Supplementary Information

Palladium(II)-Catalyzed Asymmetric C–H Carbonylation to Diverse Isoquinoline Derivatives Bearing All-Carbon Quaternary Stereocenters

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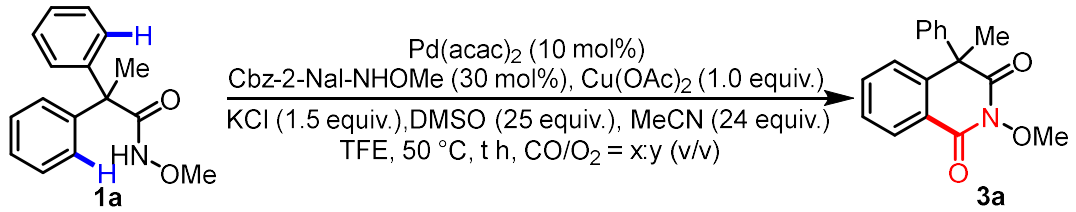
Contents

General Information

NMR spectra were recorded on Bruker-400 (400 MHz for ^{1}H; 101 MHz for ^{13}C) instruments internally referenced to $SiMe_4$ signal. High resolution mass spectra were recorded on P-SIMS-Gly of Bruker Daltonics Inc. using ESI-TOF (electrospray ionization-time of flight). Optical rotations were determined at 589 nm (sodium D line) by using an Anton-Paar MCP 200 polarimeter. HPLC analysis was performed on Shimadzu LC-20AT. Chiral column AD-H, OD-H, and ID were purchased from Daicel Chemical Industries, LTD. Trifluoroethanol was purchased from Qinba Chemie and used as received. Potassium chloride was purchased from Sinopharm and used as received. DMSO (superdry) and acetonitrile (superdry) were obtained from Adamas-Beta and used as received. Palladium acetoacetate was purchased from Strem and used as received. 2-Nal-OH was purchased from GL Biochem. And used as received.

Tables of the Optimization of Reaction Conditions

Table S1. Enantioselective C(sp^2)–H activation/CO insertion: Reaction time and Atmosphere Screening[a]

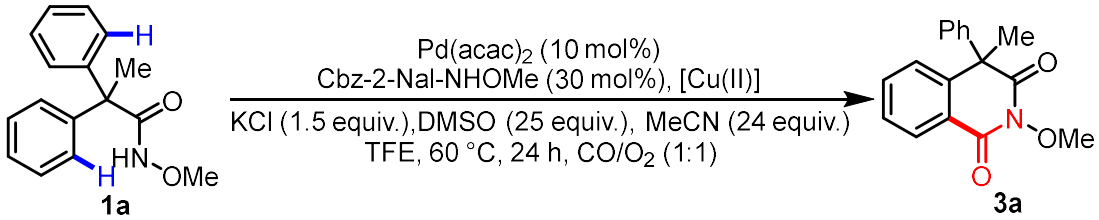


1a 3a

entry	x:y	t (h)	yield (%)[b]	ee (%)[c]
1	1:1	24	37	93
2	1:1	48	71	92
3	1:1	72	74	91
4	1:2	24	55	94
5	1:3	24	40	94
6[d]	1:1	24	77	92

[a] Reaction conditions: **1a** (0.15 mmol), $Pd(acac)_2$ (0.015 mmol, 10 mol%), Cbz-2-Nal-NHOMe (0.045 mmol, 30 mol%), $Cu(OAc)_2$ (0.15 mmol, 1.0 equiv.), KCl (0.225 mmol, 1.5 equiv.), DMSO (3.75 mmol, 25 equiv.), MeCN (3.6 mmol, 24 equiv.), TFE (1.2 mL), CO/O_2, 50 °C. [b] Isolated yield. [c] The ee value was determined by chiral HPLC analysis. [d] 60 °C.

Table S2. Enantioselective C(sp^2)–H activation/CO insertion: Copper salts Screening[a]



1a 3a

entry	[Cu(II)] (equiv.)	yield (%)[b]	ee (%)[c]
1	$Cu(OAc)_2/Cu(acac)_2$ (1.0/0.5)	82	93
2	$Cu(OAc)_2/Cu(acac)_2$ (0.5/1.0)	73	93
3	$Cu(acac)_2$ (1.0)	N.R.	-
4	$Cu(OAc)_2/Cu(hfacac)_2$ (1.0/0.5)	58	88
5	$Cu(OAc)_2/Cu(acac)_2$ (1.5/0.5)	46	93
6[d]	$Cu(OAc)_2/Cu(acac)_2$ (1.0/0.5)	88	91

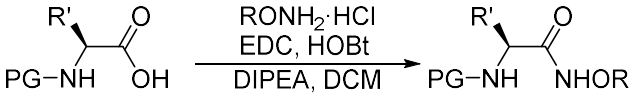
[a] Reaction conditions: **1a** (0.15 mmol), $Pd(acac)_2$ (0.015 mmol, 10 mol%), Cbz-2-Nal-NHOMe (0.045 mmol, 30 mol%), Cu(II) salts, KCl (0.225 mmol, 1.5 equiv.), DMSO (3.75 mmol, 25 equiv.), MeCN (3.6 mmol, 24 equiv.), TFE (1.2 mL), CO/O_2 (v/v = 1:1), 60 °C, 24 h. [b] Isolated yield. [c] The ee value was determined by chiral HPLC analysis. [d] 48 h.

Experimental Procedures

All of the mono-protected amino hydroxamic acid (MPAHA) ligands were prepared through the amidations of mono-protected amino acid (MPAA).[1] **L1**, **L3-7** and **L12** were prepared according to the reported procedures. **L2** was purchased from Adamas-Beta and used as received.

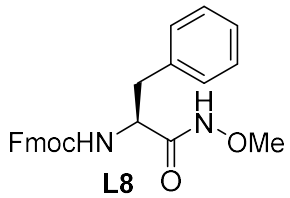
All of the substrates were prepared through amidations of diarylacetic acids.[1] The acid, which was used for preparation of **1a**, **1r** and **1s** was purchased from Fluorochem and used as received.

Preparation of Ligands (L8-11, L13-17)



To a DCM (40 mL) solution of MPAA (10 mmol), HOBt (1.49 g, 11 mmol, 1.1 equiv.) and EDCI (2.1 g, 11 mmol, 1.1 equiv.), *O*-alkyl hydroxylamine hydrochloride (15 mmol, 1.5 equiv.) and DIPEA (2.6 mL, 15 mmol, 1.5 equiv.) were added in an ice-bath. After stirring at room temperature overnight, the reaction mixture was quenched by water. The mixture was extracted by DCM for three times. The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography, and the ligand was obtained after recrystallization.

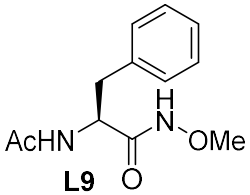
(9H-fluoren-9-yl)methyl (*S*)-(1-(methoxyamino)-1-oxo-3-phenylpropan-2-yl)carbamate (Fmoc-Phe-NHOMe, L8)



L8

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.31 (s, 1H), 7.88 (d, *J* = 7.5 Hz, 2H), 7.82 (d, *J* = 8.4 Hz, 1H), 7.66 (t, *J* = 7.4 Hz, 2H), 7.41 (td, *J* = 7.4, 3.3 Hz, 2H), 7.36 – 7.23 (m, 6H), 7.23 – 7.15 (m, 1H), 4.21 – 4.09 (m, 3H), 4.09 – 3.99 (m, 1H), 3.49 (s, 3H), 2.95 – 2.79 (m, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 167.8, 155.7, 143.7, 140.7, 137.6, 129.2, 128.1, 127.6, 127.1, 126.4, 125.4, 125.3, 120.1, 65.7, 63.1, 53.9, 46.5, 37.5. HRMS (ESI) calcd. for $C_{25}H_{25}O_4N_2$ ([M+H]$^+$): 417.1809, found: 417.1810. $[\alpha]_D^{20}$ = -21.2º (*c* 0.09, $CHCl_3$).

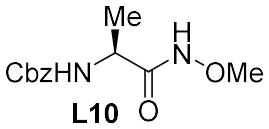
(*S*)-2-acetamido-*N*-methoxy-3-phenylpropanamide (Ac-Phe-NHOMe, L9)



L9

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 7.33 – 7.12 (m, 5H), 6.78 (d, *J* = 8.0 Hz, 1H), 4.62 (dd, *J* = 15.5, 7.9 Hz, 1H), 3.59 (s, 3H), 3.12 – 2.94 (m, 2H), 1.95 (s, 3H). ^{13}C NMR (101 MHz, Acetone-d_6) δ 170.1, 168.8, 138.4, 130.2, 129.1, 127.3, 63.8, 53.0, 38.7, 22.7. HRMS (ESI) calcd. for $C_{12}H_{17}O_3N_2$ ($[M+H]^+$): 237.1234, found: 237.1236. $[\alpha]_D^{20}$ = -2.4º (*c* 1.0, $CHCl_3$).

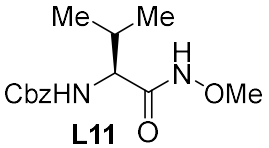
Benzyl (*S*)-(1-(methoxyamino)-1-oxopropan-2-yl)carbamate (Cbz-Ala-NHOMe, L10)



L10

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.99 (s, 1H), 7.40 – 7.29 (m, 5H), 5.21 (s, 1H), 5.16 – 5.05 (m, 2H), 4.17 – 4.05 (m, 1H), 3.75 (s, 3H), 1.39 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 156.3, 136.0, 128.7, 128.4, 128.1, 67.3, 64.4, 48.2, 18.2. HRMS (ESI) calcd. for $C_{12}H_{17}O_4N_2$ ($[M+H]^+$): 253.1183, found: 253.1184. $[\alpha]_D^{20}$ = -1.2º (*c* 1.0, $CHCl_3$).

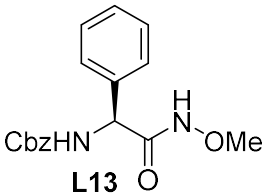
Benzyl (*S*)-(1-(methoxyamino)-3-methyl-1-oxobutan-2-yl)carbamate (Cbz-Val-NHOMe, L11)



L11

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (s, 1H), 7.44 – 7.29 (m, 5H), 5.24 (br, 1H), 5.11 (s, 2H), 3.87 – 3.61 (m, 1H), 3.77 (s, 3H), 2.19 – 2.06 (m, 1H), 1.10 – 0.82 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 156.8, 136.0, 128.7, 128.4, 128.1, 67.3, 64.4, 58.3, 31.0, 19.2, 18.5. HRMS (ESI) calcd. for $C_{14}H_{21}O_4N_2$ ($[M+H]^+$): 281.1496, found: 281.1498. $[\alpha]_D^{20}$ = -26.7º (*c* 1.0, $CHCl_3$).

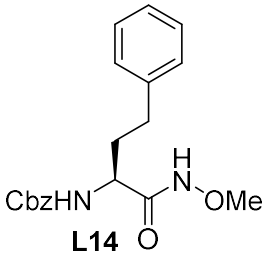
Benzyl (*S*)-(2-(methoxyamino)-2-oxo-1-phenylethyl)carbamate (Cbz-Phg-NHOMe, L13)



L13

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.35 (dd, *J* = 8.1, 5.2 Hz, 10H), 6.01 (s, 1H), 5.12 (d, *J* = 12.3 Hz, 1H), 5.06 (d, *J* = 12.2 Hz, 1H), 3.72 (s, 3H), 3.43 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.8, 156.0, 137.1, 136.0, 129.2, 128.8, 128.7, 128.4, 128.2, 127.2, 67.4, 64.6, 56.3. HRMS (ESI) calcd. for $C_{17}H_{19}O_4N_2$ ($[M+H]^+$): 315.1339, found: 315.1340. $[\alpha]_D^{20}$ = +22.8º (*c* 0.5, $CHCl_3$).

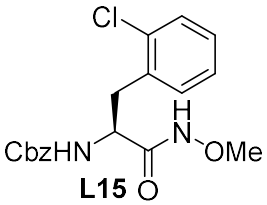
Benzyl (*S*)-(1-(methoxyamino)-1-oxo-4-phenylbutan-2-yl)carbamate (Cbz-HPh-NHOMe, L14)



L14

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (s, 1H), 7.41 – 7.31 (m, 5H), 7.30 – 7.26 (m, 2H), 7.23 – 7.19 (m, 1H), 7.19 – 7.09 (m, 2H), 5.21 (s, 1H), 5.13 (d, *J* = 12.3 Hz, 1H), 5.08 (d, *J* = 12.0 Hz, 1H), 3.97 (dd, *J* = 14.6, 7.0 Hz, 1H), 3.74 (s, 3H), 2.68 (t, *J* = 7.5 Hz, 2H), 2.22 – 2.08 (m, 1H), 2.04 – 1.89 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 156.5, 140.6, 136.0, 128.7, 128.6, 128.5, 128.4, 128.1, 126.3, 67.4, 64.4, 52.1, 33.7, 31.8. HRMS (ESI) calcd. for $C_{19}H_{23}O_4N_2$ ($[M+H]^+$): 343.1652, found: 343.1653. $[\alpha]_D^{20}$ = -24.4º (*c* 1.0, $CHCl_3$).

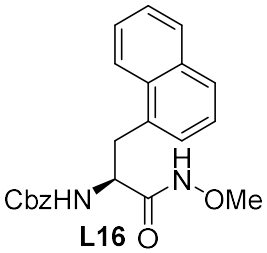
Benzyl (*S*)-(3-(2-chlorophenyl)-1-(methoxyamino)-1-oxopropan-2-yl)carbamate (Cbz-Phe(*o*-Cl)-NHOMe, L15)



L15

^{1}H NMR (400 MHz, Acetone-d_6) δ 10.44 (s, 1H), 7.57 – 7.17 (m, 9H), 6.69 (d, *J* = 8.1 Hz, 1H), 5.00 (dd, *J* = 18.9, 12.6 Hz, 2H), 4.40 (dd, *J* = 15.2, 8.6 Hz, 1H), 3.59 (s, 3H), 3.28 (dd, *J* = 13.7, 6.3 Hz, 1H), 3.08 (dd, *J* = 13.6, 8.8 Hz, 1H). ^{13}C NMR (101 MHz, Acetone-d_6) δ 168.5, 156.7, 138.1, 135.9, 134.9, 132.9, 130.2, 129.4, 129.2, 128.6, 128.5, 127.8, 66.7, 63.9, 53.2, 36.6. HRMS (ESI) calcd. for $C_{18}H_{20}ClN_2O_4$ ($[M+H]^+$): 363.1106, found: 363.1111. $[\alpha]_D^{20}$ = -14.1º (*c* 1.0, $CHCl_3$).

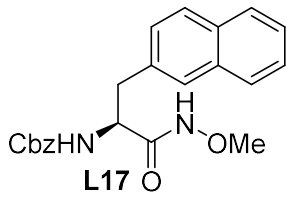
Benzyl (*S*)-(1-(methoxyamino)-3-(naphthalen-1-yl)-1-oxopropan-2-yl)carbamate (Cbz-1-Nal-NHOMe, L16)



L16

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (s, 1H), 8.05 (d, *J* = 6.4 Hz, 1H), 7.89 – 7.78 (m, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.53 – 7.38 (m, 2H), 7.38 – 7.13 (m, 7H), 5.68 (d, *J* = 7.2 Hz, 1H), 4.97 (s, 2H), 4.55 – 4.34 (m, 1H), 3.62 – 3.47 (m, 1H), 3.47 – 3.29 (m, 1H), 3.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.3, 156.1, 136.0, 134.0, 132.1, 131.8, 129.0, 128.7, 128.4, 128.3, 128.2, 128.1, 126.7, 126.0, 125.6, 123.6, 67.3, 64.3, 53.3, 36.2. HRMS (ESI) calcd. for $C_{22}H_{23}N_2O_4$ ($[M+H]^+$): 379.1652, found: 379.1657. $[\alpha]_D^{20}$ = -8.2º (*c* 0.5, MeOH).

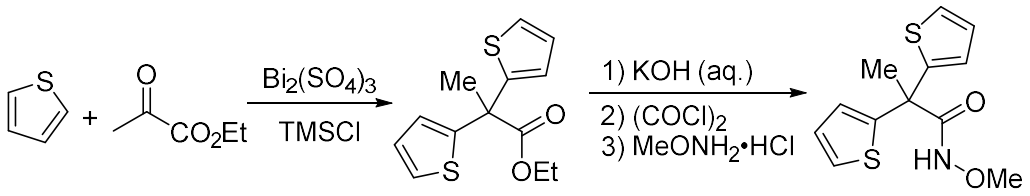
Benzyl (*S*)-(1-(methoxyamino)-3-(naphthalen-2-yl)-1-oxopropan-2-yl)carbamate (Cbz-2-Nal-NHOMe, L17)



L17

^{1}H NMR (400 MHz, Acetone-d_6) δ 10.39 (s, 1H), 7.98 – 7.66 (m, 4H), 7.59 – 7.36 (m, 3H), 7.26 (s, 5H), 6.69 (d, *J* = 8.3 Hz, 1H), 4.99 (q, *J* = 12.6 Hz, 2H), 4.39 (dd, *J* = 15.0, 8.3 Hz, 1H), 3.57 (s, 3H), 3.29 (dd, *J* = 13.6, 6.3 Hz, 1H), 3.13 (dd, *J* = 13.6, 8.5 Hz, 1H). ^{13}C NMR (101 MHz, Acetone-d_6) δ 168.8, 156.7, 138.1, 136.0, 134.5, 133.4, 129.1, 128.9, 128.7, 128.6 (2C), 128.5, 128.4, 126.8, 126.4, 66.7, 63.9, 55.2, 39.1. HRMS (ESI) calcd. for $C_{22}H_{23}N_2O_4$ ([M+H]$^+$): 379.1652, found: 379.1656. $[\alpha]_D^{20}$ = -10.0º (*c* 1.0, $CHCl_3$).

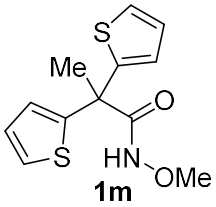
Preparation of 1m



To a mixture of TMSCl (13.0 mL, 100 mmol, 2.0 equiv.), dihydrobenzofuran (16.9 mL, 150 mmol, 3.0 equiv.), ethyl pyruvate (6.1 mL, 50 mmol), $Bi_2(SO_4)_3$ (3.5 g, 5 mmol, 10 mol%) was added in portions. The mixture was stirred at room temperature overnight vigorously. Silica gel was added after completing the reaction. To evaporate the volatile, and the residue was purified by flash column chromatography.

The ester (20 mmol) was dissolved in MeOH (11.2 mL), and was added 11.2 mL 20% KOH (aq.). The resulting solution was refluxed for 24 h. After that, the mixture was cooled to 0 ºC, acidified using 4N HCl, and extracted with Et_2O. The organic phase was dried over anhydrous Na_2SO_4. After filtration and concentration, the acid, without further purification, was dissolved in dry DCM (40 mL). Then DMF (3000 μL, 4 mmol, 0.2 equiv.) and oxalyl dichloride (3.4 mL, 40 mmol, 2.0 equiv.) was added successively in an ice-bath. The reaction mixture was stirred at room temperature for 3 h, and then concentrated in vacuo. The residue was dissolved in toluene (30 mL), *O*-methyl hydroxylamine hydrochloride (1.84 g, 22 mmol, 1.1 equiv.), which was added to a solution of Na_2CO_3 (4.24 g, 40 mmol, 2.0 equiv.) in toluene/H_2O (100 mL, v/v = 1:1) slowly at 0 ˚C. The resulting mixture was stirred at room temperature overnight. The mixture was extracted with EtOAc for three times, and the combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel.

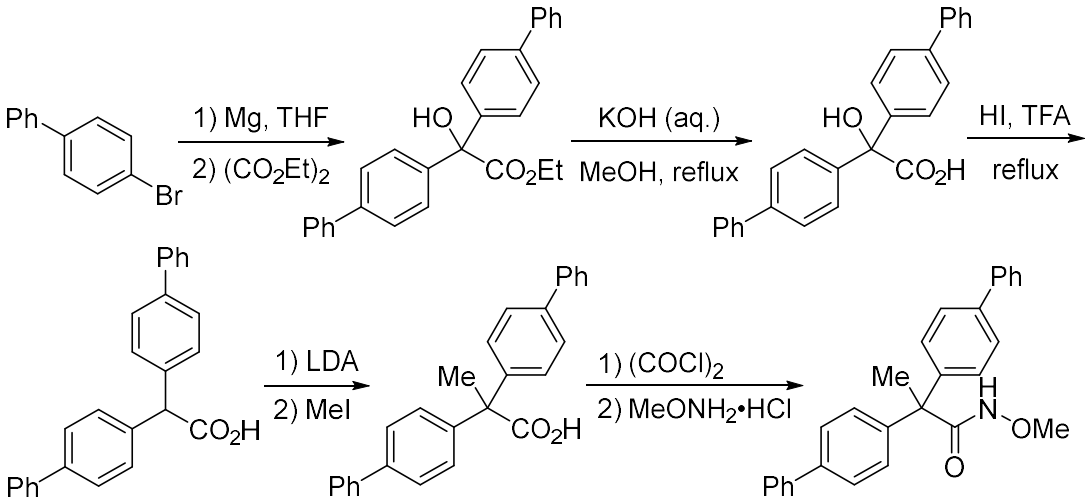
***N*-methoxy-2,2-di(thiophen-2-yl)propanamide (1m)**



1m

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (s, 1H), 7.33 – 7.21 (m, 2H), 6.98 (d, J = 2.7 Hz, 4H), 3.78 (s, 3H), 2.13 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 147.8, 126.9, 126.4, 125.7, 64.4, 50.7, 29.6. HRMS (ESI) calcd. for $C_{12}H_{14}NO_2S_2$ ($[M+H]^+$): 268.0460, found: 268.0434.

Preparation of 1f

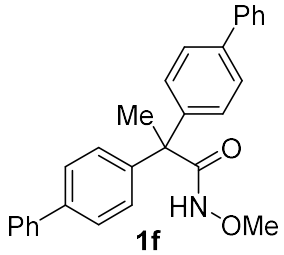


To a solution of diethyl oxalate (8.13 mL, 60 mmol) in dried THF (60 mL) was added 4-biphenylmagnesium bromide (132 mmol, 2.2 equiv., synthesized from 30.77 g 4-bromobiphenyl and 3.35 g magnesium in 90 mL dried THF) dropwise at -78 ºC, and then warmed to room temperature. After stirring overnight, the reaction was quenched with saturated NH_4Cl (aq.) at -10 ºC, and stirred for another 10 min. The mixture was extracted with DCM for three times, and the combined organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by flash col umn chromatography to give the ethyl benzilate.

The ester (23.69 g, 58 mmol) was dissolved in MeOH (58 mL), and the solution refluxed overnight after 2.5 M KOH (58 mL) was added. The reaction was cooled to 0 ºC and then acidified using 4N HCl. The mixture was extracted with Et_2O. The organic phase was dried over anhydrous Na_2SO_4. After filtration and concentration, the residue was dissolved in TFA (190 mL), and heated to reflux, then 57% HI (aq., 84 mL) was added dropwise. The solution was cooled to room temperature 5 h later, and most of the volatile was evaporated. The residue was diluted with water, added 30% NaOH (aq.) to adjust pH < 3 in an ice bath, extracted with EtOAc for three times. The combined organic phase was dried over anhydrous Na_2SO_4, filtered, concentrated. The crude acid was purified by flash column chromatography.

To a solution of di(4-biphenyl)acetic acid (3.64 g, 10 mmol) in dried THF (10 mL) was added LDA (2.0 M in THF, 11 mL, 22 mmol) under N_2 atmosphere in an ice-bath. The solution was stirred at room temperature for 4 h. After cooling the reaction mixture again to 0 ^oC, alkyl halide (15 mmol) was added dropwise. The reaction was stirred overnight at room temperature, quenched by 4N HCl at 0 ^oC and then diluted with Et_2O. The phases were separated and the aqueous phase was extracted with Et_2O (3 times). The organic layers were combined, washed with brine, dried over anhydrous Na_2SO_4. After filtration and concentration, the crude acid was purified by flash column chromatography. Then, the similar procedure according to **1m** was carried out to give **1f**.

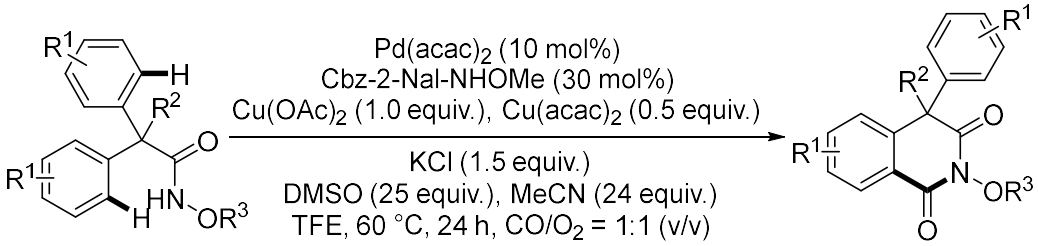
2,2-di([1,1'-biphenyl]-4-yl)-*N*-methoxypropanamide (1f)



1f

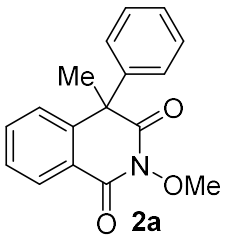
^1H NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.59 (t, J = 6.7 Hz, 8H), 7.45 (t, J = 7.5 Hz, 4H), 7.40 – 7.31 (m, 6H), 3.79 (s, 3H), 2.08 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.9, 142.9, 140.4, 140.3, 129.0, 128.6, 127.7, 127.5, 127.2, 64.5, 55.2, 27.1. HRMS (ESI) calcd. for $C_{28}H_{26}O_2N$ ($[M+H]^+$): 408.1958, found: 408.1962.

General Procedure for Pd(II)-Catalyzed Enantioselective C(sp^2)–H Carbonylation



In a 10 mL oven-dried two-necked bottle, 1.2 mL trifluoropanol, 267 μL DMSO and 150 μL MeCN were added to a mixture of diarylamide **1** (0.15 mmol), $Pd(acac)_2$ (4.6 mg, 0.015 mmol, 10 mol%), Cbz-2-Nal-NHOMe (17.0 mg, 0.045 mmol, 30 mol%), $Cu(OAc)_2$ (27.2 mg, 0.15 mmol, 1.0 equiv.), $Cu(acac)_2$ (19.6 mg, 0.075 mmol, 0.5 equiv.) and KCl (16.8 mg, 0.225 mmol, 1.5 equiv.) under CO/O_2 (v/v 1:1). The bottle was equipped with a 10 cm spherical condenser and the reaction mixture was stirred at 60 ^oC for 24 h. After cooling to room temperature, the mixture was diluted with EtOAc, washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/acetone = 7:1 to 5:1) to give the chiral imides as white to pale yellow solid.

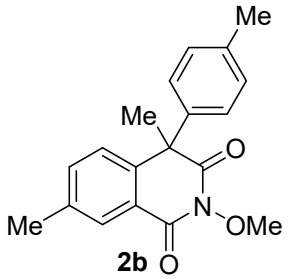
2-Methoxy-4-methyl-4-phenylisoquinoline-1,3(2*H*,4*H*)-dione (2a)



2a

82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.59 (td, *J* = 7.6, 1.4 Hz, 1H), 7.53 – 7.45 (m, 1H), 7.35 – 7.23 (m, 3H), 7.19 – 7.10 (m, 3H), 3.92 (s, 3H), 2.10 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.0, 160.8, 143.9, 142.5, 134.6, 128.9 (2C), 128.1, 128.0, 127.9, 127.2, 124.5, 63.9, 53.6, 26.9. HRMS (ESI) calcd. for $C_{17}H_{16}NO_3$ ($[M+H]^+$): 282.1125, found: 282.1131. $[\alpha]_D^{20}$ = -54.8º (*c* 0.5, $CHCl_3$). Enantiomeric excess: 93%, determined by HPLC (Chiralpak-AD-H, hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 12.278 min (minor), t_R = 16.320 min (major).

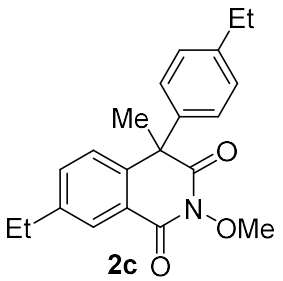
2-Methoxy-4,7-dimethyl-4-(*p*-tolyl)isoquinoline-1,3(2*H*,4*H*)-dione (2b)



2b

83% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.15 – 6.94 (m, 5H), 3.90 (s, 3H), 2.44 (s, 3H), 2.30 (s, 3H), 2.05 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 161.0, 141.1, 139.7, 138.0, 137.6, 135.5, 129.5, 128.8, 127.8, 126.9, 124.2, 63.8, 52.9, 26.8, 21.0 (2C). HRMS (ESI) calcd. for $C_{19}H_{20}NO_3$ ($[M+H]^+$): 310.1438, found: 310.1439. $[\alpha]_D^{20}$ = -48.4º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 91%, determined by HPLC (Chiralpak-AD-H, hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 14.477 min (minor), t_R = 20.040 min (major).

7-Ethyl-4-(4-ethylphenyl)-2-methoxy-4-methylisoquinoline-1,3(2*H*,4*H*)-dione (2c)

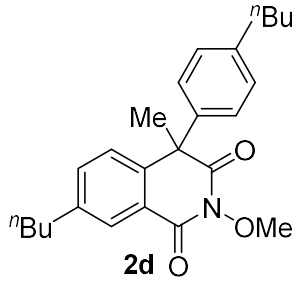


2c

80% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 1.7 Hz, 1H), 7.41 (dd, *J* = 8.1, 1.9 Hz, 1H), 7.12 (d, *J* = 8.3 Hz, 2H), 7.09 – 7.01 (m, 3H), 3.91 (s, 3H), 2.74 (q, *J* = 7.6 Hz, 2H), 2.60 (q, *J* = 7.6 Hz, 2H), 2.06 (s, 3H), 1.29 (t, *J* = 7.6 Hz, 3H), 1.19 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 161.1, 144.3, 143.8, 141.4, 139.9, 134.5, 128.3, 128.0, 127.7, 127.0, 124.3, 63.9, 53.0, 28.4 (2C), 26.8, 15.4 (2C). HRMS (ESI)

calcd. for $C_{21}H_{24}NO_3$ ([M+H]$^+$): 338.1751, found: 338.1757. $[\alpha]_D^{20}$ = -54.5º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 91%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 95/5, flow rate 0.5 mL/min, T = 25 ºC, 254 nm): t_R = 15.967 min (minor), t_R = 47.052 min (major).

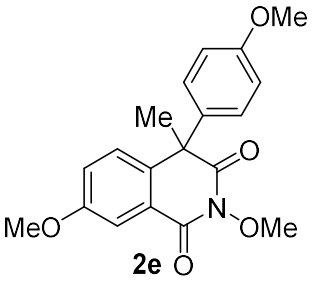
7-Butyl-4-(4-butylphenyl)-2-methoxy-4-methylisoquinoline-1,3(2*H*,4*H*)-dione (2d)



2d

82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.09 (d, *J* = 8.2 Hz, 2H), 7.06 (d, *J* = 8.2 Hz, 1H), 7.03 (d, *J* = 8.2 Hz, 2H), 3.90 (s, 3H), 2.77 – 2.63 (m, 2H), 2.63 – 2.49 (m, 2H), 2.06 (s, 3H), 1.73 – 1.59 (m, 3H), 1.59 – 1.48 (m, 2H), 1.45 – 1.23 (m, 5H), 0.95 (t, *J* = 7.4 Hz, 3H), 0.90 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 161.1, 143.0, 142.6, 141.3, 139.9, 134.9, 128.9, 128.2, 127.9, 127.0, 124.3, 63.9, 53.0, 35.2 (2C), 33.5, 33.4, 26.8, 22.5 (2C), 14.1. HRMS (ESI) calcd. for $C_{25}H_{32}NO_3$ ([M+H]$^+$): 394.2377, found: 394.2379. $[\alpha]_D^{20}$ = -45.1º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 90%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 5.533 min (minor), t_R = 9.958 min (major).

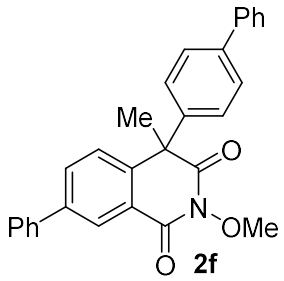
2,7-Dimethoxy-4-(4-methoxyphenyl)-4-methylisoquinoline-1,3(2*H*,4*H*)-dione (2e)



2e

68% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 2.8 Hz, 1H), 7.15 (dd, *J* = 8.7, 2.8 Hz, 1H), 7.09 – 7.00 (m, 3H), 6.86 – 6.77 (m, 2H), 3.91 (s, 3H), 3.90 (s, 3H), 3.77 (s, 3H), 2.04 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 160.8, 159.2, 159.1, 136.3, 134.8, 129.3, 128.3, 125.5, 122.9, 114.2, 110.6, 63.9, 55.9, 55.4, 52.3, 27.0. HRMS (ESI) calcd. for $C_{19}H_{20}NO_5$ ([M+H]$^+$): 342.1336, found: 342.1343. $[\alpha]_D^{20}$ = -55.1º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 90%, determined by HPLC (Chiralpak-AD-H, hexane/isopropanol = 85/15, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 20.211 min (minor), t_R = 28.263 min (major).

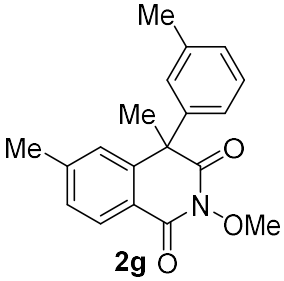
4-([1,1'-Biphenyl]-4-yl)-2-methoxy-4-methyl-7-phenylisoquinoline-1,3(2*H*,4*H*)-dione (2f)



2f

60% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (d, *J* = 2.0 Hz, 1H), 7.83 (dd, *J* = 8.2, 2.1 Hz, 1H), 7.70 – 7.64 (m, 2H), 7.58 – 7.52 (m, 4H), 7.52 – 7.46 (m, 2H), 7.46 – 7.38 (m, 3H), 7.38 – 7.30 (m, 1H), 7.28 – 7.24 (m, 3H), 3.96 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.0, 160.8, 142.5, 141.3, 141.2, 140.8, 140.2, 139.0, 133.1, 129.2, 128.9, 128.6, 128.3, 127.7, 127.6 (2C), 127.1, 127.0, 124.9, 64.0, 53.2, 27.0. HRMS (ESI) calcd. for $C_{29}H_{24}NO_3$ ($[M+H]^+$): 434.1751, found: 434.1753. $[\alpha]_D^{20}$ = -24.6º (*c* 1.5, $CHCl_3$). Enantiomeric excess: 90%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 15.790 min (minor), t_R = 26.692 min (major).

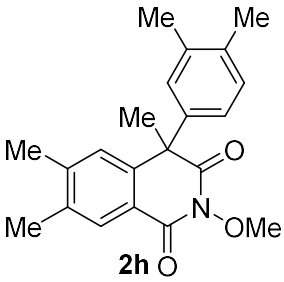
2-Methoxy-4,6-dimethyl-4-(*m*-tolyl)isoquinoline-1,3(2*H*,4*H*)-dione (2g)



2g

71% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.1 Hz, 1H), 7.28 (d, *J* = 8.1 Hz, 1H), 7.18 (t, *J* = 7.7 Hz, 1H), 7.08 (d, *J* = 7.7 Hz, 1H), 6.98 (s, 1H), 6.94 – 6.87 (m, 2H), 3.91 (s, 3H), 2.36 (s, 3H), 2.30 (s, 3H), 2.06 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.2, 160.9, 145.6, 144.1, 142.5, 138.6, 129.1, 128.8, 128.7 (2C), 128.3, 127.8, 124.2, 122.0, 63.9, 53.4, 26.9, 22.1, 21.7. HRMS (ESI) calcd. for $C_{19}H_{20}NO_3$ ($[M+H]^+$): 310.1438, found: 310.1444. $[\alpha]_D^{20}$ = -27.2º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 89%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 8.472 min (minor), t_R = 16.403 min (major).

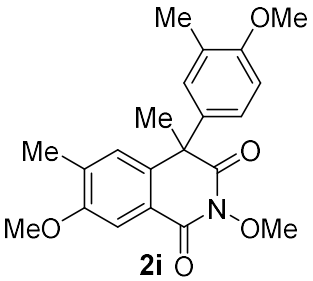
4-(3,4-Dimethylphenyl)-2-methoxy-4,6,7-trimethylisoquinoline-1,3(2*H*,4*H*)-dione (2h)



2h

85% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.04 (d, *J* = 7.9 Hz, 1H), 6.92 (s, 1H), 6.87 (s, 1H), 6.83 (d, *J* = 7.9 Hz, 1H), 3.90 (s, 3H), 2.34 (s, 3H), 2.25 (s, 3H), 2.20 (s, 3H), 2.20 (s, 3H), 2.03 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.5, 161.1, 144.5, 141.7, 140.1, 137.0, 136.9, 136.2, 130.0, 129.1, 128.8, 128.2, 124.4, 122.0, 63.8, 52.8, 26.8, 20.4, 20.1, 19.4. HRMS (ESI) calcd. for $C_{21}H_{24}NO_3$ ($[M+H]^+$): 338.1751, found: 338.1757. $[\alpha]_D^{20}$ = -15.3º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 86%, determined by HPLC (Chiralpak-AD-H, hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 11.156 min (major), t_R = 12.628 min (minor).

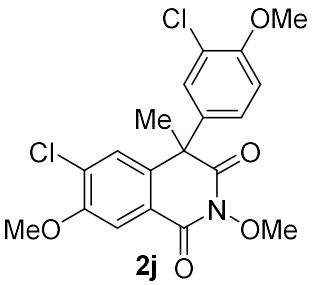
2,7-Dimethoxy-4-(4-methoxy-3-methylphenyl)-4,6-dimethylisoquinoline-1,3(2*H*,4*H*)-dione (2i)



2i

82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (s, 1H), 6.94 – 6.82 (m, 3H), 6.71 (d, *J* = 9.1 Hz, 1H), 3.95 (s, 3H), 3.90 (s, 3H), 3.78 (s, 3H), 2.21 (s, 3H), 2.14 (s, 3H), 2.01 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.7, 161.1, 157.5, 157.3, 136.4, 135.2, 134.4, 129.8, 129.4, 127.1, 125.6, 123.1, 109.9, 107.9, 63.8, 55.9, 55.4, 52.3, 27.0, 17.0, 16.6. HRMS (ESI) calcd. for $C_{21}H_{24}NO_5$ ($[M+H]^+$): 370.1649, found: 370.1657. $[\alpha]_D^{20}$ = -45.4º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 86%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 85/15, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 8.604 min (minor), t_R = 18.197 min (major).

6-Chloro-4-(3-chloro-4-methoxyphenyl)-2,7-dimethoxy-4-methylisoquinoline-1,3(2*H*,4*H*)-dione (2j)

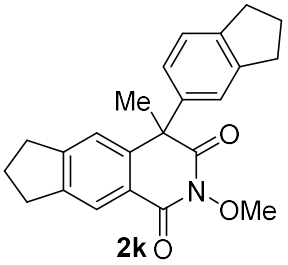


2j

46% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.08 (d, *J* = 2.5 Hz, 1H), 7.04 (s, 1H), 6.89 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.78 (d, *J* = 8.7 Hz, 1H), 3.95 (s, 3H), 3.84 (s, 3H), 3.80 (s, 3H), 1.94 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 160.0, 155.0, 154.8, 136.3, 135.0, 130.4, 129.6, 129.0, 126.6, 123.8, 123.0, 112.2, 110.4, 64.0, 56.8, 56.3, 52.1, 27.2. HRMS (ESI) calcd. for $C_{19}H_{20}Cl_2NO_5$ ($[M+H]^+$): 410.0557, found: 410.0553. $[\alpha]_D^{20}$ = +14.6º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 92%, determined by

HPLC (Chiralpak-OD-H, hexane/isopropanol = 85/15, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 16.548 min (minor), t_R = 24.704 min (major).

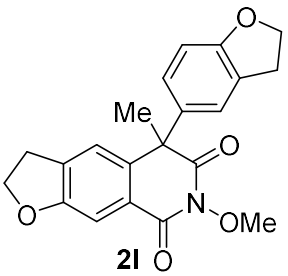
4-(2,3-Dihydro-1*H*-inden-5-yl)-2-methoxy-4-methyl-4,6,7,8-tetrahydro-1*H*-cyclopenta[*g*]isoquinoline-1,3(2*H*)-dione (2k)



2k

76% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (s, 1H), 7.12 (d, *J* = 8.0 Hz, 1H), 7.04 (s, 1H), 6.96 (s, 1H), 6.87 (d, *J* = 7.7 Hz, 1H), 3.91 (s, 3H), 2.98 (t, *J* = 7.5 Hz, 2H), 2.93 – 2.77 (m, 6H), 2.18 – 1.98 (m, 7H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.6, 161.3, 152.3, 145.0, 144.6, 143.9, 142.9, 140.8, 125.1, 124.6, 124.2, 123.6, 123.1, 122.5, 63.9, 53.3, 33.3, 33.0, 32.6, 32.4, 27.3, 25.5, 25.4. HRMS (ESI) calcd. for $C_{23}H_{24}NO_3$ ($[M+H]^+$): 362.1751, found: 362.1757. $[\alpha]_D^{20}$ = -6.9º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 84%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 8.631 min (minor), t_R = 19.846 min (major).

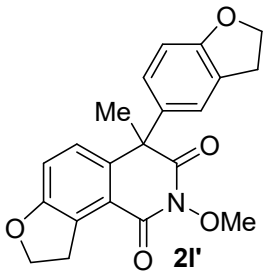
5-(2,3-Dihydrobenzofuran-5-yl)-7-methoxy-5-methyl-2,3-dihydrofuro[3,2-*g*]isoquinoline-6,8(5*H*,7*H*)-dione (2l)



2l

General Procedure B with modifications of 45 ºC, 36 h give **3m** and **3m'** in 38% and 20% yields as separable mixture. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (s, 1H), 6.99 (s, 1H), 6.94 (s,1H), 6.86 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.67 (d, *J* = 8.4 Hz, 1H), 4.62 (t, *J* = 8.7 Hz, 2H), 4.54 (t, *J* = 8.7 Hz, 2H), 3.90 (s, 3H), 3.35 – 3.02 (m, 4H), 2.01 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.5, 160.7, 159.9, 159.7, 137.2, 135.7, 134.9, 127.8, 127.0, 124.3, 123.8, 109.2, 107.5, 71.6 (2C), 63.8, 52.6, 29.8 (2C), 27.4. HRMS (ESI) calcd. for $C_{21}H_{20}NO_5$ ($[M+H]^+$): 366.1336, found: 366.1341. $[\alpha]_D^{20}$ = +42.1º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 88%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 75/25, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 13.454 min (minor), t_R = 33.552 min (major).

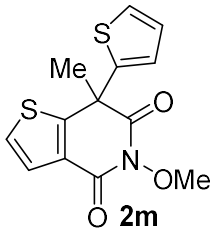
4-(2,3-Dihydrobenzofuran-5-yl)-2-methoxy-4-methyl-8,9-dihydrofuro[2,3-*h*]isoquinoline-1,3(2*H*,4*H*)-dione (2l')



2l'

General Procedure B with modifications of 45 ºC, 36 h give **3m** and **3m'** in 38% and 20% yields as separable mixture. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 (d, *J* = 8.4 Hz, 1H), 6.98 (s, 1H), 6.94 (d, *J* = 8.5 Hz, 1H), 6.84 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.67 (d, *J* = 8.4 Hz, 1H), 4.72 (ddd, *J* = 9.6, 8.2, 2.1 Hz, 2H), 4.55 (t, *J* = 8.7 Hz, 2H), 3.90 (s, 3H), 3.85 – 3.64 (m, 2H), 3.15 (t, *J* = 8.7 Hz, 2H), 2.02 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.6, 160.9, 160.5, 159.7, 135.9, 135.4, 129.6, 128.4, 127.8, 127.1, 123.8, 121.4, 115.2, 109.3, 72.6, 71.6, 63.9, 52.8, 31.4, 29.8, 27.7. HRMS (ESI) calcd. for $C_{21}H_{20}NO_5$ ($[M+H]^+$): 366.1336, found: 366.1342. $[\alpha]_D^{20}$ = -48.6º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 86%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 85/15, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 19.875 min (minor), t_R = 32.160 min (major).

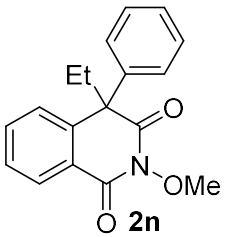
5-Methoxy-7-methyl-7-(thiophen-2-yl)thieno[3,2-*c*]pyridine-4,6(5*H*,7*H*)-dione (2m)



2m

General Procedure B with modifications of 40 ºC, 12 h give **3u** in 79% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 4.9 Hz, 1H), 7.34 (d, *J* = 4.9 Hz, 1H), 7.18 (d, *J* = 4.8 Hz, 1H), 6.84 (s, 1H), 6.77 (d, *J* = 2.9 Hz, 1H), 3.81 (s, 3H), 2.10 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.7, 156.7, 153.6, 146.1, 128.3, 127.1, 127.1, 126.4, 126.0, 125.5, 64.0, 50.1, 29.2. HRMS (ESI) calcd. for $C_{13}H_{12}NO_3S_2$ ($[M+H]^+$): 294.0253, found: 294.0255. $[\alpha]_D^{20}$ = +14.3º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 75%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 85/15, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 13.218 min (minor), t_R = 22.280 min (major).

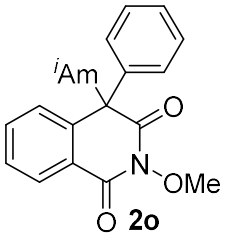
4-Ethyl-2-methoxy-4-phenylisoquinoline-1,3(2*H*,4*H*)-dione (2n)



2n

67% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 7.9 Hz, 1H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.33 – 7.21 (m, 3H), 7.16 (d, *J* = 7.5 Hz, 2H), 7.09 (d, *J* = 7.8 Hz, 1H), 3.94 (s, 3H), 3.14 – 3.00 (m, 1H), 2.41 – 2.23 (m, 1H), 0.73 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.5, 160.9, 142.7, 142.3, 134.6, 128.9, 128.7, 128.0, 127.9, 127.4, 126.0, 64.1, 58.6, 33.0, 9.5. HRMS (ESI) calcd. for $C_{18}H_{18}NO_3$ ($[M+H]^+$): 296.1281, found: 296.1276. $[\alpha]_D^{20}$ = -60.5º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 90%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 95/5, flow rate 0.5 mL/min, T = 25 ºC, 254 nm): t_R = 29.077 min (major), t_R = 32.160 min (minor).

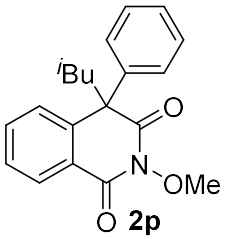
4-Isopentyl-2-methoxy-4-phenylisoquinoline-1,3(2*H*,4*H*)-dione (2o)



2o

51% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 7.8 Hz, 1H), 7.59 (t, *J* = 7.5 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.33 – 7.21 (m, 3H), 7.16 (d, *J* = 7.3 Hz, 2H), 7.10 (d, *J* = 7.8 Hz, 1H), 3.93 (s, 3H), 3.00 (td, *J* = 12.5, 3.9 Hz, 1H), 2.29 (td, *J* = 12.8, 4.0 Hz, 1H), 1.56 – 1.47 (m, 1H), 1.08 – 0.96 (m, 1H), 0.85 (d, *J* = 6.7 Hz, 3H), 0.79 (d, *J* = 6.6 Hz, 3H), 0.74 – 0.61 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.5, 160.9, 142.8, 142.6, 134.6, 128.9, 128.7, 128.0, 127.9, 127.8, 127.3, 125.8, 64.0, 57.9, 37.7, 33.8, 28.3, 22.6, 22.3. HRMS (ESI) calcd. for $C_{21}H_{24}NO_3$ ($[M+H]^+$): 338.1751, found: 338.1755. $[\alpha]_D^{20}$ = -36.3º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 86%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 95/5, flow rate 0.5 mL/min, T = 25 ºC, 254 nm): t_R = 13.887 min (major), t_R = 16.793 min (minor).

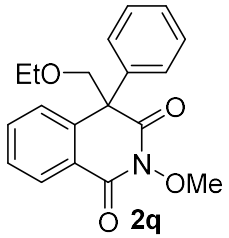
4-Isobutyl-2-methoxy-4-phenylisoquinoline-1,3(2*H*,4*H*)-dione (2p)



2p

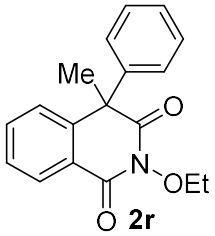
48% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 7.9 Hz, 1H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.33 – 7.19 (m, 3H), 7.19 – 7.07 (m, 3H), 3.91 (s, 3H), 3.04 (dd, *J* = 13.7, 8.4 Hz, 1H), 2.37 (dd, *J* = 13.6, 4.5 Hz, 1H), 1.43 – 1.28 (m, 1H), 0.79 (d, *J* = 6.7 Hz, 3H), 0.74 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.6, 160.8, 143.7, 142.2, 134.4, 128.9, 128.5, 128.0, 127.8, 127.2, 125.8, 63.7, 57.0, 47.5, 25.6, 24.4, 22.6. HRMS (ESI) calcd. for $C_{20}H_{22}NO_3$ ($[M+H]^+$): 324.1594, found: 324.1599. $[\alpha]_D^{20}$ = -46.0º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 86%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 95/5, flow rate 0.5 mL/min, T = 25 ºC, 254 nm): t_R = 14.793 min (major), t_R = 16.304 min (minor).

4-(Ethoxymethyl)-2-methoxy-4-phenylisoquinoline-1,3(2*H*,4*H*)-dione (2q)



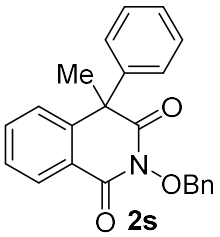
66% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, *J* = 7.8 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.33 – 7.23 (m, 3H), 7.14 (d, *J* = 7.8 Hz, 1H), 7.08 (d, *J* = 7.8 Hz, 2H), 4.73 (d, *J* = 8.1 Hz, 1H), 4.15 (d, *J* = 8.2 Hz, 1H), 3.95 (s, 3H), 3.54 – 3.42 (m, 1H), 3.42 – 3.30 (m, 1H), 1.00 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 161.0, 141.4, 139.8, 134.1, 129.1, 128.6, 128.2, 128.0, 127.8, 127.4, 126.4, 76.5, 67.4, 63.9, 59.0, 14.8. HRMS (ESI) calcd. for $C_{19}H_{20}NO_4$ ([M+H]$^+$): 326.1387, found: 326.1389. $[\alpha]_D^{20}$ = -39.2º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 92%, determined by HPLC (Chiralpak-AD-H, hexane/isopropanol = 85/15, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 13.679 min (major), t_R = 22.214 min (minor).

2-Ethoxy-4-methyl-4-phenylisoquinoline-1,3(2*H*,4*H*)-dione (2r)



66% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.58 (td, *J* = 7.6, 1.4 Hz, 1H), 7.49 (td, *J* = 7.8, 0.9 Hz, 1H), 7.34 – 7.21 (m, 3H), 7.18 – 7.07 (m, 3H), 4.20 – 4.03 (m, 2H), 2.09 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 161.1, 143.9, 142.6, 134.5, 128.9, 128.8, 128.0, 127.9 (2C), 127.1, 124.6, 72.2, 53.5, 26.9, 13.4. HRMS (ESI) calcd. for $C_{18}H_{18}NO_3$ ([M+H]$^+$): 296.1281, found: 296.1285. $[\alpha]_D^{20}$ = -65.4º (*c* 1.0, $CHCl_3$). Enantiomeric excess: 90%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 ºC, 254 nm): t_R = 11.345 min (minor), t_R = 23.988 min (major).

