Electronic Supplementary Information

Enantioselective Bromolactonization of *cis*-1,2-disubstituted olefinic acids using amino-thiocarbamate catalyst

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A. General Information

All reactions that required anhydrous conditions were carried out under nitrogen atmosphere. Commercially available reagents were used as received from Alfa Aesar, Sigma-Aldrich and TCI-Tokyo Kasei Kogyo Co. Ltd. Dichromethane and acetonitrile were dried by distillation over the appropriate drying reagents. Anhydrous THF was purchased from EM Science and used without further purification. HPLC grade chloroform was purchased from Fischer Chemicals while HPLC grade hexane and isopropanol were purchased from Tedia and used as received. Analytical thin layer chromatography (TLC) was performed with Merck pre-coated TLC plates, silica gel 60 F-254, layer thickness 0.25 mm. The plate were visualized under 254 nm UV light and stained with potassium permanganate. Flash chromatography separations were performed on Merck 60 (0.040-0.0063 mm) mesh silica gel. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker ACF 300 (300MHz) and AMX500 (500 MHz) spectrometer. Chemical shift (δ) are reported in parts per million (ppm) relative to tetramethylsilane (TMS, δ=0.00) for ^{1}H NMR and are referenced to the residual solvent peak chloroform (δ=7.26). Chemical shift for ^{13}C NMR are reported in ppm downfield from TMS and are referenced to the carbon resonance of the solvent chloroform (δ=77.0). Data are represented as follow: chemical shift, multiplicity (br. s = broad singlet, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz). Low resolution mass spectra (ESI) were obtained on a Finnnigan/MAT LCQ spectrometer. High resolution mass spectra (ESI) were obtained on Finnnigan/MAT 95XL-T spectrometer. Enantiomeric excesses were determined by HPLC analysis on Shimadzu HPLC units, including the following instruments: LC-20AD pump, SPD-20A detector and Dacicel Chiralpak IA, IB, IC or ODH columns. Optical rotations were recorded on a Jasco DIP-1000 polarimeter. Infrared spectra were recorded on a Varian 3100 FTIR spectrophotometer and reported in wave numbers (cm^{-1}). Melting points were determined on a BÜCHI B-540b melting point apparatus.

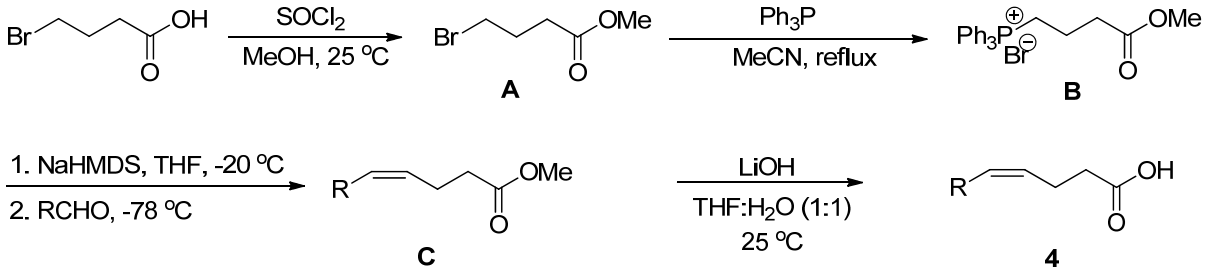
B. Experimental Procedures and Physical Data

All the amino-thiocarbamate catalysts were prepared according to our previously reported procedure. Spectral data of other catalysts were reported in previous reports.[1-4]

Amino-thiocarbamate 6a

White solid, mp 68.5-69.6 °C; $[\alpha]_D^{25}$ +140.8 (*c* 1.0, $CHCl_3$); IR (KBr): 2952, 2868, 1619, 1510, 1464, 1308, 1284, 1208, 1159, 1033 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.75 (br, 1H), 8.34 (s, 1H), 8.02 (d, *J* = 9.5 Hz, 1H), 7.66-7.44 (m, 1H), 7.38-7.34 (m, 3H), 6.50 (d, *J* = 7.6 Hz, 2H), 5.82 (br, 1H), 5.03-5.00 (m, 2H), 4.15 (br, 1H), 4.04 (br, 1H), 3.85-3.74 (m, 6H), 3.49 (br, 1H), 3.18 (br, 1H), 3.05 (br, 1H), 2.67 (m, 2H), 2.28 (br, 1H), 1.92-1.50 (m, 9H), 1.02 (d, *J* = 6.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 188.0, 158.6, 157.2, 152.0, 147.1, 144.6, 143.0, 141.6, 131.3, 127.0, 124.8, 123.8, 122.2, 119.4, 119.2, 114.3, 103.8, 102.5, 98.6, 81.5, 66.7, 59.5, 56.4, 55.4, 42.4, 39.6, 37.7, 27.4, 25.0, 24.0, 22.5, 22.5; HRMS (ESI) calcd for $C_{33}H_{42}N_3O_4S$ $[M + H]^+$: 576.2891; found: 576.2908.

Preparation of *cis*-olefinic acid 4



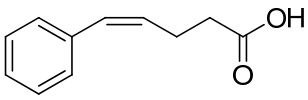
General Procedure for the preparation of *cis*-olefinic acid 4: To a solution of 4-bromobutyric acid (1.0 eq) in MeOH was added thionyl chloride (1.5 eq) dropwise at 0 °C and the reaction mixture was stirred at 25 °C overnight. The reaction was by adding sat'd $NaHCO_3$ solution and was then extracted with Et_2O. The organic layer was dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was subjected to flash chromatography to afford the desired ester product **A**.

A mixture of methyl 4-bromobutanoate **A** (1.0 eq) and triphenylphosphine (1.0 eq) was refluxed in MeCN for 6 hours to yield salt **B**. The crude phosphonium bromide **B** was recrystallized from MeCN and Et_2O to give the Wittig salt as white crystals.

The Wittig salt **B** (1.0 eq) was suspended in anhydrous THF at -20 °C. Sodium bis(trimethylsilyl)amide (1.0 eq) was added dropwise into the suspension and stirred for 20 min. The reaction mixture was further cooled to -78 °C, to which a solution of the aldehyde (1.0 eq) was added dropwise. The reaction mixture was allowed to rise to room temperature

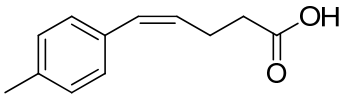
and stirred overnight. The reaction was then quenched with sat'd NH_4Cl solution and the product was then extracted with Et_2O. The organic layer was dried over Na_2SO_4, filtered and the concentrated *in vacuo*. The residue was separated by flash column chromatography on silica gel to afford the *cis*-alkenoate **C**.

To a solution of *cis*-alkenoate **C** (1.0 eq.) in THF:H_2O (1:1) was added LiOH (5.0 eq.). The reaction mixture was stirred at room temperature overnight. Upon termination, H_2O was added and an extraction with CH_2Cl_2 was performed. The organic layer was discarded. The aqueous layer was then acidified with 1 M HCl before it was extracted with CH_2Cl_2. The combined organic layer was dried over Na_2SO_4 and filtered. The solvent was removed *in vacuo* to yield *cis*-olefinic acid **4** in quantitative yield.



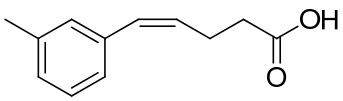
(*Z*)-5-Phenyl-4-pentenoic acid (4a)[2]

Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.37 (br, 1H), 7.44-7.31 (m, 5H), 6.57 (d, J = 11.52 Hz, 1H), 5.76-5.67 (m, 1H), 2.79-2.71 (m, 2H), 2.62-2.54 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.4, 137.2, 130.5, 129.9, 128.7, 126.9, 34.2, 23.8.



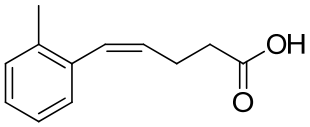
(*Z*)-5-*p*-toylpent-4-enoic acid (4b)

White solid; mp 54.4–55.0 °C; IR (KBr): 3009, 2927, 1700, 1508, 1425, 1296, 1212 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 11.57 (br, 1H), 7.28-7.24 (m, 4H), 6.55 (d, J = 11.4 Hz, 1H), 5.71-5.66 (m, 1H), 2.79-2.76 (m, 2H), 2.59-2.56 (m, 2H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 179.6, 136.4, 134.2, 130.2, 129.1, 128.9, 128.5, 34.1, 23.7, 21.0; HRMS (ESI) calcd for $C_{12}H_{14}O_2$ $[M - H]^-$: 189.0921; found:189.0919.



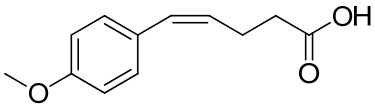
(*Z*)-5-*m*-toylpent-4-enoic acid (4c)

Yellow oil; IR (Neat): 3018, 2922, 1709, 1413, 1281, 1216 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.92 (br, 1H), 7.41-7.36 (m, 1H), 7.26-7.19 (m, 2H), 6.62 (d, J = 11.7 Hz, 1H), 5.81-5.73 (m, 1H), 2.82-2.79 (m, 2H), 2.65-2.50 (m, 2H), 2.51 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.6, 137.6, 136.9, 130.4, 129.6, 129.3, 128.0, 127.5, 125.6, 34.1, 23.6, 21.3; HRMS (ESI) calcd for $C_{12}H_{14}O_2$ $[M - H]^-$: 189.0921; found:189.0918.



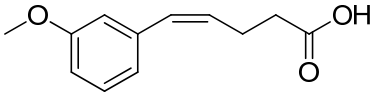
(*Z*)-5-*o*-toylpent-4-enoic acid (4d)

Pale yellow oil; IR (Neat): 3019, 1710, 1413, 1216 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.74 (br, 1H), 7.33 (s, 4H), 6.68 (d, *J* = 11.3 Hz, 1H), 5.91-5.82 (m, 1H), 2.71-2.54 (m, 4H), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.9, 136.3, 129.9, 129.9, 129.8, 128.9, 127.2, 125.5, 34.2, 23.6, 19.9; HRMS (ESI) calcd for $C_{12}H_{14}O_2$ $[M - H]^-$: 189.0921; found:189.0915.



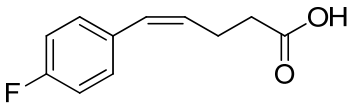
(*Z*)-5-(4-methoxyphenyl)pent-4-enoic acid (4e)

White solid; mp 64.2-65.0 °C; IR (KBr): 3031, 2837, 1698, 1508, 1251 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.25 (br, 1H), 7.30 (d, *J* = 8.7 Hz, 2H), 6.96 (d, *J* = 8.7 Hz, 2H), 6.50 (d, *J* = 11.7 Hz, 1H), 5.66-5.57 (m, 1H), 3.87 (s, 3H), 2.78-2.71 (m, 2H), 2.59-2.54 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.4, 158.3, 129.8, 129.7, 128.2, 113.6, 55.0, 34.0, 23.7; HRMS (ESI) calcd for $C_{12}H_{14}O_3$ $[M - H]^-$: 205.0870; found:205.0862.



(*Z*)-5-(3-methoxyphenyl)pent-4-enoic acid (4f)

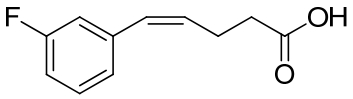
Colorless oil; IR (Neat): 3019, 1709, 1645, 1599, 1215 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.40 (br, 1H), 7.36-7.31 (m, 1H), 6.97-6.86 (m, 3H), 6.54 (d, *J* = 11.7 Hz, 1H), 5.76-5.57 (m, 1H), 3.87 (s, 3H), 2.80-2.72 (m, 2H), 2.59-2.54 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.4, 158.3, 129.8, 129.7, 128.2, 113.6, 55.0, 34.0, 23.7; HRMS (ESI) calcd for $C_{12}H_{14}O_3$ $[M - H]^-$: 205.0870; found:205.0862.



(*Z*)-5-(4-fluorophenyl)pent-4-enoic acid (4g)

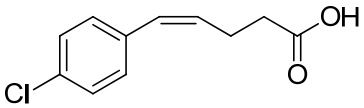
White solid; mp 45.6-46.9 °C; IR (KBr): 3030, 2927, 1699, 1604, 1405, 1241 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.79 (br, 1H), 7.32-7.27 (m, 2H), 7.12-7.06 (m, 2H) 6.50 (d, *J* = 11.7 Hz, 1H), 5.72-5.64 (m, 1H), 2.74-2.67 (m, 2H), 2.60-2.53 (m, 2H); ^{13}C NMR (75 MHz,

$CDCl_3$): δ 179.5, 161.6 (d, $J_{C,F}$ = 246 Hz), 133.1 (d, $J_{C,F}$ = 3 Hz), 130.2 (d, $J_{C,F}$ = 8 Hz), 129.7 (d, $J_{C,F}$ = 1 Hz), 129.2, 115.1 (d, $J_{C,F}$ = 21 Hz), 34.0, 23.6; HRMS (ESI) calcd for $C_{11}H_{11}FO_2$ $[M - H]^-$: 193.0670; found: 193.0672.



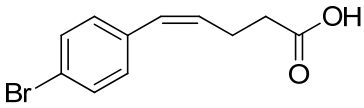
(*Z*)-5-(3-fluorophenyl)pent-4-enoic acid (4h)

Colorless oil; IR (Neat): 3019, 1708, 1611, 1580, 1487, 1440, 1234, 1136 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 11.51 (br, 1H), 7.39-7.32 (m, 1H), 7.13-6.98 (m, 3H), 6.51 (d, *J* = 11.7 Hz, 1H), 5.79-5.70 (m, 1H), 2.77-2.69 (m, 2H), 2.62-2.53 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.5, 162.6 (d, $J_{C,F}$ = 245 Hz), 139.2 (d, $J_{C,F}$ = 8 Hz), 131.0, 129.6 (d, $J_{C,F}$ = 6 Hz), 129.2 (d, $J_{C,F}$ = 2 Hz), 124.3 (3 Hz), 115.3 (d, $J_{C,F}$ = 21 Hz), 113.6 (d, $J_{C,F}$ = 21 Hz), 33.9, 23.6; HRMS (ESI) calcd for $C_{11}H_{11}FO_2$ $[M - H]^-$: 193.0670; found: 193.0668.



