Supplementary Information

Visible-Light-Induced Organocatalytic Enantioselective N–H Insertion of α-Diazoesters Enabled by Indirect Free Carbene Capture

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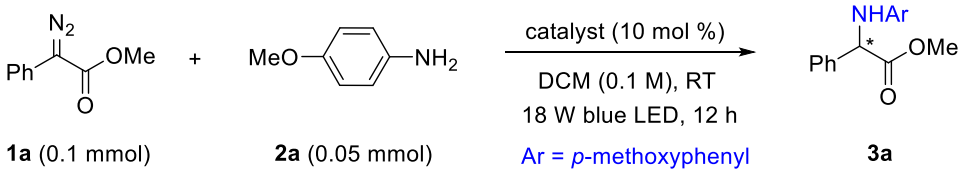
I. General Information

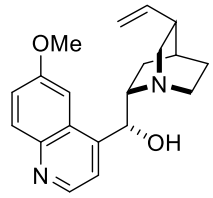
Flash column chromatography was performed over silica gel (200-300 mesh) purchased from Qindao Puke Co., China. All air or moisture sensitive reactions were conducted in oven-dried glassware under nitrogen atmosphere using anhydrous solvents. Anhydrous acetonitrile was purified by the Innovative® solvent purification system or purchased from Energy Chemical. Analytical grade dimethyl sulfoxide (DMSO), Dichloromethane (DCM), 1,2-dichloroethane (DCE), methyl *tert*-butyl ether (MTBE), tetrahydrofuran (THF), benzotrifluoride ($CF_3C_6H_5$), and chlorobenzene (ClC_6H_5) were purchased from Energy Chemical. *n*-Hexane (HPLC grade), *iso*-propanol (HPLC grade) were purchased from J&K Chemical. These solvents were directly used as received. α-Aryl acetates were purchased from Energy Chemical and Leyan. 4-Acetamidobenzenesulfonyl azide was purchased from Energy Chemical and Accela. ^1H, ^{13}C and ^{19}F NMR spectra were collected on a Bruker AV 300 and 400 MHz NMR spectrometer using residue solvent peaks as an internal standard (^1H NMR: $CDCl_3$ at 7.26 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm). Mass spectra were collected on an Agilent GC/MS 5975C system, a MALDI Micro MX mass spectrometer, or an API QSTAR XL System. Optical rotations were measured on JASCO P-2000 polarimeter with $[\alpha]^D$ values reported in degrees; concentration (c) is in 10 mg/mL. The enantiomeric excess values were determined by chiral HPLC using an Agilent 1260 LC system with a Daicel CHIRALCEL OD-H column, or a Daicel CHIRALPAK AD-H or IC column. Unless otherwise noted, the racemic samples in this study were prepared using the corresponding aryldiazoacetate and arylamine under the irradiation of the blue light.

II. Optimization of Reaction Conditions

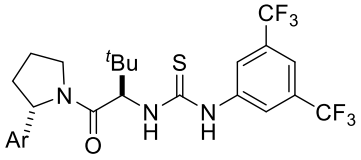
Table S1 Preliminary results[a]



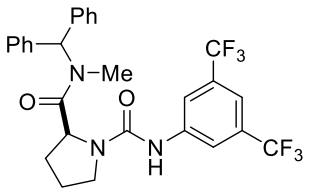
1a (0.1 mmol) **2a** (0.05 mmol) Ar = *p*-methoxyphenyl **3a**



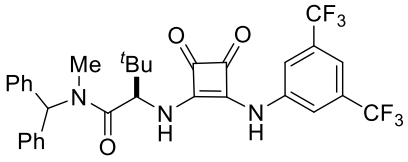
A (quinine)



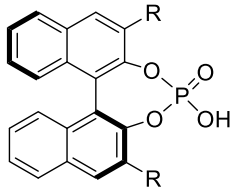
B (Ar = 1-pyrenyl)



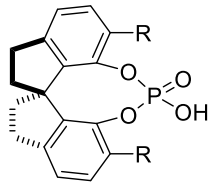
C



D



(*S*)-**E1** (R = 2,4,6-$^{i}Pr_3C_6H_2$)
(*S*)-**E2** (R = 9-anthryl)
(*S*)-**E3** (R = 9-(10-Ph)anthryl)

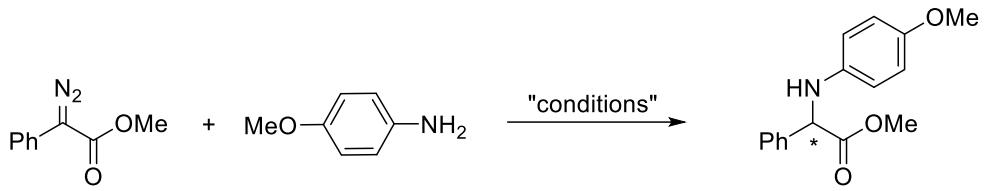


(*R*)-**F** (R = 9-anthryl)

entry	catalyst	yield[b] (%)	e.r.[c]
1	**A**	>95	50:50
2	**B**	>95	50:50
3	**C**	>95	50:50
4	**D**	>95	50:50
5	**--**	>95	50:50
6	**E1**	>95	45:55
7	**E2**	>95	40.5:59.5
8	**E3**	>95	39.5:60.5
9	**F**	>95	43:57
10	**E3** (no light)	0[d]	–

[a] **1a** (0.1 mmol), **2a** (0.05 mmol). [b] Determined by ^{1}H NMR analysis of the crude mixture. [c] Determined by chiral HPLC. [d] No reaction.

Table S2 The effect of different wavelength of light source on the enantioselectivity of reaction



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

entry	light source	T/°C	t/h	yield[a] (%)	e.r.[b]
1	blue LED	25	12	>95	90.6:9.4
2[c]	blue LED	25	12	>95	39.5:60.5
3[d]	green LED	25	48	>95	90:10
4[c,d]	green LED	25	48	>95	40.5:59:5
5[d]	purple LED	25	12	>95	85:15
6[d]	white LED	25	48	>95	88.5:11.5
7[c,d]	white LED	25	48	>95	39.5:60.5
8	blue LED	−10	24	74	85:15
9	blue LED	−20	24	51	69:31

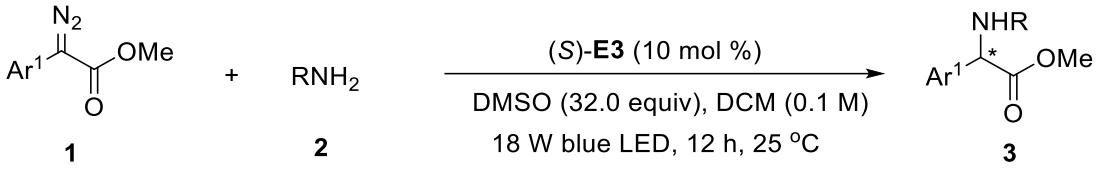
Unless specified mentioned, the reactions are conducted under otherwise identical conditions with those of optimized reaction conditions. [a] NMR yield using CH_2Br_2 as an internal standard. [b] Determined by chiral HPLC analysis. [c] Without DMSO. [d] 24 W.

III. Synthesis of α-Diazo Esters

All the α-diazo esters used this work are known compounds and were synthesized according to the literature procedure.[1]

IV. Visible-Light-Promoted Enantioselective N–H Insertion

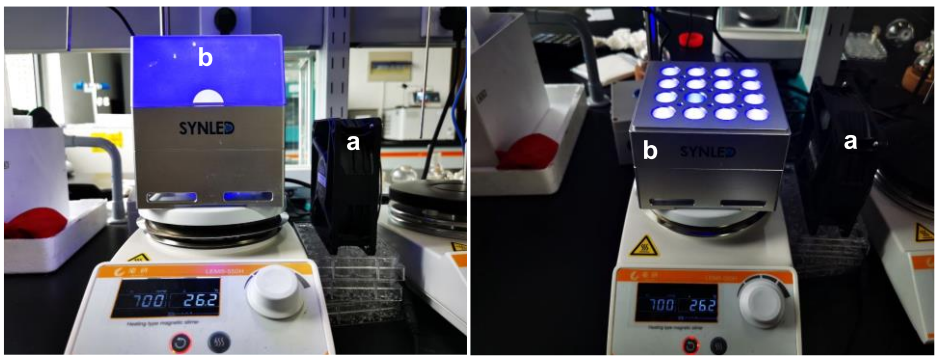
General Procedure.



An oven-dried 4-mL vial equipped with a magnetic stirring bar was charged with the arylamine **2** (0.1 mmol, 1.0 equiv), the catalyst (*S*)-**E3** (8.5 mg, 0.01 mmol, 10 mol %) and DCM (1.0 mL, analytical grade with amylene as stabilizer). The mixture was stirred at 25 °C for 2 min before the addition of the methyl α-diazo acetate **1** (0.2 mmol, 2.0 equiv) and DMSO (230 μL, 3.2 mmol, 32.0 equiv). Then, the reaction mixture was stirred at the same temperature under the irradiation of 18 W blue LED, and the reaction progress was monitored by TLC. Upon completion (12 h), the mixture was directly subjected to flash column chromatography on silica gel (eluent: *n*-hexane/ethyl acetate = 10:1 to 5:1) to give the desired product **3**.

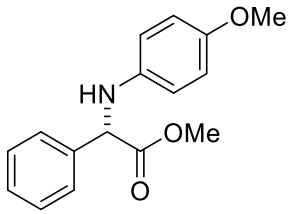
The images of the photo-reactor in current study:

1 H. M. L. Davies, T. Hansen, and M. R. Churchill, *J. Am. Chem. Soc.,* 2000, **122**, 3063–3070.



a: An additional fan was adopted to help cool the reaction mixture.

b: The photo reactor with a built-in fan (18 W output power with a peak power at 467.5 nm; beam angle: 45°; lumen: 130–140).



3a

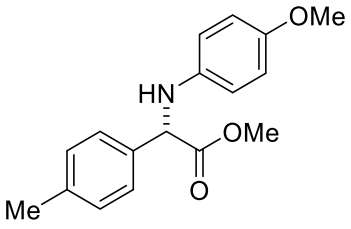
Methyl (*S*)-2-((4-methoxyphenyl)amino)-2-phenylacetate (3a) was prepared as a light yellow solid according to the General Procedure D (eluent: *n*-hexane/EtOAc = 10:1, 26.0 mg, 96% yield, 90.6:9.4 er).

$[\alpha]_D^{25}$: +75.0 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 15.6 min (major), 14.0 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 – 7.49 (m, 2H), 7.40 – 7.29 (m, 3H), 6.77 – 6.71 (m, 2H), 6.58 – 6.52 (m, 2H), 5.04 (s, 1H), 4.70 (br, 1H), 3.73 – 3.71 (m, 6H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6, 152.5, 140.2, 137.8, 128.9, 128.3, 127.3, 114.9, 114.8, 61.7, 55.7, 52.8 ppm.

HRMS (CI+) Calcd for $C_{16}H_{18}NO_3$ $[M+H]^+$: 272.1287, found: 272.1281.



3b

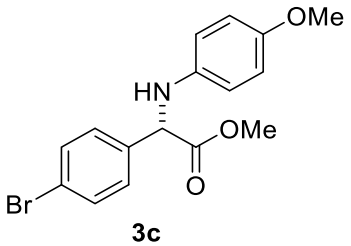
Methyl (*S*)-2-((4-methoxyphenyl)amino)-2-(*p*-tolyl)acetate (3b) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 21.1 mg, 74% yield, 92.1:7.9 er).

$[\alpha]_D^{25}$: +65.5 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 13.1 min (major), 14.1 min (minor).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.63 (d, *J* = 8.8 Hz, 2H), 6.45 (d, *J* = 8.8 Hz, 2H), 4.90 (s, 1H), 4.56 (br, 1H), 3.62 – 3.61 (m, 6H), 2.24 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.8, 152.5, 140.2, 138.1, 134.8, 129.6, 127.2, 114.9, 114.8, 61.4, 55.7, 52.7, 21.2 ppm.

HRMS (CI+) Calcd for $C_{17}H_{20}NO_3$ $[M+H]^+$: 286.1443, found: 286.1434.



3c

Methyl (*S*)-2-(4-bromophenyl)-2-((4-methoxyphenyl)amino)acetate (3c) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 30.1 mg, 86% yield, 89.8:10.2 er).

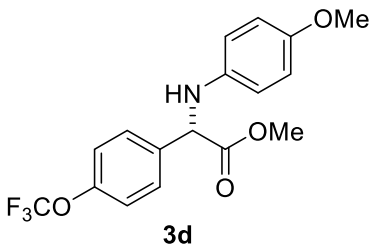
$[\alpha]_D^{25}$: +31.9 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 14.0 min (major), 12.6 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 – 7.45 (m, 2H), 7.38 – 7.35 (m, 2H), 6.73 – 6.70 (m,

2H), 6.50 – 6.47 (m, 2H), 4.97 (s, 1H), 4.72 (br, 1H), 3.72 – 3.69 (m, 6H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.0, 152.6, 139.8, 137.0, 132.0, 129.0, 122.3, 114.9, 114.8, 61.0, 55.7, 53.0 ppm.

HRMS (CI+) Calcd for $C_{16}H_{17}BrNO_3$ $[M+H]^+$: 350.0392, found: 350.0388.



3d

Methyl (*S*)-2-((4-methoxyphenyl)amino)-2-(4-(trifluoromethoxy)phenyl)acetate (3d) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 34.5 mg, 97% yield, 84.6:15:4% er).

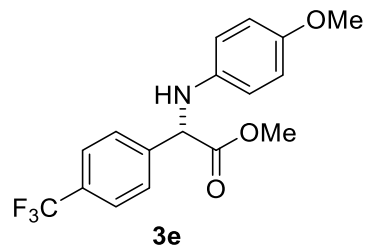
$[\alpha]_D^{25}$: +28.0 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 9.6 min (major), 8.8 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55 – 7.50 (m, 2H), 7.21 – 7.18 (m, 2H), 6.74 – 6.70 (m, 2H), 6.53 – 6.49 (m, 2H), 5.02 (s, 1H), 4.73 (br, 1H), 3.73 (s, 3H), 3.70 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.0, 152.7, 149.1, 139.8, 136.5, 128.7, 121.3, 120.4 (q, $^1J_{C\text{-}F}$ = 256 Hz), 114.9, 114.8, 60.9, 55.7, 52.9 ppm.

^{19}F NMR (282 MHz, $CDCl_3$) δ –57.8 ppm.

HRMS (CI+) Calcd for $C_{17}H_{17}F_3NO_4$ $[M+H]^+$: 356.1110, found: 356.1100.



3e

Methyl (*S*)-2-((4-methoxyphenyl)amino)-2-(4-(trifluoromethyl)phenyl)acetate (3e) was prepared as a light yellow solid according to the General Procedure (eluent: *n*-

hexane/EtOAc = 10:1, 27.5 mg, 81% yield, 84.5:15.5 er).

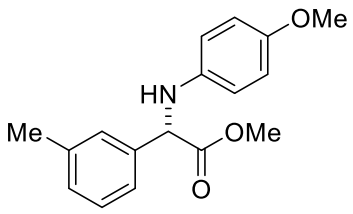
$[\alpha]_D^{25}$: +23.0 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 10.3 min (major), 9.1 min (minor).

^{1}H NMR (300 MHz, acetone-d_6) δ 7.84 – 7.74 (m, 4H), 6.76 – 6.69 (m, 4H), 5.51 – 5.49 (m, 1H), 5.36 – 5.34 (m, 1H), 3.73 (s, 3H), 3.69 (s, 3H) ppm.

^{13}C NMR (75 MHz, acetone-d_6) δ 171.5, 152.5, 143.2, 140.4, 129.5 (q, $^2J_{C\text{-}F}$ = 32 Hz), 128.3, 125.4 (q, $^3J_{C\text{-}F}$ = 3.8 Hz), 124.4 (q, $^1J_{C\text{-}F}$ = 251.1 Hz), 114.8, 114.5, 60.7, 54.8, 52.1 ppm.

^{19}F NMR (282 MHz, Acetone-d_6) δ –63.0 ppm.

HRMS (CI+) Calcd for $C_{17}H_{17}F_3NO_3$ $[M+H]^+$: 340.1161, found: 340.1156.



3f

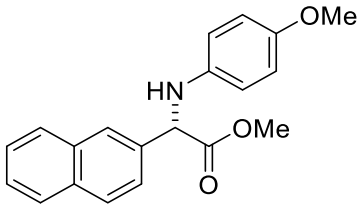
Methyl (*S*)-2-((4-methoxyphenyl)amino)-2-(*m*-tolyl)acetate (3f) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 25.7 mg, 90% yield, 87.6:12.4 er).

$[\alpha]_D^{25}$: +24.6 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 15.4 min (major), 14.5 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 7.21 (m, 3H), 7.12 – 7.10 (m, 1H), 6.74 – 6.71 (m, 2H), 6.55 – 6.52 (m, 2H), 4.97 (s, 1H), 4.64 (br, 1H), 3.71 (s, 3H), 3.70 (s, 3H), 2.34 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.7, 152.5, 140.3, 138.6, 137.7, 129.1, 128.8, 127.9, 124.4, 114.9, 114.8, 61.7, 55.7, 52.7, 21.5 ppm.

HRMS (CI+) Calcd for $C_{17}H_{20}NO_3$ $[M+H]^+$: 286.1443, found: 286.1432.



3g

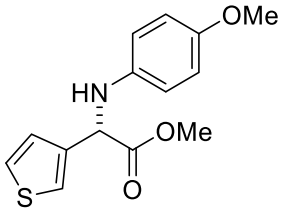
Methyl (*S*)-2-((4-methoxyphenyl)amino)-2-(naphthalen-2-yl)acetate (3g) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/ethyl acetate = 10:1 to 5:1, 28.6 mg, 89% yield, 86.5:13.5 er).

$[\alpha]_D^{25}$: +22.1 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 19.7 min (major), 17.8 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.84 – 7.80 (m, 3H), 7.61 – 7.58 (m, 1H), 7.48 – 7.45 (m, 2H), 6.72 – 6.69 (m, 2H), 6.58 – 6.55 (m, 2H), 5.17 (s, 1H), 4.82 (br, 1H), 3.71 (s, 3H), 3.67 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.5, 152.5, 140.2, 135.3, 133.4, 133.3, 128.8, 128.1, 127.7, 126.5, 126.4, 126.3, 125.0, 114.88, 114.85, 61.8, 55.7, 52.8 ppm.

HRMS (CI+) Calcd for $C_{20}H_{20}NO_3$ $[M+H]^+$: 322.1443, found: 322.1437.



3h

Methyl (*S*)-2-((4-methoxyphenyl)amino)-2-(thiophen-3-yl)acetate (3i) was prepared as a light yellow solid according to the General Procedure D (91 h at –15 °C, eluent: *n*-hexane/EtOAc = 10:1, 25.5 mg, 92% yield, 83.6:16.4 er).

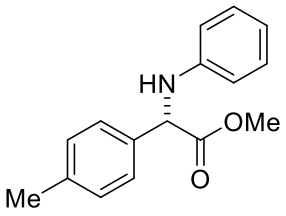
$[\alpha]_D^{25}$: +15.3 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® AD-H column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 30.0 min (major), 27.5 min (minor).

^{1}H NMR (300 MHz, acetone-d_6) δ 7.55 – 7.53 (m, 1H), 7.50 – 7.47 (m, 1H), 7.26 – 7.24

(m, 1H), 6.79 – 6.71 (m, 4H), 5.31 – 5.27 (m, 2H), 3.72 (s, 3H), 3.71 (s, 3H) ppm.

^{13}C NMR (75 MHz, acetone-d_6) δ 172.2, 152.5, 141.1, 138.9, 126.8, 126.1, 122.9, 114.7, 114.5, 57.3, 54.8, 51.7 ppm.

HRMS (CI+) Calcd for $C_{14}H_{16}NO_3S$ $[M+H]^+$: 278.0851, found: 278.0842.



3i

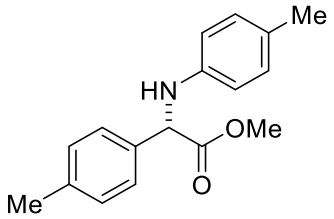
Methyl (*S*)-2-(phenylamino)-2-(*p*-tolyl)acetate (3i) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 20.7 mg, 81% yield, 89.8:10.2 er).

$[\alpha]_D^{25}$: +25.1 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 11.8 min (major), 15.0 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.35 (m, 2H), 7.16 – 7.07 (m, 4H), 6.68 (t, *J* = 7.4 Hz, 1H), 6.56 – 6.53 (m, 2H), 5.04 (s, 1H), 4.92 (br, 1H), 3.70 (s, 3H), 2.32 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6, 146.0, 138.2, 134.6, 129.6, 129.3, 127.2, 118.1, 113.4, 60.5, 52.8, 21.2 ppm.

HRMS (CI+) Calcd for $C_{16}H_{18}NO_2$ $[M+Na]^+$: 256.1338, found: 256.1322.



3j

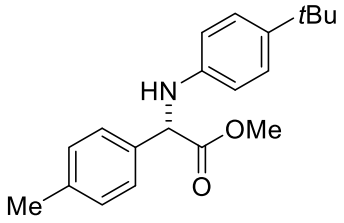
Methyl (*S*)-2-(*p*-tolyl)-2-(*p*-tolylamino)acetate (3j) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 21.5 mg, 80% yield, 91.7:8.3 er).

$[\alpha]_D^{25}$: +60.1 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 12.5 min (major), 8.8 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.34 (m, 2H), 7.15 – 7.13 (m, 2H), 6.93 – 6.90 (m, 2H), 6.49 – 6.46 (m, 2H), 5.02 (s, 1H), 4.78 (br, 1H), 3.70 (s, 3H), 2.31 (s, 3H), 2.18 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.7, 143.8, 138.1, 134.8, 129.8, 129.6, 127.3, 127.2, 113.6, 60.8, 52.7, 21.2, 20.4 ppm.

HRMS (CI+) Calcd for $C_{17}H_{20}NO_2$ $[M+H]^+$: 270.1496, found: 270.1486.



3k

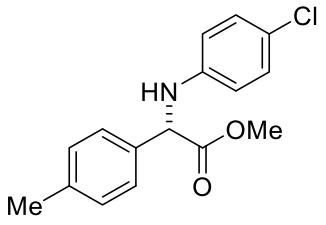
Methyl (*S*)-2-((4-(*tert*-butyl)phenyl)amino)-2-(*p*-tolyl)acetate (3k) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 23.4 mg, 75% yield, 90.4:9.6 er).

$[\alpha]_D^{25}$: +45.2 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 7.5 min (major), 6.3 min (minor).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.39 (m, 2H), 7.19 – 7.15 (m, 4H), 6.56 – 6.52 (m, 2H), 5.03 (s, 1H), 4.85 (br, 1H), 3.73 (s, 3H), 2.35 (s, 3H), 1.26 (s, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 143.7, 140.8, 138.1, 134.9, 129.6, 127.2, 126.1, 113.0, 60.8, 52.8, 33.9, 31.5, 21.2 ppm.

HRMS (CI+) Calcd for $C_{20}H_{26}NO_2$ $[M+H]^+$: 312.1964, found: 312.1955.



3l

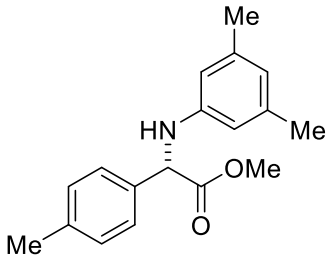
Methyl (*S*)-2-((4-chlorophenyl)amino)-2-(*p*-tolyl)acetate (3l) was prepared as a light yellow solid according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 26.7 mg, 92% yield, 85.1:14.9 er).

$[\alpha]_D^{25}$: +19.1 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 9.8 min (major), 9.2 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.32 (m, 2H), 7.17 – 7.14 (m, 2H), 7.06 – 7.03 (m, 2H), 6.47 – 6.44 (m, 2H), 4.99 (br, 2H, overlaps of the protons of NH and chiral benzylic C–H), 3.71 (s, 3H), 2.32 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.2, 144.5, 138.3, 134.1, 129.7, 129.1, 127.1, 122.7, 114.5, 60.4, 52.9, 21.2 ppm.

HRMS (CI+) Calcd for $C_{16}H_{17}ClNO_2$ $[M+H]^+$: 290.0948, found: 290.0942.



3m

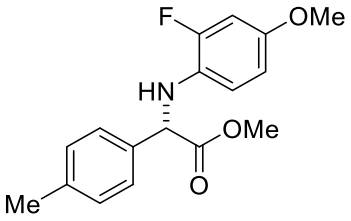
Methyl (*S*)-2-((3,5-dimethylphenyl)amino)-2-(*p*-tolyl)acetate (3m) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 19.8 mg, 70% yield, 88.3:11.7 er).

$[\alpha]_D^{25}$: +24.5 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® OD-H column; 2% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 11.6 min (major), 9.3 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 6.36 (s, 1H), 6.20 (s, 2H), 5.03 (s, 1H), 4.77 (br, 1H), 3.71 (s, 3H), 2.33 (s, 3H), 2.18 (s, 6H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.7, 146.2, 138.9, 138.0, 134.8, 129.6, 127.1, 120.2, 111.3, 60.5, 52.7, 21.5, 21.2 ppm.

HRMS (CI+) Calcd for $C_{18}H_{22}NO_2$ $[M+H]^+$: 284.1651, found: 284.1646.



3n

Methyl (*S*)-2-((2-fluoro-4-methoxyphenyl)amino)-2-(*p*-tolyl)acetate (3n) was prepared as a light yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1 to 5:1, 26.4 mg, 87% yield, 92.2:7.8 er).

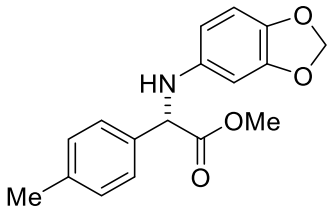
$[\alpha]_D^{25}$: +31.1 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® IC column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 12.3 min (major), 11.4 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.66 – 6.61 (m, 1H), 6.47 – 6.37 (m, 2H), 5.01 – 4.99 (m, 1H), 4.81 (br, 1H), 3.72 (s, 3H), 3.68 (s, 3H), 2.33 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.3, 152.2 (d, $^3J_{C\text{-}F}$ = 9.8 Hz), 152.1 (d, $^1J_{C\text{-}F}$ = 238.8 Hz), 138.3, 134.4, 129.6, 128.5 (d, $^3J_{C\text{-}F}$ = 12.1 Hz), 127.1, 114.0 (d, $^4J_{C\text{-}F}$ = 4.4 Hz), 109.1 (d, $^4J_{C\text{-}F}$ = 3.4 Hz), 102.6 (d, $^2J_{C\text{-}F}$ = 22.2 Hz), 61.0, 55.8, 52.8, 21.1 ppm.

^{19}F NMR (282 MHz, $CDCl_3$) δ –131.9 ppm.

HRMS (CI+) Calcd for $C_{17}H_{19}FNO_3$ $[M+H]^+$: 304.1349, found: 304.1344.



3o

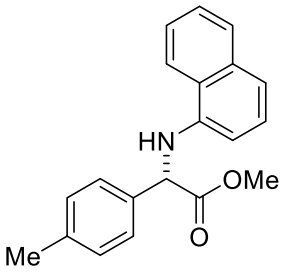
Methyl (*S*)-2-(benzo[*d*][1,3]dioxol-5-ylamino)-2-(*p*-tolyl)acetate (3o) was prepared as a yellow oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1 to 5:1, 20.7 mg, 69% yield, 90.3:9.7 er).

$[\alpha]_D^{25}$: +60.4 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® IC column; 20% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 8.8 min (major), 9.8 min (minor).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.58 (d, *J* = 8.3 Hz, 1H), 6.20 (d, *J* = 2.3 Hz, 1H), 5.98 (dd, *J* = 2.3, 8.3 Hz, 1H), 5.81 (s, 2H), 4.95 (s, 1H), 4.71 (br, 1H), 3.71 (s, 3H), 2.33 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6, 148.3, 141.7, 140.1, 138.2, 134.6, 129.6, 127.1, 108.6, 105.2, 100.6, 96.6, 61.3, 52.8, 21.2 ppm.

HRMS (CI+) Calcd for $C_{17}H_{18}NO_4$ $[M+H]^+$: 300.1236, found: 300.1232.



3p

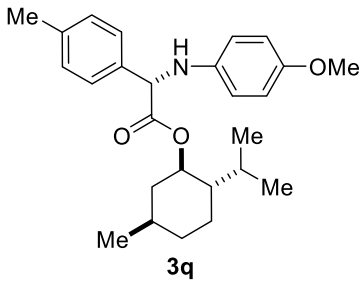
Methyl (*S*)-2-(naphthalen-1-ylamino)-2-(*p*-tolyl)acetate (3p) was prepared as a light yellow solid according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1 to 5:1, 18.6 mg, 61% yield, 90.3:9.7 er).

$[\alpha]_D^{25}$: +65.0 (*c* = 1.0, CH_2Cl_2). HPLC analysis of the product: Daicel CHIRALPAK® AD-H column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min; retention times: 9.0 min (major), 10.8 min (minor).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.90 (m, 1H), 7.68 – 7.66 (m, 1H), 7.39 – 7.33 (m, 4H), 7.12 – 7.05 (m, 4H),6.25 – 6.23 (m, 1H), 5.66 (br, 1H), 5.11 (s, 1H), 3.64 (s, 3H), 2.22 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 141.1, 138.3, 134.4, 134.4, 129.7, 128.7, 127.2, 126.4, 125.9, 125.0, 123.5, 120.2, 118.1, 105.6, 60.6, 53.0, 21.2 ppm.

HRMS (CI+) Calcd for $C_{20}H_{20}NO_2$ $[M+H]^+$: 306.1494, found: 306.1488.



3q

(1*R*,2*S*,5*R*)-2-*iso*-Propyl-5-methylcyclohexyl (*S*)-2-((4-methoxyphenyl)amino)-2-(*p*-tolyl)acetate (3q) was prepared as a colorless oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 32.8 mg, 80% yield, 7:1 dr).

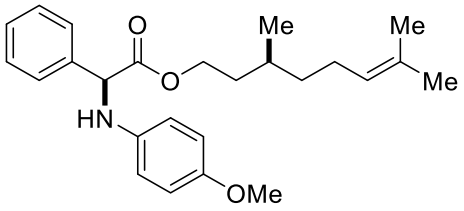
$[\alpha]_D^{25}$: +50.6 (*c* = 1.0, CH_2Cl_2).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.34 (m, 2H), 7.16 – 7.13 (m, 2H), 6.73 – 6.69 (m, 2H), 6.56 – 6.53 (m, 2H), 4.96 – 4.93 (m, 1H), 4.75 – 4.58 (m, 2H), 3.70 (s, 3H), 2.33 (s, 3H), 1.88 – 1.82 (m, 1H), 1.72 – 1.63 (m, 3H), 1.42 – 1.33 (m, 2H), 1.05 – 0.95 (m, 1H), 0.90 – 0.87 (m, 3H), 0.83 – 0.80 (m, 4H), 0.73 – 0.36 (m, 4H) ppm.

Note: The diastereoselectivity was determined by crude ^{1}H NMR analysis and the product was obtained as an inseparable diastereomeric mixture. Thus, the ^{1}H NMR integration was based on the sum of hydrogens of the two diastereomers.

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.8, 152.4, 140.4, 137.8, 134.8, 129.4, 127.0, 114.82, 114.80, 75.6, 61.5, 55.7, 46.9, 40.1, 34.1, 31.3, 26.2, 23.3, 21.9, 21.2, 20.8, 16.2 ppm.

HRMS (CI+) Calcd for $C_{26}H_{36}NO_3$ $[M+H]^+$: 410.2695, found: 410.2690.



3r

(*S*)-3,7-Dimethyloct-6-en-1-yl (*S*)-2-((4-methoxyphenyl)amino)-2-phenylacetate (3r) was prepared as a colorless oil according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 35.6 mg, 90% yield, 7:1 dr).

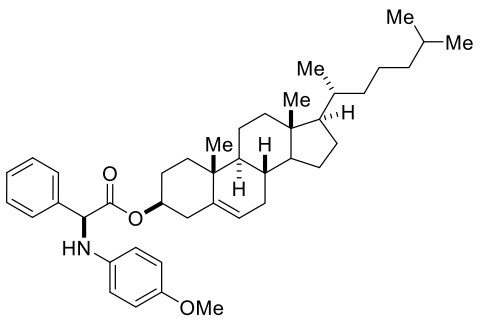
$[\alpha]_D^{25}$: +18.2 (*c* = 1.0, CH_2Cl_2).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 – 7.47 (m, 2H), 7.37 – 7.25 (m, 3H), 6.74 – 6.69 (m, 2H), 6.55 – 6.50 (m, 2H), 5.08 – 4.99 (m, 2H), 4.70 (br, 1H), 4.22 – 4.08 (m, 2H), 3.70 (s, 3H), 1.96 – 1.83 (m, 2H), 1.68 (s, 3H), 1.59 (s, 3H), 1.41 – 1.20 (m, 3H), 1.16 – 1.06 (m, 1H), 0.84 – 0.78 (m, 3H) ppm.