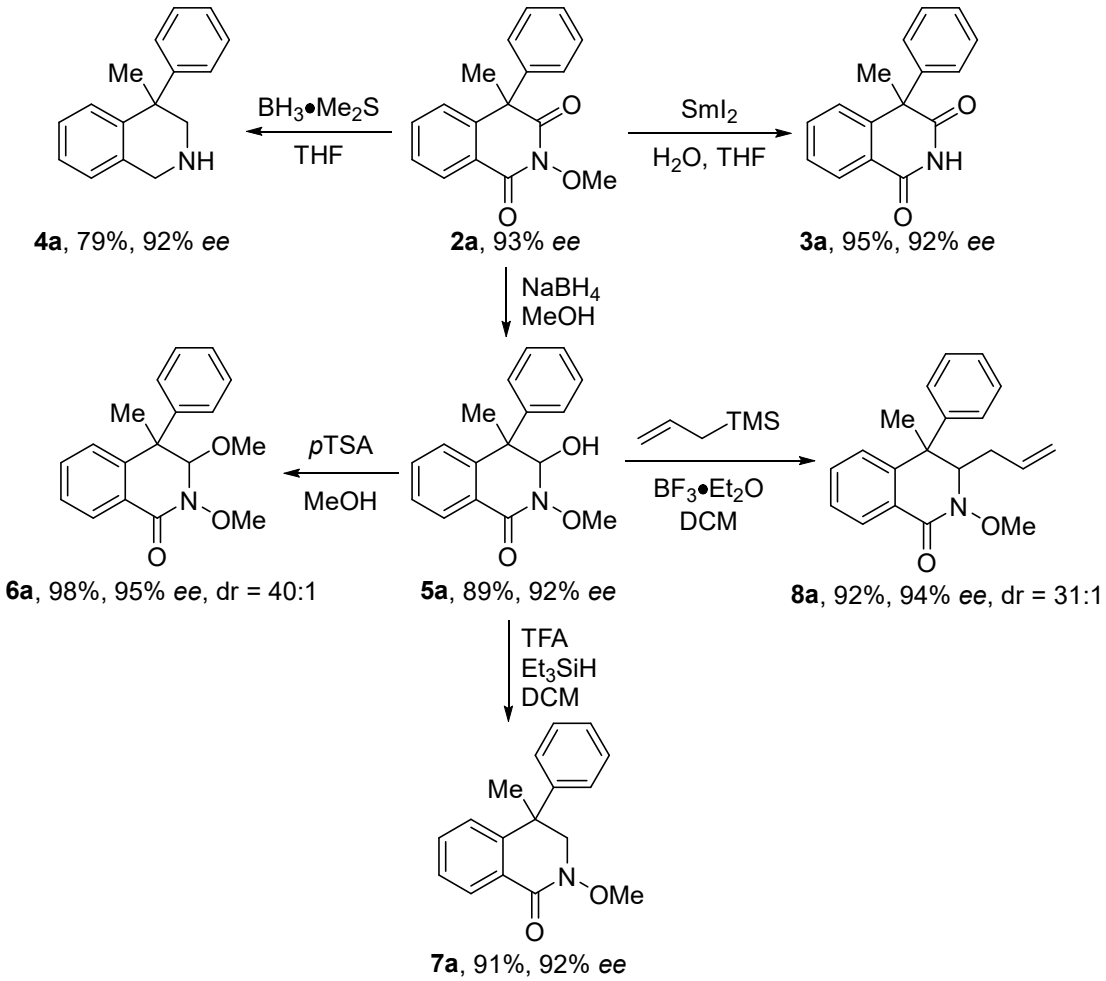
2-(Benzyloxy)-4-methyl-4-phenylisoquinoline-1,3(2*H*,4*H*)-dione (2s)



46% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, *J* = 7.8 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 1H), 7.53 – 7.44 (m, 3H), 7.36 – 7.22 (m, 6H), 7.09 (dd, *J* = 7.6, 3.0 Hz, 3H), 5.09 (s, 2H), 2.03 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.5, 161.1, 144.1, 142.5, 134.5, 133.9, 130.2, 129.2, 128.9, 128.8, 128.5, 128.0 (2C), 127.9, 127.3, 124.5, 78.2, 53.7,

27.2. HRMS (ESI) calcd. for $C_{23}H_{20}NO_3$ ([M+H]$^+$): 358.1438, found: 358.1442. $[\alpha]_D^{20}$ = -54.7° (*c* 1.0, $CHCl_3$). Enantiomeric excess: 88%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 °C, 254 nm): t_R = 12.828 min (major), t_R = 14.324 min (minor).

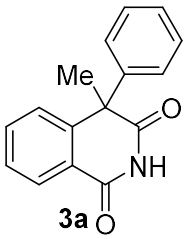
Derivatizations of 2a



Deprotection of Methoxy Group[1]

To a solution of **2a** (28.1 mg, 0.1 mmol) in THF (1 mL) and deoxygenated water (0.05 mL) was added SmI_2 (0.1 M in THF, 10 mL, 1.0 mmol, 10 equiv.) in an ice-bath. The mixture was stirred for 1 h, and diluted with EtOAc, washed with saturated $NaHCO_3$, saturated $Na_2S_2O_3$ and brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by column chromatography.

4-Methyl-4-phenylisoquinoline-1,3(2*H*,4*H*)-dione (3a)

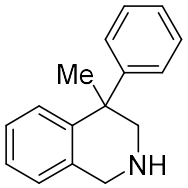


^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (dd, *J* = 7.9, 1.4 Hz, 1H), 8.17 (s, 1H), 7.60 (td, *J* = 7.6, 1.4 Hz, 1H), 7.52 – 7.44 (m, 1H), 7.35 – 7.23 (m, 3H), 7.21 – 7.16 (m, 2H), 7.14 (d, *J* = 8.0 Hz, 1H), 2.07 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.1, 164.0, 146.0, 142.6, 134.8, 128.9, 128.4, 128.3, 127.9, 127.4, 124.1, 52.1, 27.1. HRMS (ESI) calcd. for $C_{16}H_{14}NO_2$ ($[M+H]^+$): 252.1019, found: 252.1021. $[\alpha]_D^{20}$ = -14.7º (*c* 0.3, $CHCl_3$). Enantiomeric excess: 92%, determined by HPLC (Chiralpak-AS-H, hexane/isopropanol = 80/20, flow rate 0.5 mL /min, T = 25 ºC, 254 nm): t_R = 15.591 min (minor), t_R = 30.045 min (major).

Reduction of Lactam[1]

To a solution of **2a** (84.4 mg, 0.3 mmol) in dried THF (4 mL) was added $BH_3 \cdot Me_2S$ (2.0 M in THF, 0.9 mL, 1.8 mmol, 6.0 equiv.) dropwise in an ice-bath. The mixture was warmed to room temperature and stirred for 2.5 h, then heated to reflux for 48 h. After cooling to 0 ˚C, 10% HCl was added slowly to quench the reaction, and the resulting solution was refluxed for 1.5 h. The mixture was cooled to 0 ˚C again, and 12 N NaOH was added until pH > 10. The mixture was extracted with Et_2O for three times, and the combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography.

4-Methyl-4-phenyl-1,2,3,4-tetrahydroisoquinoline (4a)



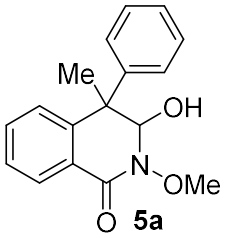
4a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.22 (m, 2H), 7.22 – 7.13 (m, 3H), 7.11 (d, *J* = 7.4 Hz, 2H), 7.06 (t, *J* = 7.3 Hz, 2H), 4.10 (s, 2H), 3.19 (d, *J* = 13.2 Hz, 1H), 3.05 (d, *J* = 13.2 Hz, 1H), 2.16 (s, 1H), 1.71 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.8, 142.5, 128.8, 128.2, 127.6, 126.7, 126.2, 126.1, 125.8, 27.0. HRMS (ESI) calcd. for $C_{16}H_{18}N$ ($[M+H]^+$): 224.1434, found: 224.1435. $[\alpha]_D^{20}$ = +10.3º (*c* 0.8, $CHCl_3$). Enantiomeric excess: 92%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 80/20, flow rate 0.5 mL/min, T = 25 ºC, 254 nm): t_R = 9.933 min (minor), t_R = 12.343 min (major).

Partial Reduction of Imide

To a solution of **2a** (28.1 mg, 0.1 mmol) in MeOH (1 mL) was added $NaBH_4$ (7.6 mg, 0.2 mmol, 2.0 equiv.) in an ice-bath. The mixture was warmed to room temperature and stirred for 2 h, then saturated $NaHCO_3$ was added slowly to quench the reaction, and the resulting solution was stirred vigorously for 15 min. The mixture was extracted with EtOAc for three times, and the combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography.

3-Hydroxy-2-methoxy-4-methyl-4-phenyl-3,4-dihydroisoquinolin-1(2*H*)-one (5a)



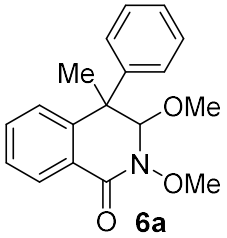
5a

^{1}H NMR (400 MHz, Acetone-d_6) δ 8.13 (dd, *J* = 7.7, 1.1 Hz, 1H), 7.68 (td, *J* = 7.6, 1.4 Hz, 1H), 7.52 (t, *J* = 8.2 Hz, 2H), 7.30 – 7.23 (m, 2H), 7.23 – 7.16 (m, 1H), 7.02 (dd, *J* = 5.4, 3.4 Hz, 2H), 5.97 (d, *J* = 5.5 Hz, 1H), 5.51 (d, *J* = 5.4 Hz, 1H), 3.33 (s, 3H), 2.86 (s, 1H), 1.91 (s, 3H). ^{13}C NMR (101 MHz, Acetone-d_6) δ 162.6, 147.1, 142.9, 133.5, 130.4, 129.0, 128.2 (3C), 127.5, 127.4, 91.1, 62.0, 50.3, 24.3. HRMS (ESI) calcd. for $C_{17}H_{18}NO_3$ ([M+H]$^+$): 284.1281, found: 284.1284. $[\alpha]_D^{20}$ = -71.9º (*c* 0.07, MeOH). Enantiomeric excess: 92%, determined by HPLC (Chiralpak-ID, hexane/isopropanol = 80/20, flow rate 0.5 mL /min, T = 25 ºC, 254 nm): t_R = 25.796 min (minor), t_R = 28.469 min (major).

Methoxylation of Hemiaminal[2]

A solution of **5a** (28.3 mg, 0.1 mmol) in MeOH (0.4 mL) was added *p*-toluenesulfonic acid (1.9 mg, 0.01 mmol, 0.1 equiv.) and stirred at room temperature for 12 h. After evaporating the volatile, the residue was purified by flash column chromatography.

2,3-Dimethoxy-4-methyl-4-phenyl-3,4-dihydroisoquinolin-1(2*H*)-one (6a)



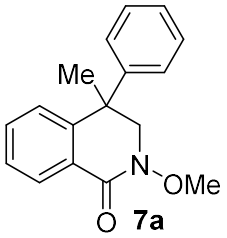
6a

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.60 (td, *J* = 7.6, 1.5 Hz, 1H), 7.48 (td, *J* = 7.6, 1.2 Hz, 1H), 7.37 (dd, *J* = 7.8, 0.7 Hz, 1H), 7.28 – 7.16 (m, 3H), 6.96 – 6.89 (m, 2H), 4.91 (s, 1H), 3.52 (s, 3H), 3.19 (s, 3H), 1.89 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.9, 145.2, 141.6, 133.1, 128.9, 128.6, 128.3, 127.8, 127.2, 126.8, 98.5, 62.1, 58.5, 49.5, 23.7. HRMS (ESI) calcd. for $C_{18}H_{20}NO_3$ ([M+H]$^+$): 298.1438, found: 298.1441. $[\alpha]_D^{20}$ = -40.6º (*c* 0.6, $CHCl_3$). Enantiomeric excess: 95%, determined by HPLC (Chiralpak-ID, hexane/isopropanol = 80/20, flow rate 0.5 mL /min, T = 25 ºC, 254 nm): t_R = 21.236 min (major), t_R = 27.765 min (minor).

Reduction of Hemiaminal[3]

To a solution of **5a** (14 mg, 0.05 mmol) in DCM (1 mL) was added triethylsilane (17 μL, 0.107 mmol, 2.0 equiv.) and trifluoroacetic acid (0.5 mL). After stirring at room temperature for 1 h, triethylsilane (17 μL, 0.107 mmol, 2.0 equiv.) was added again. After 1 h, the mixture was concentrated under reduced pressure, and the residue was purified by flash column chromatography.

2-Methoxy-4-methyl-4-phenyl-3,4-dihydroisoquinolin-1(2*H*)-one (7a)



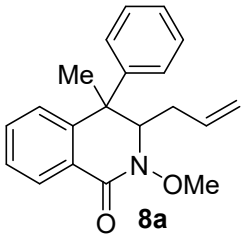
7a

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.48 (td, *J* = 7.5, 1.6 Hz, 1H), 7.42 (td, *J* = 7.5, 1.3 Hz, 1H), 7.36 – 7.22 (m, 3H), 7.22 – 7.12 (m, 2H), 7.08 (dd, *J* = 7.7, 0.9 Hz, 1H), 4.06 (d, *J* = 11.8 Hz, 1H), 3.82 (d, *J* = 11.8 Hz, 1H), 3.63 (s, 3H), 1.83 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7, 145.0, 144.9, 132.7, 128.7 (2C), 128.5, 127.6, 127.2 (2C), 126.6, 61.5, 61.1, 44.7, 25.9. HRMS (ESI) calcd. for $C_{17}H_{18}NO_2$ ($[M+H]^+$): 268.1332, found: 268.1334. $[\alpha]_D^{20}$ = +15.7º (*c* 0.8, $CHCl_3$). Enantiomeric excess: 92%, determined by HPLC (Chiralpak-AS-H, hexane/isopropanol = 80/20, flow rate 0.5 mL /min, T = 25 ºC, 254 nm): t_R = 13.166 min (major), t_R = 16.298 min (minor).

Allylation of Hemiaminal[4]

To a solution of **5a** (28.3 mg, 0.1 mmol) and allyltrimethylsilane (48 μL, 0.3 mmol, 3.0 equiv.) in DCM (2 mL) was added $BF_3{\cdot}Me_2O$ (38 μL, 0.3 mmol, 3.0 equiv.) at -60 ˚C, and stirred for 4 h. The reaction mixture was purified by flash column chromatography directly.

3-Allyl-2-methoxy-4-methyl-4-phenyl-3,4-dihydroisoquinolin-1(2*H*)-one (8a)



8a

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 7.7 Hz, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.37 – 7.13 (m, 4H), 6.94 (d, *J* = 7.2 Hz, 2H), 5.78 (dq, *J* = 10.0, 7.2 Hz, 1H), 4.99 (d, *J* = 17.2 Hz, 1H), 4.96 (d, *J* = 10.2 Hz, 1H), 4.07 (dd, *J* = 8.0, 4.7 Hz, 1H), 3.16 (s, 3H), 2.65 – 2.48 (m, 1H), 2.45 – 2.26 (m, 1H), 1.82 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.2, 147.5, 141.6, 134.6, 132.9, 129.9, 128.4, 128.2, 127.9, 127.0, 126.5, 126.4, 117.5, 70.6, 61.0, 48.1, 35.1, 24.3. HRMS (ESI) calcd. for $C_{20}H_{22}NO_2$ ($[M+H]^+$): 308.1645, found: 308.1647. $[\alpha]_D^{20}$ = -127.7º (*c* 0.3, $CHCl_3$). Enantiomeric excess: 94%, determined by HPLC (Chiralpak-OD-H, hexane/isopropanol = 95/5, flow rate 0.3 mL/min, T = 25 ºC, 254 nm): t_R = 16.123 min (minor), t_R = 16.771 min (major).

Determination of the Absolute Configuration of 2a, 5a, 6a and 8a

The crystals of **2a**, **5a**, **6a** and **8a**, which were detected by X-ray, were the major product

determined by chiral HPLC.

X-ray crystal data of the enantiomerically enriched isomer 2a:

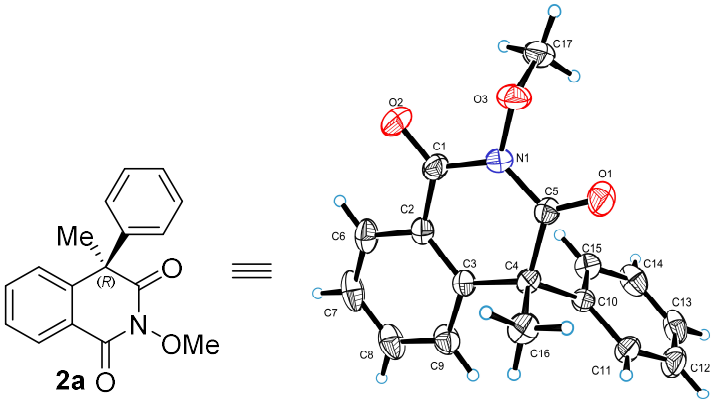


Table 1 Crystal data and structure refinement for 170412.

Identification code	170412
Empirical formula	$C_{17}H_{15}NO_3$
Formula weight	281.30
Temperature/K	292(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	8.53180(10)
b/Å	9.34100(10)
c/Å	18.0021(2)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1434.69(3)
Z	4
ρ_{calc}g/cm^3	1.302
μ/mm^{-1}	0.731
F(000)	592.0
Crystal size/mm^3	0.240 × 0.220 × 0.210
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.826 to 142.52
Index ranges	$-6 \le h \le 10$, $-11 \le k \le 10$, $-16 \le l \le 21$
Reflections collected	4843
Independent reflections	2688 [R_{int} = 0.0185, R_{sigma} = 0.0227]
Data/restraints/parameters	2688/0/193
Goodness-of-fit on F^2	1.076
Final R indexes [I>=2σ (I)]	R_1 = 0.0356, wR_2 = 0.0916

Final R indexes [all data]	R_1 = 0.0366, wR_2 = 0.0929
Largest diff. peak/hole / e Å^{-3}	0.14/-0.14
Flack parameter	0.01(10)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 170412. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O3	-415.1(19)	7890.1(16)	6223.7(10)	69.1(5)
O1	1280(2)	6518(2)	7197.0(8)	77.6(6)
N1	431(2)	6684.8(18)	6017.3(9)	51.6(4)
O2	-755(3)	6811(2)	4899.4(11)	87.6(6)
C10	3734(2)	4720.3(19)	6656.1(10)	43.1(4)
C5	1248(2)	6005(2)	6585.2(10)	47.9(4)
C4	2021(2)	4568(2)	6404.5(10)	43.4(4)
C2	1097(2)	4934(2)	5076.2(10)	50.5(4)
C15	4696(3)	5644(2)	6270.0(12)	56.9(5)
C1	175(3)	6199(2)	5297.2(12)	56.3(5)
C3	1961(2)	4162(2)	5587.9(10)	45.9(4)
C11	4330(3)	3990(3)	7261.2(12)	60.1(5)
C6	1058(3)	4519(3)	4328.2(12)	70.7(7)
C12	5888(3)	4188(3)	7467.7(16)	77.4(8)
C16	1095(3)	3445(3)	6851.2(13)	63.8(6)
C13	6828(3)	5110(3)	7087.9(16)	78.3(8)
C14	6250(3)	5840(3)	6488.5(17)	74.0(7)
C9	2740(3)	2938(3)	5342.5(15)	68.0(6)
C17	434(4)	9164(3)	6039.2(18)	79.2(8)
C8	2673(3)	2534(3)	4608.2(17)	83.3(9)
C7	1848(3)	3325(4)	4099.9(15)	86.7(9)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for 170412. The Anisotropic displacement factor exponent takes the form: -2π^2[h^{2}a*^{2}U$_{11}$+2hka*b*U$_{12}$+…].

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O3	58.5(8)	51.3(8)	97.3(12)	-2.5(8)	0.5(8)	15.1(7)

O1	78.8(10)	100.2(13)	53.9(8)	-23.7(9)	-5.1(8)	33.4(10)
N1	51.7(8)	45.6(8)	57.5(9)	1.1(7)	-1.9(8)	8.6(7)
O2	103.6(14)	75.4(11)	84.0(11)	20.1(10)	-38.0(11)	10.2(11)
C10	42.2(8)	43.8(9)	43.3(8)	-2.7(7)	0.6(7)	4.4(8)
C5	41.6(9)	56.9(11)	45.1(9)	-1.6(8)	0.7(8)	4.3(9)
C4	40.6(8)	47.9(10)	41.7(9)	5.1(7)	1.8(7)	0.2(7)
C2	52.6(9)	56.2(11)	42.8(9)	2.2(8)	-0.4(8)	-12.5(9)
C15	48.8(10)	60.0(12)	62.0(11)	3.3(10)	4.9(9)	-3.8(9)
C1	61.9(12)	52.4(11)	54.7(10)	14.2(9)	-11.8(9)	-6.5(9)
C3	39.7(8)	52(1)	46.2(9)	-1.7(8)	1.8(7)	-4.1(8)
C11	61.6(12)	64.5(13)	54.3(11)	3.9(10)	-9.6(10)	7.2(10)
C6	75.0(14)	92.8(18)	44.4(10)	-1.4(11)	-5.7(10)	-21.8(14)
C12	65.9(15)	94.9(19)	71.2(14)	-14.6(14)	-24.3(12)	24.1(14)
C16	57.1(11)	71.6(14)	62.8(12)	15.3(11)	7.5(10)	-13.0(11)
C13	44(1)	100(2)	90.6(18)	-45.1(16)	-12.1(11)	10.9(12)
C14	49.6(11)	79.9(16)	92.4(18)	-21.5(14)	11.1(12)	-14.1(12)
C9	56.5(12)	71.8(14)	75.7(15)	-21.0(12)	-2.8(11)	9.4(11)
C17	85.7(17)	47.0(11)	105(2)	-0.5(12)	-17.4(16)	3.1(12)
C8	65.1(14)	97(2)	87.5(18)	-44.6(17)	6.6(14)	2.0(15)
C7	77.5(16)	123(2)	59.8(14)	-39.7(16)	7.5(13)	-24.0(18)

Table 4 Bond Lengths for 170412.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O3	N1	1.388(2)	C2	C3	1.383(3)
O3	C17	1.432(3)	C2	C6	1.402(3)
O1	C5	1.201(2)	C2	C1	1.474(3)
N1	C5	1.391(3)	C15	C14	1.394(3)
N1	C1	1.391(3)	C3	C9	1.394(3)
O2	C1	1.212(3)	C11	C12	1.392(3)
C10	C15	1.379(3)	C6	C7	1.366(5)
C10	C11	1.382(3)	C12	C13	1.361(5)
C10	C4	1.537(2)	C13	C14	1.369(4)
C5	C4	1.530(3)	C9	C8	1.376(3)
C4	C3	1.519(3)	C8	C7	1.371(5)
C4	C16	1.540(3)			

Table 5 Bond Angles for 170412.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N1	O3	C17	110.40(18)	C3	C2	C1	121.54(17)
O3	N1	C5	115.75(16)	C6	C2	C1	117.9(2)
O3	N1	C1	115.61(17)	C10	C15	C14	120.4(2)
C5	N1	C1	127.94(17)	O2	C1	N1	120.0(2)
C15	C10	C11	119.14(19)	O2	C1	C2	124.6(2)
C15	C10	C4	118.40(17)	N1	C1	C2	115.40(17)
C11	C10	C4	122.45(18)	C2	C3	C9	118.09(19)
O1	C5	N1	120.22(19)	C2	C3	C4	122.14(18)
O1	C5	C4	122.31(18)	C9	C3	C4	119.71(18)
N1	C5	C4	117.41(16)	C10	C11	C12	119.7(2)
C3	C4	C5	114.22(15)	C7	C6	C2	120.2(3)
C3	C4	C10	109.89(14)	C13	C12	C11	120.8(2)
C5	C4	C10	105.44(15)	C12	C13	C14	119.9(2)
C3	C4	C16	108.54(17)	C13	C14	C15	120.0(3)
C5	C4	C16	105.37(15)	C8	C9	C3	120.6(3)
C10	C4	C16	113.40(16)	C7	C8	C9	121.0(3)
C3	C2	C6	120.6(2)	C6	C7	C8	119.5(2)

Table 6 Torsion Angles for 170412.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C17	O3	N1	C5	99.9(2)	C3	C2	C1	N1	9.8(3)
C17	O3	N1	C1	-88.9(2)	C6	C2	C1	N1	-171.1(2)
O3	N1	C5	O1	-5.0(3)	C6	C2	C3	C9	-2.2(3)
C1	N1	C5	O1	-174.9(2)	C1	C2	C3	C9	176.88(19)
O3	N1	C5	C4	172.29(15)	C6	C2	C3	C4	-179.39(19)
C1	N1	C5	C4	2.4(3)	C1	C2	C3	C4	-0.3(3)
O1	C5	C4	C3	-175.4(2)	C5	C4	C3	C2	-8.3(3)
N1	C5	C4	C3	7.4(2)	C10	C4	C3	C2	-126.53(19)
O1	C5	C4	C10	-54.6(2)	C16	C4	C3	C2	108.9(2)
N1	C5	C4	C10	128.12(17)	C5	C4	C3	C9	174.59(19)
O1	C5	C4	C16	65.6(2)	C10	C4	C3	C9	56.3(2)
N1	C5	C4	C16	-111.7(2)	C16	C4	C3	C9	-68.2(2)
C15	C10	C4	C3	55.1(2)	C15	C10	C11	C12	-0.5(3)
C11	C10	C4	C3	-126.2(2)	C4	C10	C11	C12	-179.1(2)
C15	C10	C4	C5	-68.5(2)	C3	C2	C6	C7	1.4(3)
C11	C10	C4	C5	110.2(2)	C1	C2	C6	C7	-177.7(2)

C15	C10	C4	C16	176.76(19)	C10	C11	C12	C13	1.0(4)
C11	C10	C4	C16	-4.6(3)	C11	C12	C13	C14	-0.9(4)
C11	C10	C15	C14	-0.2(3)	C12	C13	C14	C15	0.3(4)
C4	C10	C15	C14	178.6(2)	C10	C15	C14	C13	0.2(4)
O3	N1	C1	O2	-1.0(3)	C2	C3	C9	C8	1.4(3)
C5	N1	C1	O2	169.0(2)	C4	C3	C9	C8	178.6(2)
O3	N1	C1	C2	178.95(17)	C3	C9	C8	C7	0.3(4)
C5	N1	C1	C2	-11.1(3)	C2	C6	C7	C8	0.3(4)
C3	C2	C1	O2	-170.3(2)	C9	C8	C7	C6	-1.1(5)
C6	C2	C1	O2	8.8(3)					

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 170412.

Atom	*x*	*y*	*z*	U(eq)
H15	4307	6138	5861	68
H11	3694	3369	7529	72
H6	493	5058	3986	85
H12	6291	3684	7870	93
H16A	1561	2519	6782	96
H16B	29	3426	6682	96
H16C	1119	3691	7369	96
H13	7863	5244	7235	94
H14	6892	6467	6227	89
H9	3311	2389	5678	82
H17A	504	9252	5509	119
H17B	1469	9112	6246	119
H17C	-102	9982	6239	119
H8	3195	1712	4454	100
H7	1826	3052	3603	104

Crystal structure determination of [170412]

Crystal Data for $C_{17}H_{15}NO_3$ (*M* =281.30 g/mol): orthorhombic, space group $P2_12_12_1$ (no. 19), *a* = 8.53180(10) Å, *b* = 9.34100(10) Å, *c* = 18.0021(2) Å, *V* = 1434.69(3) Å^3, *Z* = 4, *T* = 292(2) K, μ(CuKα) = 0.731 mm^{-1}, *Dcalc* = 1.302 g/cm^3, 4843 reflections measured (9.826° ≤ 2Θ ≤ 142.52°), 2688 unique (R_{int} = 0.0185, R_{sigma} = 0.0227) which were used in all calculations. The final R_1 was 0.0356 (I > 2σ(I)) and wR_2 was 0.0929 (all data).

X-ray crystal data of the enantiomerically enriched isomer 5a:

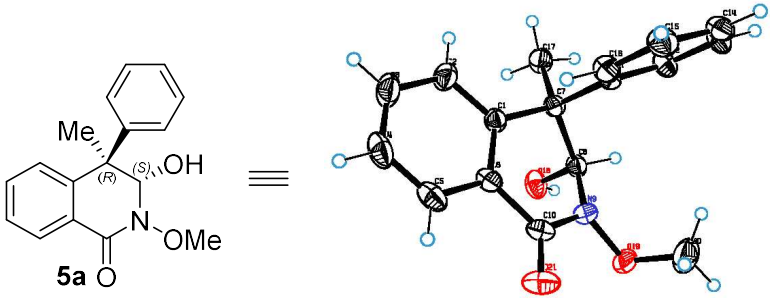


Table 1 Crystal data and structure refinement for exp_47.

Identification code	exp_47
Empirical formula	$C_{17}H_{17}NO_3$
Formula weight	283.32
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	7.37420(10)
b/Å	8.59040(10)
c/Å	12.08780(10)
α/°	90
β/°	101.0020(10)
γ/°	90
Volume/Å^3	751.656(15)
Z	2
ρ_{calc}g/cm^3	1.252
μ/mm^{-1}	0.698
F(000)	300.0
Crystal size/mm^3	0.6 × 0.4 × 0.1
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.45 to 147.672
Index ranges	-9 ≤ h ≤ 7, -9 ≤ k ≤ 10, -14 ≤ l ≤ 14
Reflections collected	9272
Independent reflections	2808 [R_{int} = 0.0284, R_{sigma} = 0.0193]
Data/restraints/parameters	2808/1/193
Goodness-of-fit on F^2	1.248
Final R indexes [I>=2σ (I)]	R_1 = 0.0606, wR_2 = 0.1325
Final R indexes [all data]	R_1 = 0.0608, wR_2 = 0.1331
Largest diff. peak/hole / e Å^{-3}	0.42/-0.66
Flack parameter	0.13(8)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_47. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O18	7307(2)	4902(2)	5281.1(12)	51.0(4)
O19	3424(2)	6034(2)	5258.1(13)	54.8(5)
O21	4642(3)	8418(2)	6602.1(16)	64.9(5)
N9	4930(2)	5873(2)	6150.2(15)	43.0(4)
C11	5842(2)	3503(2)	7908.5(15)	35.3(4)
C8	6129(2)	4559(3)	6028.9(15)	38.2(4)
C16	5546(3)	4210(3)	8887.3(17)	45.5(5)
C7	7224(2)	4137(2)	7215.5(15)	35.2(4)
C17	8616(3)	2844(3)	7099(2)	47.8(5)
C1	8186(3)	5609(2)	7739.7(16)	37.8(4)
C12	4847(3)	2154(3)	7548.3(18)	46.4(5)
C14	3281(3)	2287(3)	9118(2)	53.7(6)
C13	3570(3)	1561(3)	8155(2)	55.7(6)
C10	5524(3)	7201(3)	6709.1(16)	44.0(5)
C6	7334(3)	7048(3)	7496.3(15)	41.3(4)
C15	4282(3)	3604(3)	9498.4(19)	54.3(6)
C2	9883(3)	5572(3)	8475(2)	51.7(5)
C5	8161(4)	8407(3)	7986(2)	55.7(6)
C3	10706(3)	6926(4)	8960(2)	63.5(7)
C4	9853(4)	8331(4)	8717(2)	64.9(7)
C20	1787(4)	5519(8)	5608(4)	108.0(18)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_47. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O18	54.8(8)	59.6(11)	40.9(7)	-6.2(7)	14.7(6)	-18.7(8)
O19	38.4(7)	72.5(13)	48.9(8)	21.4(8)	-3.3(6)	-5.4(7)
O21	84.2(12)	48.2(11)	59.5(10)	9.3(8)	6.7(9)	22.7(9)
N9	39.9(7)	44.8(11)	41.0(7)	9.1(7)	-1.0(6)	0.7(6)

C11	37.9(7)	27.4(9)	39.3(8)	4.9(7)	4.3(6)	1.6(7)
C8	40.3(8)	37.2(11)	36.3(8)	-0.5(7)	5.6(6)	-7.9(7)
C16	50.8(10)	44.5(11)	41.0(9)	-2.0(8)	8.4(8)	-3.6(8)
C7	37.2(8)	29.8(10)	37.6(8)	-1.3(7)	4.2(6)	-2.4(7)
C17	44.7(10)	37.2(12)	61.6(12)	-6.3(9)	10.5(8)	2.5(8)
C1	39.4(8)	34.2(10)	39.3(8)	-1.7(8)	6.1(6)	-5.0(8)
C12	54.7(10)	31.6(11)	53.7(11)	-0.8(9)	12.1(8)	-6.3(8)
C14	49.5(10)	52.1(14)	61.7(12)	22.4(11)	16.0(9)	3.7(9)
C13	55.8(11)	39.6(13)	72.9(14)	9.3(10)	15.3(10)	-11.2(9)
C10	54.7(10)	37.1(11)	41.6(9)	9.8(8)	12.6(8)	7.3(9)
C6	52.9(10)	33.6(10)	37.8(9)	2.9(8)	9.8(7)	-3.8(8)
C15	58.8(11)	60.9(16)	45.4(10)	5.4(9)	15.4(8)	2.3(11)
C2	44.8(10)	48.2(13)	56.9(11)	-6.5(10)	-3.7(8)	-5.1(10)
C5	78.0(14)	33.7(12)	56.9(12)	-0.7(9)	16.5(10)	-9.8(11)
C3	51.9(11)	66.3(18)	66.7(14)	-12.0(14)	-2.7(10)	-19.8(13)
C4	77.7(16)	50.5(16)	66.2(15)	-13.6(12)	12.9(12)	-30.3(13)
C20	44.9(12)	163(5)	112(3)	63(3)	6.1(14)	-19.1(19)

Table 4 Bond Lengths for exp_47.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O18	C8	1.399(2)	C7	C1	1.527(3)
O19	N9	1.400(2)	C1	C6	1.393(3)
O19	C20	1.424(3)	C1	C2	1.391(3)
O21	C10	1.225(3)	C12	C13	1.396(3)
N9	C8	1.458(3)	C14	C13	1.372(4)
N9	C10	1.355(3)	C14	C15	1.381(4)
C11	C16	1.384(3)	C10	C6	1.490(3)
C11	C7	1.537(2)	C6	C5	1.395(3)
C11	C12	1.396(3)	C2	C3	1.389(4)
C8	C7	1.549(2)	C5	C4	1.386(4)
C16	C15	1.396(3)	C3	C4	1.367(5)
C7	C17	1.537(3)			

Table 5 Bond Angles for exp_47.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N9	O19	C20	109.50(19)	C6	C1	C7	119.51(16)
O19	N9	C8	113.98(17)	C2	C1	C7	122.5(2)

C10	N9	O19	115.72(17)	C2	C1	C6	118.0(2)
C10	N9	C8	123.87(16)	C13	C12	C11	120.4(2)
C16	C11	C7	122.65(18)	C13	C14	C15	119.7(2)
C16	C11	C12	118.14(18)	C14	C13	C12	120.6(2)
C12	C11	C7	119.21(17)	O21	C10	N9	123.4(2)
O18	C8	N9	111.39(18)	O21	C10	C6	122.4(2)
O18	C8	C7	111.67(14)	N9	C10	C6	114.15(18)
N9	C8	C7	107.87(15)	C1	C6	C10	121.73(19)
C11	C16	C15	121.3(2)	C1	C6	C5	120.65(19)
C11	C7	C8	107.74(14)	C5	C6	C10	117.6(2)
C17	C7	C11	108.50(16)	C14	C15	C16	119.8(2)
C17	C7	C8	108.90(16)	C3	C2	C1	121.2(2)
C1	C7	C11	111.78(15)	C4	C5	C6	119.9(3)
C1	C7	C8	108.03(16)	C4	C3	C2	120.2(2)
C1	C7	C17	111.77(15)	C3	C4	C5	120.0(2)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for exp_47.

Atom	*x*	*y*	*z*	U(eq)
H001	6987.93	4402.96	4698.19	77
H006	5355.37	3668.34	5733.67	46
H007	6202.49	5105.47	9142.94	55
H00A	9182.91	2477.25	7834.42	72
H00B	9549.34	3249.01	6722.53	72
H00C	7985.91	1999.21	6669.04	72
H00D	5036.26	1647.42	6899.91	56
H00E	2415.57	1893.61	9511.84	64
H00F	2908.19	665.41	7904.59	67
H00G	4114.38	4085.92	10160.41	65
H00H	10478.91	4623.73	8645.82	62
H00I	7578.51	9361.92	7821.92	67
H00J	11840.56	6876.71	9451.96	76
H00K	10406.81	9236.35	9042.7	78
H00L	780.25	5517.3	4973.15	162
H00M	1504.3	6207.07	6177.12	162
H00N	1970.94	4484.04	5908.23	162

Crystal structure determination of [exp_47]

Crystal Data for $C_{17.000001}H_{17.000001}NO_3$ (M =283.32 g/mol): monoclinic, space group $P2_1$ (no. 4), a = 7.37420(10) Å, b = 8.59040(10) Å, c = 12.08780(10) Å, β = 101.0020(10)°, V = 751.656(15) Å^3, Z = 2, T = 293(2) K, μ(CuKα) = 0.698 mm^{-1}, *Dcalc* = 1.252 g/cm^3, 9272 reflections measured (7.45° ≤ 2Θ ≤ 147.672°), 2808 unique (R_{int} = 0.0284, R_{sigma} = 0.0193) which were used in all calculations. The final R_1 was 0.0606 (I > 2σ(I)) and wR_2 was 0.1331 (all data).

X-ray crystal data of the enantiomerically enriched isomer 6a:

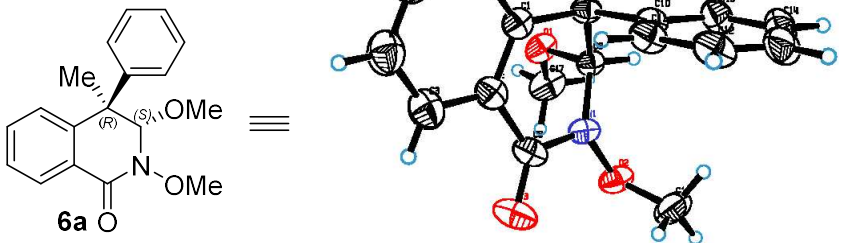


Table 1 Crystal data and structure refinement for liyan-cxf-co-meoh.

Identification code	liyan-cxf-co-meoh
Empirical formula	$C_{18}H_{19}NO_3$
Formula weight	297.34
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.87176(11)
b/Å	12.32287(17)
c/Å	16.6389(3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1614.02(4)
Z	4
ρ_{calc}g/cm^3	1.224
μ/mm^{-1}	0.673
F(000)	632.0
Crystal size/mm^3	0.4 × 0.3 × 0.2
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.93 to 147.734
Index ranges	-9 ≤ h ≤ 9, -15 ≤ k ≤ 13, -20 ≤ l ≤ 19

Reflections collected	12148
Independent reflections	3210 [R_{int} = 0.0280, R_{sigma} = 0.0186]
Data/restraints/parameters	3210/37/202
Goodness-of-fit on F^2	1.066
Final R indexes [I>=2σ (I)]	R_1 = 0.0436, wR_2 = 0.1289
Final R indexes [all data]	R_1 = 0.0464, wR_2 = 0.1331
Largest diff. peak/hole / e $Å^{-3}$	0.21/-0.22
Flack parameter	-0.06(9)

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

Atom	*x*	*y*	*z*	U(eq)
O1	4271.9(19)	102.7(13)	1403.9(10)	56.7(4)
N1	5171(2)	-1291.9(14)	2294.7(11)	50.4(4)
C8	4076(2)	-357.9(15)	2173.3(12)	44.5(4)
C10	1990(2)	-1079.5(17)	3156.1(13)	48.6(5)
C7	2217(2)	-726.0(16)	2272.6(12)	46.2(4)
C2	3267(3)	-2368.6(19)	1516.5(13)	57.9(5)
O2	6791.6(19)	-1044.4(15)	2588.7(10)	62.8(4)
C9	4980(3)	-2224.0(17)	1877.5(15)	57.7(5)
C1	1920(3)	-1669.7(18)	1699.9(13)	52.0(5)
O3	6112(3)	-2904.7(16)	1836.2(17)	87.3(6)
C16	1047(3)	249.1(18)	2096.5(17)	62.0(6)
C15	2331(4)	-347(2)	3764.7(16)	64.8(6)
C11	1449(3)	-2115.4(19)	3365.3(15)	60.4(6)
C17	5877(3)	597(3)	1290.6(19)	75.7(8)
C6	328(3)	-1888(2)	1373.1(17)	69.1(6)
C12	1303(4)	-2408(2)	4169.4(18)	75.9(8)
C4	1421(5)	-3433(3)	683(2)	89.5(7)
C13	1705(5)	-1678(3)	4765.5(16)	85.7(10)
C3	3026(4)	-3248(3)	1010.3(18)	78.9(7)
C14	2201(5)	-642(3)	4563.5(17)	82.5(9)
C18	6960(4)	-1439(3)	3387.4(17)	86.9(9)
C5	93(4)	-2769(3)	864(2)	82.2(7)

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

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	53.8(8)	61.8(8)	54.5(9)	20.1(7)	-0.9(6)	-8.5(6)
N1	42.5(8)	51.4(9)	57.5(10)	9.2(8)	0.0(7)	4.5(6)
C8	44.2(9)	43.6(9)	45.8(10)	9.4(8)	1.0(7)	0.5(7)
C10	44.5(8)	46.9(10)	54.3(11)	8.7(8)	8.8(8)	7.8(7)
C7	43.1(9)	43.9(9)	51.6(10)	9.2(8)	2.4(8)	3.0(7)
C2	60.1(9)	55.0(9)	58.5(9)	3.6(7)	8.8(8)	-1.8(8)
O2	42.7(7)	76.4(10)	69.2(10)	18.3(8)	-3.9(7)	0.7(6)
C9	56.1(11)	49.9(11)	67.1(13)	7.2(10)	15.3(10)	7.7(9)
C1	52.4(10)	53.3(11)	50.2(10)	7.7(9)	3.4(9)	-4.8(8)
O3	73.8(11)	64.3(10)	123.9(17)	-5.1(11)	12.7(12)	23.1(9)
C16	50.9(10)	58.4(12)	76.6(15)	15.4(11)	-0.6(11)	13.2(9)
C15	78.9(15)	57.4(13)	58.2(12)	1.7(11)	14.1(12)	6.4(11)
C11	62.6(12)	51.5(11)	67.0(13)	12.3(11)	14.7(11)	7.2(9)
C17	57.8(12)	89.5(18)	79.9(17)	32.3(16)	4.4(12)	-15.3(12)
C6	64.1(10)	73.2(10)	70.0(11)	4.7(8)	-4.9(9)	-7.7(8)
C12	83.2(16)	65.2(14)	79.2(17)	29.5(13)	29.9(15)	19.5(13)
C4	94.6(11)	87.0(12)	87.0(12)	-15.6(10)	-1.2(10)	-15.0(9)
C13	106(2)	95(2)	55.7(13)	22.7(14)	26.9(15)	39.1(18)
C3	84.6(10)	72.5(11)	79.6(12)	-11.0(9)	6.8(10)	-4.1(9)
C14	104(2)	88(2)	55.3(13)	-2.7(13)	15.3(14)	20.7(18)
C18	73.4(16)	116(3)	70.8(16)	22.4(17)	-15.9(14)	15.7(16)
C5	81.0(10)	84.3(11)	81.4(12)	-2.8(9)	-6.8(10)	-18.8(9)

Table 4 Bond Lengths for liyan-cxf-co-meoh.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C8	1.409(2)	C2	C1	1.400(3)
O1	C17	1.415(3)	C2	C3	1.386(4)
N1	C8	1.452(2)	O2	C18	1.421(3)
N1	O2	1.400(2)	C9	O3	1.226(3)
N1	C9	1.350(3)	C1	C6	1.392(3)
C8	C7	1.541(3)	C15	C14	1.382(4)
C10	C7	1.544(3)	C11	C12	1.390(3)

C10	C15	1.383(4)	C6	C5	1.389(4)
C10	C11	1.390(3)	C12	C13	1.376(5)
C7	C1	1.522(3)	C4	C3	1.394(5)
C7	C16	1.542(3)	C4	C5	1.362(5)
C2	C9	1.487(3)	C13	C14	1.376(5)

Table 5 Bond Angles for liyan-cxf-co-meoh.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C8	O1	C17	113.09(17)	C3	C2	C1	120.7(2)
O2	N1	C8	114.63(17)	N1	O2	C18	109.72(19)
C9	N1	C8	122.46(18)	N1	C9	C2	114.23(18)
C9	N1	O2	117.79(18)	O3	C9	N1	122.0(2)
O1	C8	N1	112.38(16)	O3	C9	C2	123.7(2)
O1	C8	C7	108.66(16)	C2	C1	C7	119.4(2)
N1	C8	C7	108.38(15)	C6	C1	C7	122.1(2)
C15	C10	C7	119.40(19)	C6	C1	C2	118.5(2)
C15	C10	C11	118.4(2)	C14	C15	C10	121.2(3)
C11	C10	C7	122.2(2)	C10	C11	C12	120.3(3)
C8	C7	C10	107.17(16)	C5	C6	C1	120.6(3)
C8	C7	C16	108.51(16)	C13	C12	C11	120.4(3)
C1	C7	C8	107.70(17)	C5	C4	C3	120.7(3)
C1	C7	C10	111.28(16)	C12	C13	C14	119.7(3)
C1	C7	C16	112.62(18)	C2	C3	C4	119.3(3)
C16	C7	C10	109.36(17)	C13	C14	C15	120.0(3)
C1	C2	C9	121.7(2)	C4	C5	C6	120.2(3)
C3	C2	C9	117.6(2)				

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for liyan-cxf-co-meoh.

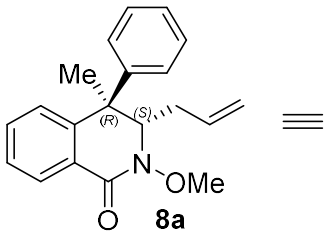
Atom	*x*	*y*	*z*	U(eq)
H8	4335.9	191.65	2580.9	53
H16A	1458.3	876.53	2377.64	93
H16B	-85.87	85.71	2272.87	93
H16C	1039.3	392.04	1529.33	93
H15	2653.13	356.95	3634.01	78
H11	1184.46	-2615.16	2965.68	72
H17A	5900.49	955.43	778.48	114

H17B	6748.55	52.33	1307.03	114
H17C	6071.27	1118.18	1709.4	114
H6	-585.93	-1440.57	1496.7	83
H12	930.74	-3100.09	4305.11	91
H4	1255.94	-4016.91	337.31	107
H13	1642.03	-1883.25	5302.46	103
H3	3924.72	-3710.12	890.03	95
H14	2449.56	-141.3	4964.55	99
H18A	6884.99	-2216.3	3386.06	130
H18B	6068.17	-1144.39	3714.91	130
H18C	8041.27	-1221.9	3600.71	130
H5	-974.05	-2905.28	646.81	99

Crystal structure determination of [liyan-cxf-co-meoh]

Crystal Data for $C_{18}H_{19}NO_3$ (M =297.34 g/mol): orthorhombic, space group $P2_12_12_1$ (no. 19), a = 7.87176(11) Å, b = 12.32287(17) Å, c = 16.6389(3) Å, V = 1614.02(4) Å^3, Z = 4, T = 293(2) K, μ(CuKα) = 0.673 mm^{-1}, $Dcalc$ = 1.224 g/cm^3, 12148 reflections measured (8.93° ≤ 2Θ ≤ 147.734°), 3210 unique (R_{int} = 0.0280, R_{sigma} = 0.0186) which were used in all calculations. The final R_1 was 0.0436 (I > 2σ(I)) and wR_2 was 0.1331 (all data).

X-ray crystal data of the enantiomerically enriched isomer 8a:



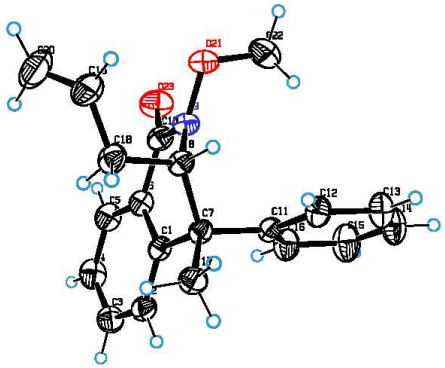


Table 1 Crystal data and structure refinement for liyan-cxf-co-allyl.

