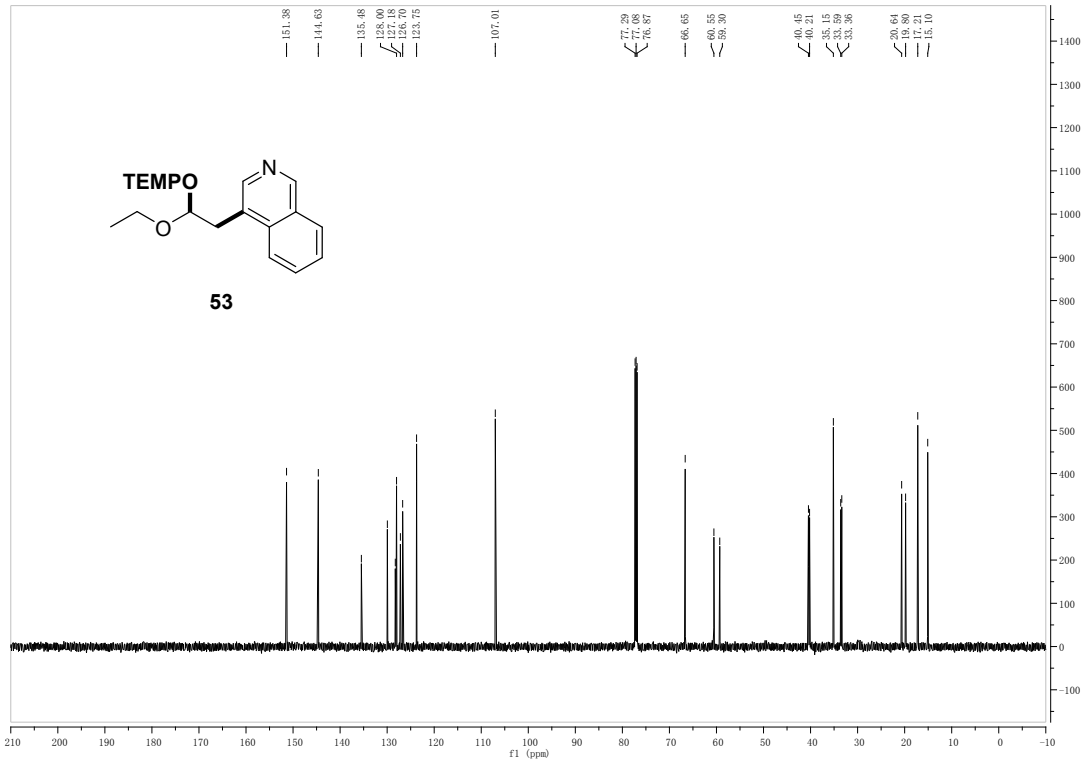

TEMPO
BocN
51
f1 (ppm)

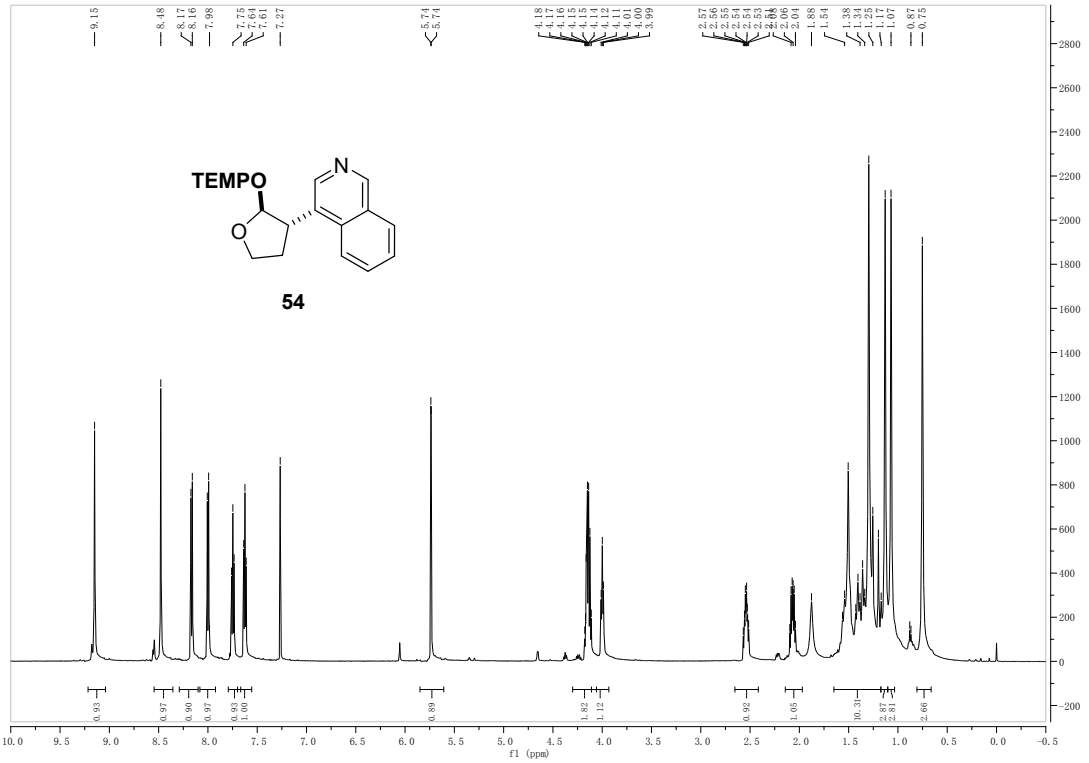

TEMPO
BocN
51
f1 (ppm)

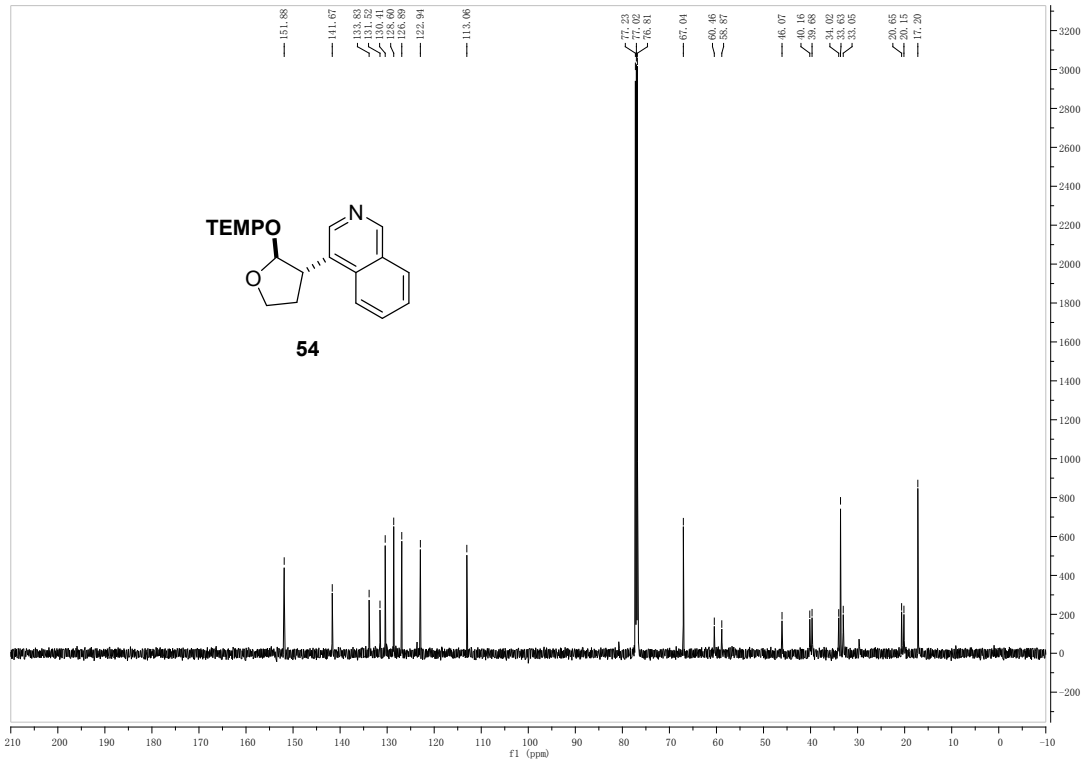

TEMPO
N
52
f1 (ppm)

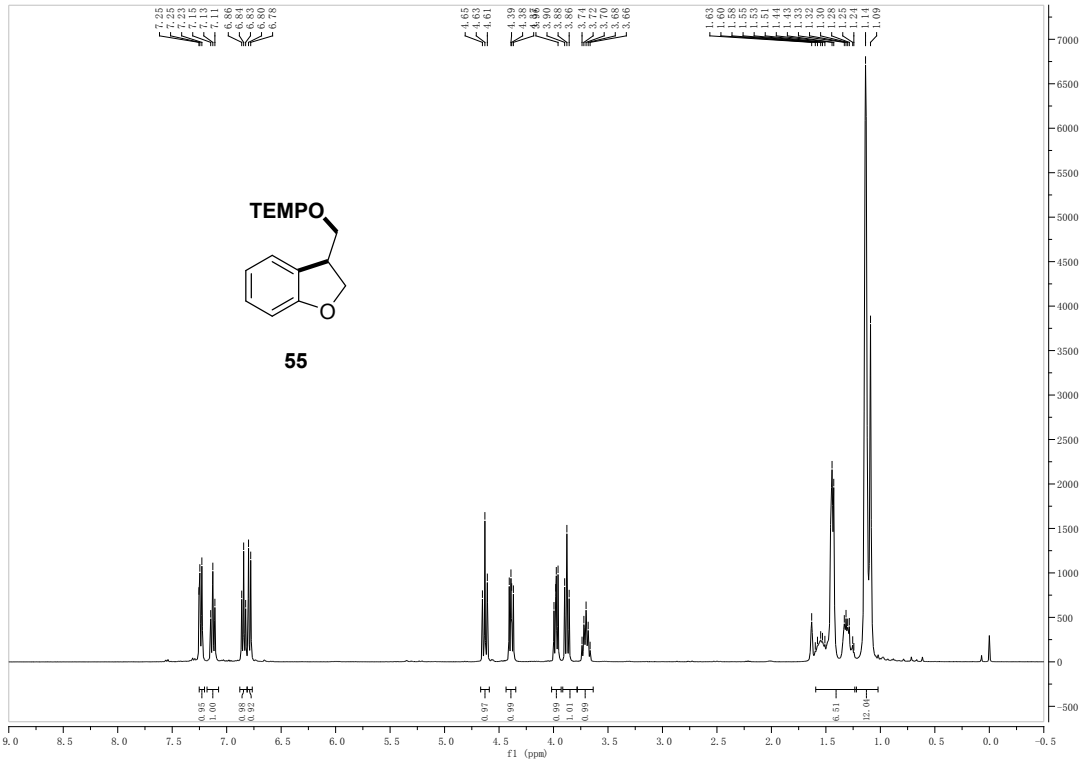

TEMPO
N
52
f1 (ppm)

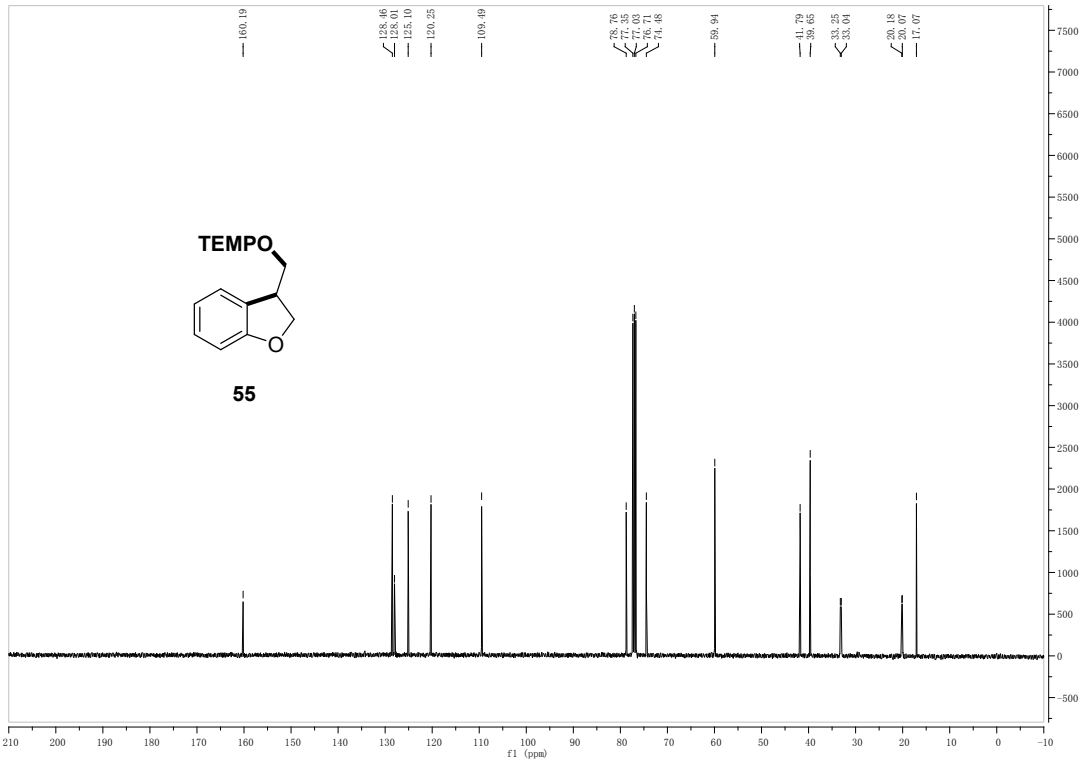

TEMPO
N
O
53
f1 (ppm)

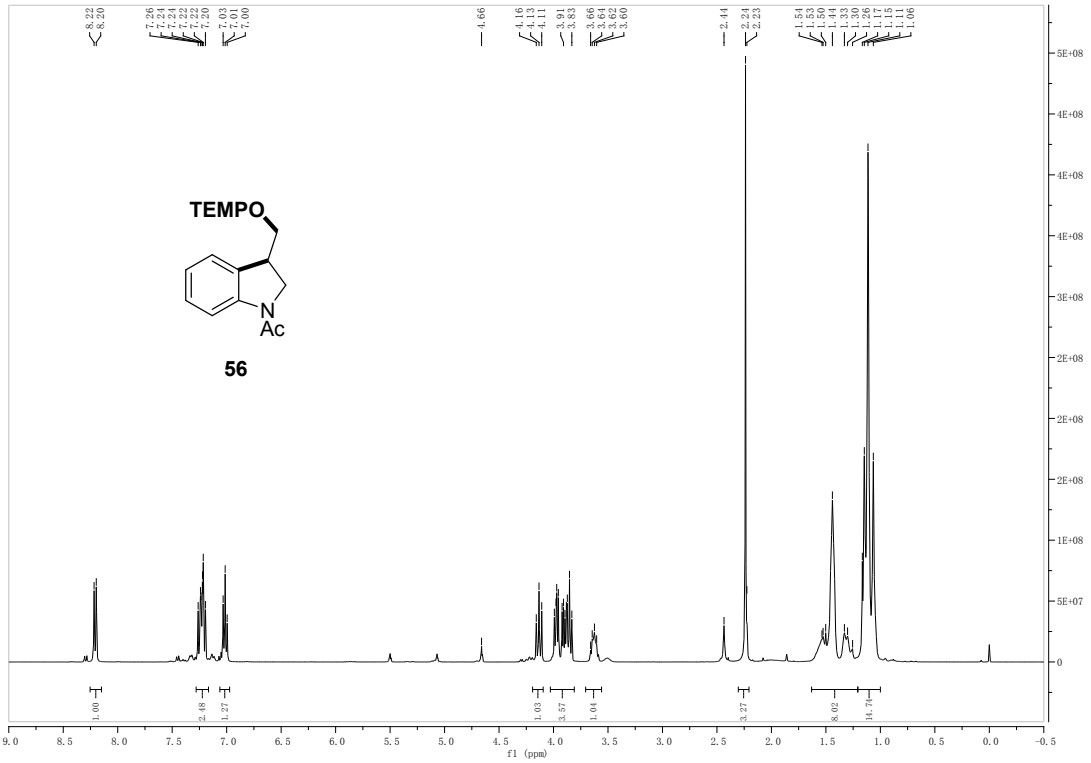

TEMPO
N
O
53
f1 (ppm)