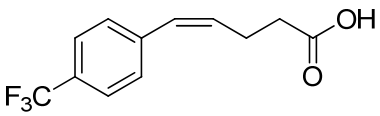
(*Z*)-5-(4-chlorophenyl)pent-4-enoic acid (4i)

White solid; mp 48.1-49.1 °C; IR (KBr): 2927, 1700, 1491, 1424, 1216, 1097 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 11.64 (br, 1H), 7.35 (d, *J* = 8.6 Hz, 2H), 7.24 (d, *J* = 8.6 Hz, 2H), 6.46 (d, *J* = 11.7 Hz, 1H), 5.74-5.66 (m, 1H), 2.72-2.65 (m, 2H), 2.59-2.51 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 178.9, 135.3, 132.3, 130.4, 129.8, 129.0, 128.1, 67.6, 33.8, 33.8, 25.3, 23.5; HRMS (ESI) calcd for $C_{11}H_{11}ClO_2$ $[M - H]^-$: 209.0375; found: 209.0366.



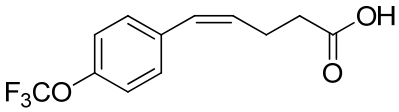
(*Z*)-5-(4-bromophenyl)pent-4-enoic acid (4j)

Pale yellow solid; mp 62.2-63.5 °C; IR (KBr): 2926, 1700, 1488, 1425, 1287, 1217, 1009 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.46 (d, *J* = 8.2 Hz, 2H), 7.13 (d, *J* = 8.2 Hz, 2H), 6.41 (d, *J* = 11.4 Hz, 1H), 5.67-5.65 (m, 1H), 2.64-2.60 (m, 1H), 2.54-2.48 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.2, 135.9, 131.3, 130.6, 130.3, 129.3, 34.0, 23.6; HRMS (ESI) calcd for $C_{11}H_{11}BrO_2$ $[M - H]^-$: 252.9870; found: 252.9861.



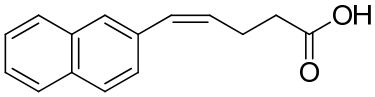
(*Z*)-5-[(4-trifluoromethyl)phenyl]pent-4-enoic acid (4k)

White semi-solid; IR (Neat): 3020, 1711, 1326, 1215, 1167, 1128, 1068 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.79 (br, 1H), 7.66 (d, J = 8.2 Hz, 2H), 7.43 (d, J = 8.2 Hz, 2H), 6.56 (d, J = 11.7 Hz, 1H), 5.86-5.77 (m, 1H), 2.76-2.68 (m, 2H), 2.63-2.54 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.6, 140.7 (q, J_{C-F} = 1 Hz), 132.0, 130.5, 129.2, 128.9, 125.2 (q, J_{C-F} = 3 Hz), 124.2 (q, J_{C-F} = 272 Hz), 33.9, 23.6; HRMS (ESI) calcd for $C_{12}H_{11}F_3O_2$ $[M - H]^-$: 243.0638; found: 243.0631.



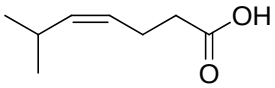
(*Z*)-5-[(4-trifluoromethoxy)phenyl]pent-4-enoic acid (4l)

Yellow oil; IR (Neat): 3020, 1712, 1507, 1415, 1262, 1215, 1170, cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.37 (br, 1H), 7.34 (d, J = 8.7 Hz, 2H), 7.24 (d, J = 8.1 Hz, 2H), 6.51 (d, J = 11.5 Hz, 1H), 5.77-5.69 (m, 1H), 2.71-2.69 (m, 2H), 2.58-2.53 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.6, 147.9 (q, J_{C-F} = 2 Hz), 135.8, 130.7, 130.0, 129.0, 120.7, 120.5 (q, J_{C-F} = 257 Hz), 34.0, 23.6; HRMS (ESI) calcd for $C_{12}H_{11}F_3O_3$ $[M - H]^-$: 259.0588; found: 259.0581.



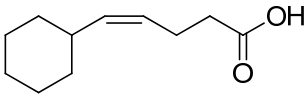
(Z)-5-(naphthalen-2-yl)pent-4-enoic acid (4m)

White solid; mp 87.7-88.4 °C; IR (KBr): 3010, 2921, 1700, 1632, 1405, 1272, 1253, 1180 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.88 (br, 1H), 7.97-7.62 (m, 4H), 7.61-7.55 (m, 3H) 6.78 (d, J = 11.7 Hz, 1H), 5.90-5.81 (m, 1H), 2.95-2.88 (m, 2H), 2.71-2.63 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ179.7, 134.5, 133.2, 132.2, 130.3, 130.2, 127.9, 127.7, 127.5, 1274, 126.9, 126.0, 125.7, 34.1, 23.7; HRMS (ESI) calcd for $C_{15}H_{14}O_2$ $[M - H]^-$: 225.0921; found: 225.0914.



(Z)-6-methylhept-4-enoic acid (4n)

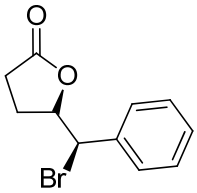
Colorless oil; IR (Neat): 3019, 2961, 1708, 1645, 1216 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 10.62 (br, 1H), 5.29-5.18 (m, 2H), 2.67-2.55 (m, 1H), 2.39 (s, 4H), 0.94 (d, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.9, 139.3, 124.5, 34.4, 26.5, 23.0, 22.6; HRMS (ESI) calcd for $C_8H_{14}O_2$ $[M - H]^-$: 141.0921; found: 141.0925.



(Z)-5-cyclohexylpent-4-enoic acid (4o)

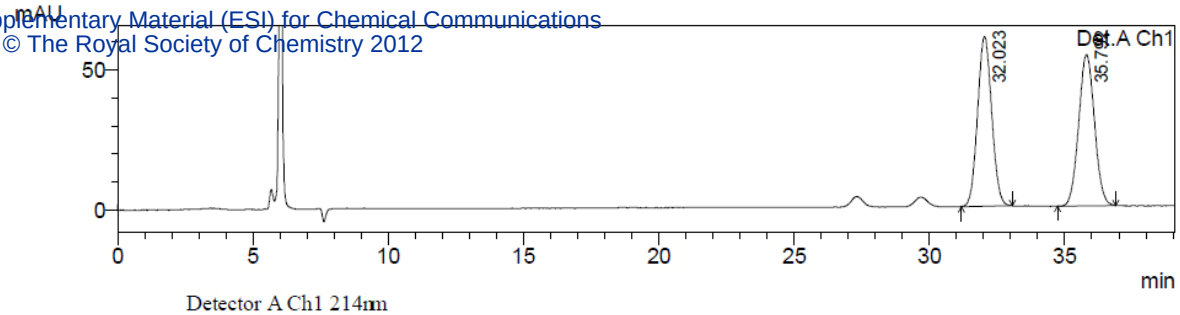
Colorless oil; IR (Neat): 3020, 2928, 1708, 1644, 1215 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.36 (br, 1H), 5.30-5.17 (m, 2H), 2.38 (s, 4H), 2.37-2.21 (m, 1H), 1.71-1.55 (m, 5H), 1.35-0.98 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 180.0, 137.8, 125.0, 36.3, 26.0, 25.9, 22.7; HRMS (ESI) calcd for $C_{11}H_{18}O_2$ $[M - H]^-$: 181.1234; found: 181.1231.

General procedure for Bromolactonization: The corresponding *cis*-alkenoic acid **4** (0.05 mmol, 1.0 eq) and catalyst **7** (3.0 mg, 0.005 mmol, 0.1 eq) were dissolved in $CHCl_3$ (0.5 ml) and *n*-hexane (1.0 ml) and stirred at -78 ^{o}C in the dark under nitrogen atmosphere. *N*-bromosuccinimide (10.6mg, 0.06 mmol, 1.2 eq) was added. The reaction mixture was stirred at the corresponding temperature and monitored by TLC. Sat'd Na_2SO_3 (3.0 ml) was added to quench the reaction mixture. The resulting biphasic mixture was warmed to 25 ^{o}C before it was diluted with water and extracted with CH_2Cl_2 (3 x 5 ml). The combined organic extracts were dried over $MgSO_4$, filtered and concentrated *in vacuo*. The residue was purified by flash column chromatography over silica gel to provide the corresponding product **5**.



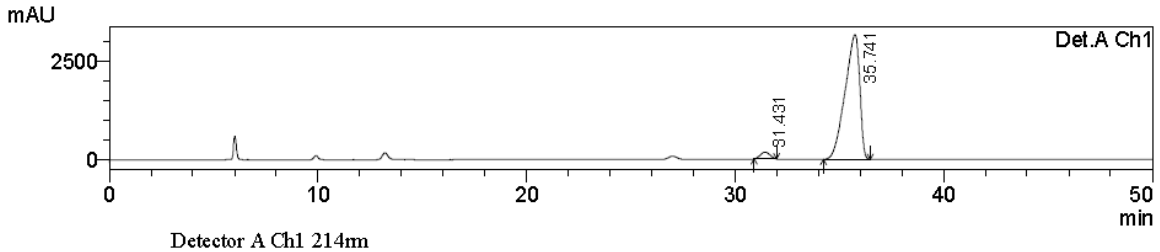
5-bromo(phenyl)methyldihydrofuran-2(3H)-one (5a)[2]

White solid; $[\alpha]_D^{25}$ -101.8 (*c* 0.8, $CHCl_3$, 93% ee); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.39-7.27 (m, 5H), 4.91 (d, *J* = 5.61 Hz, 1H), 4.86-4.80 (m, 1H), 2.47-2.26 (m, 2H), 2.21-2.09 (m, 1H), 2.02-1.89 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.8, 136.9, 129.1, 128.9, 128.5, 81.9, 55.1, 28.3, 25.6; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 31.4 min (minor), t_2 = 35.7 min (major).



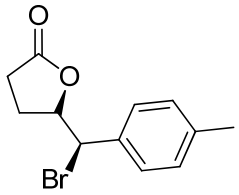
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.023	2190304	60568	49.932	52.891
2	35.792	2196230	53948	50.068	47.109
Total		4386534	114516	100.000	100.000



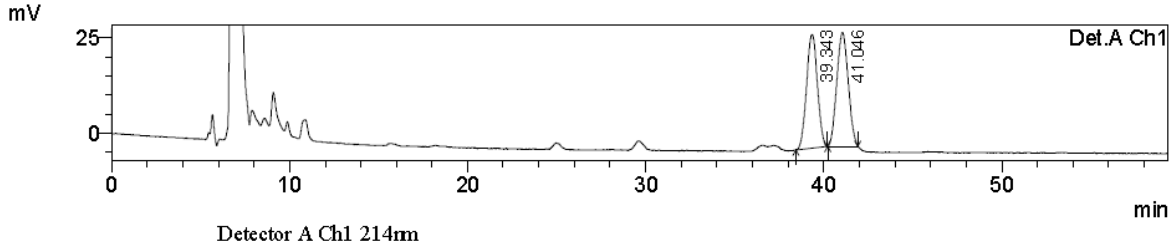
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.431	5562444	174883	3.475	5.236
2	35.741	154513311	3164890	96.525	94.764
Total		160075755	3339773	100.000	100.000



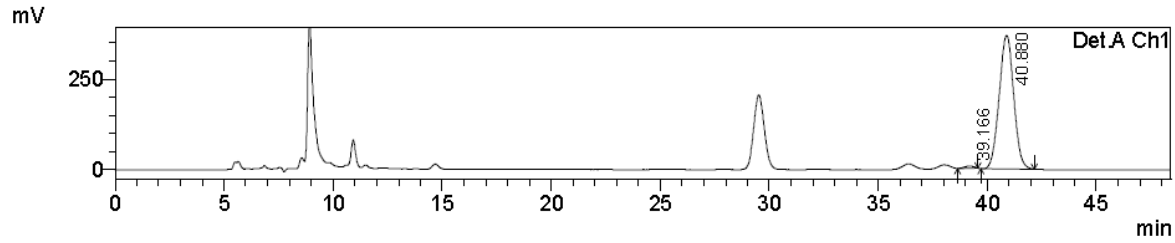
5-bromo(p-toyl)methyldihydrofuran-2(3H)-one (5b)

Colorless oil; $[\alpha]_D^{25}$ -34.0 (*c* 0.5, $CHCl_3$, 98% ee); IR (Neat): 3019, 1776, 1739, 1713, 1215 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.34 (d, *J* = 8.0 Hz, 2 H), 7.16 (d, *J* = 7.8 Hz, 2 H), 4.97 (d, *J* = 5.5 Hz, 1 H), 4.92-4.88 (m, 2H), 2.50-2.41 (m, 1H), 2.39 (s, 1H), 2.39-2.35 (m, 1H), 2.27-2.01 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.0, 139.2, 133.9, 129.5, 128.3, 82.1, 55.2, 28.3, 25.6, 21.1; HRMS (EI) calcd for $C_{12}H_{13}BrO_2$ $[M]^+$: 268.0099; found: 268.0089; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 20/80, 0.6 mL/min, 214 nm) t_1= 39.2 min (minor), t_2 = 40.9 min (major).