Identification code	liyan-cxf-co-allyl
Empirical formula	$C_{20}H_{21}NO_2$
Formula weight	307.38
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	7.84850(10)
b/Å	12.7347(2)

c/Å	9.08320(10)
α/°	90
β/°	90.4380(10)
γ/°	90
Volume/Å^3	907.82(2)
Z	2
ρ_{calc}g/cm^3	1.124
μ/mm^{-1}	0.571
F(000)	328.0
Crystal size/mm^3	0.3 × 0.2 × 0.1
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	11.274 to 148.064
Index ranges	-9 ≤ h ≤ 9, -15 ≤ k ≤ 13, -11 ≤ l ≤ 11
Reflections collected	8523
Independent reflections	3231 [R_{int} = 0.0204, R_{sigma} = 0.0151]
Data/restraints/parameters	3231/1/218
Goodness-of-fit on F^2	1.026
Final R indexes [I>=2σ (I)]	R_1 = 0.0388, wR_2 = 0.1096
Final R indexes [all data]	R_1 = 0.0390, wR_2 = 0.1103
Largest diff. peak/hole / e Å^{-3}	0.14/-0.24
Flack parameter	0.15(8)

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

Atom	*x*	*y*	*z*	U(eq)
O21	3143.9(13)	5628.3(12)	4754.8(14)	56.3(3)
O23	3936.5(19)	3725.6(12)	5835(2)	69.6(4)
N9	4764.0(14)	5381.2(11)	5307.6(14)	44.9(3)
C11	7858.3(16)	5694.1(13)	3633.3(15)	41.3(3)
C12	7499(2)	6483.0(15)	2613.8(17)	50.2(4)
C10	5005.4(19)	4424.4(14)	5882.8(17)	47.7(3)
C6	6766(2)	4261.6(13)	6505.8(16)	45.2(3)
C1	8085.0(18)	4968.6(13)	6209.4(15)	41.6(3)
C7	7720.1(16)	5941.6(11)	5287.4(15)	39.0(3)

C8	5860.9(18)	6291.2(12)	5603.3(16)	41.6(3)
C2	9713(2)	4731.1(15)	6732.1(17)	49.6(4)
C18	5606(2)	6721.5(15)	7171.5(19)	53.9(4)
C5	7063(3)	3359.5(15)	7330(2)	57.2(4)
C3	9999(2)	3820.8(17)	7548.7(19)	59.7(4)
C4	8685(3)	3143.9(18)	7865(2)	63.6(5)
C17	8947.0(19)	6851.5(15)	5662.8(18)	49.9(4)
C16	8343(3)	4715.5(18)	3115(2)	60.0(4)
C13	7628(3)	6296(2)	1112.9(19)	64.9(5)
C19	3778(3)	6856(3)	7571(3)	79.2(7)
C14	8105(4)	5328(2)	607(2)	76.1(6)
C15	8464(4)	4530(2)	1609(2)	78.4(7)
C22	3019(3)	5322(3)	3250(2)	75.7(6)
C20	3052(5)	6396(4)	8679(4)	115.4(15)

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

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O21	37.9(5)	63.7(8)	67.3(7)	-0.5(6)	-1.4(4)	1.7(5)
O23	60.4(7)	50.3(8)	98.0(10)	7.8(7)	-1.2(7)	-17.3(6)
N9	35.6(5)	45.8(8)	53.3(6)	-1.0(5)	1.1(4)	-2.4(5)
C11	41.1(6)	42.8(8)	40.0(6)	-3.0(5)	5.5(5)	-4.9(5)
C12	61.9(8)	44.2(8)	44.6(7)	0.4(6)	6.4(6)	-3.5(7)
C10	44.1(7)	45.4(8)	53.7(7)	0.2(7)	5.9(6)	-5.9(6)
C6	49.0(7)	39.4(8)	47.1(7)	0.8(6)	4.6(6)	0.9(6)
C1	43.2(6)	42.3(8)	39.5(6)	-3.0(5)	5.5(5)	1.6(5)
C7	39.7(6)	37.1(8)	40.3(6)	-2.8(5)	4.5(4)	-3.4(5)
C8	42.3(6)	37.7(8)	44.9(6)	-3.3(6)	4.9(5)	-0.7(5)
C2	44.6(7)	54.4(10)	49.7(7)	0.4(7)	4.6(5)	6.2(6)
C18	57.0(8)	52.3(10)	52.6(8)	-13.4(7)	7.6(6)	2.0(7)
C5	67.0(9)	44.0(10)	60.5(9)	7.9(8)	6.1(7)	-0.4(7)
C3	58.8(8)	62.4(11)	58.0(8)	0.0(8)	-0.8(7)	19.4(8)
C4	79.0(11)	49.6(10)	62.3(10)	8.8(8)	0.6(8)	15.1(9)
C17	49.1(7)	47.9(9)	52.7(7)	-5.3(6)	-0.8(6)	-9.9(6)
C16	77.0(10)	51.2(10)	52.0(8)	-5.4(7)	7.1(7)	8.9(8)
C13	86.9(12)	66.2(13)	41.7(8)	1.7(8)	2.4(7)	-3.7(10)

C19	66.2(11)	100(2)	71.2(12)	-23.6(12)	12.7(9)	21.3(11)
C14	105.1(16)	82.2(16)	41.0(7)	-10.4(9)	4.2(9)	4.1(13)
C15	114.8(18)	64.5(14)	56.0(10)	-16.9(9)	8.4(10)	12.0(13)
C22	67.2(11)	90.4(17)	69.2(11)	-5.9(11)	-18.3(9)	-4.3(10)
C20	89.0(18)	175(4)	83.2(16)	-35(2)	38.2(15)	-22(2)

Table 4 Bond Lengths for liyan-cxf-co-allyl.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O21	N9	1.3993(16)	C1	C2	1.393(2)
O21	C22	1.424(3)	C7	C8	1.5544(18)
O23	C10	1.224(2)	C7	C17	1.543(2)
N9	C10	1.339(2)	C8	C18	1.541(2)
N9	C8	1.467(2)	C2	C3	1.394(3)
C11	C12	1.394(2)	C18	C19	1.493(3)
C11	C7	1.5398(18)	C5	C4	1.387(3)
C11	C16	1.386(3)	C3	C4	1.376(3)
C12	C13	1.388(2)	C16	C15	1.392(3)
C10	C6	1.504(2)	C13	C14	1.368(4)
C6	C1	1.399(2)	C19	C20	1.299(5)
C6	C5	1.390(2)	C14	C15	1.391(4)
C1	C7	1.522(2)			

Table 5 Bond Angles for liyan-cxf-co-allyl.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N9	O21	C22	109.78(13)	C11	C7	C17	108.71(11)
O21	N9	C8	114.74(13)	C1	C7	C11	110.86(12)
C10	N9	O21	118.04(13)	C1	C7	C8	107.74(11)
C10	N9	C8	124.50(12)	C1	C7	C17	112.04(13)
C12	C11	C7	119.04(14)	C17	C7	C8	109.22(12)
C16	C11	C12	118.48(14)	N9	C8	C7	106.87(12)
C16	C11	C7	122.48(14)	N9	C8	C18	111.71(12)
C13	C12	C11	120.85(17)	C18	C8	C7	113.66(12)
O23	C10	N9	123.60(15)	C1	C2	C3	120.41(16)
O23	C10	C6	122.74(17)	C19	C18	C8	113.43(15)
N9	C10	C6	113.54(13)	C4	C5	C6	120.14(17)
C1	C6	C10	121.18(14)	C4	C3	C2	120.92(16)
C5	C6	C10	117.80(15)	C3	C4	C5	119.36(18)

C5	C6	C1	120.95(15)	C11	C16	C15	120.48(19)
C6	C1	C7	119.57(13)	C14	C13	C12	120.42(19)
C2	C1	C6	118.19(15)	C20	C19	C18	124.4(3)
C2	C1	C7	122.20(14)	C13	C14	C15	119.54(17)
C11	C7	C8	108.18(11)	C14	C15	C16	120.2(2)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for liyan-cxf-co-allyl.

Atom	*x*	*y*	*z*	U(eq)
H12	7169.12	7143.33	2943.05	60
H8	5556.49	6846.62	4902.29	50
H2	10613.55	5182.77	6535.02	59
H18A	6139.58	6245.54	7869.63	65
H18B	6176.29	7394.82	7255.19	65
H5	6172	2899.47	7522.37	69
H3	11094.11	3668.62	7884.55	72
H4	8882.74	2547.65	8432.33	76
H17A	10058.73	6695.3	5287.54	75
H17B	9013.3	6936.8	6711.78	75
H17C	8532.28	7488.34	5222.64	75
H16	8587.87	4178.96	3777.79	72
H13	7388.02	6831.36	446.94	78
H19	3113.63	7298.03	6988.51	95
H14	8187.65	5204.43	-398.51	91
H15	8787.39	3870.86	1271.06	94
H22A	3917.7	5646.94	2703.05	114
H22B	1937.13	5539.48	2855.85	114
H22C	3117.85	4572.19	3178	114
H20A	1990(80)	6590(50)	9060(60)	170(20)
H20B	3830(60)	5740(50)	9270(50)	139(16)

Crystal structure determination of [liyan-cxf-co-allyl]

Crystal Data for $C_{20}H_{21}NO_2$ (M =307.38 g/mol): monoclinic, space group $P2_1$ (no. 4), a = 7.84850(10) Å, b = 12.7347(2) Å, c = 9.08320(10) Å, β = 90.4380(10)°, V = 907.82(2) Å^3, Z = 2,T = 293(2) K, μ(CuKα) = 0.571 mm^{-1}, *Dcalc* = 1.124 g/cm^3, 8523 reflections measured (11.274° ≤ 2Θ ≤ 148.064°), 3231 unique (R_{int} = 0.0204, R_{sigma} = 0.0151) which were used in all calculations. The final R_1 was 0.0388 (I > 2σ(I)) and wR_2 was 0.1103 (all data).

References

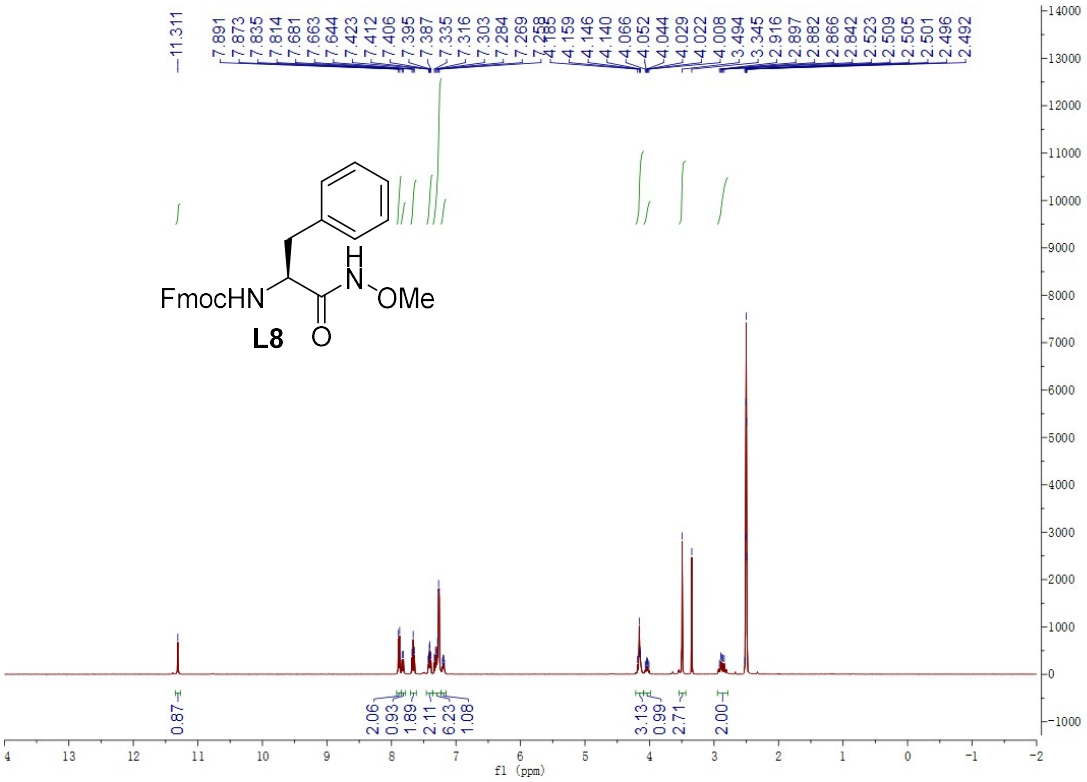
[1] X.-F. Cheng, F. Fei, Y. Li, Y.-M. Hou, X. Zhou and X.-S. Wang, *Org. Lett*. 2020, DOI: 10.1021/acs.orglett.0c02216.

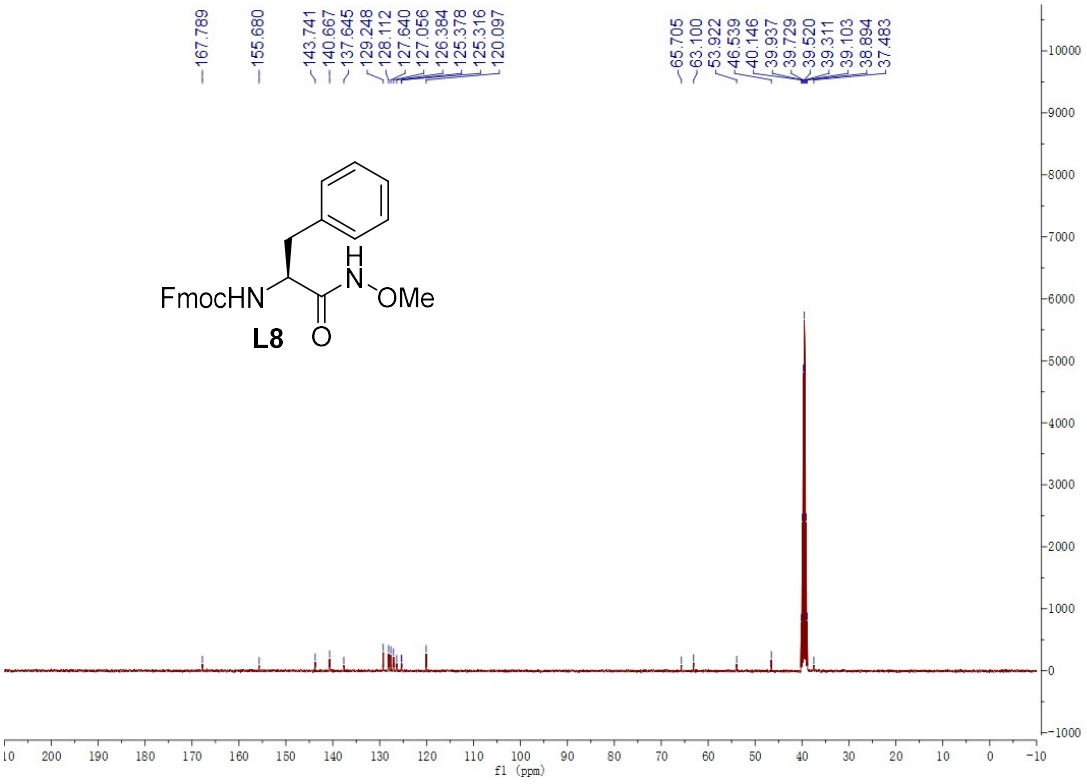
[2] M. A. Kroc, A. Patil, A. Carlos, J. Ballantine, S. Aguilar, D.-L. Mo, H.-Y. Wang, D. S. Mueller, D. J. Wink and L. L. Anderson, *Tetrahedron* 2017, **73**, 4125.

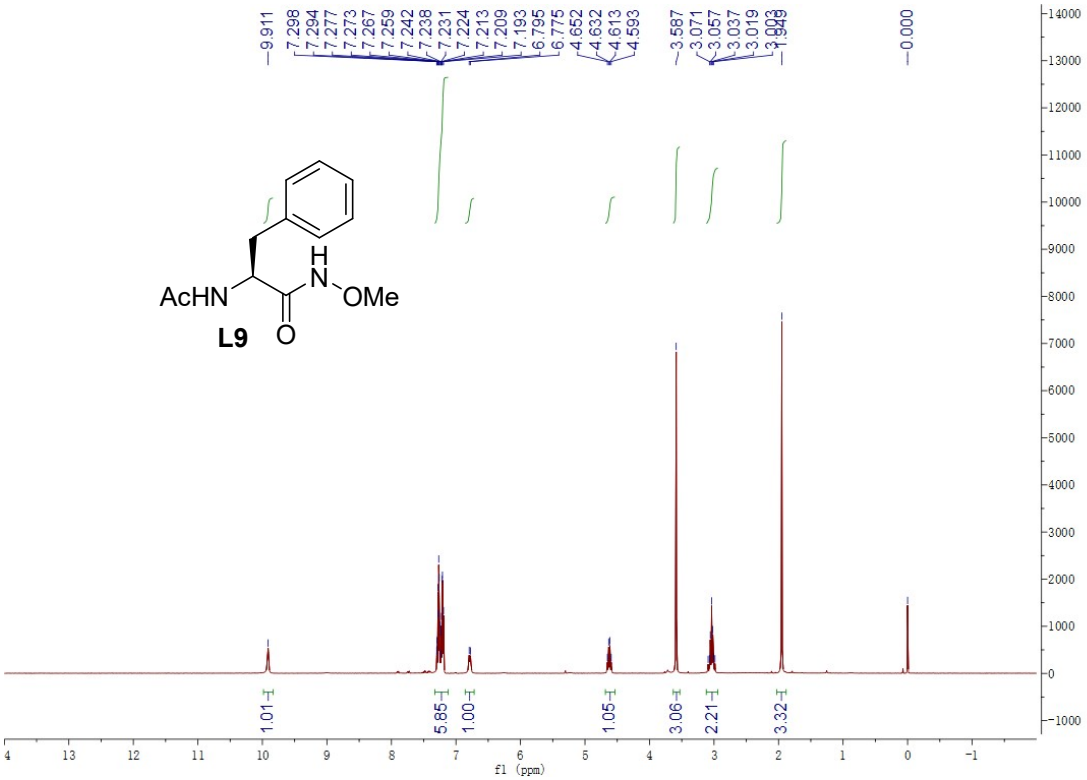
[3] D. J. Wardrop, E. G. Bowen, R. E. Forslund, A. D. Sussman and S. L. Weerasekera, *J. Am. Chem. Soc.* 2010, **132**, 1188.

[4] Z.-Q. He, Q. Zhou, L. Wu and Y.-C. Chen, *Adv. Synth. Catal.* 2010, **352**, 1904.