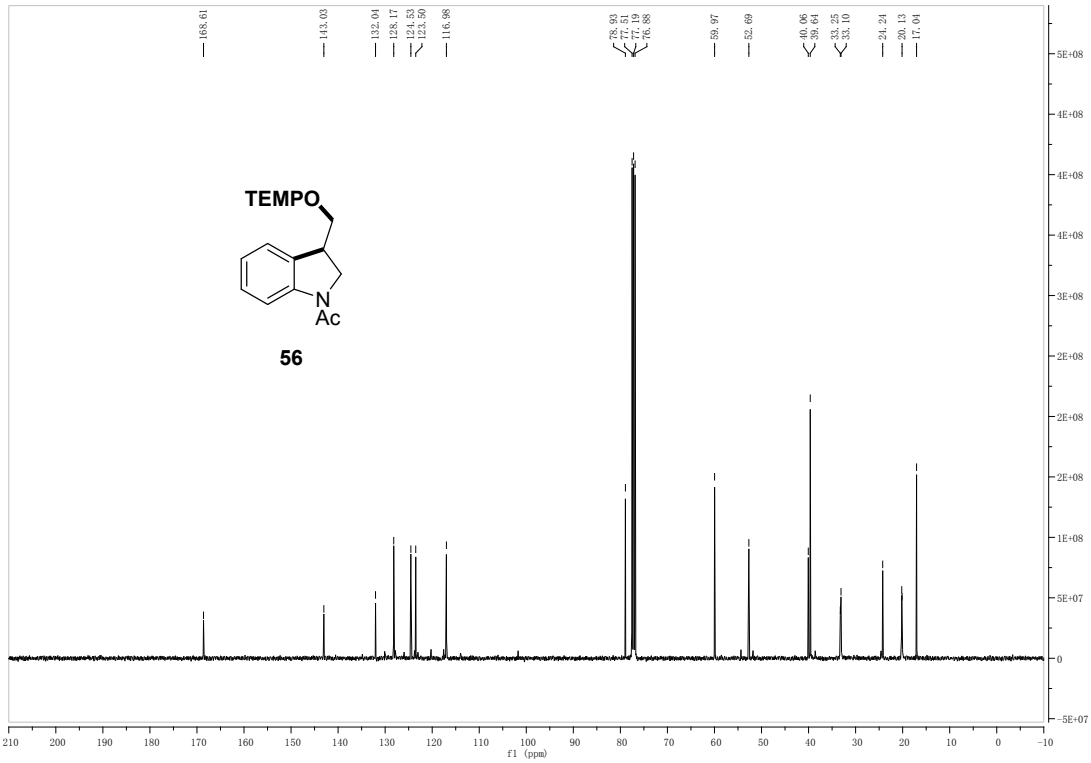

TEMPO
54
f1 (ppm)

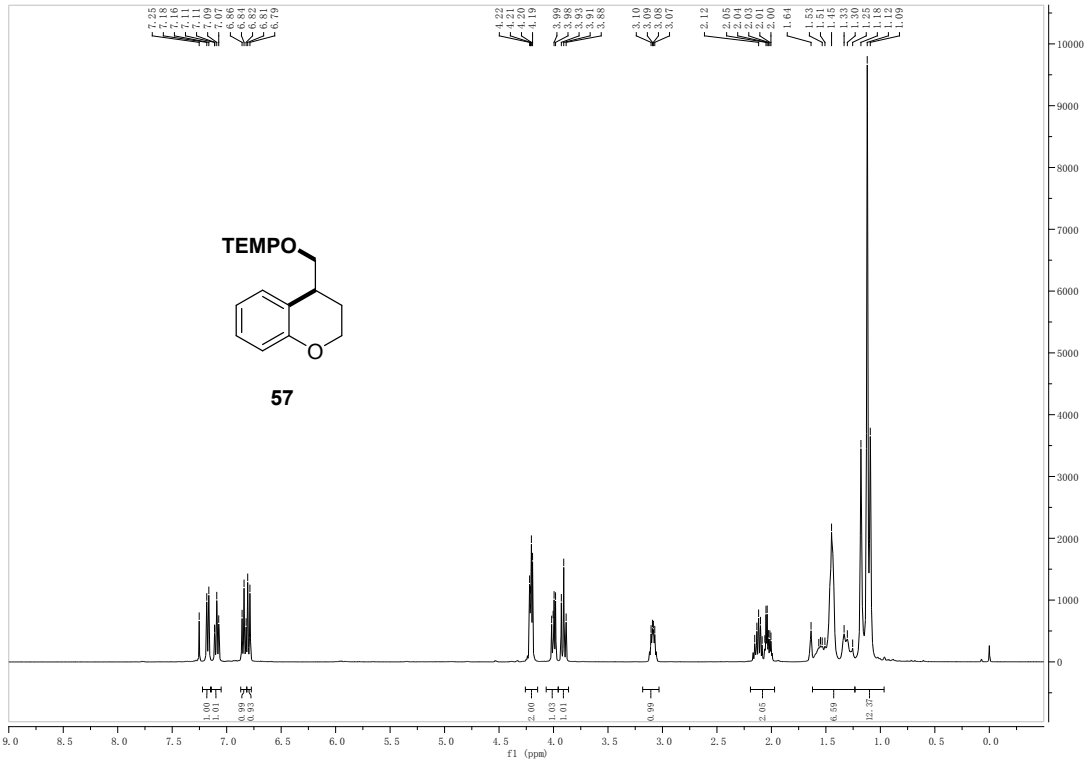

TEMPO
54
f1 (ppm)

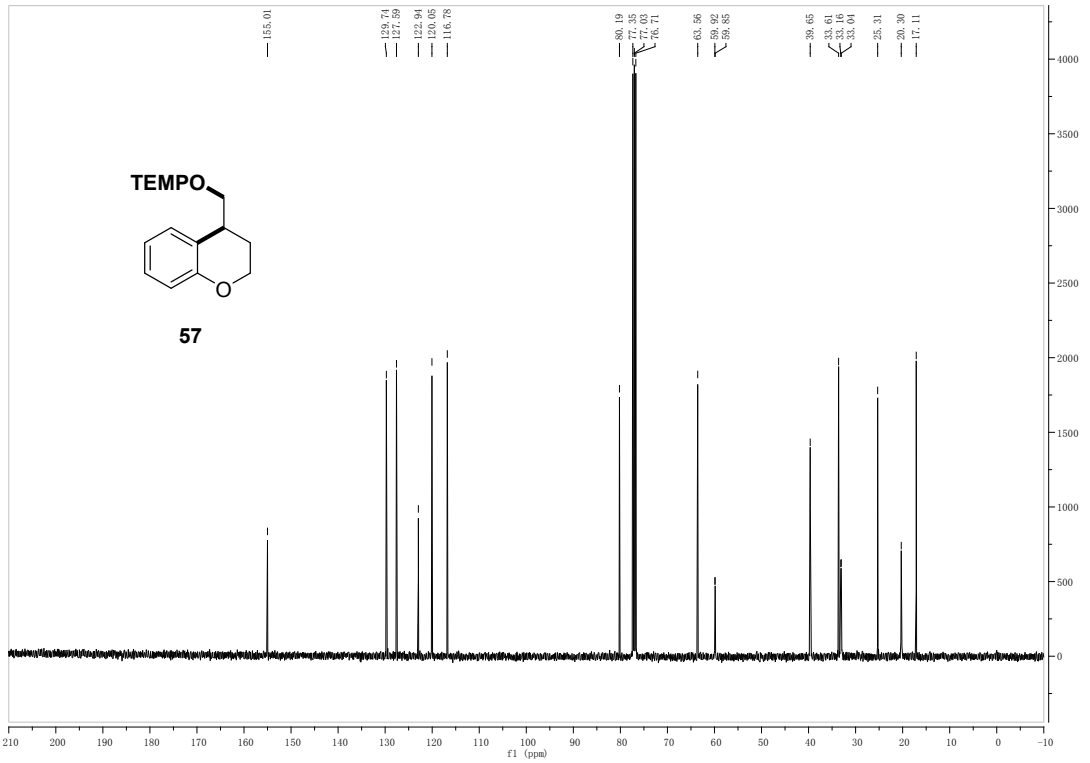

TEMPO
55
f1 (ppm)

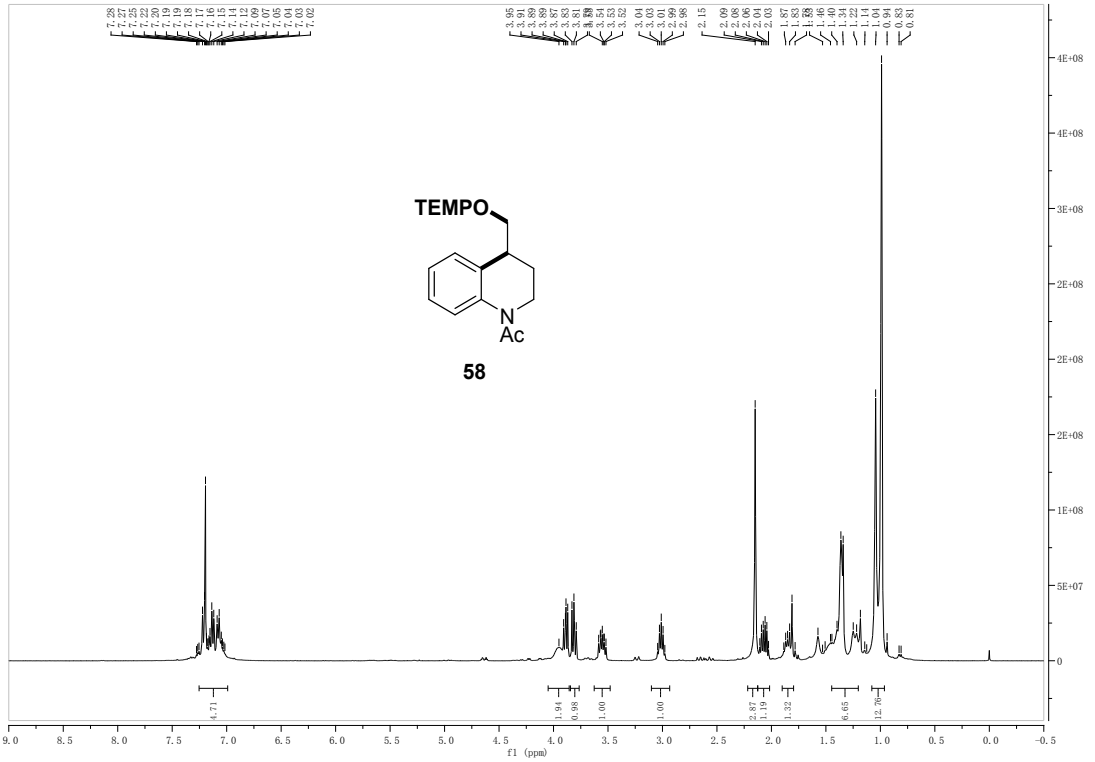

TEMPO
55
f1 (ppm)

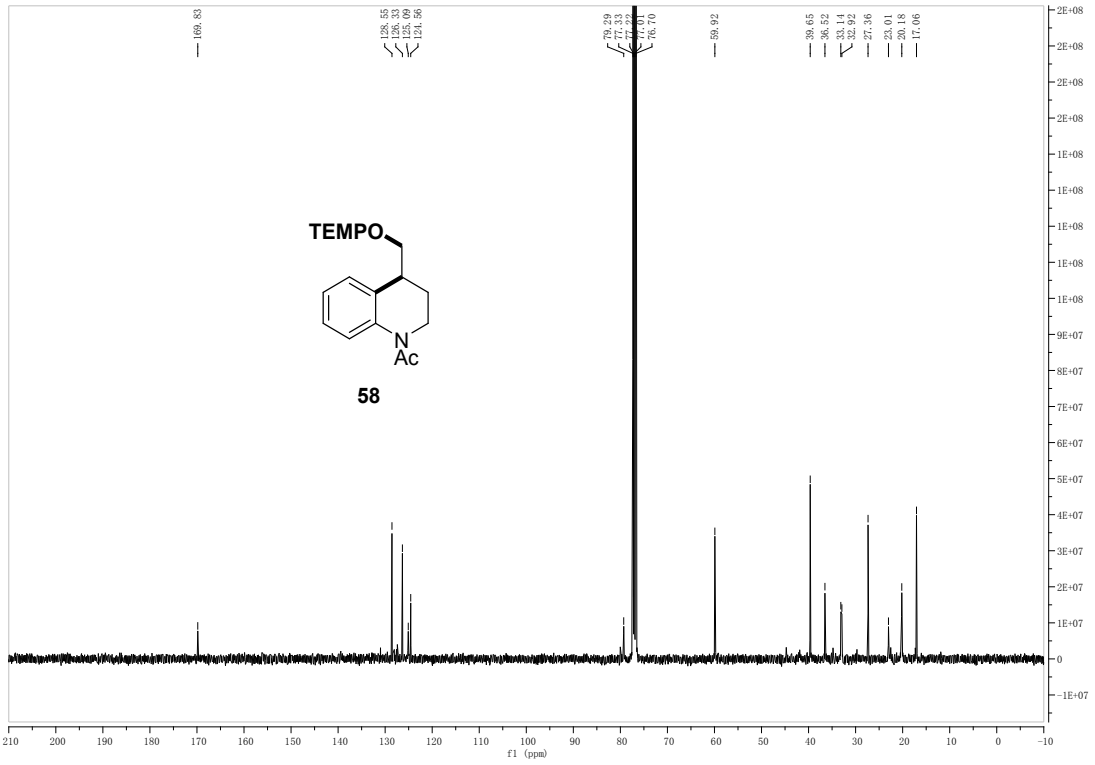

TEMPO
N
Ac
56
f1 (ppm)