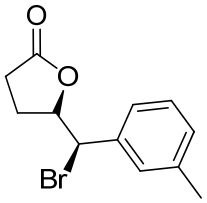
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	39.343	1254300	29695	48.603	49.909
2	41.046	1326395	29803	51.397	50.091
Total		2580695	59498	100.000	100.000



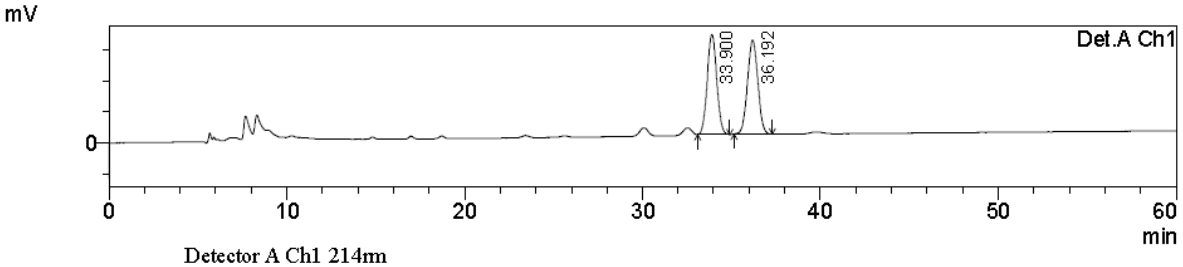
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	39.166	177592	5880	1.030	1.572
2	40.880	17060333	368158	98.970	98.428
Total		17237924	374038	100.000	100.000



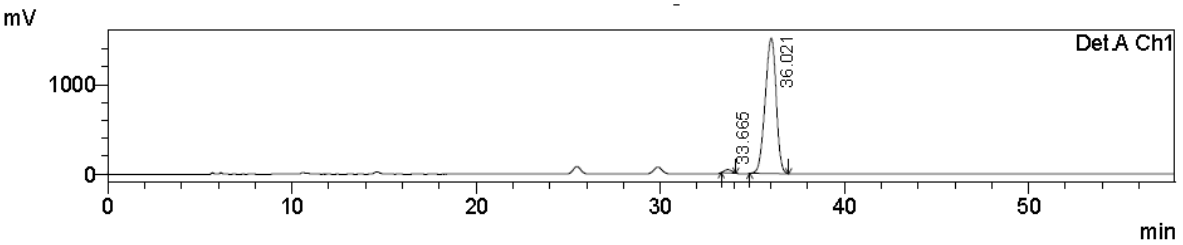
5-bromo(m-toyl)methyldihydrofuran-2(3H)-one (5c)

Colorless oil; $[\alpha]_D^{25}$ -111.7 (*c* 1.1, $CHCl_3$, 97% ee); IR (Neat): 3020, 1780, 1216 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.26-7.15 (m, 4H), 4.96-4.88 (m, 2H), 2.52-2.41 (m, 2H), 2.36 (s, 3H), 2.30-2.17 (m, 1H), 2.11-1.99 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.3, 138.7, 136.8, 129.9, 129.0, 128.8, 125.5, 121.8, 82.1, 55.3, 28.4, 25.8, 21.4; HRMS (EI) calcd for $C_{12}H_{13}BrO_2$ $[M]^+$: 268.0099; found: 268.0091; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 20/80, 0.6 mL/min, 214 nm) t_1= 33.7 min (minor), t_2 = 36.0 min (major).



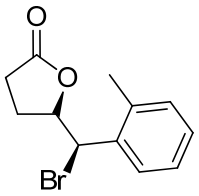
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	33.900	5973241	160716	49.442	51.520
2	36.192	6108023	151233	50.558	48.480
Total		12081264	311949	100.000	100.000



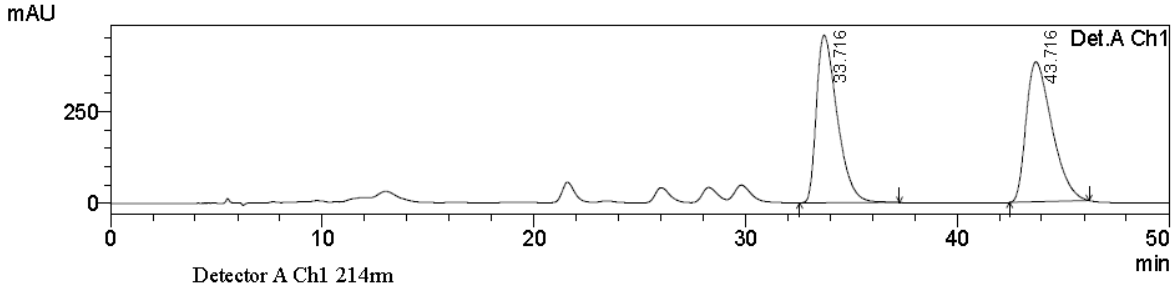
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	33.665	979211	35032	1.548	2.275
2	36.021	62296213	1504840	98.452	97.725
Total		63275424	1539871	100.000	100.000



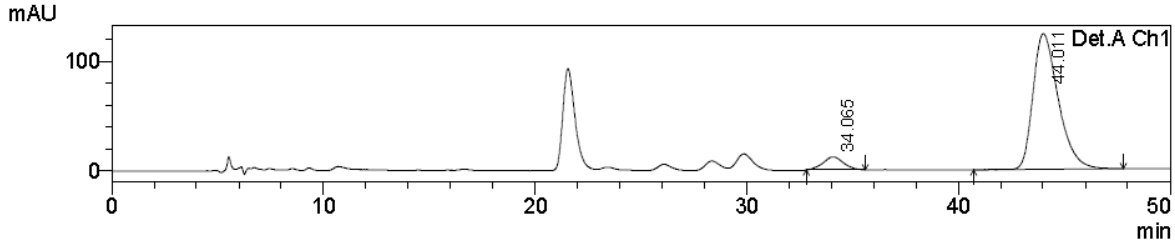
5-bromo(o-toyl)methyldihydrofuran-2(3H)-one (5d)

(5-*exo* : 6-*endo* = 11:1) Colorless oil; $[\alpha]_D^{25}$ -20.1 (*c* 0.6, $CHCl_3$, 86% ee); IR (Neat): 3018, 2972, 1732, 1216 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.56-7.54 (m, 1H), 7.26-7.16 (m, 3H), 5.21 (d, *J* = 6.4 Hz, 1H), 5.04-5.00 (m, 1H), 2.55-2.52 (m, 2H), 2.40 (s, 3H), 2.30-2.26 (m, 1H), 1.97-1.93 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.7, 135.6, 130.9, 128.9, 128.2, 126.9, 82.0, 51.5, 28.5, 26.3, 19.4; HRMS (EI) calcd for $C_{12}H_{13}BrO_2$ $[M]^+$: 268.0099; found: 268.0087; HPLC (Daicel Chiralpak ODH, *i*-PrOH/*n*-hexane = 20/80, 0.6 mL/min, 214 nm) t_1= 34.1 min (minor), t_2 = 44.0 min (major).



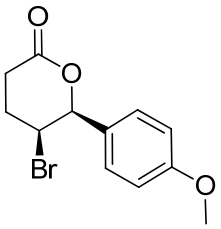
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	33.716	31278590	456815	49.007	54.486
2	43.716	32546445	381592	50.993	45.514
Total		63825035	838408	100.000	100.000



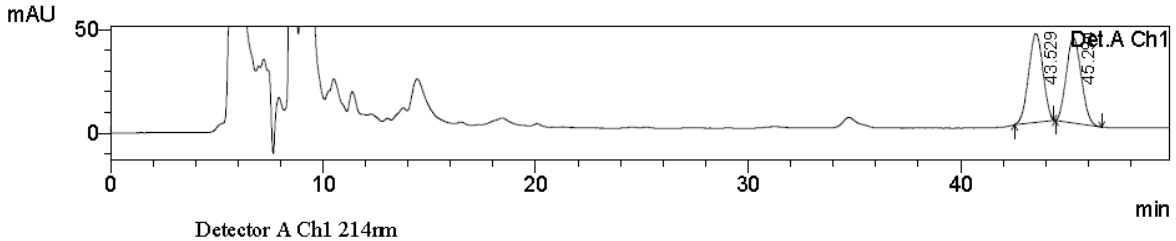
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.065	800386	11857	7.249	8.769
2	44.011	10241387	123361	92.751	91.231
Total		11041772	135218	100.000	100.000



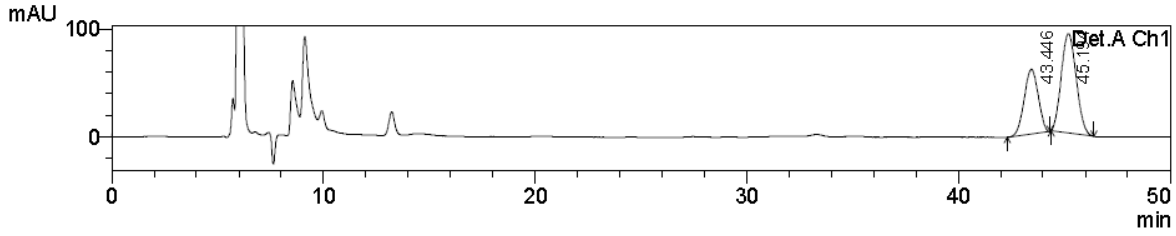
(5R,6S)-5-bromo-6-(4-methoxyphenyl)tetrahydro-2H-pyran-2-one (8e)

White solid; $[\alpha]_D^{25}$ -7.8 (*c* 0.2, $CHCl_3$, 24% ee); ^1H NMR (300 MHz, $CDCl_3$): δ 7.25 (d, *J* = 8.4 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 5.49 (d, *J* = 6.8 Hz, 1H), 4.37-4.31 (m, 1H), 3.82 (s, 3H), 2.99-2.88 (m, 1H), 2.76-2.65 (m, 1H), 2.49-2.40 (m, 1H), 2.34-2.23 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.2, 160.1, 129.3, 127.8, 114.1, 85.4, 55.3, 47.4, 28.5, 28.0; HRMS (EI) calcd for $C_{12}H_{13}BrO_3$ $[M]^+$: 284.0048; found: 284.0046; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 43.4 min (minor), t_2 = 45.2 min (major).



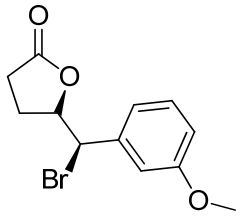
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	43.529	2011358	42908	49.922	51.078
2	45.294	2017675	41097	50.078	48.922
Total		4029033	84005	100.000	100.000



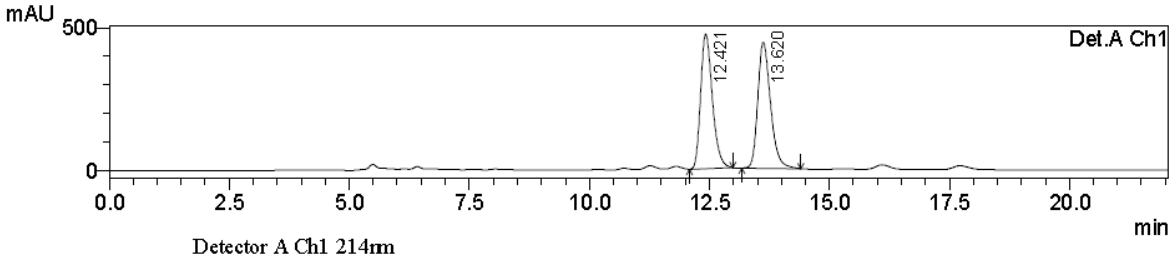
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	43.446	2762825	59964	37.842	39.300
2	45.194	4538209	92614	62.158	60.700
Total		7301035	152578	100.000	100.000



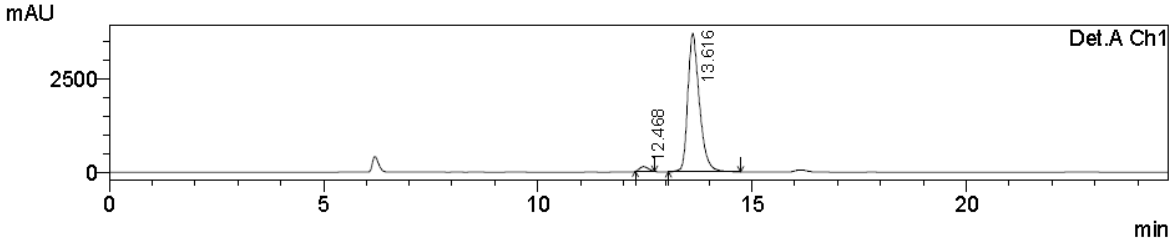
5-bromo(o-toyl)methyldihydrofuran-2(3H)-one (5f)

Pale yellow oil; $[\alpha]_D^{25}$ -122.9 (*c* 1.0, $CHCl_3$, 95% ee); IR (Neat): 3020, 1780, 1215 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.30-7.25 (m, 1H), 7.03-6.98 (m, 2H), 6.89-6.85 (m, 1H), 4.96-4.87 (m, 2H), 3.82 (s, 3H), 2.55-2.42 (m, 2H), 2.40-2.18 (m, 1H), 2.11-1.98 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 175.9, 159.8, 138.2, 130.0, 120.6, 114.5, 114.2, 82.0, 55.3, 55.0, 28.3, 25.7; HRMS (EI) calcd for $C_{12}H_{13}BrO_2$ $[M]^+$: 284.0048; found: 284.0044; HPLC (Daicel Chiralpak IA, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 12.5 min (minor), t_2 = 13.6 min (major).