^{1}H and ^{13}C Spectra



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6.775
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3.037
3.019
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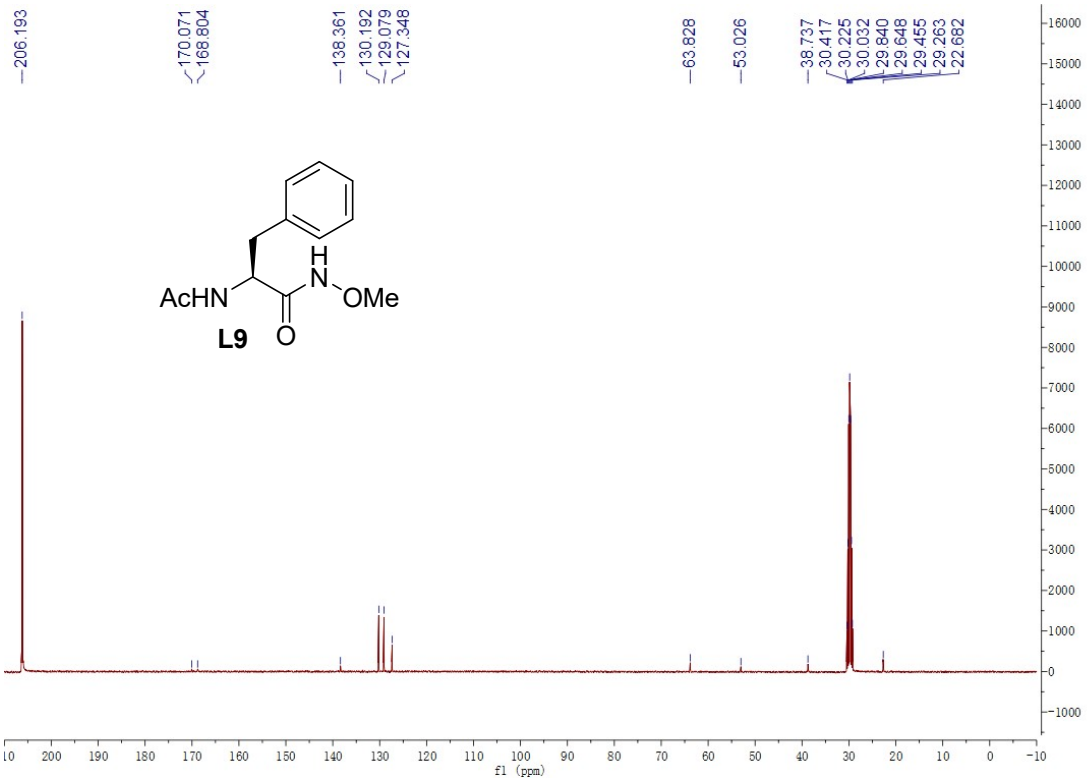
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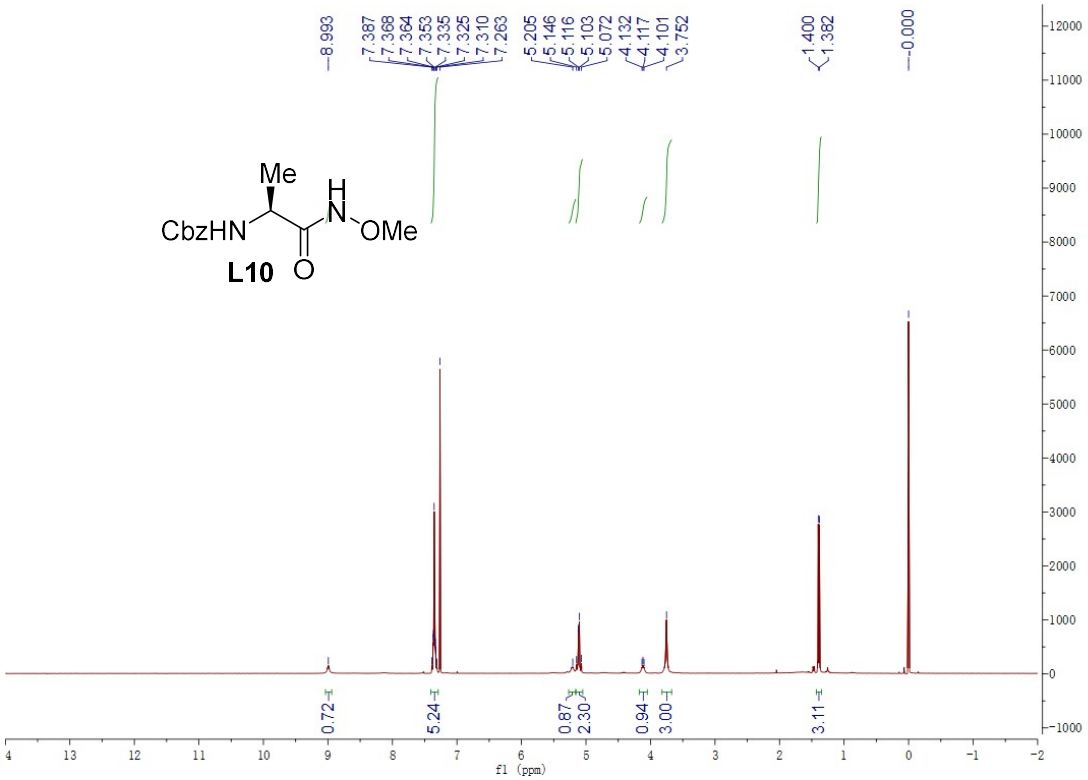
f1 (ppm)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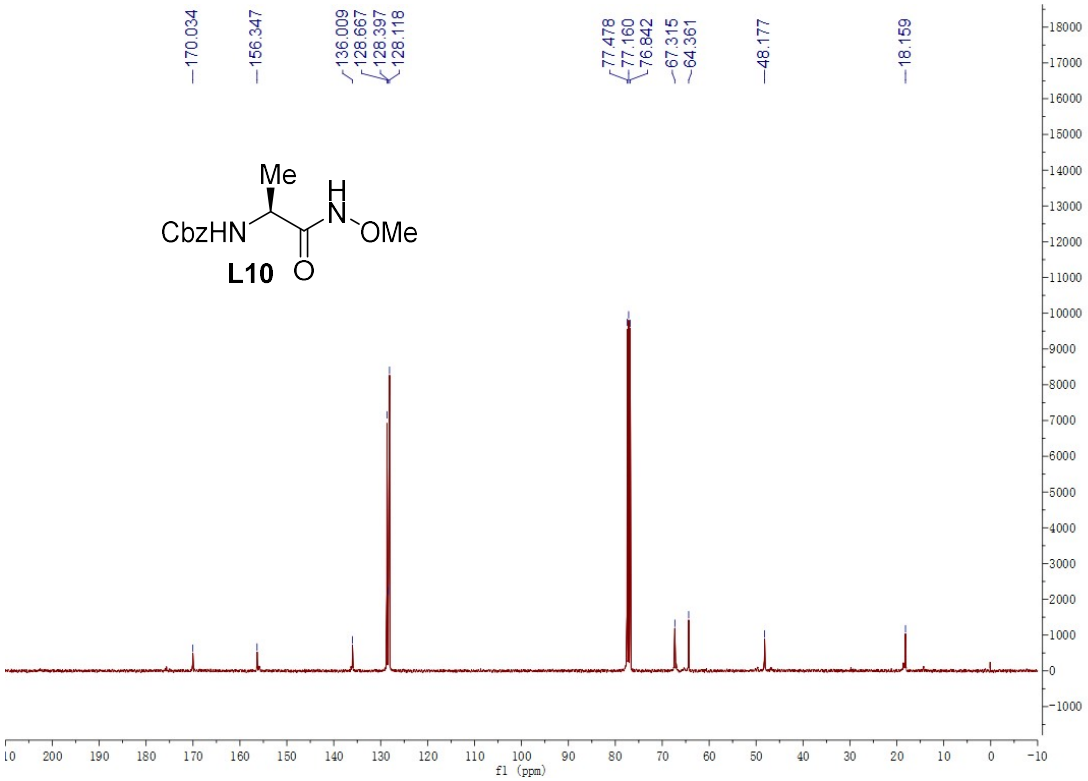
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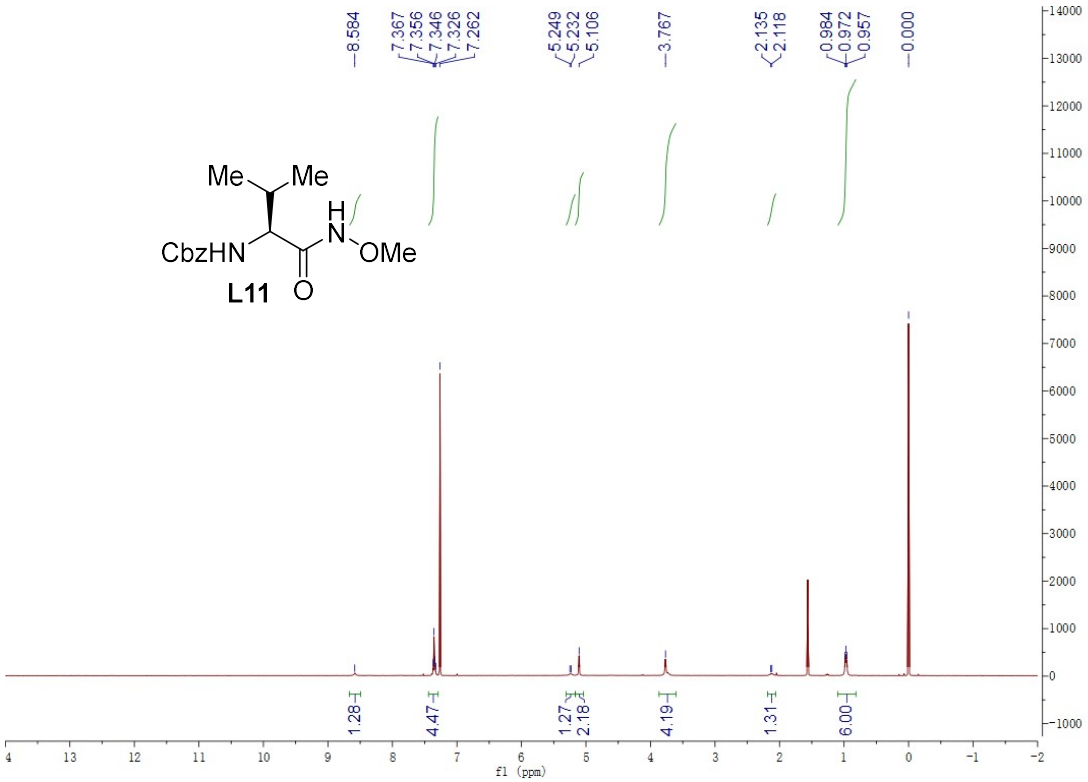
OMe
O
L10
f1 (ppm)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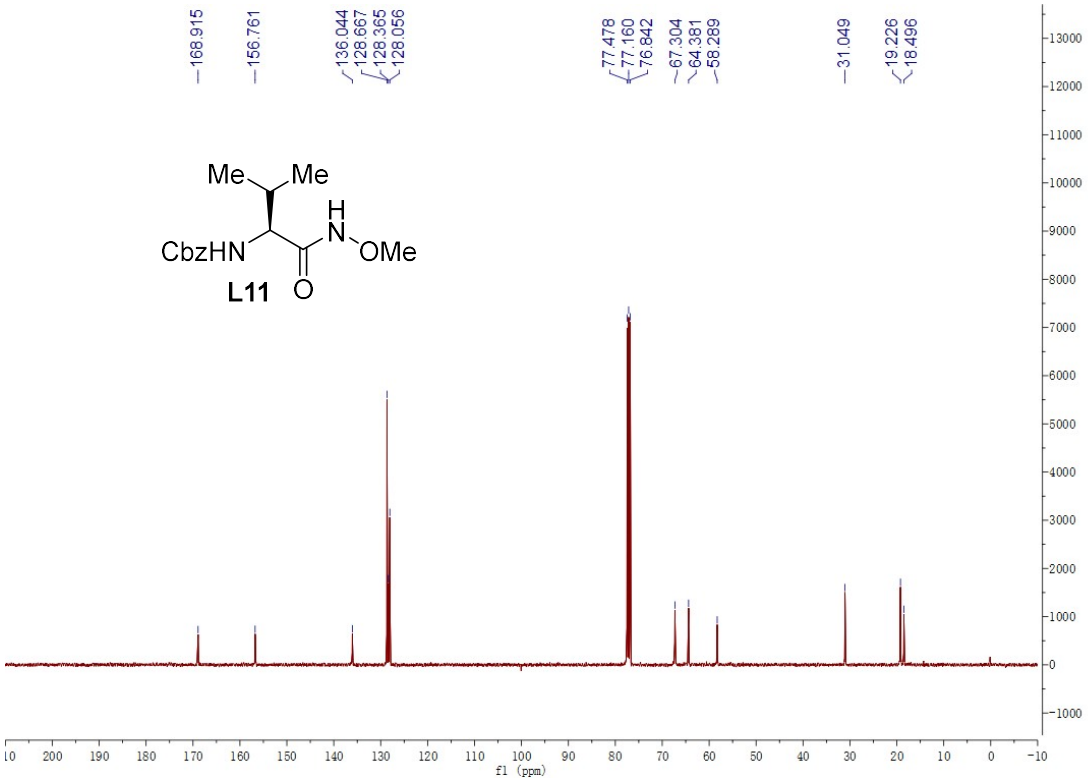
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O
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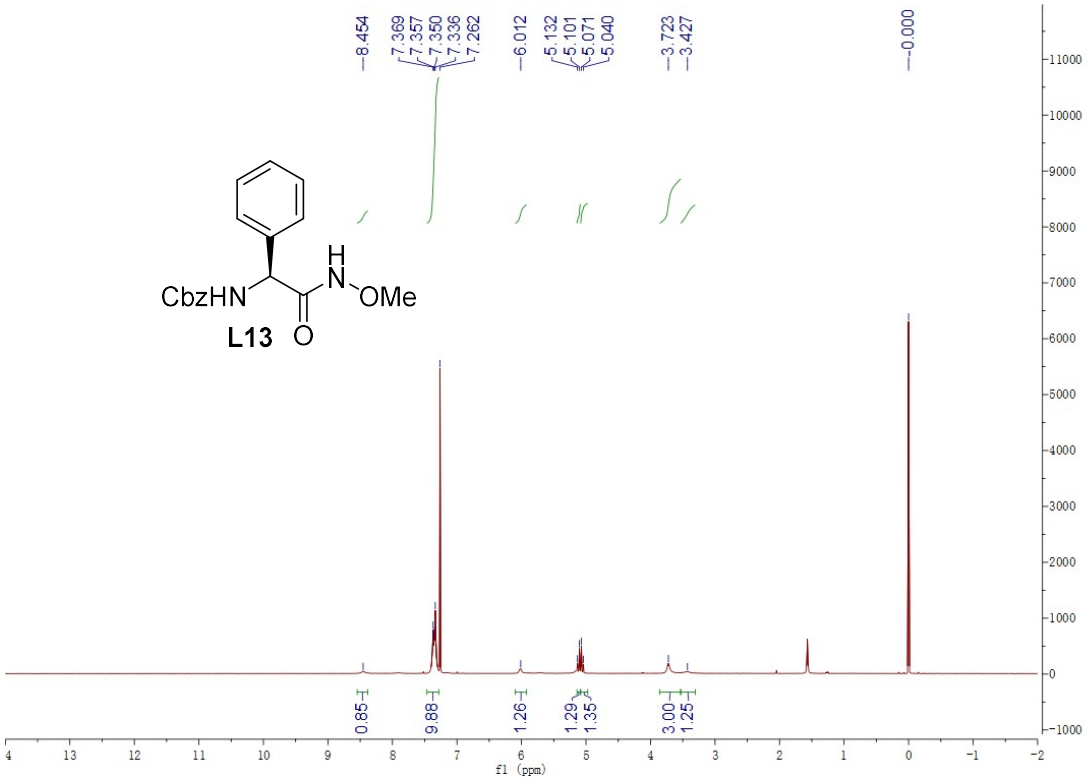
f1 (ppm)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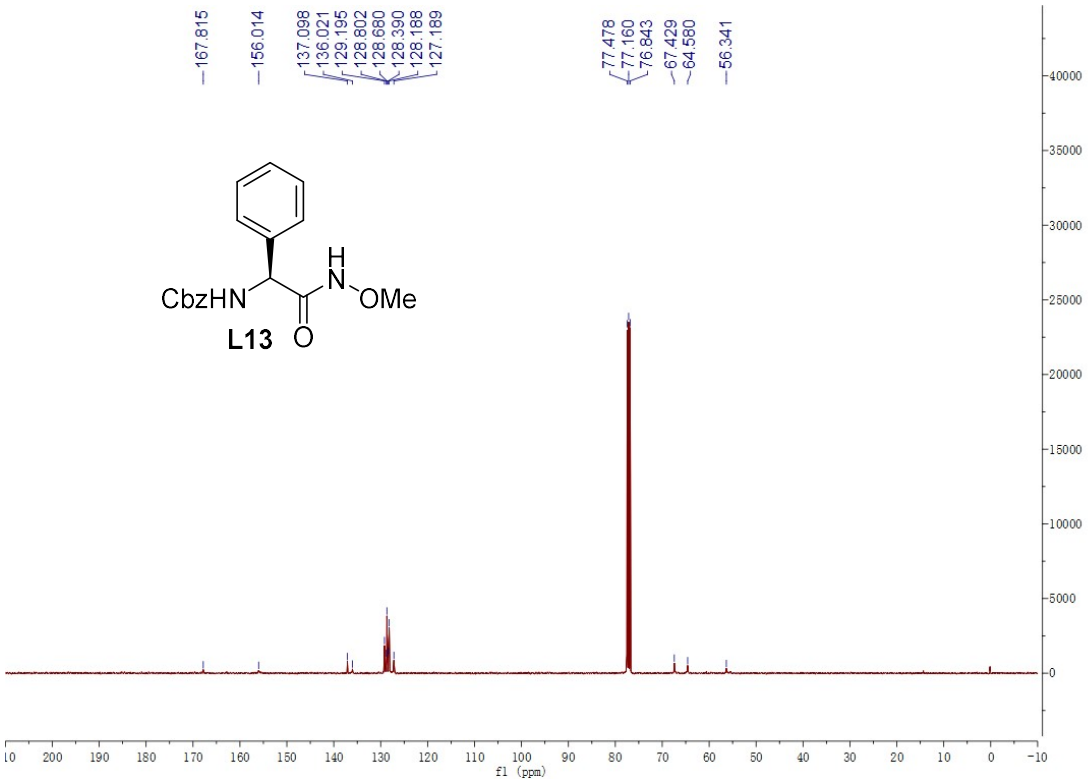
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O
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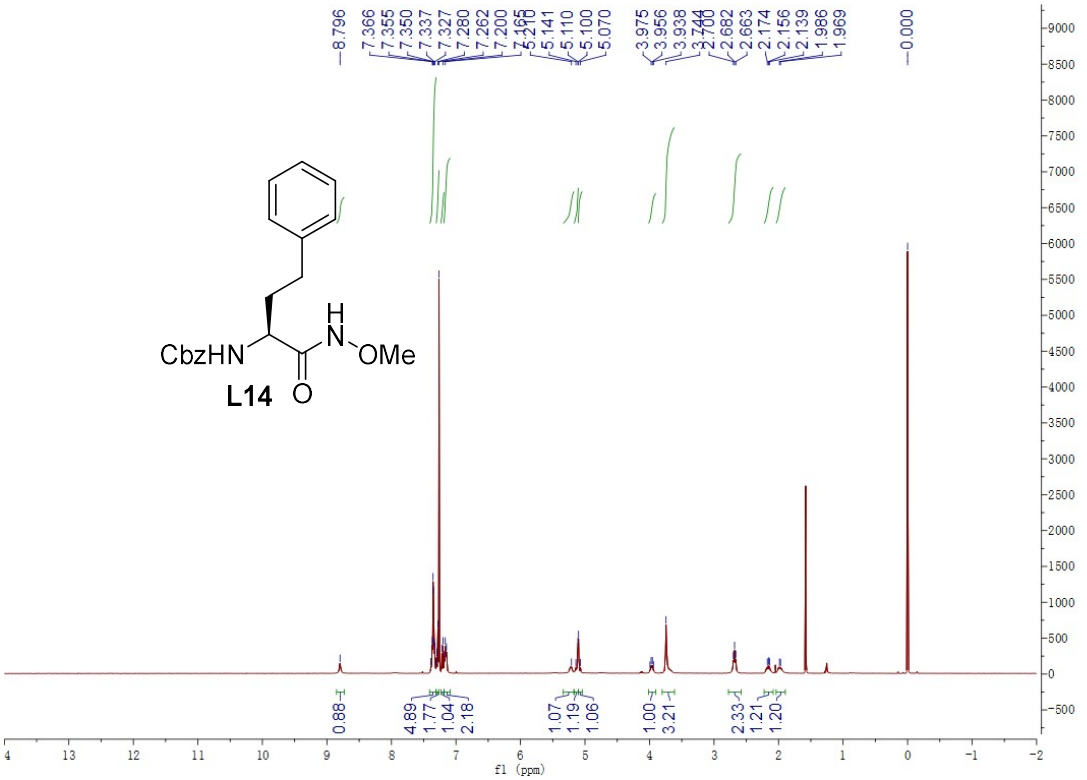
f1 (ppm)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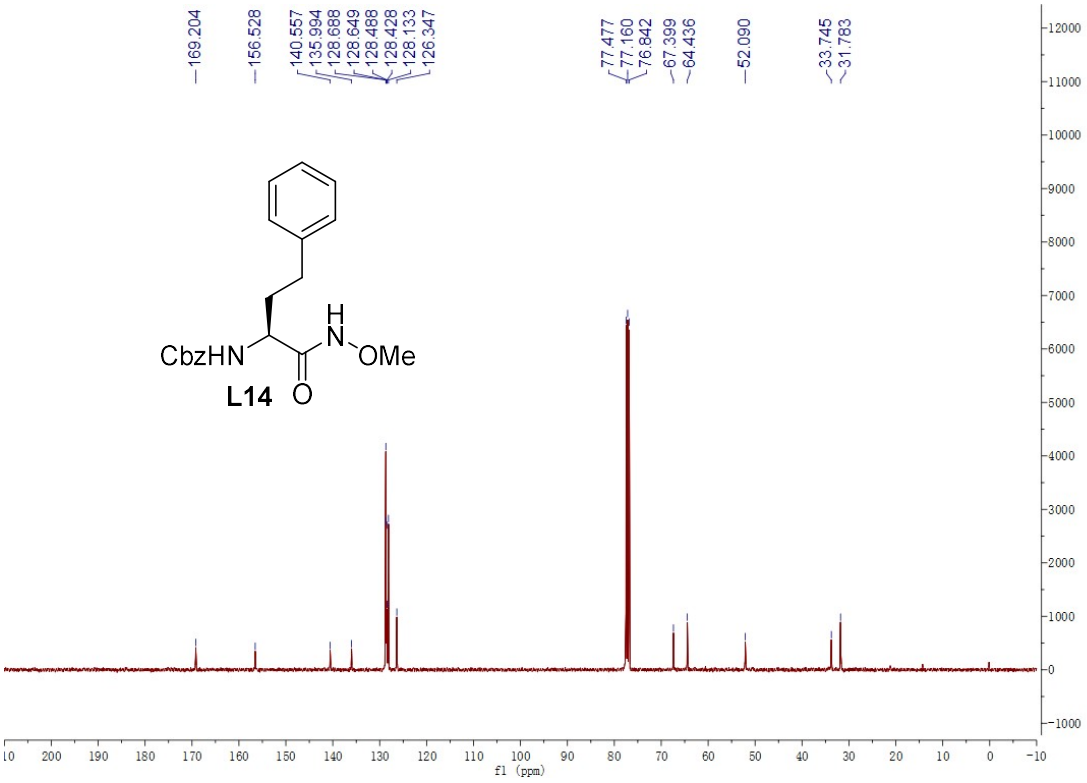
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N
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O
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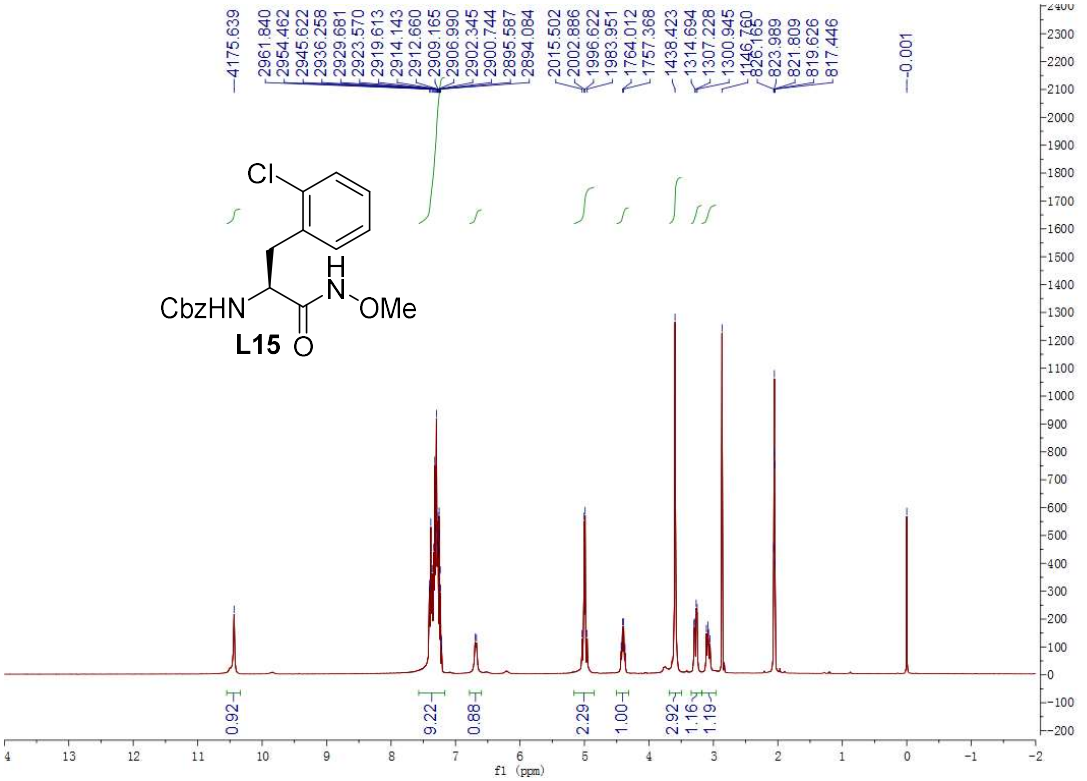
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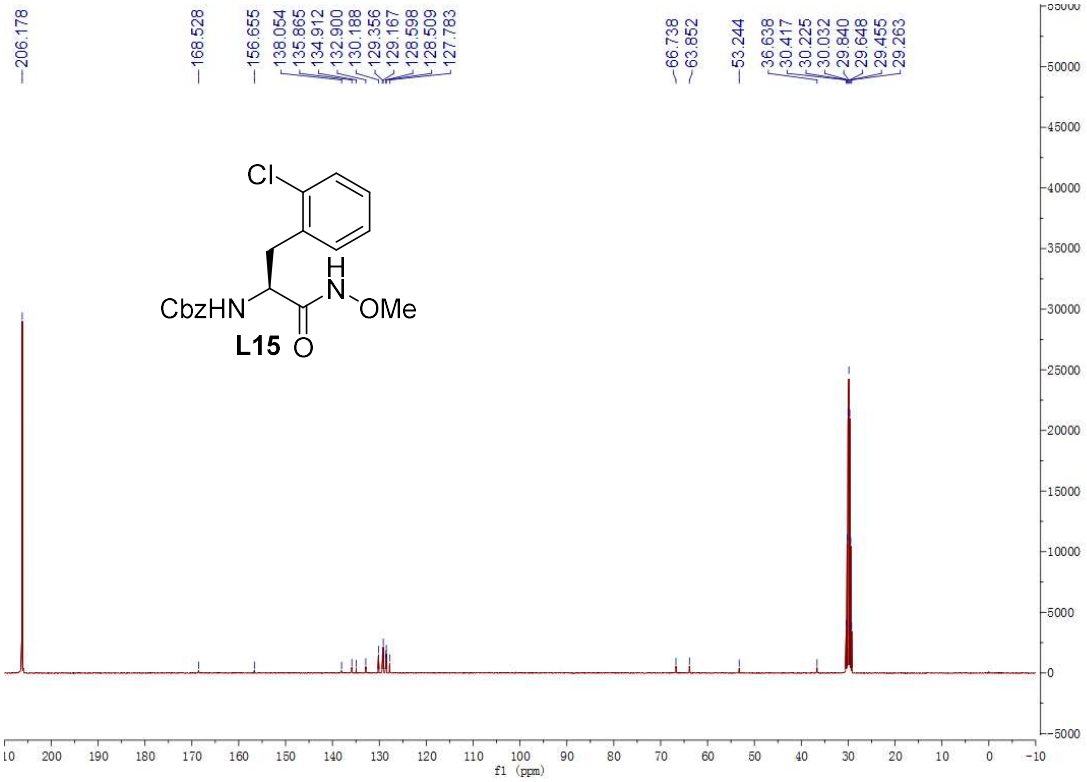
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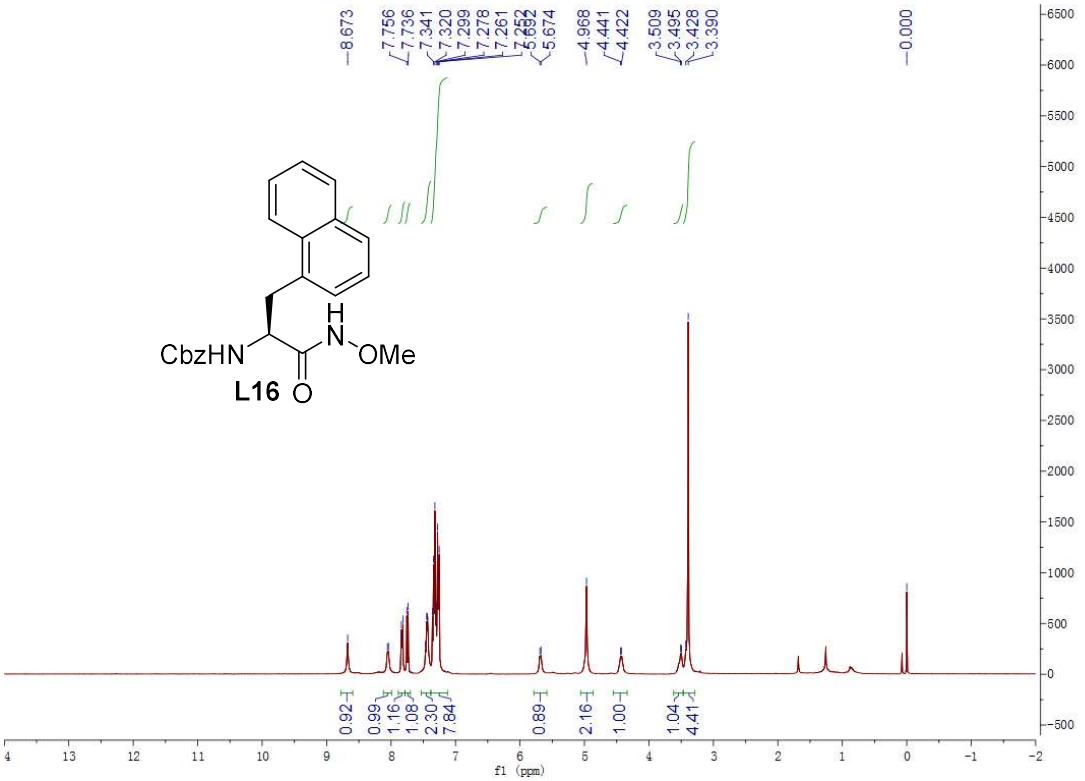
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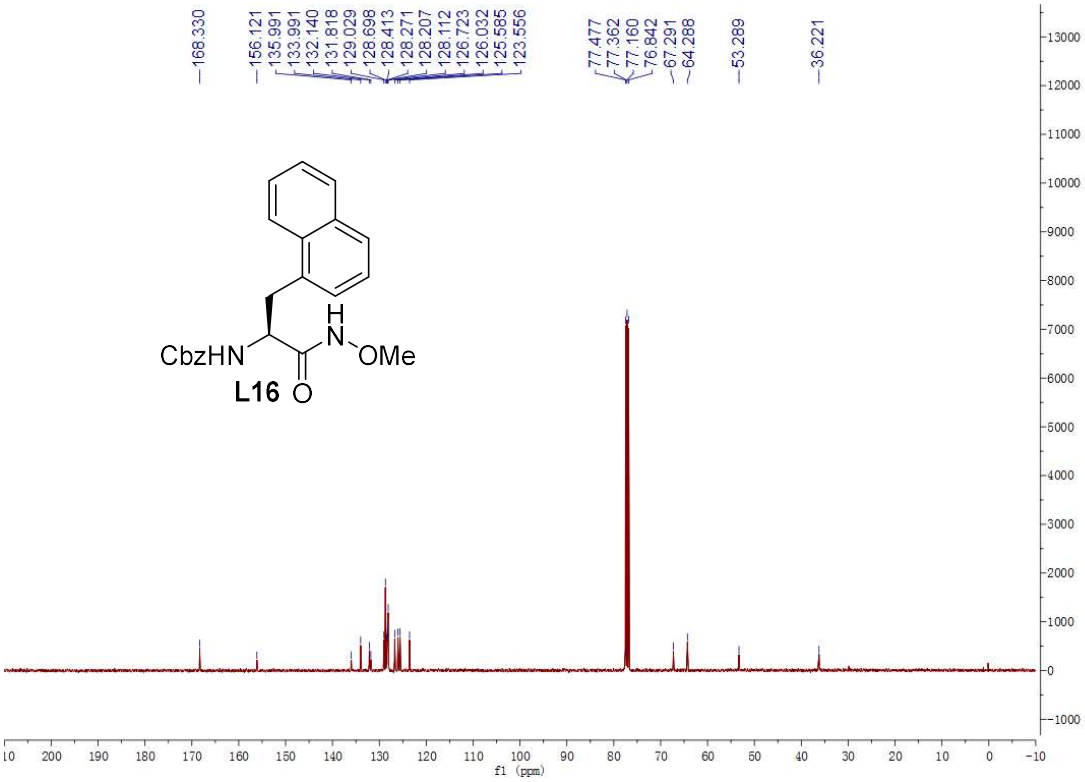
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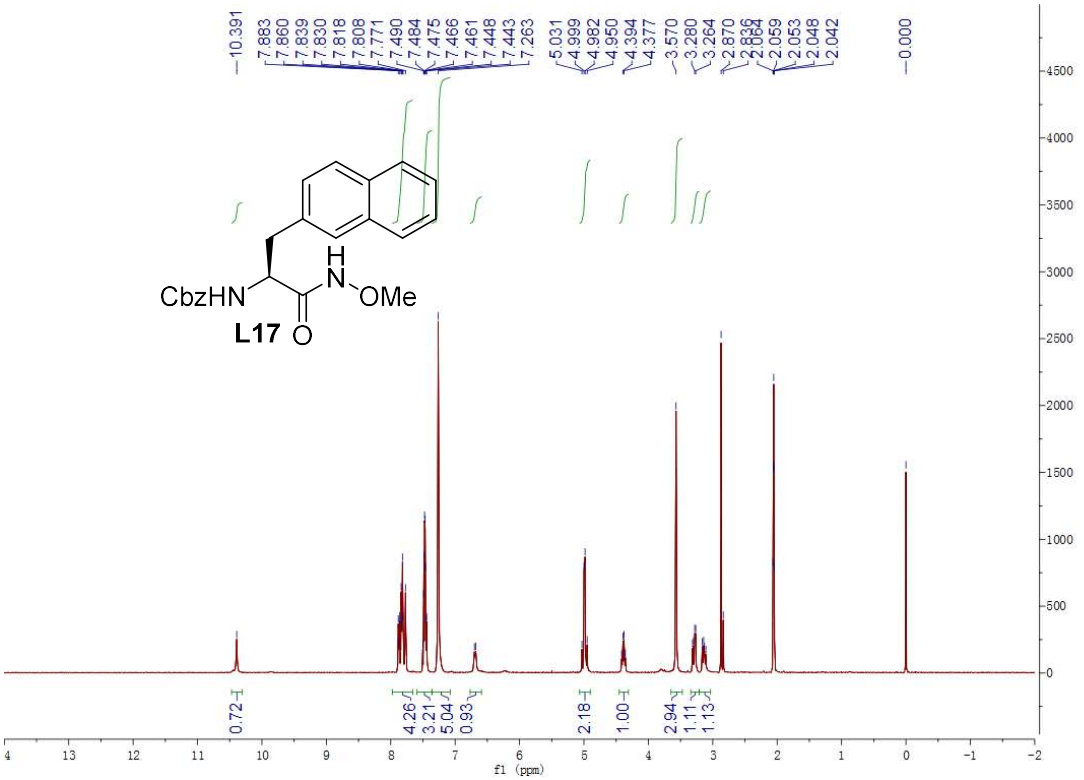
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L17
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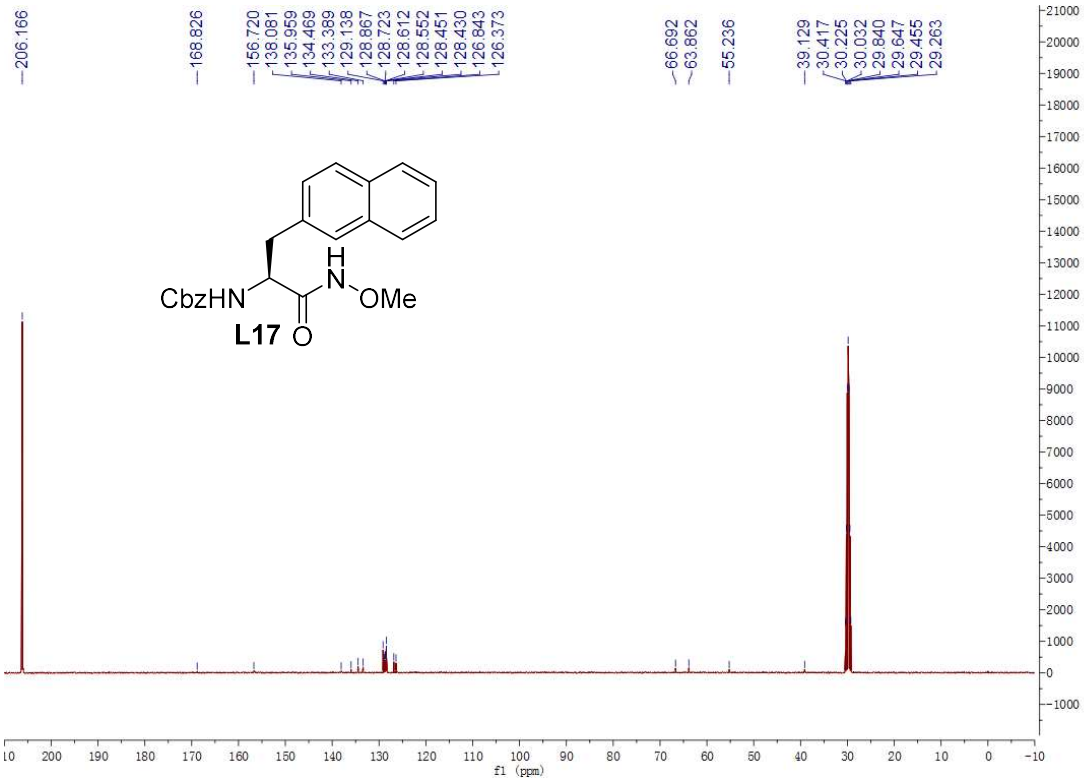
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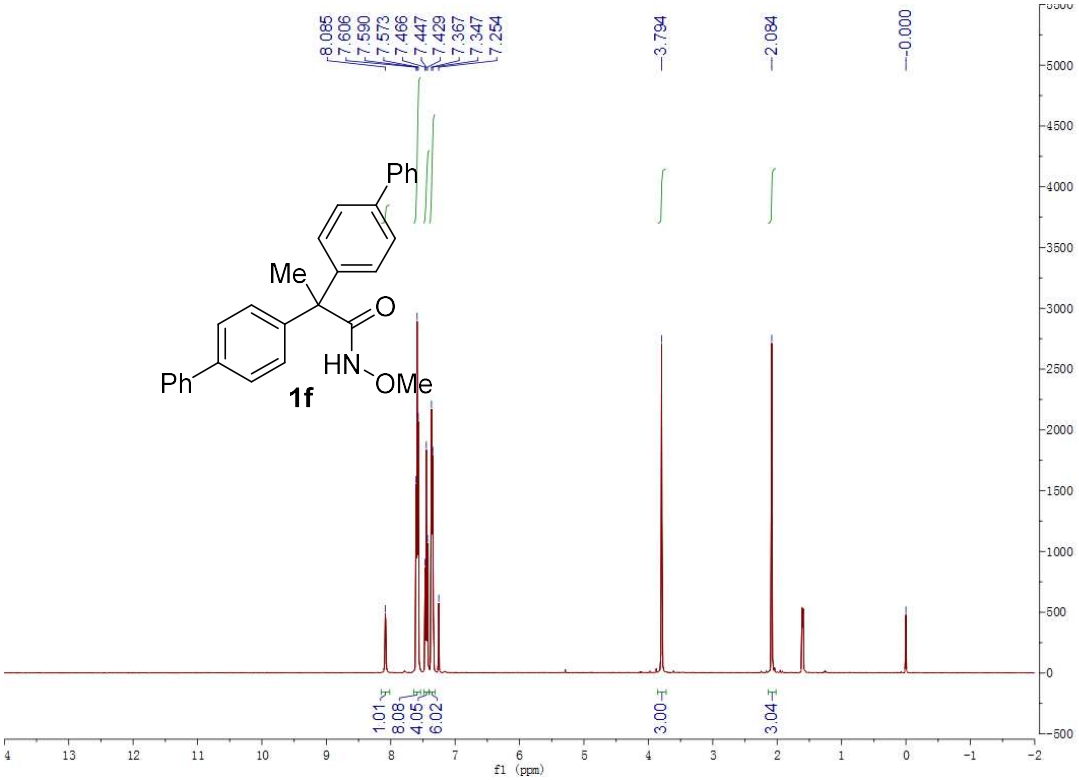
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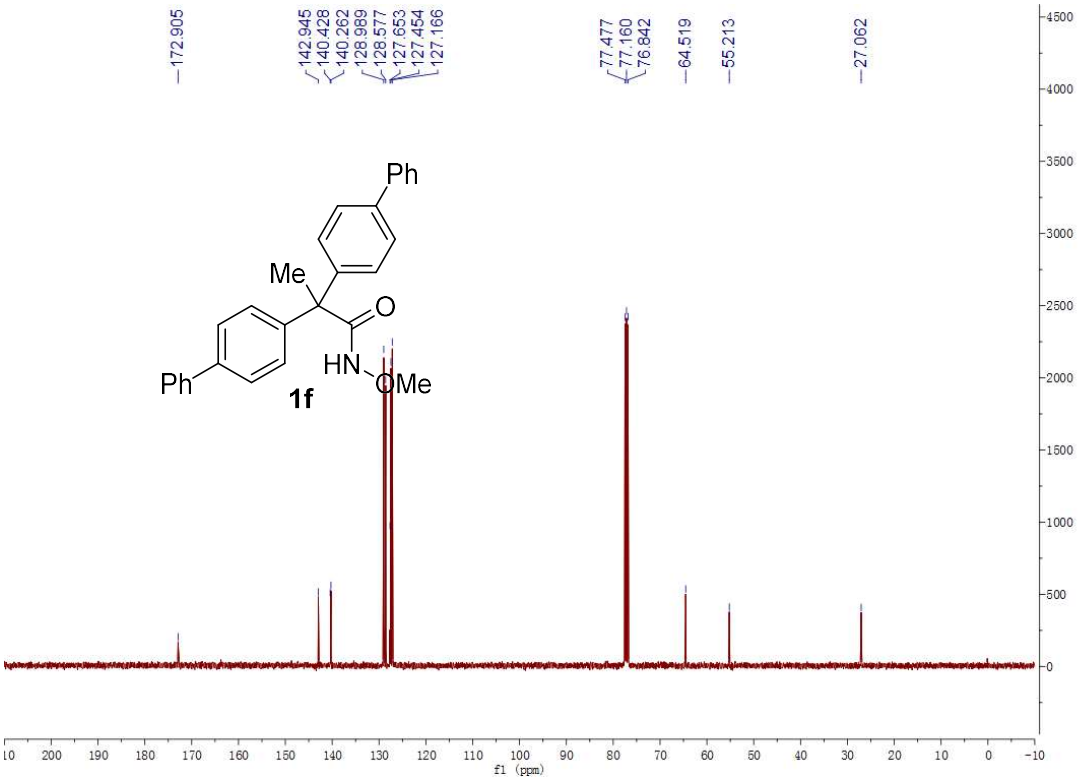
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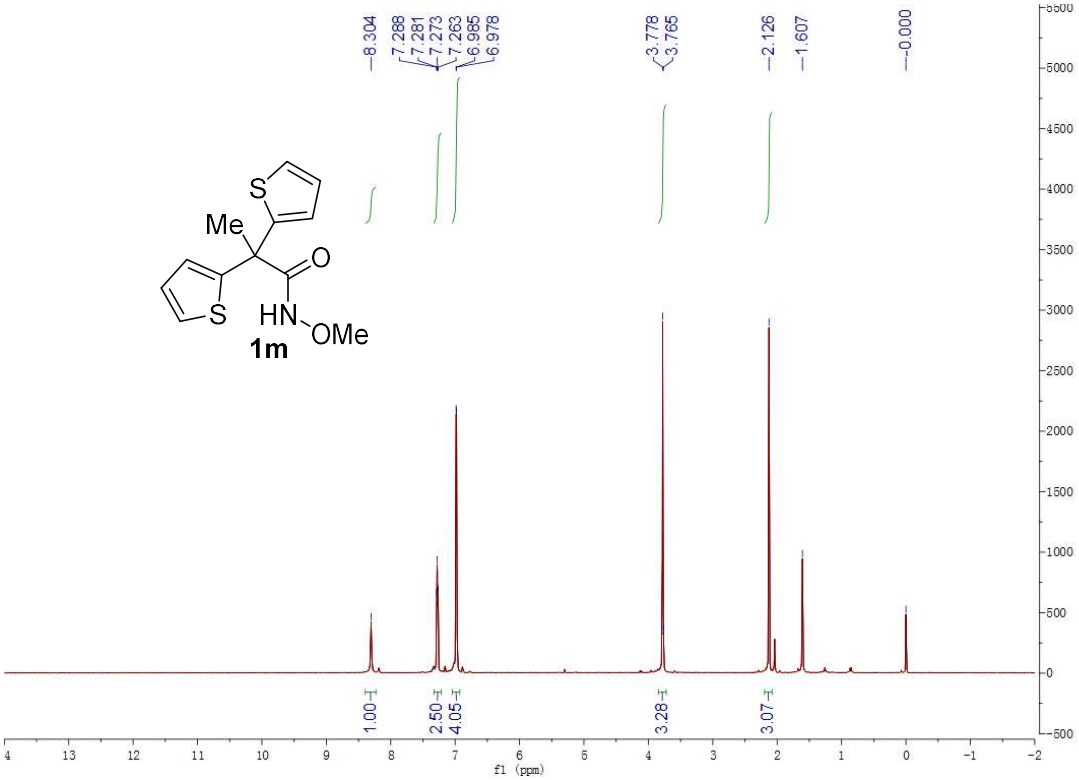
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Me
S
O
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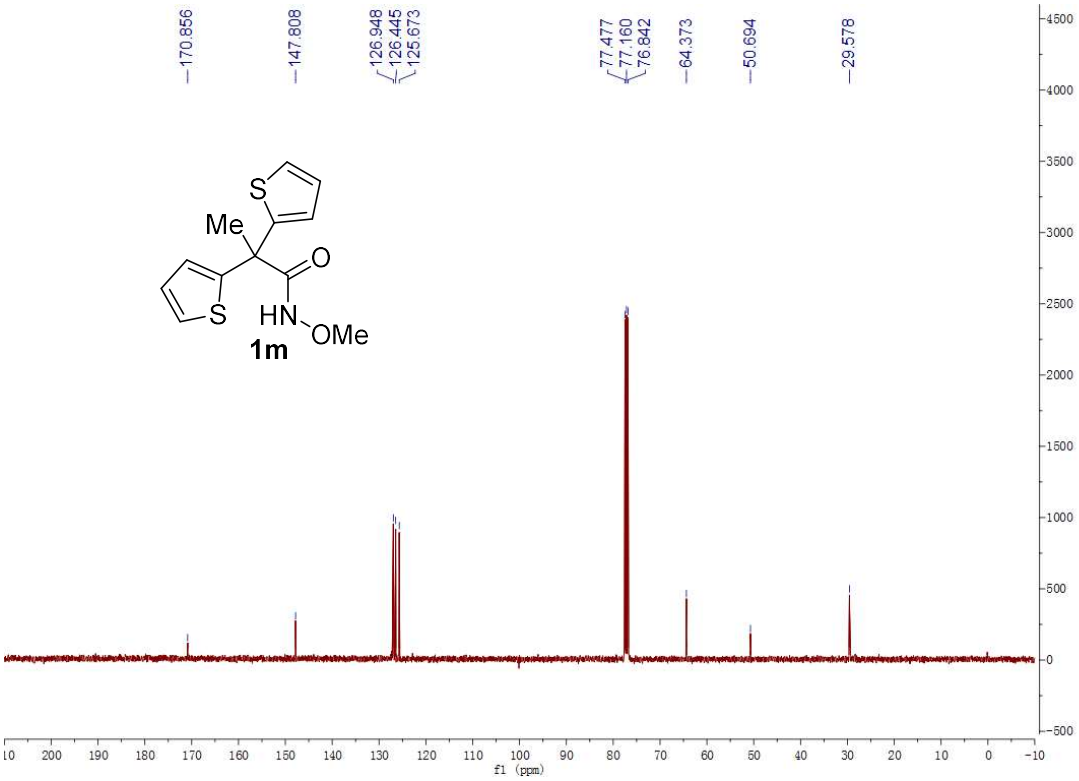
OMe
1m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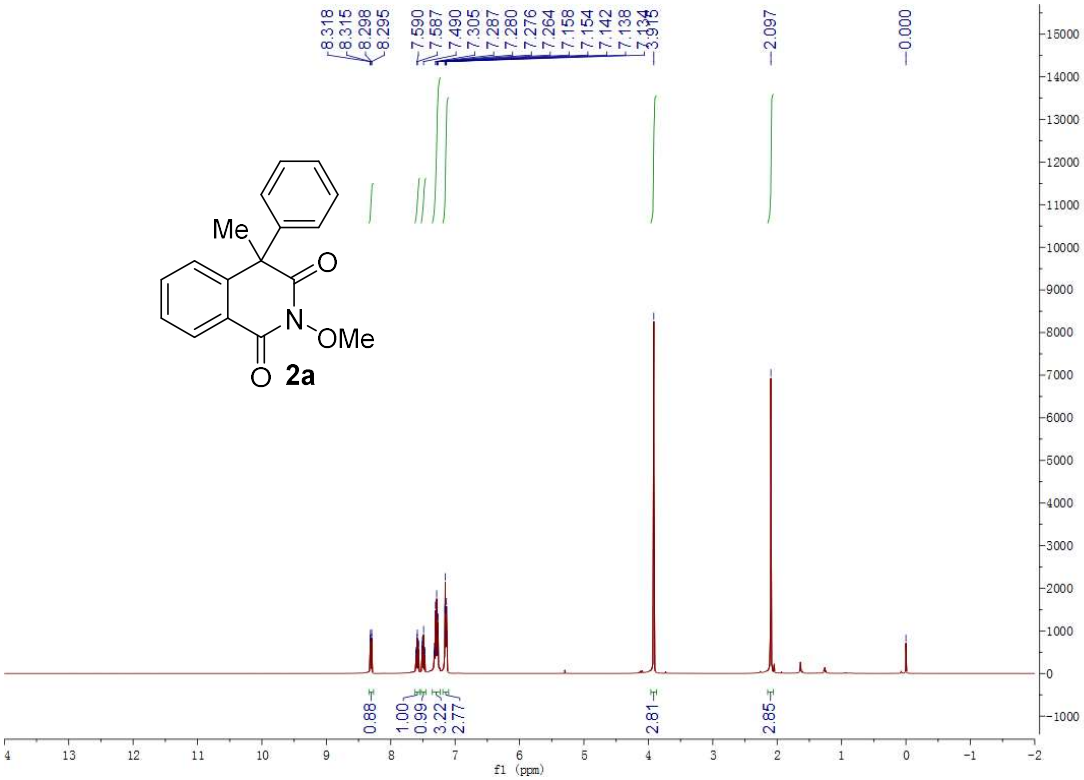
1m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8.295
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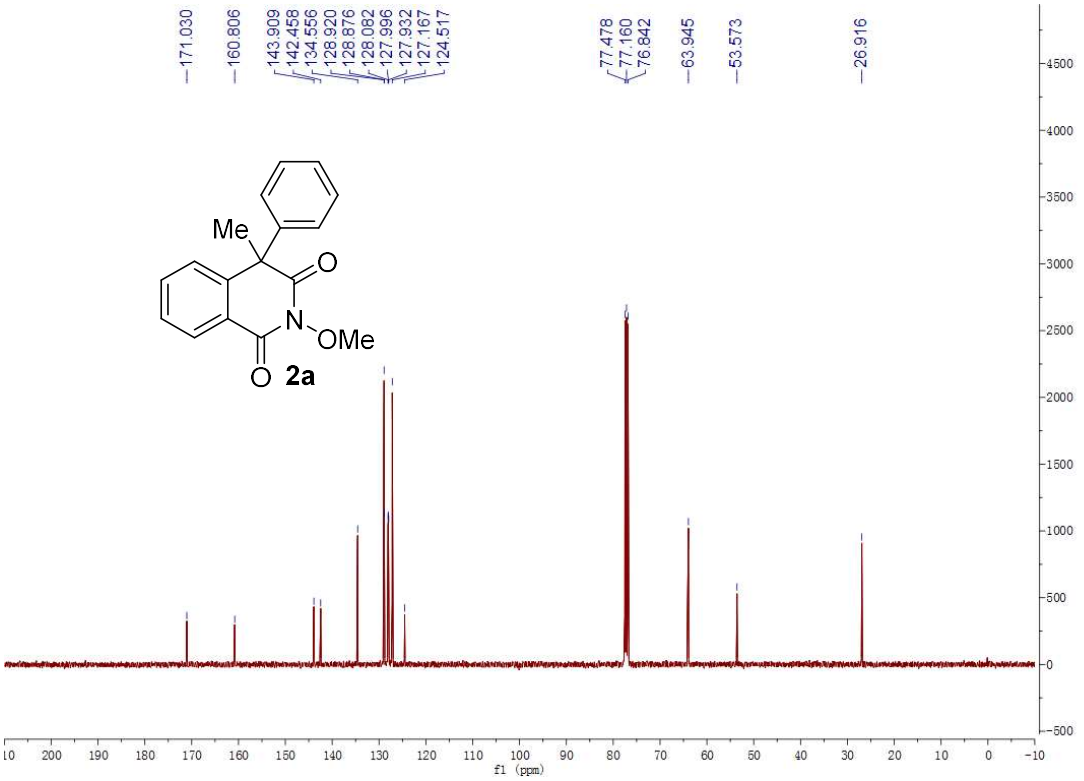
7.587
7.490
7.305
7.287
7.280
7.276
7.264
7.158
7.154
7.142
7.138
7.134
3.915
2.097
0.000
Me
O
N
OMe
O
2a
0.88
1.00
0.99
3.22
2.77
2.81
2.85
f1 (ppm)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128.082
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127.932
127.167
124.517
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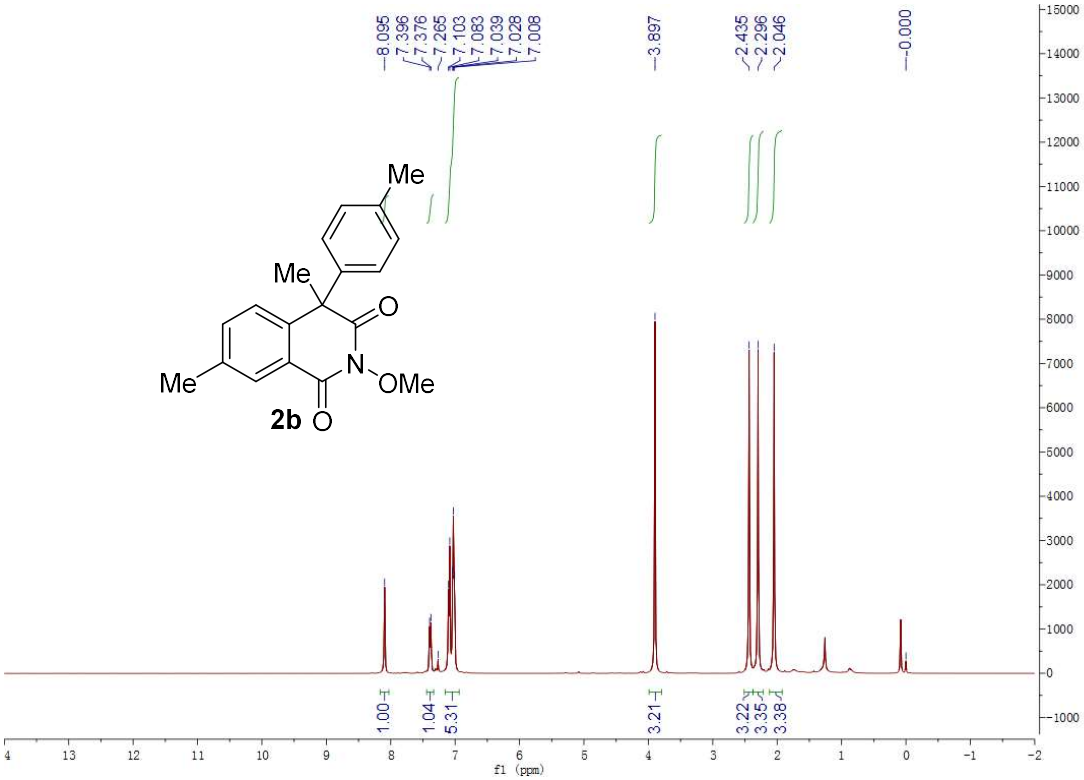
77.160
76.842
63.945
53.573
26.916
Me
O
N
OMe
O
2a
f1 (ppm)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7.028
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3.897
2.435
2.296
2.046
0.000
Me
Me
O
N
OMe
Me
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O
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1.04
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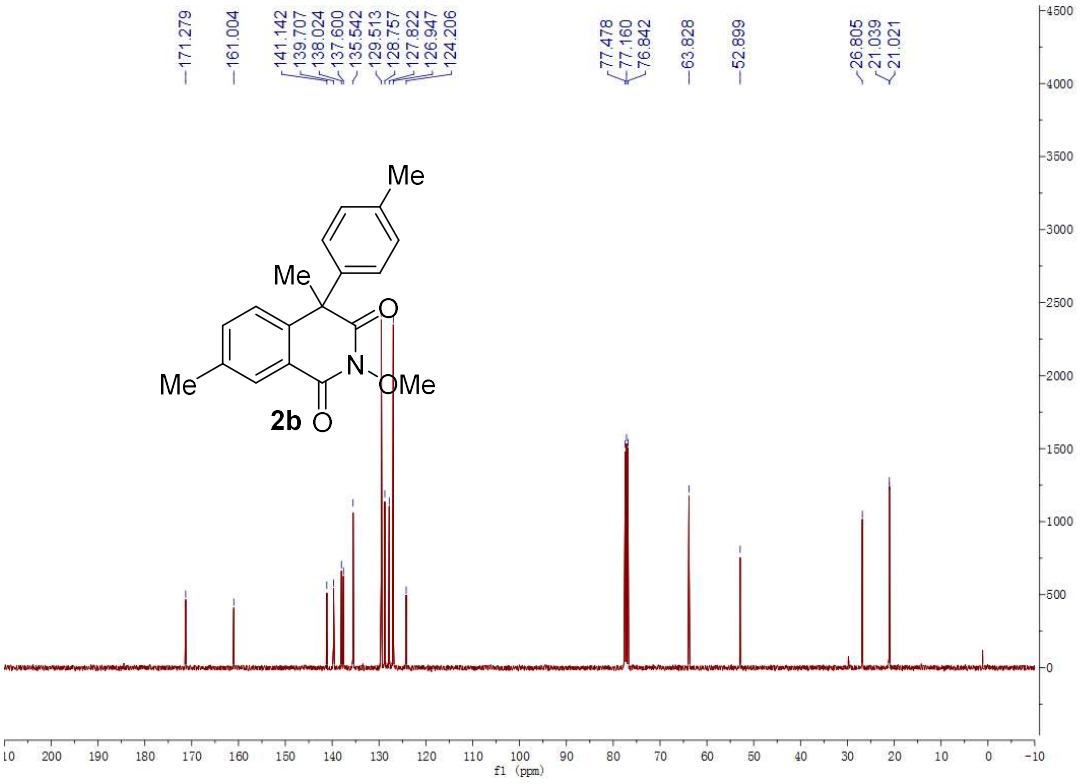
3.22
3.35
3.38
f1 (ppm)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77.160
76.842
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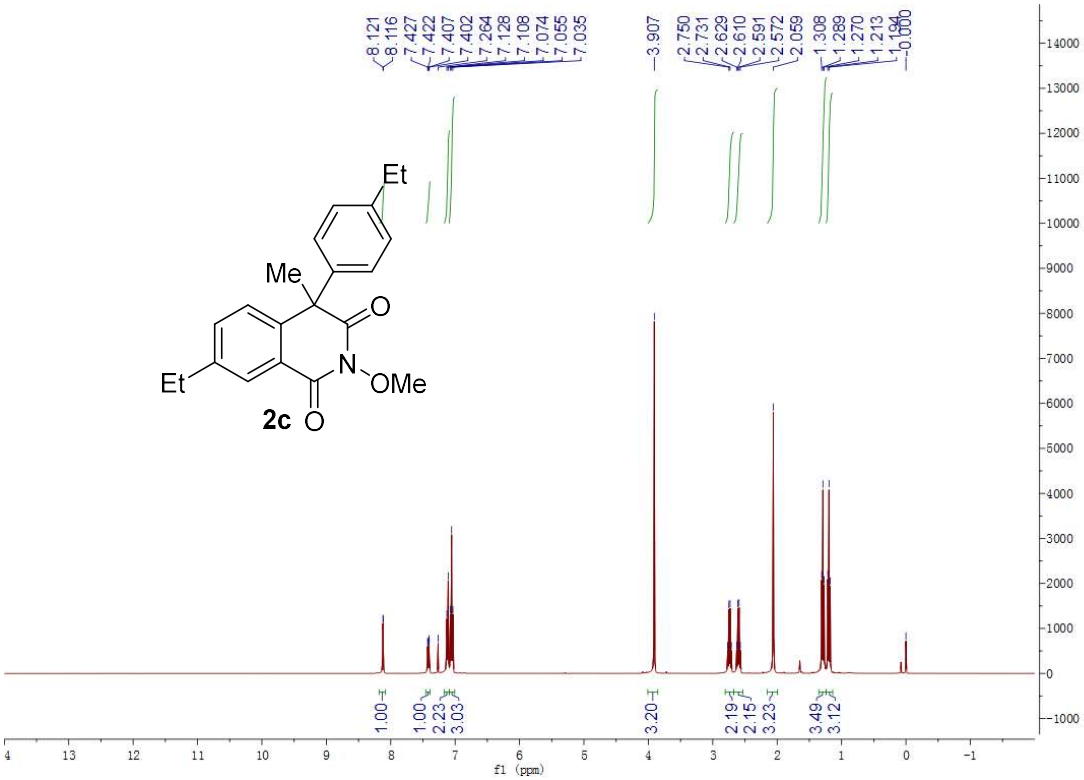
26.805
21.039
21.021
Me
Me
O
N
OMe
Me
2b
O
f1 (ppm)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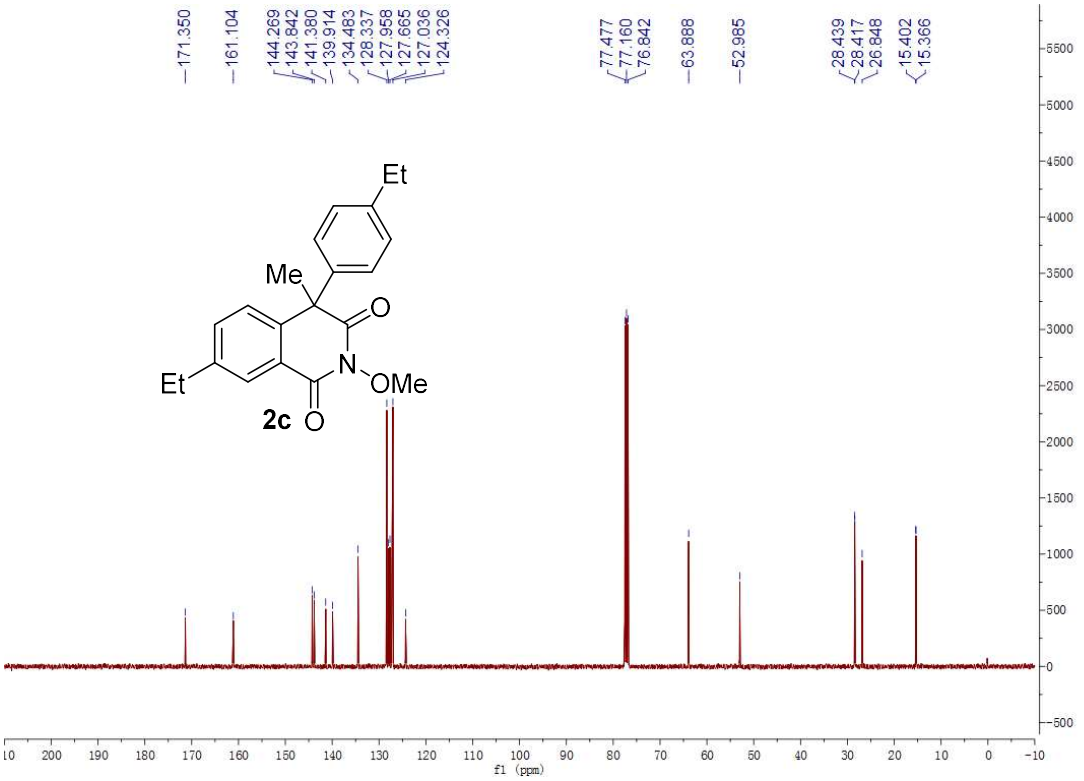
7.407
7.402
7.264
7.128
7.108
7.074
7.055
7.035
3.907
2.750
2.731
2.629
2.610
2.591
2.572
2.059
1.308
1.289
1.270
1.213
1.194
0.000
Et
Me
O
N
OMe
Et
O
2c
1.00
1.00
2.23
3.03
3.20
2.19
2.15
3.23
3.49
3.12
f1 (ppm)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77.477
77.160
76.842
63.888
52.985
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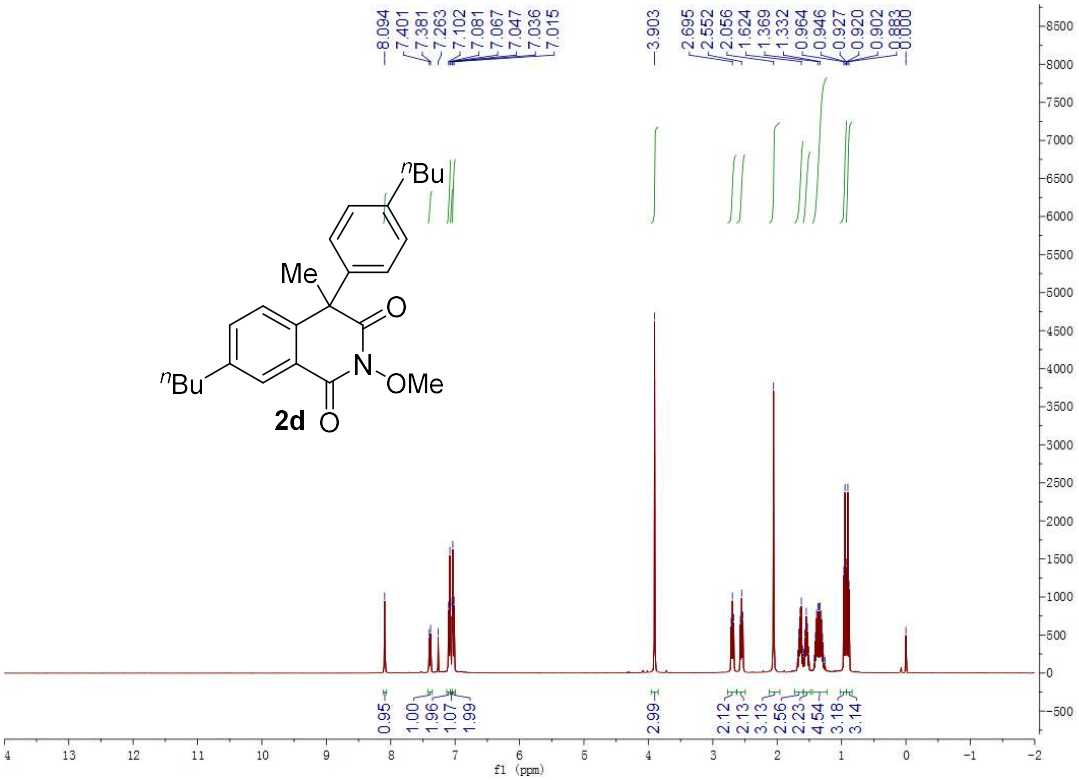
15.402
15.366
Et
Me
O
N
OMe
Et
O
2c
f1 (ppm)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7.081
7.067
7.047
7.036
7.015
3.903
2.695
2.552
2.056
1.624
1.369
1.332
0.964
0.946
0.927
0.920
0.902
0.883
0.866
nBu
Me
O
N
OMe
nBu
2d
O
0.95
1.00
1.96
1.07
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3.13
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2.23
4.54
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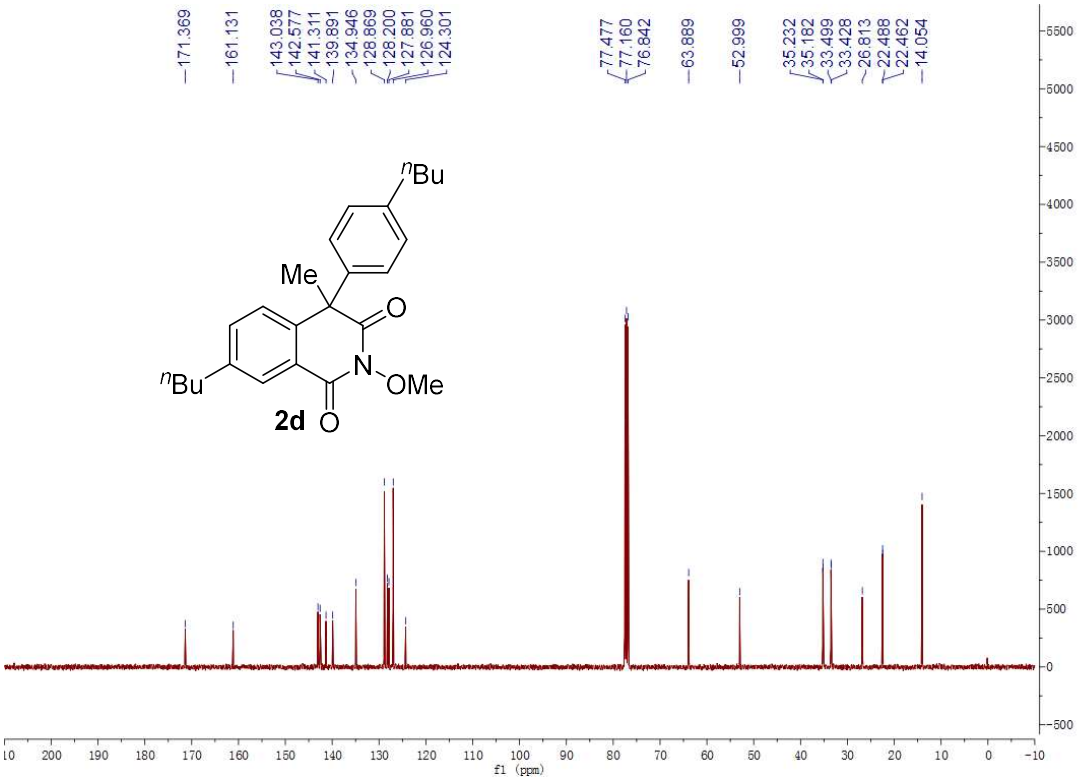
3.14
f1 (ppm)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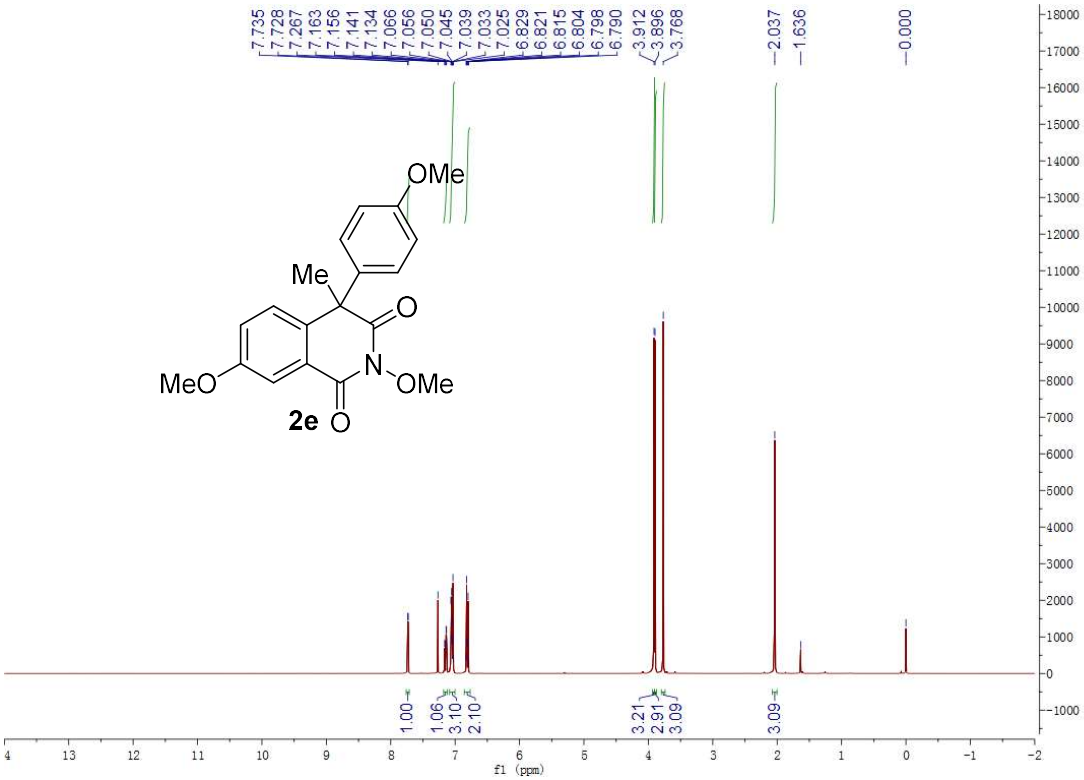
139.891
134.946
128.869
128.200
127.881
126.960
124.301
77.477
77.160
76.842
63.889
52.999
35.232
35.182
33.499
33.428
26.813
22.488
22.462
14.054
nBu
Me
O
N
OMe
nBu
2d
O
f1 (ppm)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7.267
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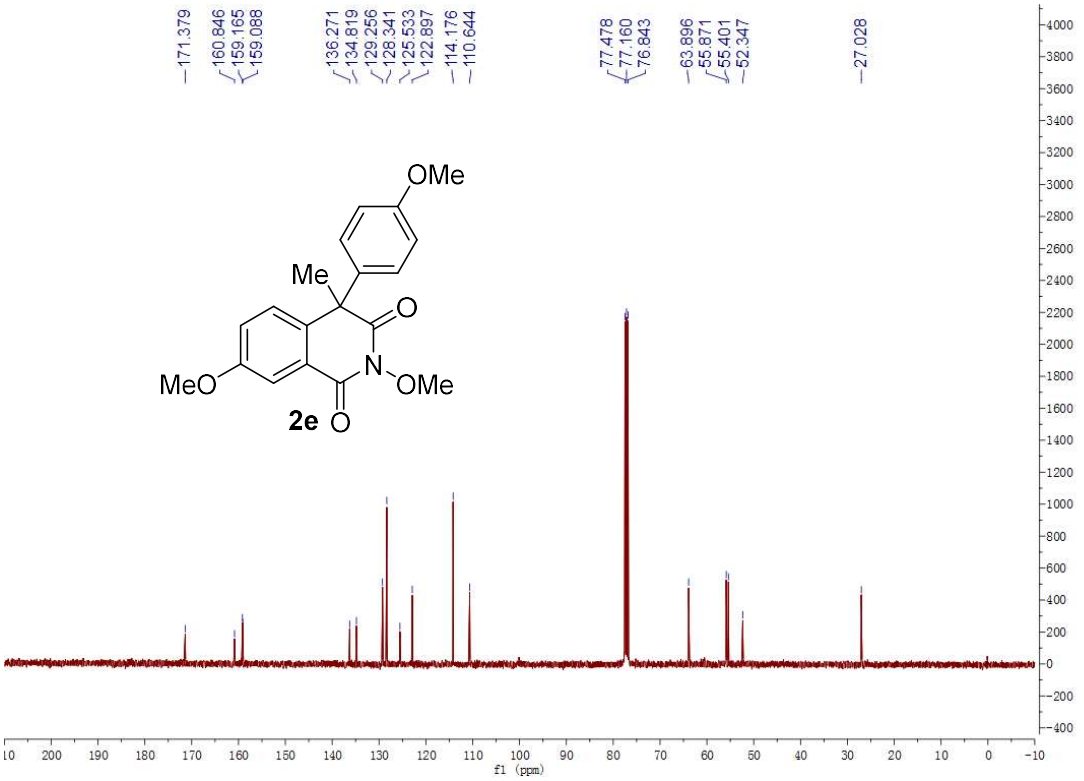
7.141
7.134
7.066
7.056
7.050
7.045
7.039
7.033
7.025
6.829
6.821
6.815
6.804
6.798
6.790
3.912
3.896
3.768
2.037
1.636
0.000
OMe
Me
O
N
OMe
MeO
O
2e
1.00
1.06
3.10
2.10
3.21
2.91
3.09
3.09
f1 (ppm)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128.341
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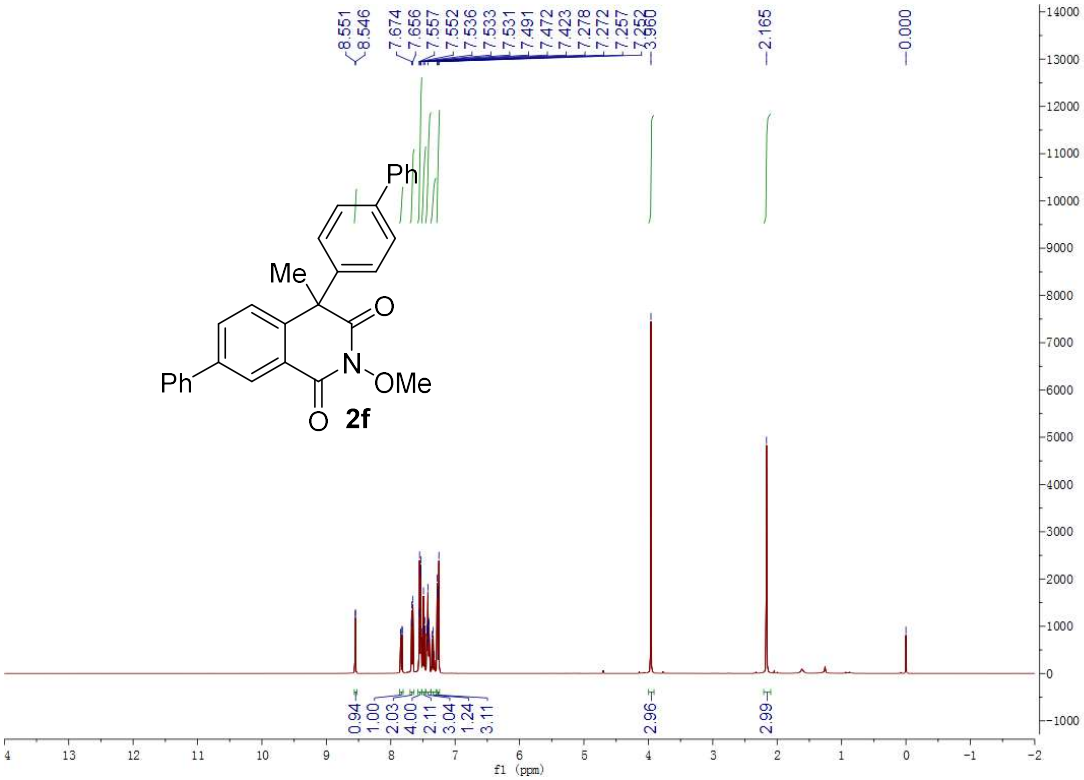
110.644
77.478
77.160
76.843
63.896
55.871
55.401
52.347
27.028
OMe
Me
O
N
OMe
MeO
O
2e
f1 (ppm)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7.533
7.531
7.491
7.472
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7.278
7.272
7.257
7.252
3.960
2.165
0.000
Ph
Me
O
N
OMe
Ph
O
2f
0.94
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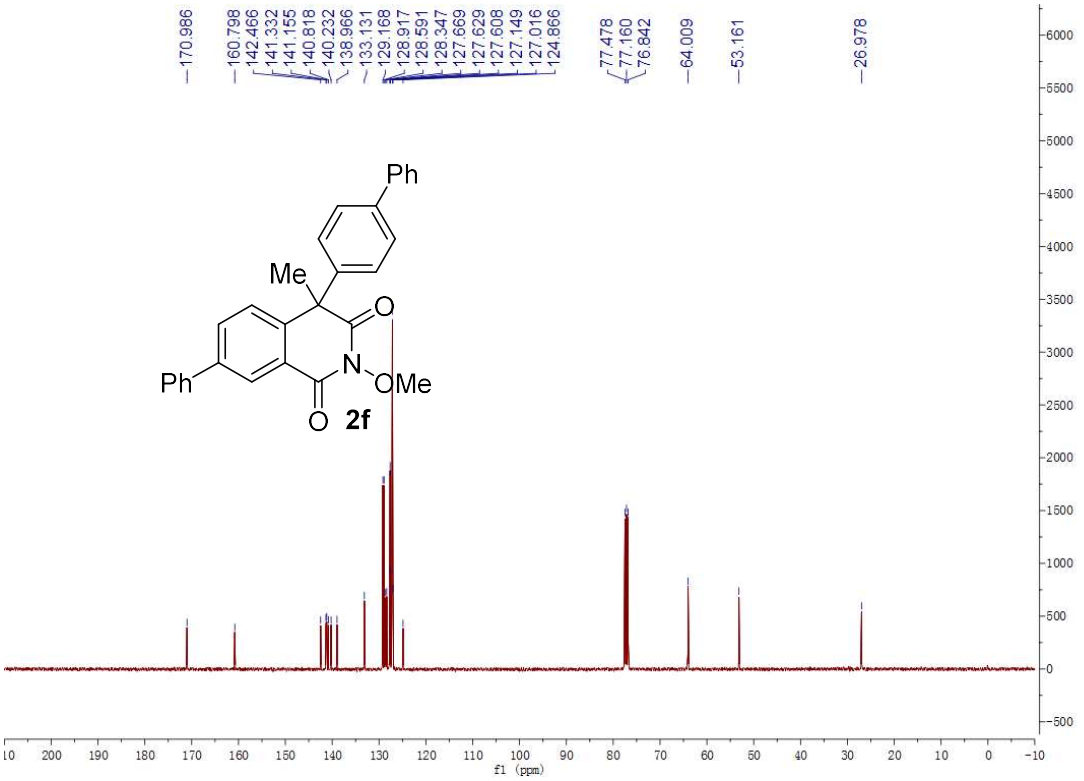
2.03
4.00
2.11
3.04
1.24
3.11
2.96
2.99
f1 (ppm)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141.332
141.155
140.818
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133.131
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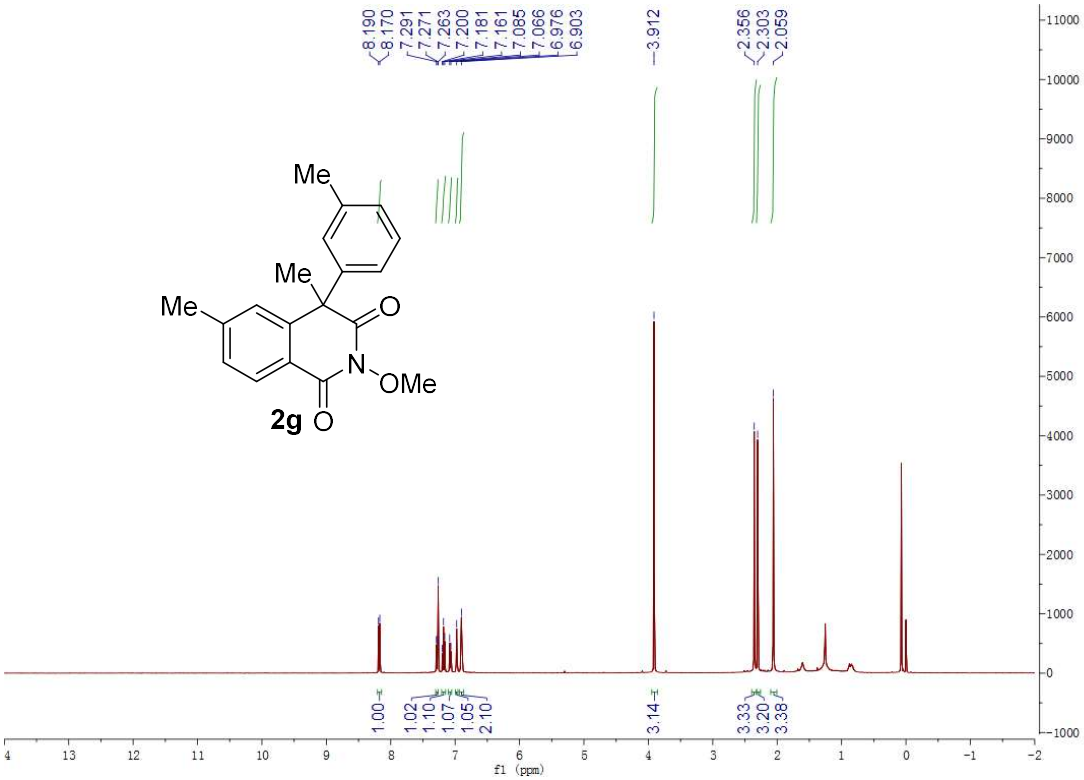
128.347
127.669
127.629
127.608
127.149
127.016
124.866
77.478
77.160
76.842
64.009
53.161
26.978
Ph
Me
O
N
OMe
Ph
O
2f
f1 (ppm)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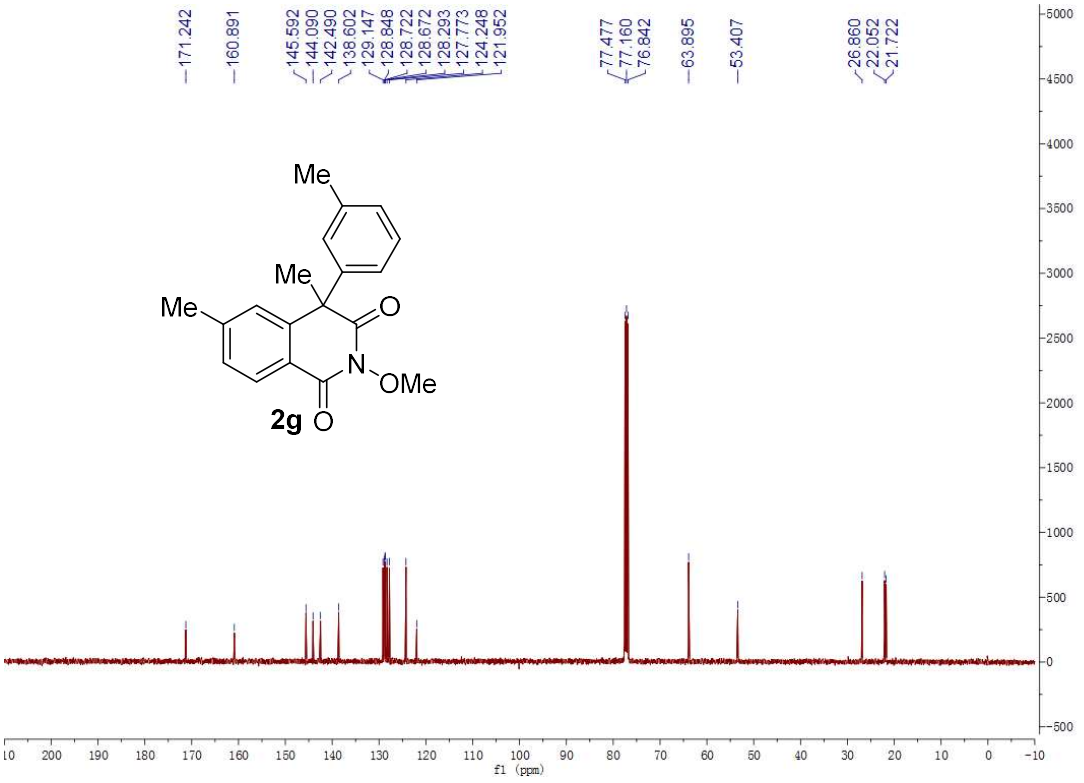
7.161
7.085
7.066
6.976
6.903
3.912
2.356
2.303
2.059
Me
Me
Me
O
N
OMe
2g
O
1.00
1.02
1.10
1.07
1.05
2.10
3.14
3.33
3.20
3.38
f1 (ppm)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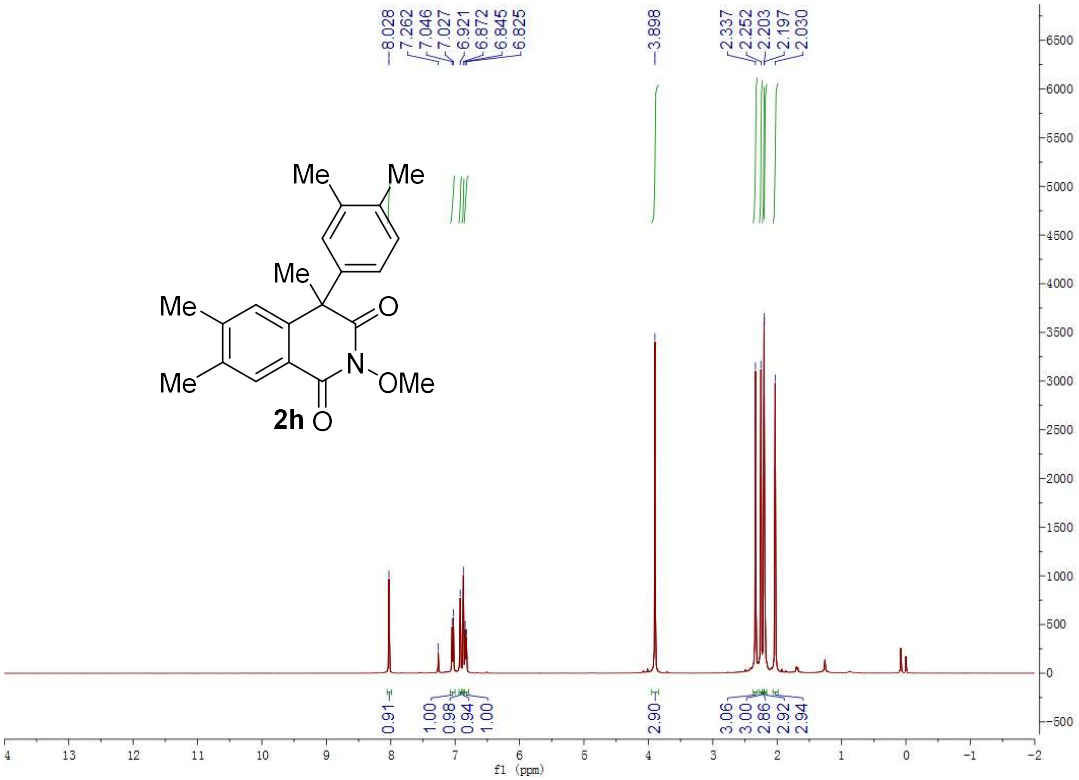
128.848
128.722
128.672
128.293
127.773
124.248
121.952
77.477
77.160
76.842
63.895