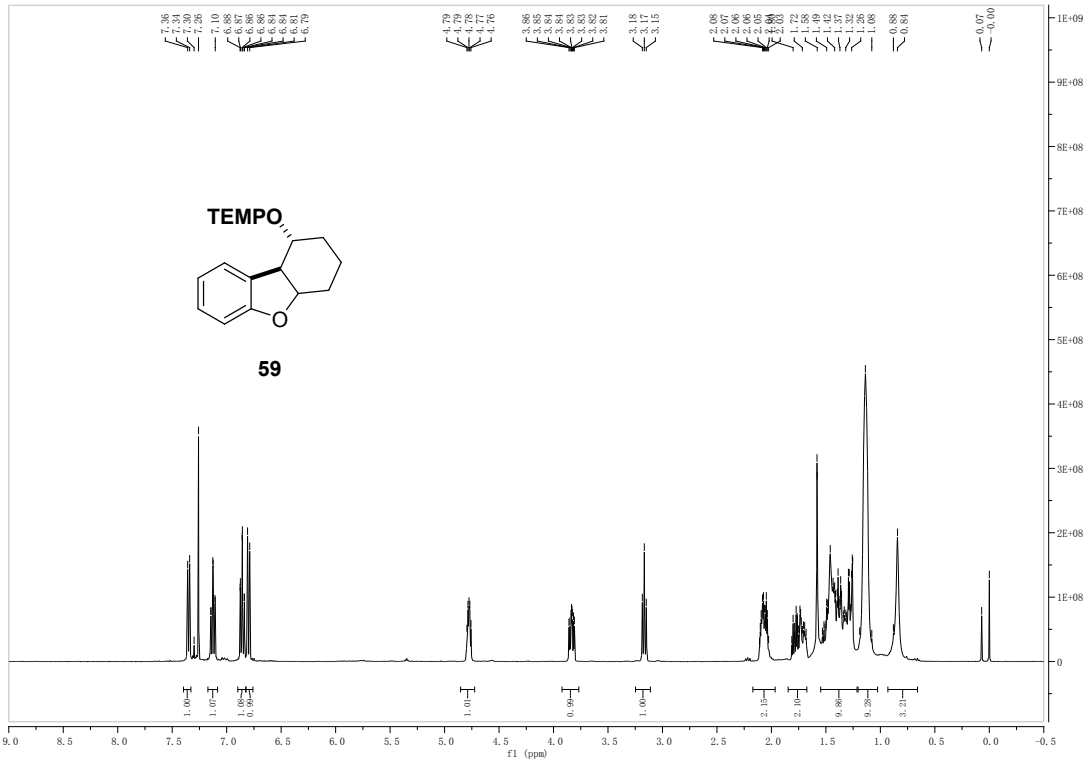

TEMPO
N
Ac
56
f1 (ppm)

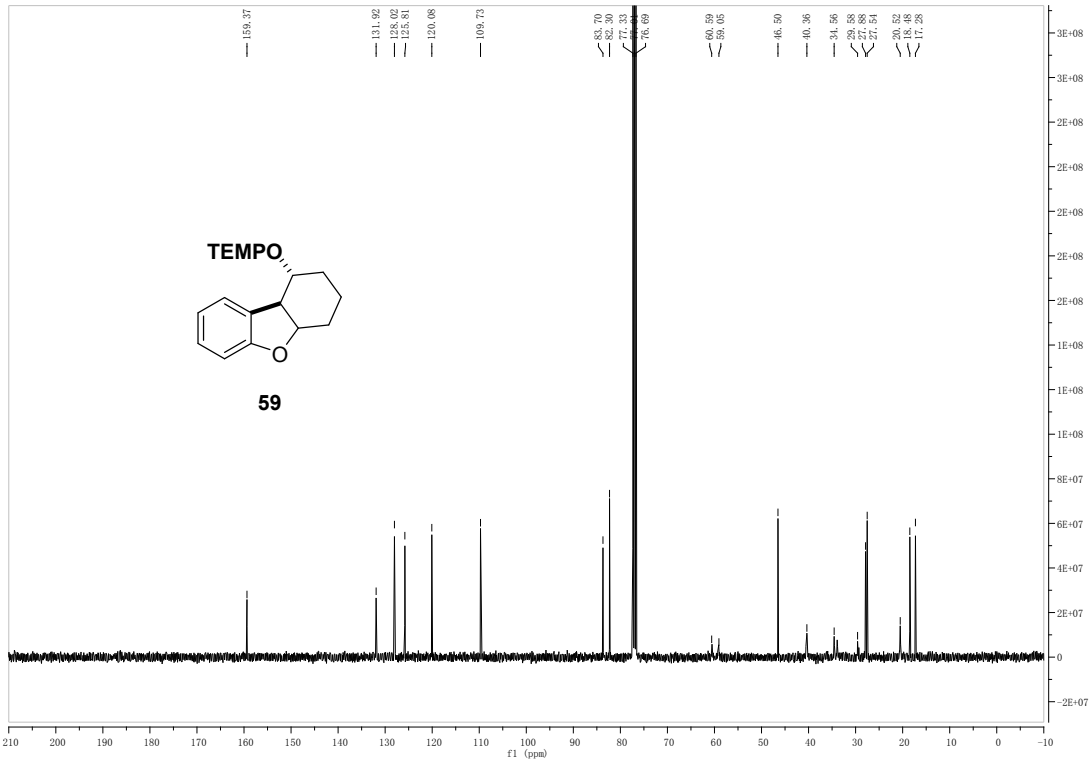

TEMPO
O
57
7.25
7.18
7.16
7.11
7.11
7.09
7.07
6.86
6.84
6.82
6.81
6.79
4.22
4.21
4.20
4.19
3.99
3.98
3.93
3.91
3.88
3.10
3.09
3.08
3.07
2.12
2.05
2.04
2.03
2.01
2.00
1.64
1.53
1.51
1.51
1.45
1.33
1.30
1.25
1.16
1.12
1.09
1.00
1.01
0.99
0.93
2.00
1.03
1.01
0.99
2.05
6.59
12.37
f1 (ppm)

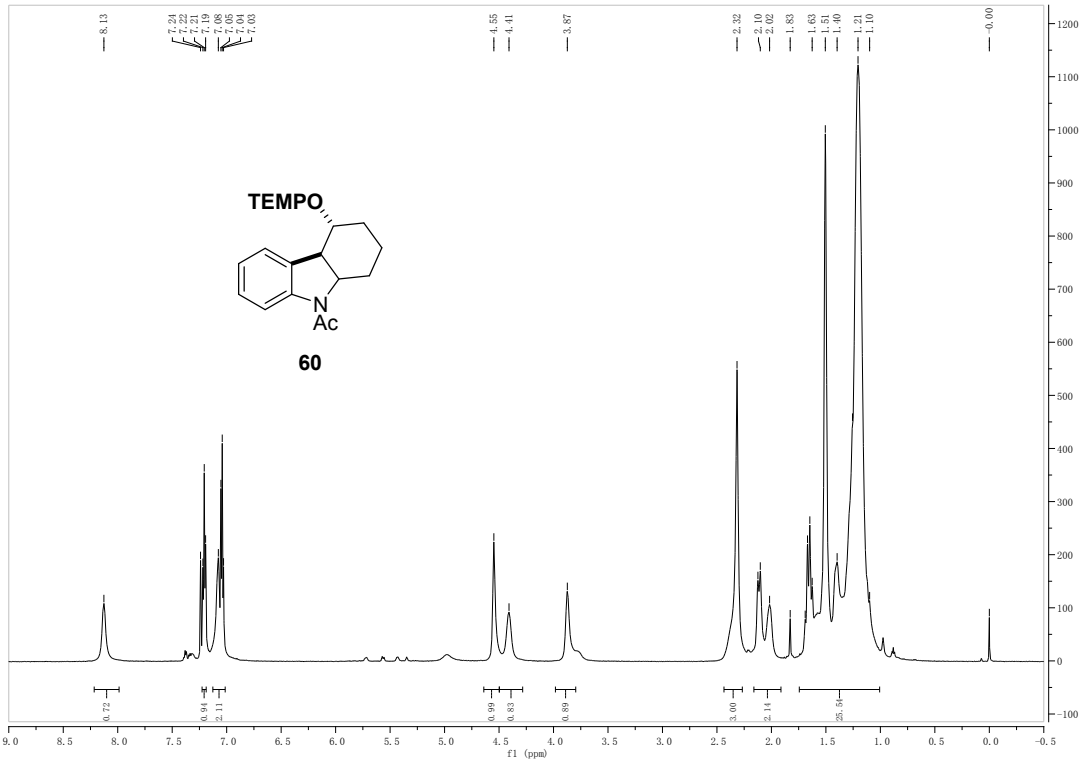

TEMPO
O
57
155.01
129.74
127.59
122.94
120.65
116.78
80.19
77.35
77.03
76.71
63.56
59.92
59.85
39.65
33.61
33.16
33.04
25.31
20.30
17.11
f1 (ppm)

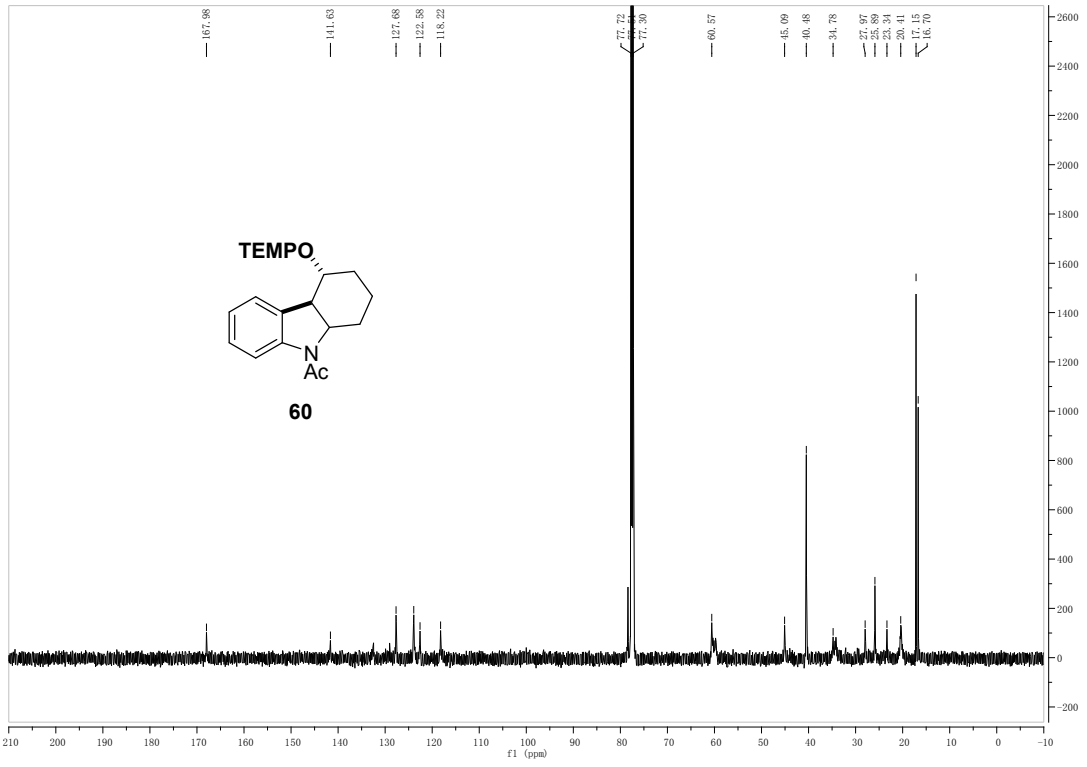

TEMPO
N
Ac
58
f1 (ppm)

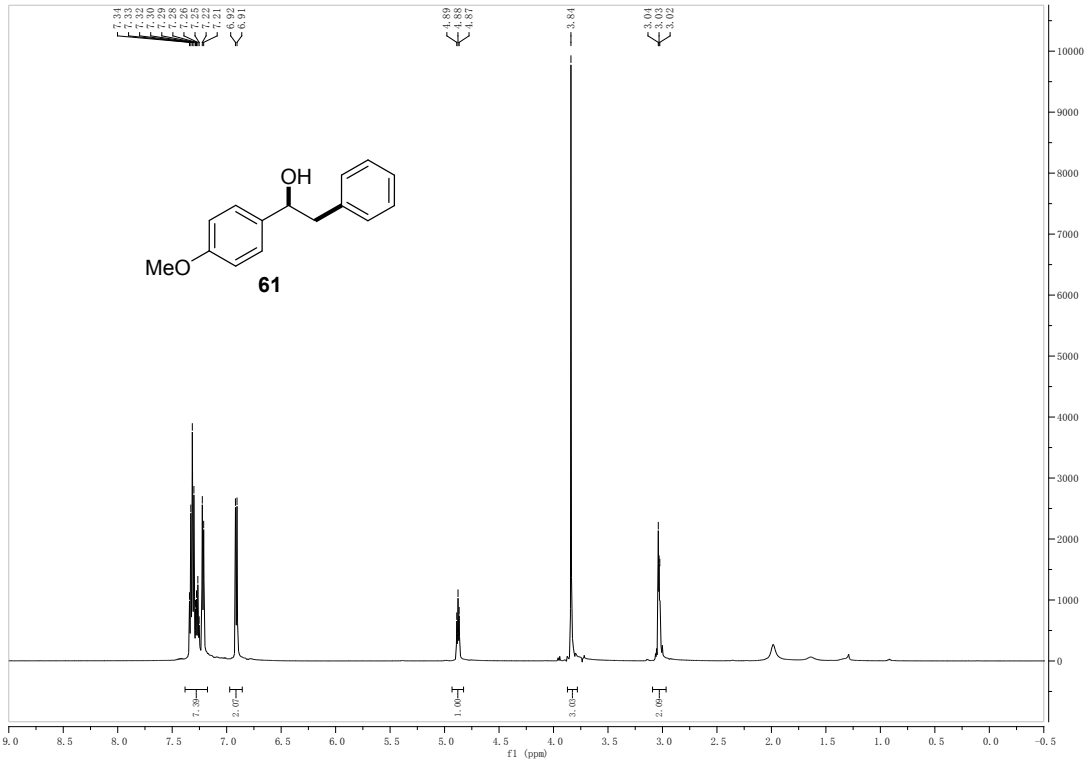

TEMPO
N
Ac
58
f1 (ppm)