Note: The diastereoselectivity was determined by crude ^{1}H NMR analysis, the product was obtained as an inseparable diastereomeric mixture. Thus, the ^{1}H NMR integration was based on the sum of hydrogens of the two diastereomers.

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.1, 152.4, 140.2, 138.0, 131.4, 128.8, 128.2, 127.2, 124.5, 114.8, 114.7, 64.2, 61.7, 55.7, 36.9, 35.3, 29.3, 25.8, 25.4, 19.2, 17.7 ppm.

HRMS (CI+) Calcd for $C_{25}H_{34}NO_3$ $[M+H]^+$: 396.2539, found: 396.2531.



3s

(3*S*,8*S*,9*S*,10*R*,13*R*,17*R*)-10,13-Dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[a]phenanthren-3-yl (2*S*)-2-((4-methoxyphenyl)amino)-2-phenylacetate (3s) was prepared as a light

yellow foam according to the General Procedure (eluent: *n*-hexane/EtOAc = 10:1, 28.2 mg, 45% yield, 7:1 dr).

$[\alpha]_D^{25}$: +39.5 (*c* = 1.0, CH_2Cl_2).

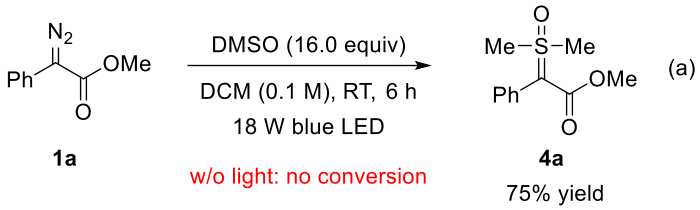
Note: The diastereoselectivity was determined by crude ^1H NMR analysis, the product was obtained as an inseparable diastereomeric mixture. Thus, the ^1H NMR integration was based on the sum of hydrogens of the two diastereomers.

^1H NMR (300 MHz, $CDCl_3$) δ 7.50 – 7.47 (m, 2H), 7.37 – 7.25 (m, 3H), 6.74 – 6.69 (m, 2H), 6.56 – 6.52 (m, 2H), 5.29 – 5.27 (m, 1H), 4.98 (br, 1H), 4.69 – 4.58 (m, 2H), 3.70 (s, 3H), 2.15 – 1.78 (m, 7H), 1.64 – 1.26 (m, 12H), 1.18 – 0.85 (m, 22H), 0.66 (s, 3H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 152.4, 140.3, 139.2, 138.0, 128.8, 128.1, 127.2, 122.9, 114.8, 114.7, 75.4, 61.8, 56.7, 56.1, 55.7, 50.0, 42.3, 39.7, 39.5, 37.6, 36.9, 36.5, 36.2, 35.8, 31.9, 31.8, 28.3, 28.0, 27.7, 24.3, 23.8, 22.9, 22.6, 21.0, 19.3, 18.7, 11.9 ppm.

HRMS (CI+) Calcd for $C_{42}H_{60}NO_3$ $[M+H]^+$: 626.4573, found: 626.4587.

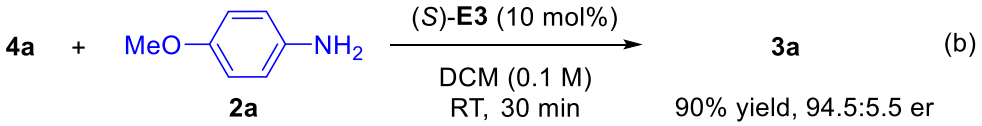
V. Control Experiments



An oven-dried 4-mL vial equipped with a magnetic stirring bar was charged with the diazoester **1a** (35.2 mg, 0.2 mmol, 1.0 equiv) and DMSO (230 μL, 3.2 mmol, 16.0 equiv) in DCM (1.0 mL). The resulting red reaction mixture was stirred at room temperature under the irradiation of 18 W blue LED, and the reaction progress was monitored by TLC. Upon completion (about 6 h), a colorless turbid mixture was obtained, due to the slightly poor solubility of the sulfoxonium ylide, which was diluted with DCM (2.0 mL) and then directly subjected to flash column chromatography on silica gel (eluent: DCM/MeOH = 50:1) to give the desired sulfoxonium ylide **4a** as a light yellow solid (33.9 mg, 75% yield).

It's a known compound, and the spectral data are consistent with the literature report.[2]

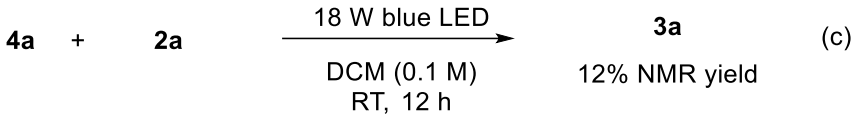
Note: Without light irradiation, this process did proceed.



To an oven-dried 4-mL vial were added the freshly prepared sulfoxonium ylide **4a** (11.3 mg, 0.05 mmol, 1.0 equiv), *p*-anisidine **2a** (6.8 mg, 0.055 mmol, 1.1 equiv), (*S*)-**E3** (4.3 mg, 0.005 mmol, 10 mol %), and then DCM (0.5 mL). The resulting suspension was stirred at room temperature, during which a clear solution was gradually formed. Upon completion (30 min), it was directly subjected to flash column chromatography on silica gel (eluent: petroleum ether/EtOAc = 10:1) to give the desired product as a

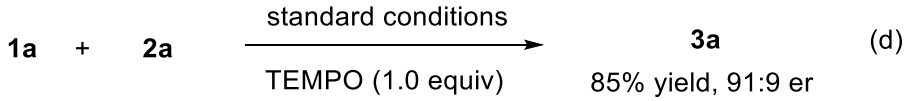
2 (a) J. Lu, L. Li, X.-K. He, G.-Y. Xu, and J. Xuan, *Chin. J. Chem.*, 2021, **39**, 1646–1650; (b) L. S. Munaretto, C. Y. dos Santos, R. D. C. Gallo, C. Y. Okada, V. M. Deflon and I. D. Jurberg, *Org. Lett.*, 2021, **23**, 9292–9296.

light yellow oil (12.2 mg, 90% yield, 94.5:5.5 er). The spectral data were consistent with those obtained from the standard protocol.



(c)

To an oven-dried 4-mL vial were added the freshly prepared sulfoxonium ylide **4a** (11.3 mg, 0.05 mmol, 1.0 equiv), $PMPNH_2$ (6.8 mg, 0.055 mmol, 1.1 equiv), and then DCM (0.5 mL). The resulting suspension was stirred at room temperature under the irradiation of blue LED for 12 h. Then, DCM was removed, and the yield of **3a** was determined by ^1H NMR analysis of the crude mixture using CH_2Br_2 as an internal standard.

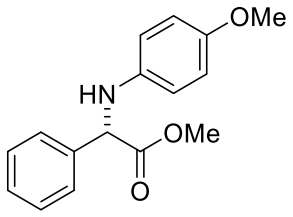


(d)

An oven-dried 4-mL vial equipped with a magnetic stirring bar was charged with the arylamine **2** (0.1 mmol, 1.0 equiv), the catalyst (*S*)-**E3** (8.5 mg, 0.01 mmol, 10 mol %) and DCM (1.0 mL, analytical grade with amylene as stabilizer). The mixture was stirred at 25 °C for 2 min before the addition of the methyl α-diazo acetate **1** (0.2 mmol, 2.0 equiv), DMSO (230 μL, 3.2 mmol, 32.0 equiv), and TEMPO (15.6 mg, 0.1 mmol, 1.0 equiv). Then, the reaction mixture was stirred at the same temperature under the irradiation of 18 W blue LED, and the reaction progress was monitored by TLC. Upon completion (12 h), the mixture was directly subjected to flash column chromatography on silica gel (eluent: *n*-hexane/ethyl acetate = 10:1 to 5:1) to give the desired product **3** (23.1 mg, 85% yield, 91:9 er). The spectral data were consistent with those obtained from the standard protocol.

VI. Determination of the Product Stereochemistry

The absolute stereochemistry of the α-amino ester **3a** was determined by comparing its optical rotation value with the literature. The stereochemistry of the other products was assumed by analogy.

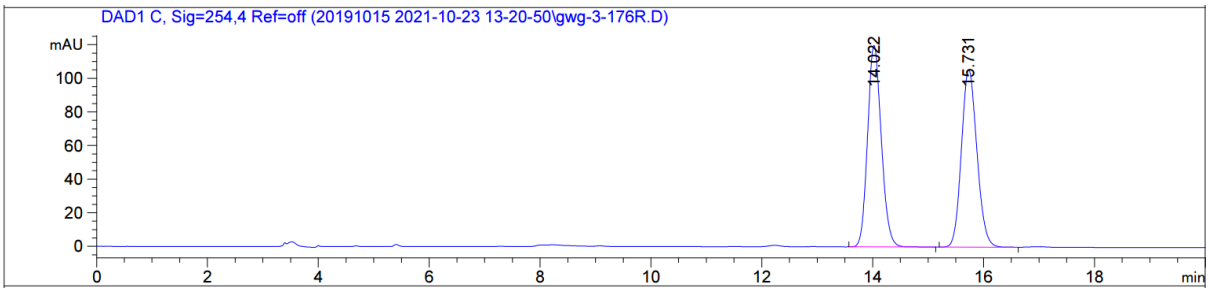


3a

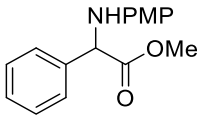
The optical rotation value for (*S*)-**3a** in 95% ee was reported to be $[\alpha]_D^{25}$ = +106.1 (*c* = 1.0, DCM).[3] The measured value of **3a** obtained from our reaction in 81% ee was $[\alpha]_D^{25}$: +75.0 (*c* = 1.0, DCM). Thus, the absolute configuration of **3a** obtained from our reaction was assigned to be *S*.

3 W. Guo, M. Wang, Z. Han, H. Huang and J. Sun, *Chem. Sci.*, 2021, **12**, 11191–11196.

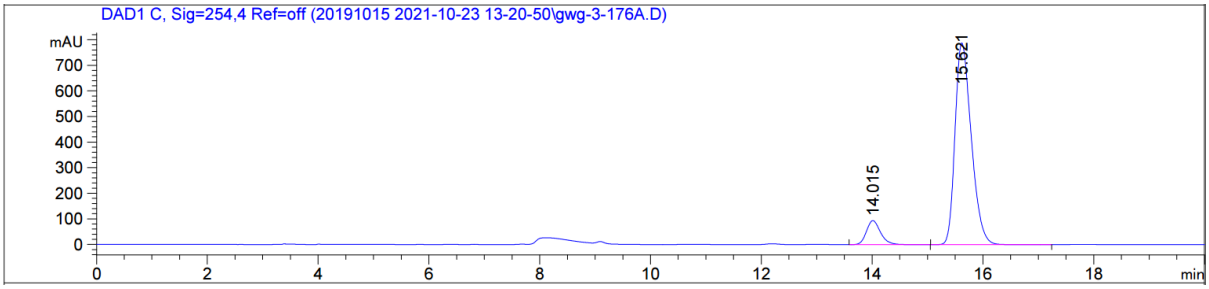
3a, HPLC conditions: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 254 nm.



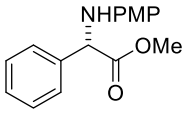
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	14.022	BB	0.2645	2060.76025	119.88544	50.0944
2	15.731	BB	0.3006	2052.99561	105.68841	49.9056



3a
racemic

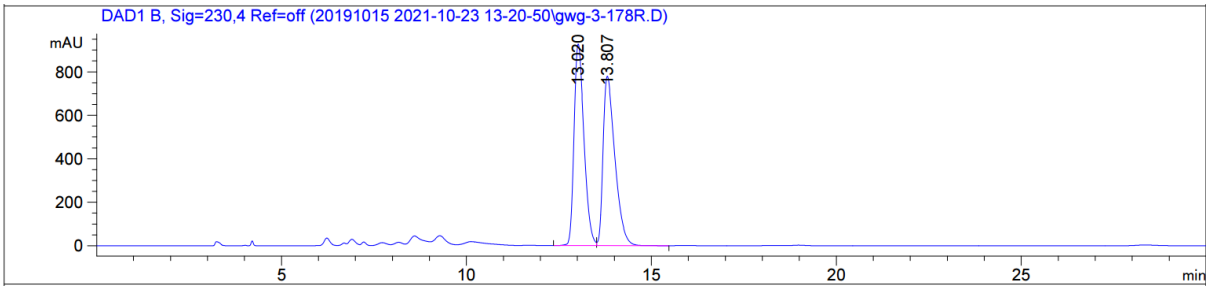


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	14.015	BB	0.2693	1653.18762	93.93599	9.3613
2	15.621	BB	0.3128	1.60067e4	788.45569	90.6387

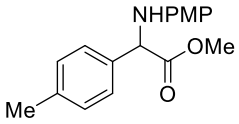


3a
enantioenriched

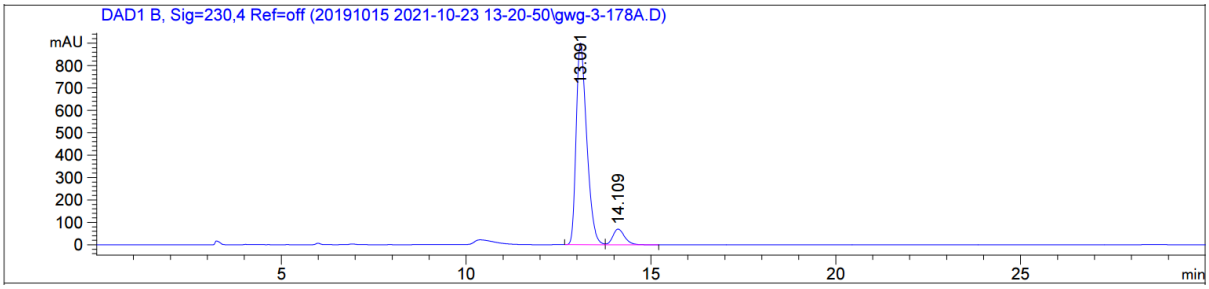
3b, HPLC conditions: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



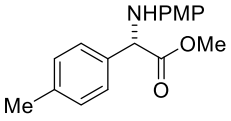
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	13.020	BV	0.2891	1.72794e4	928.39545	49.9736
2	13.807	VB	0.3388	1.72977e4	779.68225	50.0264



3b
racemic

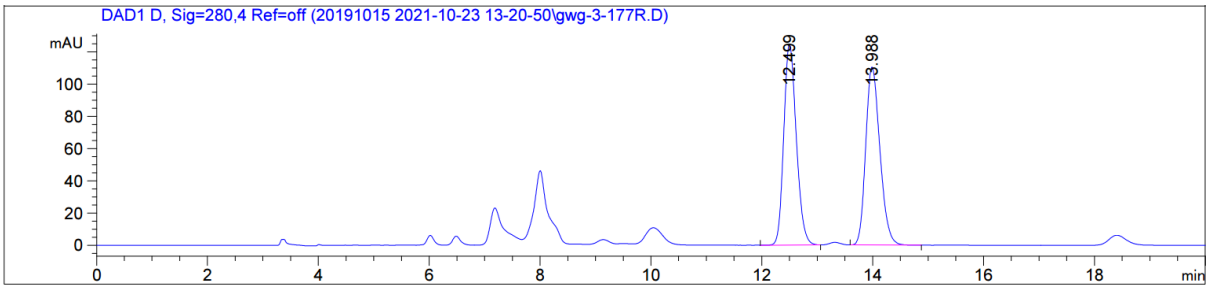


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	13.091	BV	0.3082	1.79147e4	900.24731	92.1226
2	14.109	VB	0.3376	1531.88477	69.92228	7.8774

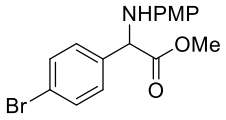


3b
enantioenriched

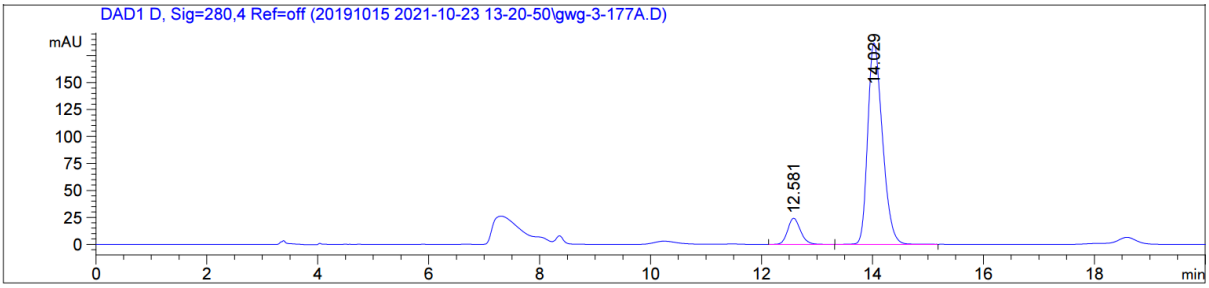
3c, HPLC conditions: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 280 nm.



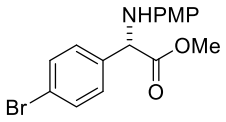
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	12.499	BB	0.2431	1957.96533	124.77911	49.9417
2	13.988	BB	0.2754	1962.53931	110.39389	50.0583



3c
racemic

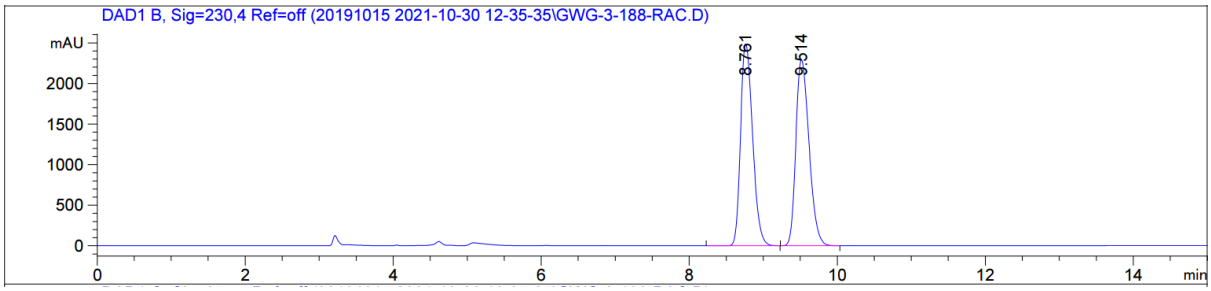


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	12.581	BB	0.2482	390.69864	24.21399	10.1878
2	14.029	BB	0.2851	3444.25879	186.77283	89.8122

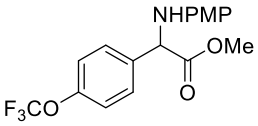


3c
enantioenriched

3d, HPLC conditions: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.

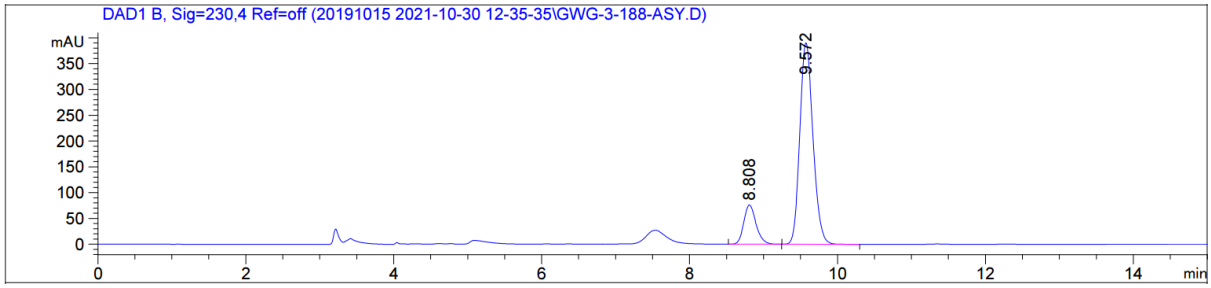


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	8.761	BB	0.1777	2.81031e4	2489.16309	49.7817
2	9.514	BB	0.1923	2.83496e4	2291.41846	50.2183

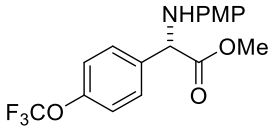


3d

racemic



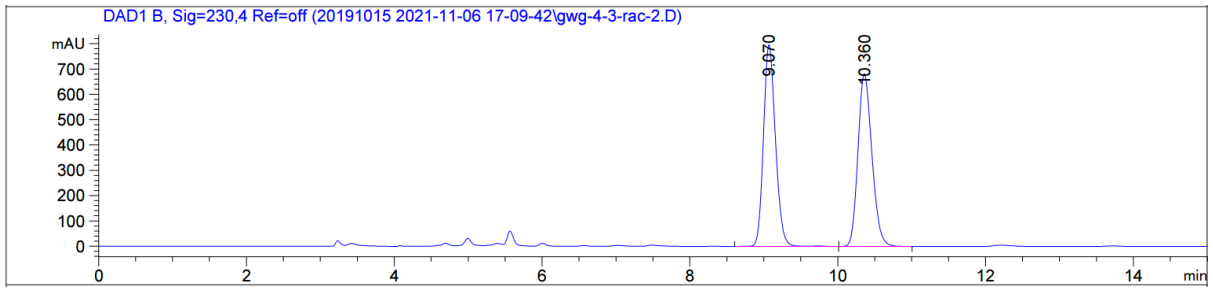
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	8.808	BB	0.1764	876.51385	76.08511	15.3784
2	9.572	BB	0.1901	4823.13672	390.49551	84.6216



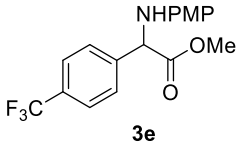
3d

enantioenriched

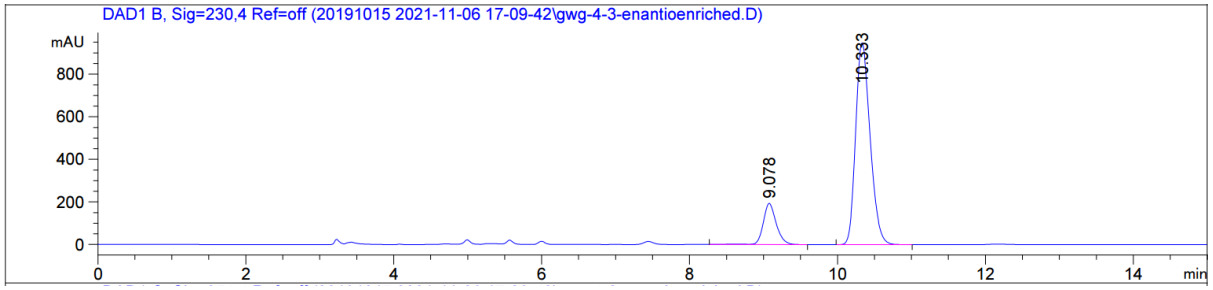
3e, HPLC conditions: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



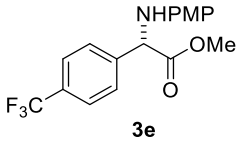
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	9.070	BV R	0.1745	8943.17285	797.82147	50.0151
2	10.360	BB	0.2027	8937.76563	682.71185	49.9849



3e
racemic

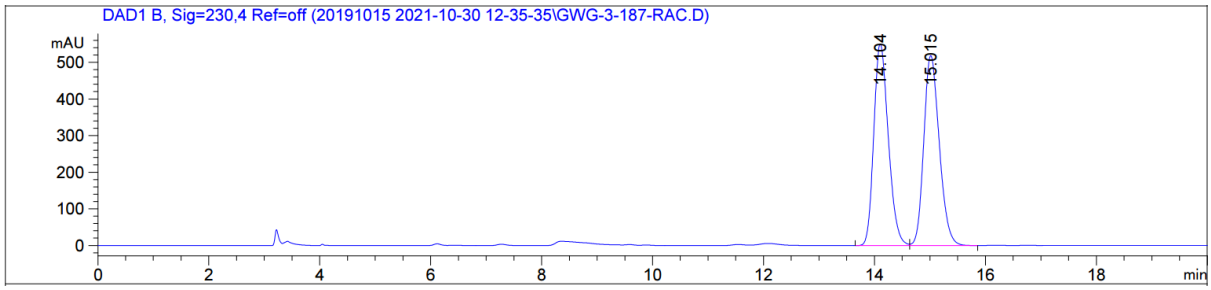


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	9.078	VB R	0.1822	2356.71143	193.51453	15.4764
2	10.333	BB	0.2083	1.28711e4	948.46655	84.5236

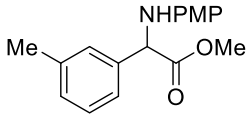


3e
enantioenriched

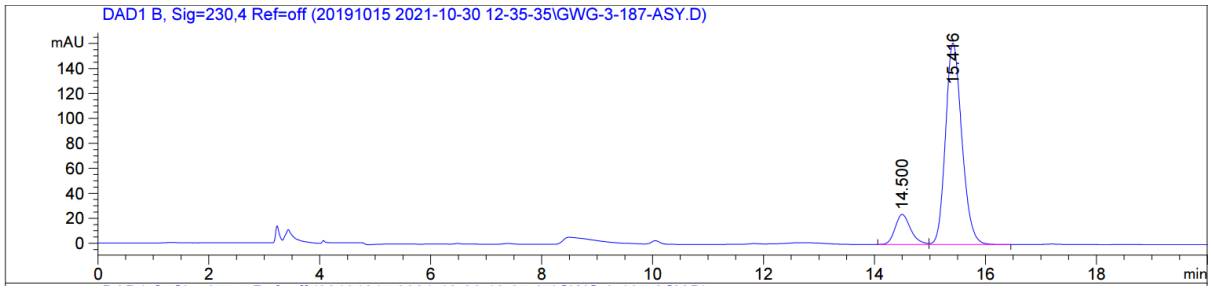
3f, HPLC conditions: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



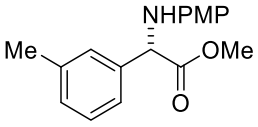
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	14.104	BV	0.2821	1.00171e4	550.99640	49.9132
2	15.015	VB	0.3001	1.00519e4	518.74847	50.0868



3f
racemic

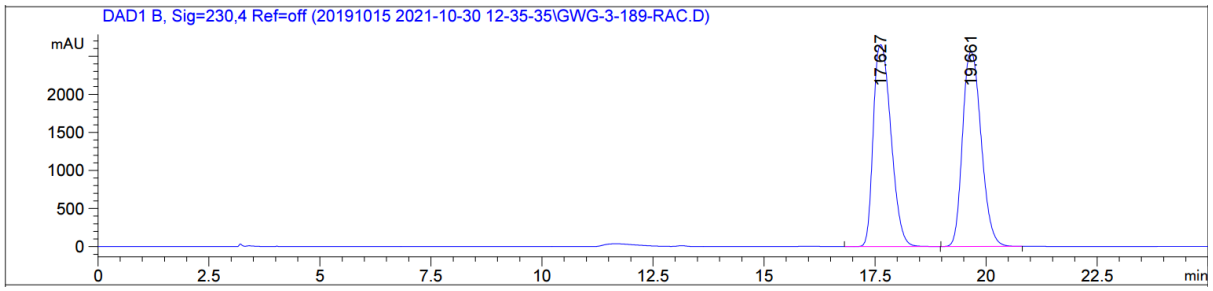


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	14.500	BV	0.2970	468.18585	24.05792	12.4200
2	15.416	VB	0.3146	3301.43066	161.41809	87.5800

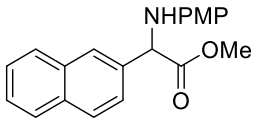


3f
enantioenriched

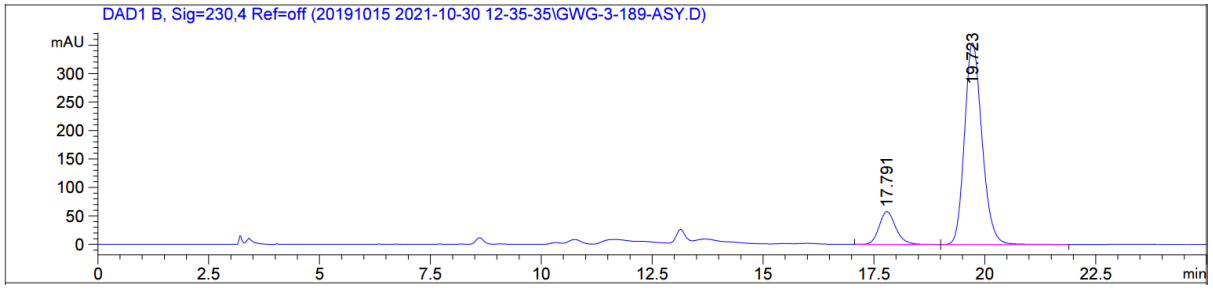
3g, HPLC conditions: Daicel CHIRALPAK® IC column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



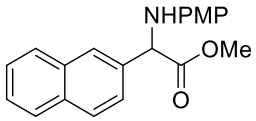
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	17.627	BB	0.4390	7.34088e4	2651.38354	49.6171
2	19.661	BB	0.4580	7.45420e4	2557.87256	50.3829



3g
racemic



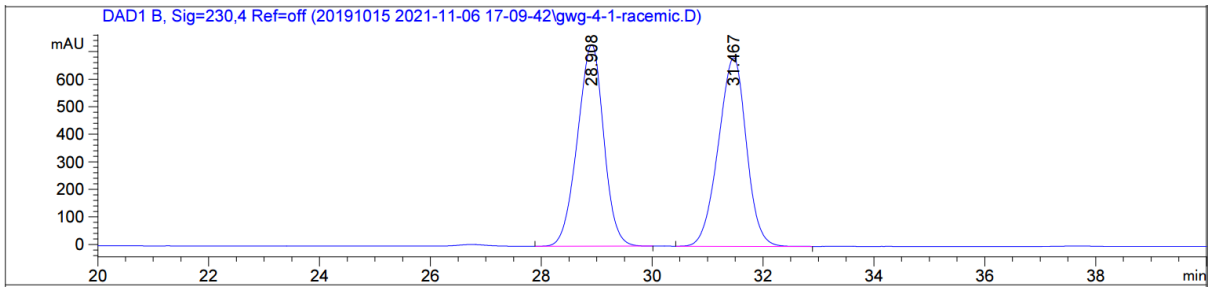
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	17.791	BB	0.4012	1506.67969	57.59026	13.4631
2	19.723	BB	0.4210	9684.53418	354.19104	86.5369



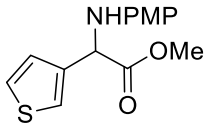
3g

enantioenriched

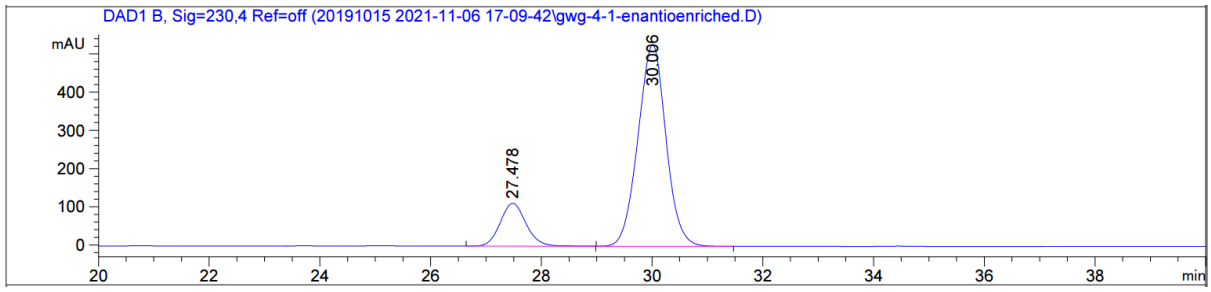
3h, HPLC conditions: Daicel CHIRALPAK® AD-H column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



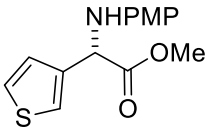
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	28.908	BB	0.5093	2.40305e4	731.49628	49.9879
2	31.467	BB	0.5420	2.40422e4	684.17914	50.0121



3h
racemic

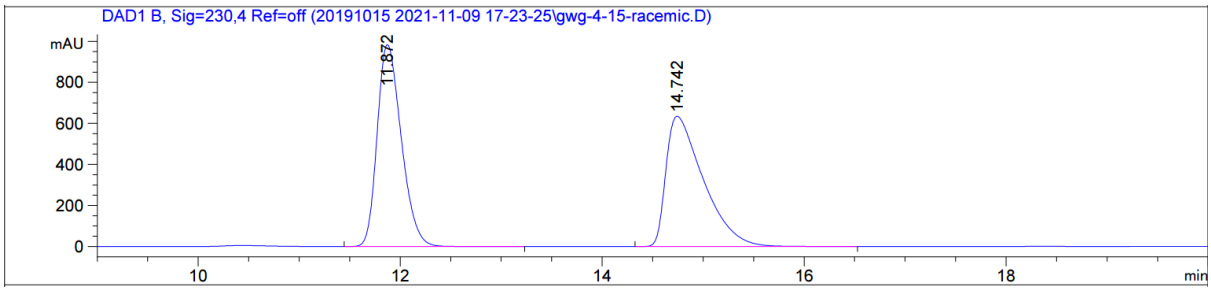


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	27.478	BB	0.5024	3633.85815	112.03840	16.4215
2	30.006	BB	0.5432	1.84948e4	527.35742	83.5785