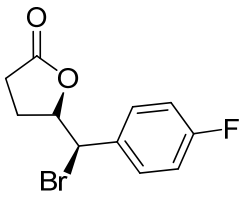
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.421	7998445	471912	49.260	51.617
2	13.620	8238685	442336	50.740	48.383
Total		16237130	914248	100.000	100.000



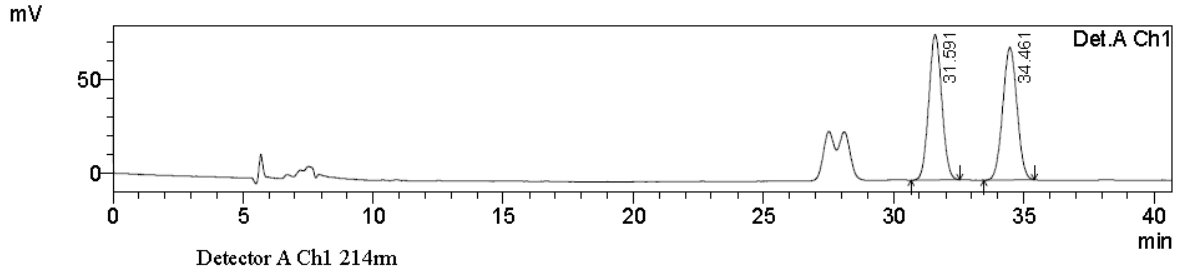
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.468	1733340	123692	2.420	3.226
2	13.616	69901043	3710688	97.580	96.774
Total		71634383	3834380	100.000	100.000



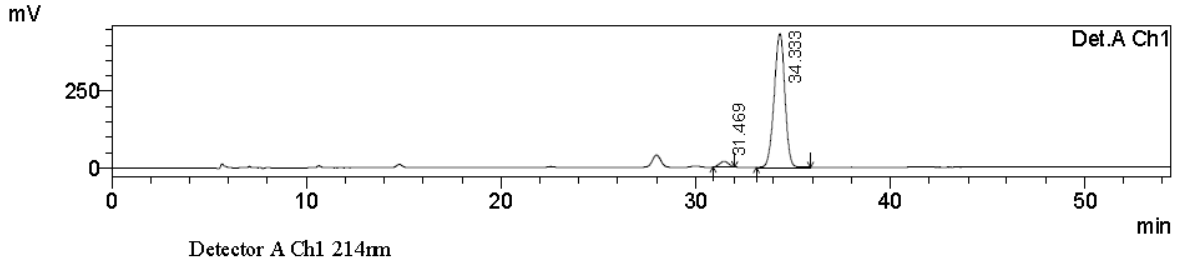
5-bromo(p-fluorophenyl)methyldihydrofuran-2(3H)-one (5g)

(5-*exo* : 6-*endo* = 23:1) Yellow oil; $[\alpha]_D^{25}$ -100.8 (*c* 0.6, $CHCl_3$, 93% ee); IR (Neat): 3020, 1780, 1644, 1607, 1511, 1216 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.47-7.44 (m, 2H), 7.07-7.03 (m, 2H), 4.99 (d, *J* = 5.0 Hz, 1H), 4.88-4.84 (m, 1H), 2.52-2.44 (m, 2H), 2.31-2.23 (m, 1H), 2.11-2.04 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 175.8, 162.8 (d, J_{C-F} = 249 Hz), 133.0 (d, J_{C-F} = 4 Hz), 130.3 (d, J_{C-F} = 8 Hz), 115.8 (d, J_{C-F} = 22 Hz), 81.7, 54.5, 28.2, 25.7; HRMS (EI) calcd for $C_{11}H_{10}BrFO_2$ $[M]^+$: 271.9848; found: 271.9851; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 20/80, 0.6 mL/min, 214 nm) t_1= 31.5 min (minor), t_2 = 34.3 min (major).



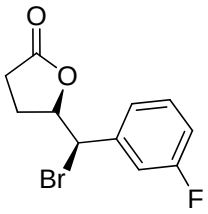
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.591	2692427	77488	50.050	52.319
2	34.461	2687094	70620	49.950	47.681
Total		5379522	148108	100.000	100.000



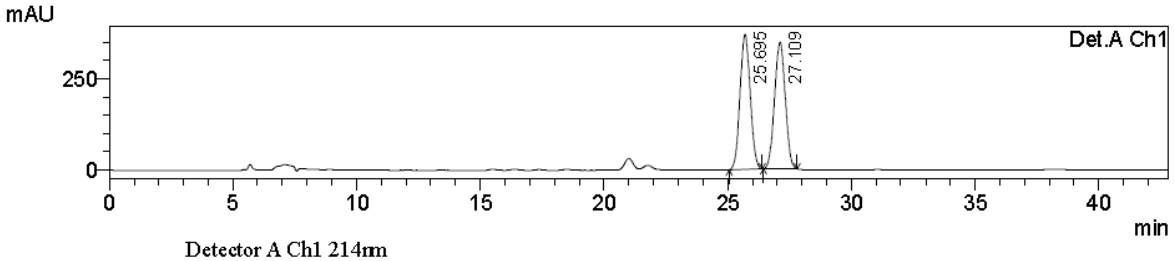
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.469	583532	18457	3.425	4.090
2	34.333	16453813	432858	96.575	95.910
Total		17037344	451315	100.000	100.000



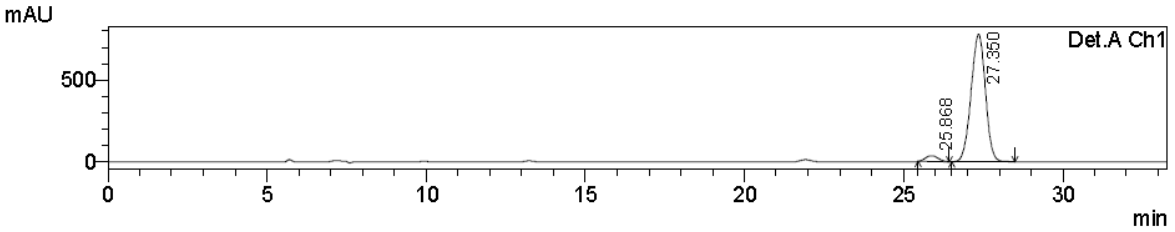
5-bromo(m-fluorophenyl)methyldihydrofuran-2(3H)-one (5h)

Colorless oil; $[\alpha]_D^{25}$ -128.8 (*c* 0.9, $CHCl_3$, 92% ee); IR (Neat): 3020, 1782, 1216 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.34-7.31 (m, 1H), 7.23-7.20 (m, 2H), 7.05-7.01 (m, 1H), 4.97 (d, J = 5.1 Hz, 1H), 4.88-4.84 (m, 1H), 2.55-2.44 (m, 2H), 2.32-2.25 (m, 1H), 2.11-2.05 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.7, 162.6 (d, J_{C-F} = 248 Hz), 139.4 (d, J_{C-F} = 7 Hz), 130.4 (d, J_{C-F} = 8 Hz), 124.1 (d, J_{C-F} = 3 Hz), 116.1 (d, J_{C-F} = 21 Hz), 115.7 (d, J_{C-F} = 23 Hz), 81.5, 54.2 (d, J_{C-F} = 2 Hz), 28.2, 25.7; HRMS (EI) calcd for $C_{11}H_{10}BrFO_2$ $[M]^+$: 271.9848; found: 271.9857; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 25.9 min (minor), t_2 = 27.4 min (major).



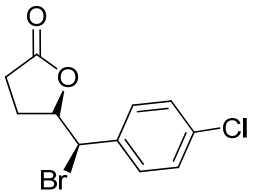
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.695	10593981	368772	50.338	51.574
2	27.109	10451836	346267	49.662	48.426
Total		21045817	715039	100.000	100.000



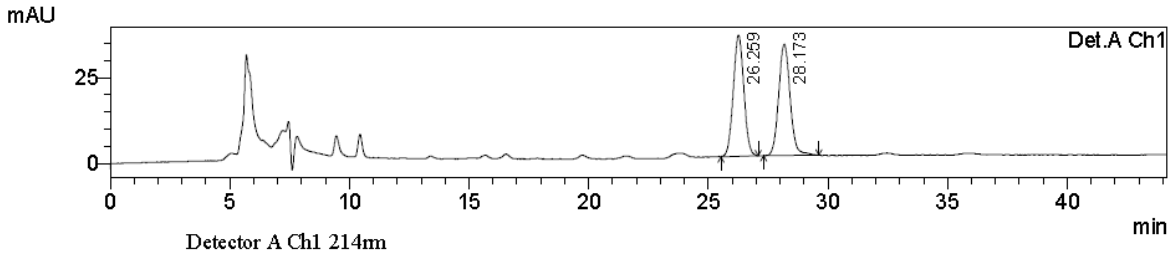
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.868	977775	35869	3.862	4.413
2	27.350	24342333	777010	96.138	95.587
Total		25320109	812879	100.000	100.000



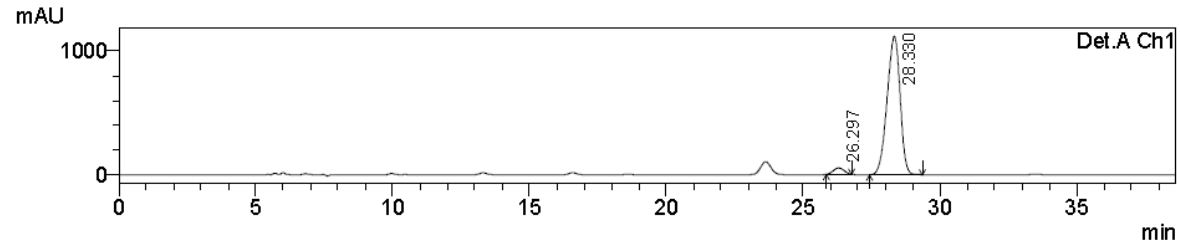
5-bromo(p-chlorophenyl)methyldihydrofuran-2(3H)-one (5i)

Colorless oil; $[\alpha]_D^{25}$ -115.1 (*c* 0.8, $CHCl_3$, 93% ee); IR (Neat): 3020, 1780, 1215 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.42-7.28 (m, 4H), 4.97 (d, *J* = 3.0 Hz, 1H), 4.87-4.84 (m, 1H), 2.55-2.42 (m, 2H), 2.32-2.25 (m, 1H), 2.11-2.04 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.8, 135.5, 135.1, 129.8, 129.1, 81.5, 54.3, 28.2, 25.6; HRMS (EI) calcd for $C_{11}H_{10}ClO_2$ $[M\text{-}Br]^+$: 209.0364; found: 209.0370; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 26.3 min (minor), t_2 = 28.3 min (major).



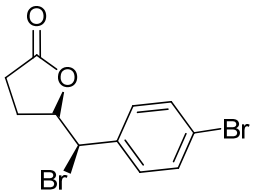
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.259	1074607	35211	49.903	52.144
2	28.173	1078780	32316	50.097	47.856
Total		2153387	67526	100.000	100.000



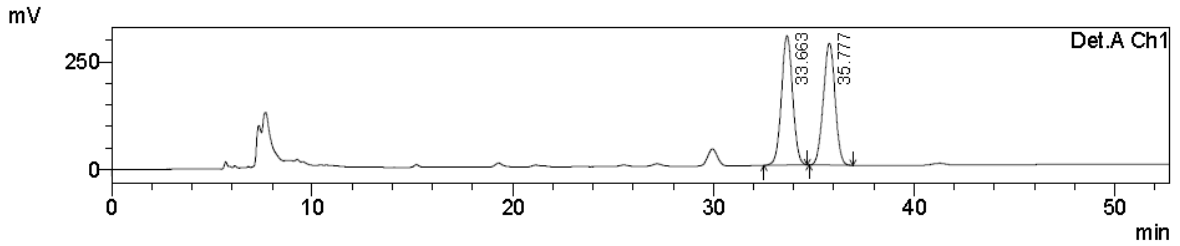
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.297	1409239	51005	3.672	4.360
2	28.330	36972729	1118820	96.328	95.640
Total		38381968	1169824	100.000	100.000



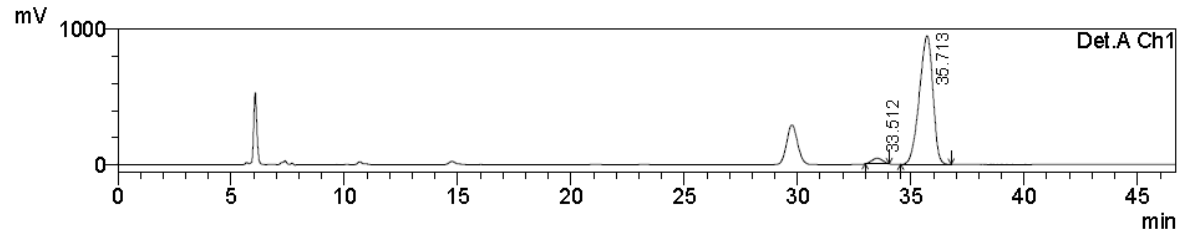
5-bromo(p- bromophenyl)methyldihydrofuran-2(3H)-one (5j)

(5-*exo* : 6-*endo* = 5:1) Orange semi-solid; $[\alpha]_D^{25}$ -91.7 (*c* 1.3, $CHCl_3$, 94% ee); IR (Neat): 3020, 1780, 1216 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.50 (d, *J* = 8.8 Hz, 2H), 7.35 (d, *J* = 8.2 Hz, 2H), 4.95 (d, *J* = 4.9 Hz, 1H), 4.88-4.82 (m, 1H), 2.55-2.41 (m, 2H), 2.35-2.23 (m, 1H), 2.13-2.01 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.7, 136.1, 132.0, 130.1, 123.3, 81.4, 54.4, 28.2, 25.6; HRMS (EI) calcd for $C_{11}H_{10}Br_2O_2$ $[M]^+$: 331.9048; found: 331.9054; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 20/80, 0.6 mL/min, 214 nm) t_1= 33.5 min (minor), t_2 = 35.7 min (major).