53.407
26.860
22.052
21.722
Me
Me
Me
O
N
OMe
2g
O
f1 (ppm)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6.845
6.825
3.898
2.337
2.252
2.203
2.197
2.030
Me
Me
Me
Me
Me
O
N
OMe
O
2h
0.91
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1.00
2.90
3.06
3.00
2.86
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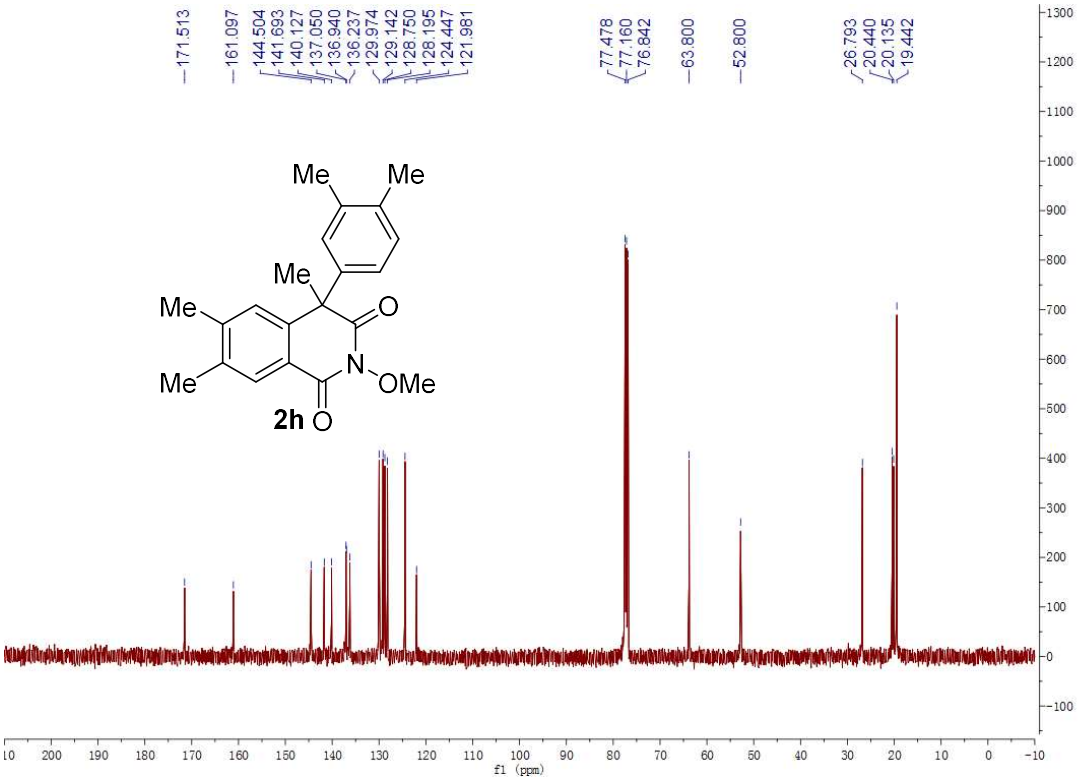
2.94
f1 (ppm)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136.237
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128.195
124.447
121.981
77.478
77.160
76.842
63.800
52.800
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Me
Me
O
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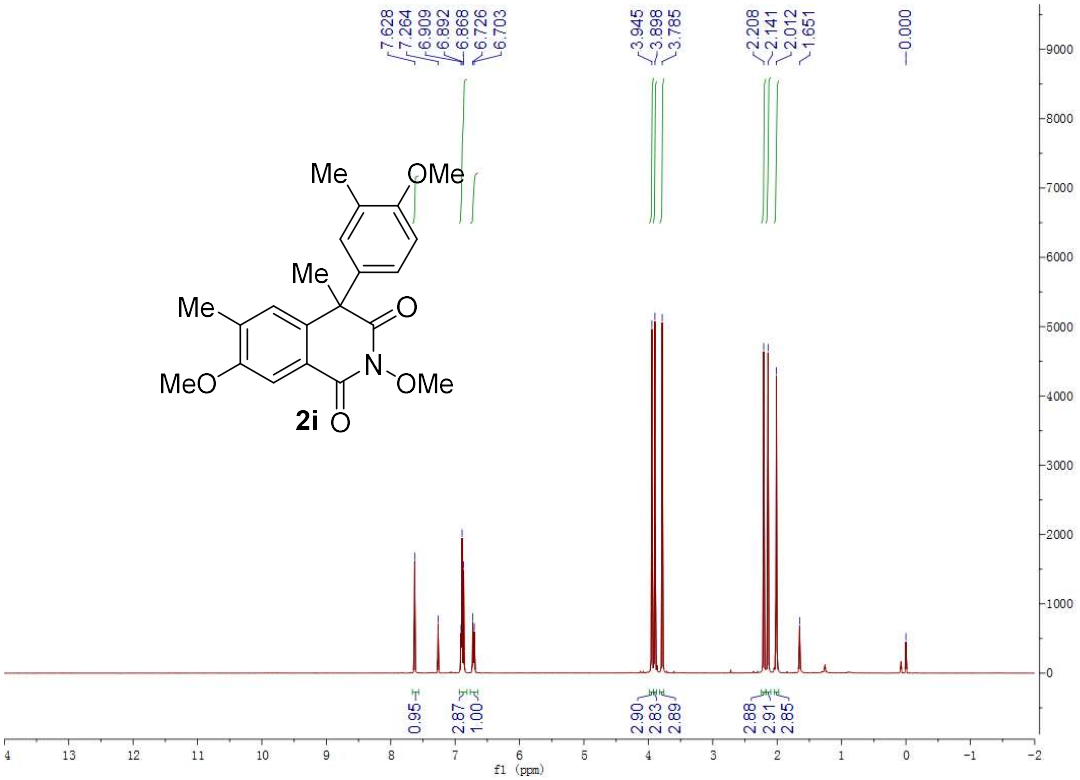
OMe
O
2h
f1 (ppm)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3.898
3.785
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2.141
2.012
1.651
0.000
Me
OMe
Me
Me
O
MeO
N
OMe
2i
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1.00
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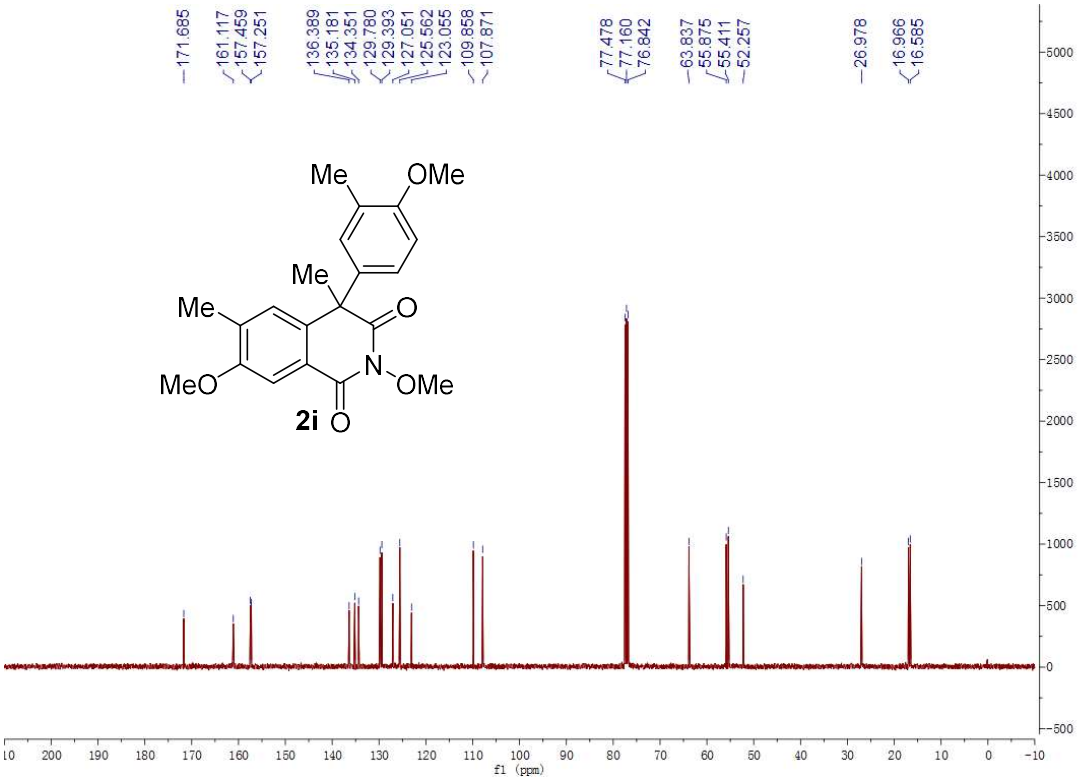
2.88
2.91
2.85
f1 (ppm)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123.055
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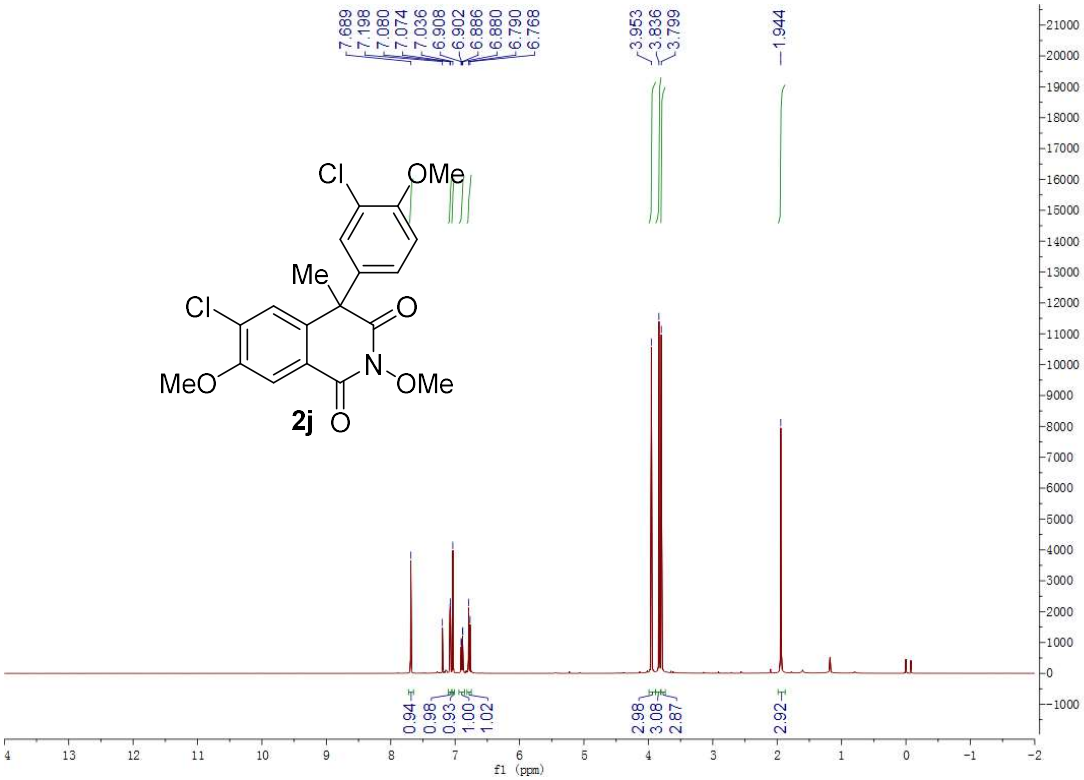
107.871
77.478
77.160
76.842
63.837
55.875
55.411
52.257
26.978
16.966
16.585
Me
OMe
Me
Me
O
MeO
N
OMe
2i
O
f1 (ppm)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6.886
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6.790
6.768
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3.836
3.799
1.944
Cl
OMe
Me
Cl
O
N
MeO
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O
2j
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0.98
0.93
1.00
1.02
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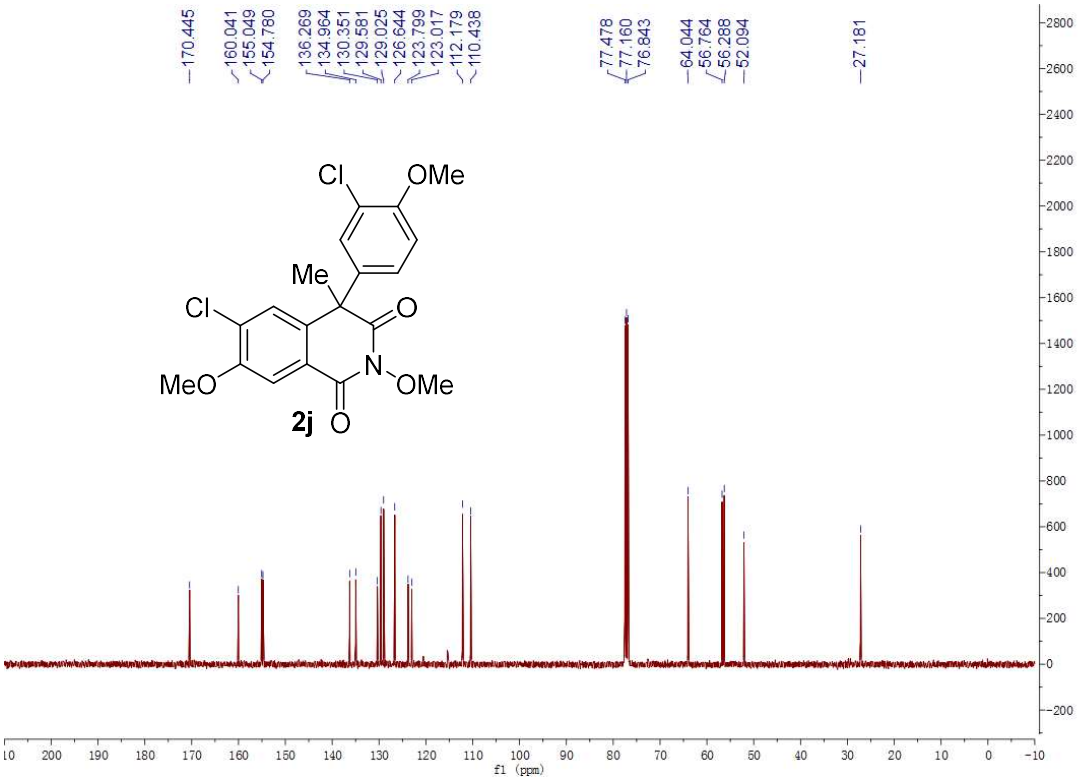
3.08
2.87
2.92
f1 (ppm)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129.581
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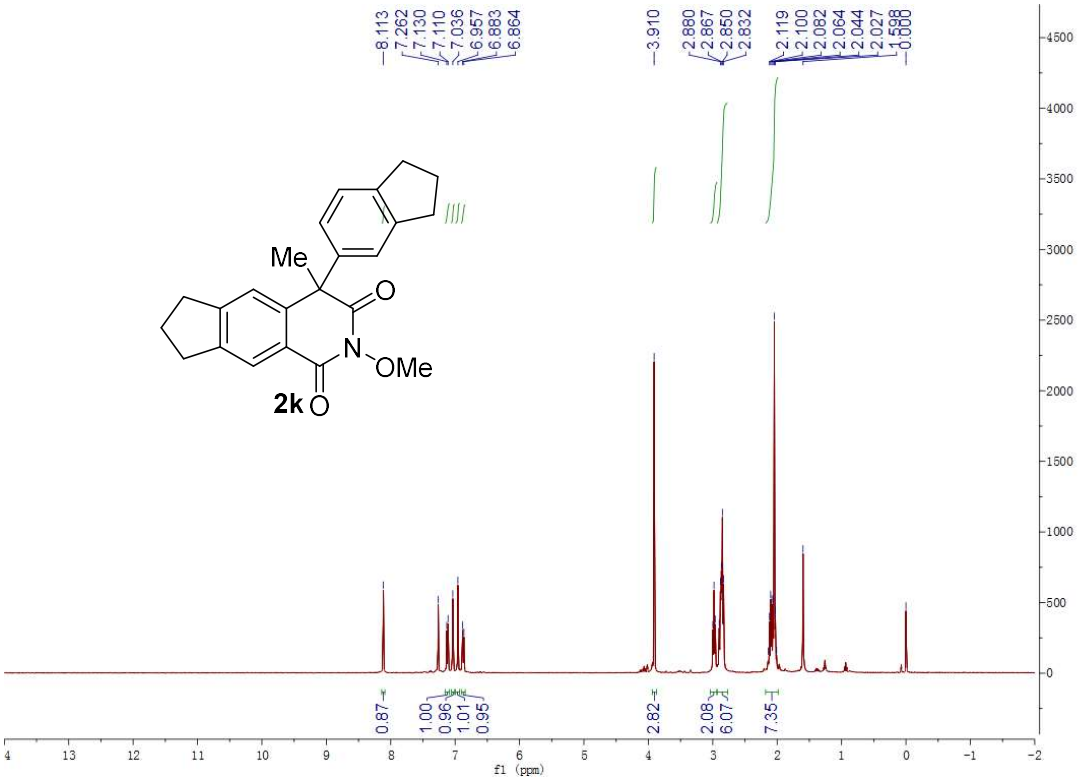
112.179
110.438
77.478
77.160
76.843
64.044
56.764
56.288
52.094
27.181
Cl
OMe
Me
Cl
O
N
MeO
OMe
O
2j
f1 (ppm)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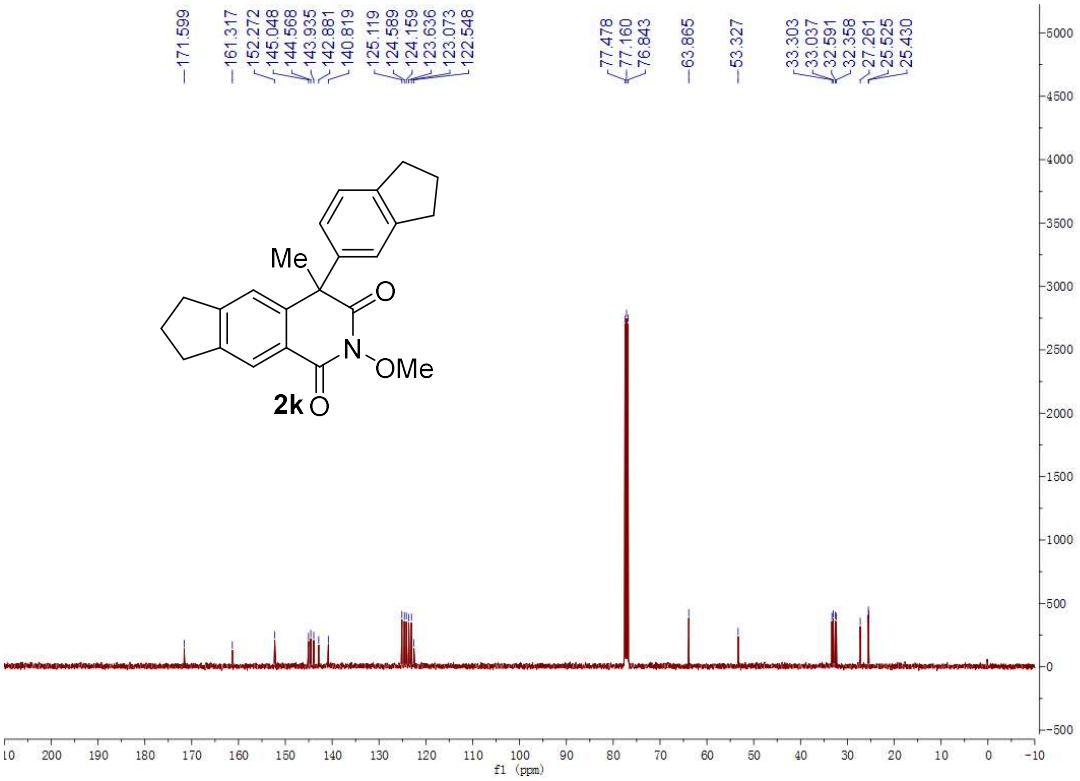
2.119
2.100
2.082
2.064
2.044
2.027
1.598
0.000
Me
O
N
OMe
O
2k
0.87
1.00
0.96
1.01
0.95
2.82
2.08
6.07
7.35
f1 (ppm)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140.819
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123.636
123.073
122.548
77.478
77.160
76.843
63.865
53.327
33.303
33.037
32.591
32.358
27.261
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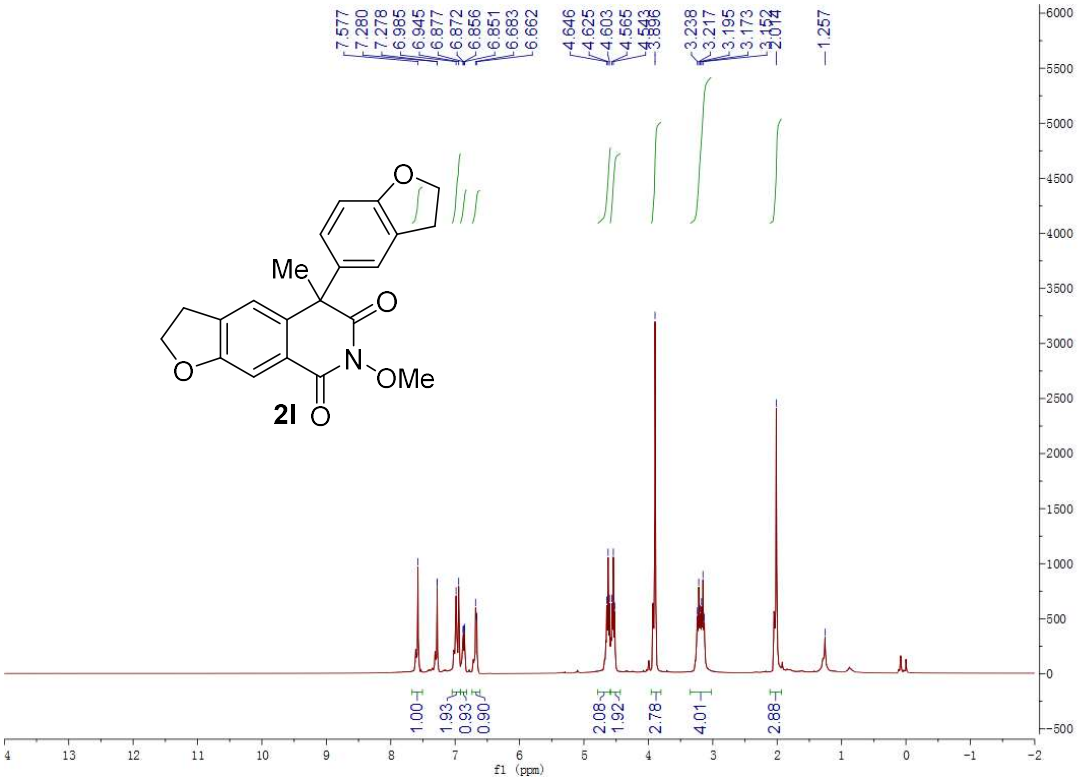
Me
O
N
OMe
O
2k
f1 (ppm)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6.856
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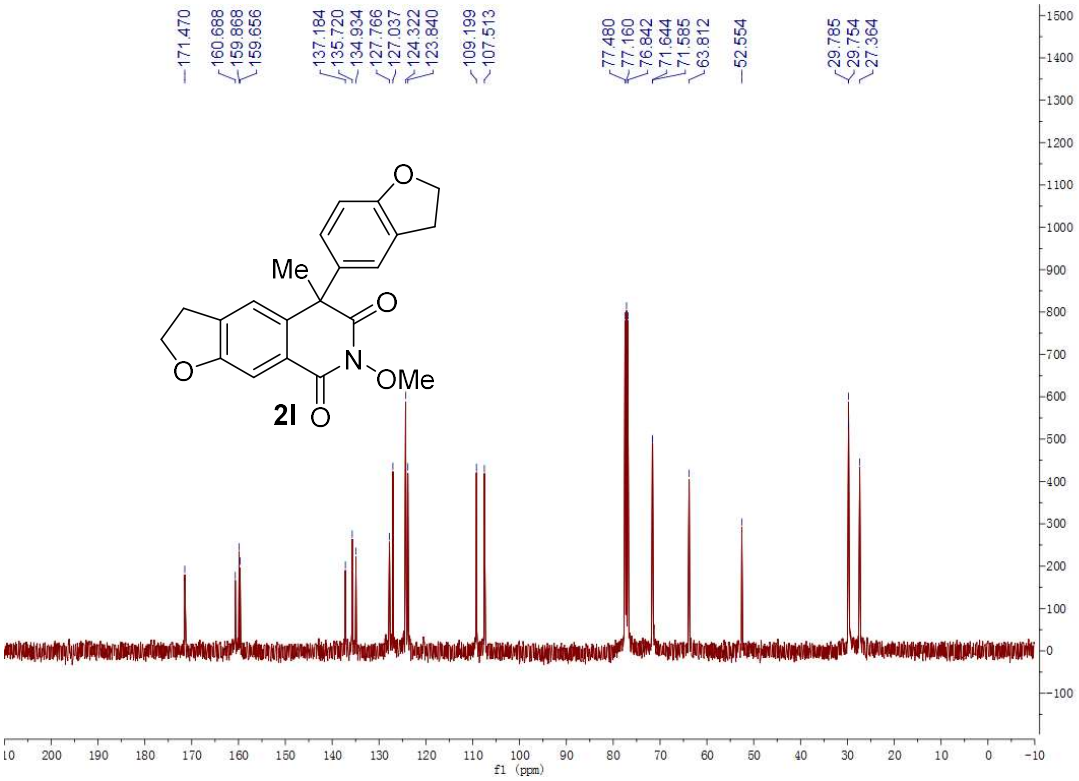
4.603
4.565
4.543
3.896
3.238
3.217
3.195
3.173
3.152
3.014
1.257
Me
O
N
OMe
2l
1.00
1.93
0.93
0.90
2.08
1.92
2.78
4.01
2.88
f1 (ppm)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127.766
127.037
124.322
123.840
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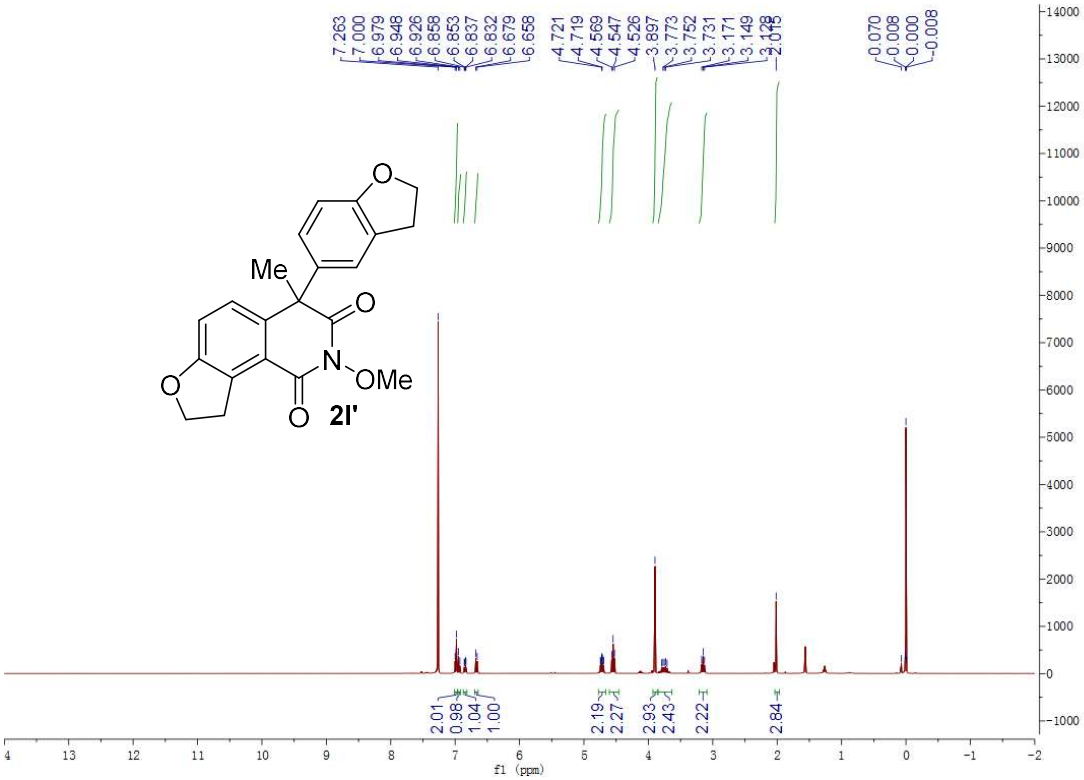
107.513
77.480
77.160
76.842
71.644
71.585
63.812
52.554
29.785
29.754
27.364
Me
O
N
OMe
2l
f1 (ppm)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6.853
6.837
6.832
6.679
6.658
4.721
4.719
4.569
4.547
4.526
3.897
3.773
3.752
3.731
3.171
3.149
3.128
2.015
0.070
0.008
0.000
-0.008
Me
O
N
OMe
O
O
2l'
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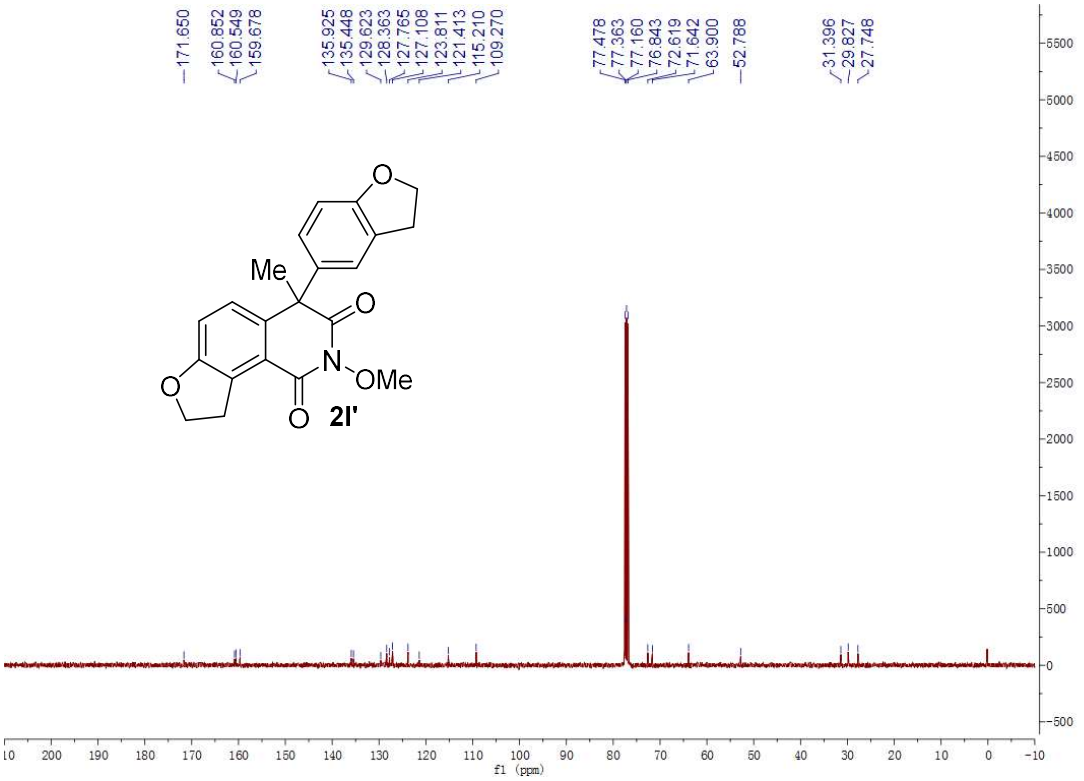
0.98
1.04
1.00
2.19
2.27
2.93
2.43
2.22
2.84
f1 (ppm)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127.108
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115.210
109.270
77.478
77.363
77.160
76.843
72.619
71.642
63.900
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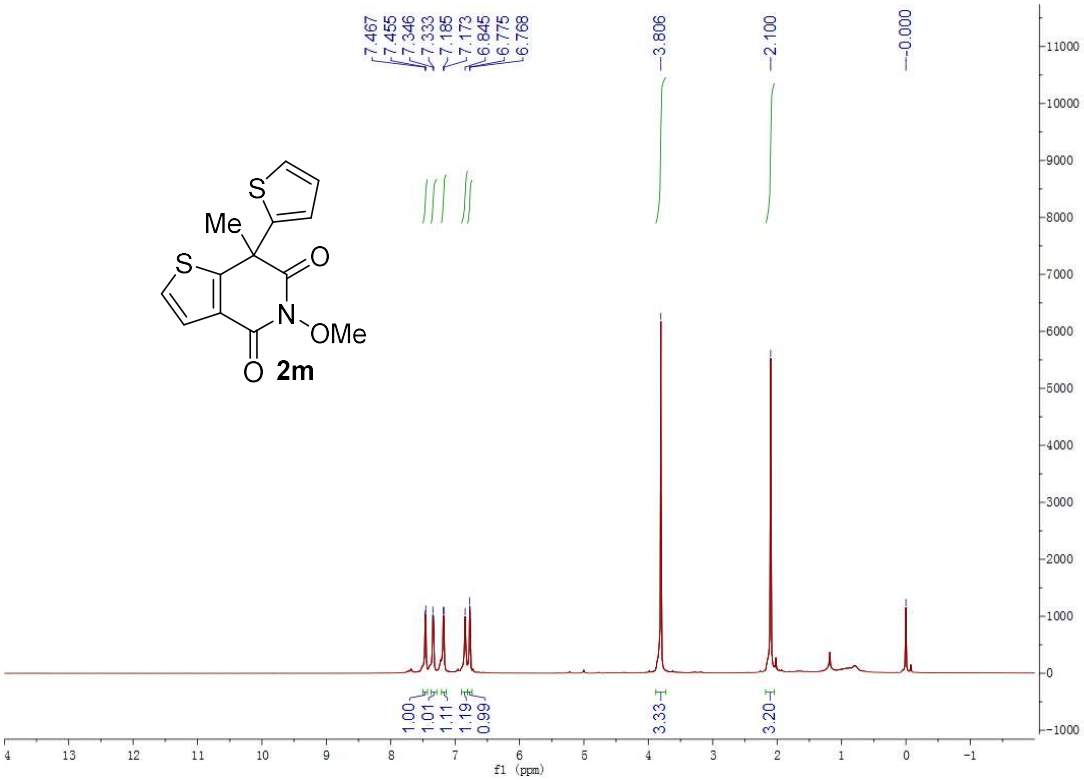
31.396
29.827
27.748
Me
O
N
OMe
O
O
2l'
f1 (ppm)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7.173
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6.775
6.768
3.806
2.100
0.000
Me
S
O
N
OMe
O
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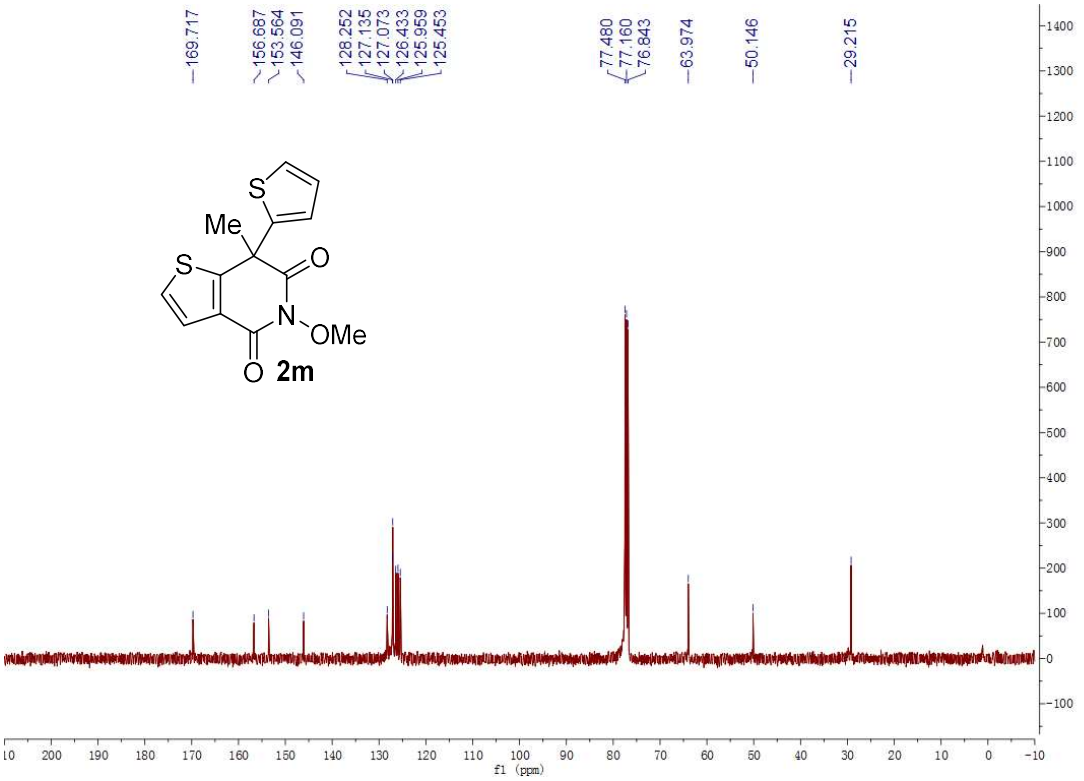
1.00
1.01
1.11
1.19
0.99
3.33
3.20
f1 (ppm)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127.135
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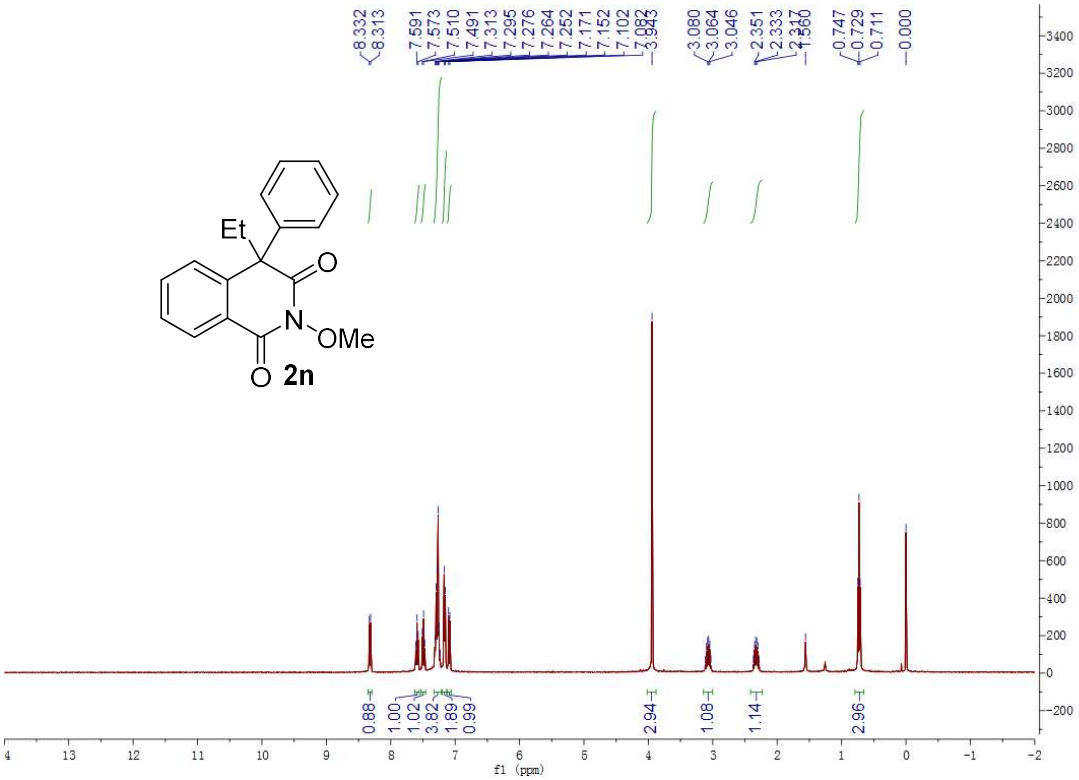
125.959
125.453
77.480
77.160
76.843
63.974
50.146
29.215
Me
S
O
N
OMe
O
2m
f1 (ppm)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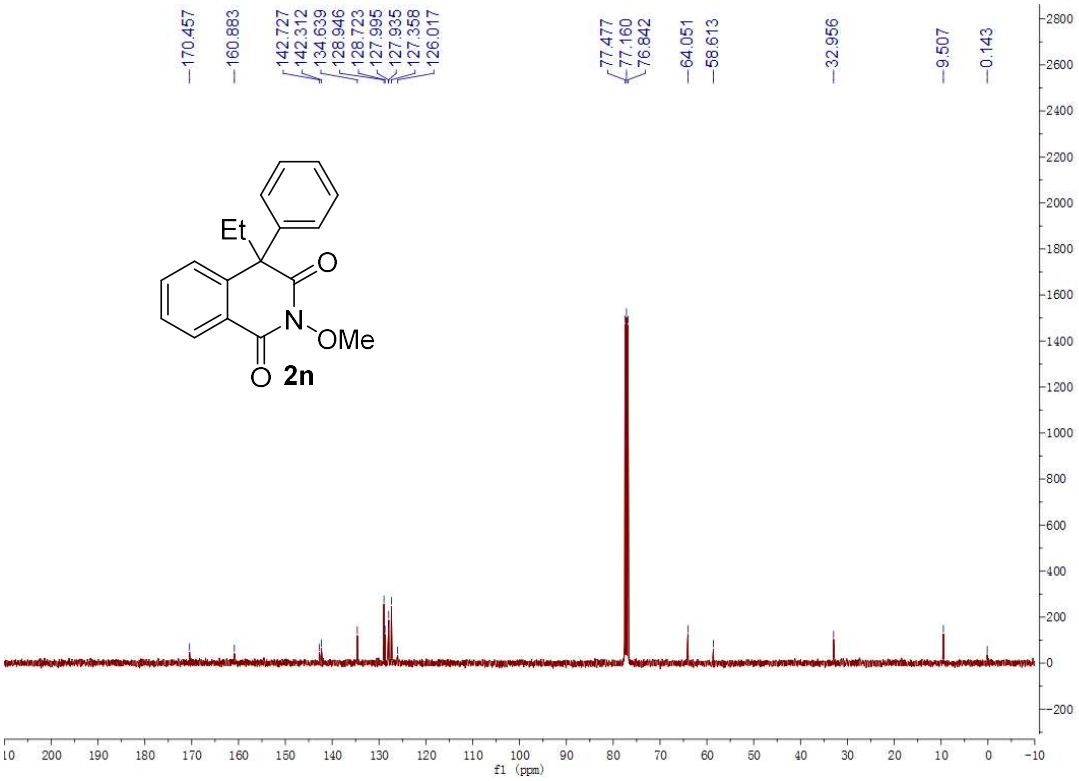
8.313
7.591
7.573
7.510
7.491
7.313
7.295
7.276
7.264
7.252
7.171
7.152
7.102
7.082
7.043
3.080
3.064
3.046
2.351
2.333
2.317
1.560
0.747
0.729
0.711
0.000
Et
O
N
OMe
O
2n
0.88
1.00
1.02
3.82
1.89
0.99
2.94
1.08
1.14
2.96
f1 (ppm)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128.723
127.995
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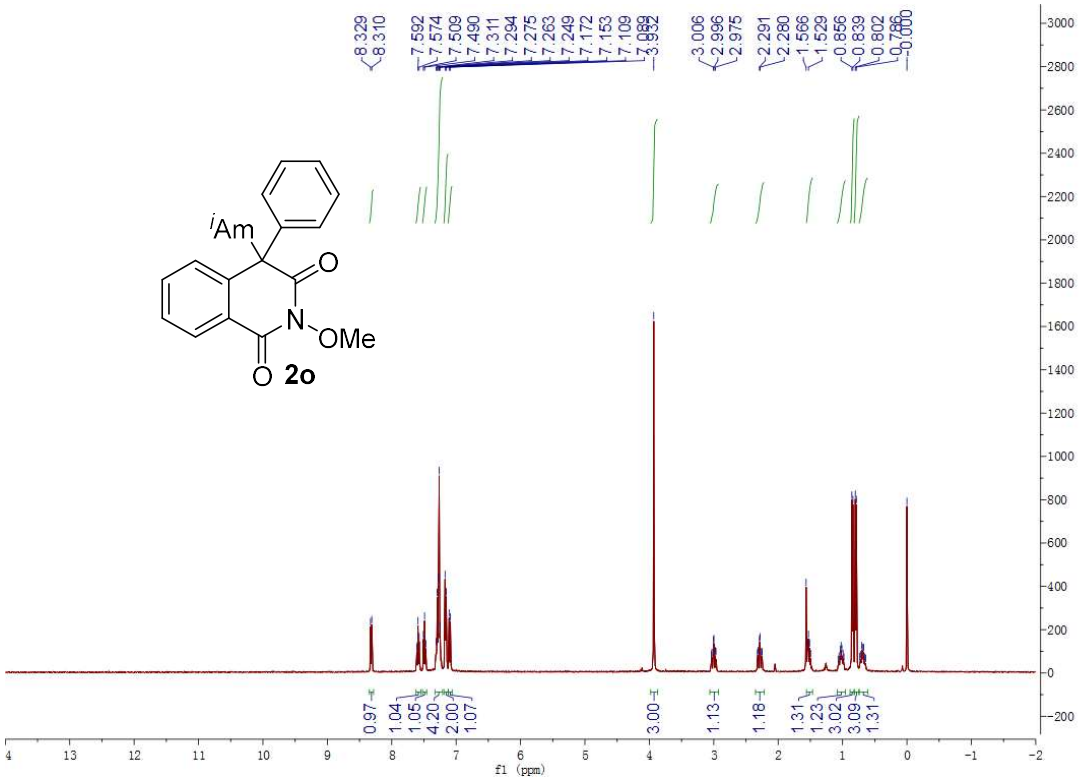
127.358
126.017
77.477
77.160
76.842
64.051
58.613
32.956
9.507
0.143
Et
O
N
OMe
O
2n
f1 (ppm)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8.310
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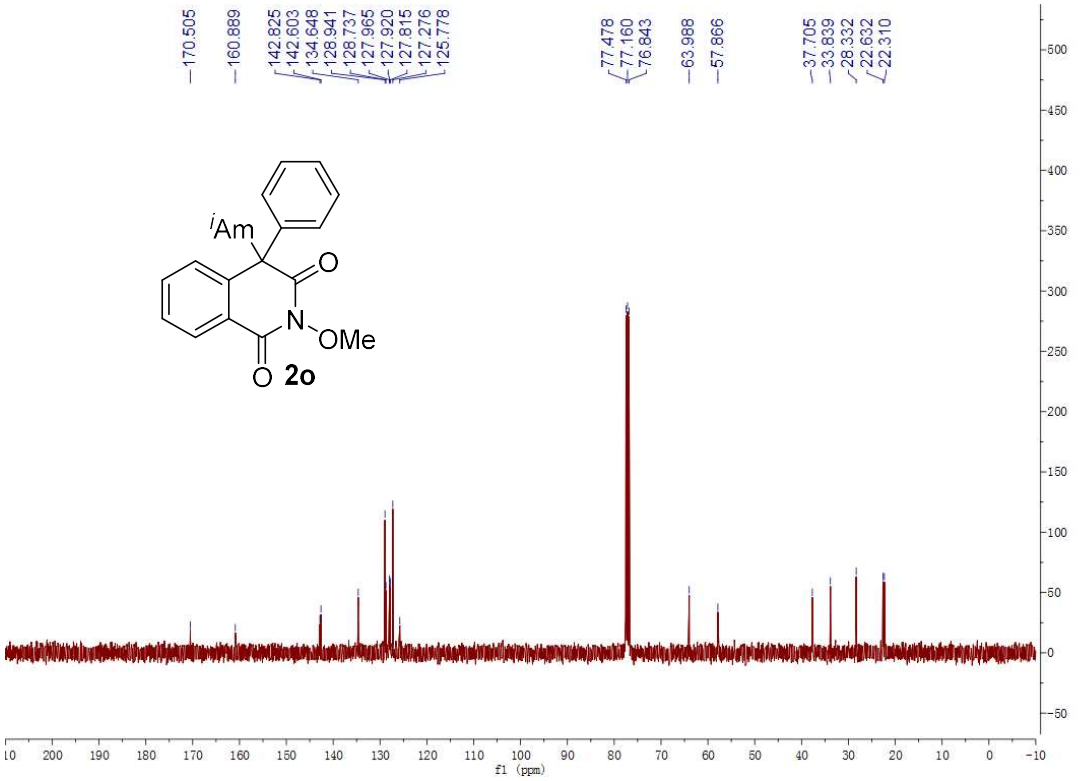
7.574
7.509
7.490
7.311
7.294
7.275
7.263
7.249
7.172
7.153
7.109
7.089
3.932
3.006
2.996
2.975
2.291
2.280
1.566
1.529
0.856
0.839
0.802
0.786
0.000
0.97
1.04
1.05
4.20
2.00
1.07
3.00
1.13
1.18
1.31
1.23
3.02
3.09
1.31
f1 (ppm)
iAm
O
N
OMe
O
2o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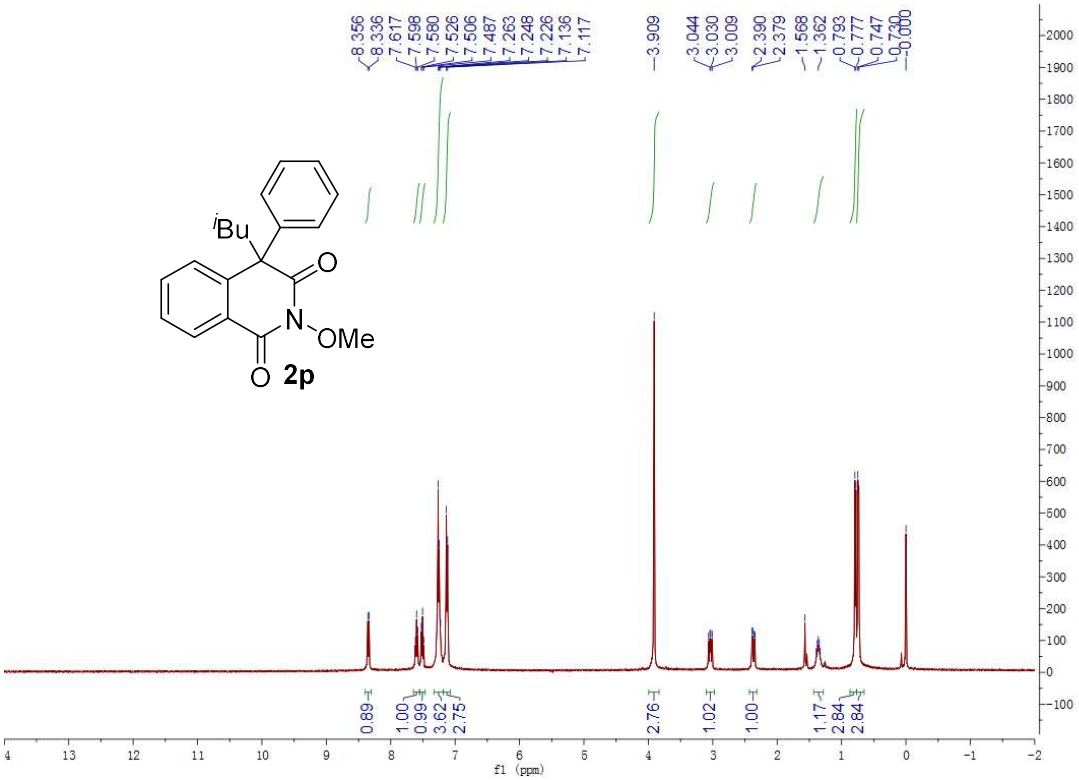
127.276
125.778
77.478
77.160
76.843
63.988
57.866
37.705
33.839
28.332
22.632
22.310
f1 (ppm)
iAm
O
N
OMe
O
2o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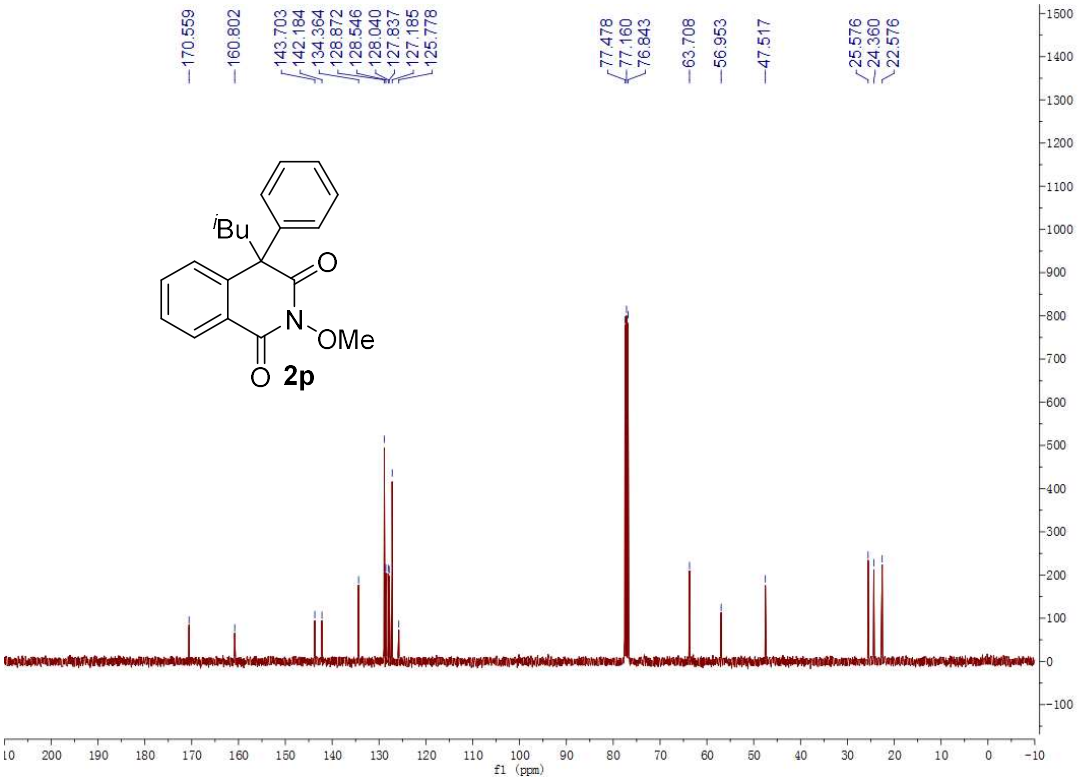
7.598
7.580
7.526
7.506
7.487
7.263
7.248
7.226
7.136
7.117
3.909
3.044
3.030
3.009
2.390
2.379
1.568
1.362
0.793
0.777
0.747
0.730
0.000
0.89
1.00
0.99
3.62
2.75
2.76
1.02
1.00
1.17
2.84
2.84
fl (ppm)
iBu
O
N
OMe
O
2p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134.364
128.872
128.546
128.040
127.837
127.185
125.778
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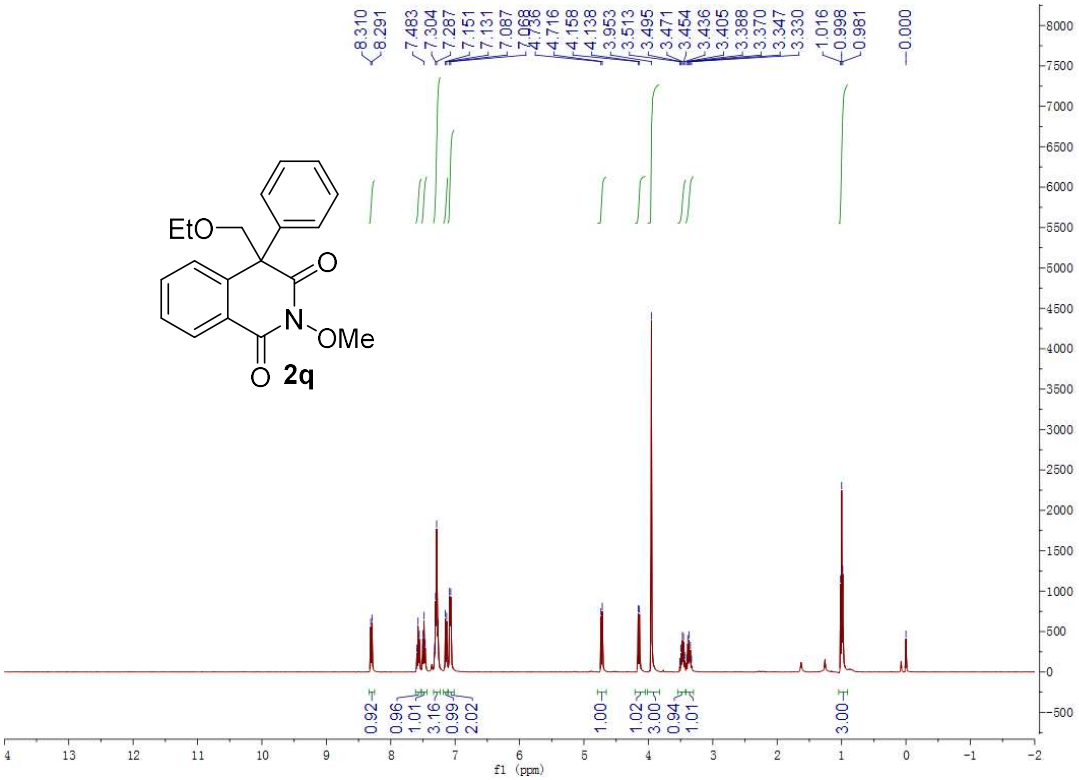
77.160
76.843
63.708
56.953
47.517
25.576
24.360
22.576
fl (ppm)
iBu
O
N
OMe
O
2p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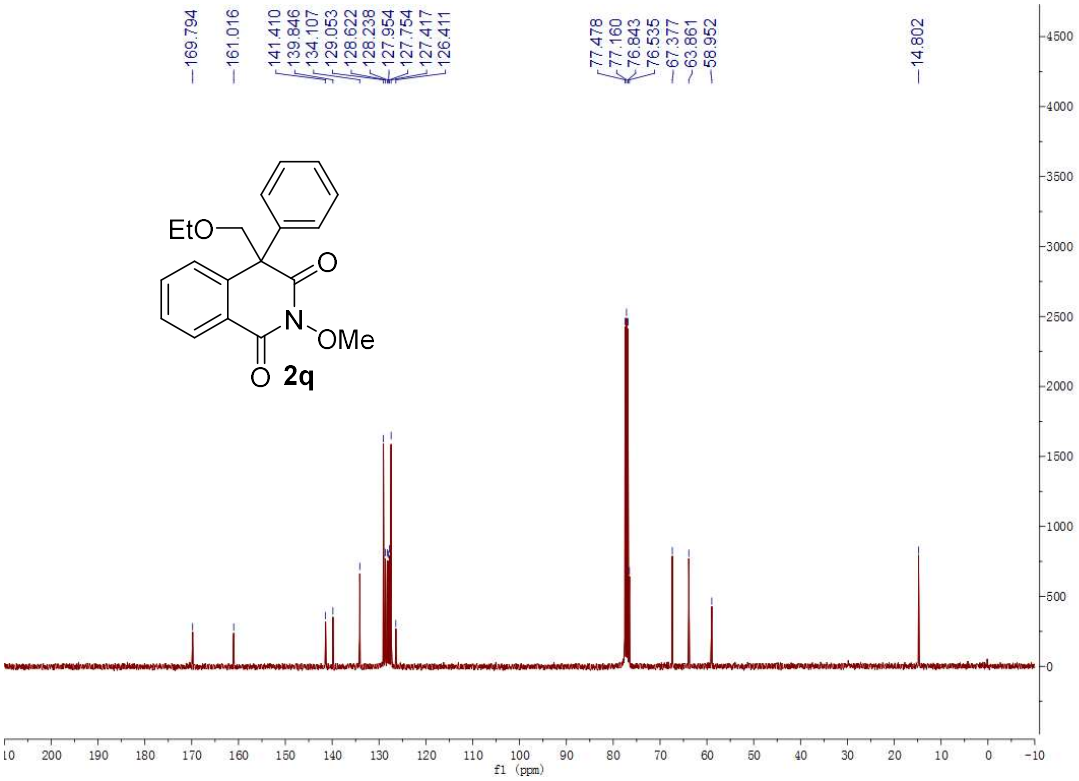
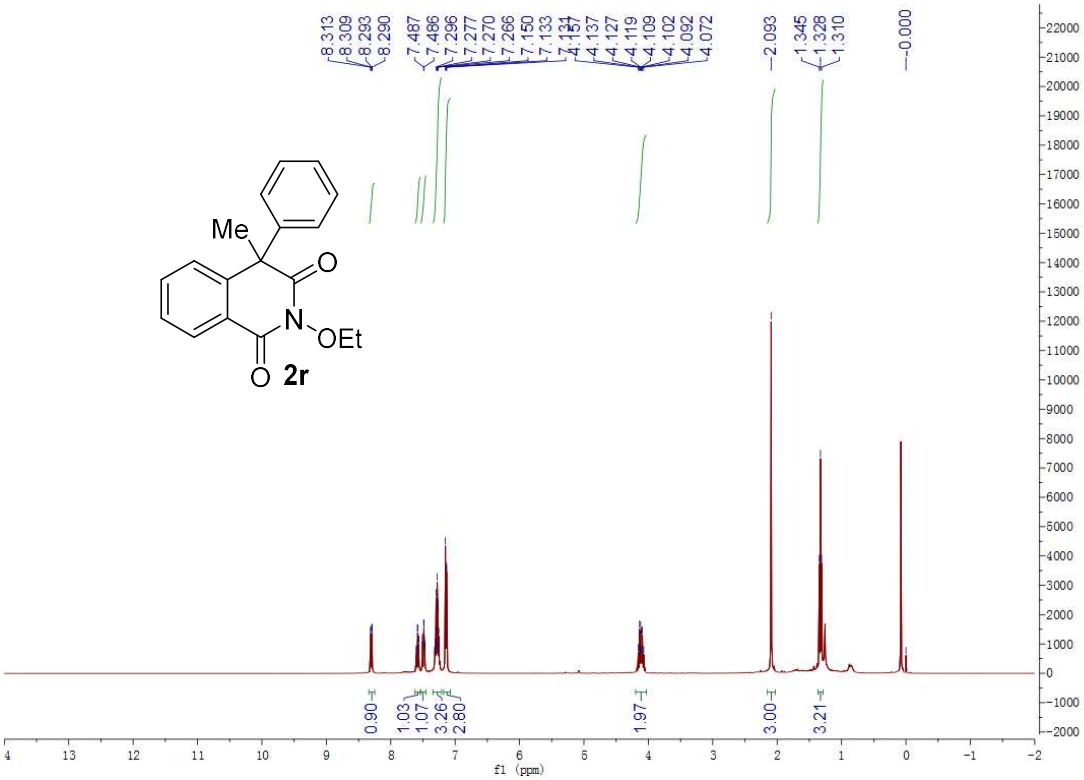
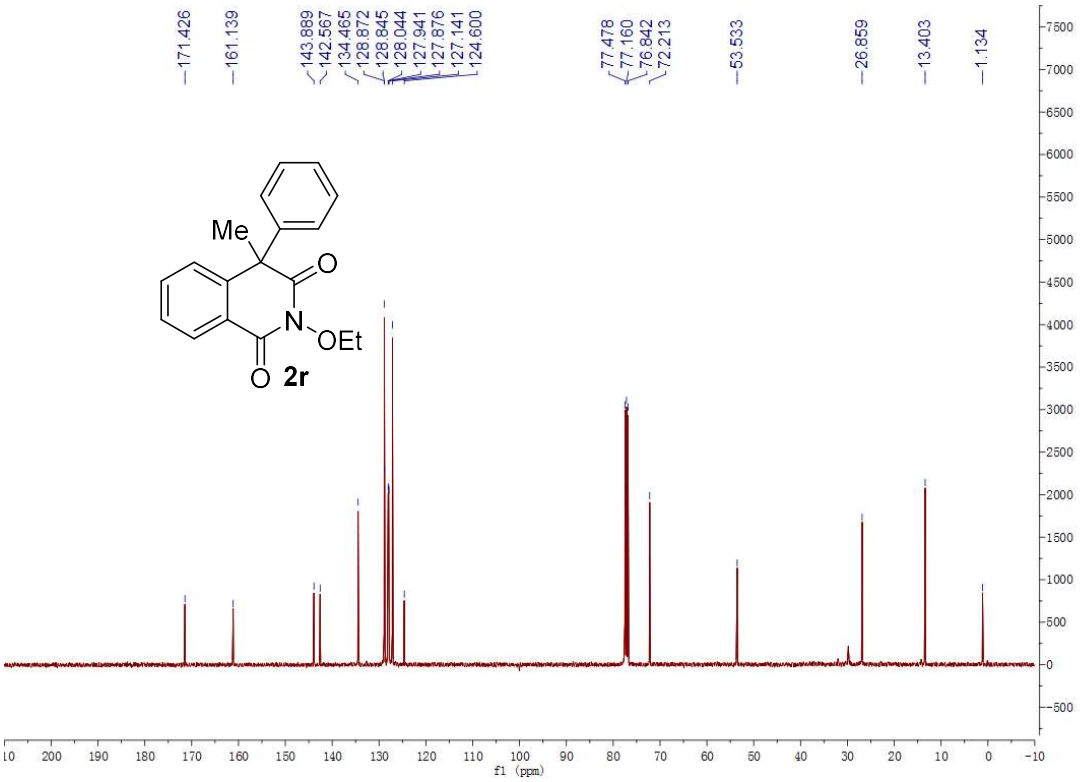
8.291
7.483
7.304
7.287
7.151
7.131
7.087
4.716
4.158
4.138
3.953
3.513
3.495
3.471
3.454
3.436
3.405
3.388
3.370
3.347
3.330
1.016
0.998
0.981
0.000
EtO
O
N
OMe
O
2q
0.92
0.96
1.01
3.16
0.99
2.02
1.00
1.02
3.00
0.94
1.01
3.00
f1 (ppm)