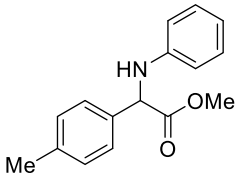


3h
enantioenriched

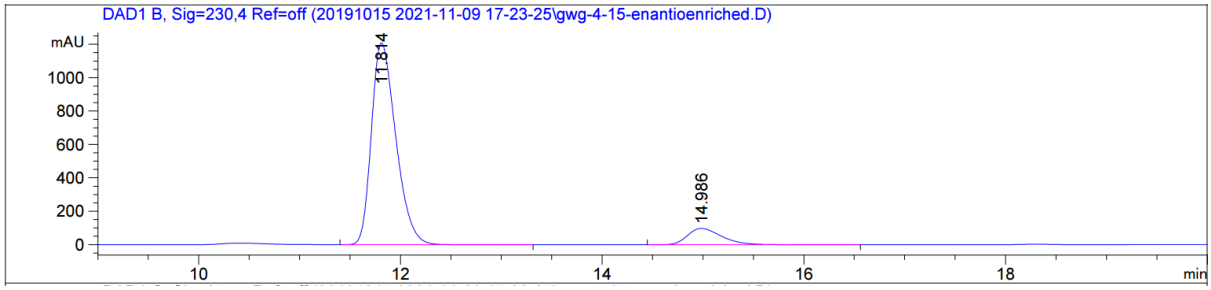
3i, HPLC conditions: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



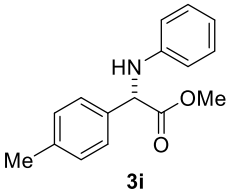
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	11.872	BB	0.2574	1.63158e4	984.09009	50.0247
2	14.742	BB	0.3855	1.62997e4	634.96027	49.9753



3i
racemic

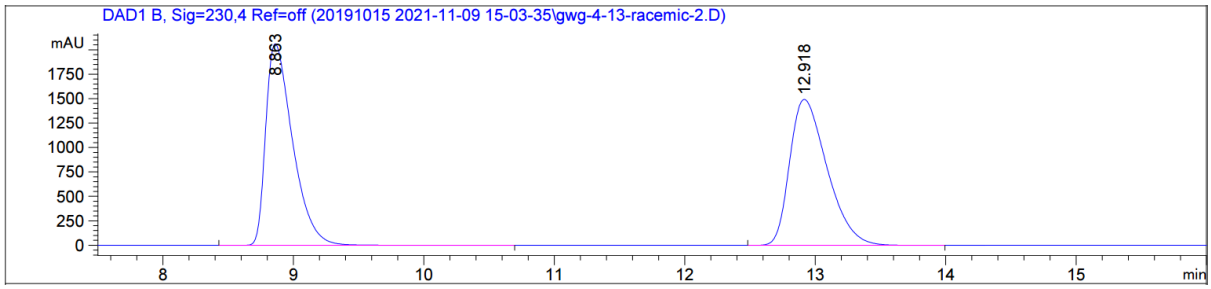


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	11.814	BB	0.2599	2.03010e4	1209.11328	89.7657
2	14.986	BB	0.3616	2314.55078	97.96838	10.2343

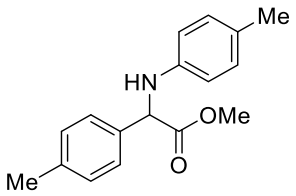


3i
enantioenriched

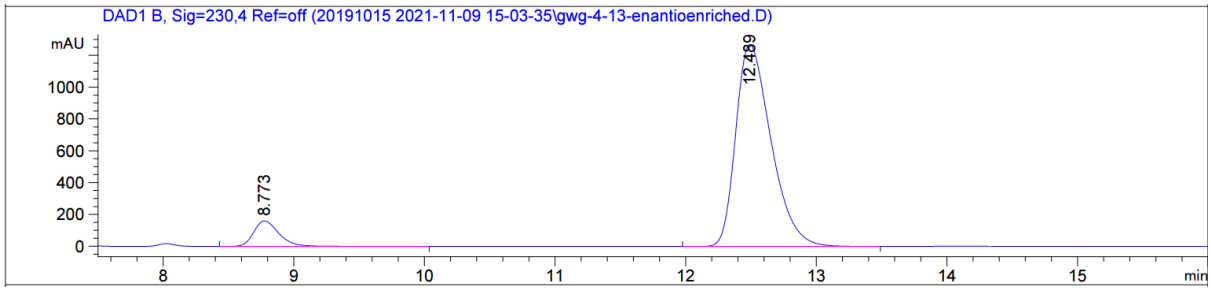
3j, HPLC conditions: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



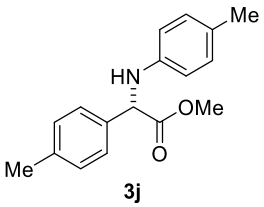
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	8.863	BB	0.2169	2.95105e4	2063.14307	49.8045
2	12.918	BB	0.3084	2.97421e4	1493.35608	50.1955



3j
racemic

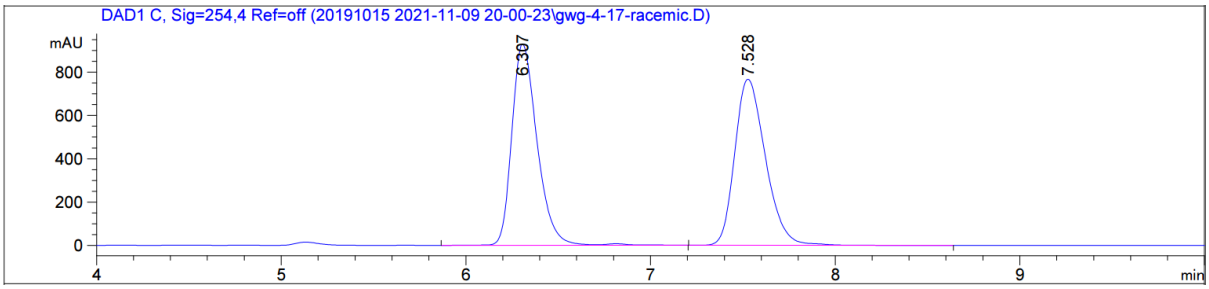


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	8.773	BB	0.2050	2155.88599	160.16408	8.3233
2	12.489	BB	0.2862	2.37459e4	1269.53259	91.6767

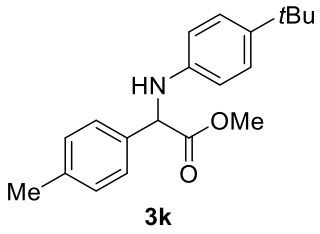


3j
enantioenriched

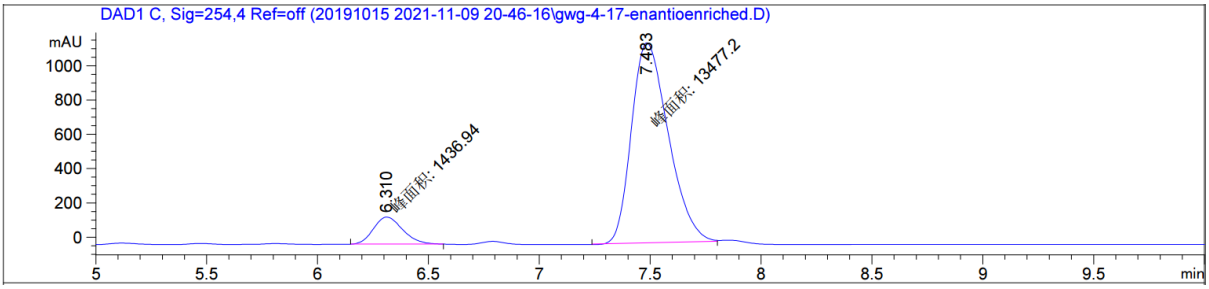
3k, HPLC conditions: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 254 nm.



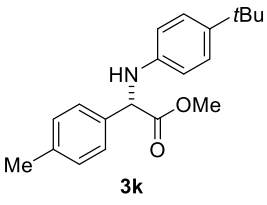
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	6.307	BV R	0.1459	8823.31543	929.33679	49.9784
2	7.528	BB	0.1785	8830.95117	766.28735	50.0216



3k
racemic

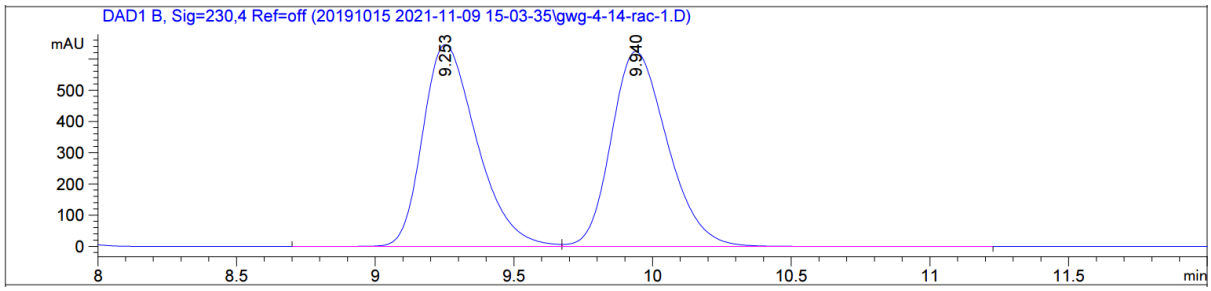


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	6.310	MM	0.1518	1436.94385	157.81908	9.6347
2	7.483	MM	0.1923	1.34772e4	1168.14136	90.3653

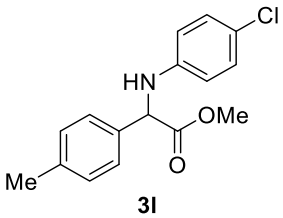


3k
enantioenriched

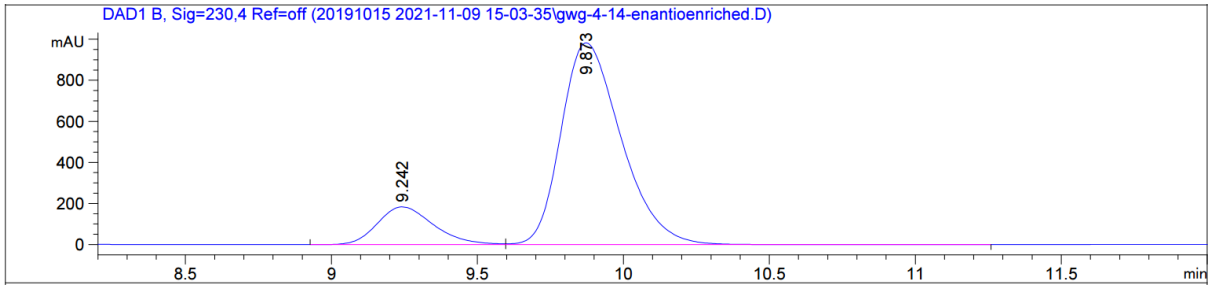
3l, HPLC conditions: Daicel CHIRALPAK® OD-H column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



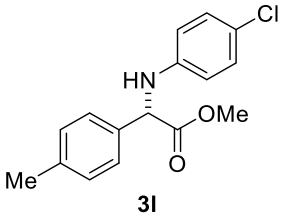
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	9.253	BV	0.2079	8751.49707	646.45105	49.8836
2	9.940	VB	0.2182	8792.34961	624.55621	50.1164



3l
racemic

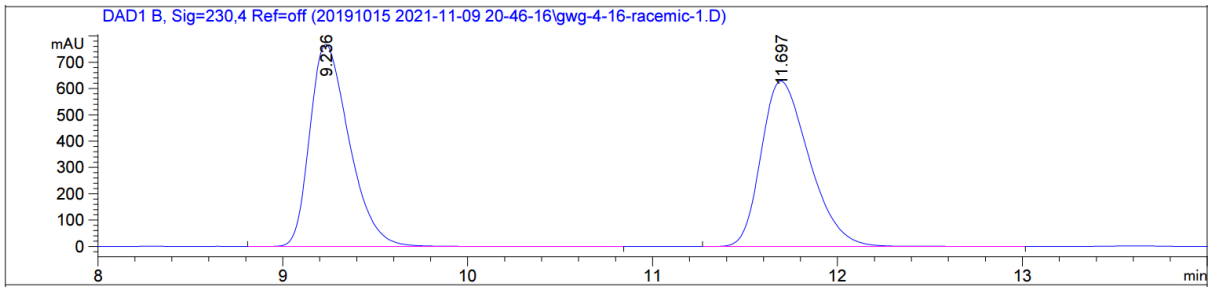


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	9.242	BV	0.2063	2464.50464	183.90395	14.8894
2	9.873	VB	0.2212	1.40875e4	982.93018	85.1106

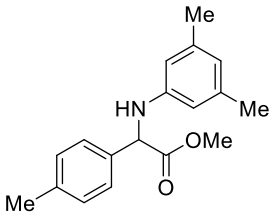


3l
enantioenriched

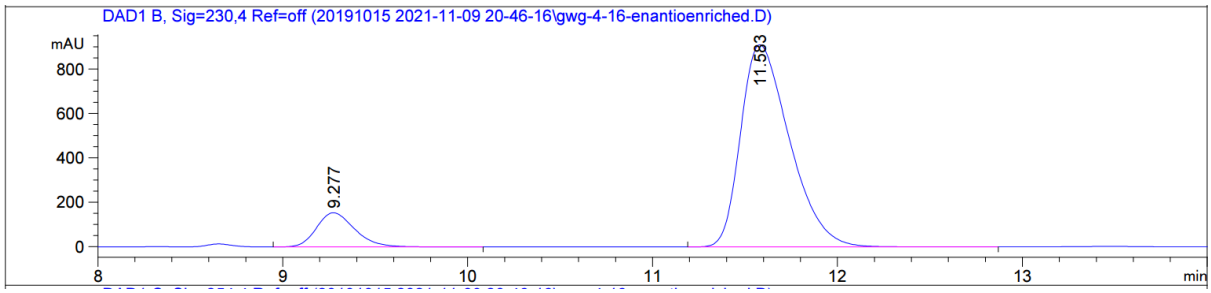
3m, HPLC conditions: Daicel CHIRALPAK® OD-H column; 2% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



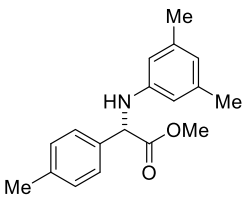
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	9.236	BB	0.2286	1.13511e4	767.46350	49.9691
2	11.697	BB	0.2786	1.13652e4	629.50305	50.0309



3m
racemic

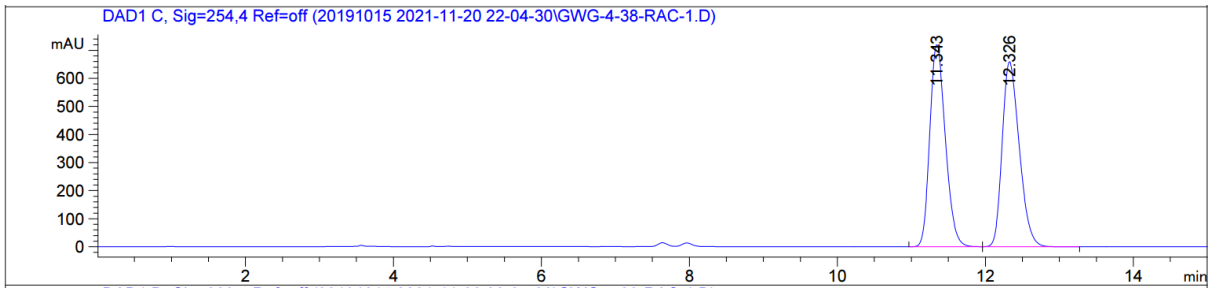


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	9.277	VB	0.2216	2205.47119	153.50519	11.6581
2	11.583	BB	0.2822	1.67124e4	910.15906	88.3419

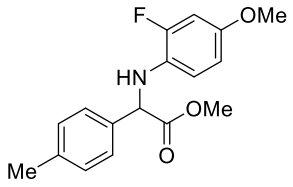


3m
enantioenriched

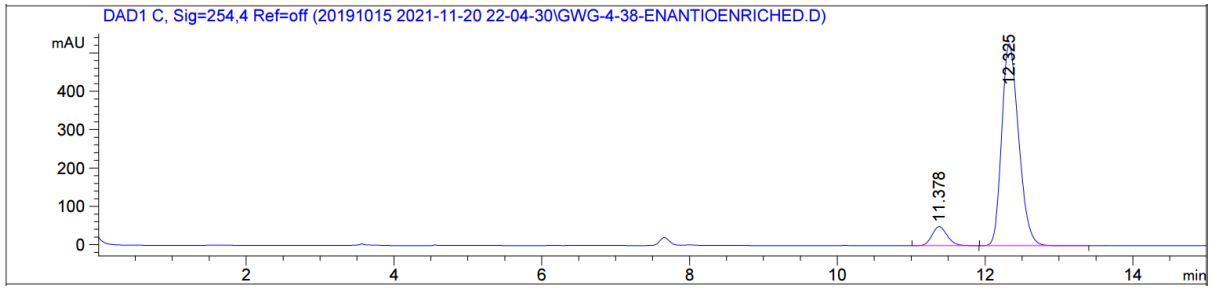
3n, HPLC conditions: Daicel CHIRALPAK® IC column; 3% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 254 nm.



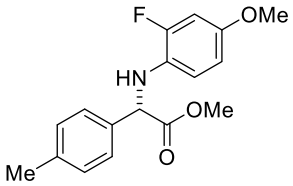
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	11.343	BB	0.2267	1.05354e4	720.06750	49.9260
2	12.326	BB	0.2490	1.05666e4	659.27466	50.0740



3n
racemic

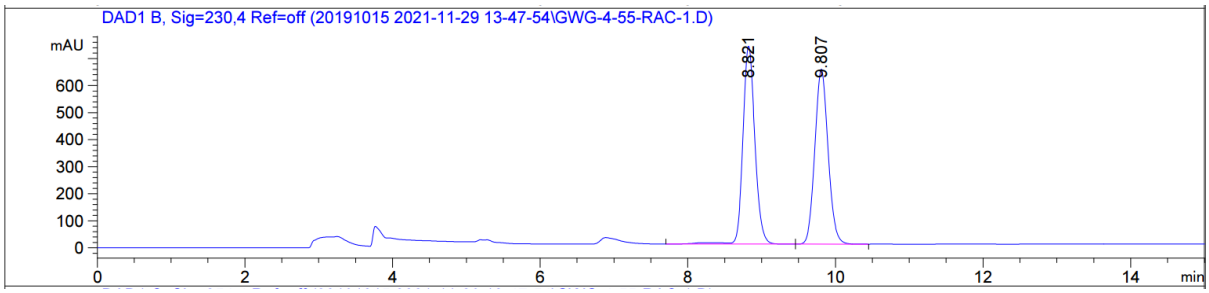


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	11.378	BB	0.2207	710.44806	49.71100	7.8279
2	12.325	BB	0.2474	8365.36816	526.36951	92.1721

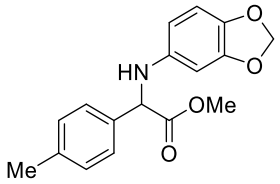


3n
enantioenriched

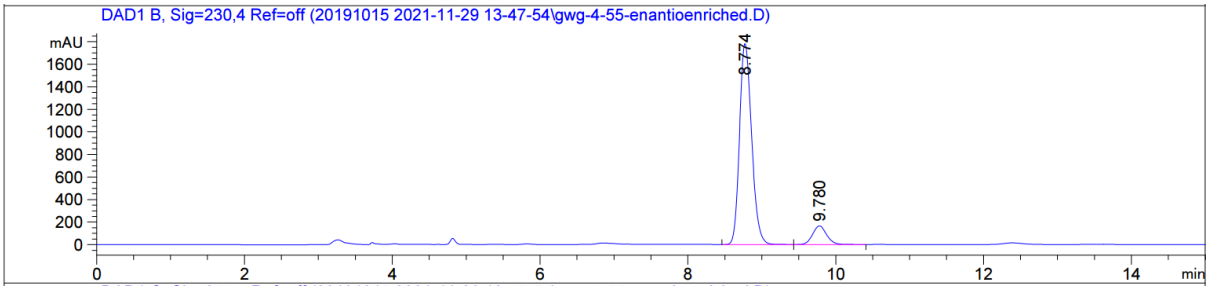
3o, HPLC conditions: Daicel CHIRALPAK® IC column; 20% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 230 nm.



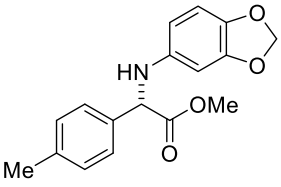
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	8.821	VB R	0.1733	8278.42773	732.69775	50.6115
2	9.807	BB	0.1917	8078.37451	646.95306	49.3885



3o
racemic

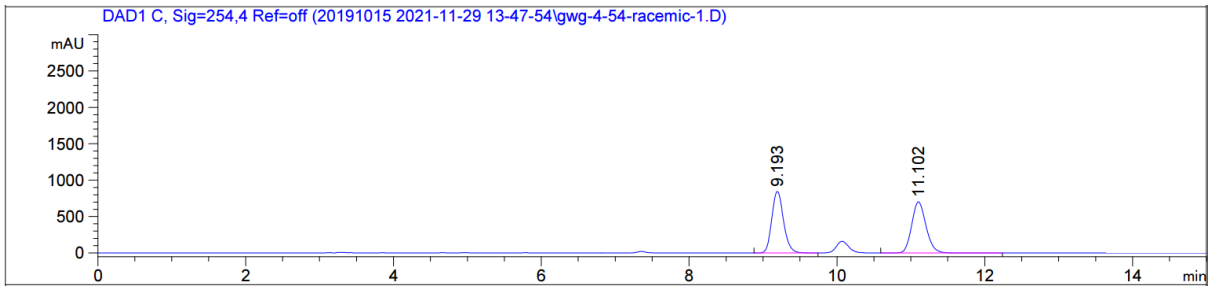


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	8.774	BB	0.1743	2.02085e4	1782.27161	90.2786
2	9.780	BB	0.2039	2176.09399	164.92154	9.7214

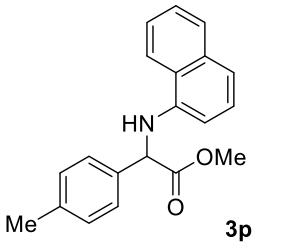


3o
enantioenriched

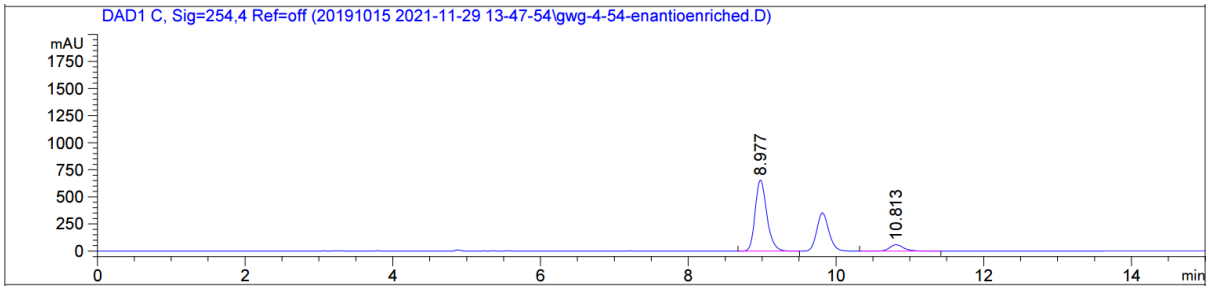
3p, HPLC conditions: Daicel CHIRALPAK® AD-H column; 5% *i*-PrOH in *n*-hexane; 1.0 mL/min, λ = 254 nm.



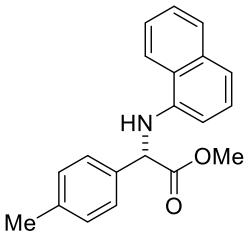
Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	9.193	BB	0.1707	9321.87988	845.11517	49.7879
2	11.102	BB	0.2081	9401.32031	702.62036	50.2121



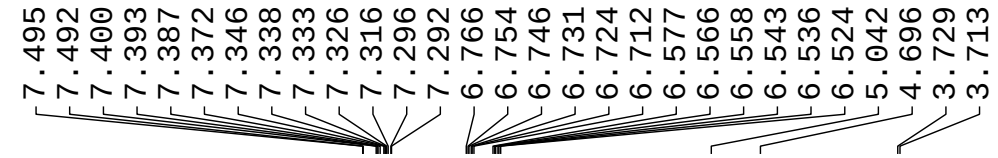
3p
racemic

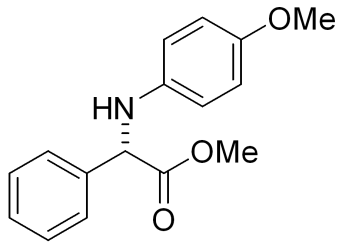


Peak	RetTime[min]	Type	Width[min]	Area[mAu*s]	Height[mAU]	Area[%]
1	8.977	BB	0.1682	7197.26758	654.83057	90.3025
2	10.813	BB	0.2021	772.90686	58.49901	9.6975



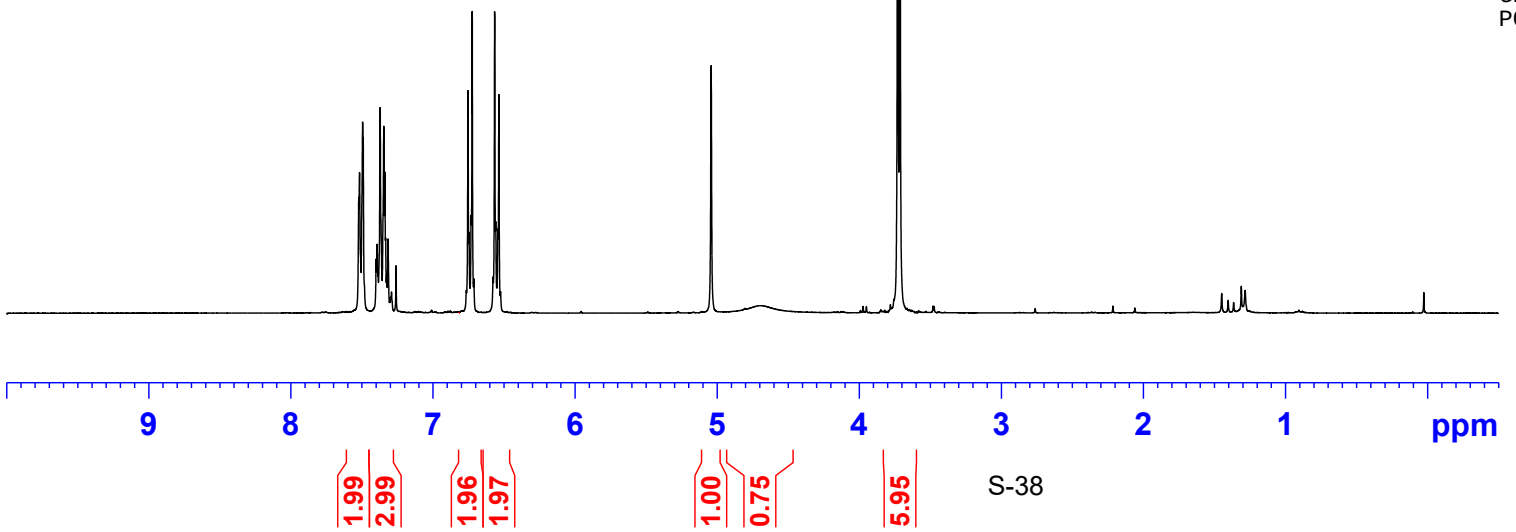
3p
enantioenriched

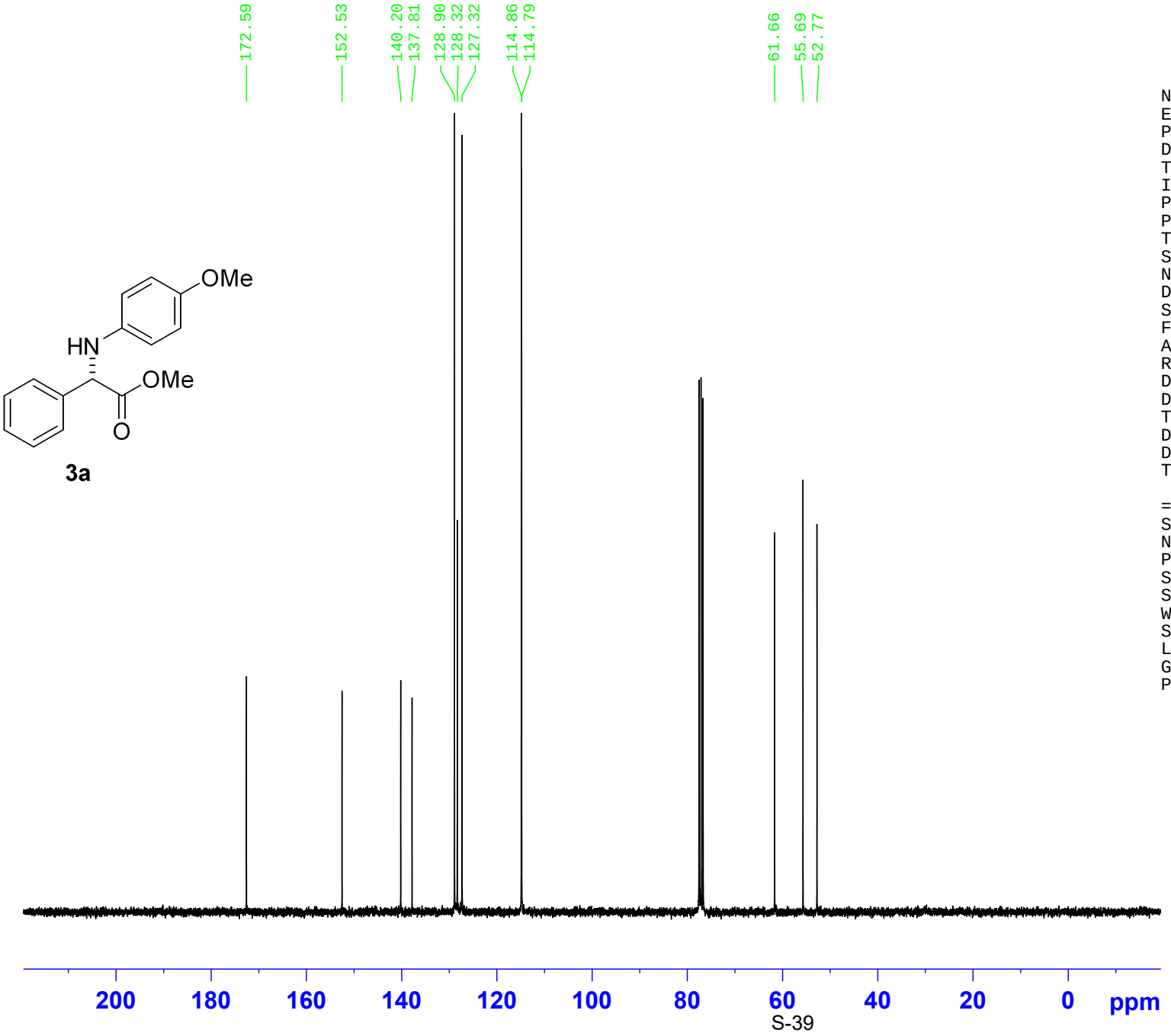




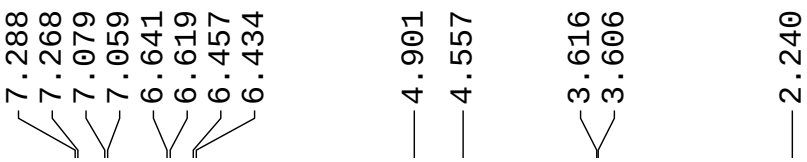
3a

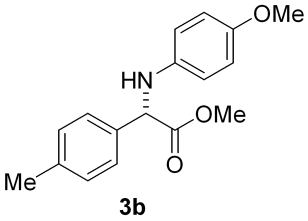
```
NAME      HNMR-gwg-3-176
EXPNO              5560
PROCNO                1
Date_          20211026
Time               9.13
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6009.615 Hz
FIDRES        0.091699 Hz
AQ            5.4526453 sec
RG                 80.6
DW               83.200 usec
DE                6.50 usec
TE                -59.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        300.1318534 MHz
NUC1                  1H
P1                10.00 usec
SI                65536
SF          300.1300072 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```





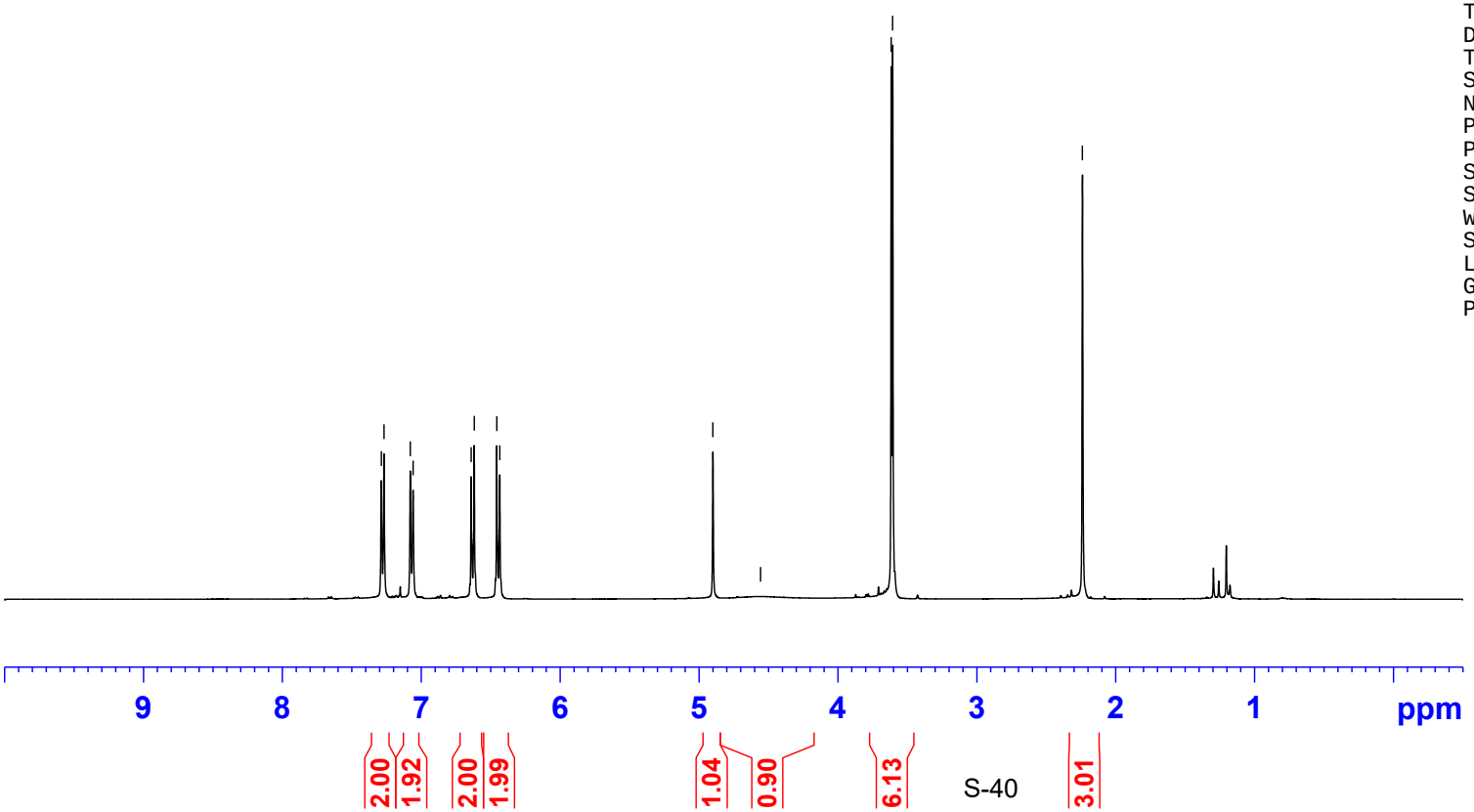