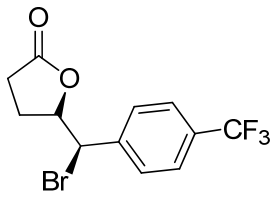
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	33.663	11653935	299599	50.345	51.478
2	35.777	11494112	282395	49.655	48.522
Total		23148046	581995	100.000	100.000



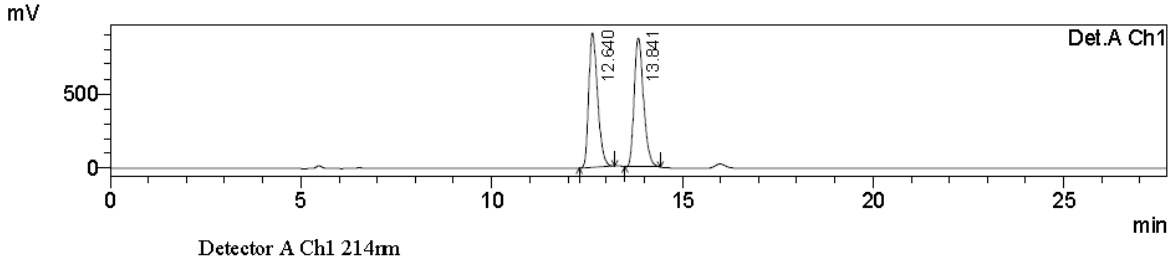
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	33.512	1303128	39621	3.206	4.017
2	35.713	39347033	946756	96.794	95.983
Total		40650160	986378	100.000	100.000



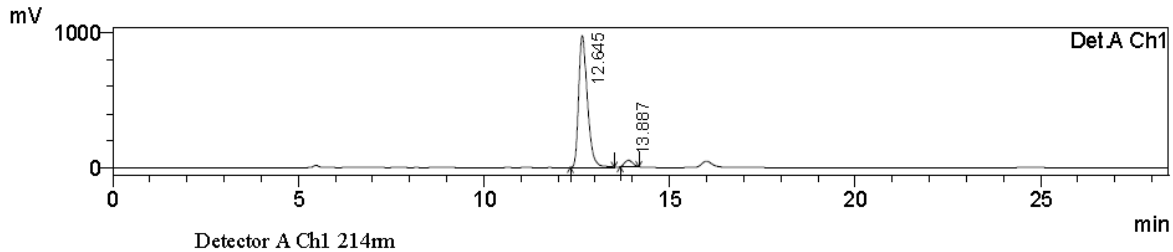
5-bromo-[(p-trifluoromethyl)phenyl]methyldihydrofuran-2(3H)-one (5k)

Yellow semi-solid; $[\alpha]_D^{25}$ -100.9 (*c* 0.6, $CHCl_3$, 91% ee); IR (Neat): 3021, 1781, 1326, 1216, 1170, 1115, 1068 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.66-7.59 (m, 4H), 5.03 (d, J = 4.6 Hz, 1H), 4.90-4.84 (m, 1H), 2.55-2.44 (m, 2H), 2.38-2.27 (m, 1H), 2.17-2.05 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.7, 141.0, 131.2 (q, $J_{C,F}$ = 33 Hz), 129.0, 125.8 (q, $J_{C,F}$ = 4 Hz), 123.7 (q, $J_{C,F}$ = 272 Hz), 81.2, 54.1, 28.2, 25.7; HRMS (EI) calcd for $C_{12}H_{10}BrF_3O_2$ $[M]^+$: 321.9816; found: 321.9803; HPLC (Daicel Chiralpak IA, *i*-PrOH/*n*-hexane = 20/80, 0.6 mL/min, 214 nm) t_1= 12.6 min (major), t_2 = 13.9 min (minor).



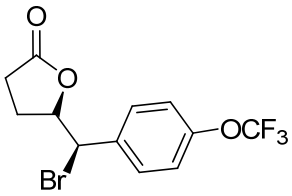
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.640	14544631	903926	49.409	51.038
2	13.841	14892792	867147	50.591	48.962
Total		29437423	1771073	100.000	100.000



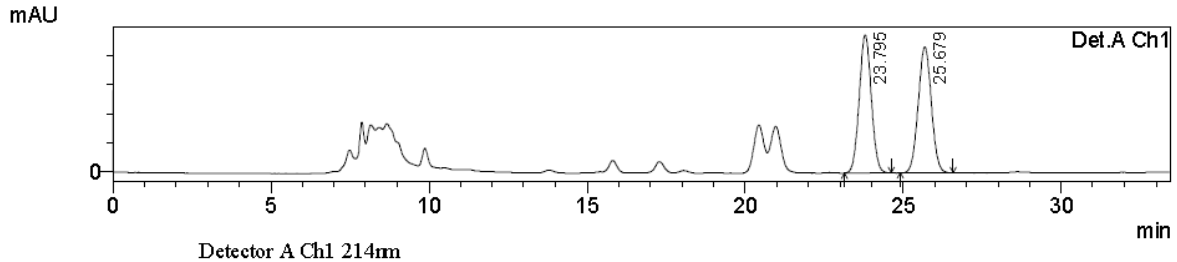
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.645	16050367	978616	95.658	95.282
2	13.887	728626	48462	4.342	4.718
Total		16778993	1027078	100.000	100.000



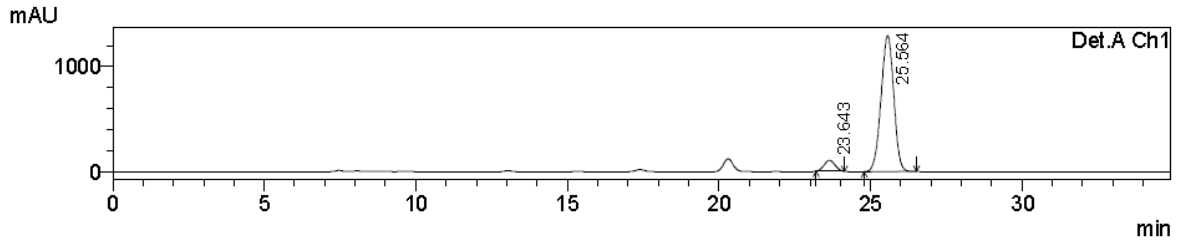
5-bromo-[(p-trifluoromethoxy)phenyl]methyldihydrofuran-2(3H)-one (5l)

Yellow semi-solid; $[\alpha]_D^{25}$ -96.2 (*c* 1.0, $CHCl_3$, 87% ee); IR (Neat): 3020, 1781, 1216 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.53-7.50 (m, 2H), 7.21 (d, *J* = 8.2 Hz, 2H), 5.01 (d, *J* = 3.0 Hz, 1H), 4.87-4.83 (m, 1H), 2.53-2.49 (m, 2H), 2.35-2.27 (m, 1H), 2.13-2.08 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.8, 149.4 (q, $J_{C,F}$ = 1.8 Hz), 135.8, 130.1, 121.1, 120.3 (q, $J_{C,F}$ = 258 Hz), 81.4, 54.2, 28.2, 25.7; HRMS (EI) calcd for $C_{12}H_{10}BrF_3O_3$ $[M]^+$: 337.9765; found: 337.9769; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 23.6 min (minor), t_2 = 25.6 min (major).



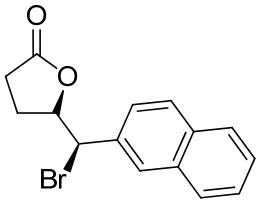
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.795	2517899	94703	50.106	52.262
2	25.679	2507247	86505	49.894	47.738
Total		5025145	181208	100.000	100.000



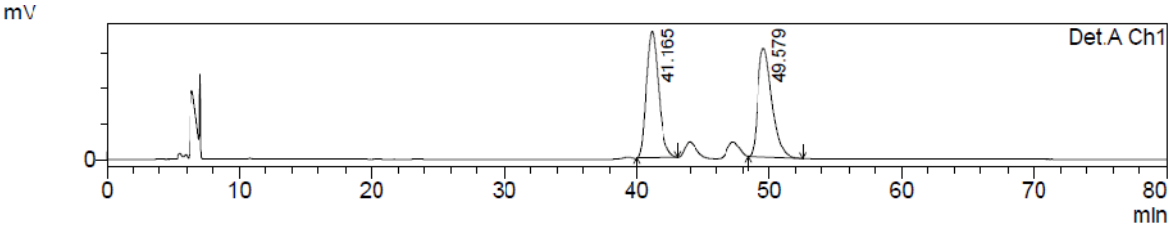
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.643	2617723	103788	6.501	7.435
2	25.564	37651339	1292232	93.499	92.565
Total		40269062	1396020	100.000	100.000



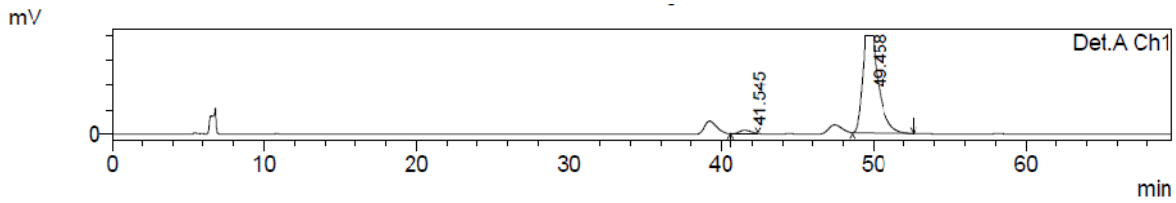
5-bromo(naphthalene-2-yl)methyldihydrofuran-2(3H)-one (5m)

Yellow semi-solid; $[\alpha]_D^{25}$ -102.0 (*c* 1.0, $CHCl_3$, 95% ee); IR (Neat): 3019, 1776, 1326, 1221, 1210, 1169, 1136, 1068 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.88-7.83 (m, 4H), 7.60-7.51 (m, 3H), 5..15 (d, *J* = 5.6 Hz, 1H), 5.05-4.98 (m, 1H), 2.52-2.45 (m, 2H), 2.31-2.19 (m, 1H), 2.16-2.06 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 178.6, 134.3, 133.3, 132.9, 128.9, 128.1, 127.7, 127.6, 127.0, 126.8, 125.6, 82.0, 55.7, 28.4, 25.9; HRMS (EI) calcd for $C_{15}H_{13}BrO_2$ $[M]^+$: 304.0099; found: 304.0096; HPLC (Daicel Chiralpak IA, *i*-PrOH/*n*-hexane = 5/95, 0.6 mL/min, 214 nm) t_1= 41.5 min (minor), t_2 = 49.5 min (major).



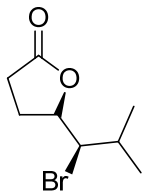
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	41.165	112834642	1786832	49.949	53.509
2	49.579	113064004	1552454	50.051	46.491
Total		225898646	3339286	100.000	100.000



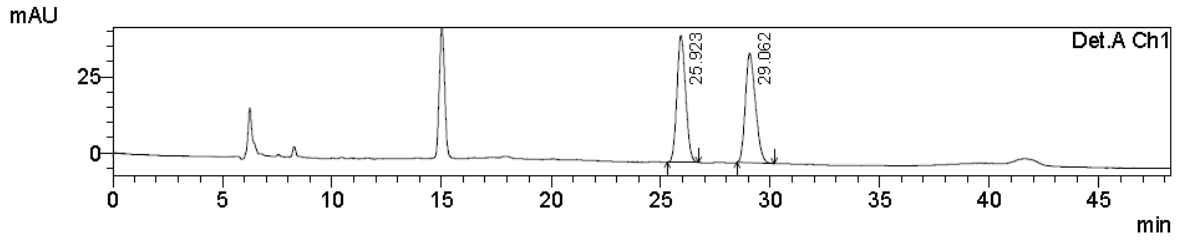
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	41.545	6226762	128075	2.149	3.143
2	49.458	283510956	3946707	97.851	96.857
Total		289737718	4074782	100.000	100.000



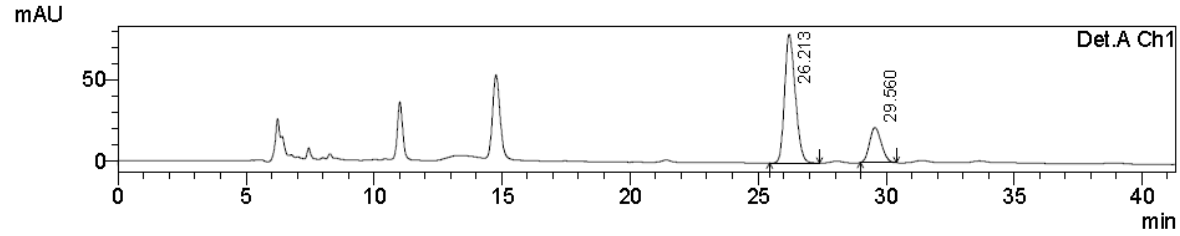
5-bromo(isopropyl)methyldihydrofuran-2(3H)-one (5n)

Yellow oil; $[\alpha]_D^{25}$ -19.9 (*c* 1.1, $CHCl_3$, 53% ee); IR (Neat): 3019, 1772, 1216 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 4.76-4.70 (m, 1H), 3.88-3.84 (m, 1H), 2.77-2.65 (m, 1H), 2.58-2.43 (m, 1H), 2.41-2.44 (m, 1H), 2.22-2.05 (m, 1H), 1.09 (d, *J* = 1.0 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 176.3, 79.9, 66.8, 32.9, 28.3, 26.7, 21.2, 20.3; HRMS (EI) calcd for $C_8H_{13}O_2$ $[M-Br]^+$: 141.0910; found: 141.0909; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 26.2 min (major), t_2 = 29.6 min (minor).