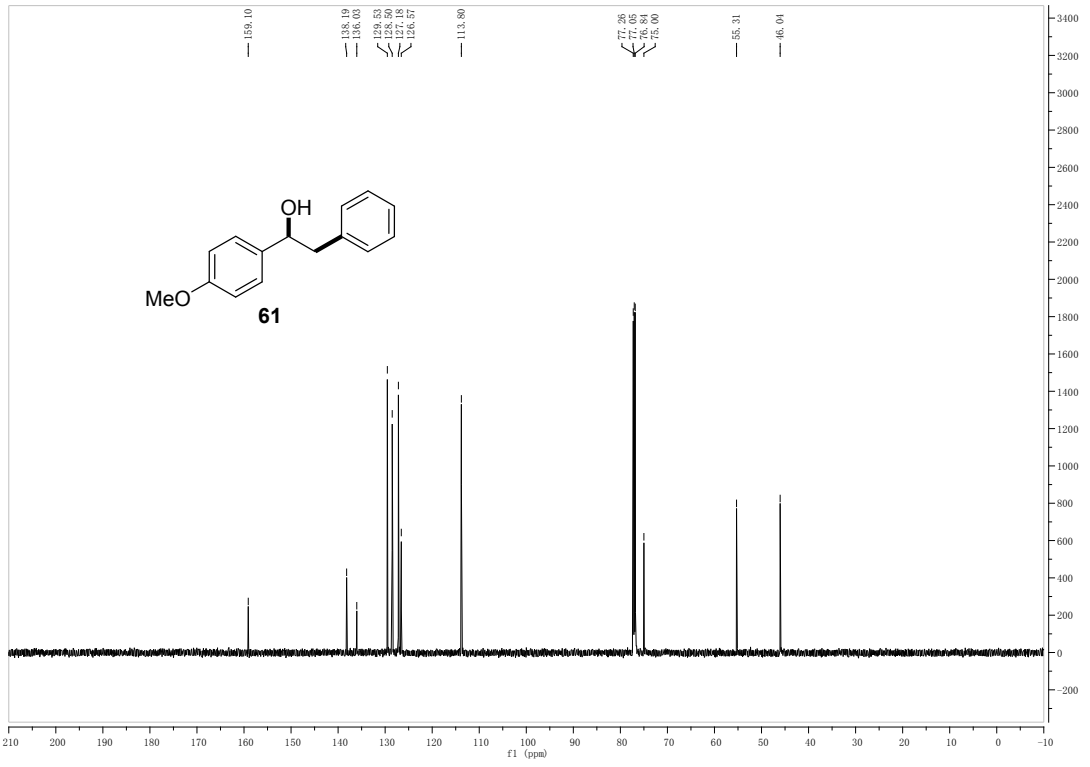

TEMPO
59
f1 (ppm)

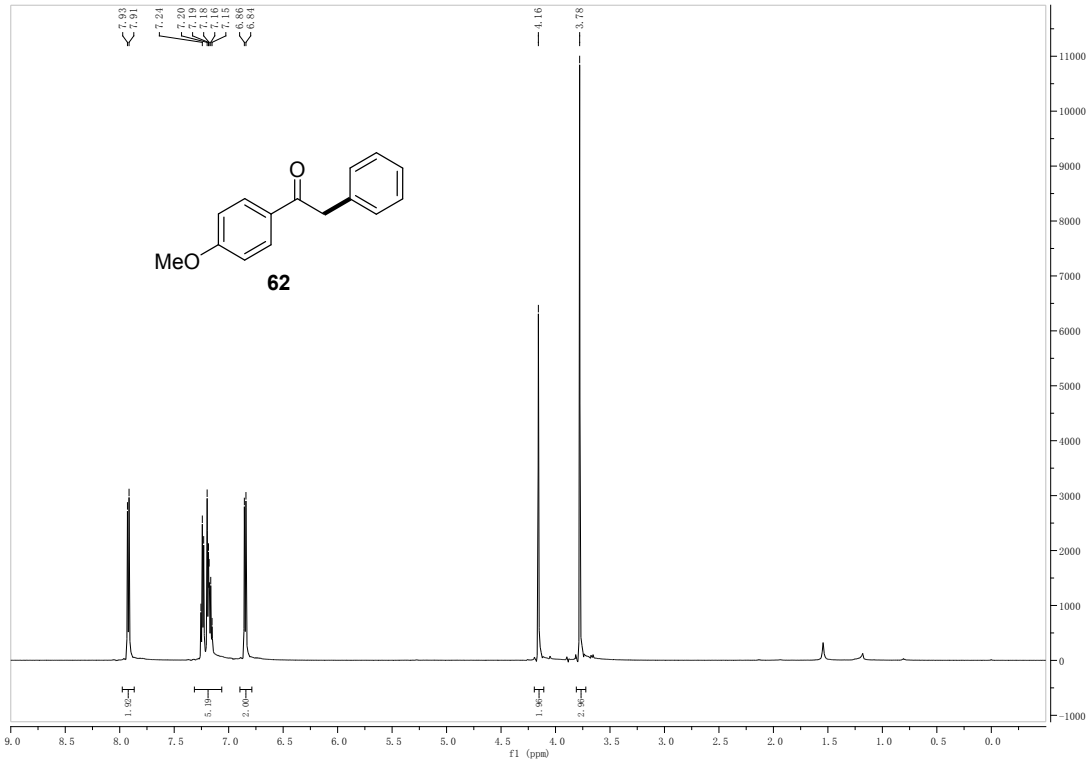

TEMPO
59
f1 (ppm)

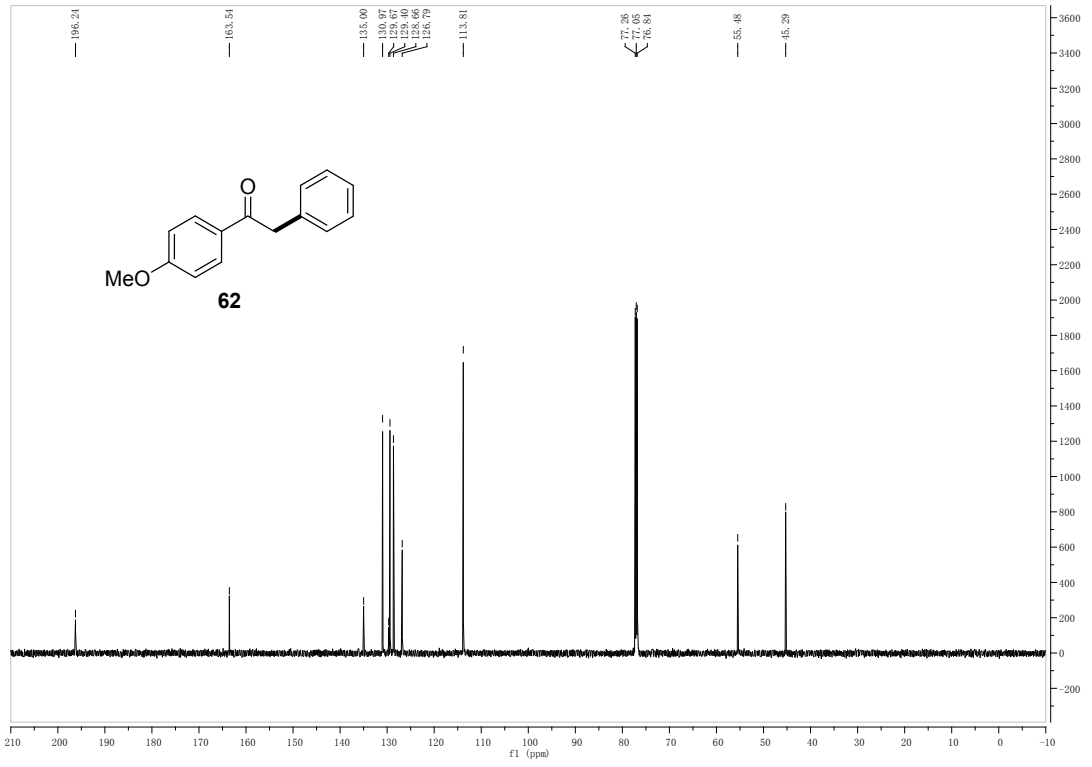

8.13
7.24
7.22
7.21
7.19
7.08
7.05
7.04
7.03
4.55
4.41
3.87
2.32
2.10
2.02
1.83
1.63
1.51
1.40
1.21
1.10
0.00
TEMPO
N
Ac
60
0.72
0.94
2.11
0.99
0.83
0.89
3.00
2.14
25.54
f1 (ppm)

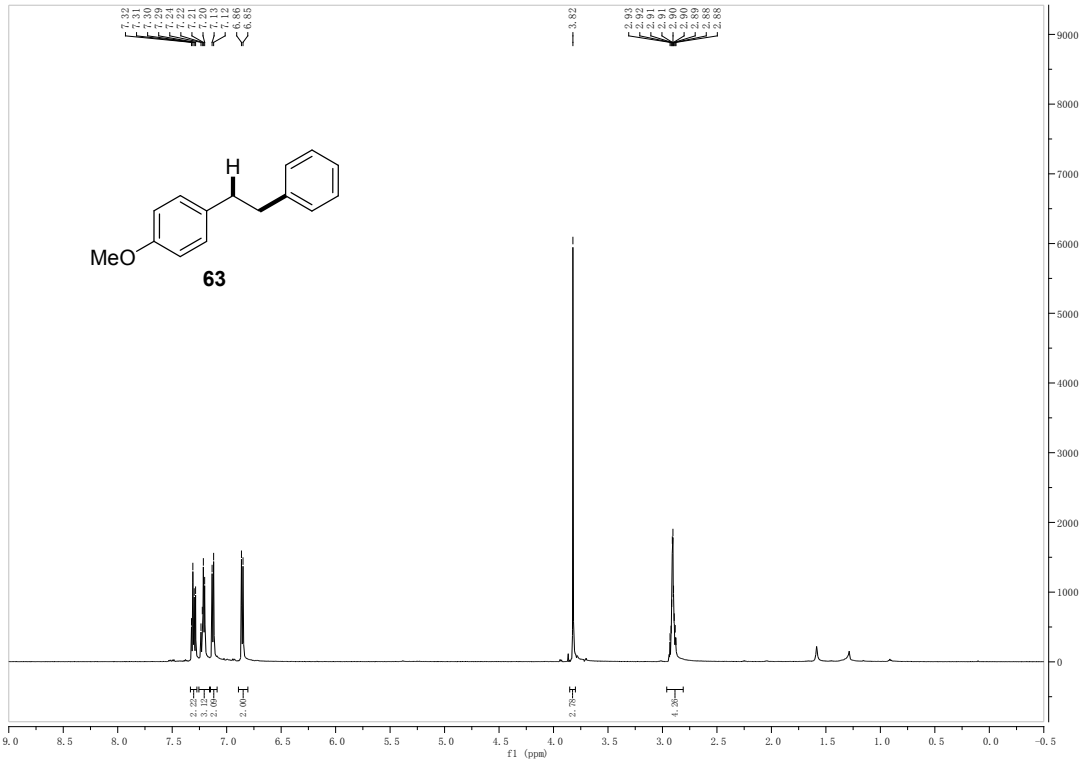

167.98
141.63
127.68
122.58
118.22
77.72
77.41
77.30
60.57
45.09
40.48
34.78
27.97
25.89
23.34
20.41
17.15
16.70
TEMPO
N
Ac
60
f1 (ppm)

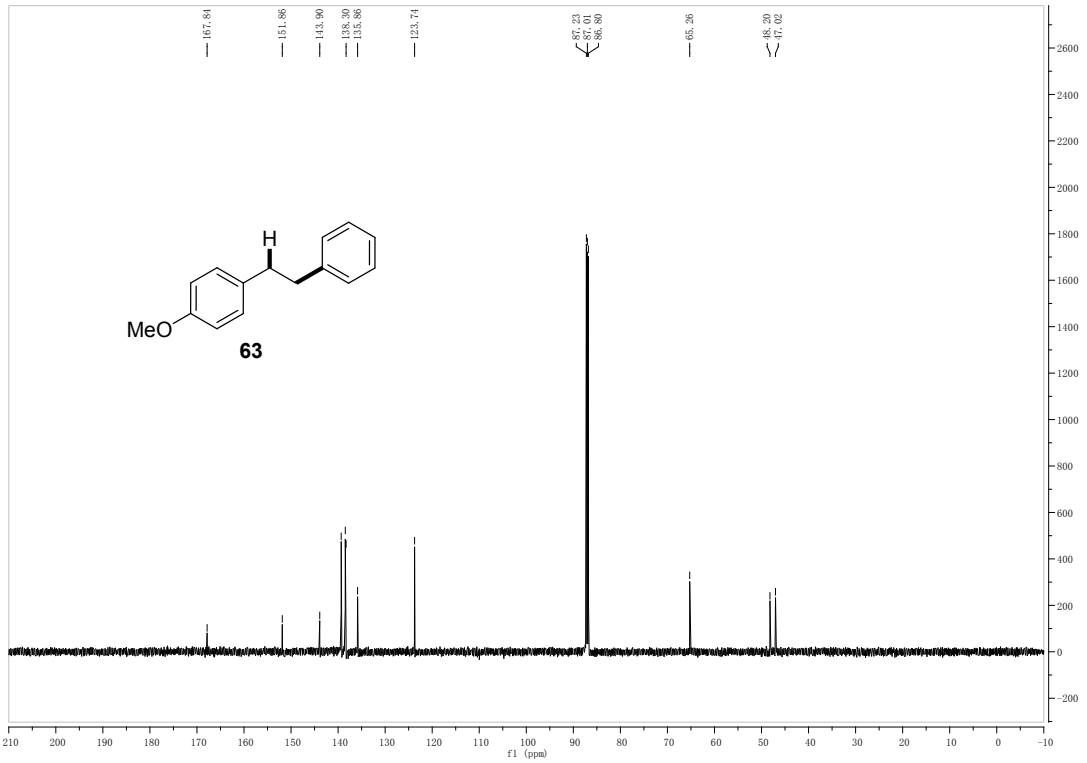

7.34
7.33
7.32
7.30
7.29
7.28
7.26
7.25
7.22
7.21
6.92
6.91
4.89
4.88
4.87
3.84
3.04
3.03
3.02
OH
MeO
61
7.39
2.07
1.00
3.03
2.09
f1 (ppm)

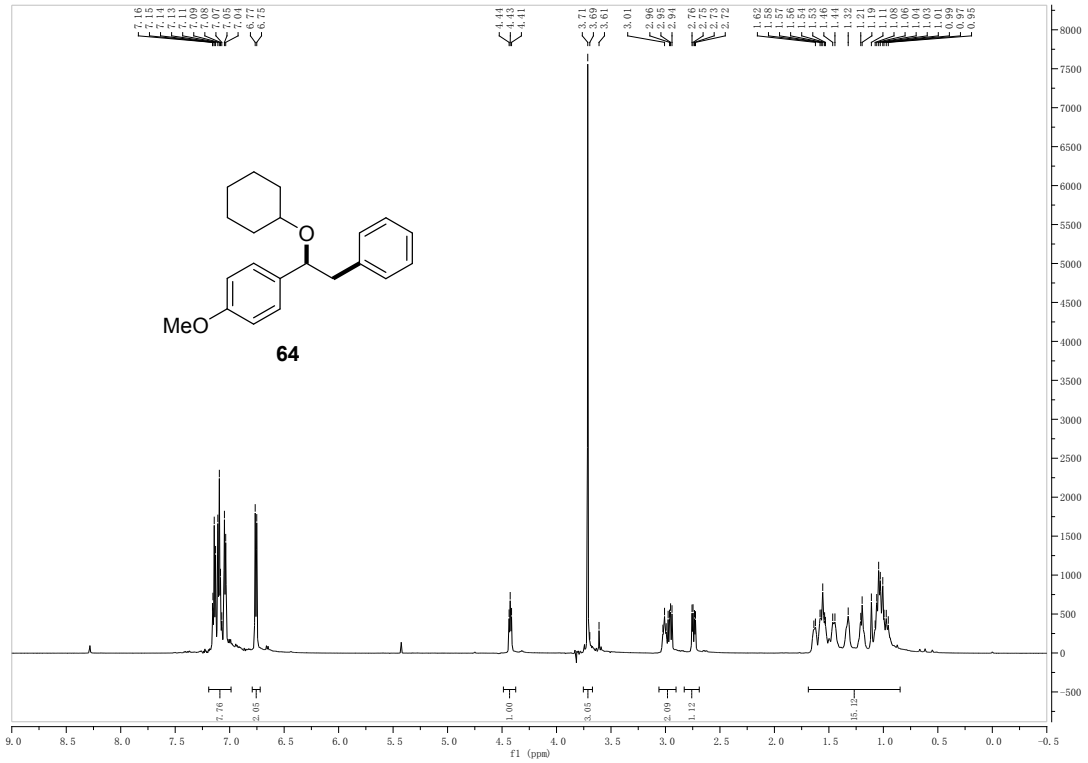

159.10
138.19
136.03
129.53
128.59
127.18
126.57
113.80
77.26
77.05
76.84
75.00
55.31
46.04
OH
MeO
61
f1 (ppm)