```
NAME      CNMR-gwg-3-176
EXPNO              5561
PROCNO                1
Date_          20211026
Time               9.59
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  700
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW               27.733 usec
DE                6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

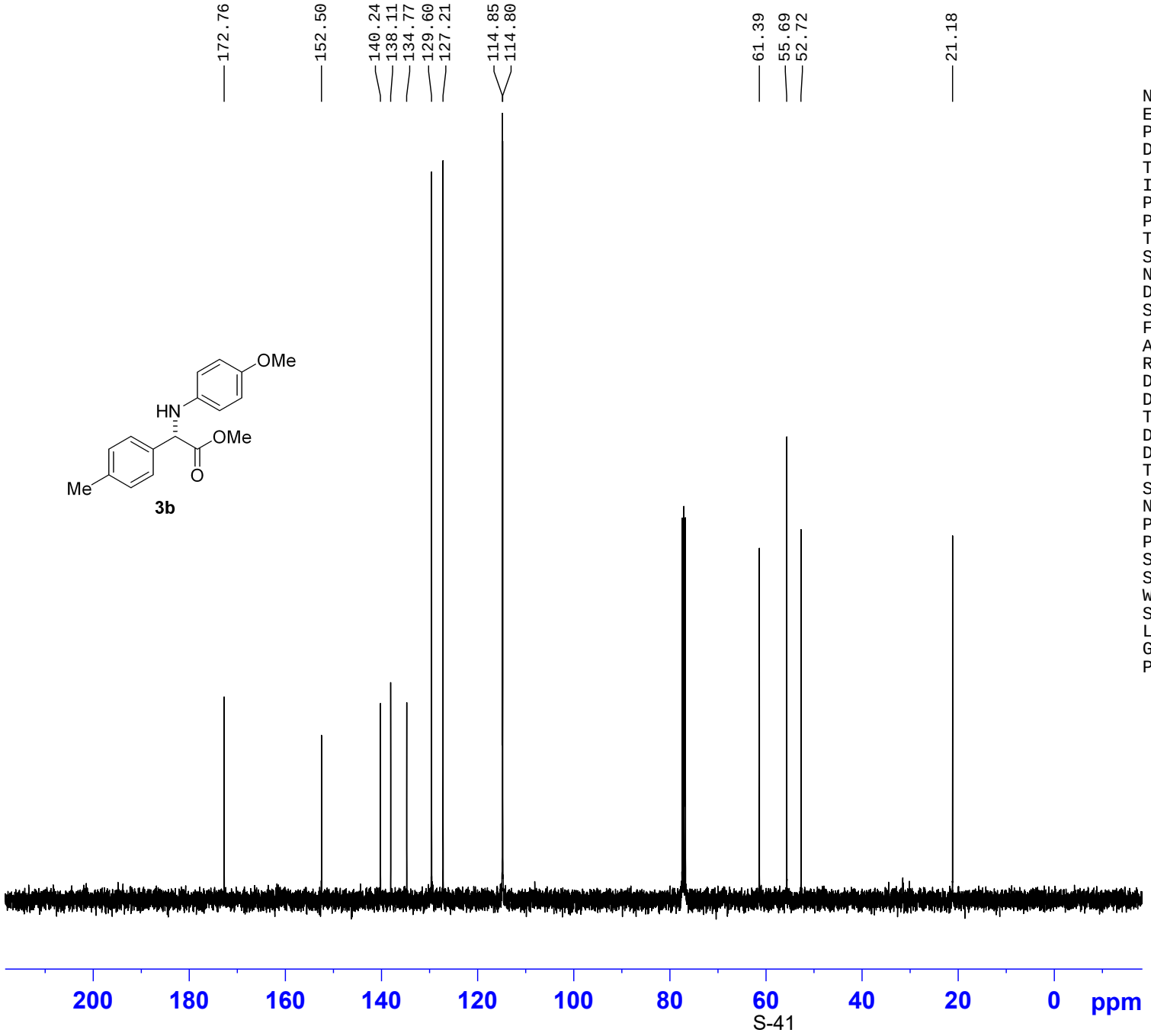




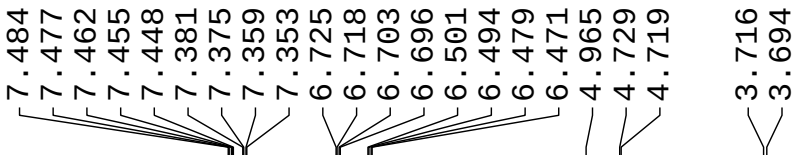
3b

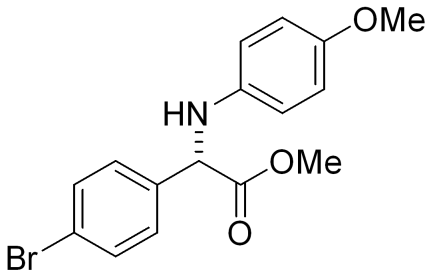
```
NAME        HNMR-gwg-3-178
EXPNO                   1
PROCNO                  1
Date_            20221102
Time                18.50 h
INSTRUM           Avance
PROBHD    Z116098_0833 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8196.722 Hz
FIDRES           0.250144 Hz
AQ               3.9977460 sec
RG                  47.619
DW                  61.000 usec
DE                  13.54 usec
TE                  294.5 K
D1              1.00000000 sec
TD0                     1
SFO1           400.1324708 MHz
NUC1                   1H
P0                  3.33 usec
P1                 10.00 usec
SI                  65536
SF             400.1300535 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```





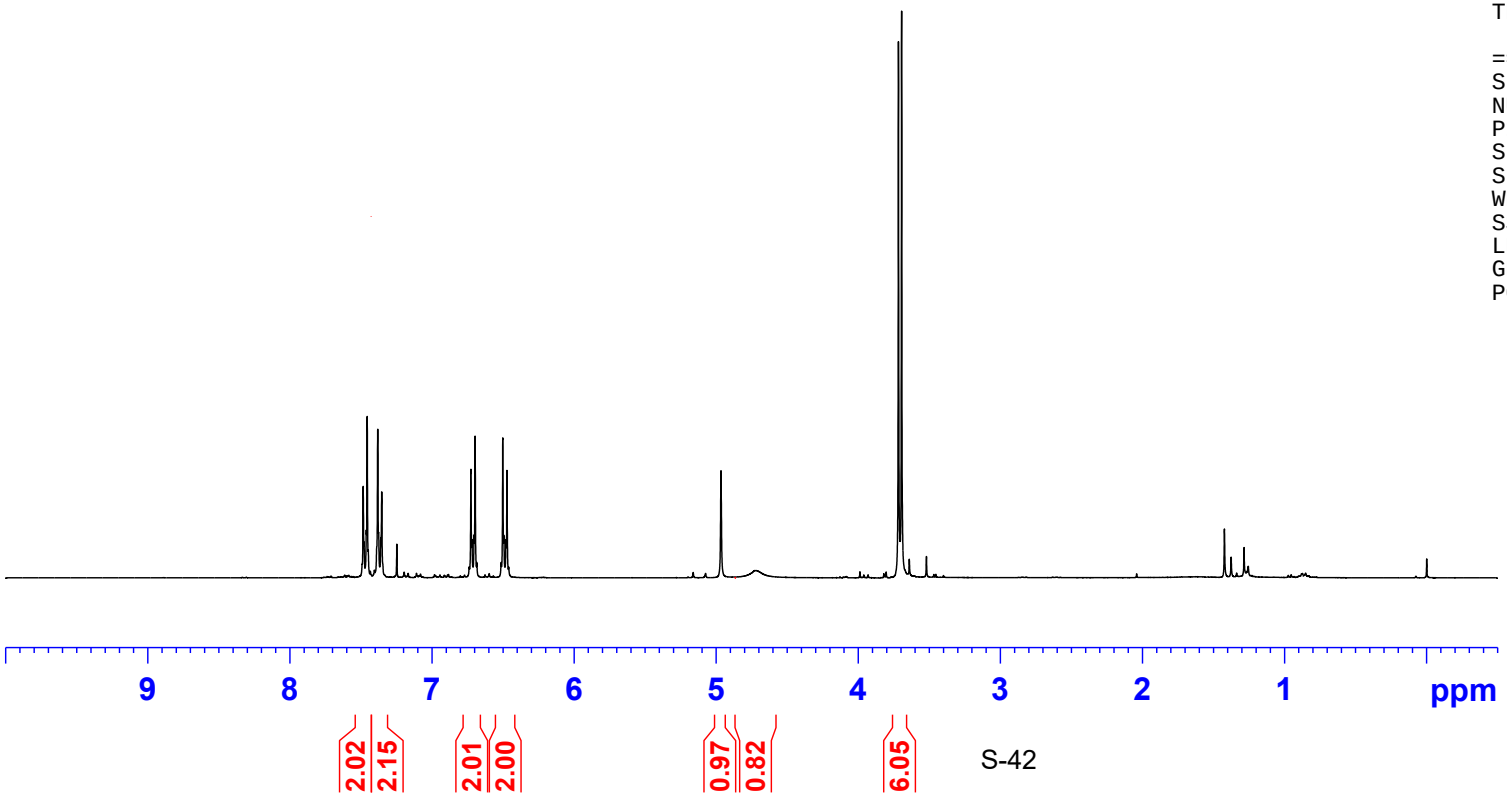
NAME	CNMR-gwg-3-178
EXPNO	2
PROCNO	1
Date_	20221102
Time	18.54 h
INSTRUM	Avance
PROBHD	Z116098_0833 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	50
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	47.4244
DW	21.000 usec
DE	6.50 usec
TE	295.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P0	3.33 usec
P1	10.00 usec
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

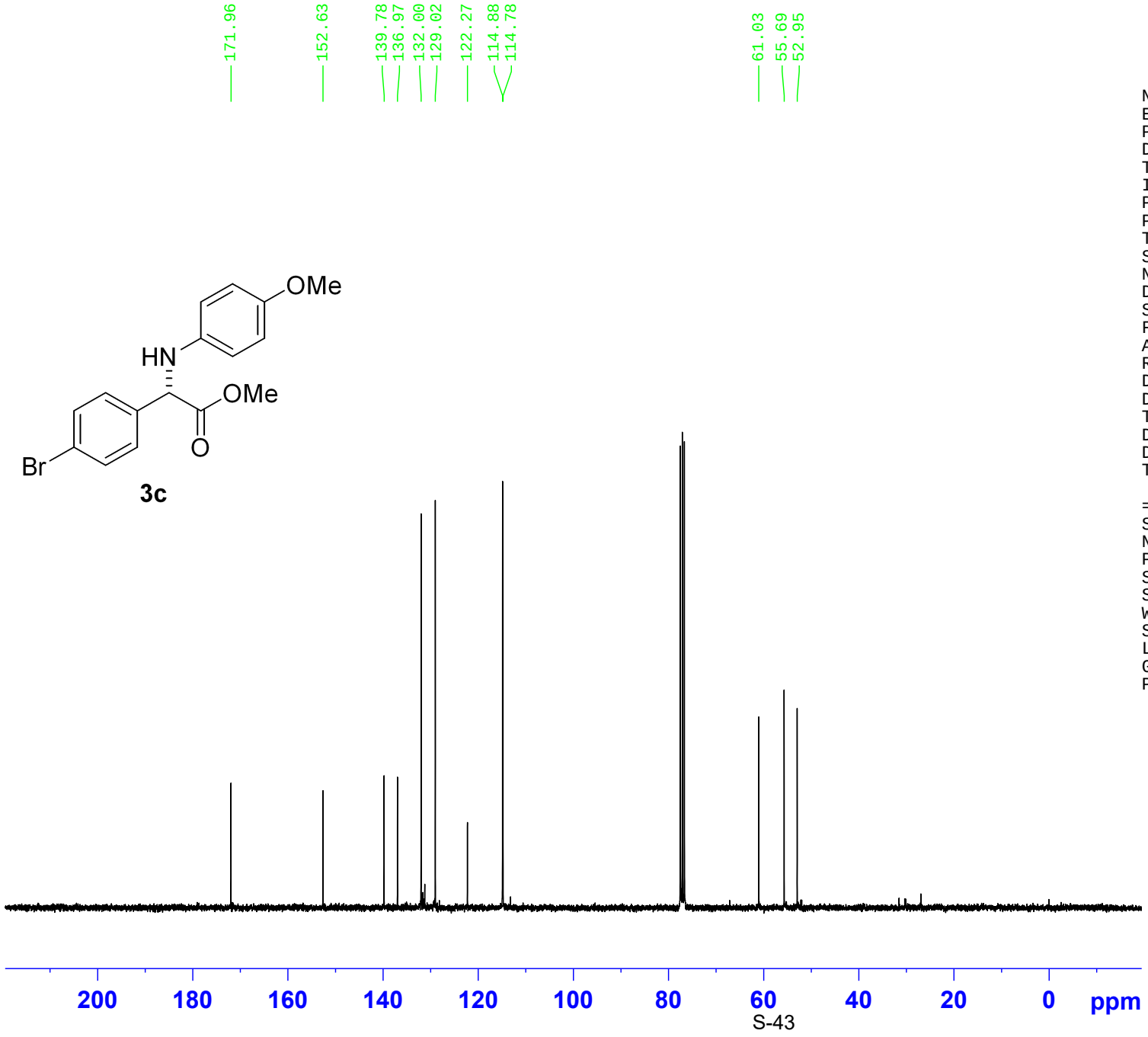




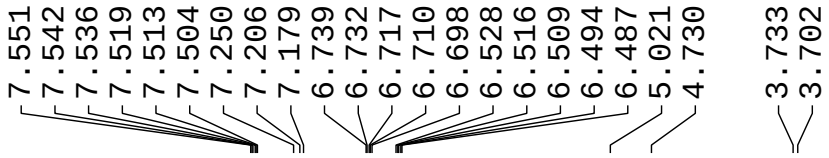
3c

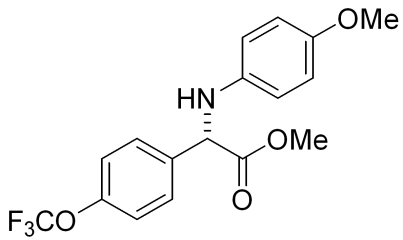
```
NAME      HNMR-gwg-3-177
EXPNO               5562
PROCNO                 1
Date_           20211026
Time               10.04
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ             5.4526453 sec
RG                   101
DW                83.200 usec
DE                 6.50 usec
TE                -59.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 10.00 usec
SI                 65536
SF           300.1300111 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```





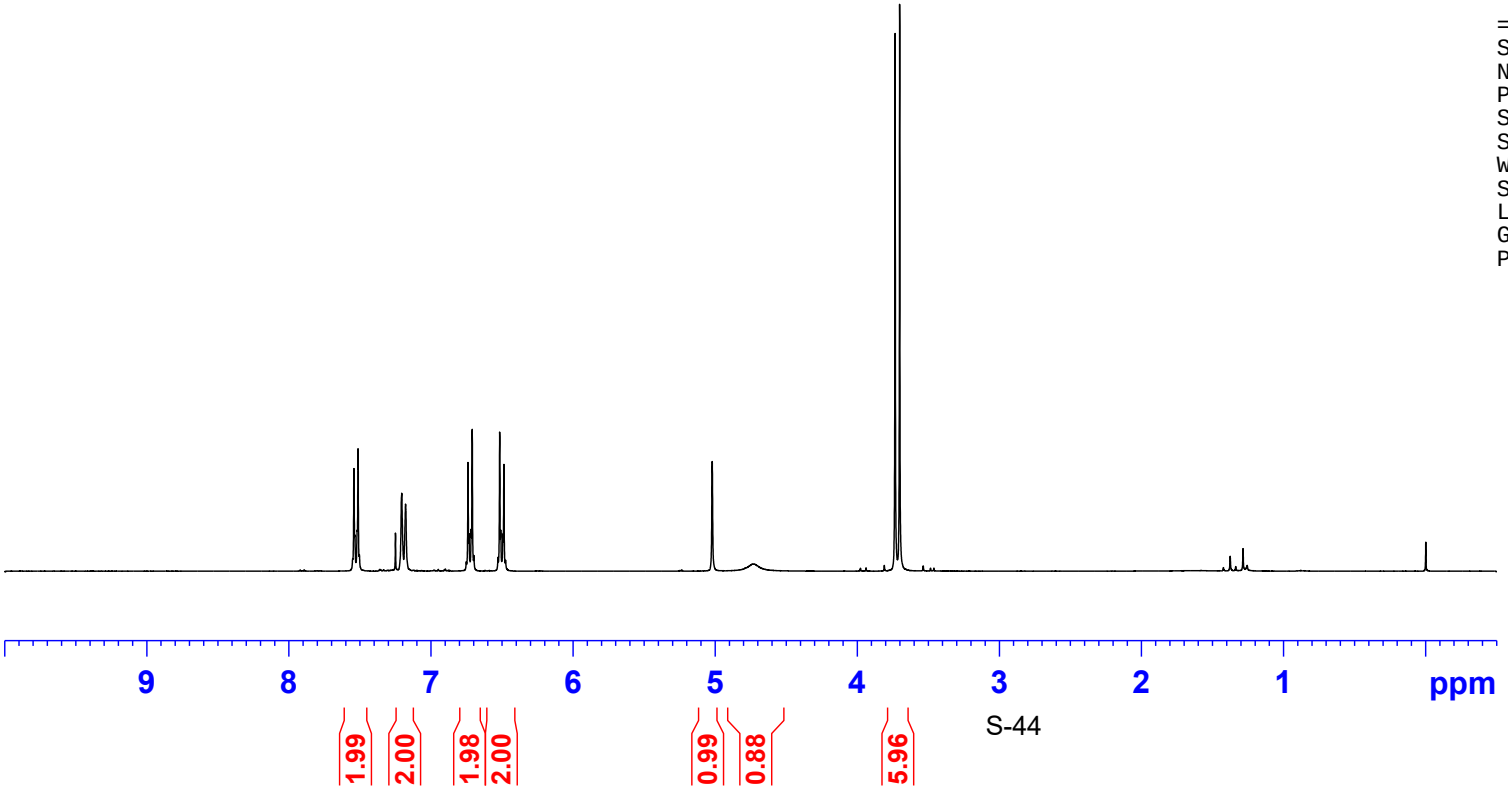

```
NAME      CNMR-gwg-3-177
EXPNO              5563
PROCNO                1
Date_          20211026
Time              10.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  700
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW               27.733 usec
DE                6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

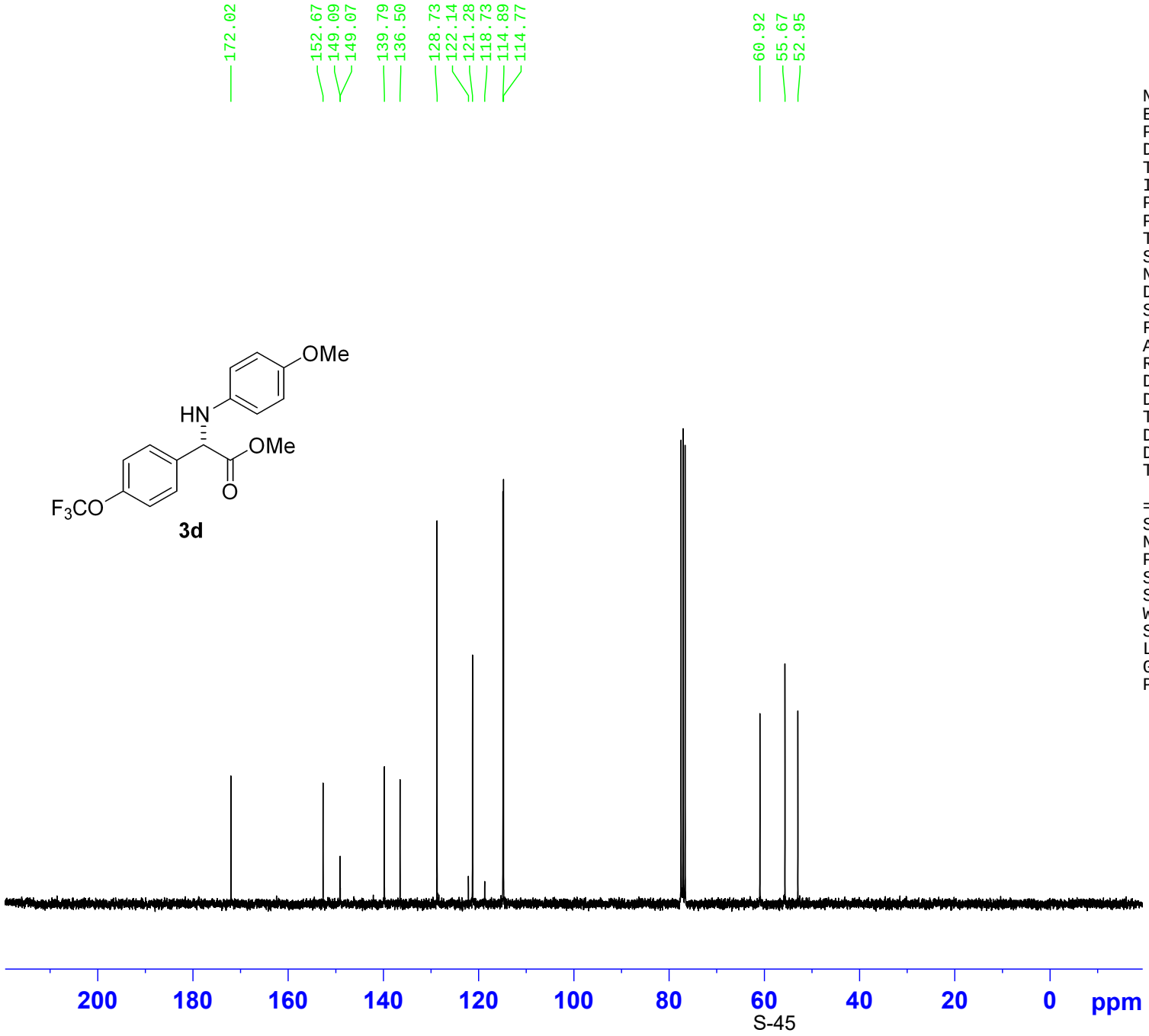




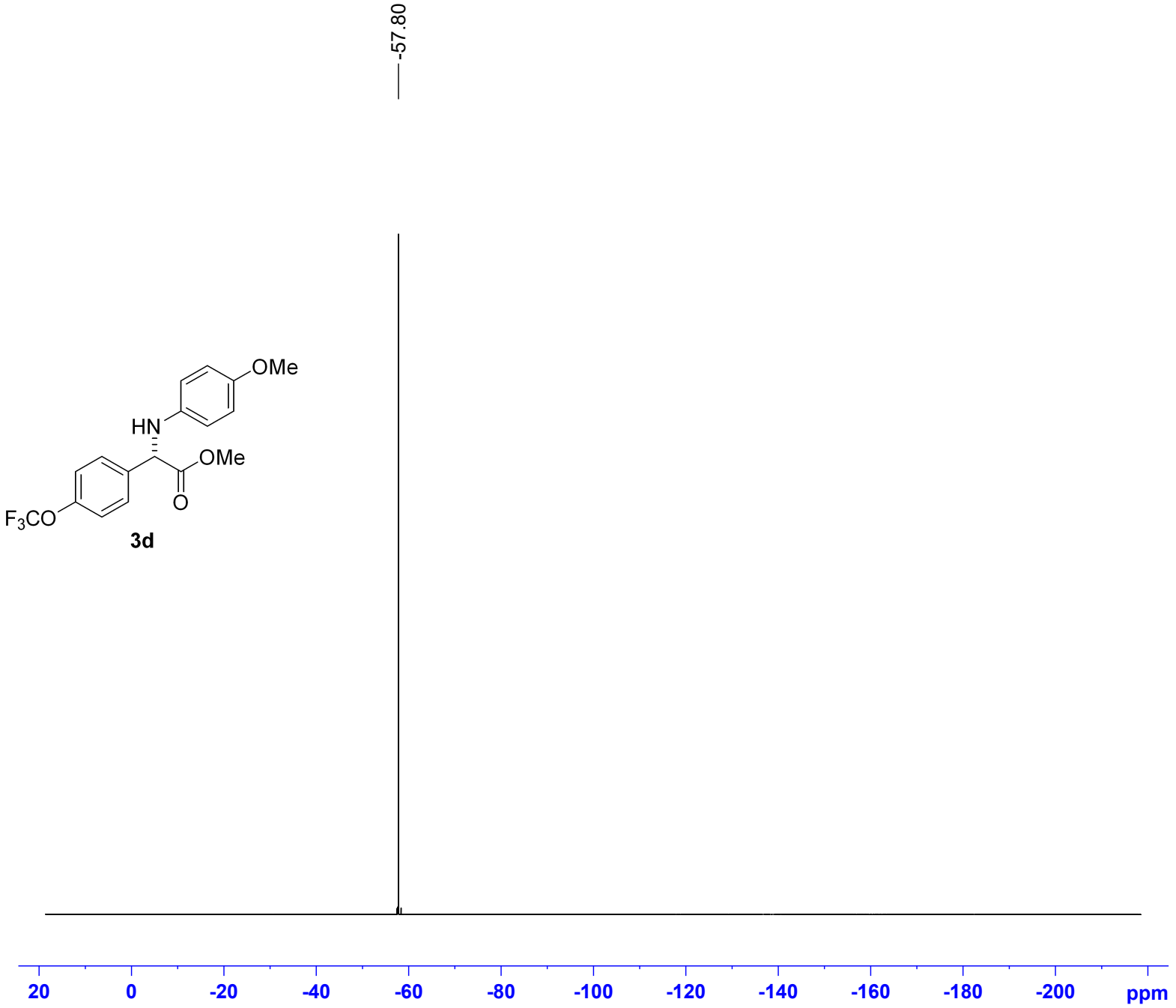
3d

```
NAME      HNMR-gwg-3-191
EXPNO              5586
PROCNO                1
Date_          20211029
Time              23.13
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6009.615 Hz
FIDRES        0.091699 Hz
AQ            5.4526453 sec
RG                  114
DW               83.200 usec
DE                6.50 usec
TE                -59.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        300.1318534 MHz
NUC1                  1H
P1                10.00 usec
SI                65536
SF          300.1300102 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