169.794
161.016
141.410
139.846
134.107
129.053
128.622
128.238
127.954
127.754
127.417
126.411
77.478
77.160
76.843
76.535
67.377
63.861
58.952
14.802
EtO
O
N
OMe
O
2q
f1 (ppm)


8.313
8.309
8.293
8.290
7.487
7.486
7.296
7.277
7.270
7.266
7.150
7.133
4.137
4.127
4.119
4.109
4.102
4.092
4.072
2.093
1.345
1.328
1.310
0.000
Me
O
N
OEt
O
2r
0.90
1.03
1.07
3.26
2.80
1.97
3.00
3.21
f1 (ppm)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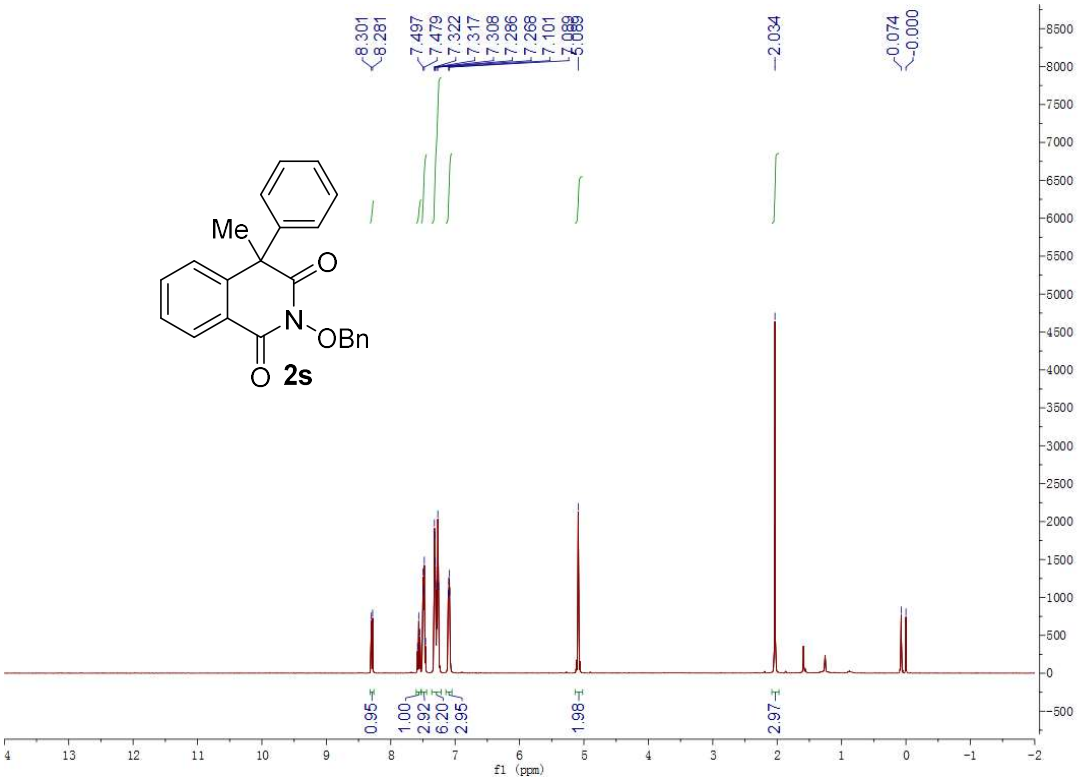
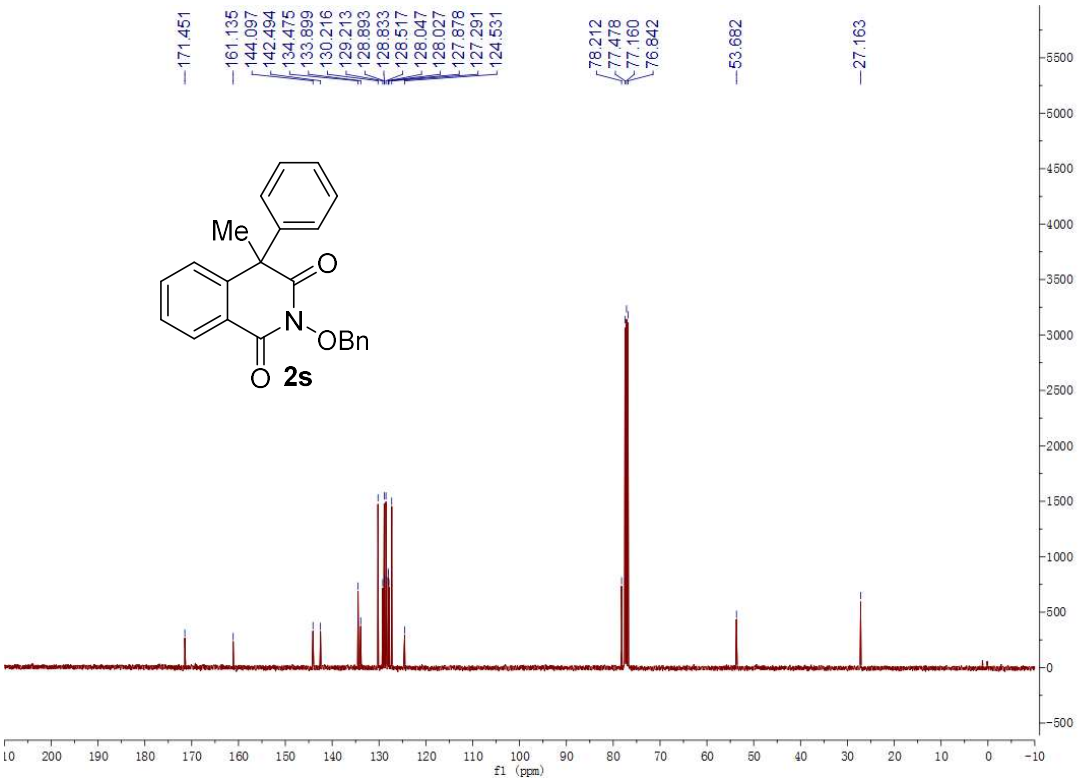
143.889
142.567
134.465
128.872
128.845
128.044
127.941
127.876
127.141
124.600
77.478
77.160
76.842
72.213
53.533
26.859
13.403
1.134
Me
O
N
OEt
O
2r
f1 (ppm)


8.301
8.281
7.497
7.479
7.322
7.317
7.308
7.286
7.268
7.101
5.089
2.034
0.074
-0.000
Me
O
N
OBn
O
2s
0.95
1.00
2.92
6.20
2.95
1.98
2.97
f1 (ppm)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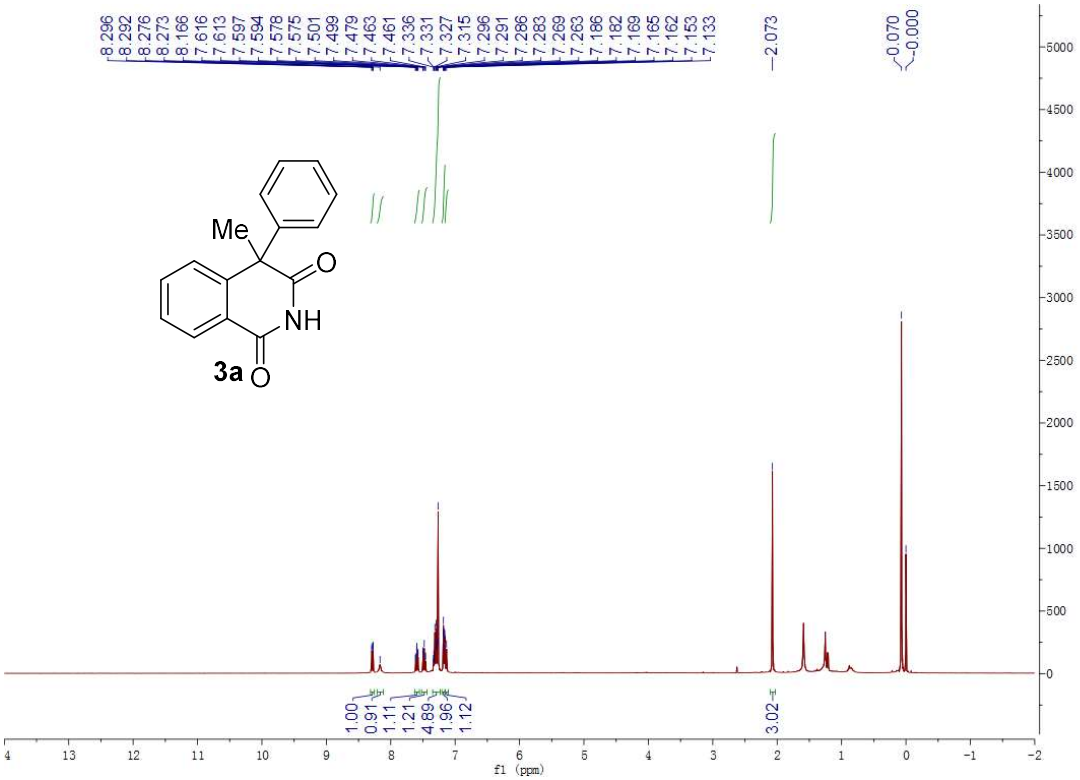
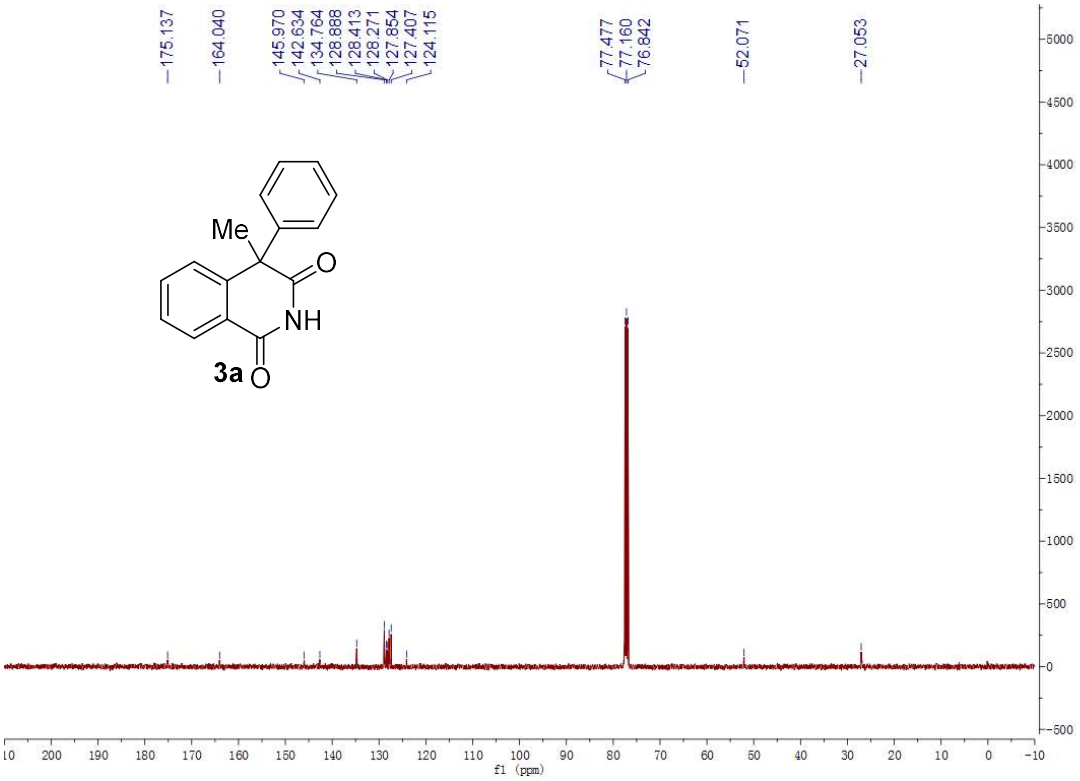
144.097
142.494
134.475
133.899
130.216
129.213
128.893
128.833
128.517
128.047
128.027
127.878
127.291
124.531
78.212
77.478
77.160
76.842
53.682
27.163
Me
O
N
OBn
O
2s
f1 (ppm)


8.296
8.292
8.276
8.273
8.166
7.616
7.613
7.597
7.594
7.578
7.575
7.501
7.499
7.479
7.463
7.461
7.336
7.331
7.327
7.315
7.296
7.291
7.286
7.283
7.269
7.263
7.186
7.182
7.169
7.165
7.162
7.153
7.133
2.073
0.070
0.000
Me
O
NH
O
3a
1.00
0.91
1.11
1.21
4.89
1.96
1.12
3.02
f1 (ppm)


175.137
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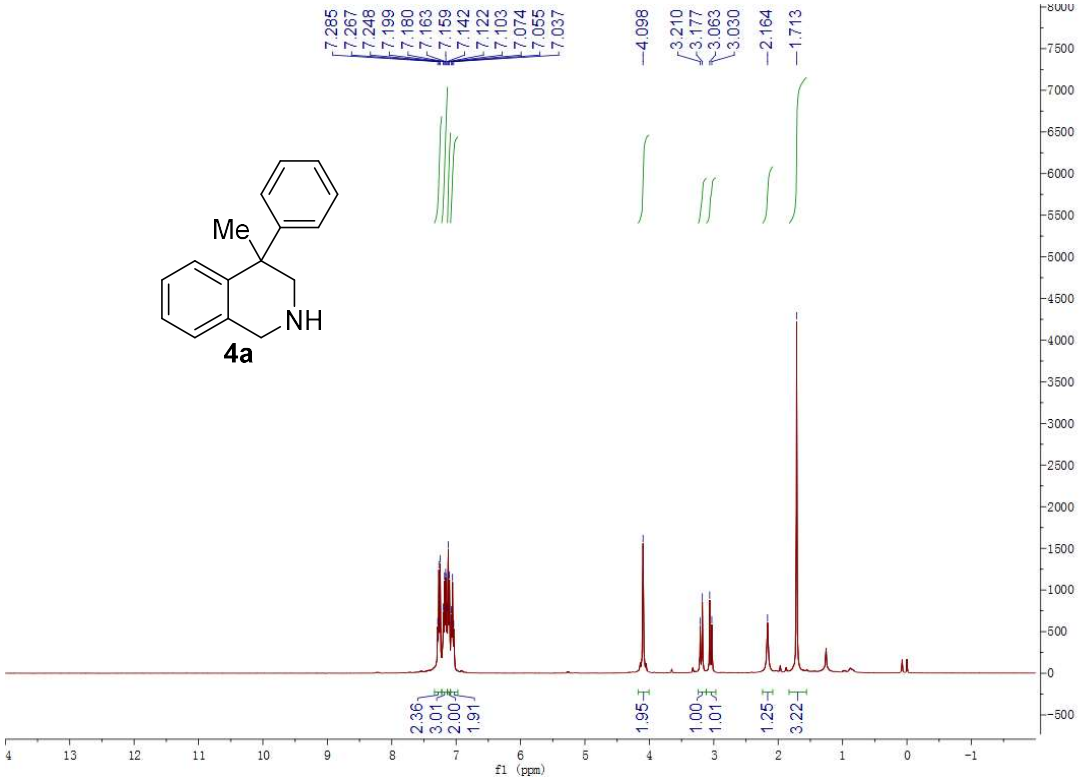
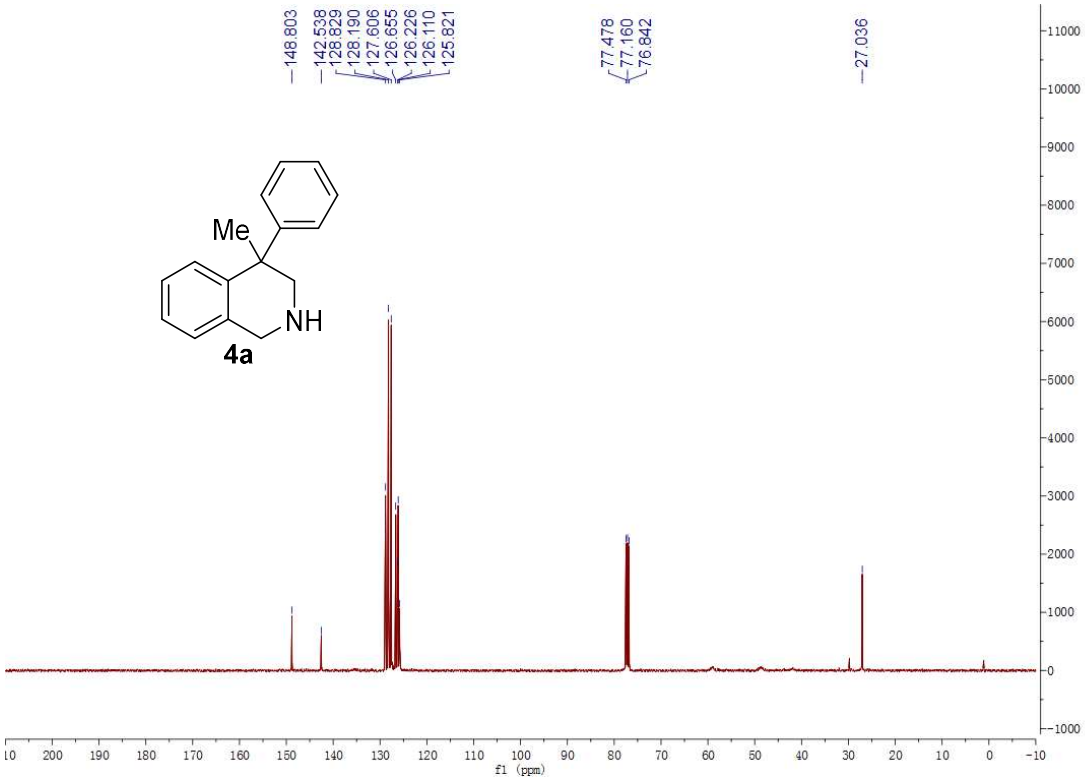
145.970
142.634
134.764
128.888
128.413
128.271
127.854
127.407
124.115
77.477
77.160
76.842
52.071
27.053
Me
O
NH
O
3a
f1 (ppm)


7.285
7.267
7.248
7.199
7.180
7.163
7.159
7.142
7.122
7.103
7.074
7.055
7.037
4.098
3.210
3.177
3.063
3.030
2.164
1.713
Me
NH
4a
2.36
3.01
2.00
1.91
1.95
1.00
1.01
1.25
3.22
f1 (ppm)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128.829
128.190
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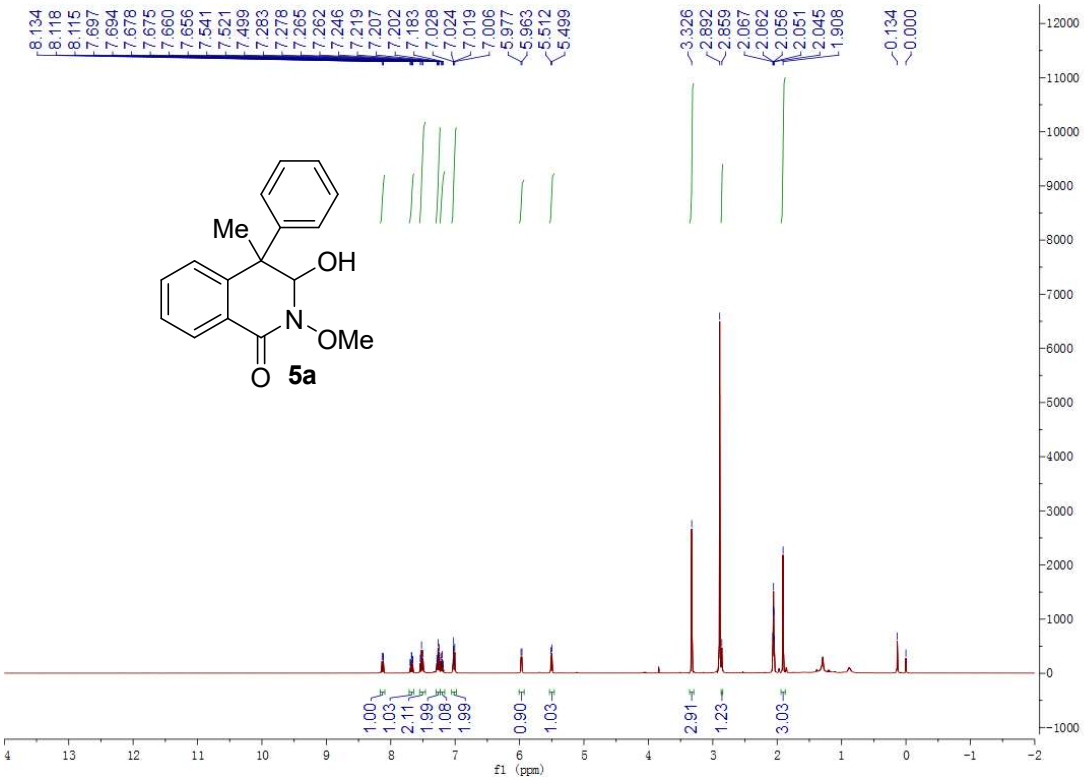
126.226
126.110
125.821
77.478
77.160
76.842
27.036
Me
NH
4a
f1 (ppm)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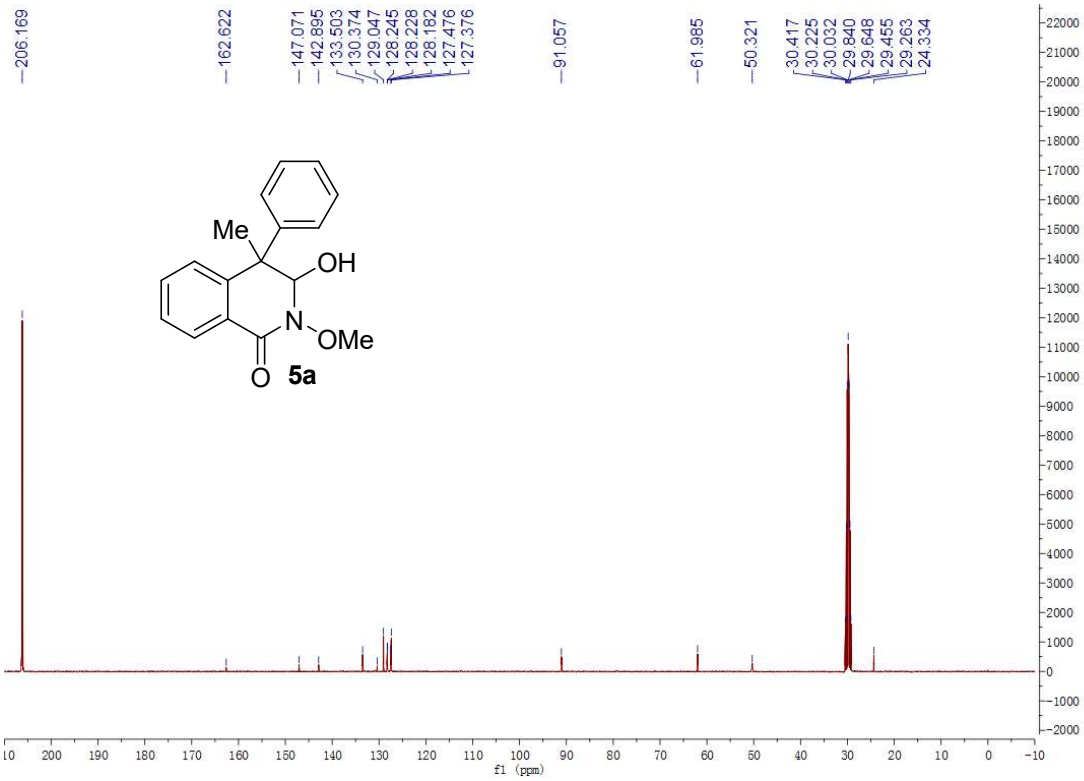
8.118
8.115
7.697
7.694
7.678
7.675
7.660
7.656
7.541
7.521
7.499
7.283
7.278
7.265
7.262
7.246
7.219
7.207
7.202
7.183
7.028
7.024
7.019
7.006
5.977
5.963
5.512
5.499
3.326
2.892
2.859
2.067
2.062
2.056
2.051
2.045
1.908
0.134
0.000
Me
OH
N
OMe
O
5a
1.00
1.03
2.11
1.99
1.08
1.99
0.90
1.03
2.91
1.23
3.03
f1 (ppm)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142.895
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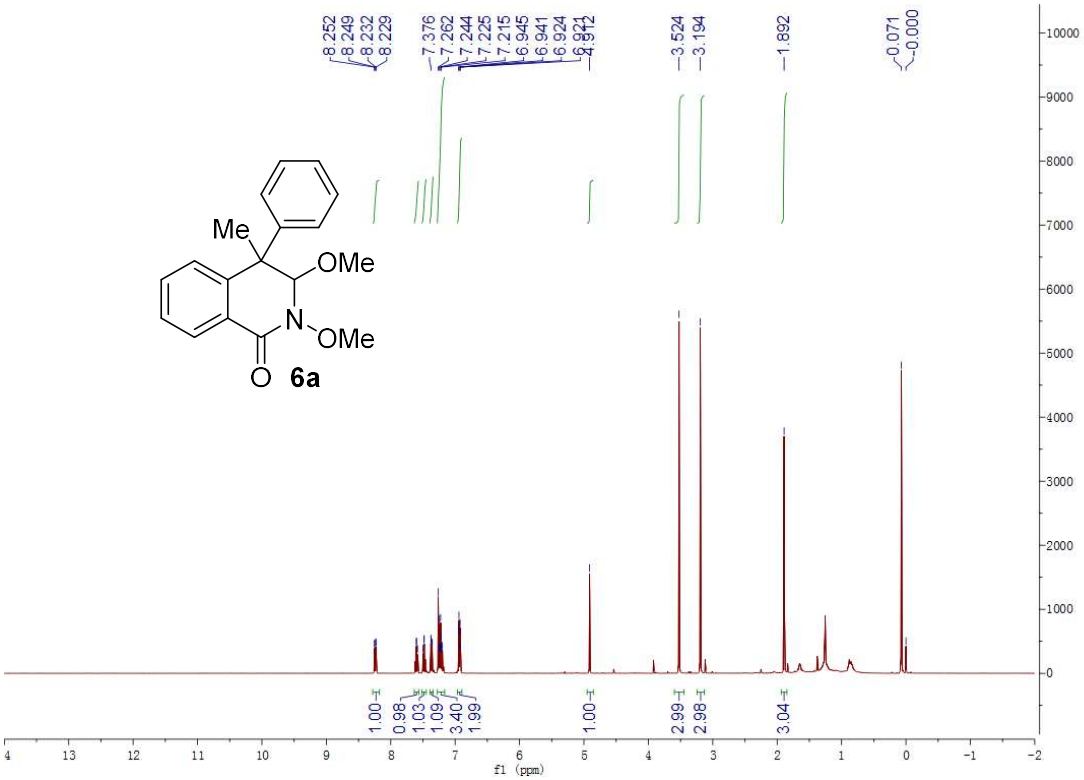
130.374
129.047
128.245
128.228
128.182
127.476
127.376
91.057
61.985
50.321
30.417
30.225
30.032
29.840
29.648
29.455
29.263
24.334
Me
OH
N
OMe
O
5a
f1 (ppm)


8.252
8.249
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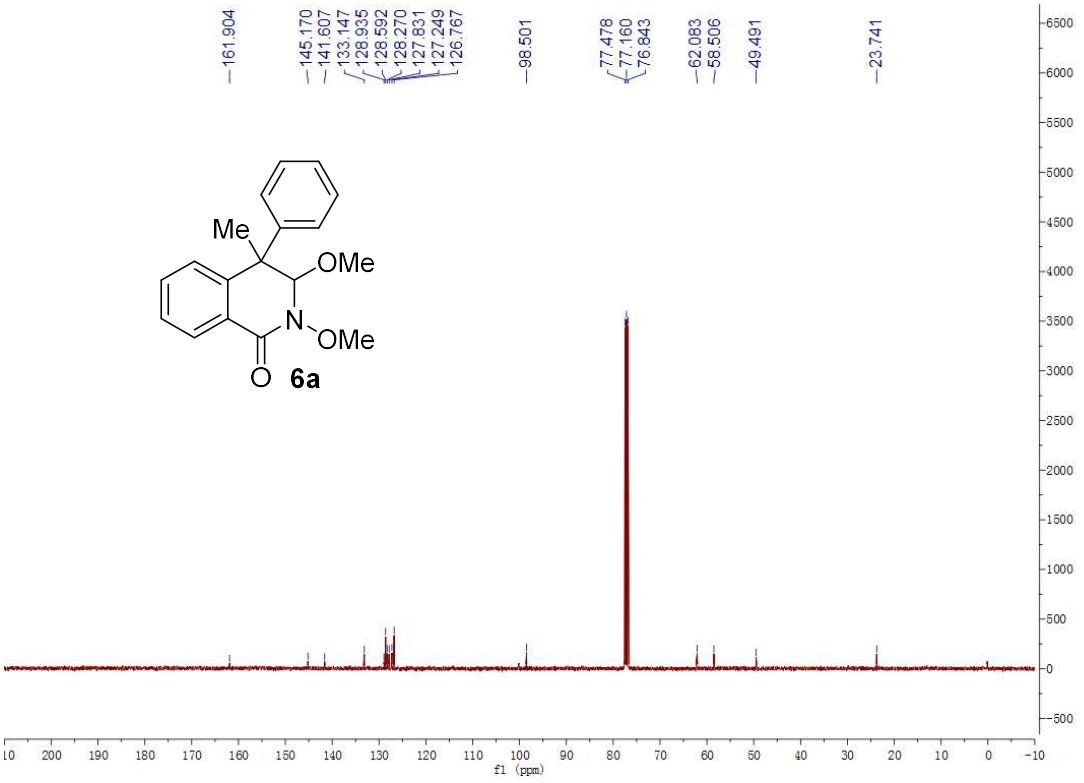
8.229
7.376
7.262
7.244
7.225
7.215
6.945
6.941
6.924
4.912
3.524
3.194
1.892
0.071
0.000
Me
OMe
N
OMe
O
6a
1.00
0.98
1.03
1.09
3.40
1.99
1.00
2.99
2.98
3.04
f1 (ppm)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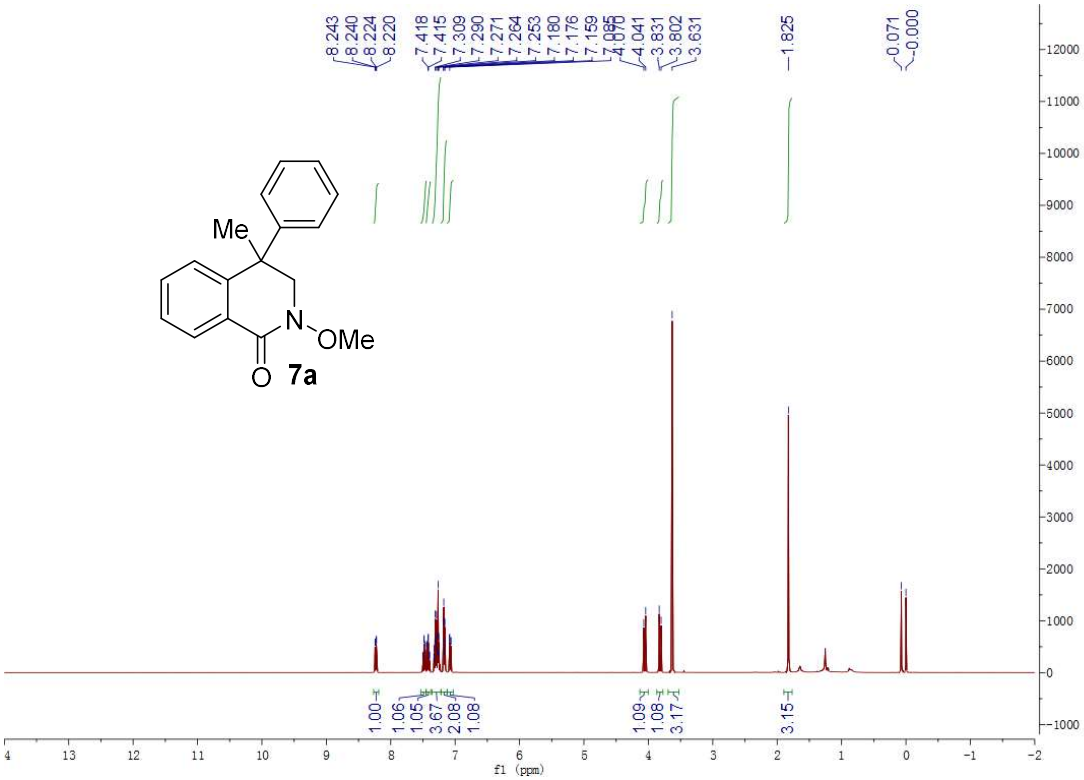
141.607
133.147
128.935
128.592
128.270
127.831
127.249
126.767
98.501
77.478
77.160
76.843
62.083
58.506
49.491
23.741
Me
OMe
N
OMe
O
6a
f1 (ppm)