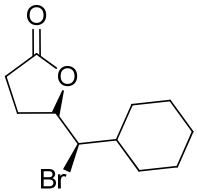
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.923	1152502	41618	50.049	53.650
2	29.062	1150224	35954	49.951	46.350
Total		2302727	77572	100.000	100.000



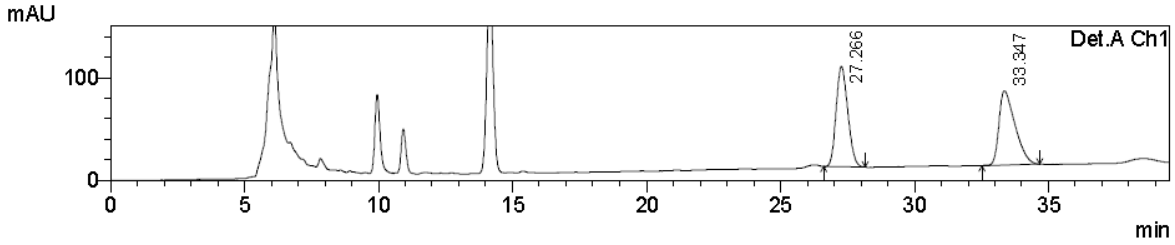
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.213	2295322	79603	76.786	78.529
2	29.560	693909	21765	23.214	21.471
Total		2989231	101368	100.000	100.000



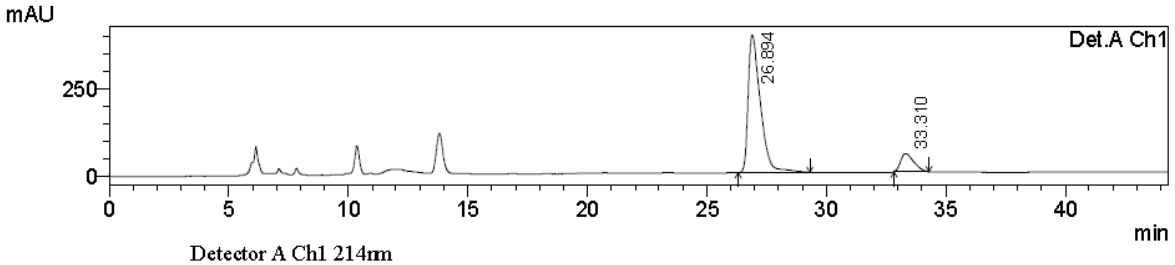
5-bromo(cyclohexyl)methyldihydrofuran-2(3H)-one (5o)

Colorless oil; $[\alpha]_D^{25}$ -31.1 (*c* 1.0, $CHCl_3$, 75% ee); IR (Neat): 3020, 2933, 1775, 1216 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 4.82-4.76 (m, 1H), 3.87-3.84 (m, 1H), 2.78-2.67 (m, 1H), 2.57-2.45 (m, 1H), 2.42-2.30 (m, 1H), 2.26-2.14 (m, 1H), 2.05 (d, *J* = 12.0 Hz, 1H), 1.89-1.74 (m, 4H), 1.67 (d, *J* = 12.0 Hz, 1H), 1.35-1.05 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.3, 78.8, 66.3, 54.3, 42.4, 31.3, 31.2, 28.2, 26.6, 26.1, 26.0, 25.9; HRMS (EI) calcd for $C_{11}H_{17}BrO_2$ $[M]^+$: 260.0412; found: 260.0409; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 26.9 min (major), t_2 = 33.3 min (minor).



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.266	2969120	97785	49.680	57.533
2	33.347	3007346	72178	50.320	42.467
Total		5976466	169962	100.000	100.000



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.894	14067363	393692	87.561	88.323
2	33.310	1998503	52048	12.439	11.677
Total		16065865	445740	100.000	100.000

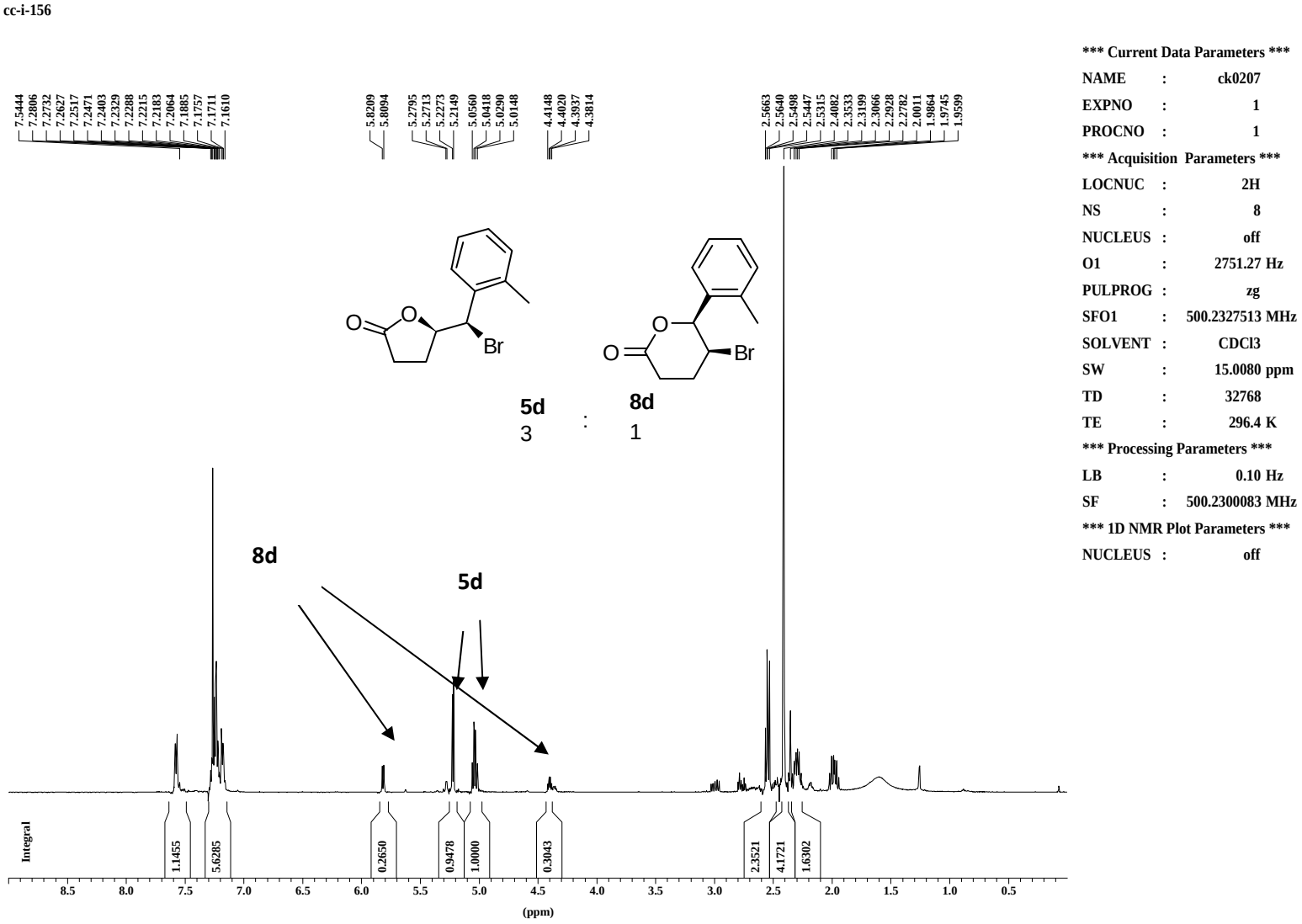
C. Study on the effect on regioselectivity by various catalysts

The regioselectivities of the reaction were determined via the ratio of the integration values of both the 5-*exo* and 6-*endo* lactone products (spectrums attached in the following pages). The ^{1}H signals corresponding to the 5-*exo* and 6-*endo* lactones are in agreement with the literature values.[5]

General procedure for the bromolactonization of 4d

4d (95 mg, 0.05 mmol, 1.0 eq) and the corresponding catalyst (3.0 mg, 0.005 mmol, 0.1 eq) were dissolved in CH_2Cl_2 (1.5 ml) at 25 ^oC in the dark under nitrogen atmosphere. *N*-bromosuccinimide (10.6 mg, 0.06 mmol, 1.2 eq) was then added. The reaction mixture was stirred at 25 ^oC for 24 h before subjecting to the work up procedure for the bromolactonization as described above.

Scheme 2, with $Ph_3P=S$ (10 mol%) as catalyst



*** Current Data Parameters ***
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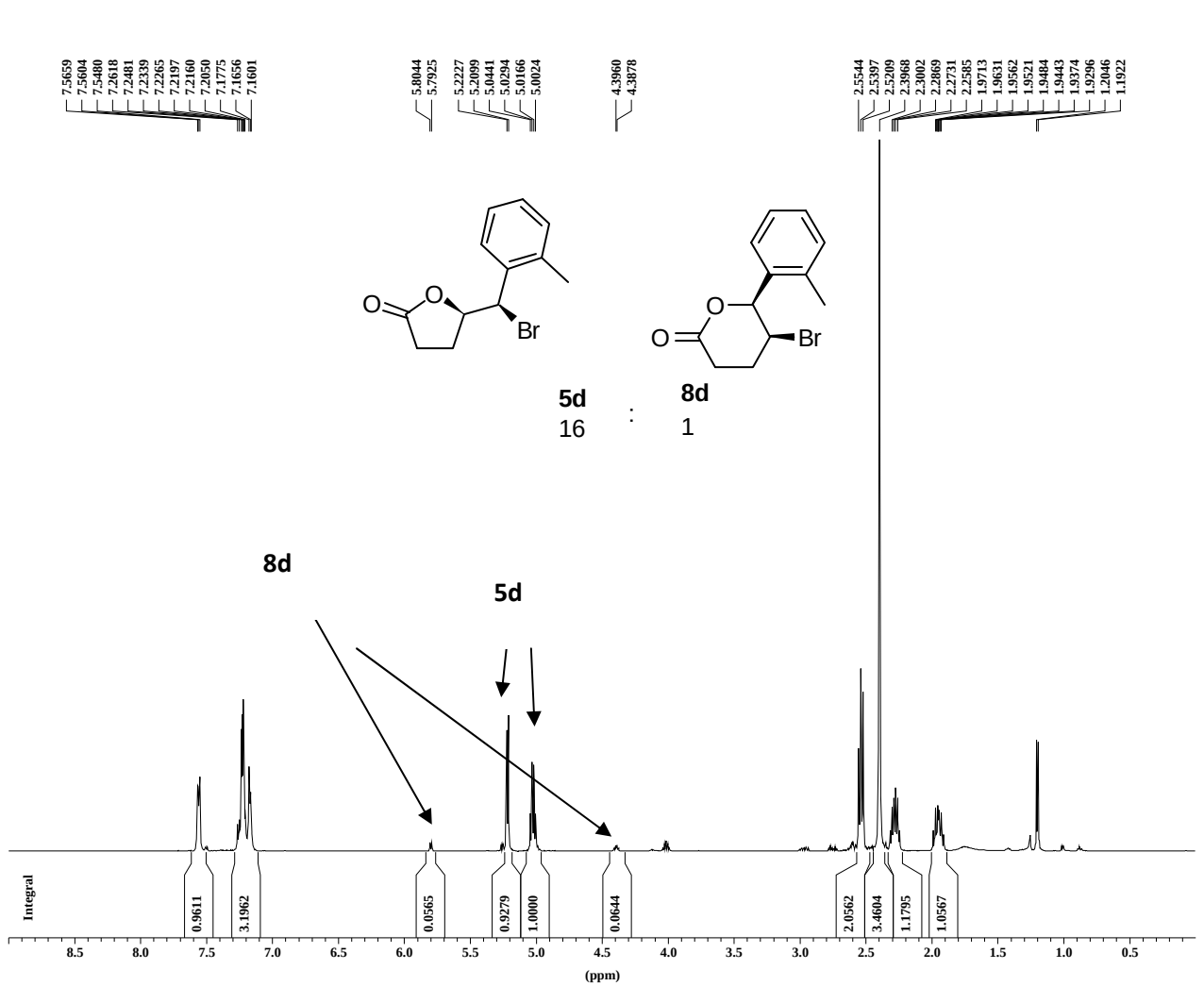
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
LOCNUC : 2H
NS : 8
NUCLEUS : off
O1 : 2751.27 Hz
PULPROG : zg
SFO1 : 500.2327513 MHz
SOLVENT : CDCl3
SW : 15.0080 ppm
TD : 32768
TE : 296.4 K
*** Processing Parameters ***
LB : 0.10 Hz
SF : 500.2300083 MHz
*** 1D NMR Plot Parameters ***
NUCLEUS : off