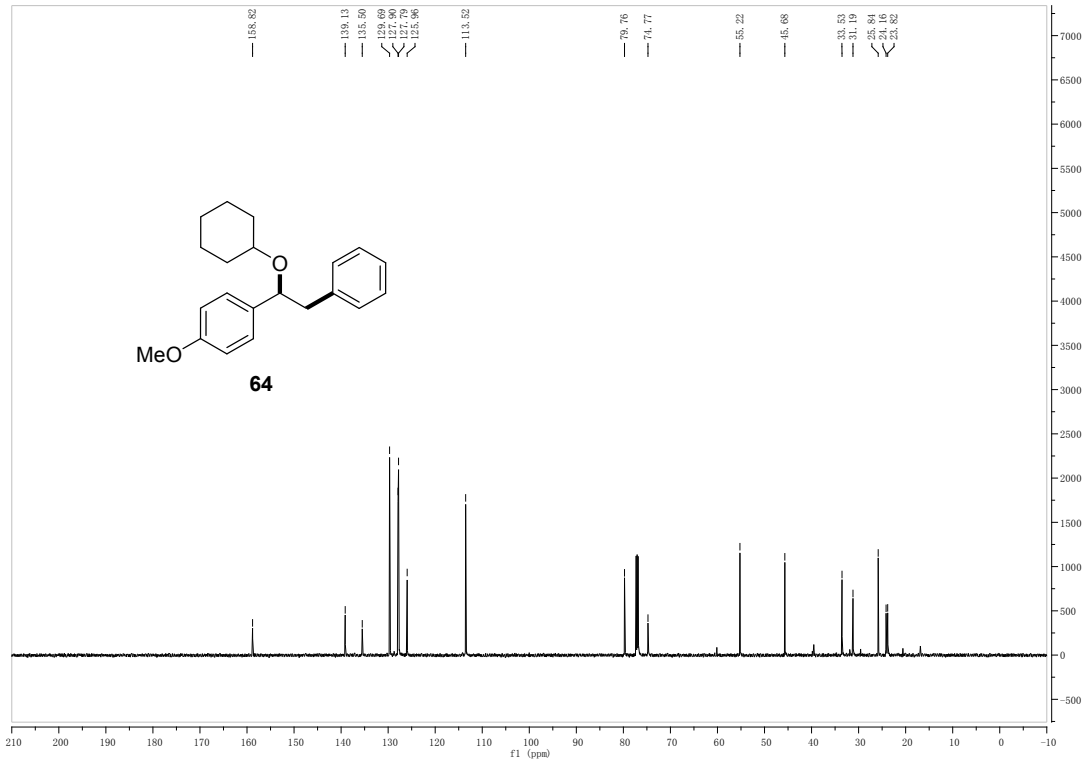

7.93
7.91
7.24
7.20
7.19
7.18
7.16
7.15
6.86
6.84
4.16
3.78
O
MeO
62
1.92
5.19
2.00
1.96
2.96
f1 (ppm)

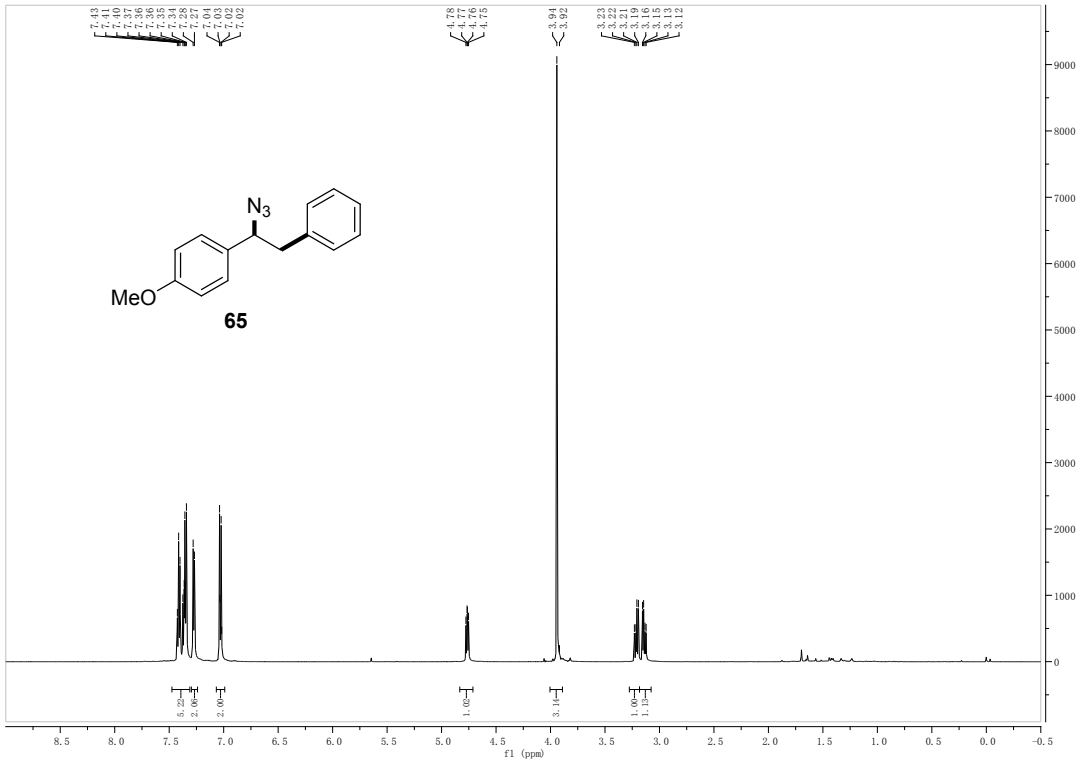

196.24
163.54
135.00
130.97
129.67
129.40
128.66
126.79
113.81
77.26
77.05
76.84
55.48
45.29
O
MeO
62
f1 (ppm)

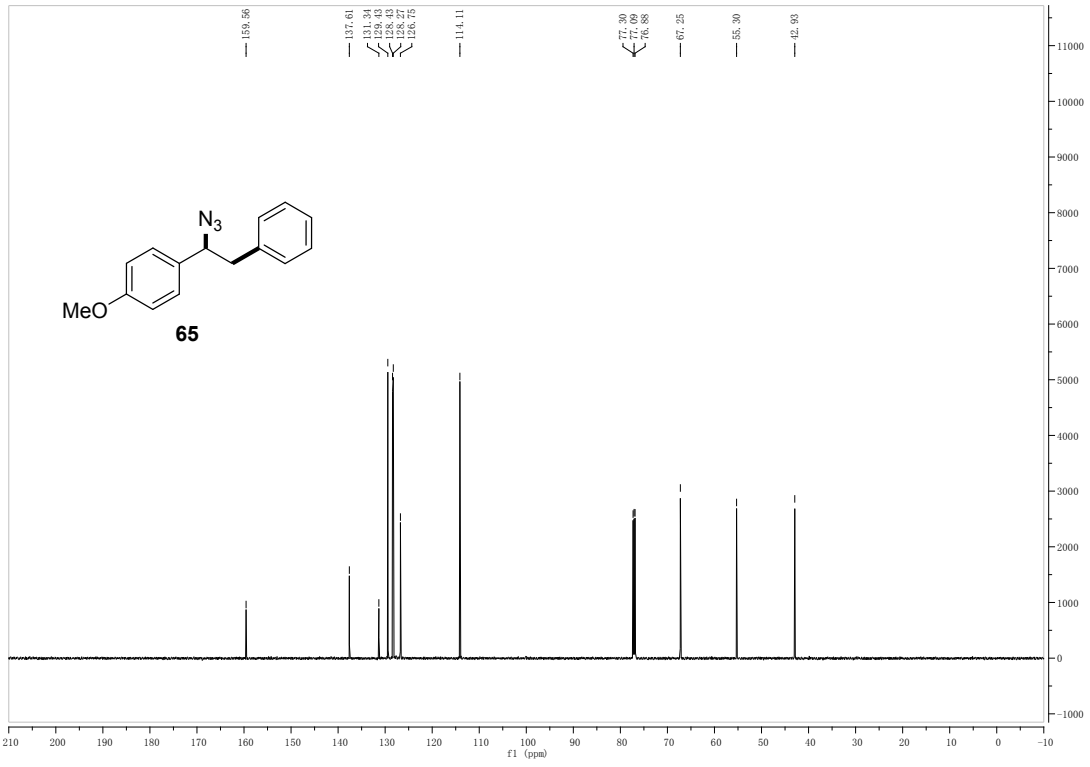

7.32
7.31
7.30
7.29
7.24
7.22
7.21
7.20
7.13
7.12
6.86
6.85
3.82
2.93
2.92
2.91
2.90
2.89
2.89
2.88
2.88
H
MeO
63
2.23
3.12
2.09
2.00
2.78
4.20
f1 (ppm)

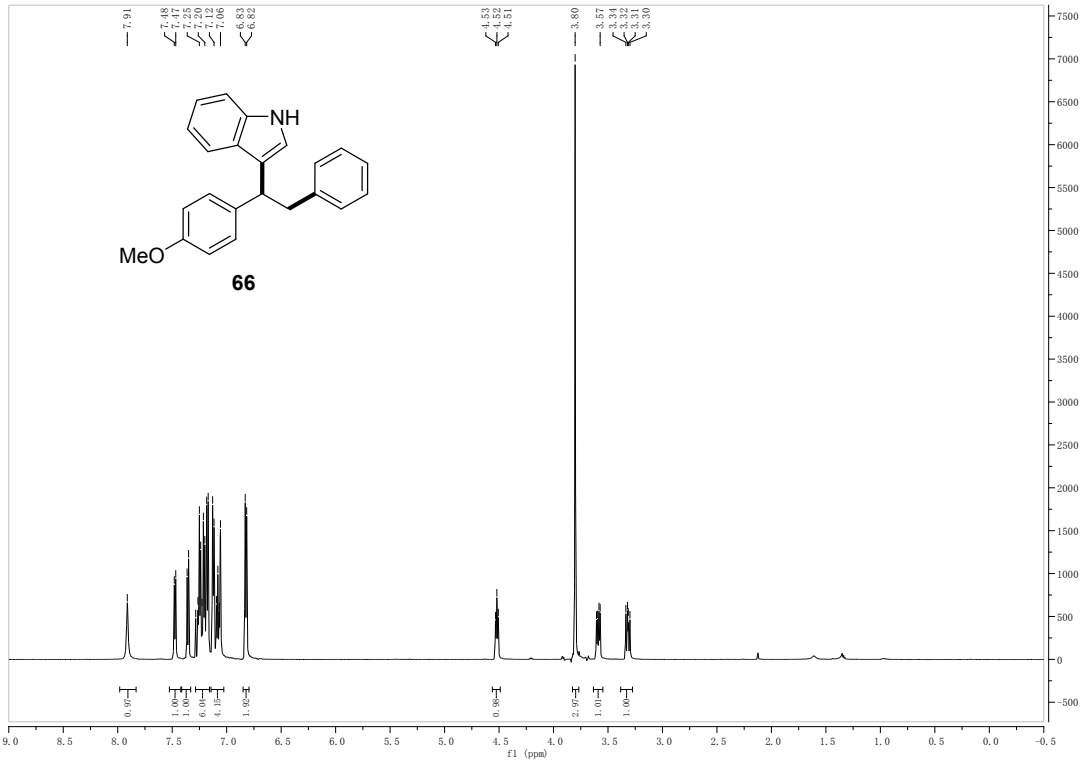

167.84
151.86
143.90
138.30
135.86
123.74
87.23
87.01
86.80
65.26
48.30
47.02
H
MeO
63
f1 (ppm)

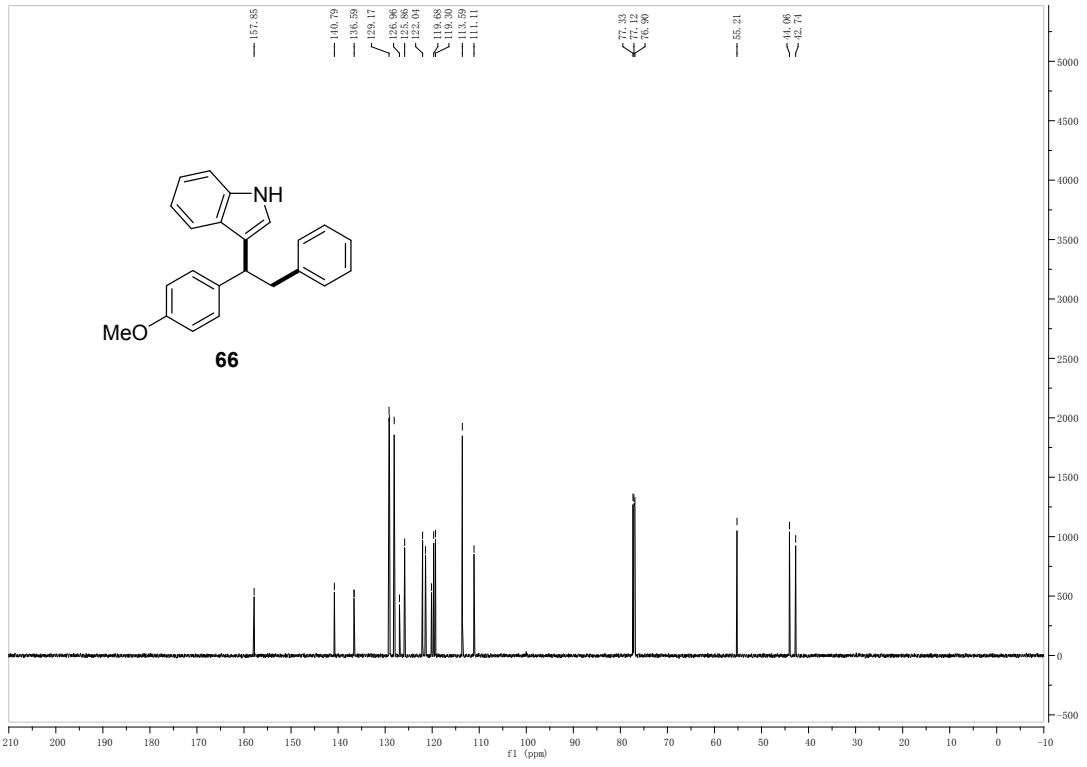

MeO
64
f1 (ppm)