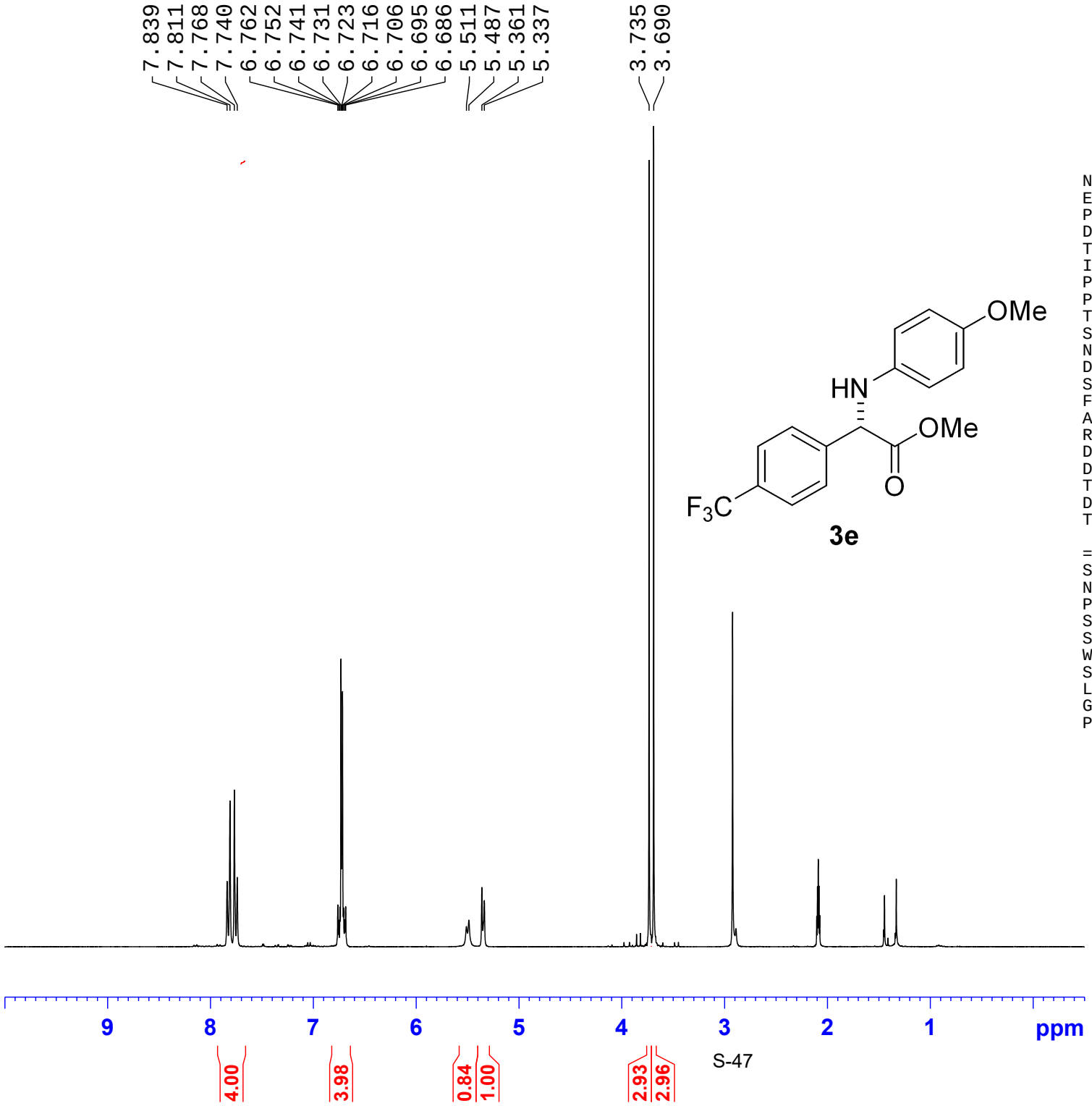




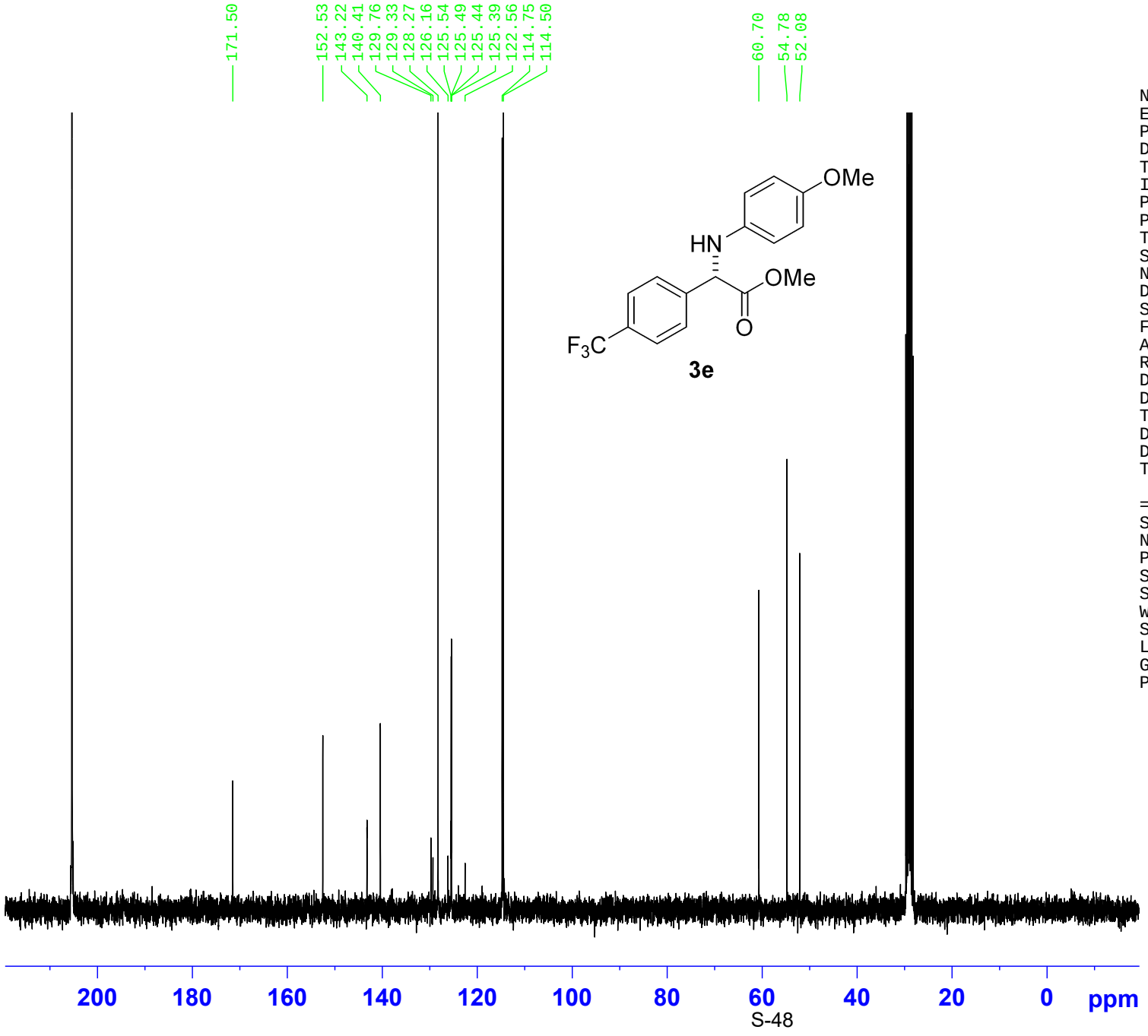

```
NAME      CNMR-gwg-3-191
EXPNO              5588
PROCNO                1
Date_          20211029
Time              23.43
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  400
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW               27.733 usec
DE                6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



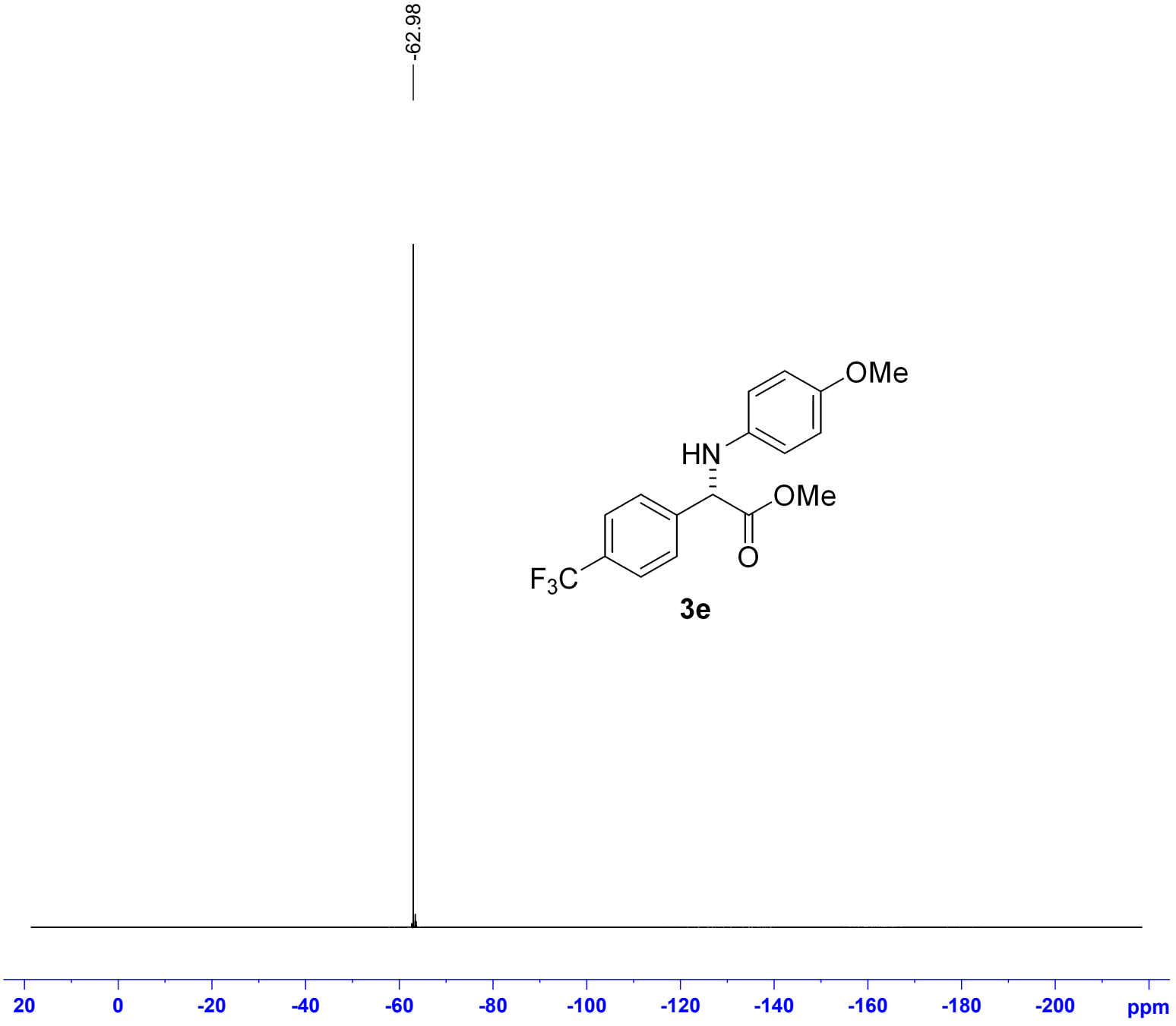

```
NAME      FNMR-gwg-3-191
EXPNO               5587
PROCNO                 1
Date_           20211029
Time               23.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG       zgfhigqn.2
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            66964.289 Hz
FIDRES          0.510897 Hz
AQ              0.9787210 sec
RG                   203
DW                 7.467 usec
DE                 6.50 usec
TE                -59.1 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         282.3761148 MHz
NUC1                 19F
P1                 14.50 usec
SI                 65536
SF           282.4043552 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



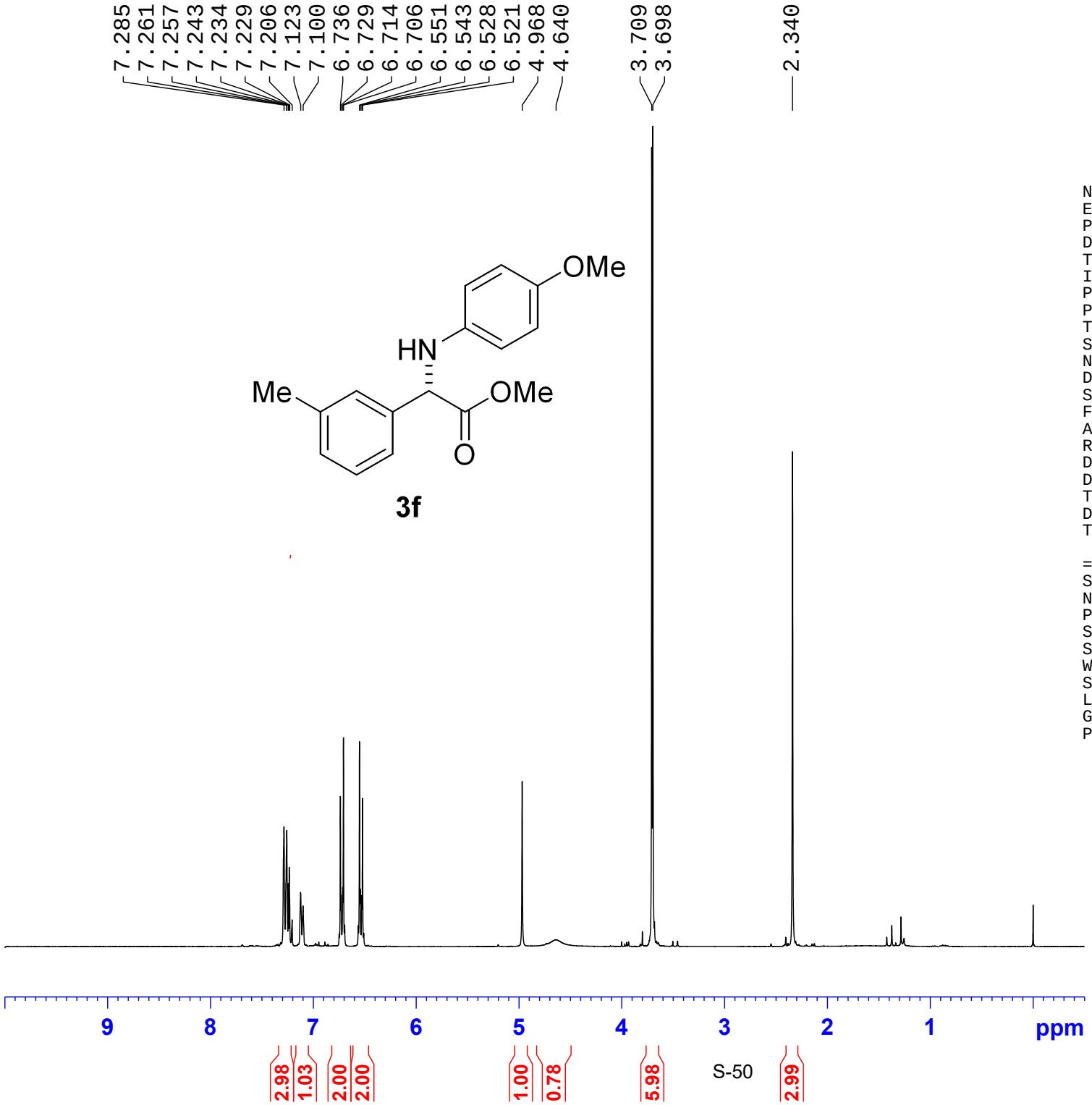
NAME	HNMR-gwg-4-3
EXPNO	5625
PROCNO	1
Date_	20211116
Time	13.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	Acetone
NS	16
DS	2
SWH	6009.615 Hz
FIDRES	0.091699 Hz
AQ	5.4526453 sec
RG	128
DW	83.200 usec
DE	6.50 usec
TE	-59.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	10.00 usec
SI	65536
SF	300.1299930 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



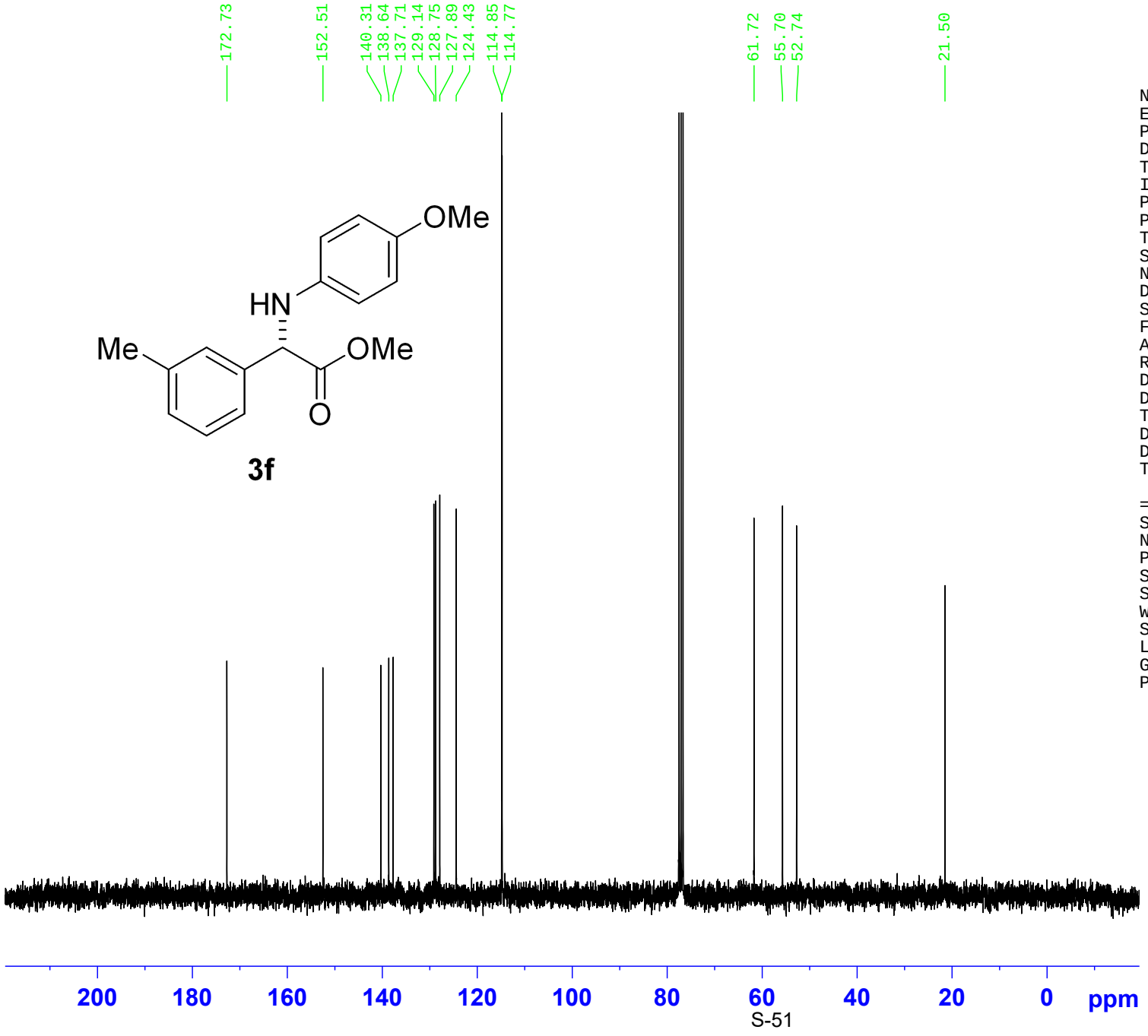

```
NAME          CNMR-gwg-4-3
EXPNO                 5627
PROCNO                   1
Date_             20211116
Time                 13.25
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT             Acetone
NS                      267
DS                        4
SWH               18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175818 sec
RG                      203
DW                   27.733 usec
DE                    6.50 usec
TE                   -59.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1              75.4752949 MHz
NUC1                     13C
P1                    9.50 usec
SI                    32768
SF                75.4677485 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```



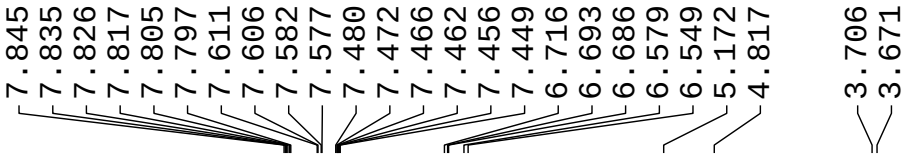

```
NAME        FNMR-gwg-4-3
EXPNO               5626
PROCNO                 1
Date_           20211116
Time               13.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG       zgfhigqn.2
TD                131072
SOLVENT          Acetone
NS                    16
DS                     4
SWH             66964.289 Hz
FIDRES          0.510897 Hz
AQ              0.9787210 sec
RG                   203
DW                 7.467 usec
DE                 6.50 usec
TE                -59.1 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         282.3761148 MHz
NUC1                 19F
P1                 14.50 usec
SI                 65536
SF           282.4043552 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

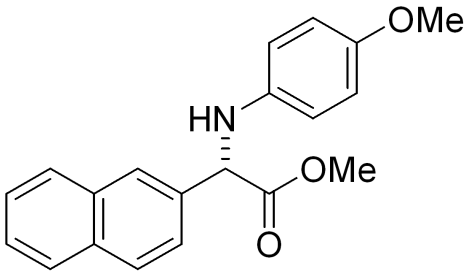



```
NAME      HNMR-gwg-3-190
EXPNO              5582
PROCNO                1
Date_          20211029
Time              22.28
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6009.615 Hz
FIDRES        0.091699 Hz
AQ            5.4526453 sec
RG                  101
DW               83.200 usec
DE                6.50 usec
TE                -59.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        300.1318534 MHz
NUC1                  1H
P1                10.00 usec
SI                65536
SF          300.1300121 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



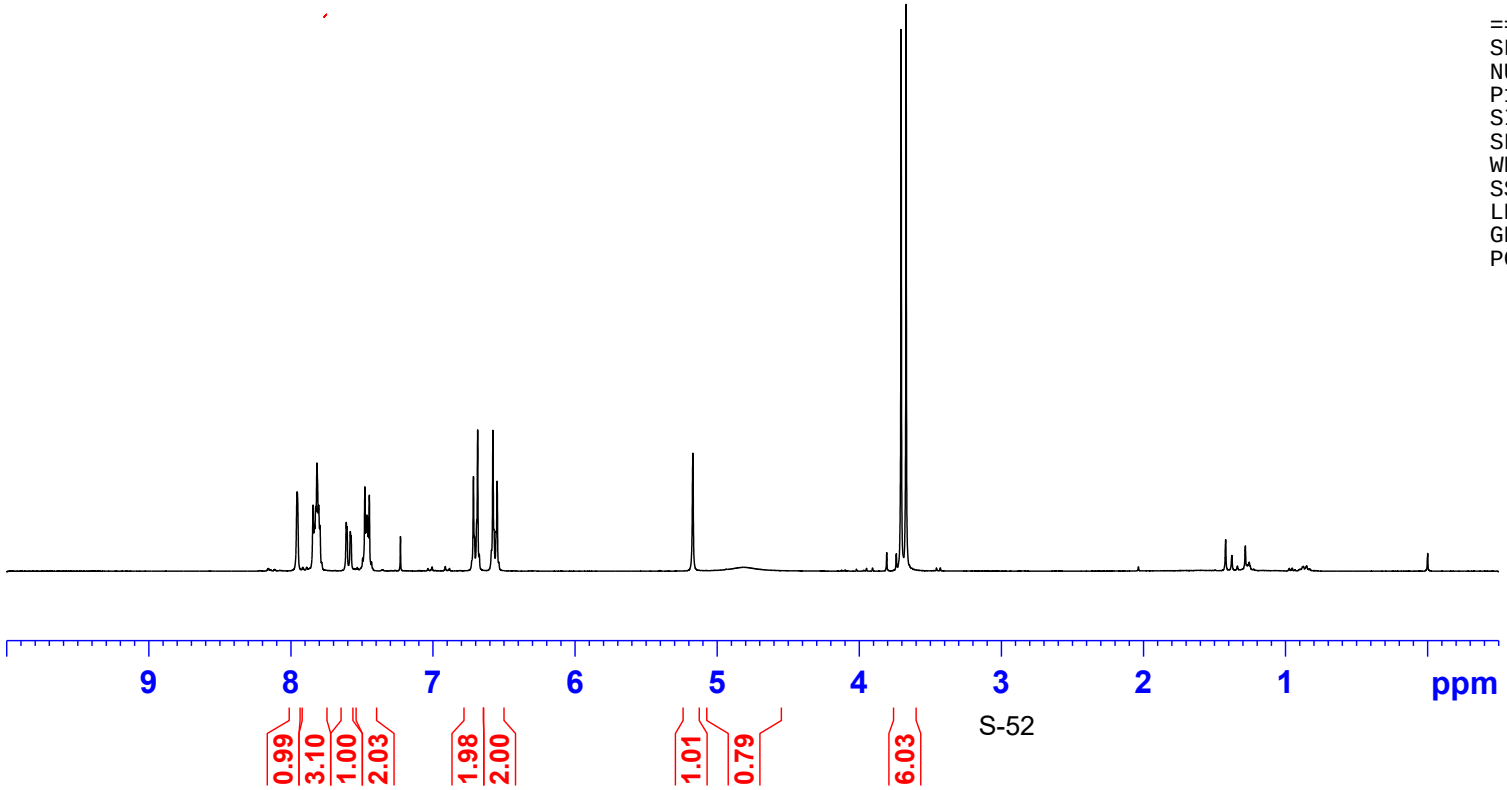

```
NAME      CNMR-gwg-3-190
EXPNO               5583
PROCNO                 1
Date_           20211029
Time               22.42
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  200
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW                27.733 usec
DE                 6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

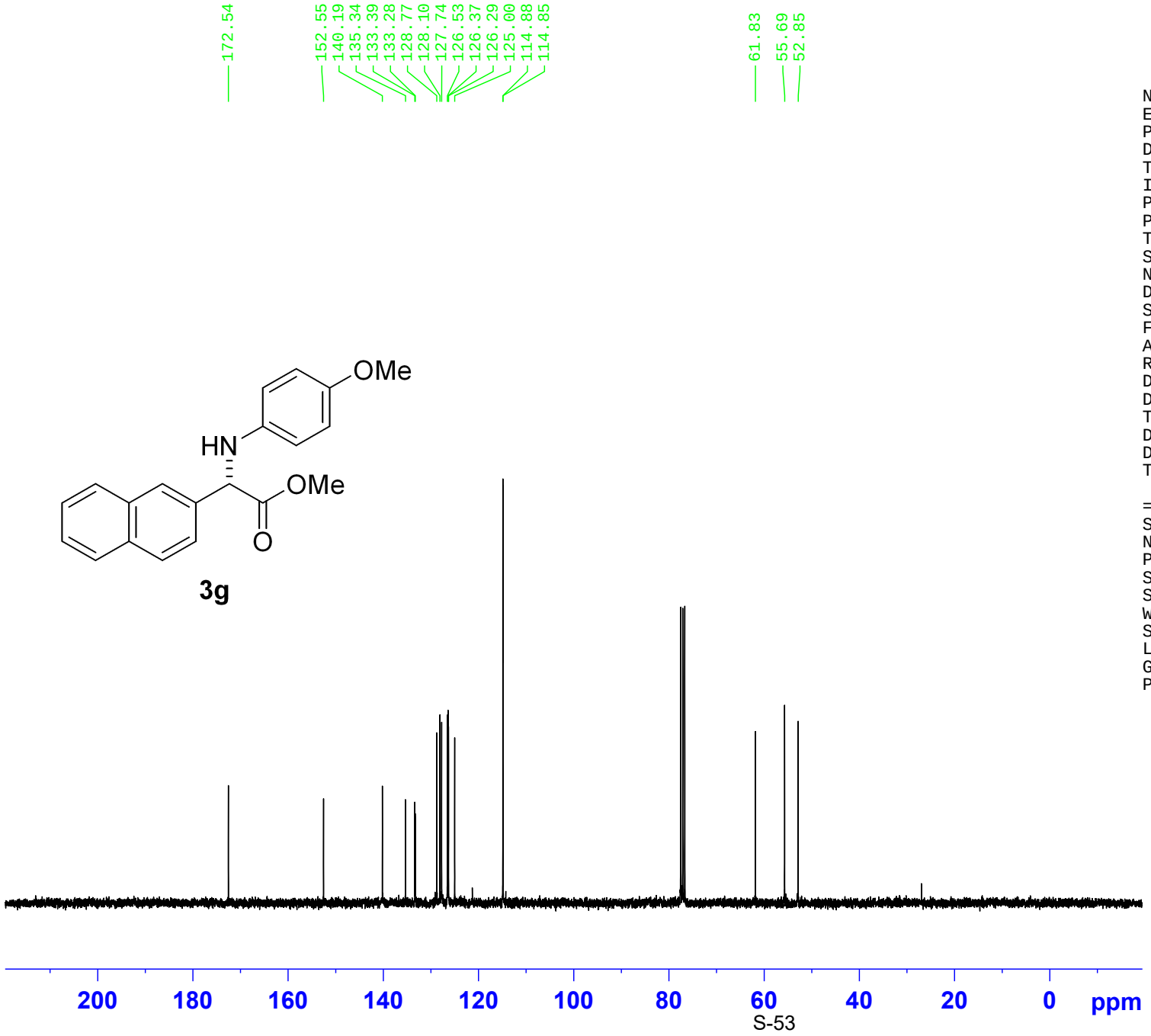




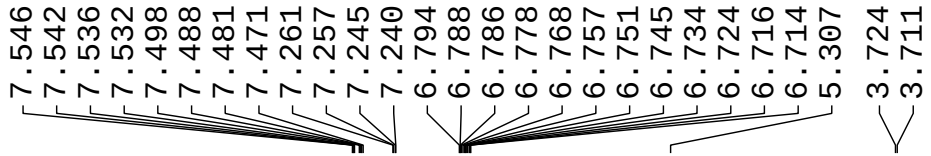
3g

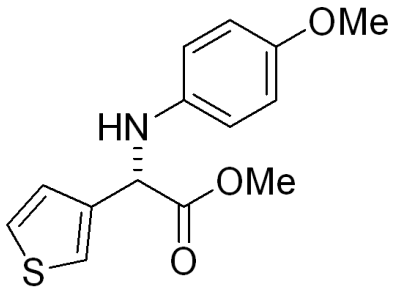
```
NAME      HNMR-gwg-3-192
EXPNO              5584
PROCNO                1
Date_          20211029
Time              22.47
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6009.615 Hz
FIDRES        0.091699 Hz
AQ            5.4526453 sec
RG                  101
DW               83.200 usec
DE                6.50 usec
TE                -59.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        300.1318534 MHz
NUC1                  1H
P1                10.00 usec
SI                65536
SF          300.1300161 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```





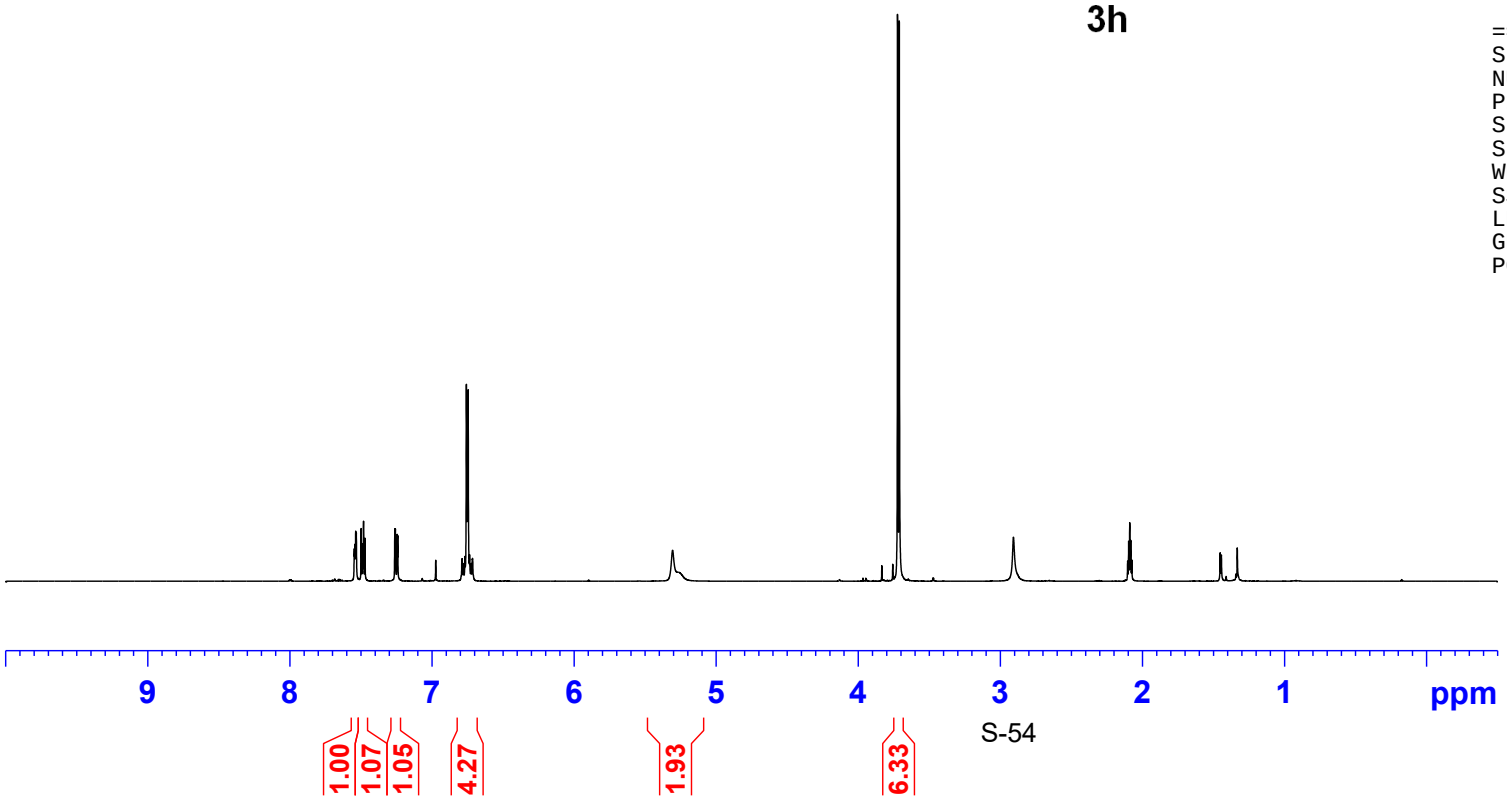

```
NAME      CNMR-gwg-3-192
EXPNO              5585
PROCNO                1
Date_          20211029
Time              23.08
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  300
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW               27.733 usec
DE                6.50 usec
TE                -59.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