Scheme 2, with 6b:7 (1:1, 10 mol%) as catalyst

ck-v-119



*** Current Data Parameters ***

NAME : ck0131

EXPNO : 1

PROCNO : 1

*** Acquisition Parameters ***

LOCNUC : 2H

NS : 8

NUCLEUS : off

O1 : 2751.27 Hz

PULPROG : zg

SFO1 : 500.2327513 MHz

SOLVENT : CDCl3

SW : 15.0080 ppm

TD : 32768

TE : 298.0 K

*** Processing Parameters ***

LB : 0.10 Hz

SF : 500.2300083 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS : off

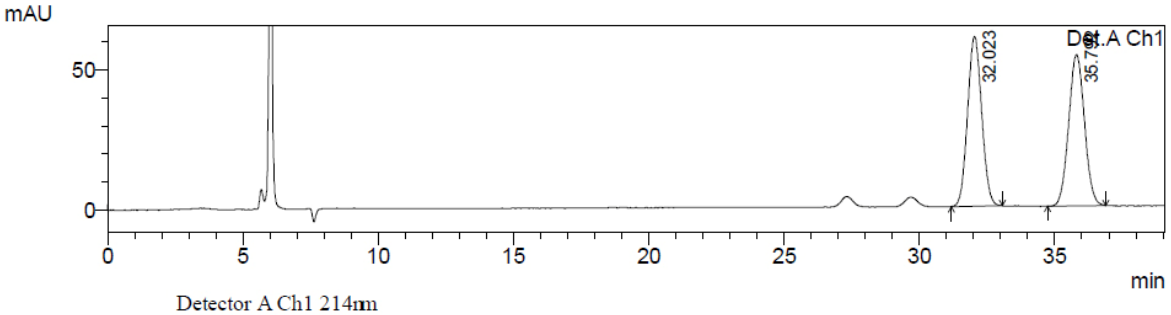
D. Study on the effect of various halogen sources

Olefinic acid **2** was synthesized according to our previous report.[2]

General procedure for the halolactonization with various halogen sources

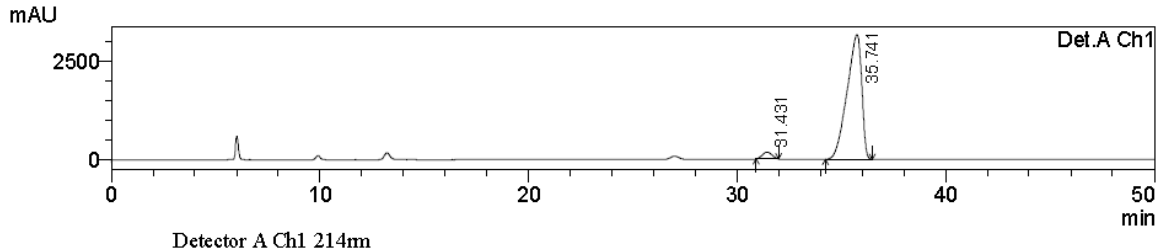
The corresponding olefinic acid (0.05 mmol, 1.0 eq) and the corresponding catalyst (0.005 mmol, 0.1 eq) were dissolved in the stated solvent blends at -78 °C in the dark under nitrogen atmosphere. The corresponding halogen source (0.06 mmol, 1.2 eq) was then added. The reaction mixture was stirred at -78 °C and monitored using TLC until completion. Upon completion the mixture was subjected to the work up procedure for the bromolactonization as described above.

Bromolactonization with NBS (Table 3, entry 1)



Detector A Ch1 214nm

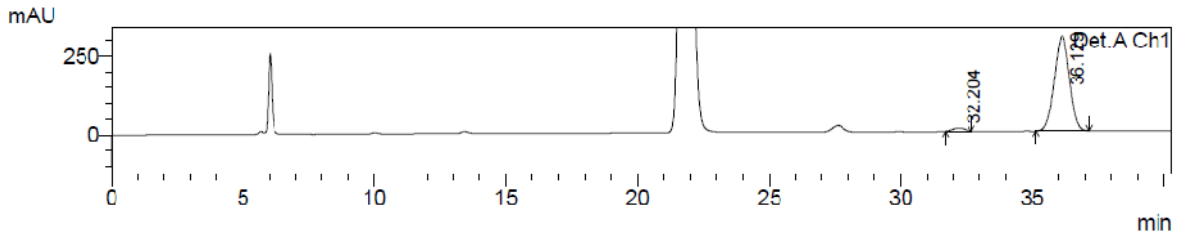
Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.023	2190304	60568	49.932	52.891
2	35.792	2196230	53948	50.068	47.109
Total		4386534	114516	100.000	100.000



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.431	5562444	174883	3.475	5.236
2	35.741	154513311	3164890	96.525	94.764
Total		160075755	3339773	100.000	100.000

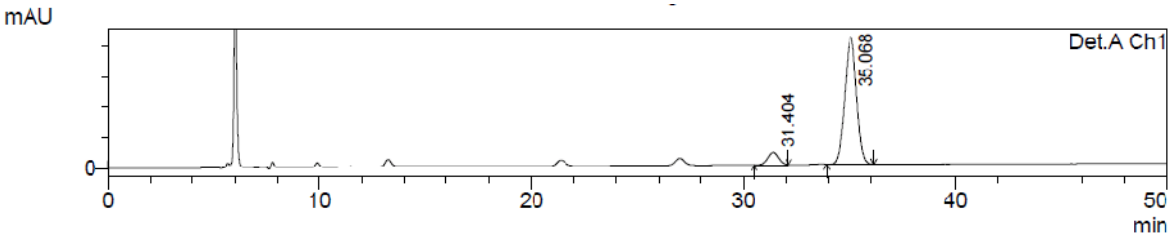
Bromolactonization with NBP (Table 3, entry 2)



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.204	303356	9928	2.415	3.157
2	36.129	12256482	304511	97.585	96.843
Total		12559838	314439	100.000	100.000

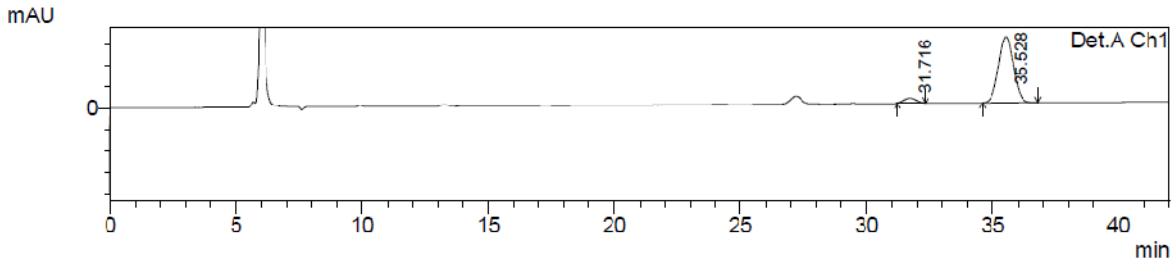
Bromolactonization with DBH (Table 3, entry 3)



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.404	689756	20094	7.801	8.817
2	35.068	8152338	207799	92.199	91.183
Total		8842094	227893	100.000	100.000

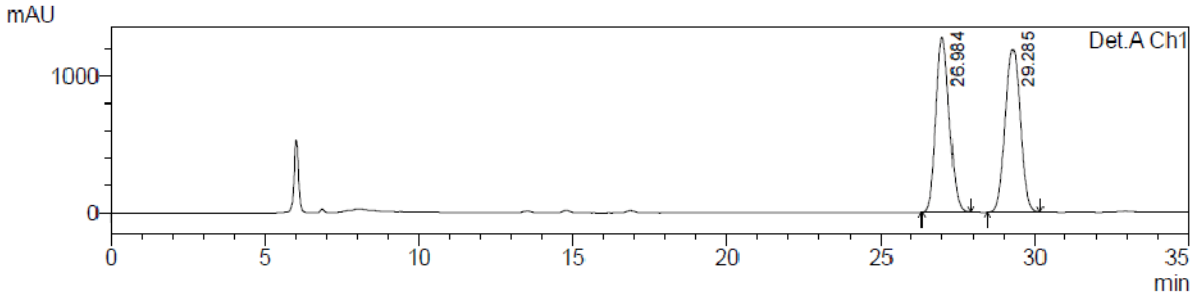
Bromolactonization with TABCO (Table 3, entry 4)



Detector A Ch1 214nm

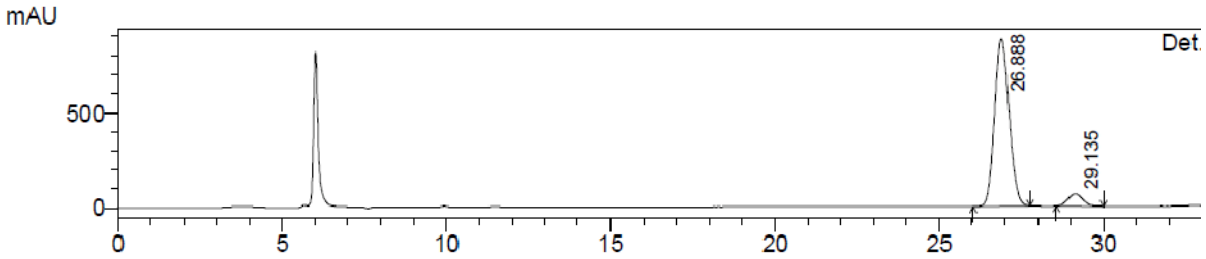
Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.716	363221	11206	5.502	6.764
2	35.528	6238251	154460	94.498	93.236
Total		6601471	165666	100.000	100.000

Bromolactonization of acid 2with TABCO (Table 3, entry 5)



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.984	39751616	1271491	49.873	51.686
2	29.285	39954651	1188519	50.127	48.314
Total		79706268	2460010	100.000	100.000

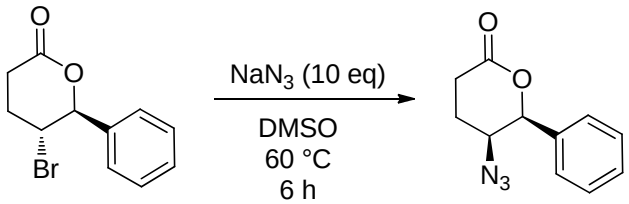


Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.888	26957774	877305	92.981	93.204
2	29.135	2034852	63972	7.019	6.796
Total		28992626	941277	100.000	100.000

E. Synthetic application of bromolactones 2 and 5a

Bromolactone **3** was prepared using the procedure described in our previous report.[2]

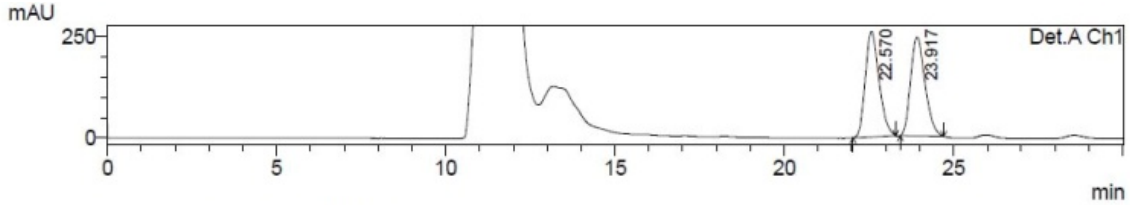


3 **10**

Synthesis of (5S,6S)-5-azido-6-phenyltetrahydro-2H-pyran-2-one (10)

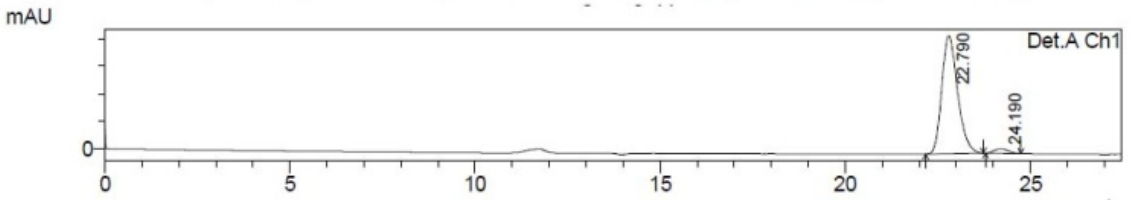
Bromolactone **3** (434 mg, 2 mmol, 1.0 eq) and NaN_3 (1.30 g, 20 mmol, 10.0 eq) were first evacuated *in vacuo* and flushed with N_2 for 3 cycles. DMSO (6 ml) was injected and the reaction mixture was stirred at 60 °C for 6 hours. Upon termination, the reaction mixture was cooled to r.t. before quenching with water (30 ml). The product was extracted with ethyl acetate (3 x 20 ml). The combined ethyl acetate layer was further washed with brine (50 ml), dried over anhydrous sodium sulfate, filtered and concentrated *in vacuo*. The product was purified over silica gel with hexane:ethyl acetate (1:1) as the eluent.

Pale yellow oil; Yield: 63%; $[\alpha]_D^{25}$ +36.0 (*c* 1.0, $CHCl_3$, 93% ee); IR (Neat): 3020, 2110, 1712, 1216 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.40-7.36 (m, 5H), 5.48 (d, *J* = 2.0 Hz, 1H), 4.09-4.06 (m, 1H), 2.84-2.61 (m, 2H), 2.37-2.15 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.3, 135.8, 128.7, 128.6, 126.0, 81.7, 59.0, 25.3, 24.8; HPLC (Daicel Chiralpak IC, *i*-PrOH (1% Ethanolamine)/*n*-hexane = 80/20, 0.3 mL/min, 214 nm) t_1 = 22.8 min (major), t_2 = 24.2 min (minor).