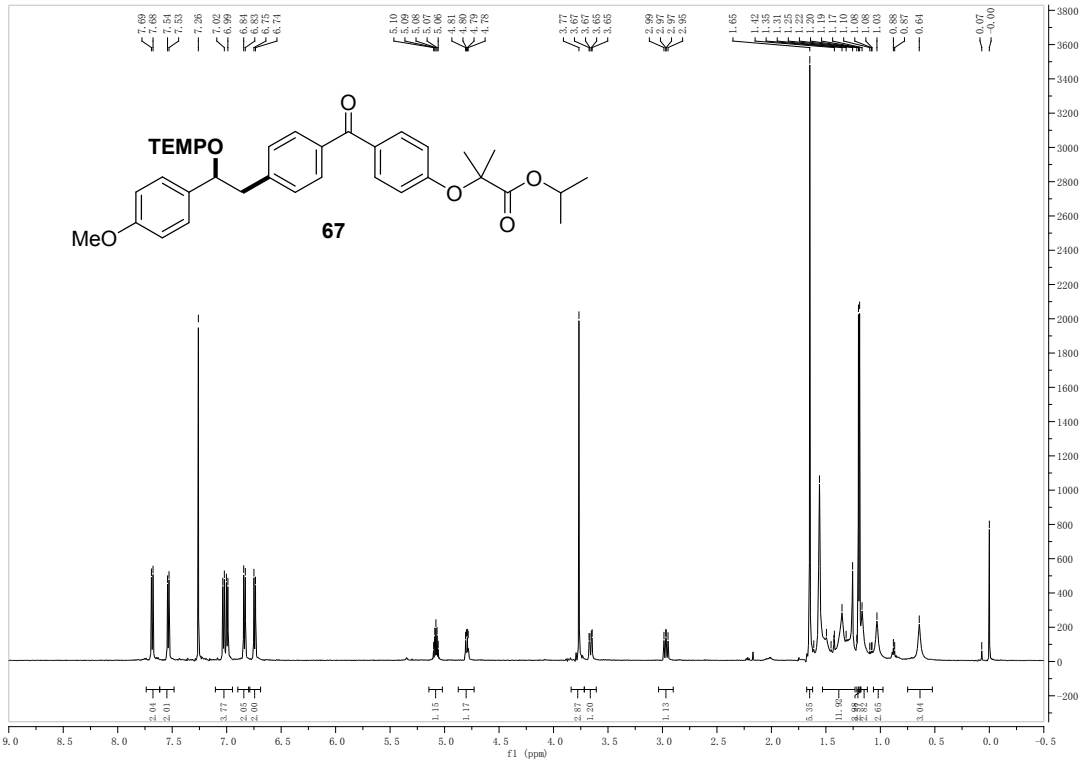

MeO
64
f1 (ppm)

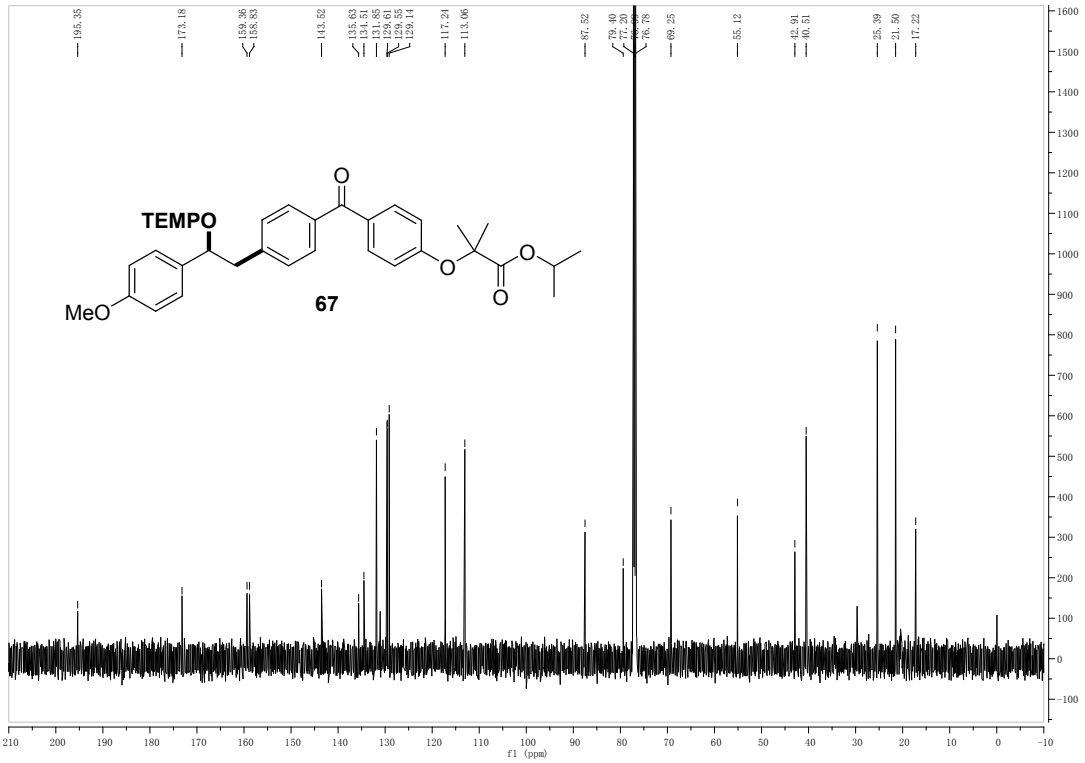

7.43
7.41
7.40
7.37
7.36
7.36
7.35
7.34
7.28
7.27
7.04
7.03
7.02
7.02
4.78
4.77
4.76
4.75
3.94
3.92
3.23
3.22
3.21
3.19
3.16
3.15
3.13
3.12
N3
MeO
65
5.22
2.06
2.00
1.02
3.14
1.04
1.13
f1 (ppm)

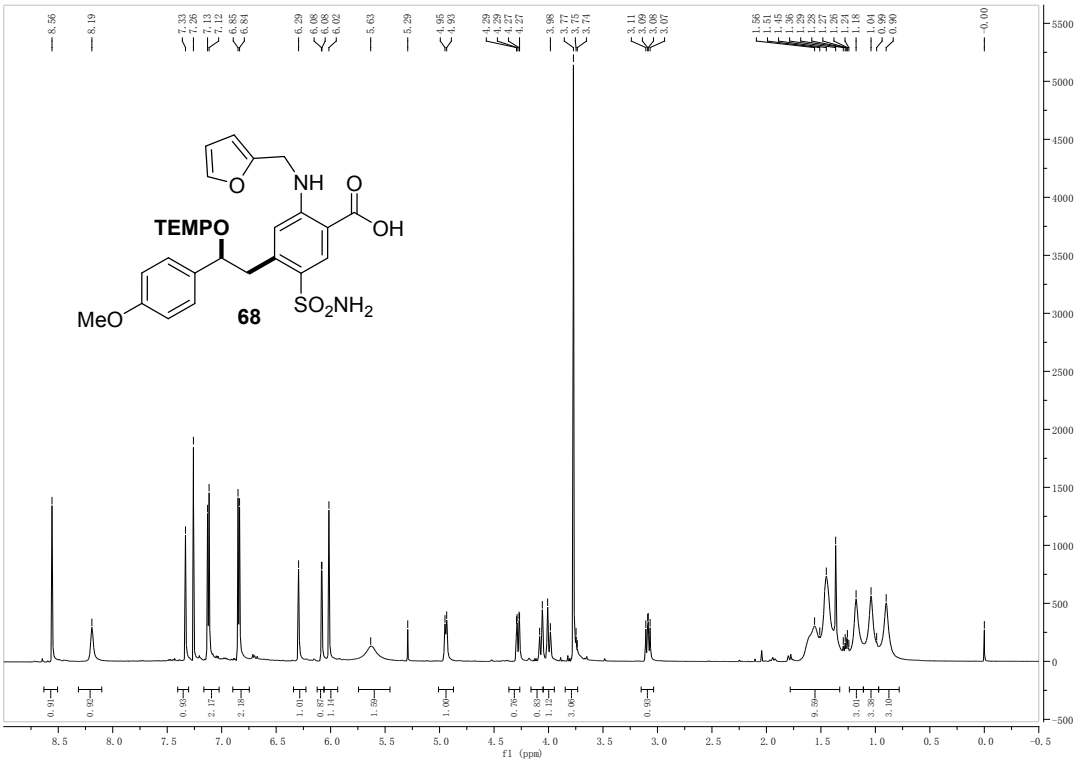

159.56
137.61
131.34
129.43
128.43
128.27
126.75
114.11
77.30
77.09
76.88
67.25
55.30
42.93
N3
MeO
65
f1 (ppm)

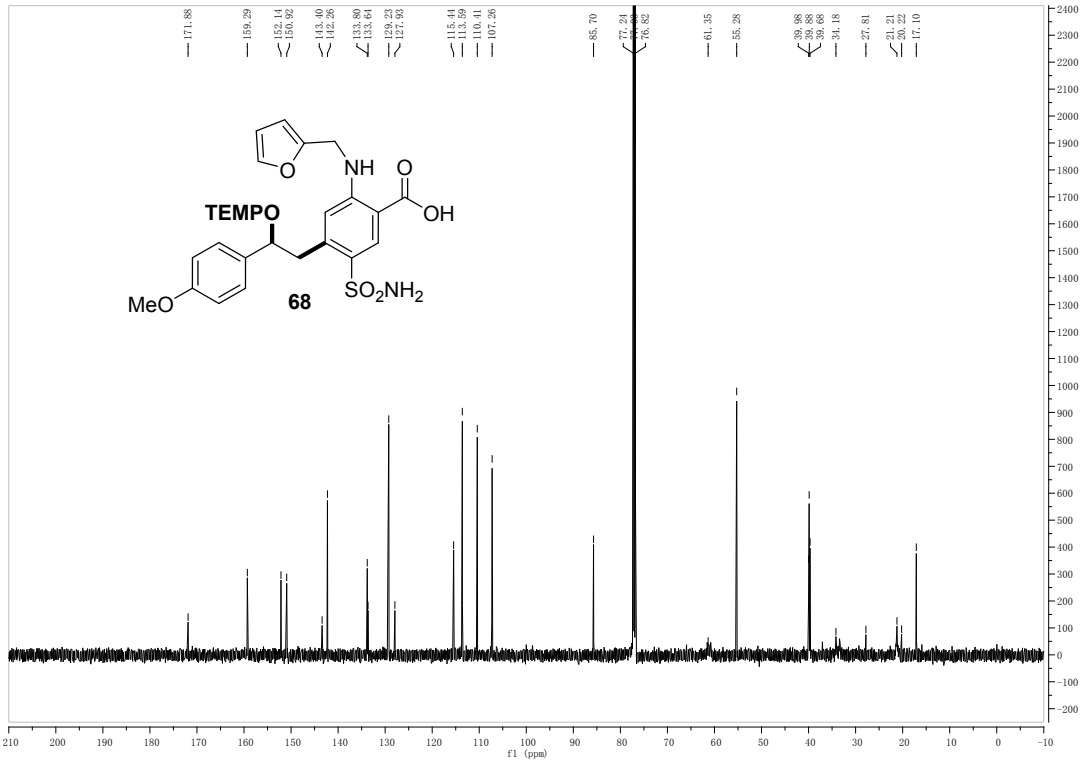

7.91
7.48
7.47
7.25
7.20
7.12
7.06
6.83
6.82
4.53
4.52
4.51
3.80
3.57
3.34
3.32
3.31
3.30
NH
MeO
66
f1 (ppm)

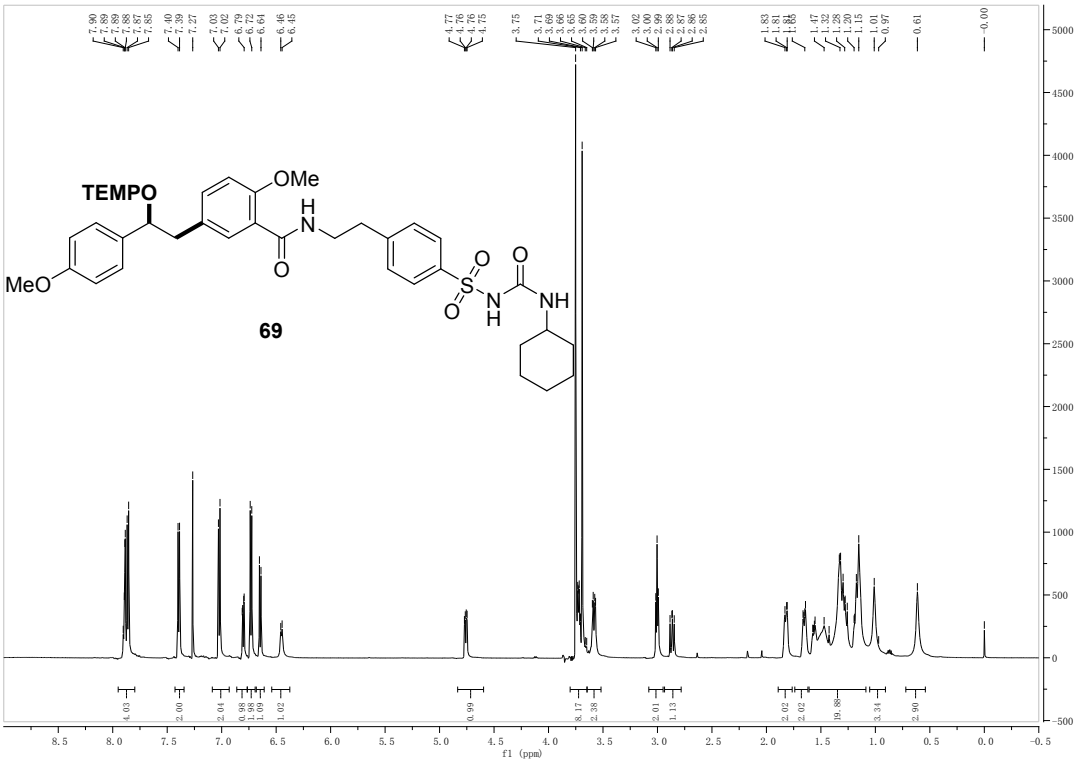

157.85
140.79
136.59
129.17
126.96
125.98
122.04
119.68
119.30
113.59
111.11
77.33
77.12
76.90
55.21
44.06
42.74
NH
MeO
66
f1 (ppm)