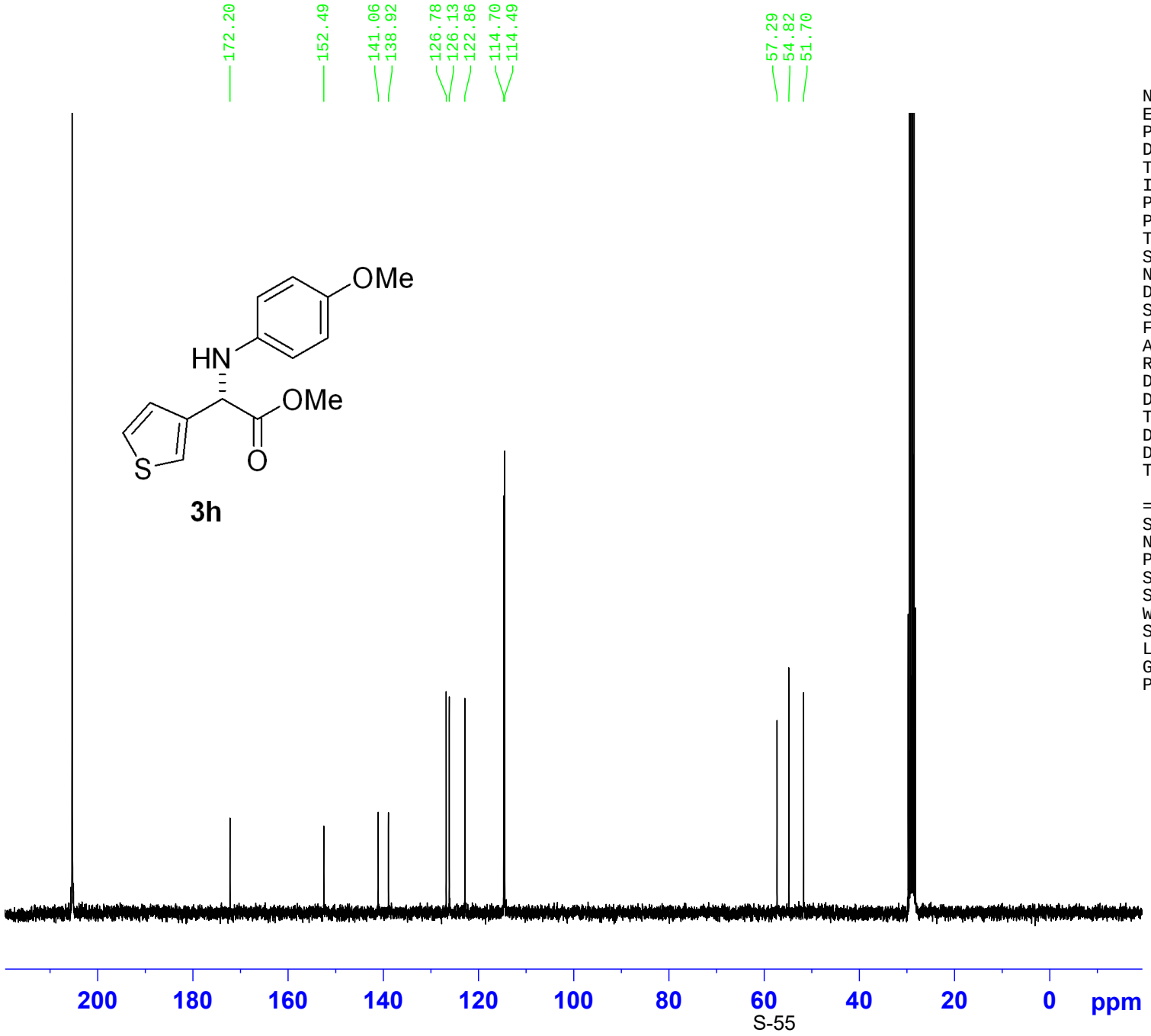




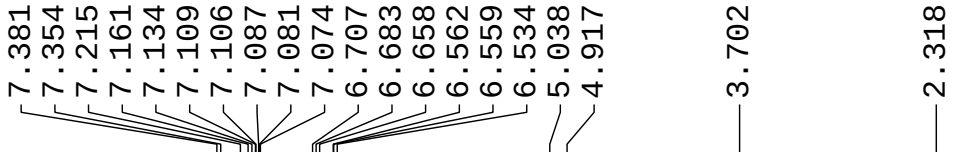
3h

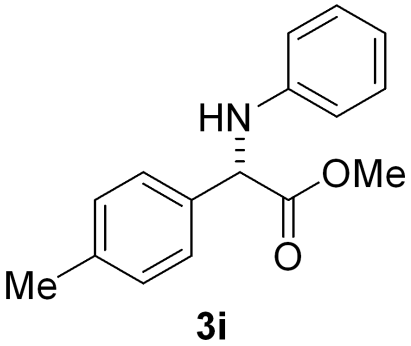
```
NAME        HNMR-gwg-4-1
EXPNO               5605
PROCNO                 1
Date_           20211113
Time               10.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4526453 sec
RG                   144
DW                83.200 usec
DE                 6.50 usec
TE                -59.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 10.00 usec
SI                 65536
SF           300.1299931 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```





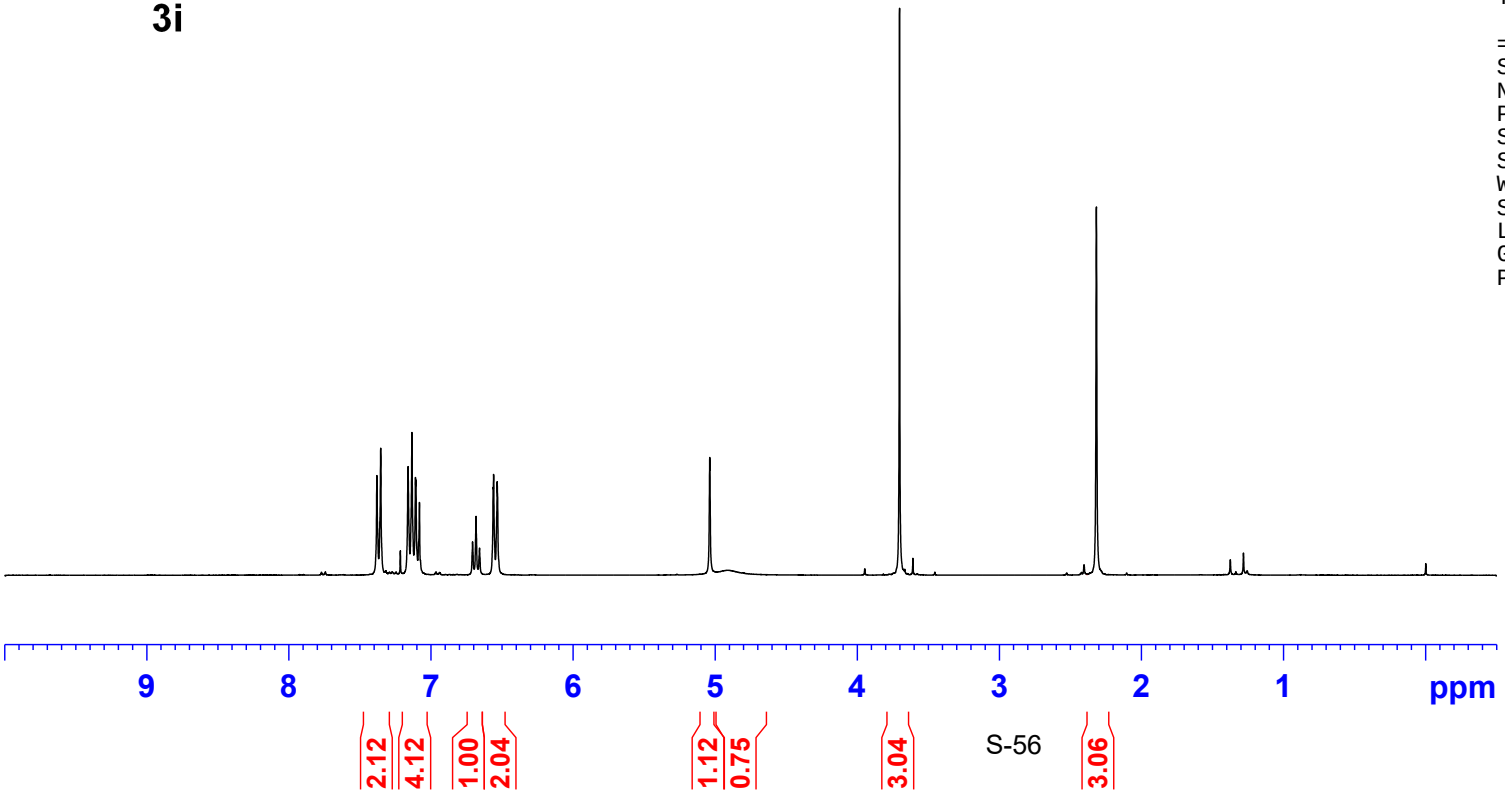

```
NAME        CNMR-gwg-4-1
EXPNO               5606
PROCNO                 1
Date_           20211113
Time               11.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT          Acetone
NS                   300
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ             1.8175818 sec
RG                   203
DW                27.733 usec
DE                 6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

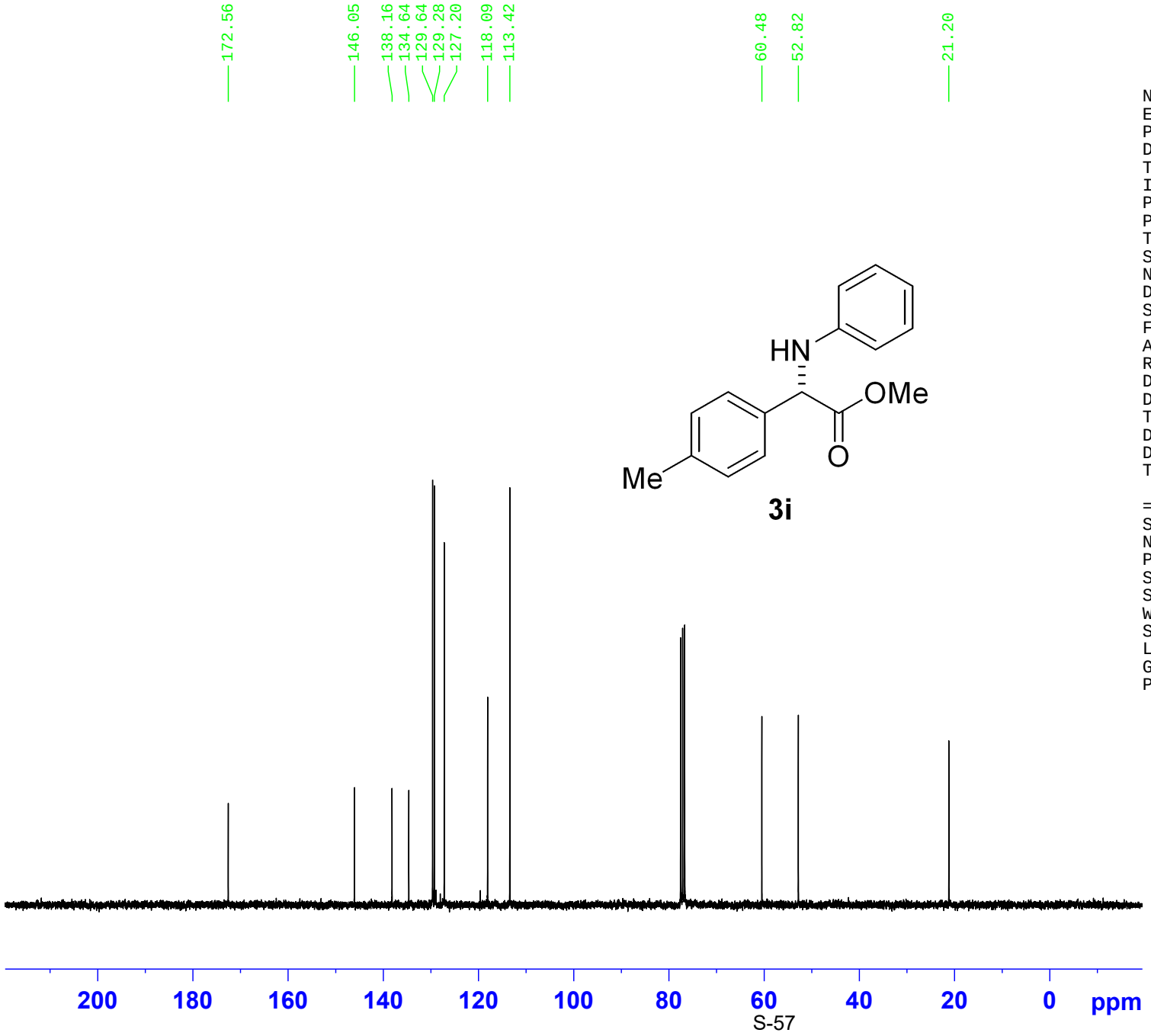




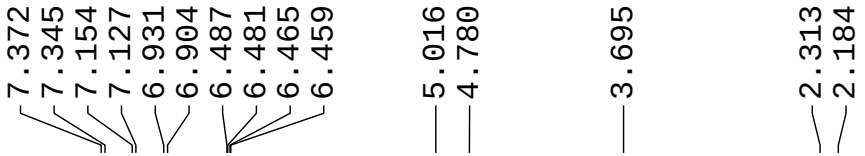
3i

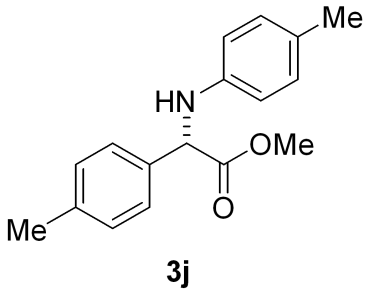
```
NAME        HNMR-gwg-4-15
EXPNO                5621
PROCNO                  1
Date_            20211120
Time                 9.26
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4526453 sec
RG                    80.6
DW                  83.200 usec
DE                    6.50 usec
TE                   -59.1 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                    1H
P1                  10.00 usec
SI                  65536
SF            300.1300206 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```





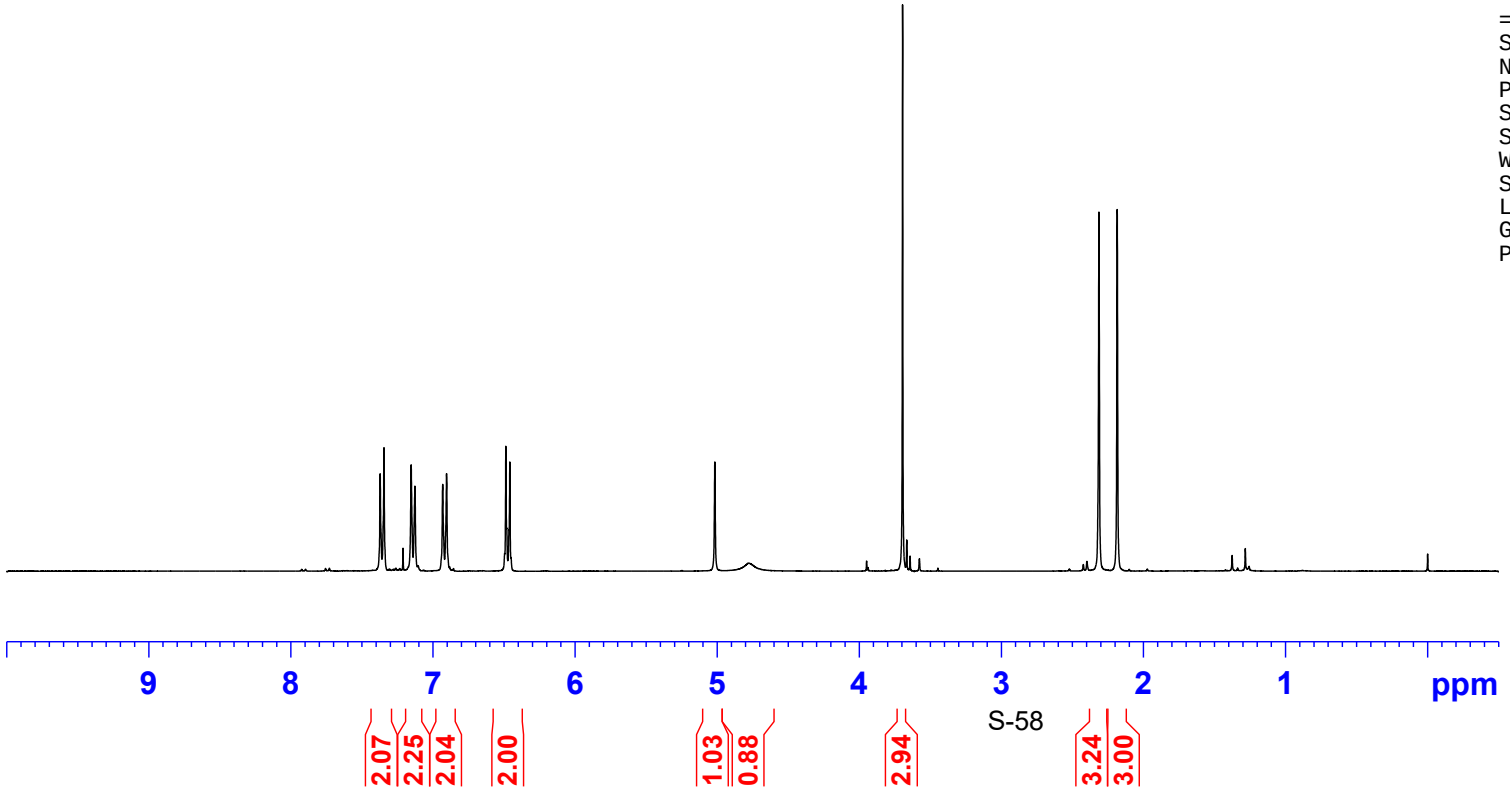

```
NAME       CNMR-gwg-4-15
EXPNO               5622
PROCNO                 1
Date_           20211120
Time                9.41
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   200
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ             1.8175818 sec
RG                   203
DW                27.733 usec
DE                 6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

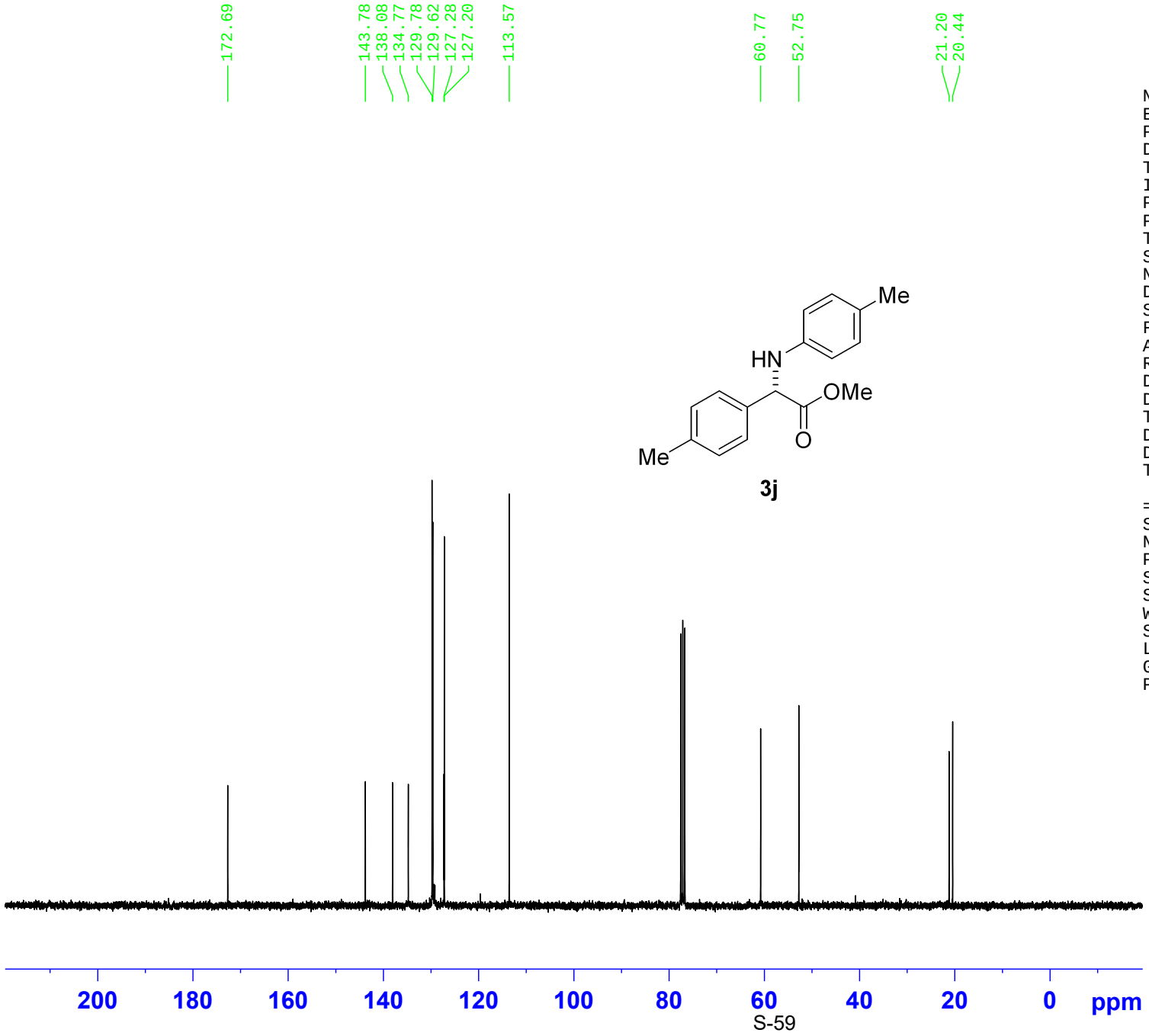




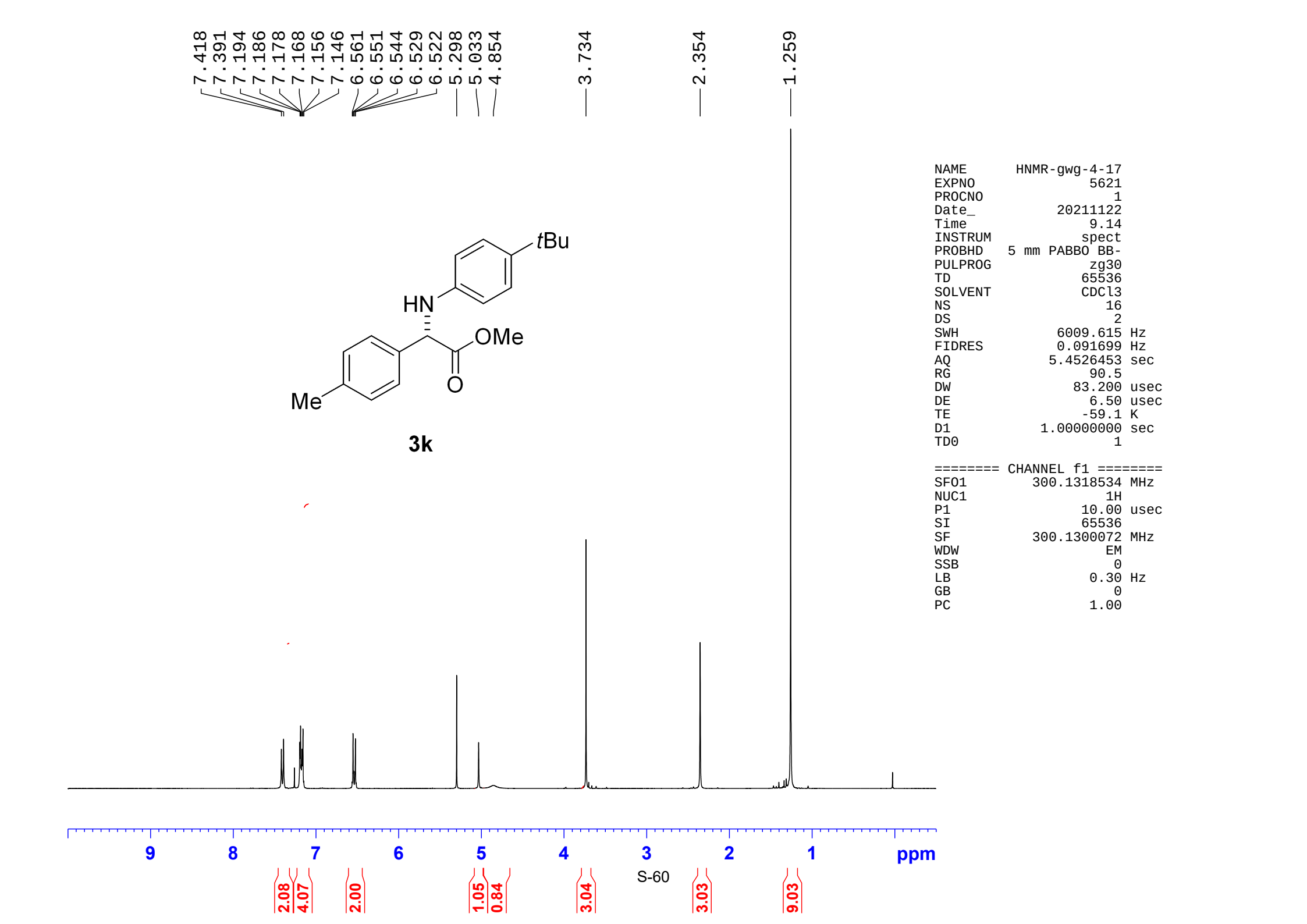
3j

```
NAME        HNMR-gwg-4-13
EXPNO                5612
PROCNO                  1
Date_            20211117
Time                 9.20
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4526453 sec
RG                   71.8
DW                 83.200 usec
DE                  6.50 usec
TE                 -59.1 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                   1H
P1                  10.00 usec
SI                  65536
SF            300.1300221 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```






```
NAME        CNMR-gwg-4-13
EXPNO                5613
PROCNO                  1
Date_            20211117
Time                9.35
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    200
DS                      4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                    203
DW               27.733 usec
DE                6.50 usec
TE                -59.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```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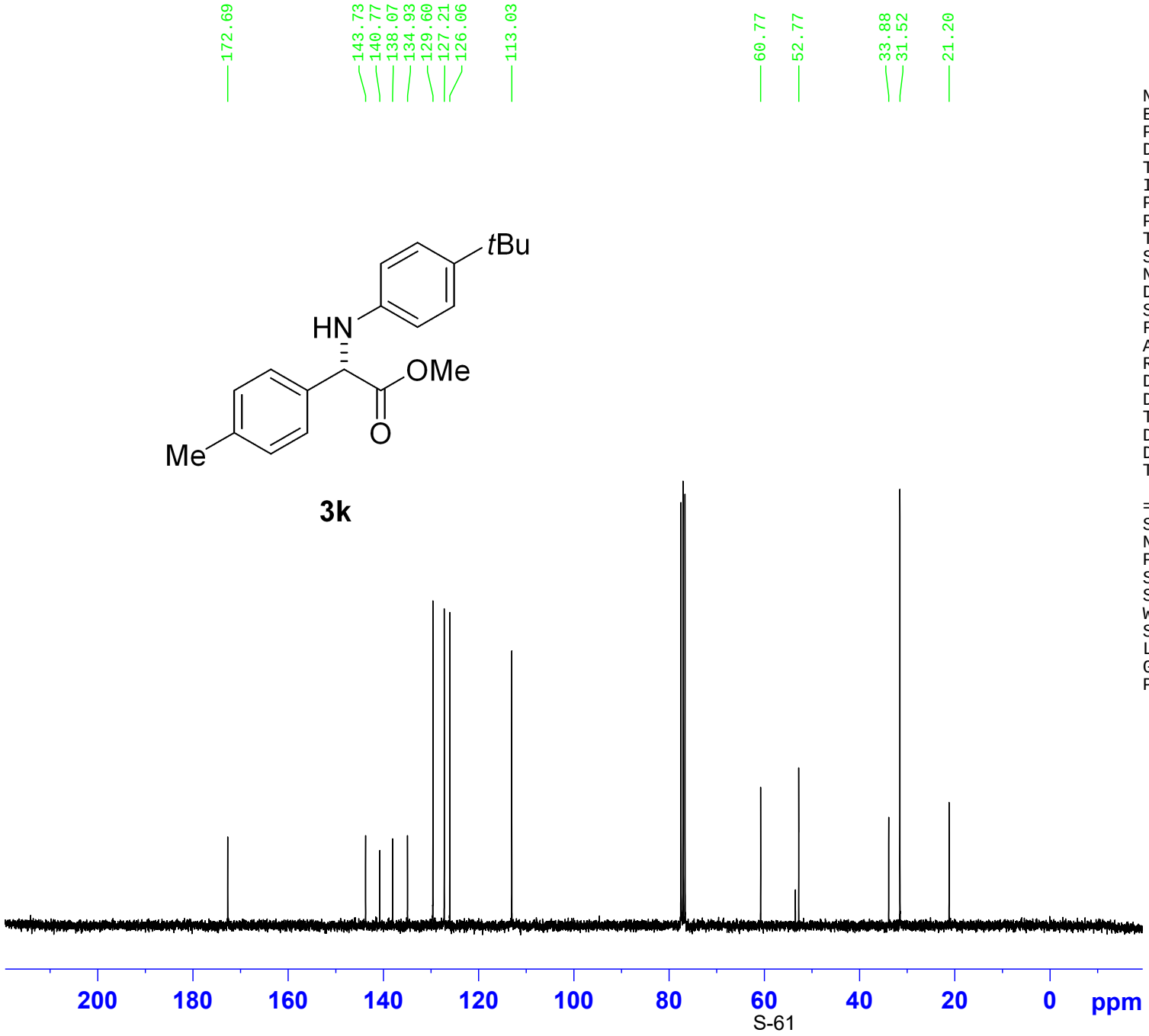
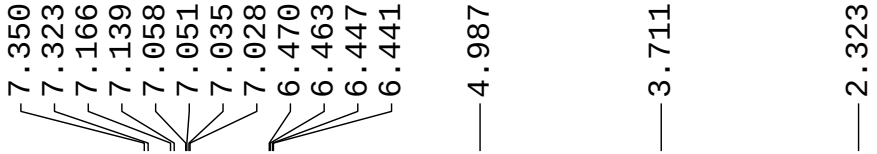
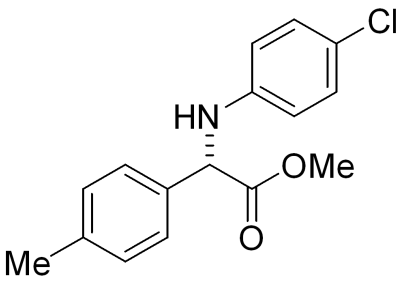
7.391
7.194
7.186
7.178
7.168
7.156
7.146
6.561
6.551
6.544
6.529
6.522
5.298
5.033
4.854
3.734
2.354
1.259
tBu
HN
OMe
O
Me
3k
NAME HNMR-gwg-4-17
EXPNO 5621
PROCNO 1
Date_ 20211122
Time 9.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4526453 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE -59.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 300.1300072 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
2.08
4.07
2.00
1.05
0.84
3.04
3.03
9.03