8.243
8.240
8.224
8.220
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7.415
7.309
7.290
7.271
7.264
7.253
7.180
7.176
7.159
4.070
4.041
3.831
3.802
3.631
1.825
0.071
0.000
Me
N
OMe
O
7a
1.00
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1.05
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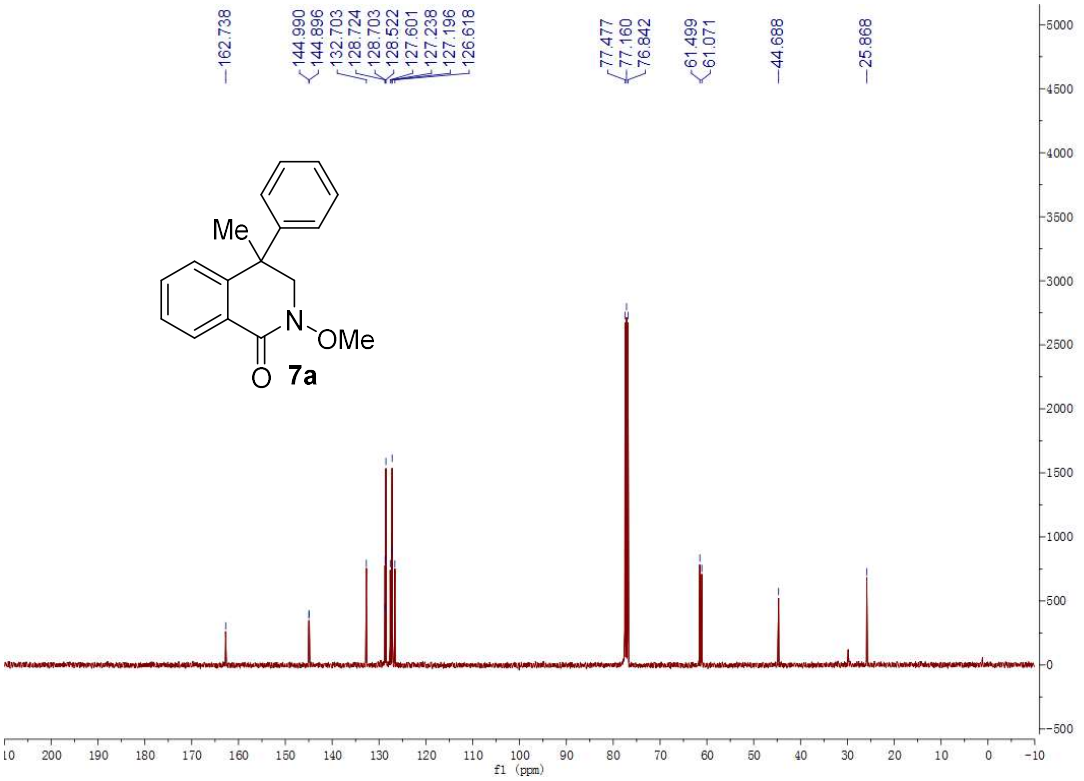
2.08
1.08
1.09
1.08
3.17
3.15
f1 (ppm)


162.738
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128.522
127.601
127.238
127.196
126.618
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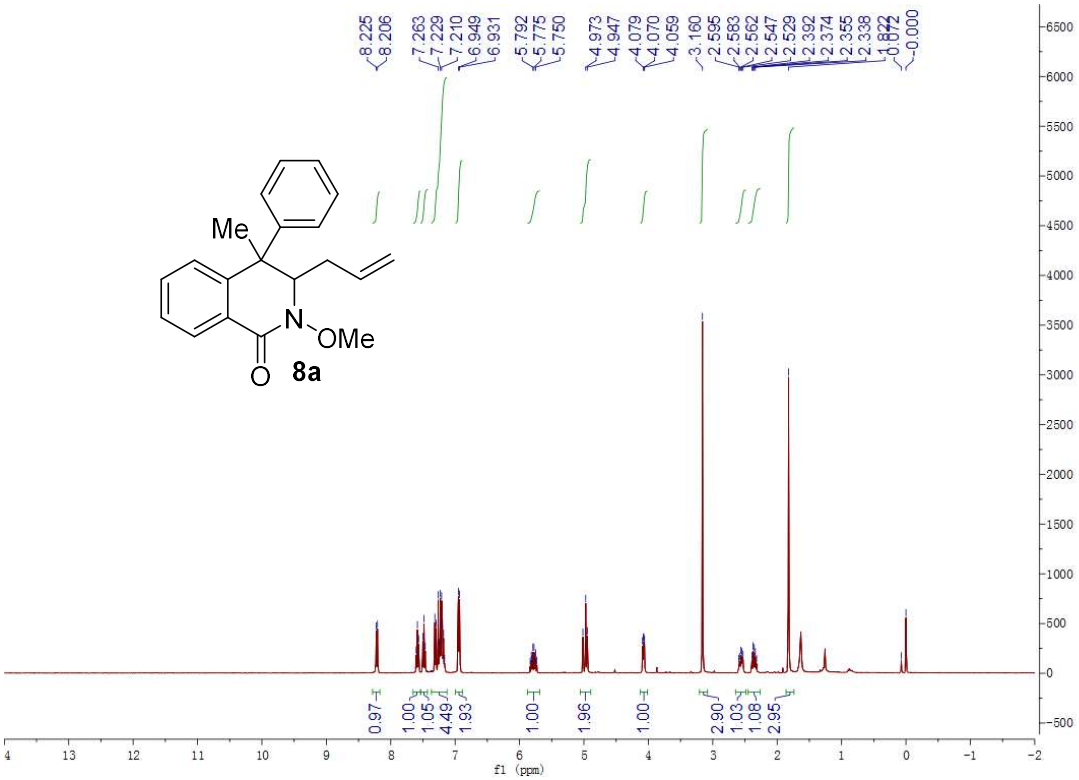
76.842
61.499
61.071
44.688
25.868
Me
N
OMe
O
7a
f1 (ppm)


8.225
8.206
7.263
7.229
7.210
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6.931
5.792
5.775
5.750
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4.947
4.079
4.070
4.059
3.160
2.595
2.583
2.562
2.547
2.529
2.392
2.374
2.355
2.338
1.822
0.872
0.000
Me
N
OMe
O
8a
0.97
1.00
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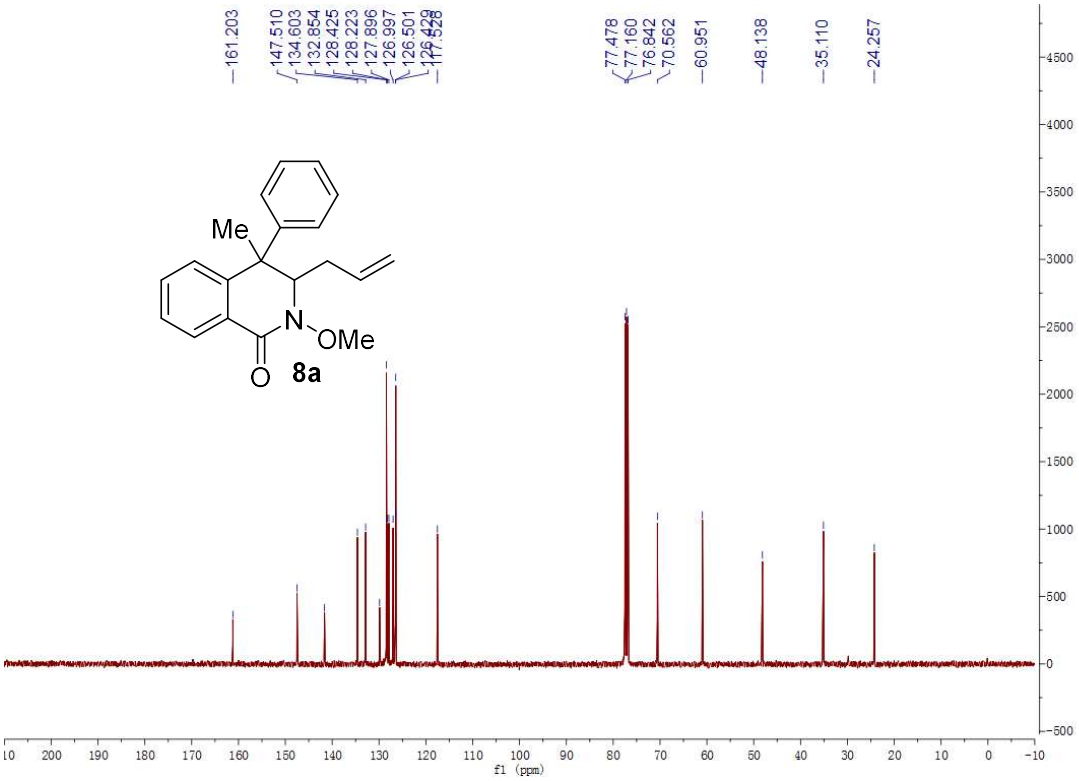
1.93
1.00
1.96
1.00
2.90
1.03
1.08
2.95
f1 (ppm)


161.203
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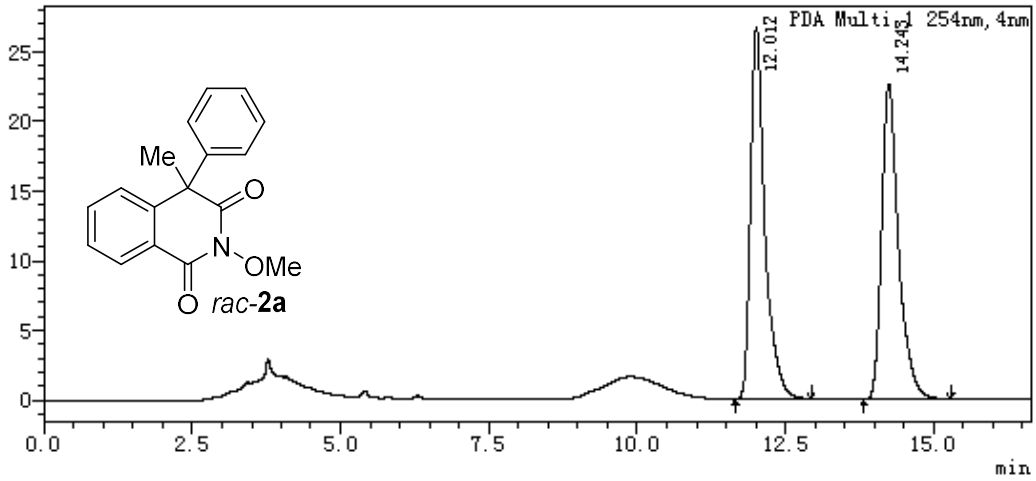
128.425
128.223
127.896
126.997
126.501
77.478
77.160
76.842
70.562
60.951
48.138
35.110
24.257
Me
N
OMe
O
8a
f1 (ppm)

Chiral HPLC Data

Chromatography

mAU



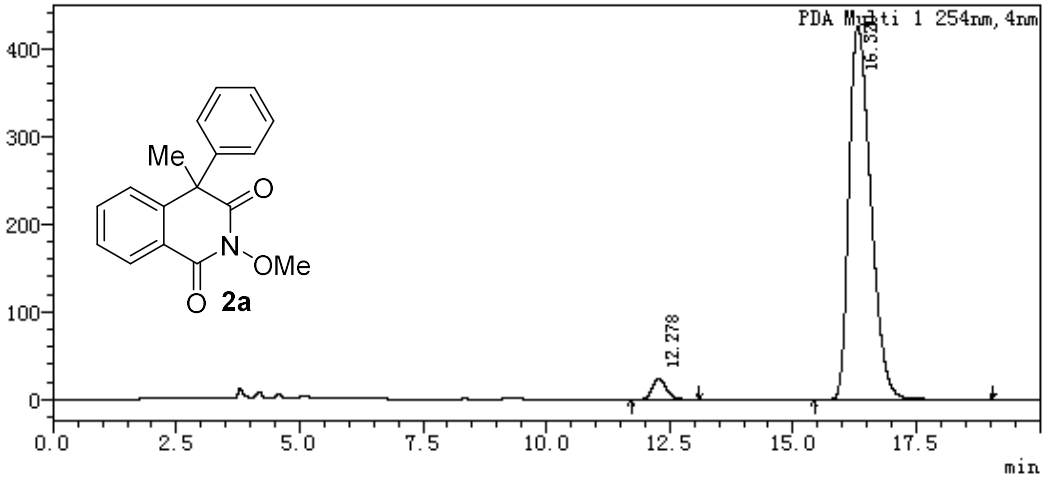
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	12.012	26728	54.127	448429	49.976
2	14.243	22652	45.873	448859	50.024
总计		49380	100.000	897287	100.000

Chromatography

mAU



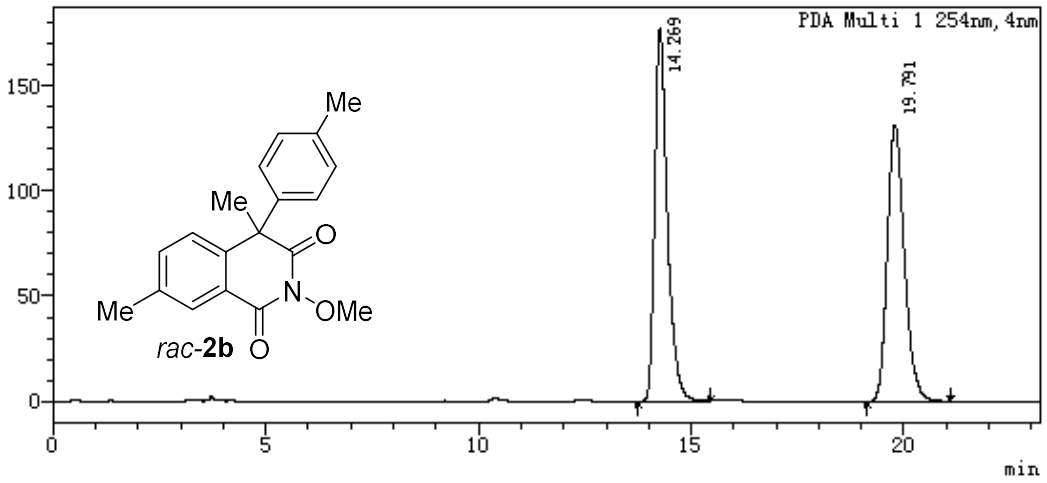
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	12.278	24079	5.351	489522	3.613
2	16.320	425893	94.649	13061208	96.387
总计		449972	100.000	13550730	100.000

Chromatography

mAU



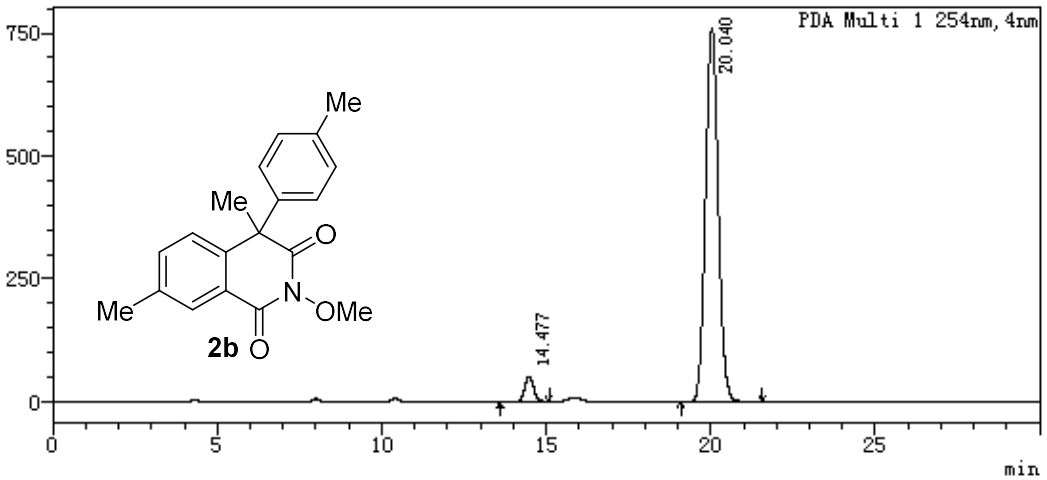
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	14.269	177498	57.433	3663039	50.008
2	19.791	131556	42.567	3661835	49.992
总计		309053	100.000	7324874	100.000

Chromatography

mAU



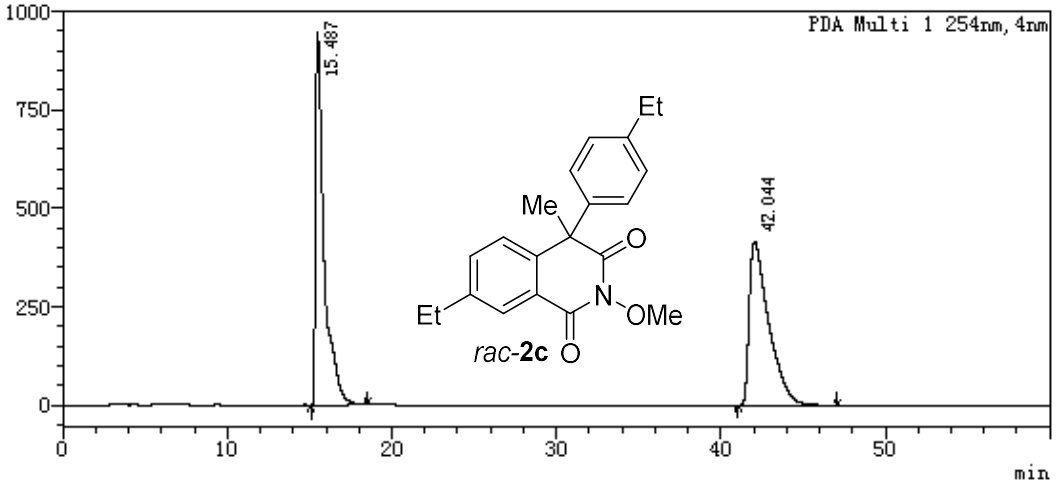
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	14.477	51394	6.330	967434	4.526
2	20.040	760484	93.670	20408739	95.474
总计		811878	100.000	21376174	100.000

Chromatography

mAU



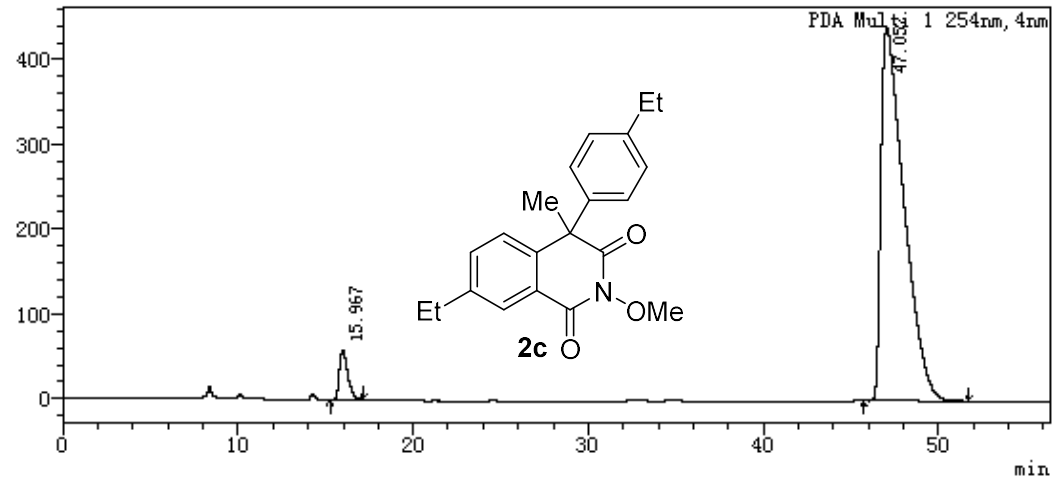
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.487	947391	69.487	33773119	49.537
2	42.044	416014	30.513	34403811	50.463
总计		1363405	100.000	68176930	100.000

Chromatography

mAU



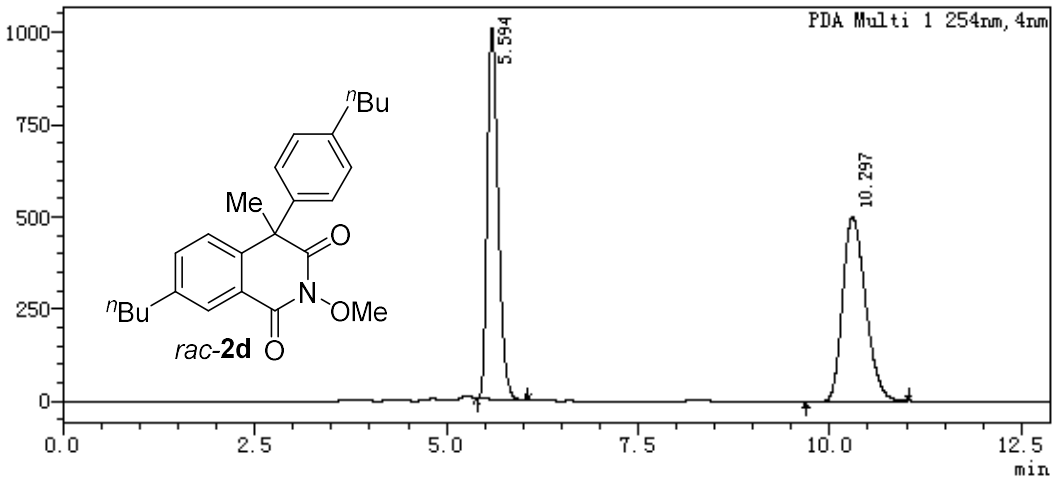
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.967	58290	11.703	1969173	4.660
2	47.052	439798	88.297	40289206	95.340
总计		498088	100.000	42258380	100.000

Chromatography

mAU



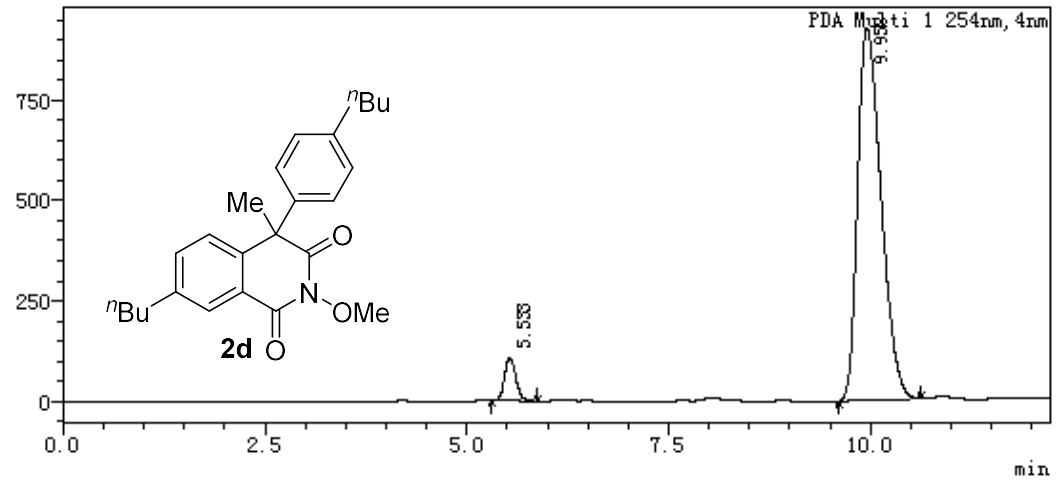
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	5.594	1005259	66.846	10120894	49.358
2	10.297	498589	33.154	10384343	50.642
总计		1503848	100.000	20505237	100.000

Chromatography

mAU



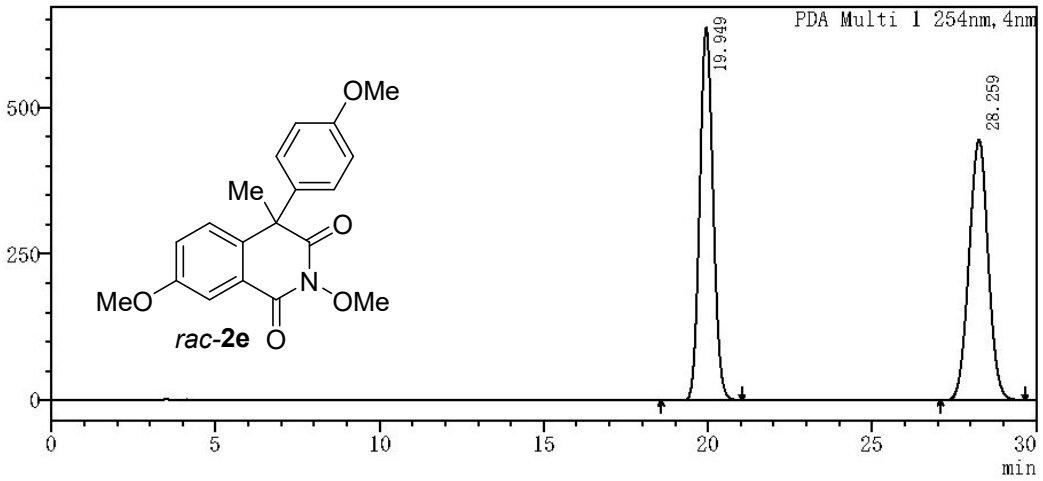
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	5.533	108332	10.459	1016570	5.162
2	9.958	927410	89.541	18676373	94.838
总计		1035743	100.000	19692944	100.000

Chromatography

mAU



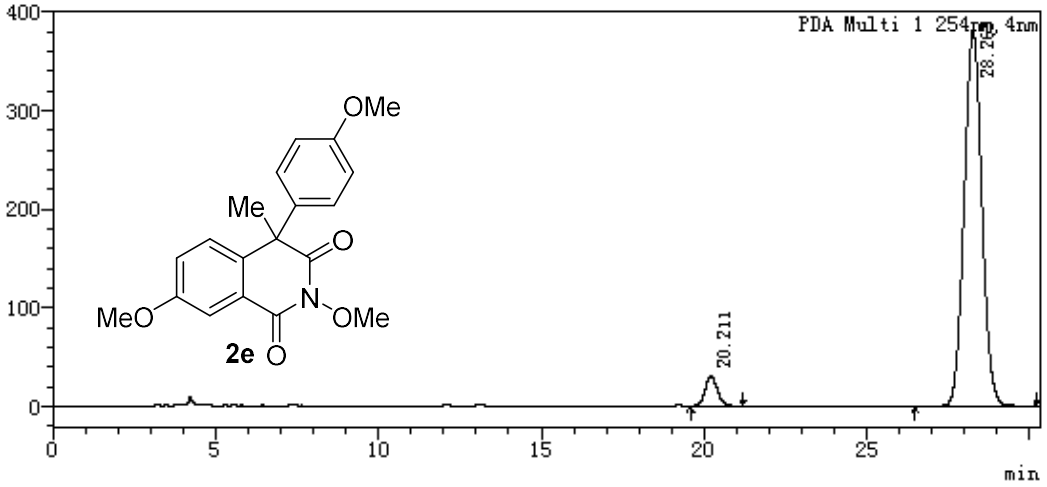
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	19.949	635736	58.832	17975106	49.957
2	28.259	444853	41.168	18005952	50.043
总计		1080589	100.000	35981058	100.000

Chromatography

mAU



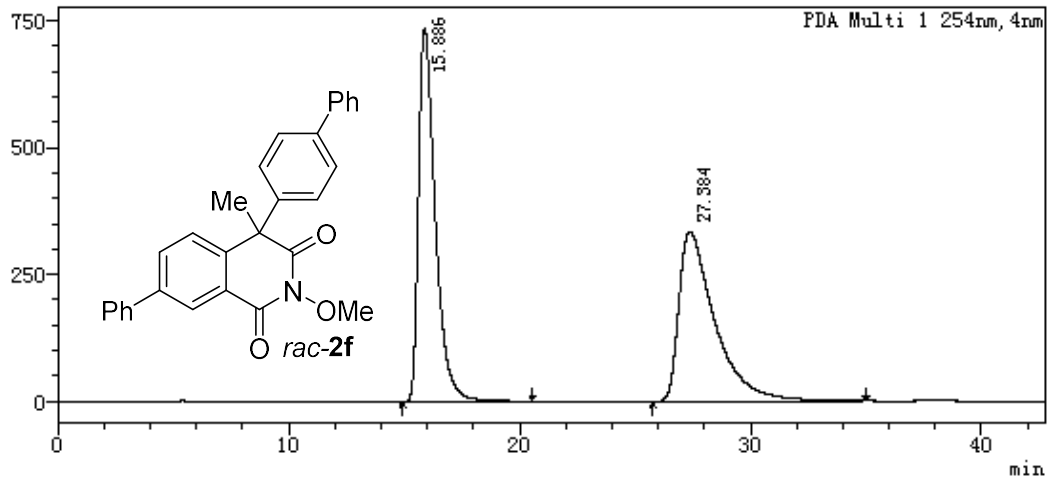
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	20.211	30617	7.465	793116	5.245
2	28.263	379538	92.535	14328078	94.755
总计		410155	100.000	15121193	100.000

Chromatography

mAU



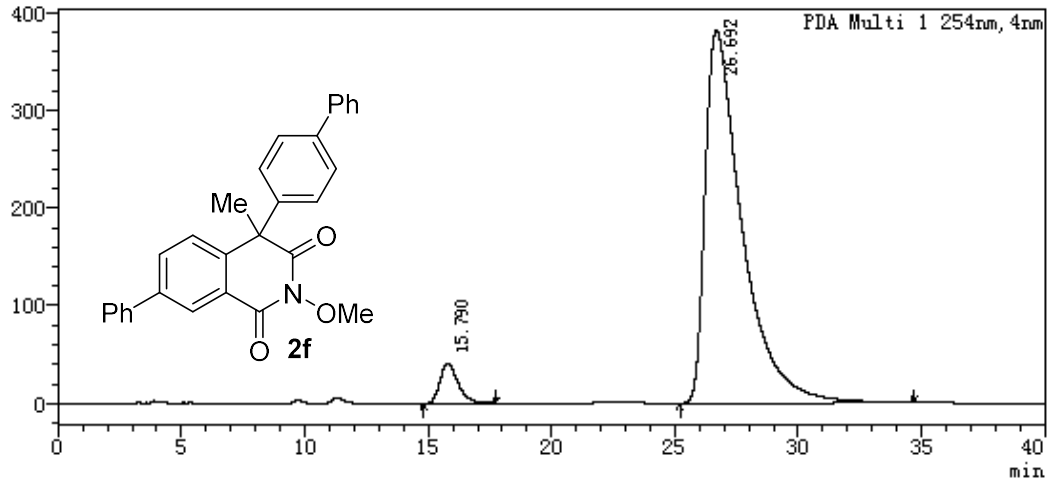
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.886	735587	68.736	36976535	50.445
2	27.384	334569	31.264	36324387	49.555
总计		1070156	100.000	73300923	100.000

Chromatography

mAU



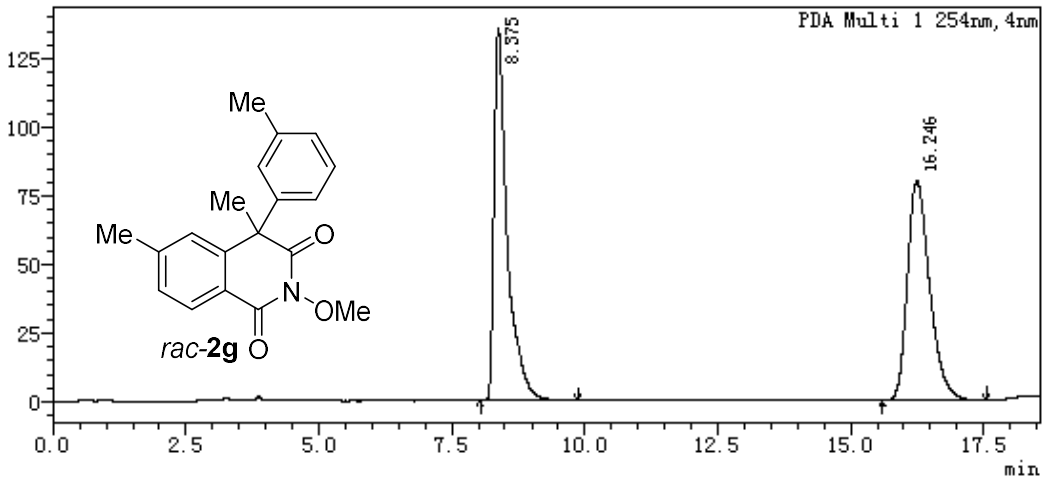
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.790	41416	9.777	2073750	4.943
2	26.692	382200	90.223	39877797	95.057
总计		423616	100.000	41951547	100.000

Chromatography

mAU



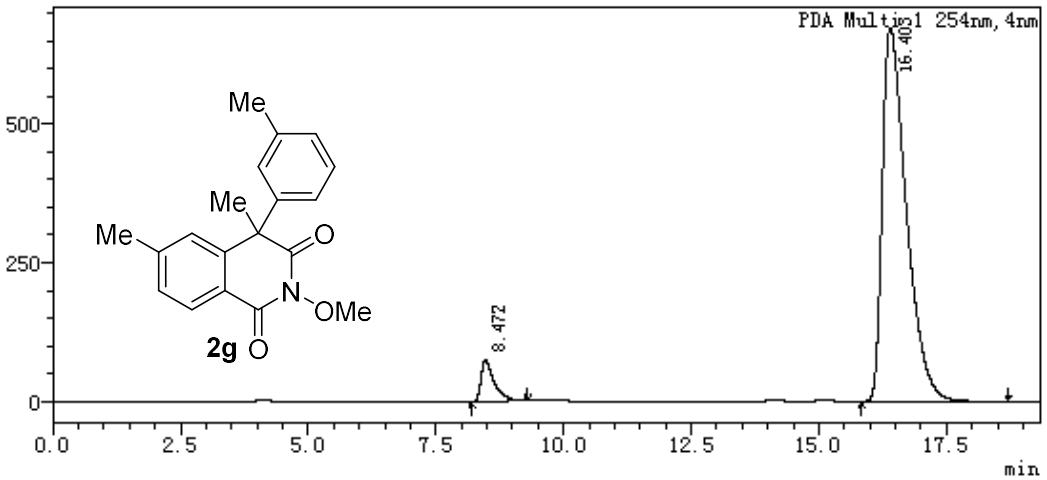
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	8.375	135927	62.914	2352746	50.154
2	16.246	80125	37.086	2338278	49.846
总计		216052	100.000	4691023	100.000

Chromatography

mAU



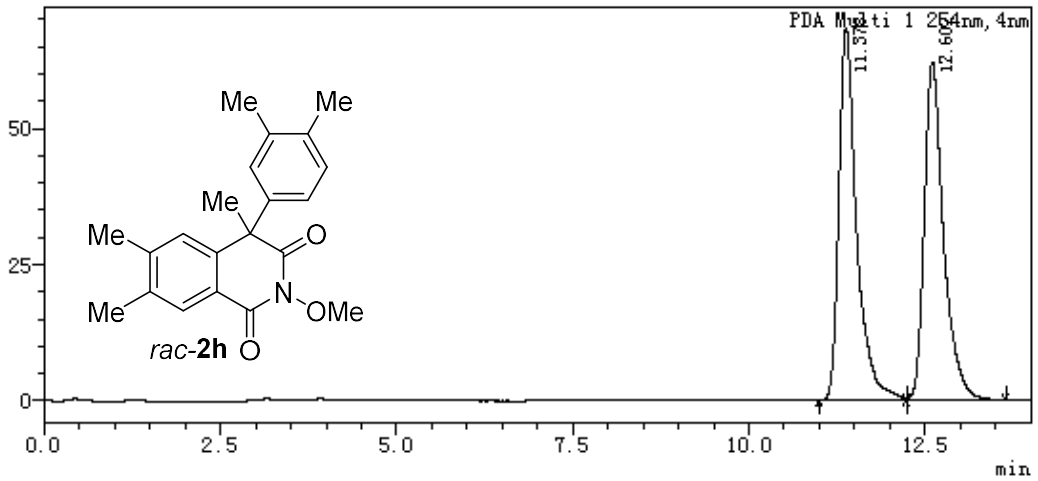
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	8.472	74578	9.979	1327839	5.688
2	16.403	672807	90.021	22018609	94.312
总计		747386	100.000	23346447	100.000

Chromatography

mAU



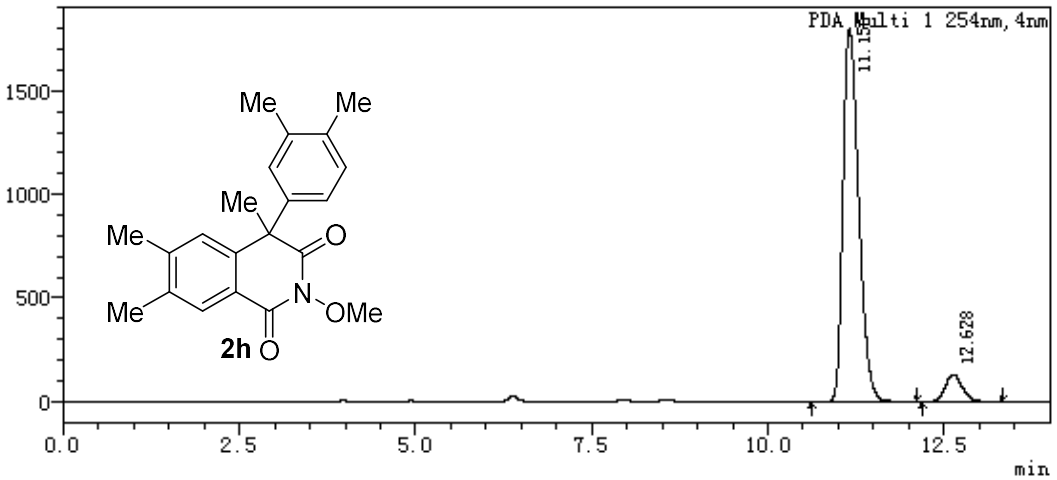
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	11.378	68303	52.394	1194430	50.296
2	12.602	62061	47.606	1180386	49.704
总计		130364	100.000	2374816	100.000

Chromatography

mAU



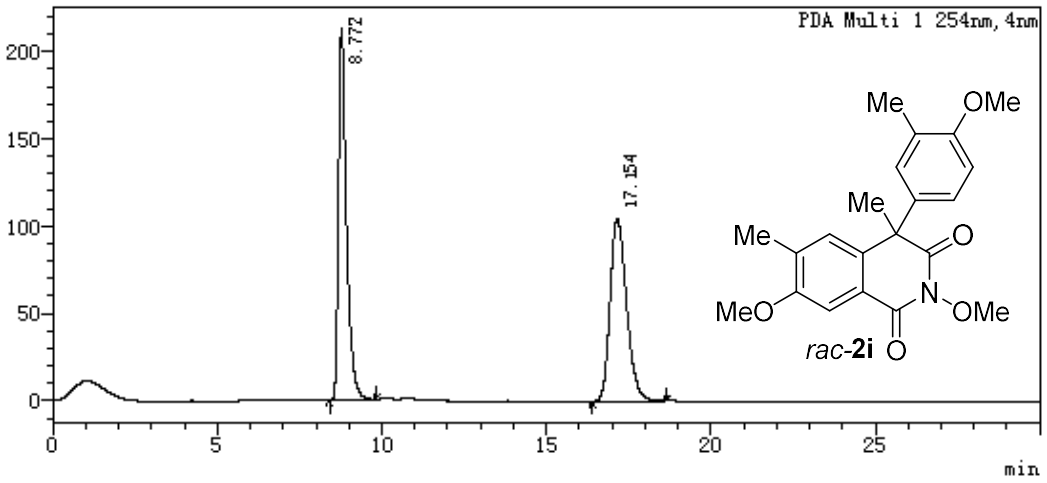
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	11.156	1801867	93.244	27922425	92.817
2	12.628	130564	6.756	2160784	7.183
总计		1932431	100.000	30083208	100.000

Chromatography

mAU



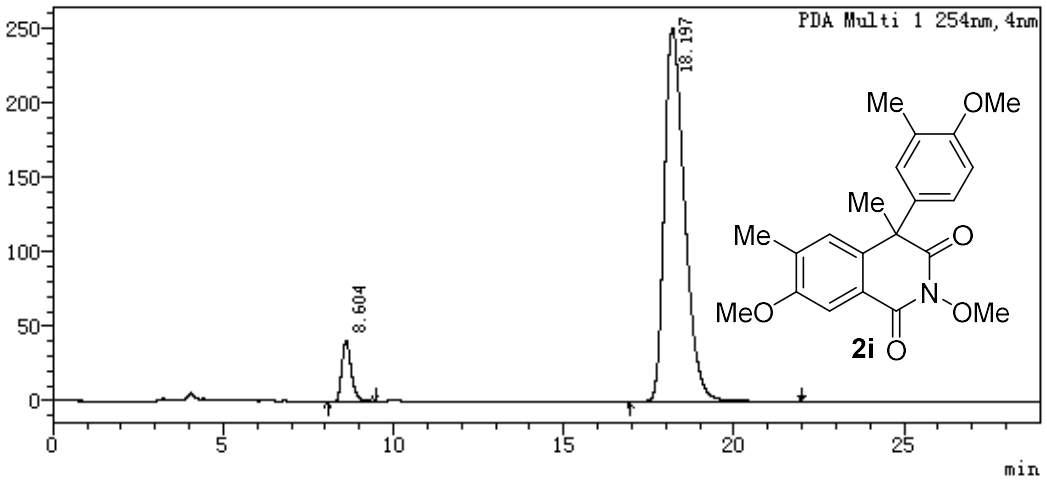
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	8.772	213219	67.027	3628938	50.022
2	17.154	104891	32.973	3625702	49.978
总计		318110	100.000	7254640	100.000

Chromatography

mAU



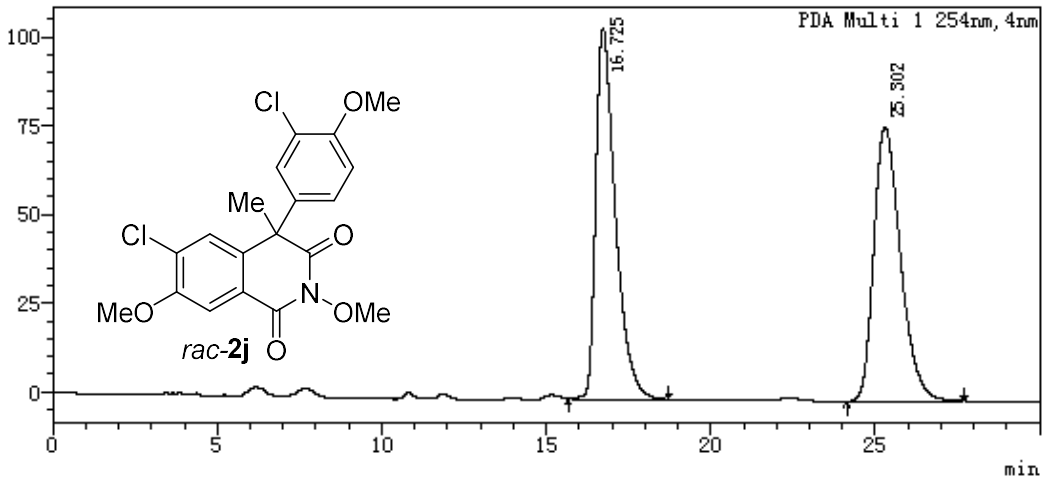
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	8.604	40700	13.964	766697	6.899
2	18.197	250775	86.036	10346496	93.101
总计		291476	100.000	11113193	100.000

Chromatography

mAU



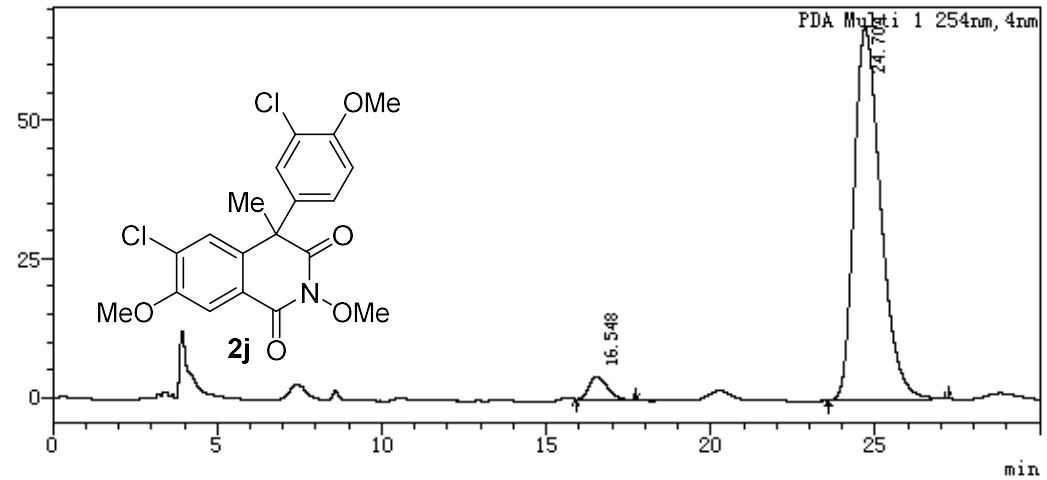
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	16.725	104654	57.503	4339283	49.808
2	25.302	77344	42.497	4372761	50.192
总计		181998	100.000	8712044	100.000

Chromatography

mAU



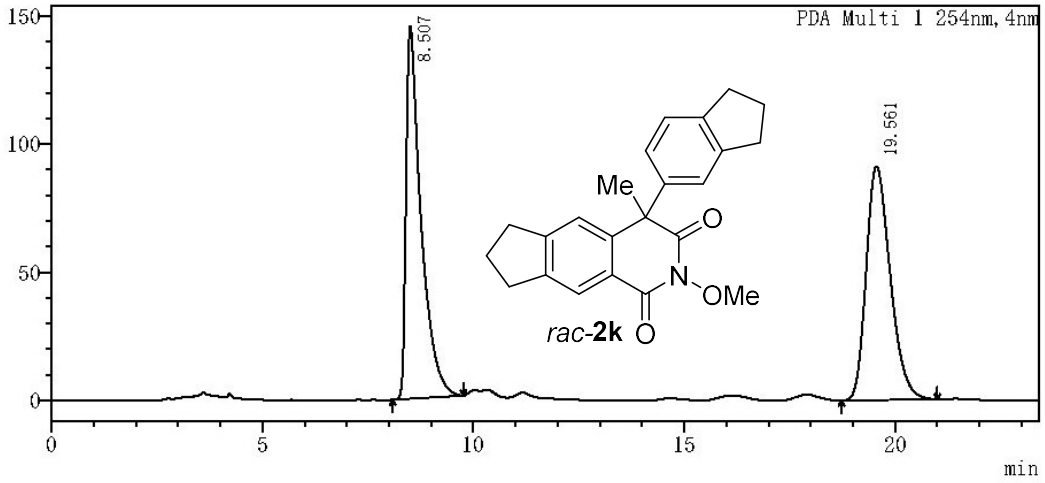
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	16.548	4181	5.862	167162	4.226
2	24.704	67148	94.138	3788355	95.774
总计		71329	100.000	3955517	100.000

Chromatography

mAU



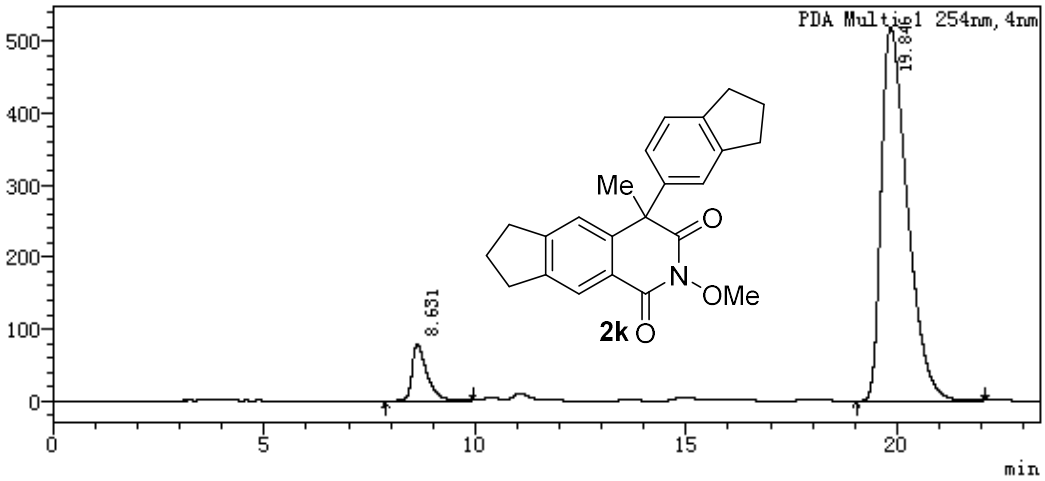
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	8.507	145257	61.443	3593626	49.791
2	19.561	91152	38.557	3623807	50.209
总计		236409	100.000	7217433	100.000

Chromatography

mAU



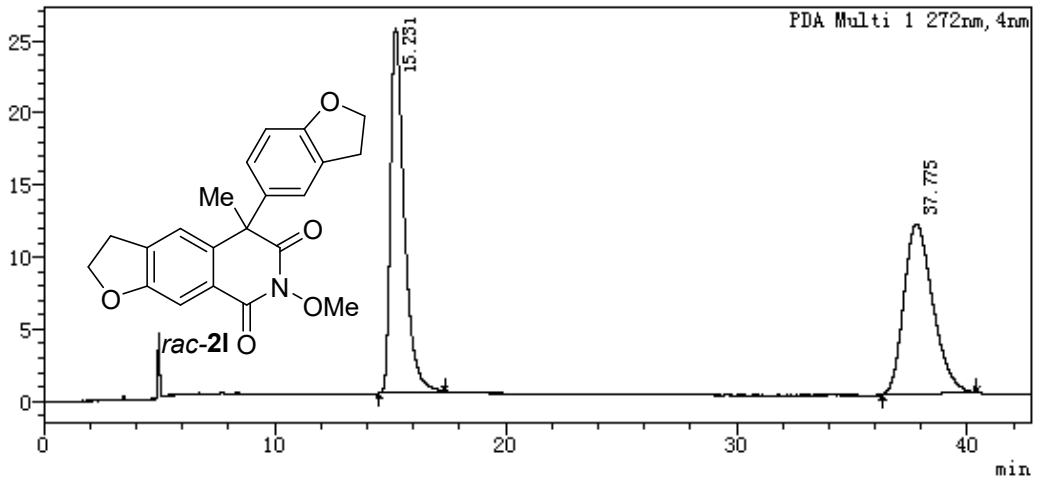
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	8.631	79013	13.213	1951307	8.006
2	19.846	518978	86.787	22421496	91.994
总计		597991	100.000	24372803	100.000

Chromatography

mAU



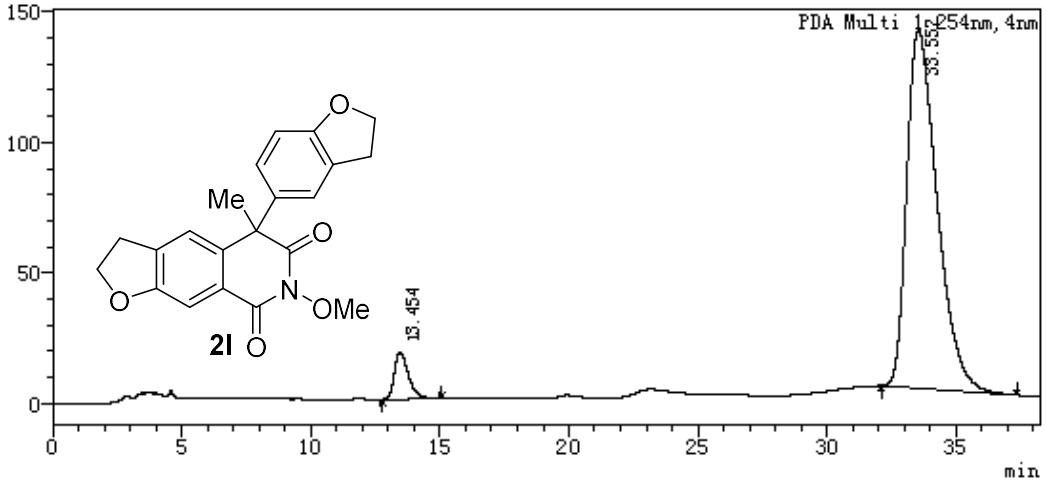
Table

PDA Ch1 272nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.231	25308	68.229	1042661	50.355
2	37.775	11785	31.771	1027941	49.645
总计		37094	100.000	2070602	100.000