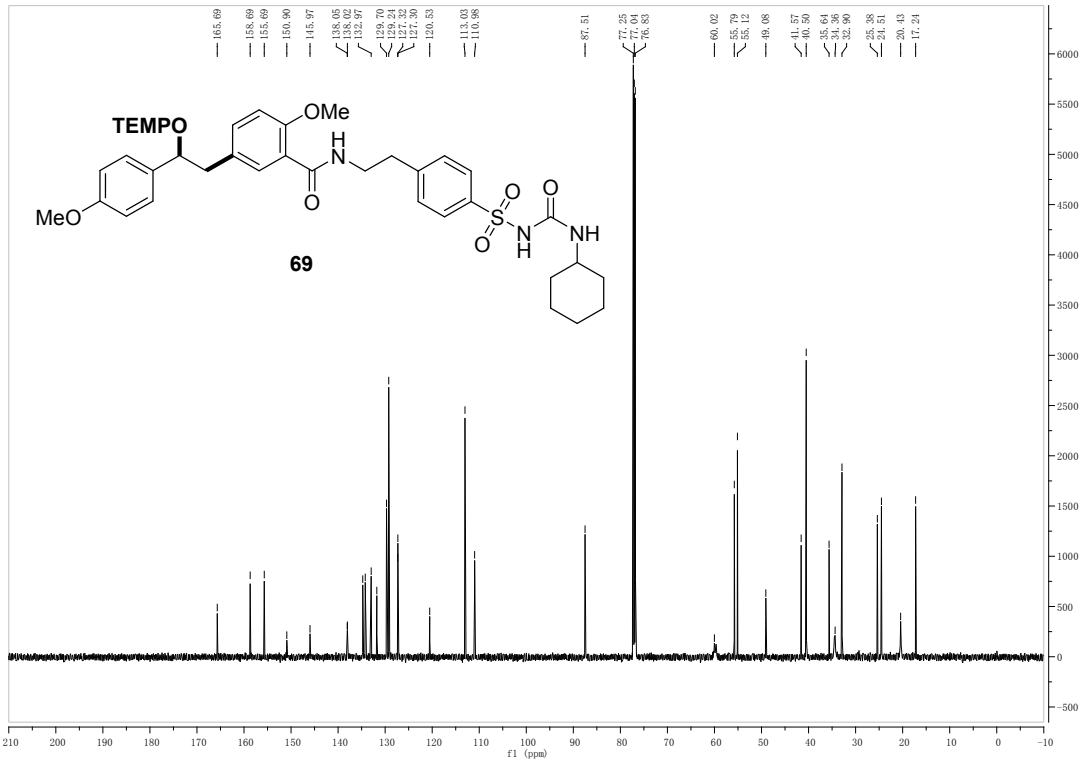

TEMPO
MeO
67

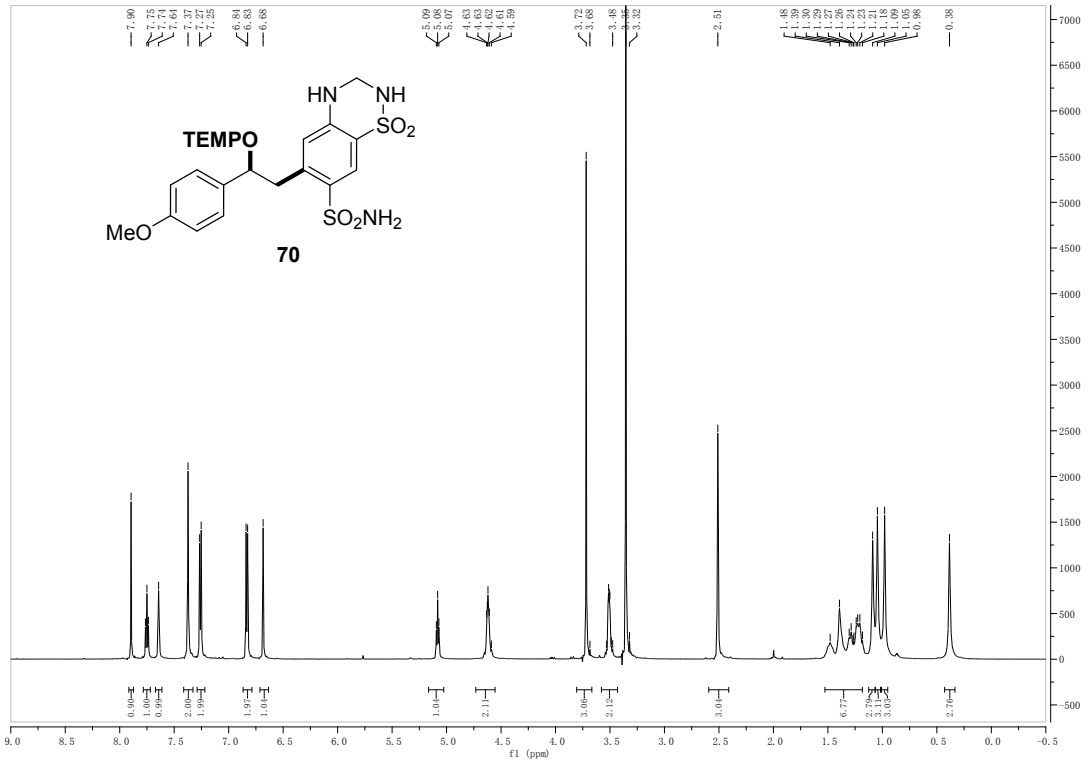

TEMPO
MeO
67

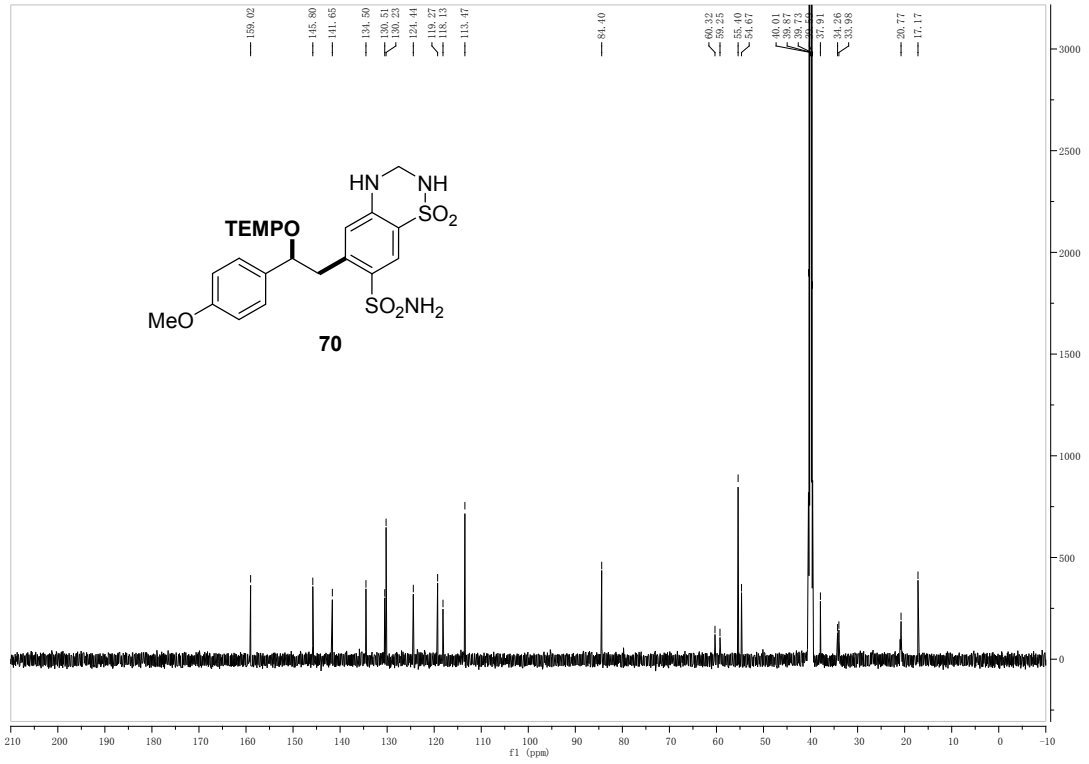

TEMPO
MeO
68
NH
O
OH
SO2NH2
f1 (ppm)

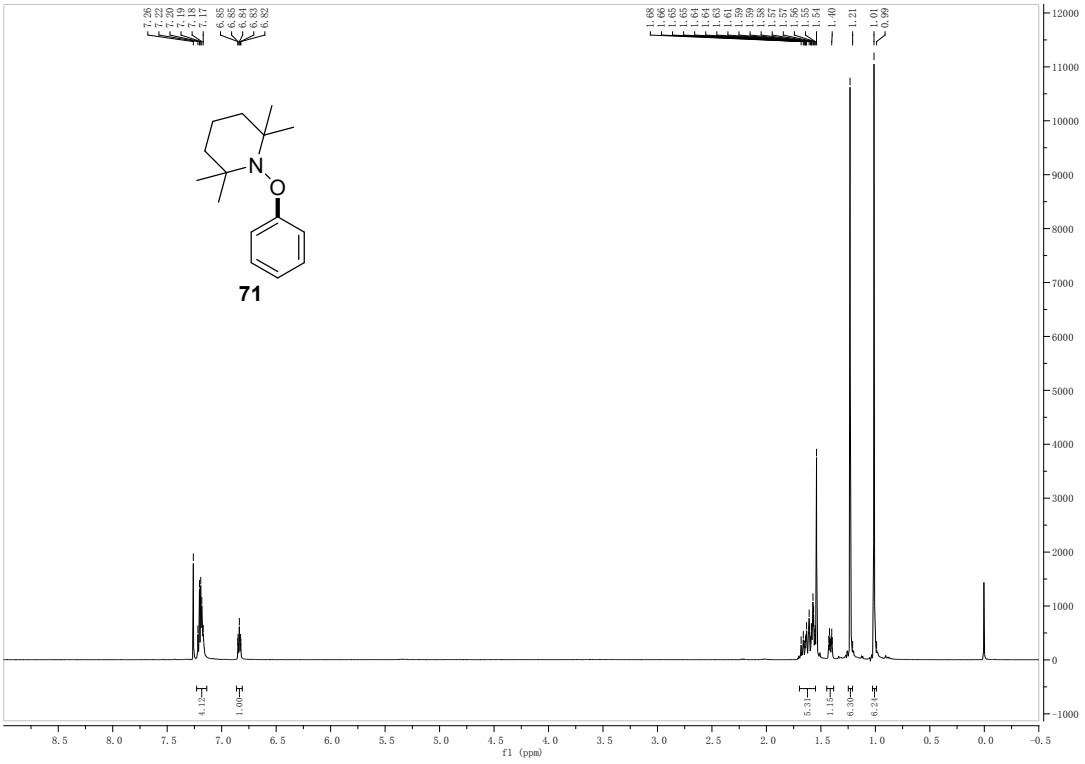

TEMPO
MeO
68
NH
O
OH
SO2NH2
f1 (ppm)