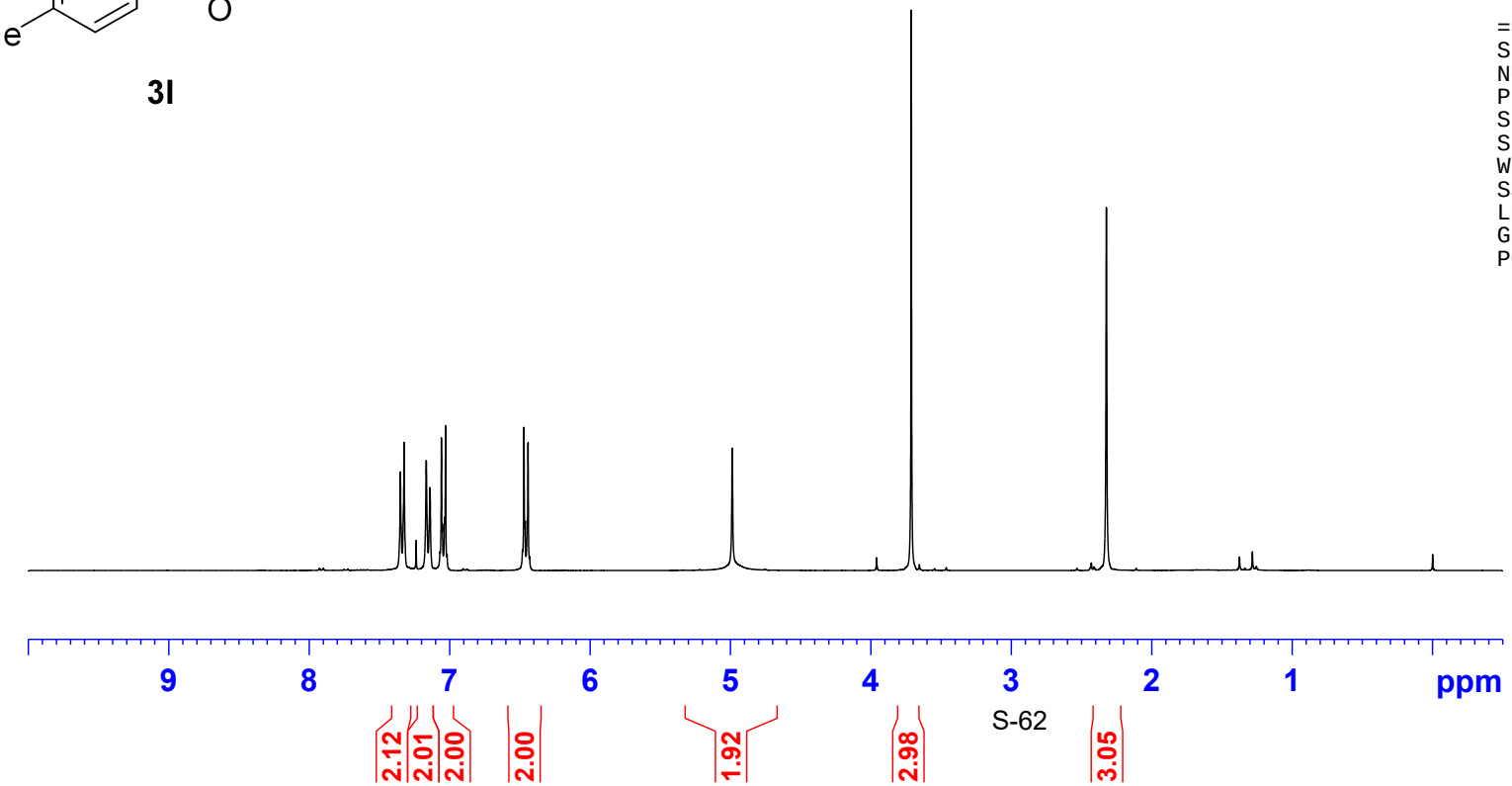

```
NAME        CNMR-gwg-4-17
EXPNO                5622
PROCNO                  1
Date_            20211122
Time                 9.28
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   200
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                   203
DW                27.733 usec
DE                 6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

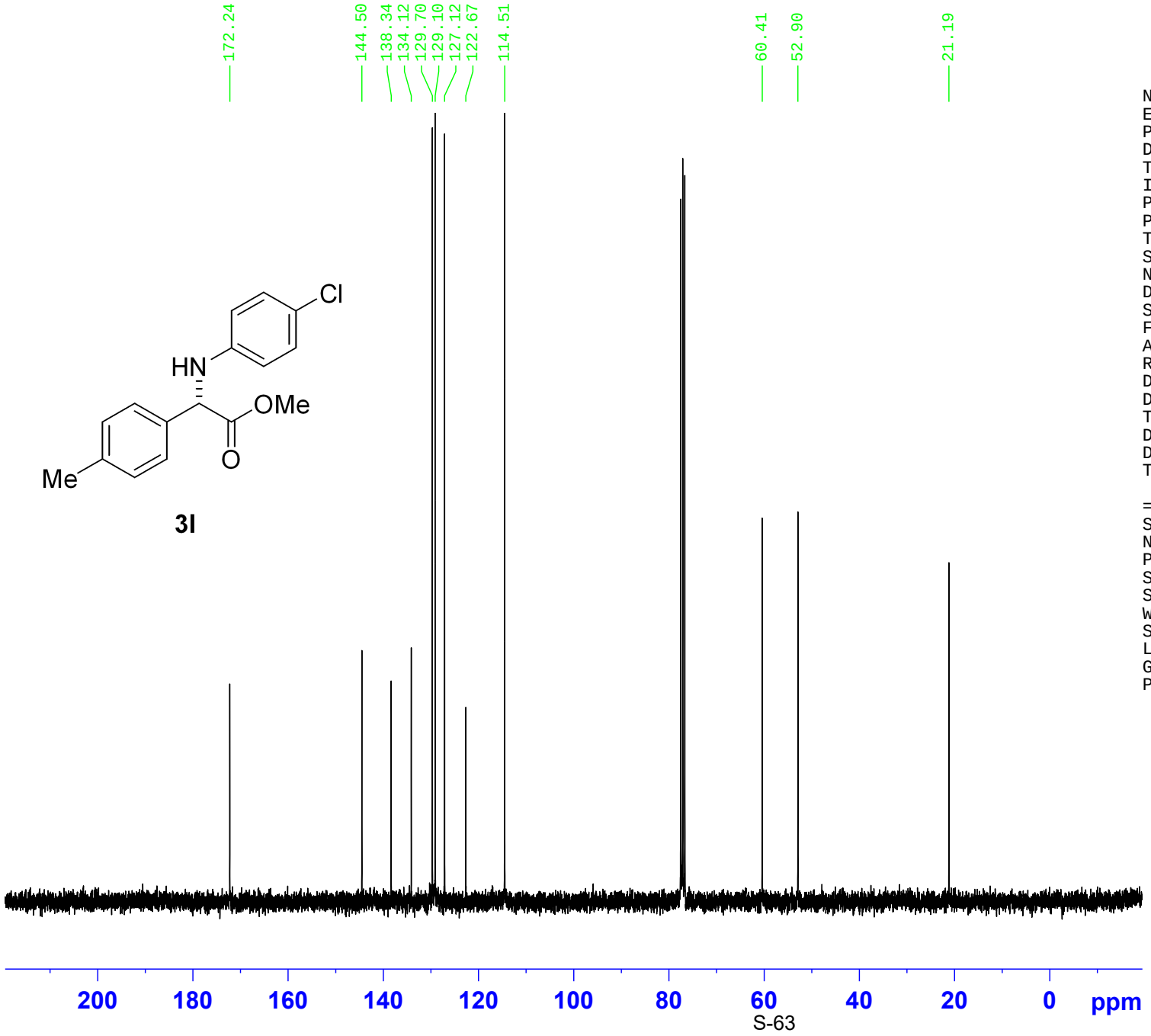




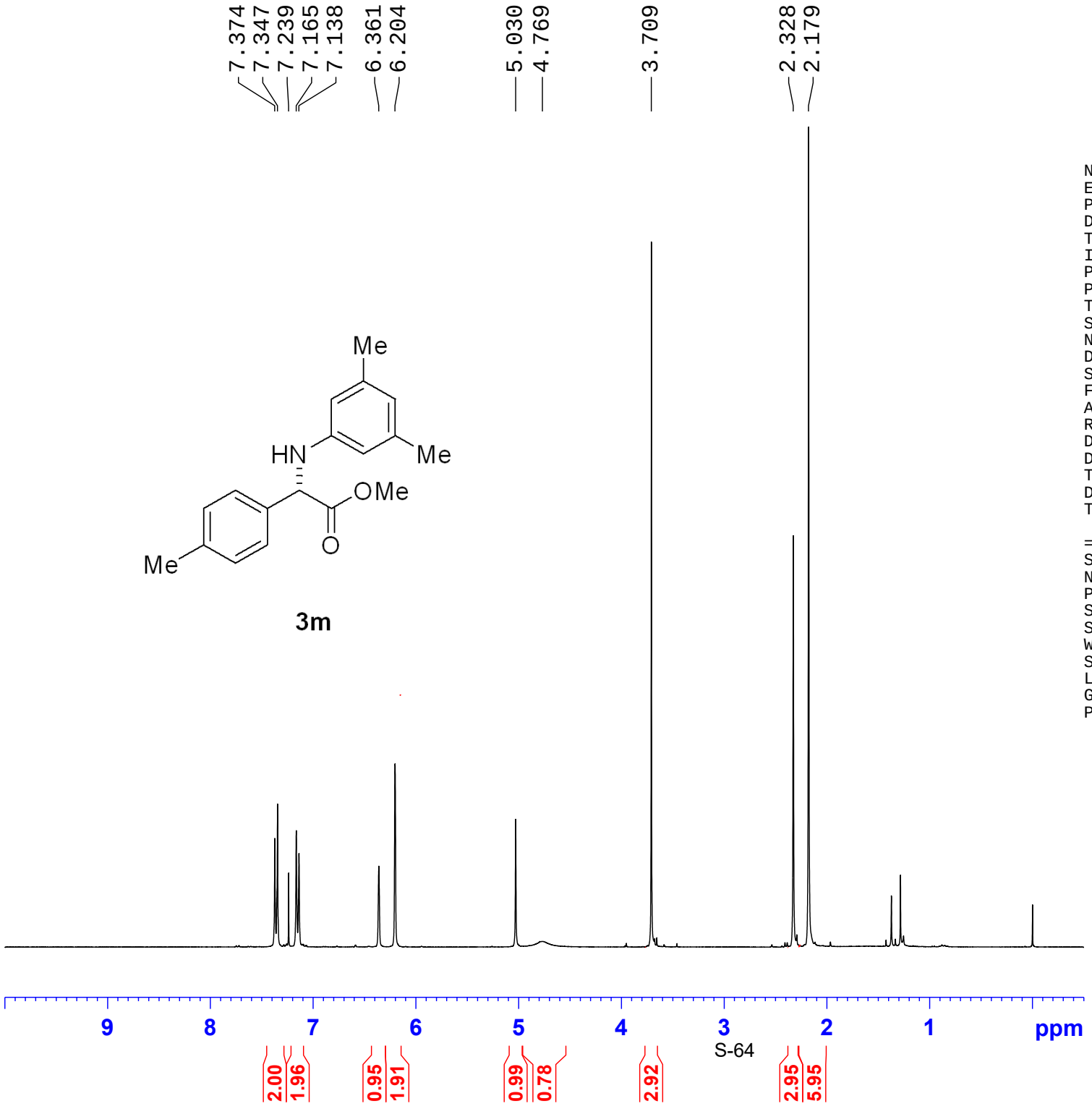
3l

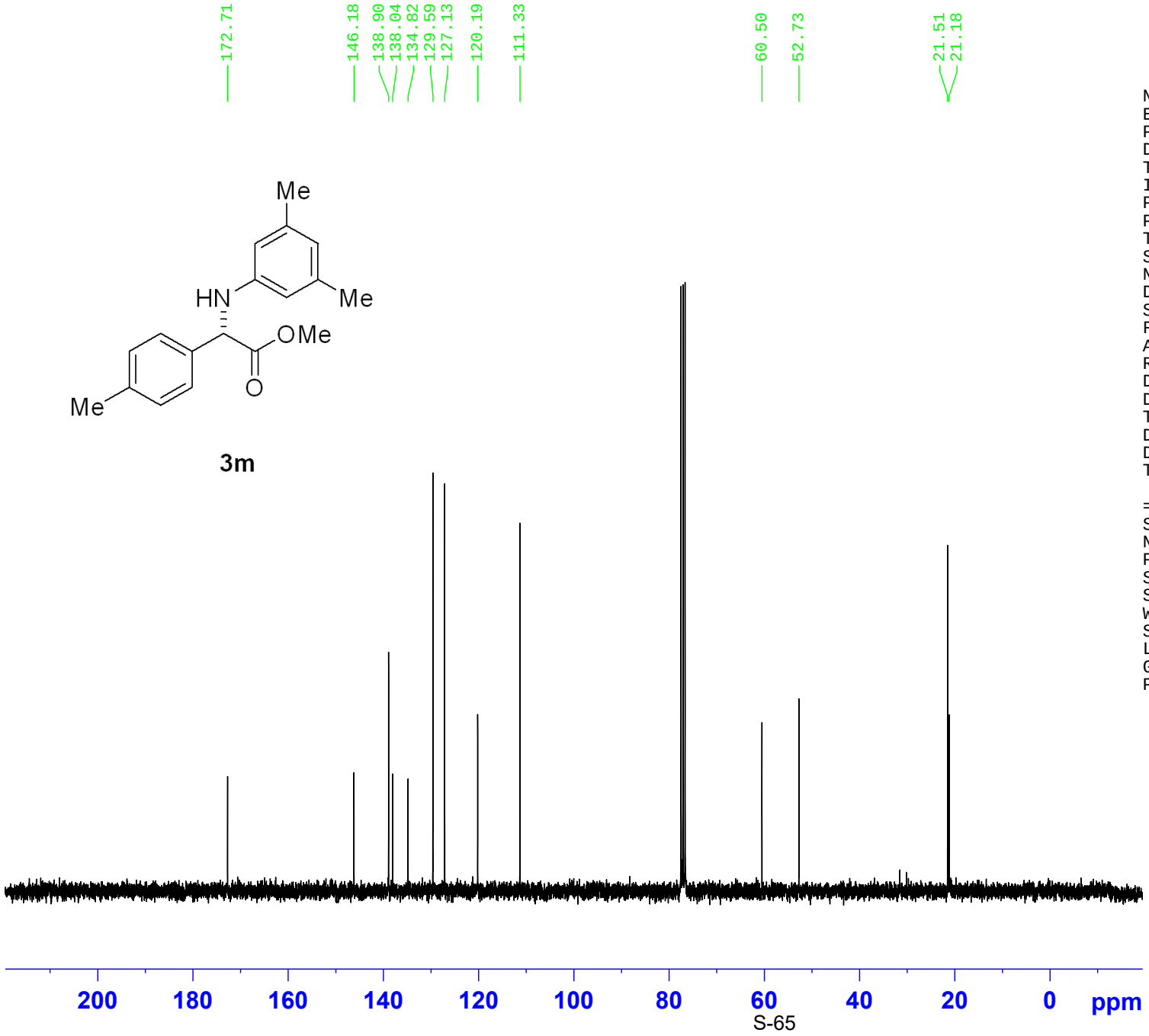
```
NAME        HNMR-gwg-4-14
EXPNO               5614
PROCNO                 1
Date_           20211117
Time                9.39
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4526453 sec
RG                   101
DW                83.200 usec
DE                 6.50 usec
TE                -59.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 10.00 usec
SI                 65536
SF           300.1300139 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



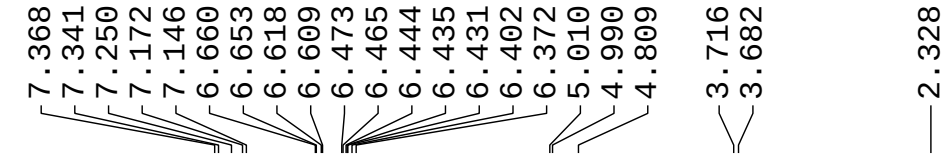


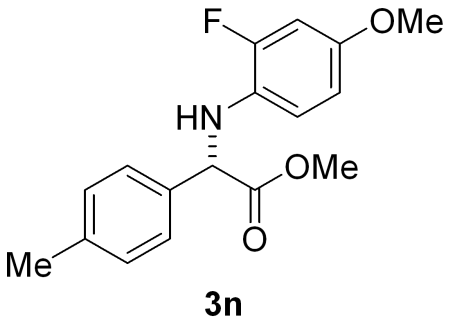

```
NAME        CNMR-gwg-4-14
EXPNO                5615
PROCNO                  1
Date_            20211117
Time                9.54
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    200
DS                      4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                    203
DW               27.733 usec
DE                6.50 usec
TE                -59.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


7.374
7.347
7.239
7.165
7.138
6.361
6.204
5.030
4.769
3.709
2.328
2.179
Me
HN
Me
OMe
O
Me
3m
NAME HNMR-gwg-4-16
EXPNO 5623
PROCNO 1
Date_ 20211120
Time 9.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4526453 sec
RG 128
DW 83.200 usec
DE 6.50 usec
TE -59.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 300.1300137 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
2.00
1.96
0.95
1.91
0.99
0.78
2.92
2.95
5.95



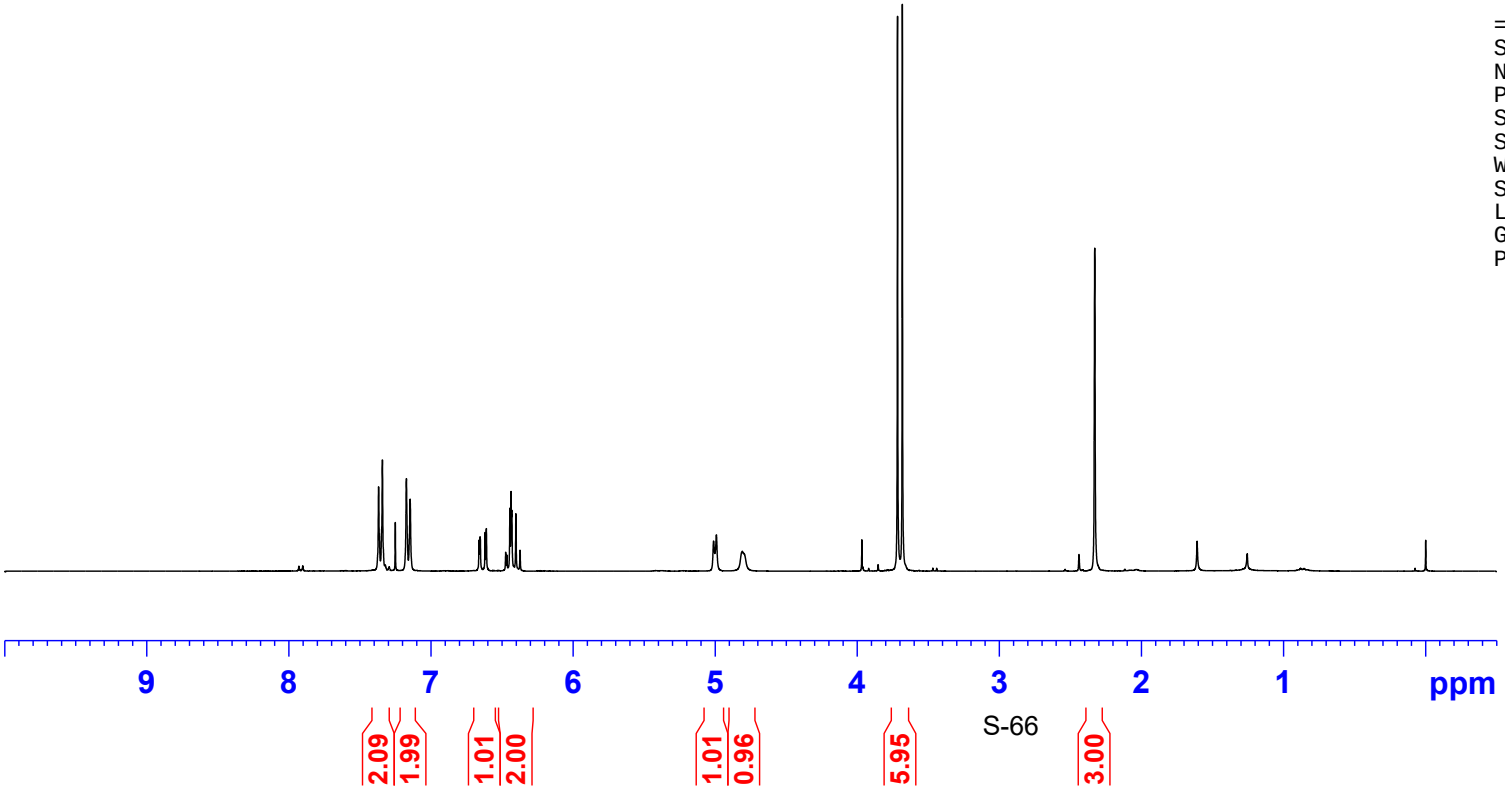

```
NAME        CNMR-gwg-4-16
EXPNO                5624
PROCNO                  1
Date_            20211120
Time                10.00
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    200
DS                      4
SWH             18028.846 Hz
FIDRES           0.275098 Hz
AQ               1.8175818 sec
RG                    203
DW                 27.733 usec
DE                  6.50 usec
TE                 -59.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1            75.4752949 MHz
NUC1                   13C
P1                   9.50 usec
SI                  32768
SF              75.4677485 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

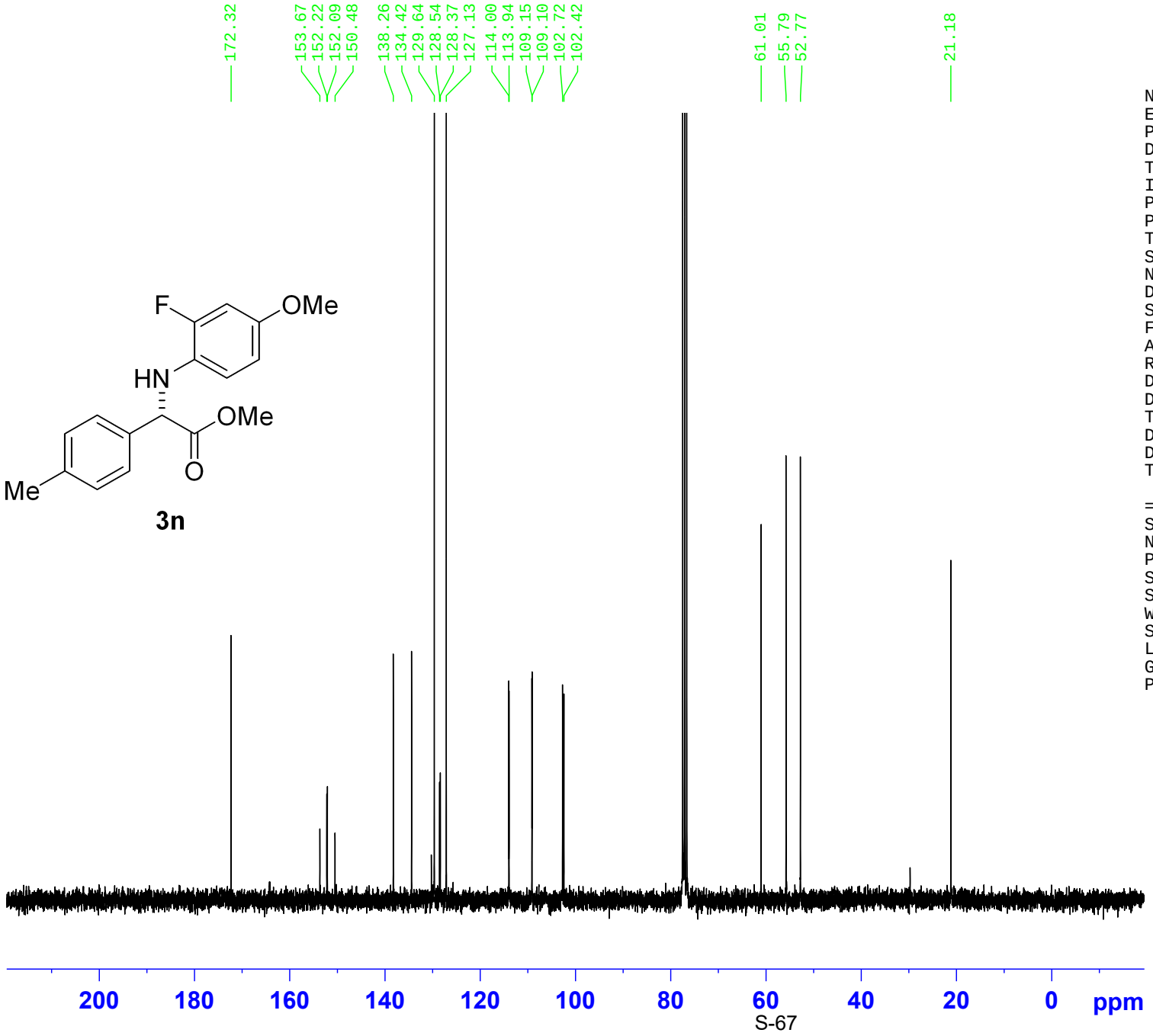




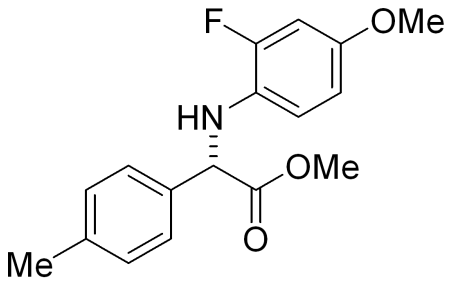
3n

```
NAME        HNMR-gwg-4-38
EXPNO               5623
PROCNO                 1
Date_           20211122
Time                9.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4526453 sec
RG                   128
DW                83.200 usec
DE                 6.50 usec
TE                -59.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 10.00 usec
SI                 65536
SF           300.1300102 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



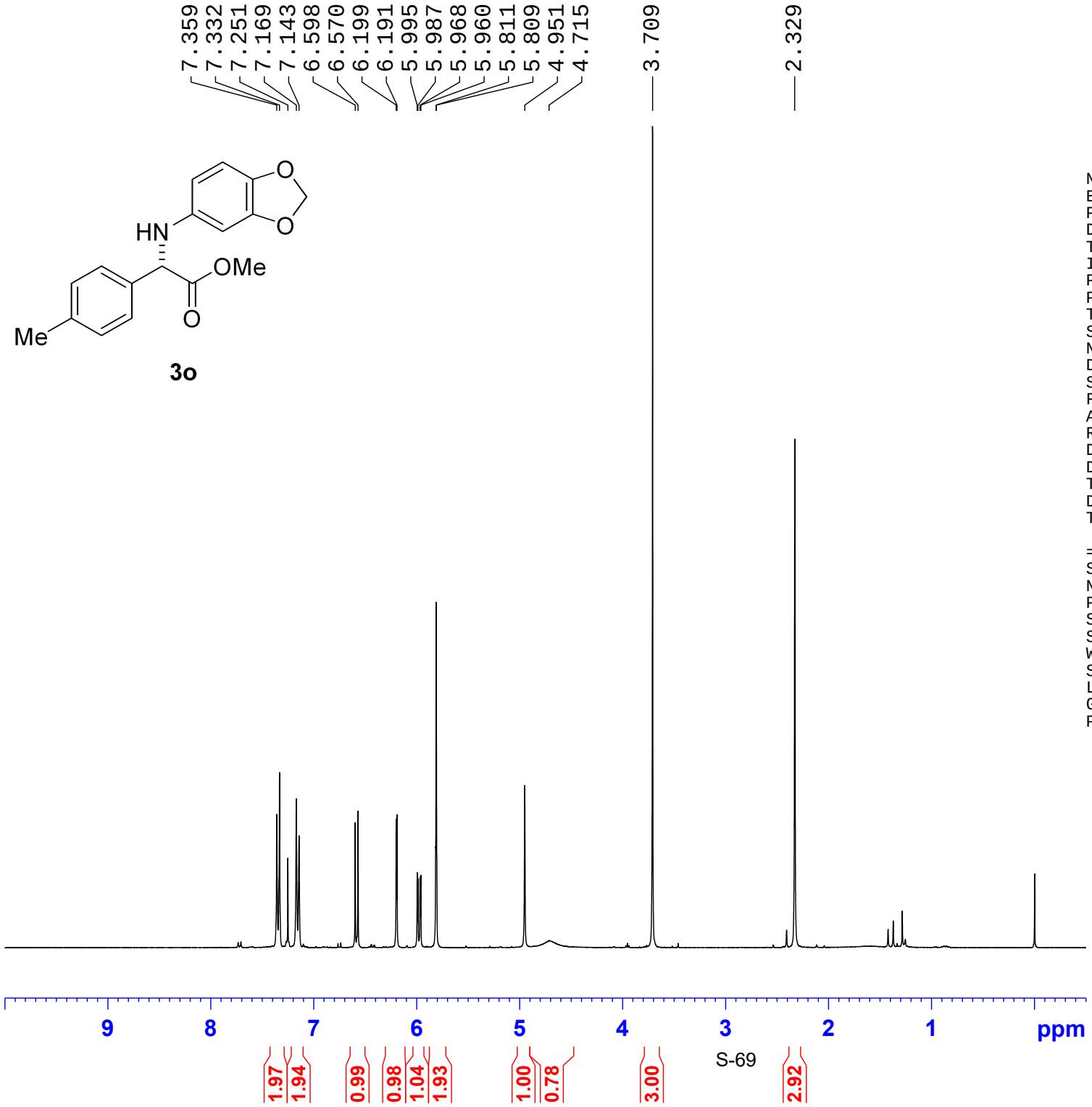


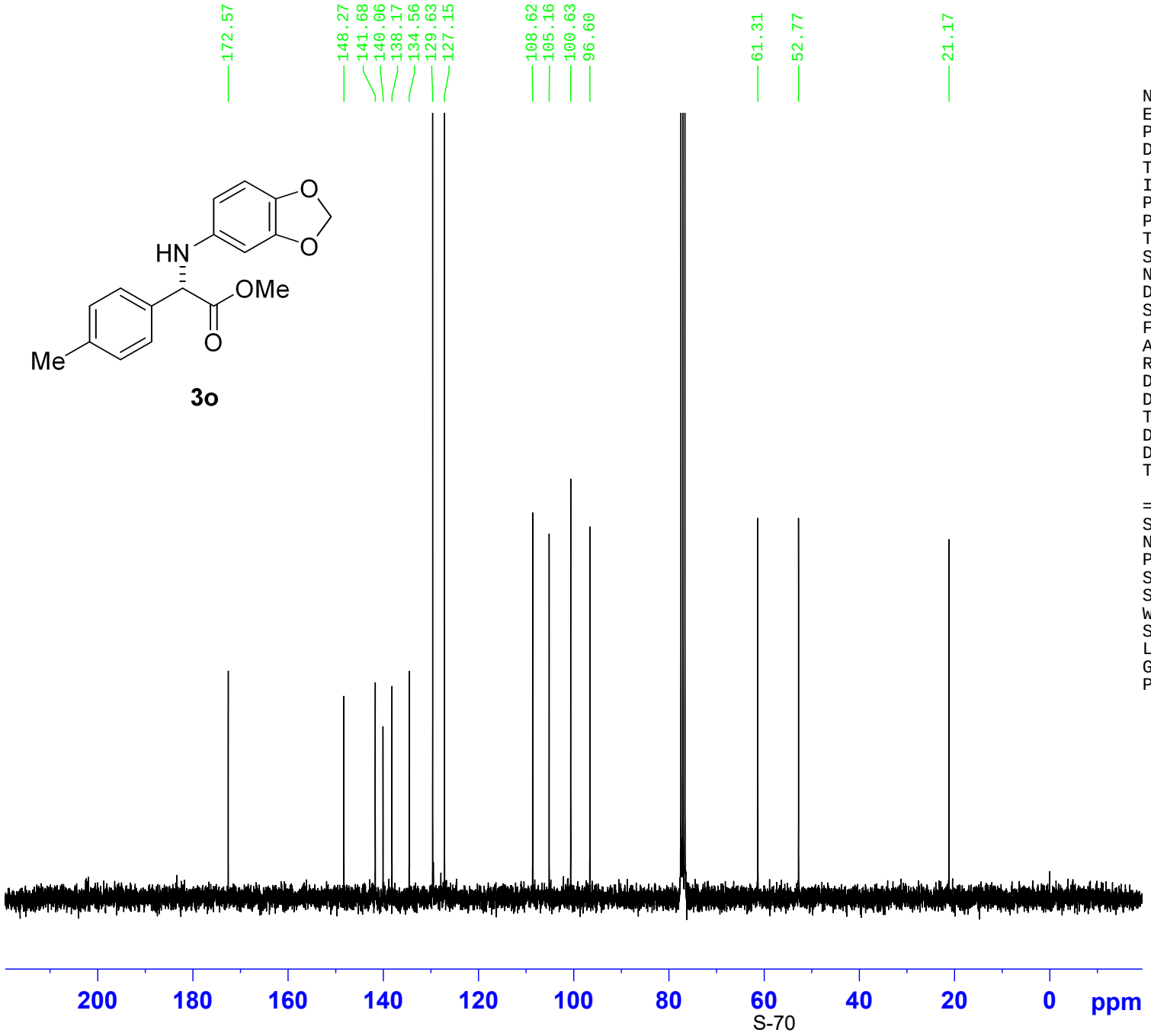

```
NAME       CNMR-gwg-4-38
EXPNO               5625
PROCNO                 1
Date_           20211122
Time               10.09
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW               27.733 usec
DE                6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