Chromatography

mAU



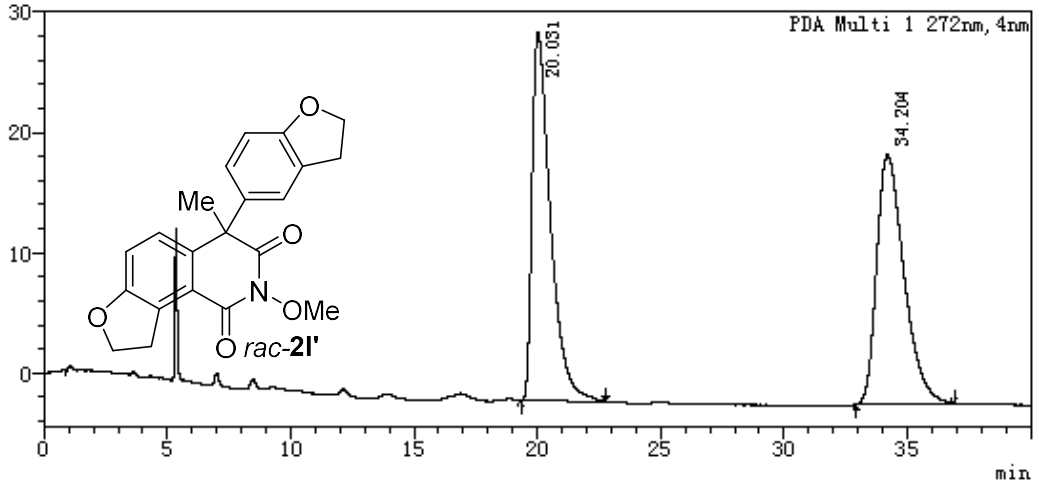
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	13.454	18093	11.651	691508	5.792
2	33.552	137204	88.349	11246797	94.208
总计		155298	100.000	11938305	100.000

Chromatography

mAU



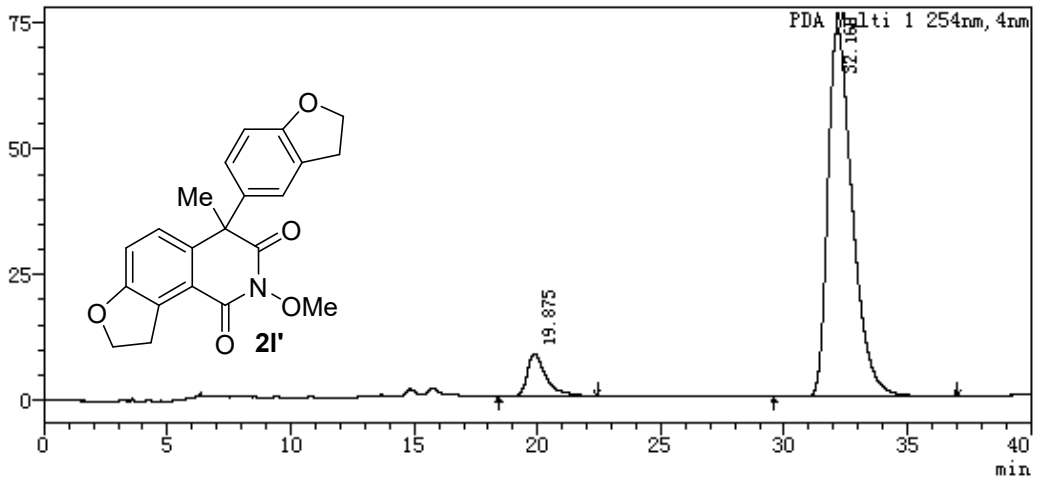
Table

PDA Ch1 272nm

Number	Retention Time	Height	Height%	Area	Area%
1	20.031	30676	59.694	1592917	49.936
2	34.204	20713	40.306	1597017	50.064
总计		51388	100.000	3189934	100.000

Chromatography

mAU



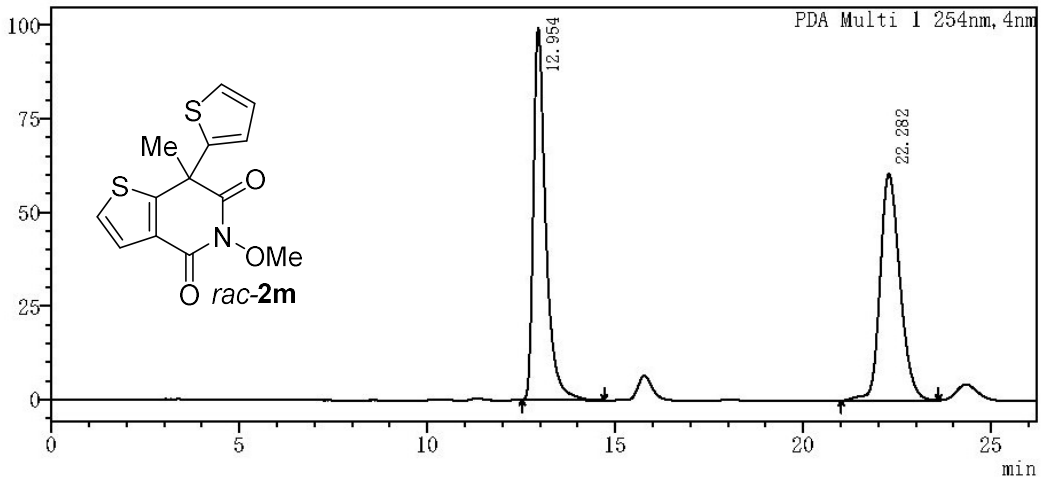
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	19.875	8294	10.166	426622	7.726
2	32.160	73291	89.834	5095240	92.274
总计		81585	100.000	5521862	100.000

Chromatography

mAU



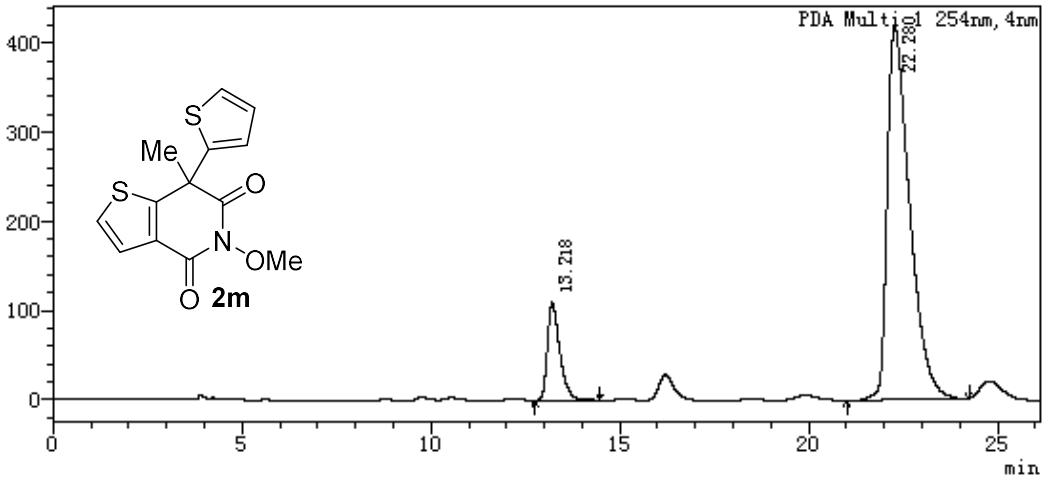
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	12.954	99287	62.117	2260542	50.456
2	22.282	60551	37.883	2219647	49.544
总计		159838	100.000	4480189	100.000

Chromatography

mAU



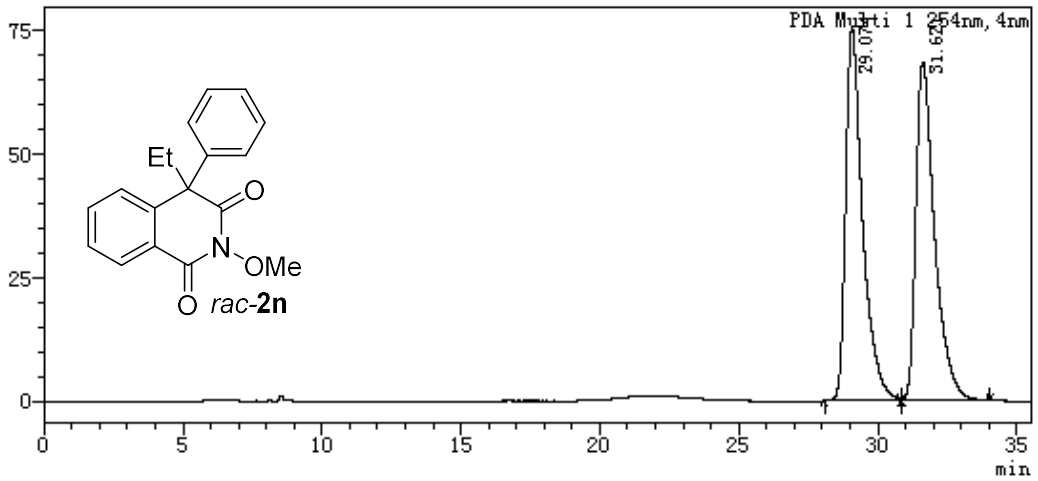
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	13.218	109626	20.760	2505698	12.686
2	22.280	418434	79.240	17245995	87.314
总计		528060	100.000	19751693	100.000

Chromatography

mAU



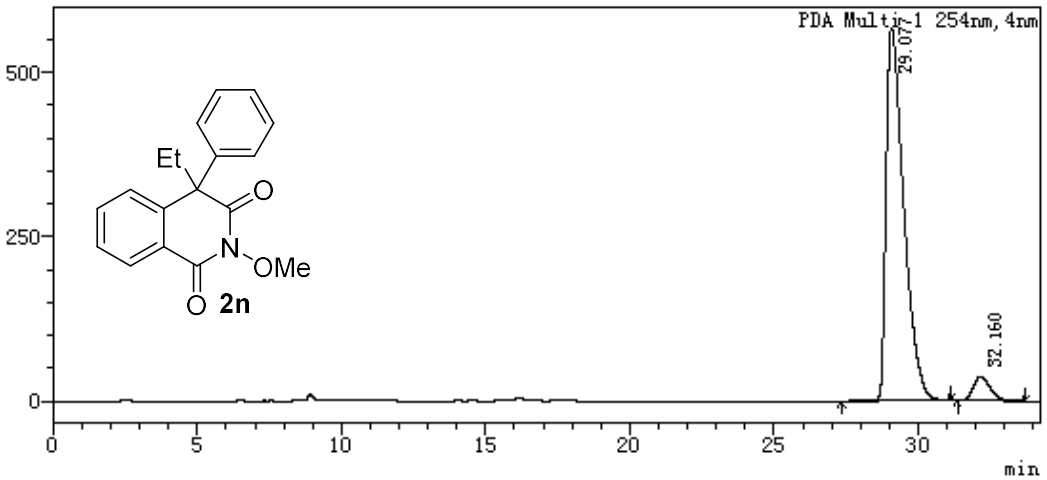
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	29.074	75302	52.390	3223085	49.951
2	31.621	68430	47.610	3229459	50.049
总计		143733	100.000	6452544	100.000

Chromatography

mAU



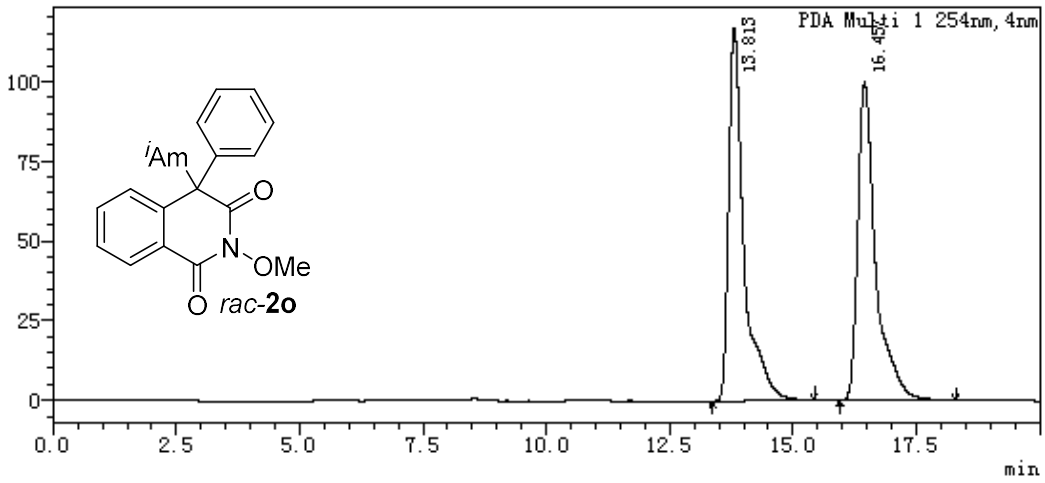
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	29.077	567312	93.897	24136628	94.097
2	32.160	36873	6.103	1514198	5.903
总计		604185	100.000	25650825	100.000

Chromatography

mAU



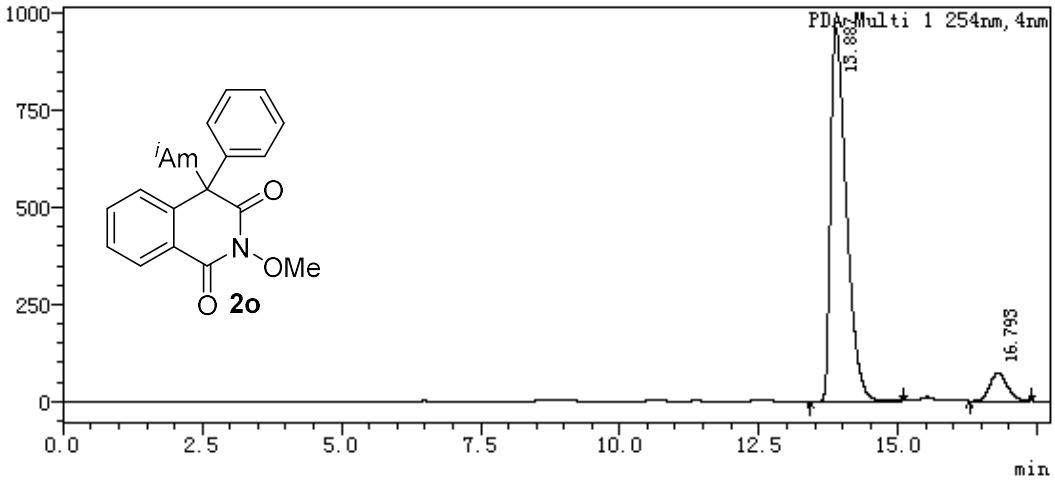
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	13.813	117086	53.974	2543885	49.926
2	16.457	99846	46.026	2551389	50.074
总计		216931	100.000	5095274	100.000

Chromatography

mAU



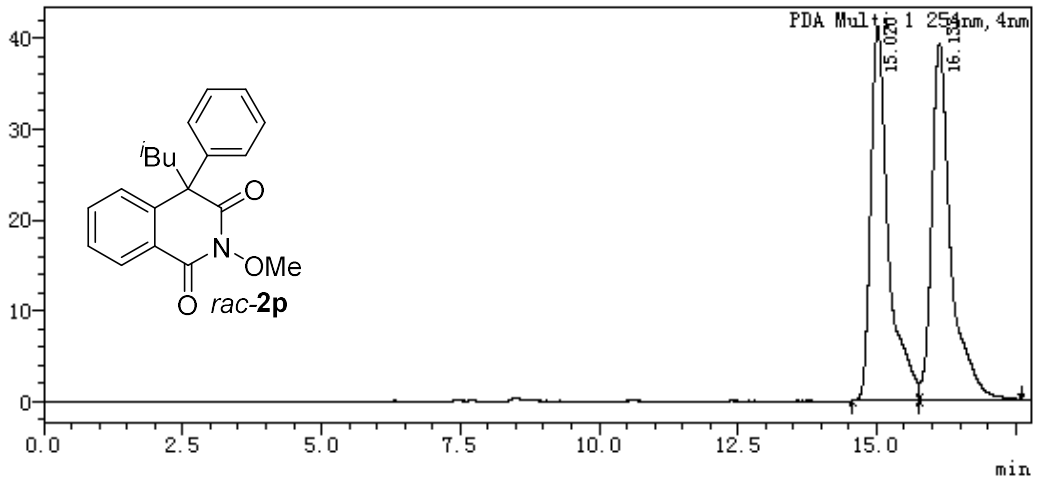
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	13.887	961743	92.945	18936789	92.296
2	16.793	73000	7.055	1580576	7.704
总计		1034742	100.000	20517365	100.000

Chromatography

mAU



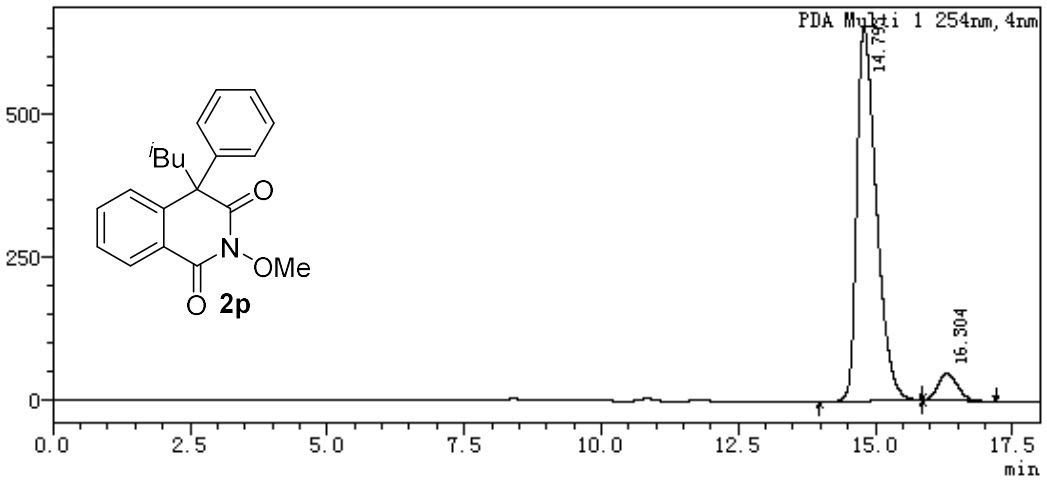
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.020	41061	51.101	913240	49.140
2	16.133	39293	48.899	945222	50.860
总计		80354	100.000	1858463	100.000

Chromatography

mAU



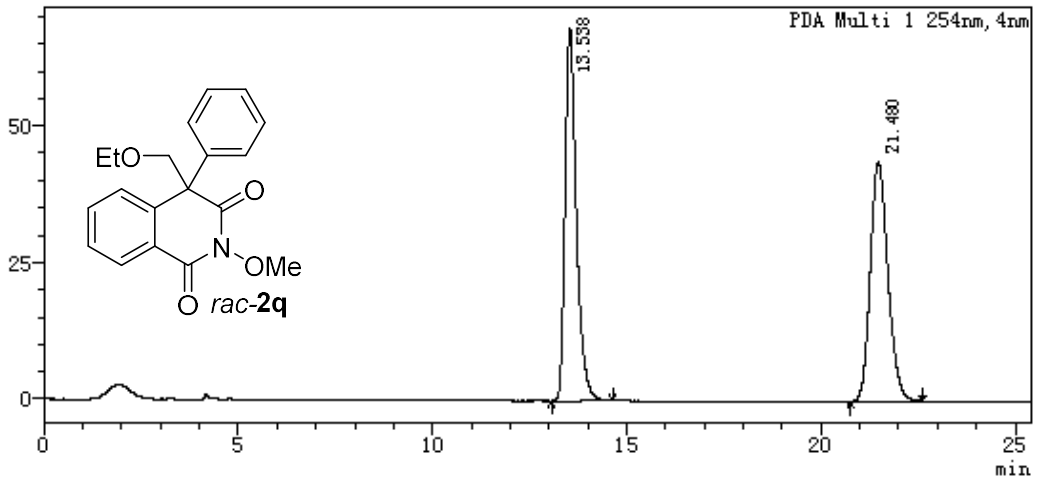
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	14.793	650428	93.147	16210142	93.239
2	16.304	47854	6.853	1175390	6.761
总计		698282	100.000	17385532	100.000

Chromatography

mAU



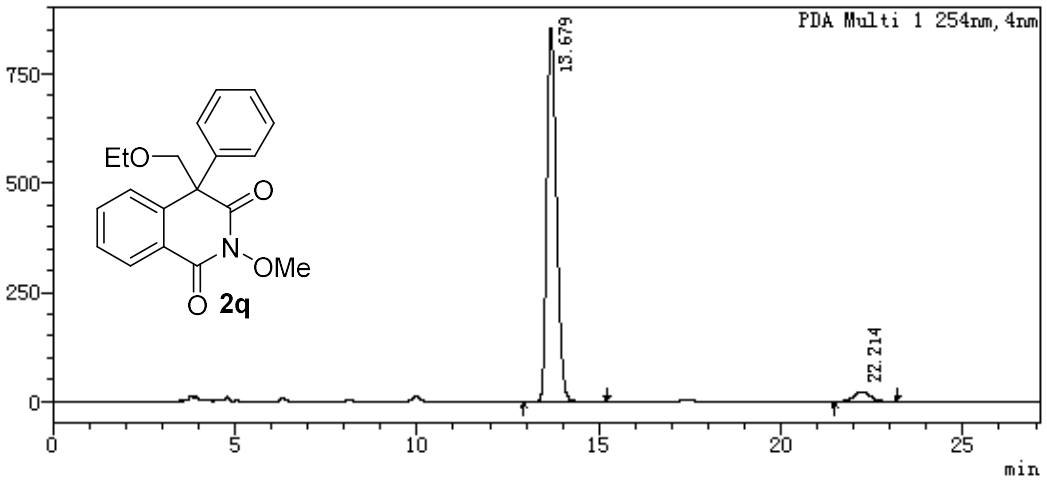
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	13.538	68246	60.972	1376769	50.039
2	21.480	43683	39.028	1374608	49.961
总计		111929	100.000	2751377	100.000

Chromatography

mAU



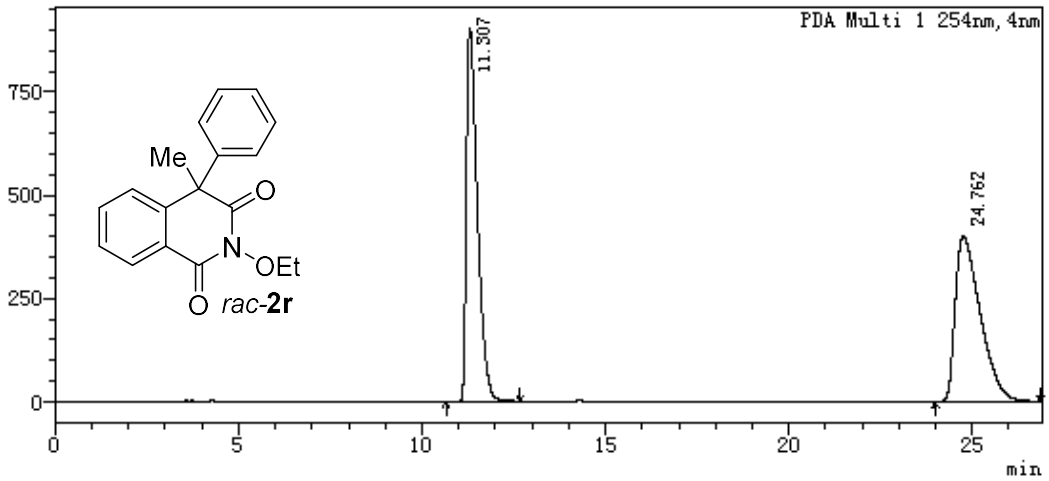
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	13.679	854572	97.500	15780836	95.818
2	22.214	21913	2.500	688690	4.182
总计		876485	100.000	16469526	100.000

Chromatography

mAU



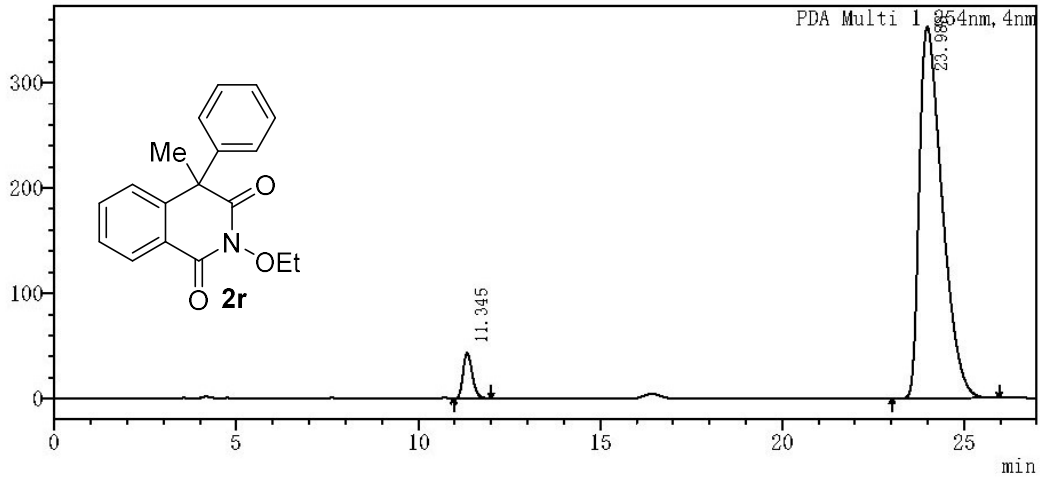
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	11.307	904632	69.211	18760134	49.681
2	24.762	402435	30.789	19000898	50.319
总计		1307067	100.000	37761032	100.000

Chromatography

mAU



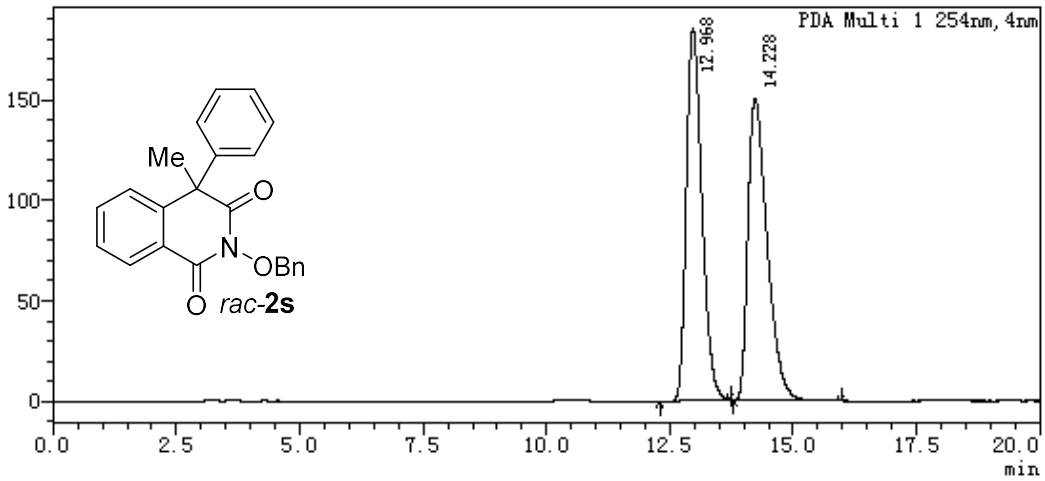
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	11.345	43382	10.952	769411	4.762
2	23.988	352713	89.048	15386362	95.238
总计		396095	100.000	16155773	100.000

Chromatography

mAU



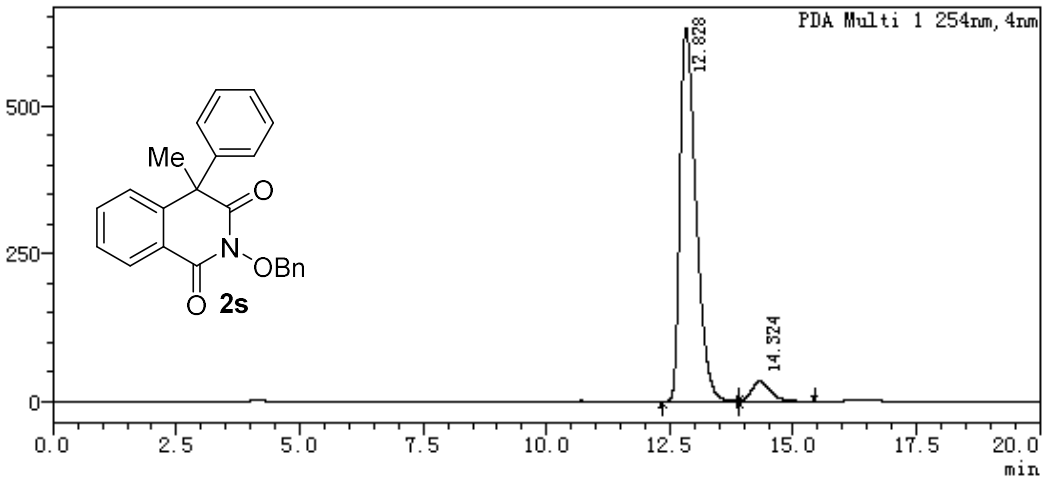
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	12.968	185496	55.224	4055201	49.960
2	14.228	150402	44.776	4061625	50.040
总计		335898	100.000	8116826	100.000

Chromatography

mAU



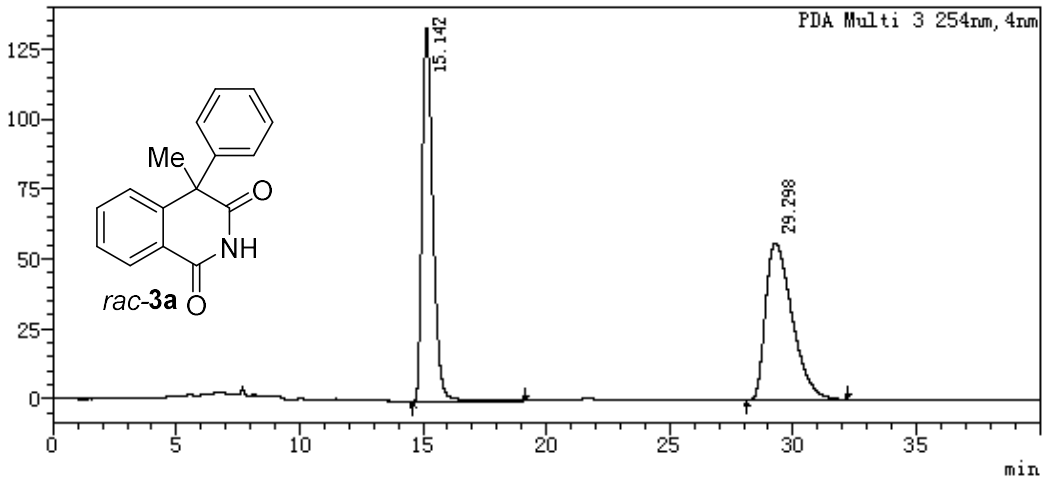
Table

PDA Ch1 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	12.828	631649	94.833	14318809	93.946
2	14.324	34417	5.167	922708	6.054
总计		666067	100.000	15241517	100.000

Chromatography

mAU



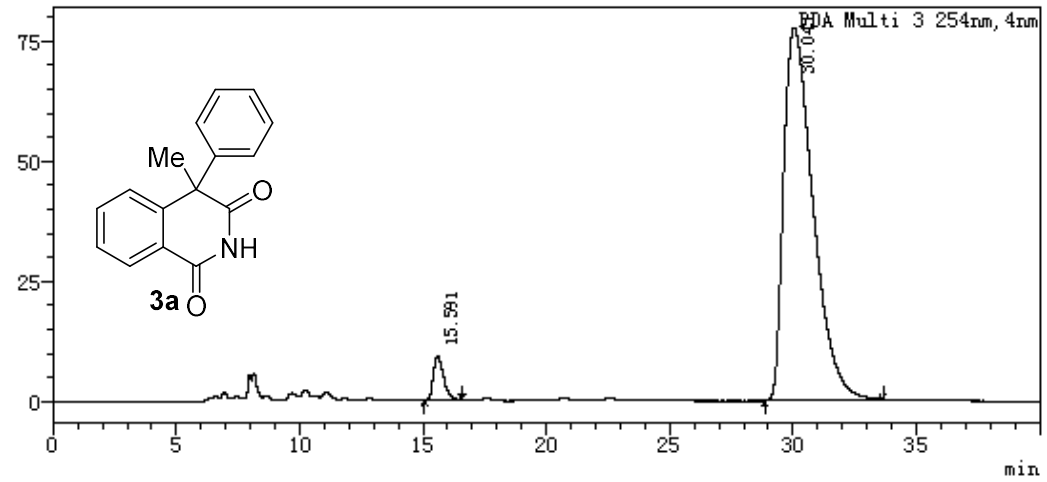
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.142	133589	70.307	4081574	48.871
2	29.298	56419	29.693	4270172	51.129
总计		190008	100.000	8351746	100.000

Chromatography

mAU



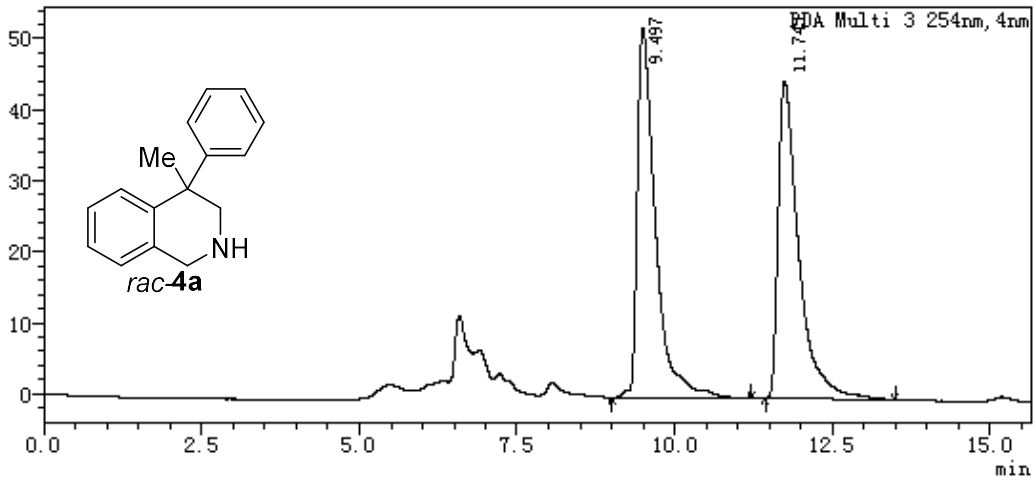
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.591	9465	10.871	270465	4.028
2	30.045	77604	89.129	6444705	95.972
总计		87069	100.000	6715170	100.000

Chromatography

mAU



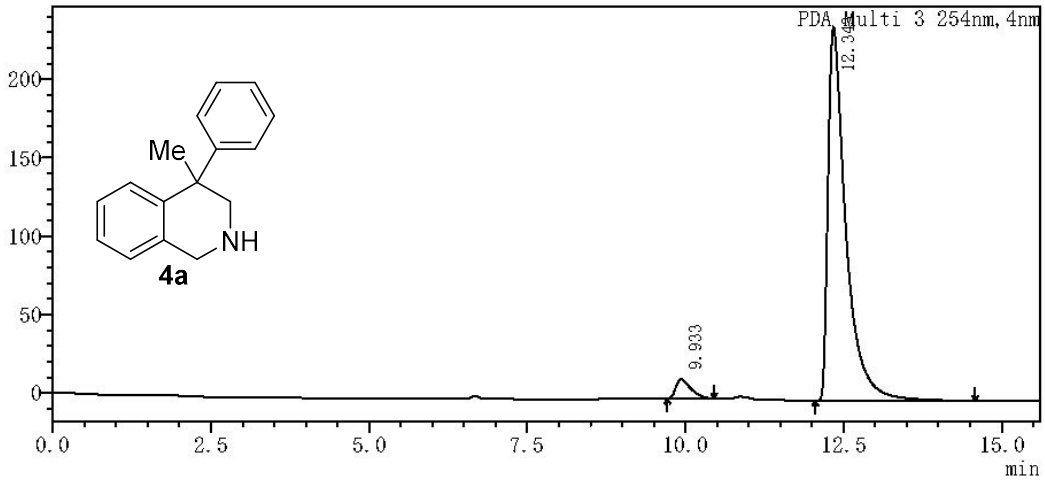
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	9.497	52046	53.790	1051415	50.746
2	11.745	44712	46.210	1020499	49.254
总计		96758	100.000	2071914	100.000

Chromatography

mAU



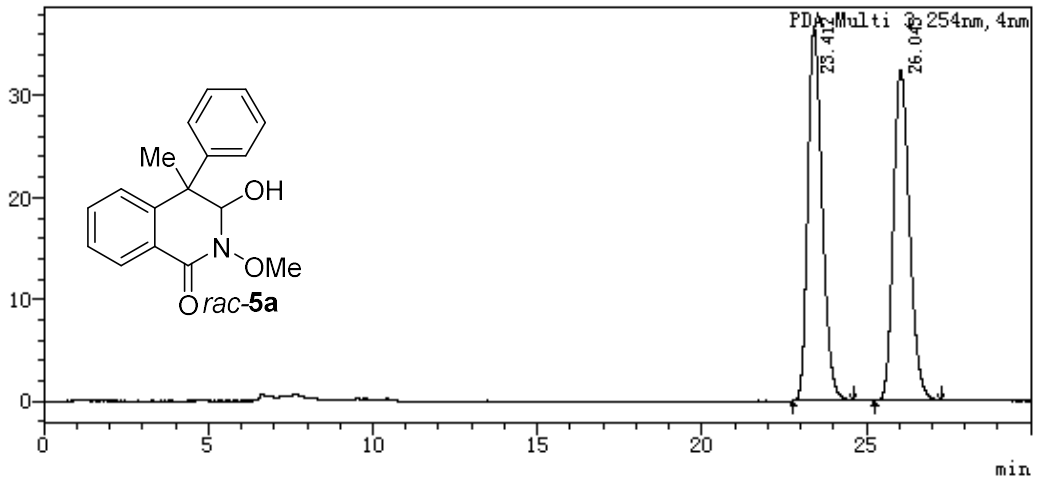
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	9.933	12375	4.941	204507	4.166
2	12.343	238051	95.059	4704103	95.834
总计		250426	100.000	4908610	100.000

Chromatography

mAU



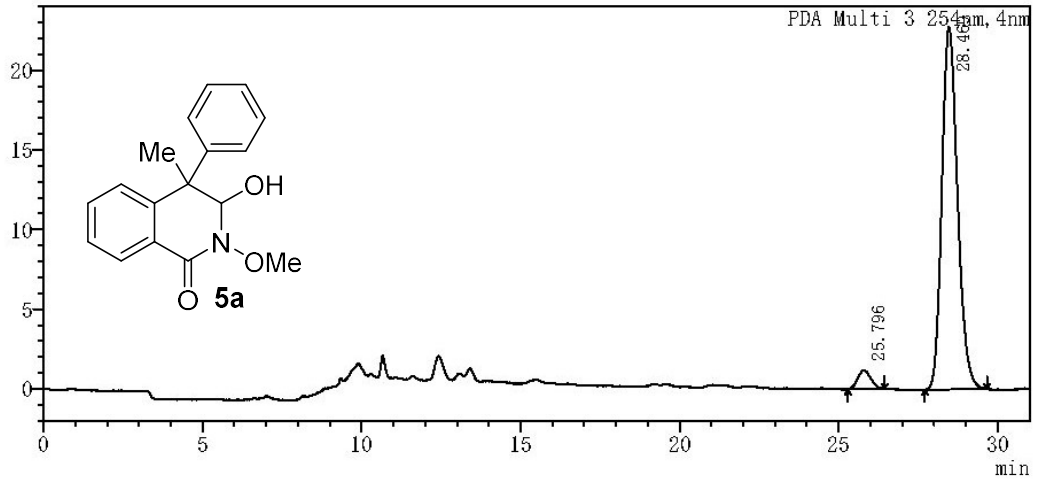
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	23.412	36621	52.952	1111001	49.977
2	26.045	32537	47.048	1112020	50.023
总计		69158	100.000	2223021	100.000

Chromatography

mAU



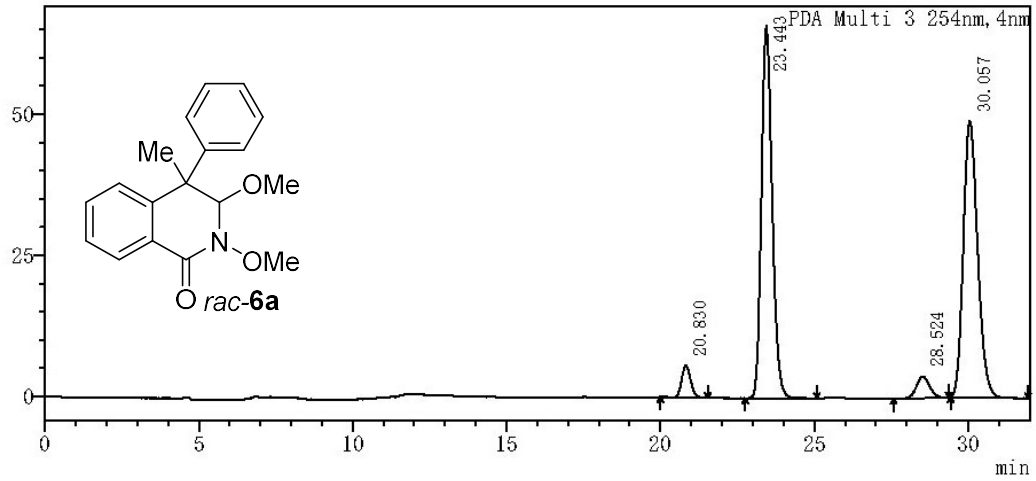
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	25.796	1187	4.958	33992	4.179
2	28.469	22755	95.042	779414	95.821
总计		23942	100.000	813406	100.000

Chromatography

mAU



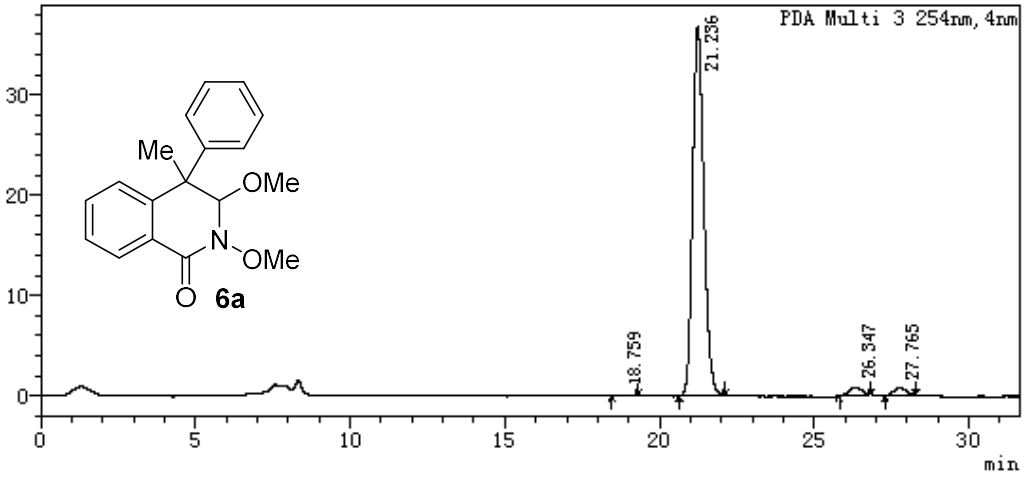
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	20.830	5684	4.579	113813	3.342
2	23.443	65706	52.925	1597998	46.927
3	28.524	3847	3.099	114784	3.371
4	30.057	48912	39.398	1578711	46.360
总计		124149	100.000	3405306	100.000

Chromatography

mAU



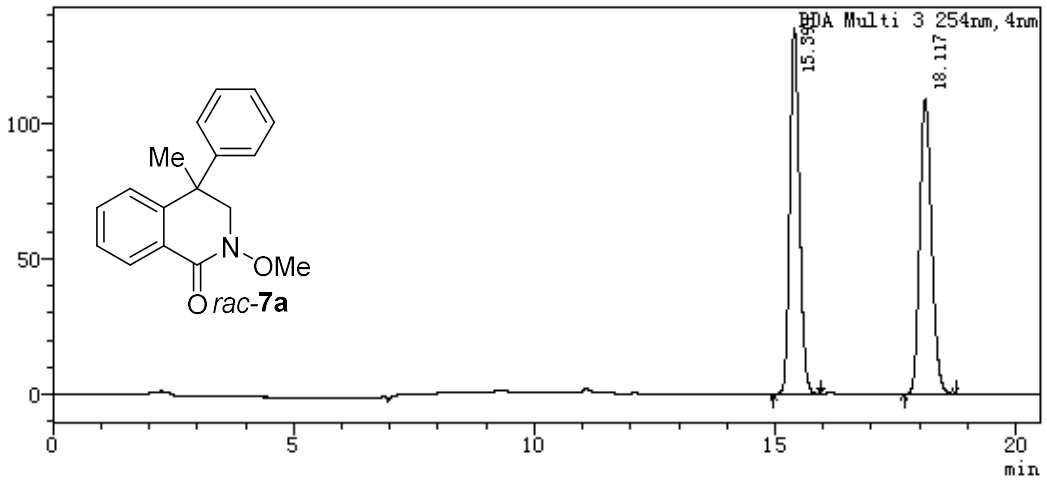
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	18.759	86	0.223	1504	0.155
2	21.236	36810	95.801	928262	95.411
3	26.347	793	2.063	22248	2.287
4	27.765	735	1.913	20892	2.147
总计		38424	100.000	972906	100.000

Chromatography

mAU



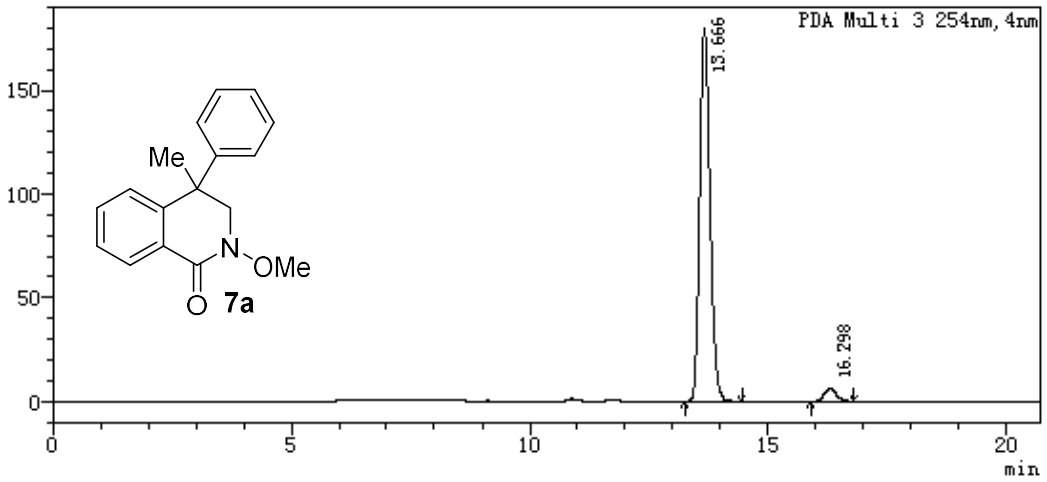
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	15.399	135024	55.361	1870807	50.021
2	18.117	108875	44.639	1869254	49.979
总计		243899	100.000	3740060	100.000

Chromatography

mAU



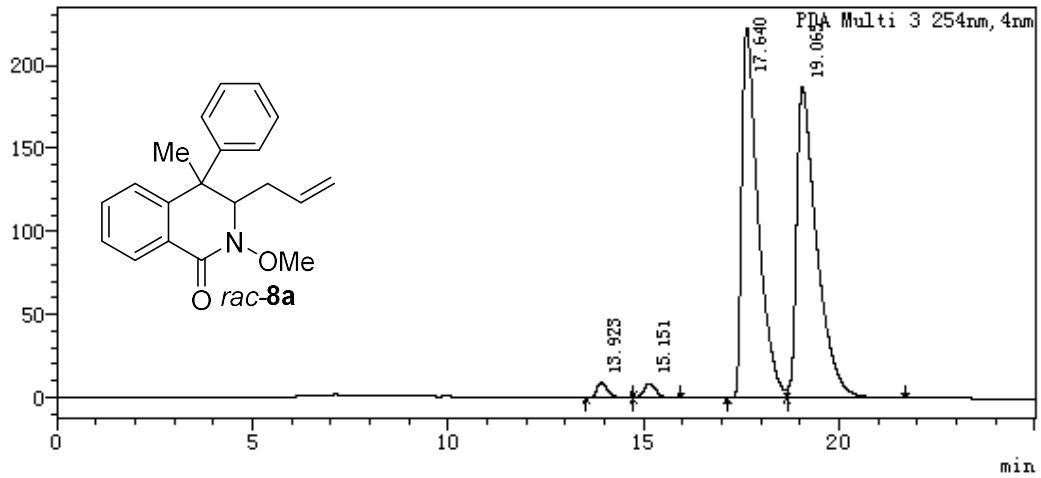
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	13.666	180101	96.546	2722365	95.973
2	16.298	6444	3.454	114220	4.027
总计		186545	100.000	2836585	100.000

Chromatography

mAU



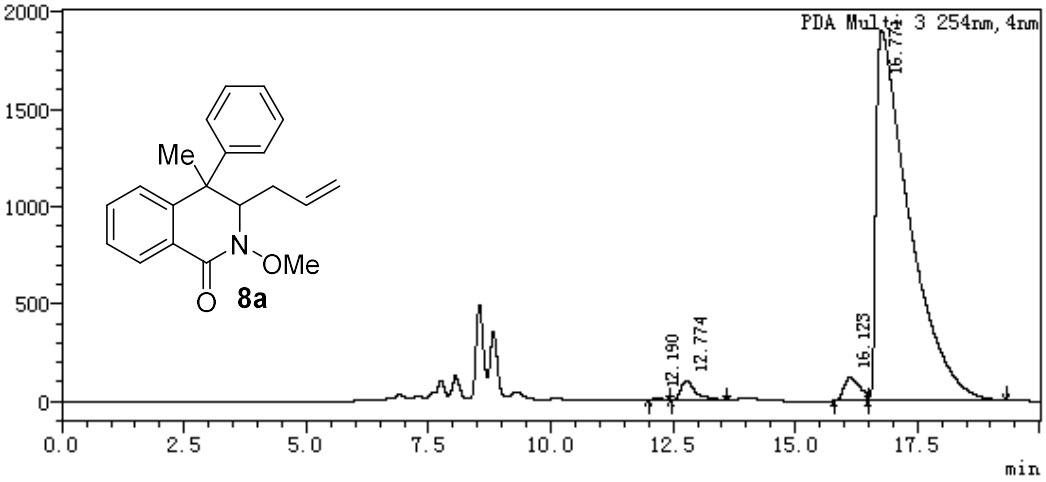
Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	13.923	9370	2.185	184508	1.351
2	15.151	8819	2.057	192334	1.408
3	17.640	223131	52.042	6526640	47.784
4	19.065	187431	43.716	6755201	49.457
总计		428751	100.000	13658684	100.000

Chromatography

mAU



Table

PDA Ch3 254nm

Number	Retention Time	Height	Height%	Area	Area%
1	12.190	11628	0.544	150548	0.159
2	12.774	101314	4.738	2246735	2.366
3	16.123	120194	5.621	2776532	2.924
4	16.771	1905125	89.097	89799014	94.552
总计		2138262	100.000	94972828	100.000