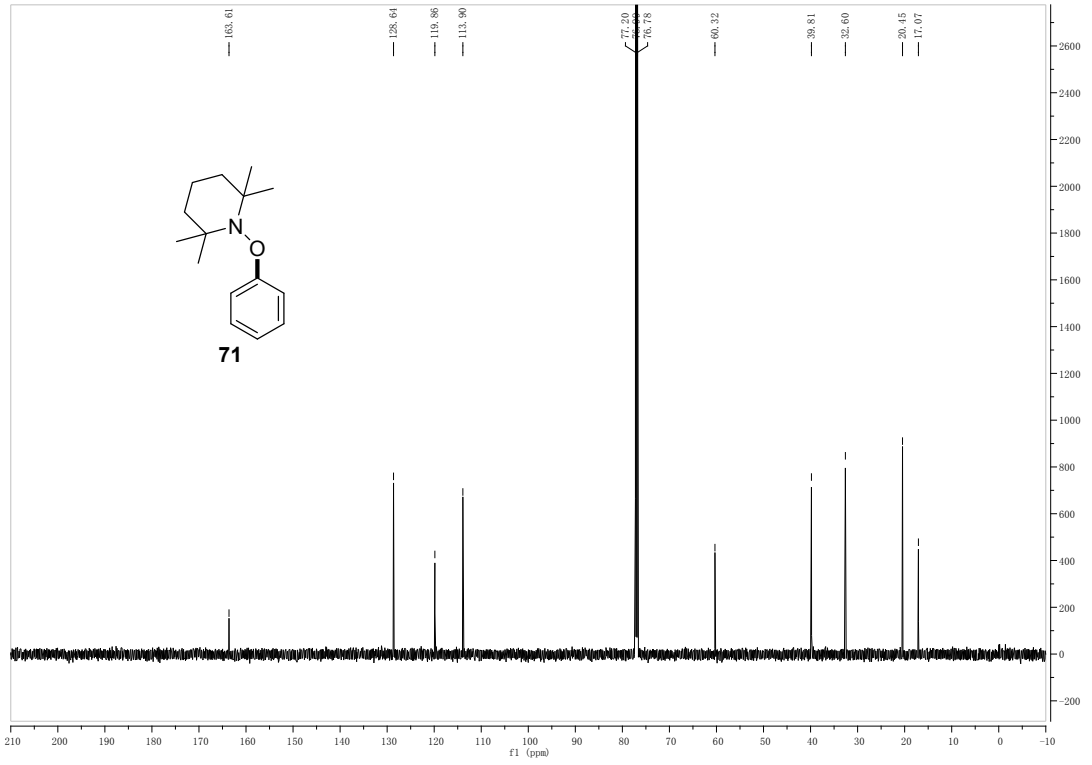

TEMPO
OMe
MeO
69

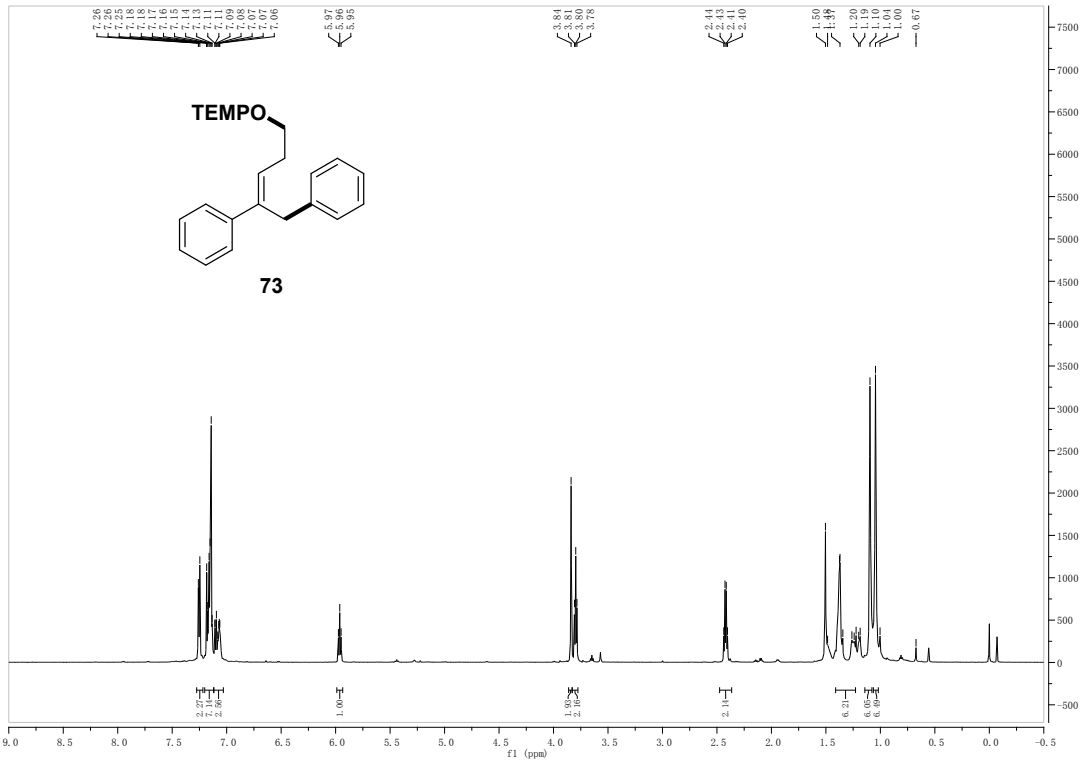

TEMPO
OMe
MeO
69

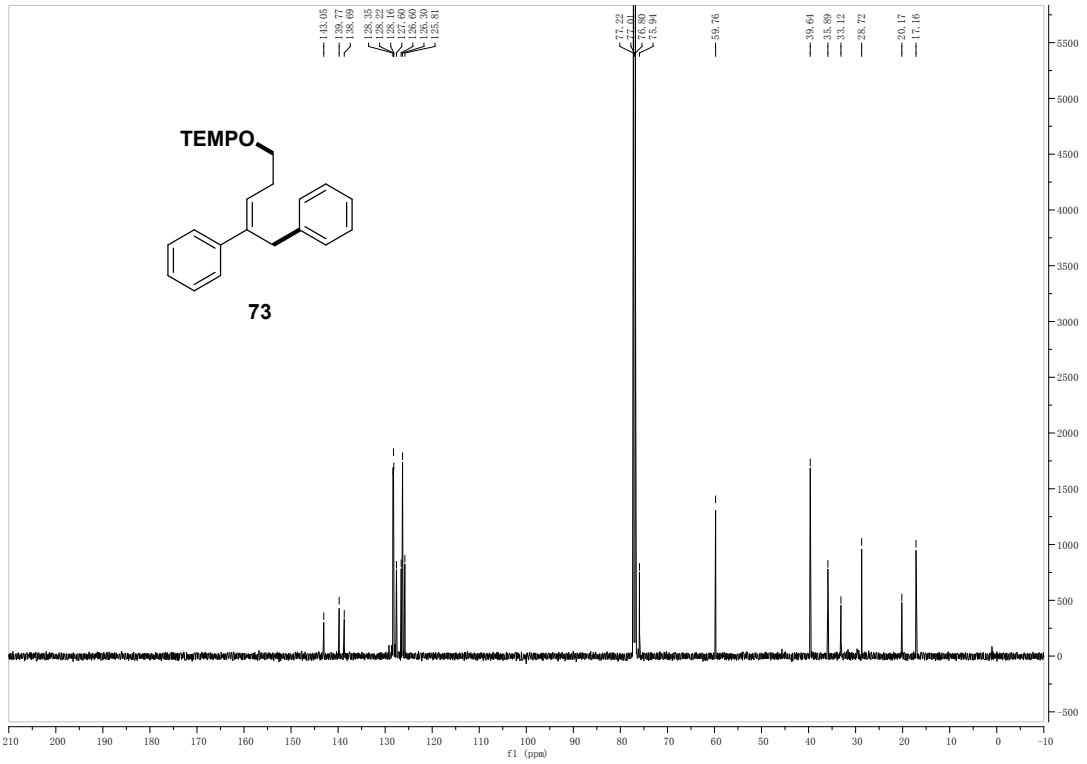

HN
NH
SO2
TEMPO
SO2NH2
MeO
70
f1 (ppm)

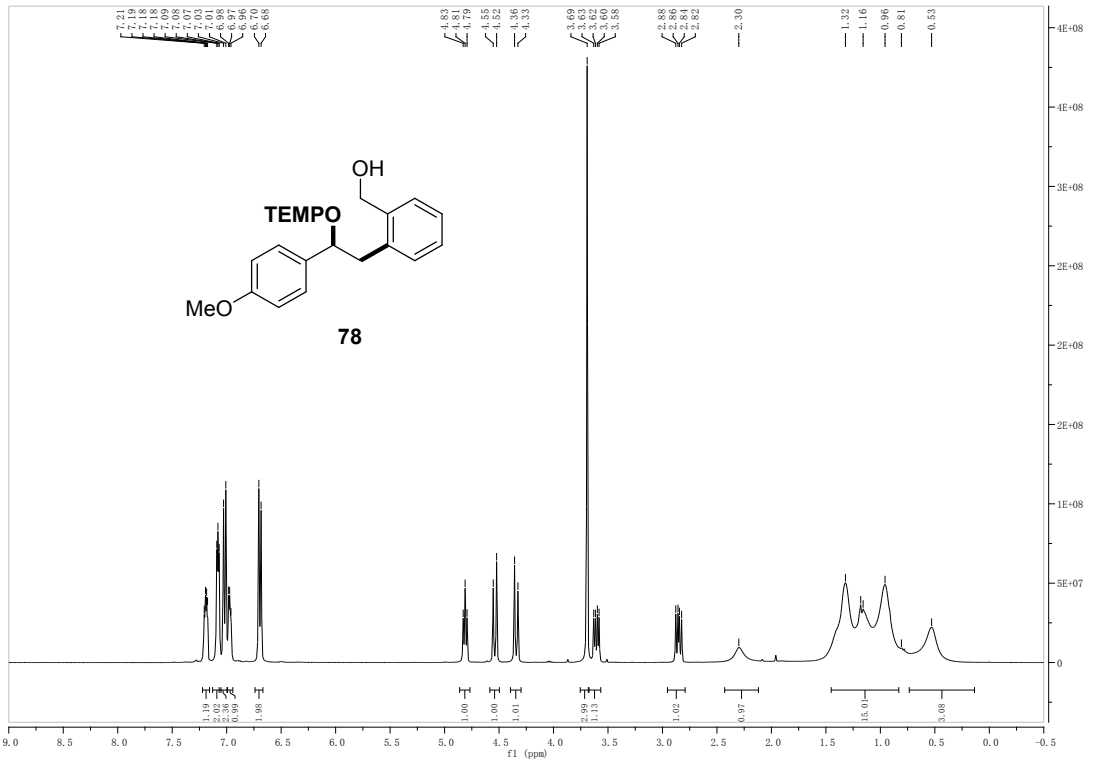

HN
NH
SO2
TEMPO
SO2NH2
MeO
70
f1 (ppm)

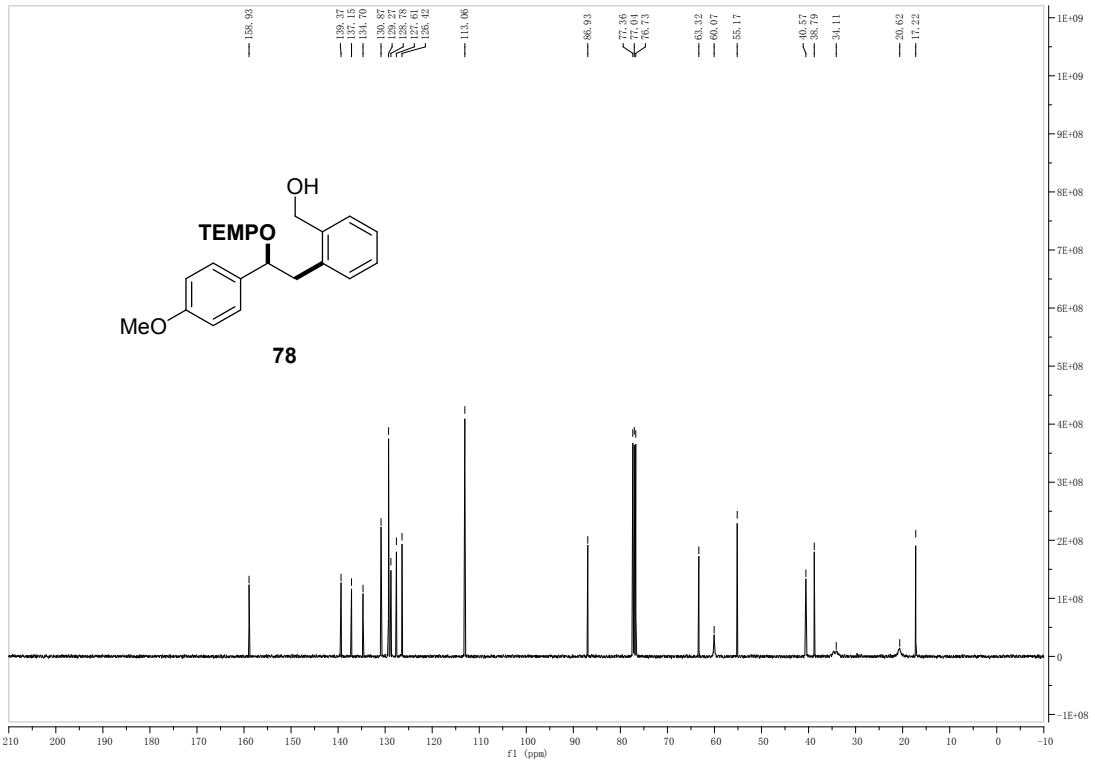

71

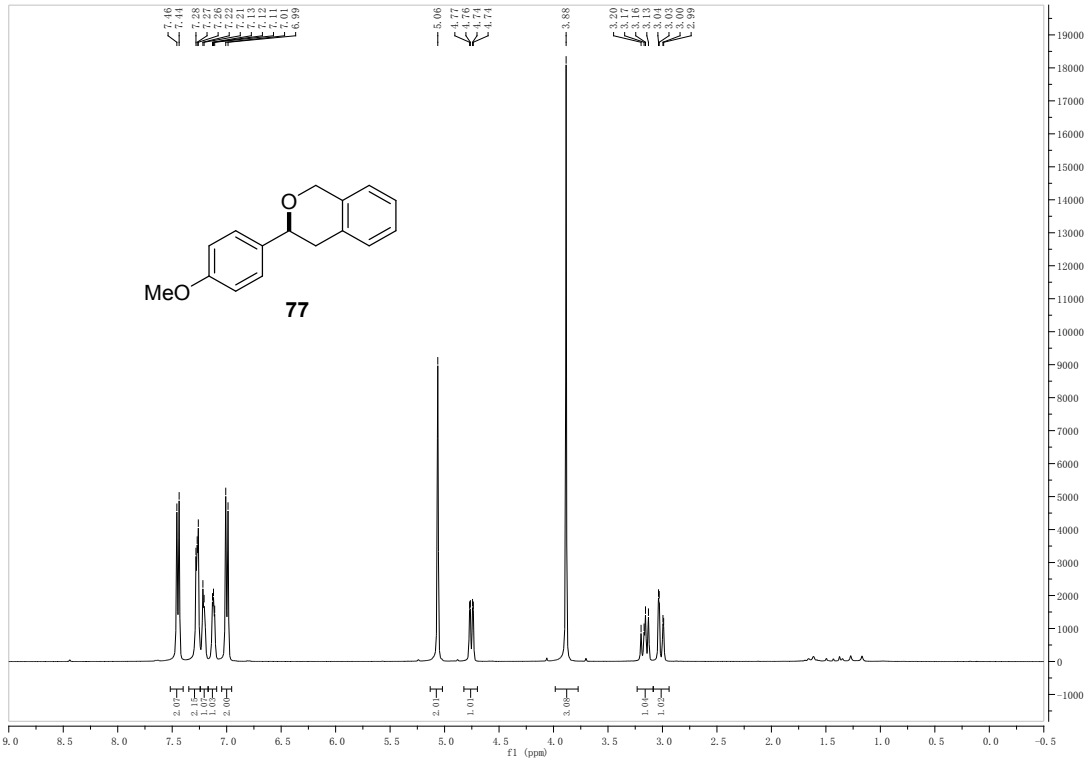

71

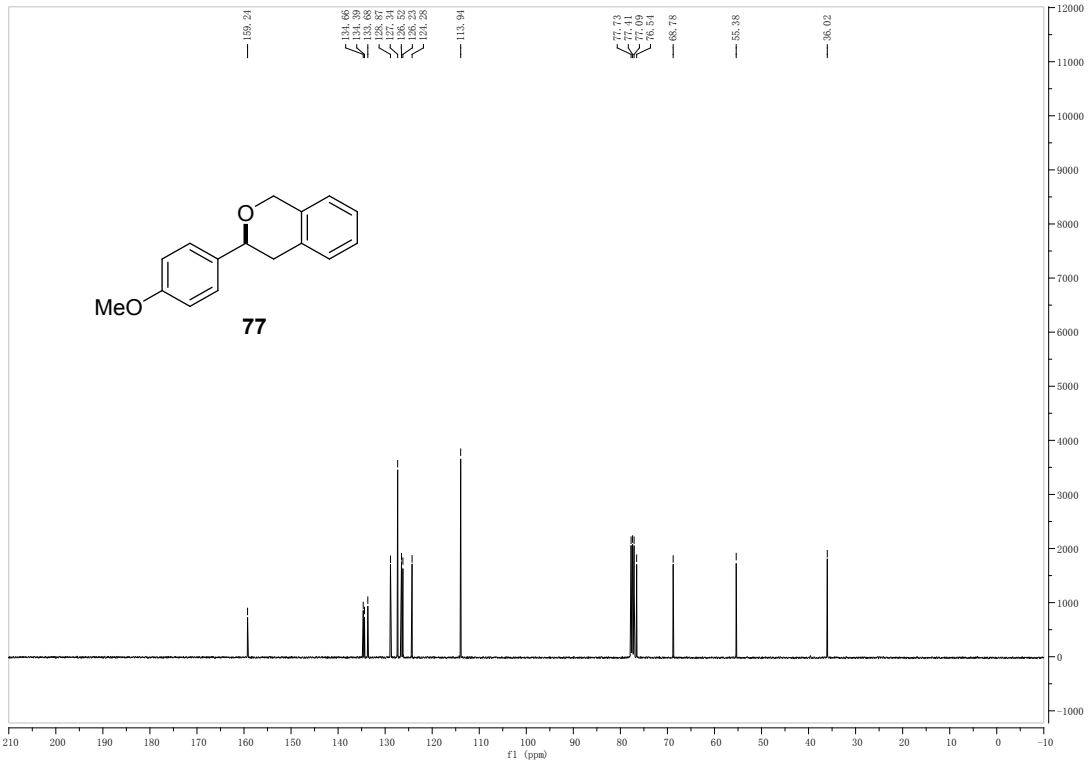

TEMPO
73
f1 (ppm)


TEMPO
73
f1 (ppm)


OH
TEMPO
MeO
78
f1 (ppm)


OH
TEMPO
MeO
78
f1 (ppm)


7.46
7.44
7.28
7.27
7.26
7.22
7.21
7.13
7.12
7.11
7.01
6.99
5.06
4.77
4.76
4.74
4.74
3.88
3.20
3.17
3.16
3.13
3.04
3.03
3.00
2.99
O
MeO
77
f1 (ppm)


159.24
134.66
134.39
133.68
128.87
127.34
126.52
126.23
124.28
113.94
77.73
77.41
77.09
76.54
68.78
55.38
36.02
O
MeO
77
f1 (ppm)