3n

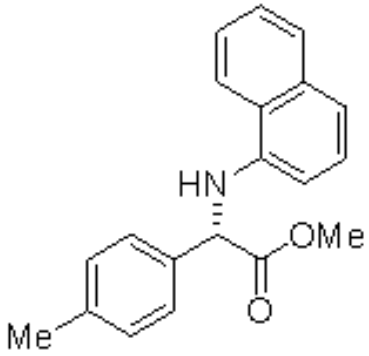

```
NAME          FNMR-gwg-4-38
EXPNO                  5624
PROCNO                    1
Date_              20211122
Time                   9.35
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG         zgfhigqn.2
TD                    131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH               66964.289 Hz
FIDRES             0.510897 Hz
AQ                 0.9787210 sec
RG                      203
DW                    7.467 usec
DE                    6.50 usec
TE                   -59.1 K
D1                1.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            282.3761148 MHz
NUC1                    19F
P1                    14.50 usec
SI                    65536
SF              282.4043552 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```


7.359
7.332
7.251
7.169
7.143
6.598
6.570
6.199
6.191
5.995
5.987
5.968
5.960
5.811
5.809
4.951
4.715
3.709
2.329
HN
O
O
OMe
O
Me
3o
NAME HNMR-gwg-4-55
EXPNO 5674
PROCNO 1
Date_ 20211129
Time 9.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4526453 sec
RG 161
DW 83.200 usec
DE 6.50 usec
TE -59.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 300.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.97
1.94
0.99
0.98
1.04
1.93
1.00
0.78
3.00
2.92



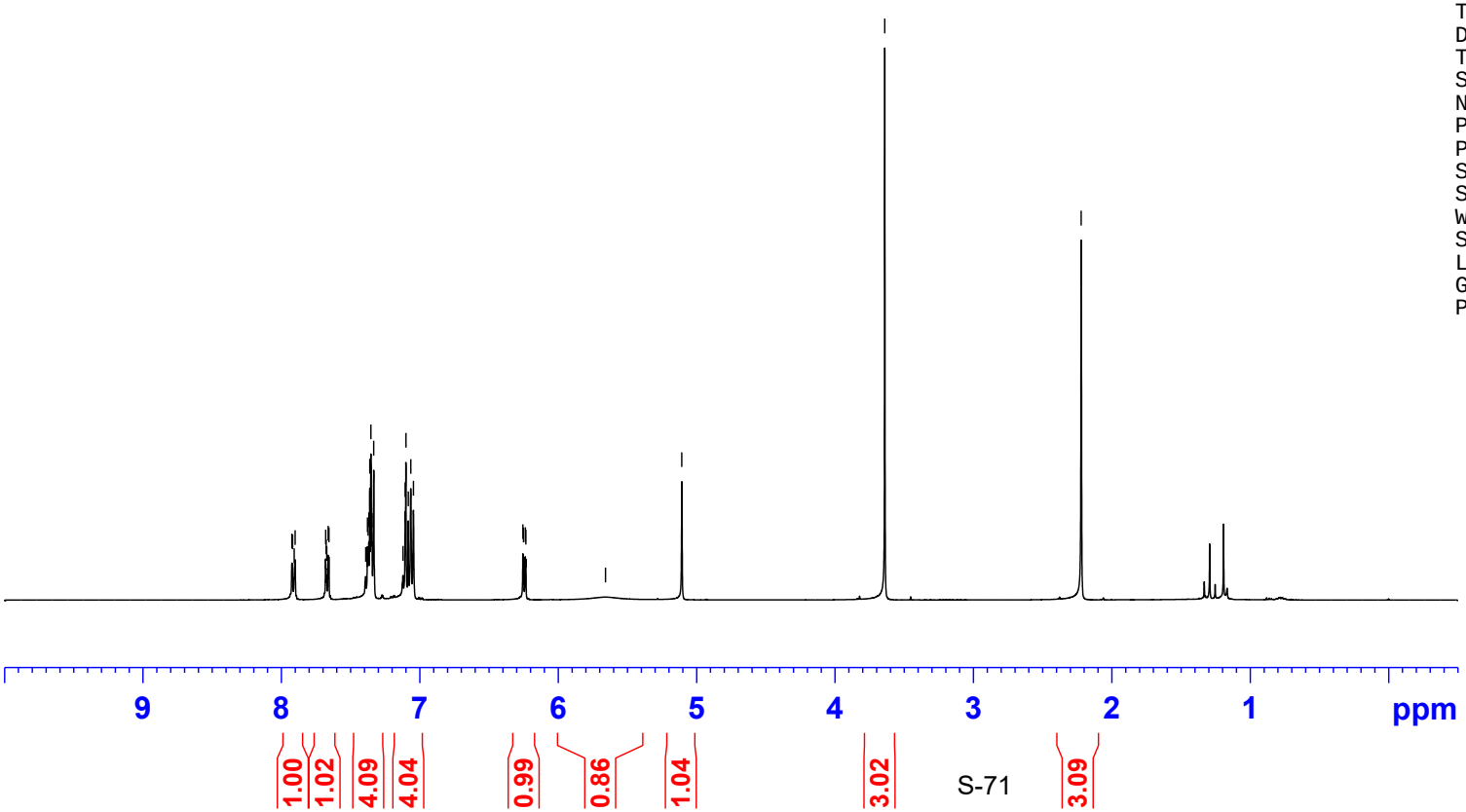

```
NAME       CNMR-gwg-4-55
EXPNO              5675
PROCNO                1
Date_          20211129
Time              10.32
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW               27.733 usec
DE                6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.50 usec
SI                32768
SF            75.4677485 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

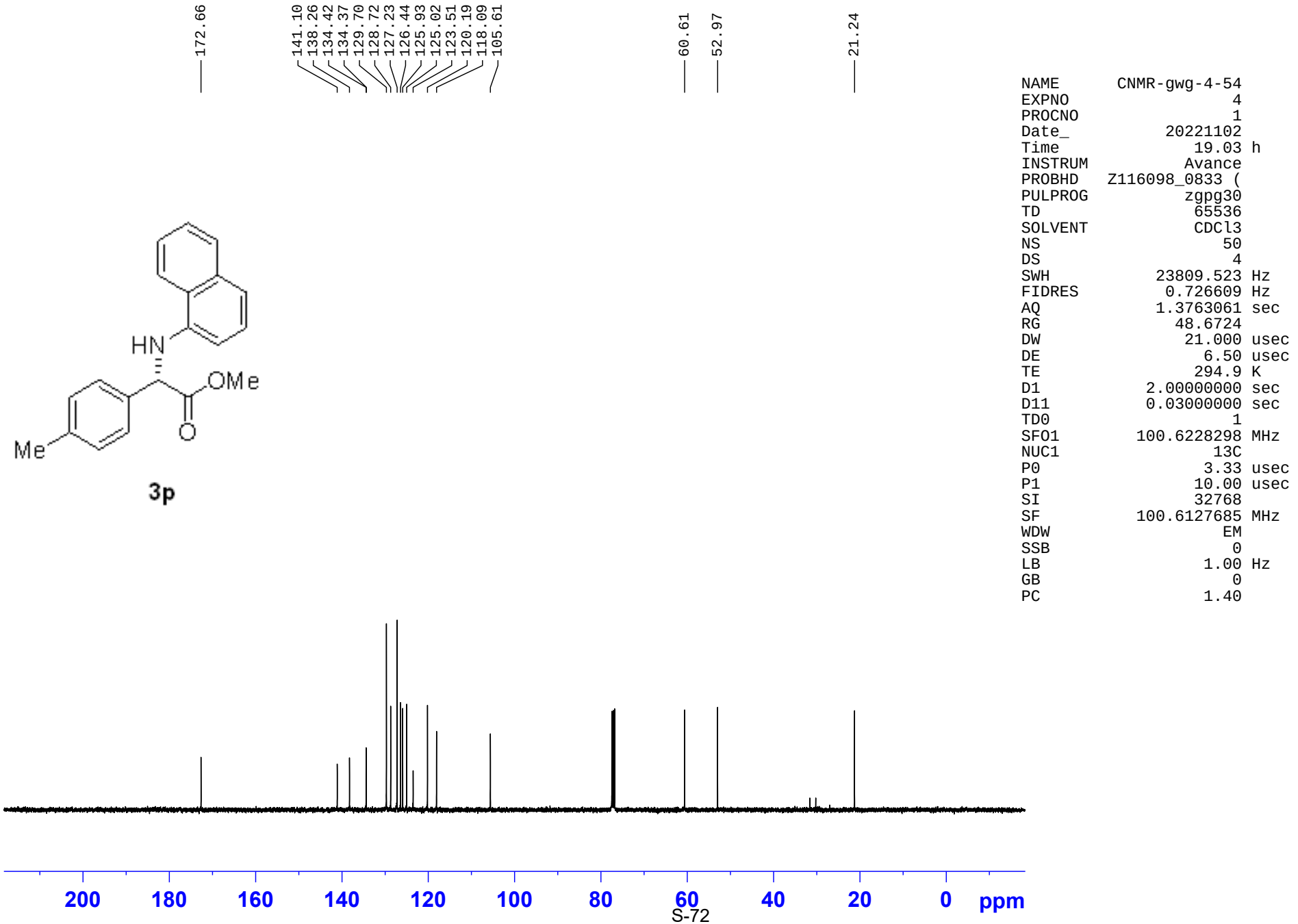
7.900
7.680
7.676
7.672
7.662
7.656
7.391
7.378
7.373
7.367
7.361
7.353
7.345
7.332
7.121
7.105
7.100
7.083
7.065
7.045
6.254
6.250
6.237
6.233
5.658
5.107
3.641
2.221



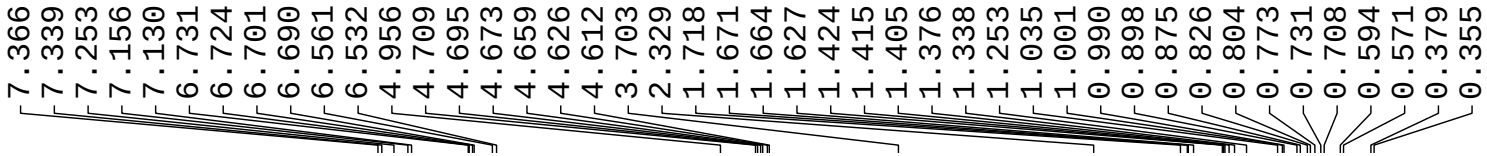
3p

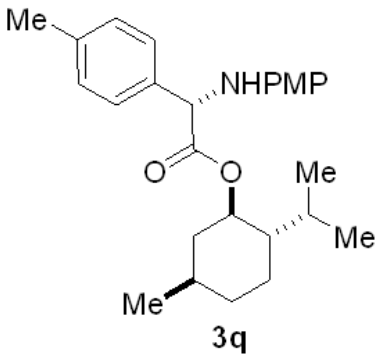
```
NAME          HNMR-gwg-4-54
EXPNO                    3
PROCNO                   1
Date_             20221102
Time                 18.59 h
INSTRUM             Avance
PROBHD    Z116098_0833 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               8196.722 Hz
FIDRES            0.250144 Hz
AQ                3.9977460 sec
RG                  45.4545
DW                  61.000 usec
DE                  13.54 usec
TE                  295.0 K
D1              1.00000000 sec
TD0                      1
SFO1           400.1324708 MHz
NUC1                    1H
P0                   3.33 usec
P1                  10.00 usec
SI                    65536
SF             400.1300752 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```





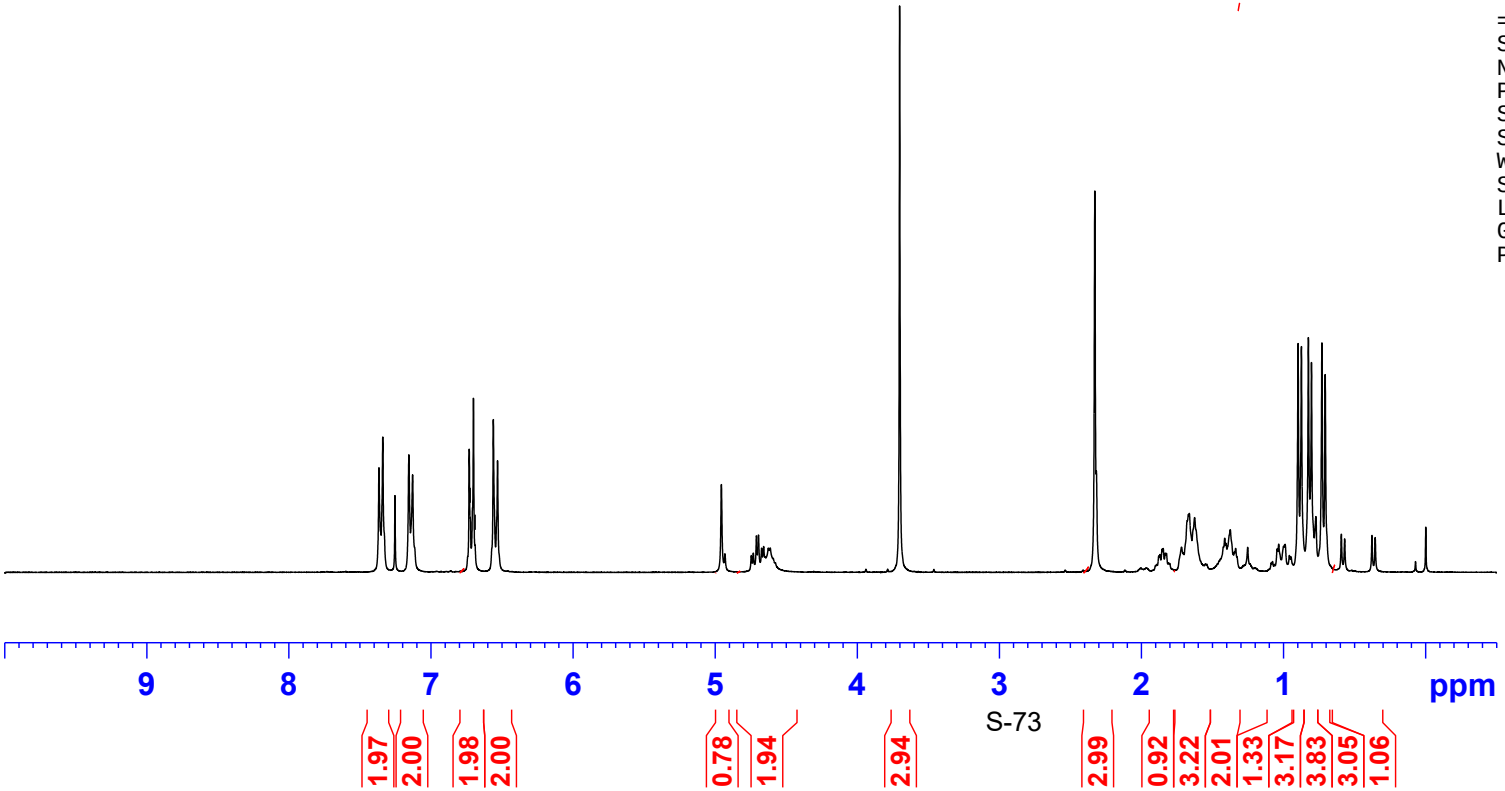
NAME	CNMR-gwg-4-54
EXPNO	4
PROCNO	1
Date_	20221102
Time	19.03 h
INSTRUM	Avance
PROBHD	Z116098_0833 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	50
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	48.6724
DW	21.000 usec
DE	6.50 usec
TE	294.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P0	3.33 usec
P1	10.00 usec
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

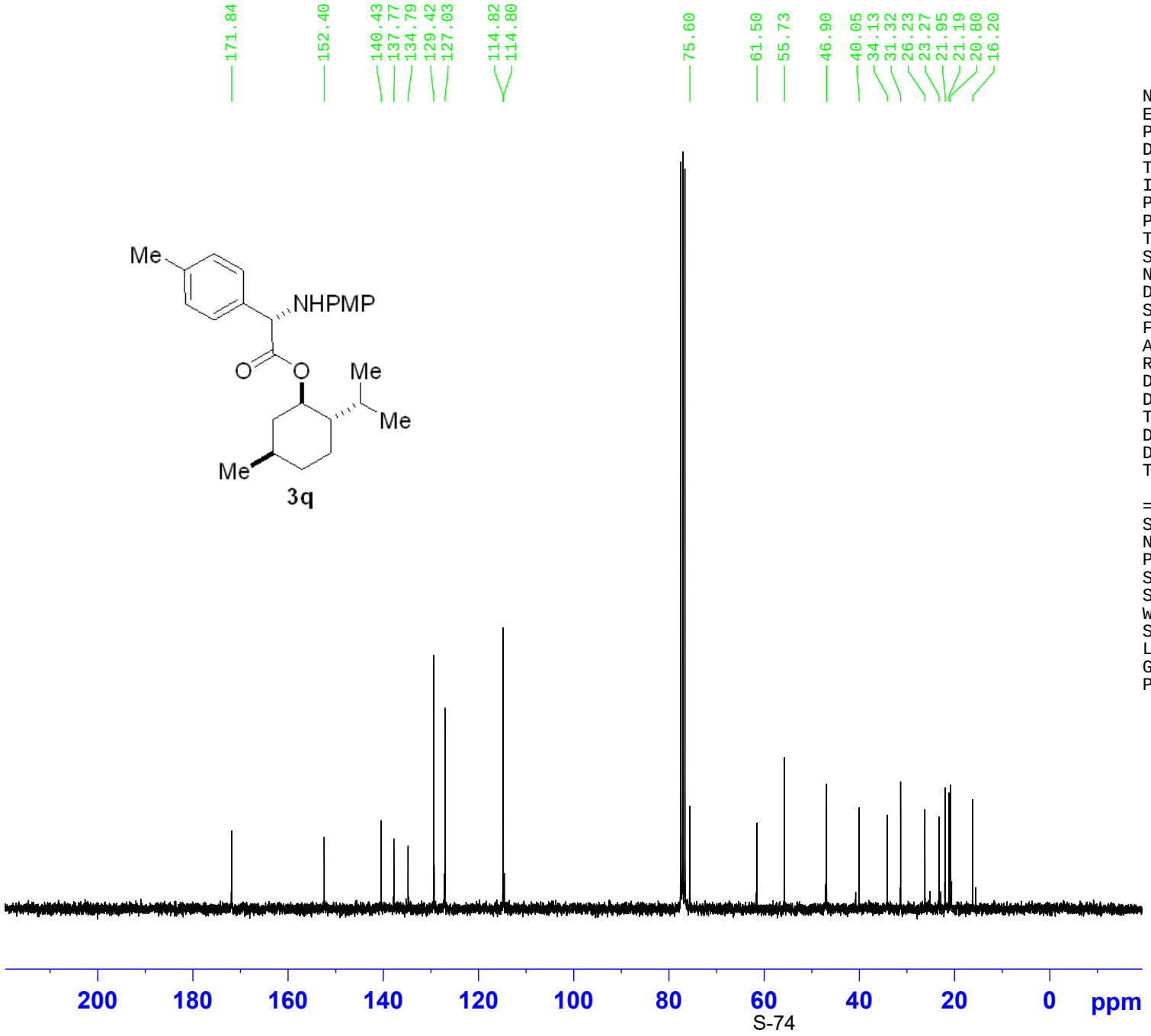




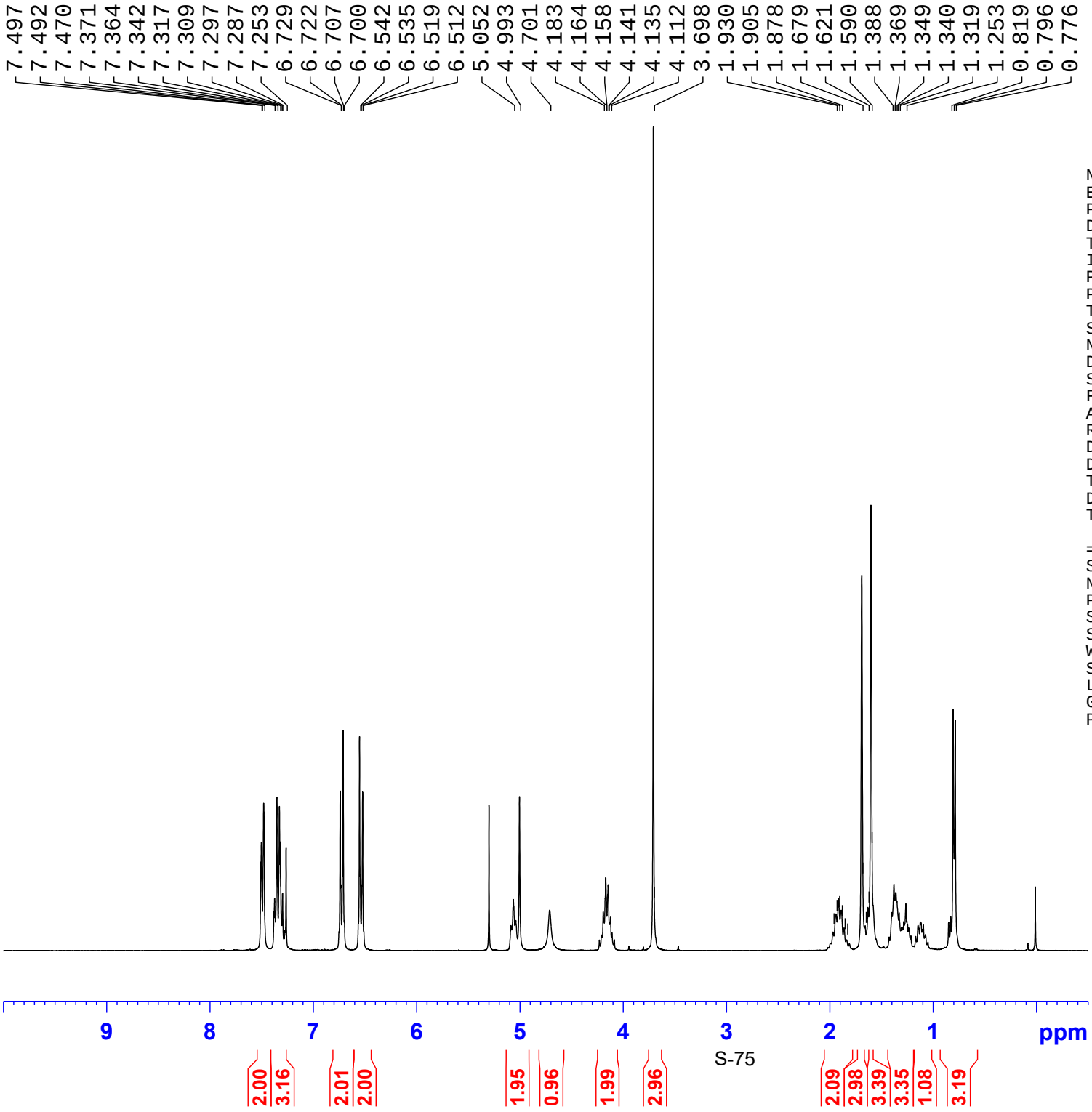
3q

```
NAME        HNMR-gwg-4-45
EXPNO               5629
PROCNO                 1
Date_           20211124
Time                9.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4526453 sec
RG                   114
DW                83.200 usec
DE                 6.50 usec
TE                -59.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 10.00 usec
SI                 65536
SF           300.1300090 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

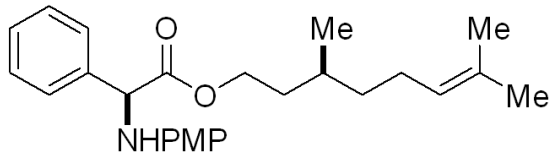




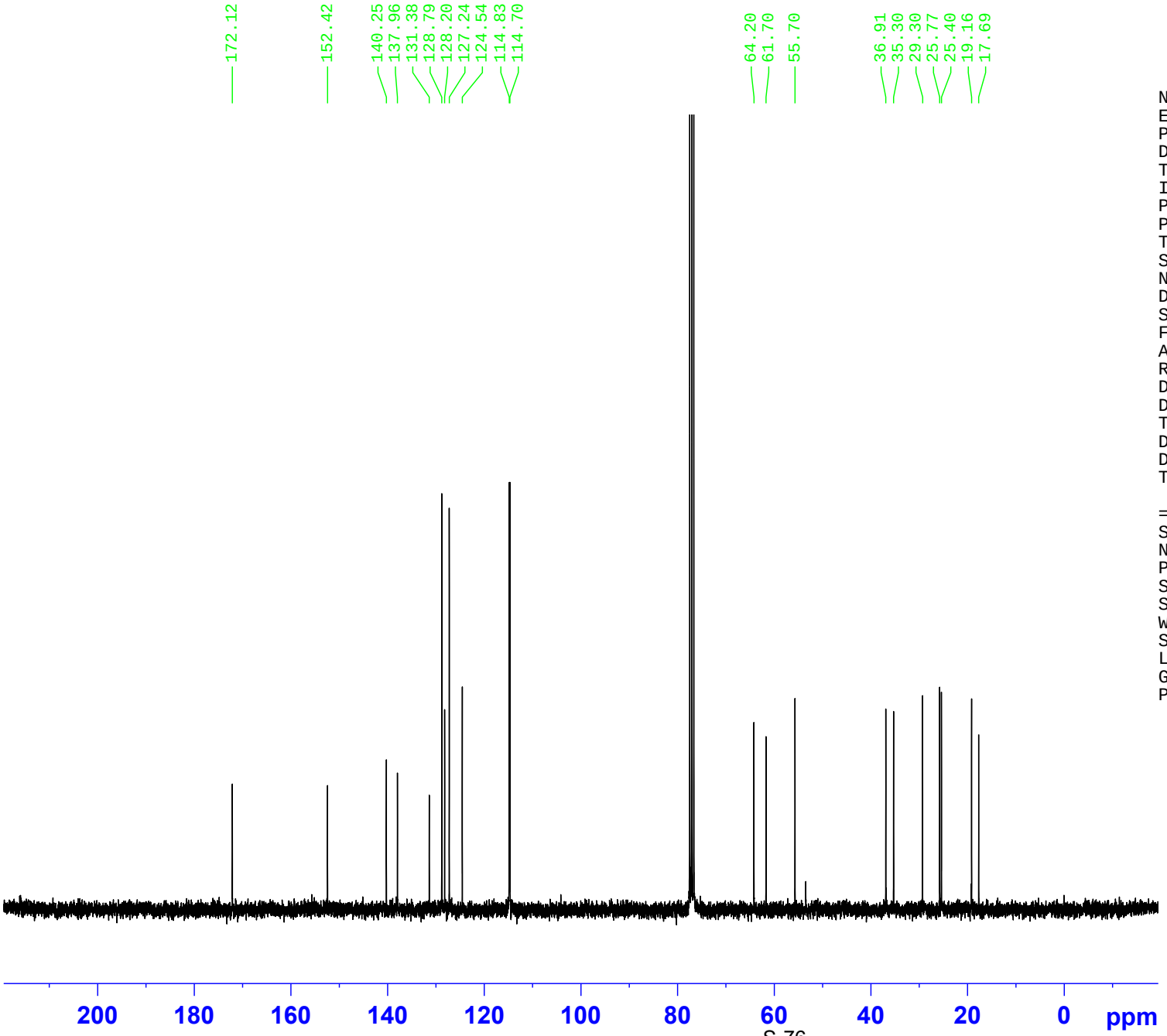

```
NAME        CNMR-gwg-4-45
EXPNO                5630
PROCNO                  1
Date_            20211124
Time                9.48
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    500
DS                      4
SWH             18028.846 Hz
FIDRES           0.275098 Hz
AQ               1.8175818 sec
RG                    203
DW                 27.733 usec
DE                  6.50 usec
TE                 -59.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1            75.4752949 MHz
NUC1                   13C
P1                   9.50 usec
SI                  32768
SF              75.4677485 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



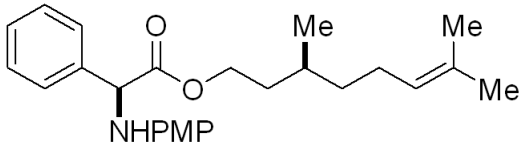

```
NAME        HNMR-gwg-4-46
EXPNO                5631
PROCNO                  1
Date_            20211124
Time                9.53
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4526453 sec
RG                    114
DW                 83.200 usec
DE                  6.50 usec
TE                 -59.1 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                   1H
P1                 10.00 usec
SI                  65536
SF            300.1300062 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```



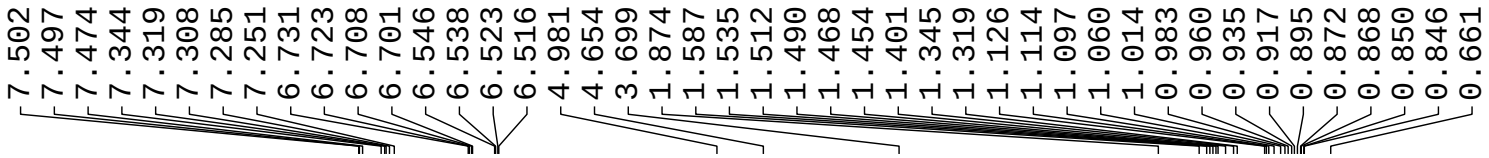
3r

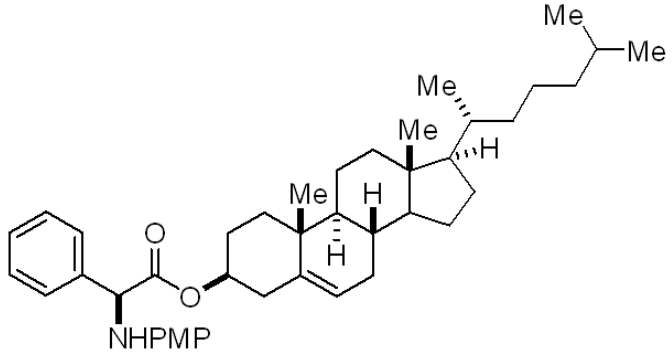



```
NAME        CNMR-gwg-4-46
EXPNO                5632
PROCNO                  1
Date_            20211124
Time                10.26
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    500
DS                      4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                -59.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                 9.50 usec
SI                 32768
SF            75.4677485 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



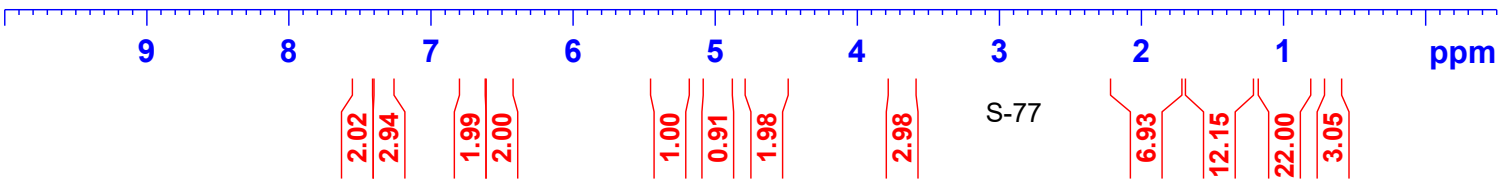
3r

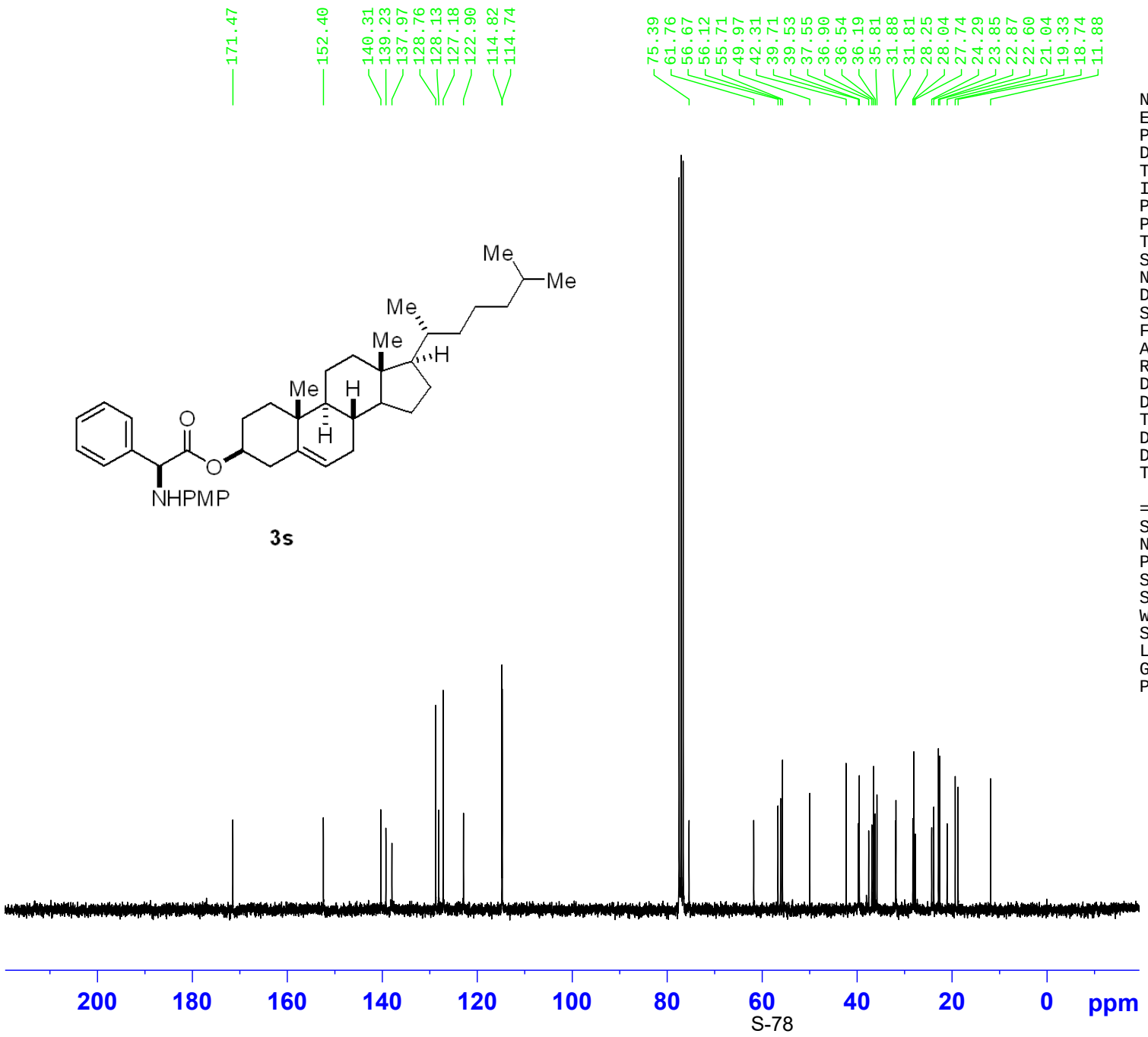




3s

```
NAME      HNMR-gwg-4-51-2
EXPNO               5644
PROCNO                 1
Date_           20211125
Time               11.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ             5.4526453 sec
RG                  80.6
DW                83.200 usec
DE                 6.50 usec
TE                -59.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 10.00 usec
SI                 65536
SF           300.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```






```
NAME        CNMR-gwg-4-51-2
EXPNO                  5645
PROCNO                    1
Date_              20211125
Time                  11.59
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      500
DS                        4
SWH               18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175818 sec
RG                      203
DW                   27.733 usec
DE                    6.50 usec
TE                   -59.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1              75.4752949 MHz
NUC1                    13C
P1                    9.50 usec
SI                    32768
SF                75.4677485 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```