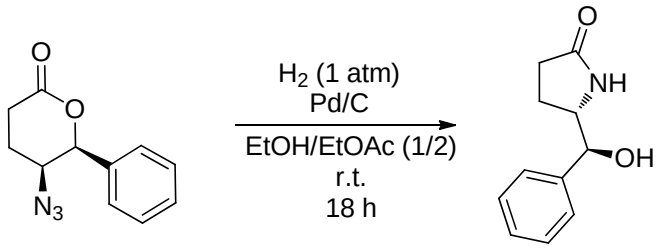
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.570	7311235	259486	50.450	51.656
2	23.917	7180931	242852	49.550	48.344
Total		14492166	502337	100.000	100.000



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.790	12774805	429456	96.705	96.283
2	24.190	435218	16580	3.295	3.717
Total		13210024	446036	100.000	100.000



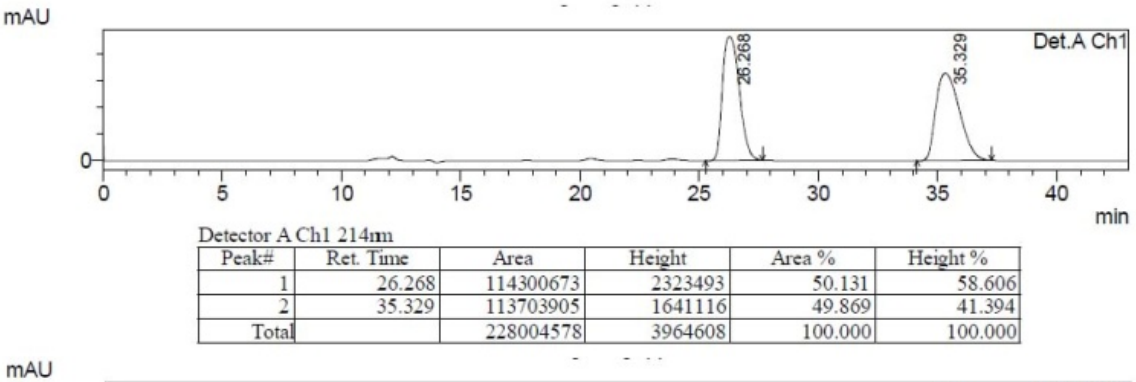
10 **11**

Synthesis of (S)-5-((S)-hydroxy(phenyl)methyl)pyrrolidin-2-one (11)[6]

The azide **10** (217 mg, 1 mmol) was dissolved in ethanol (2 ml) and ethyl acetate (4 ml). Pd/C (80 mg) was added before the attachment of a hydrogen balloon. The reaction mixture was stirred at r.t. for 18 h. The reacted mixture was filtered over celite with several washings of chloroform. The filtrate was dried over anhydrous sodium sulfate, filtered and concentrated *in vacuo*. The product was then purified over silica gel with MeOH:chloroform (1:20) and 1% ammonia solution as the eluent.

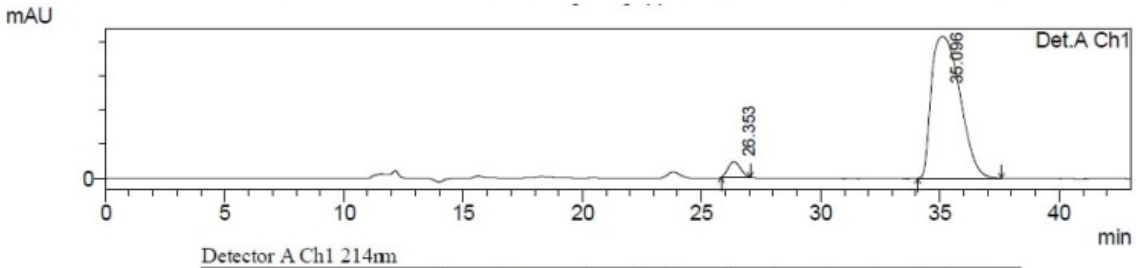
White solid, mp 136.6-138.3; Yield: 55%; $[\alpha]_D^{25}$ +27.0 (*c* 1.0, $CHCl_3$, 91% ee); IR (KBr): 2937, 1682, 1541, 1350, 1164 cm^{-1}; ^1H NMR (300 MHz, MeOD): δ 7.38-7.27 (m, 5H), 4.49 (d, *J* = 6.9 Hz, 1H), 3.90-3.30 (m, 1H), 2.23-2.03 (m, 2H), 1.93-1.77 (m, 2H); ^{13}C NMR (75 MHz, MeOD): δ 181.1, 142.3, 129.4, 128.9, 128.0, 78.0, 61.7, 31.0, 23.8; HRMS (ESI) calcd for $C_{11}H_{13}NNaO_2$ $[M]^+$: 214.0844; found: 214.0846. HPLC (Daicel Chiralpak IC, *i*-PrOH

(1% Ethanolamine)/*n*-hexane = 80/20, 0.3 mL/min, 214 nm) t_1 = 26.4 min (minor), t_2 = 35.1 min (major).



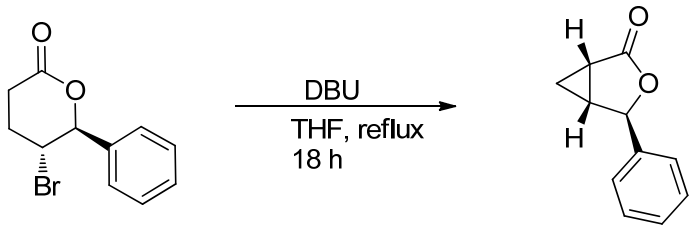
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.268	114300673	2323493	50.131	58.606
2	35.329	113703905	1641116	49.869	41.394
Total		228004578	3964608	100.000	100.000



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.353	8070361	226032	4.457	9.844
2	35.096	172983509	2070159	95.543	90.156
Total		181053871	2296191	100.000	100.000



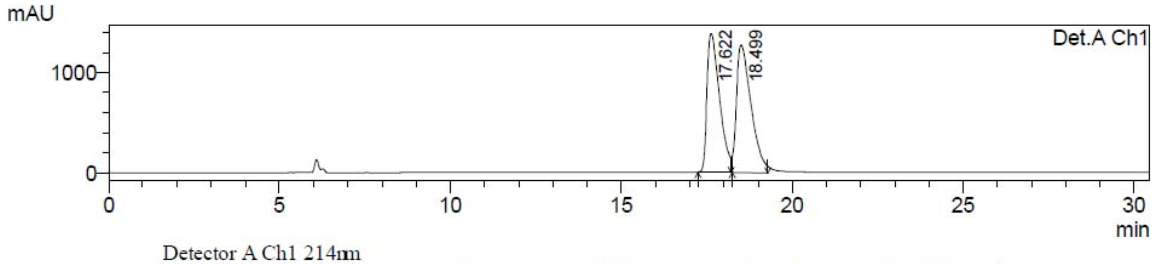
3 **12**

Synthesis of (1S,4R,5R)-4-phenyl-3-oxabicyclo[3.1.0]hexan-2-one (12) [7]

The bromolactone **3** (147 mg, 0.67 mmol, 1.0 equiv) was evacuated *in vacuo* and flushed with N_2 for 3 cycles. THF (6 ml) was injected followed by DBU (110 µL, 0.74 mmol, 1.1 equiv). The reaction mixture was then refluxed for 18 h. The reacted mixture was then quenched with water (30 ml) and extracted with ethyl acetate (3 x 15 ml). The ethyl acetate layers were combined and was dried over anhydrous sodium sulfate, filtered and concentrated *in vacuo*. The product was then purified over silica gel with EtOAc:Hexane (1:3) as the eluent.

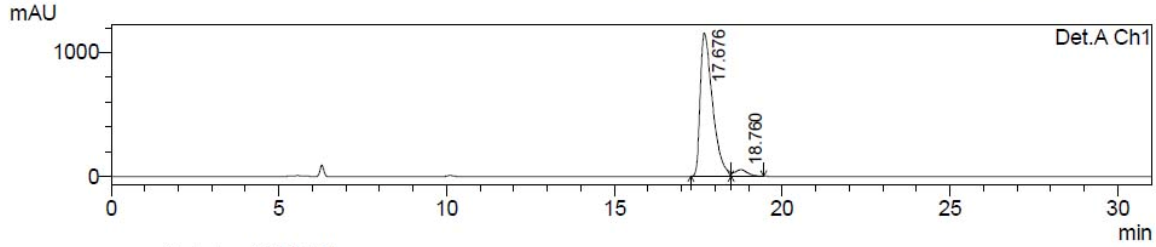
Colorless oil; Yield: 42%; $[\alpha]_D^{25}$ +36.0 (*c* 1.0, $CHCl_3$, 91% ee); IR (Neat): 3020, 1767, 1641, 1216 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.39-7.33 (m, 5H), 5.31 (s, 1H), 2.28-2.19 (m, 2H),

1.34 (ddd, J = 8.8, 7.6, 5 Hz, 1H), 1.07-1.04 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.9, 139.6, 128.8, 128.7, 125.5, 81.5, 24.6, 17.7, 12.8; DEPT 135 (125 MHz, $CDCl_3$) 128.9 (CH), 128.8 (CH), 125.6 (CH), 81.6 (CH), 24.7 (CH), 17.8 (CH), 12.9 (CH_2); HRMS (ESI) calcd for $C_{11}H_{10}NaO_2$ $[M]^+$: 197.0578; found: 197.0573. HPLC (Daicel Chiralpak IB, *i*-PrOH/*n*-hexane = 15/85, 0.6 mL/min, 214 nm) t_1 = 17.6 min (major), t_2 = 18.7 min (minor).



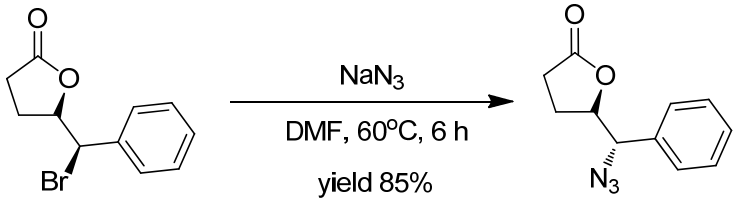
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.622	35235891	1380707	48.528	52.218
2	18.499	37373991	1263434	51.472	47.782
Total		72609883	2644141	100.000	100.000



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.676	29402239	1154421	95.270	95.506
2	18.760	1459749	54316	4.730	4.494
Total		30861988	1208737	100.000	100.000

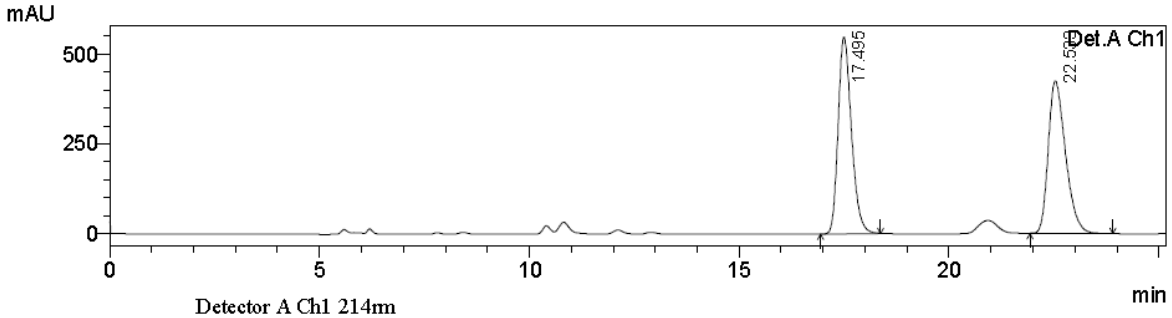


93% ee — **5a** — 93% ee — **13**

Synthesis of 5-azido(phenyl)methyldihydro-furan-2(3H)-one (13)

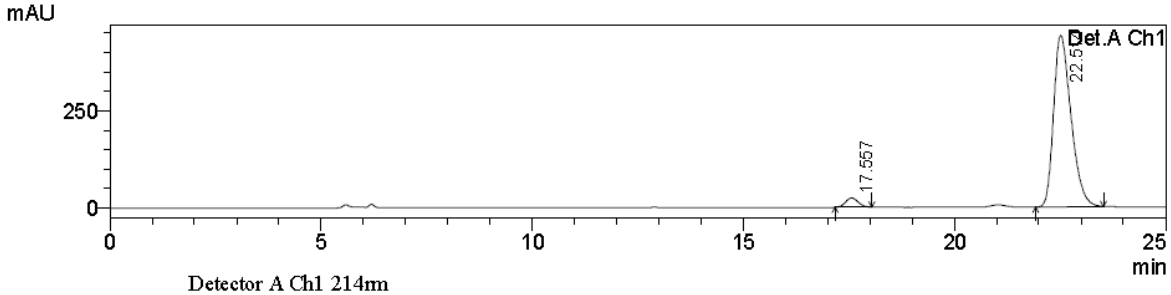
5a (213 mg, 0.84 mmol, 1.0 eq) and sodium azide (82 mg, 1.26 mmol, 1.5 eq) were dissolved in DMF (15 ml) and the reaction mixture was stirred for 6 hours at 60 °C. Upon completion, H_2O (50 ml) was added before extracting with diethyl ether (3 x 50 ml). The combined organic layer were washed with brine (2 x 100 ml), dried over sodium sulfate and the solvent was removed *in vacuo*. The product was purified using flash column chromatography over silica gel with EtOAc:Hexane (1:3) as the eluent. Light brown oil; $[\alpha]_D^{25}$ -146.7 (*c* 1.0, $CHCl_3$,

93% ee); IR (Neat): 3020, 2111, 1778, 1642, 1216 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.44-7.35(m, 5H), 4.90 (d, *J* = 4.4 Hz, 1H), 4.70-4.66 (m, 1H), 2.58-2.42 (m, 2H), 2.23-2.16 (m, 1H), 2.13-2.05 (m, 1H) ; ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.4, 134.5, 129.1, 128.9, 127.2, 81.4, 67.5, 28.1, 22.3; HRMS (EI) calcd for $C_{11}H_{17}BrO_2$ $[M]^+$: 217.0851; found: 217.0847; HPLC (Daicel Chiralpak IB, *i*-PrOH/*n*-hexane = 25/75, 0.6 mL/min, 214 nm) t_1= 17.6 min (minor), t_2 = 22.5 min (major).



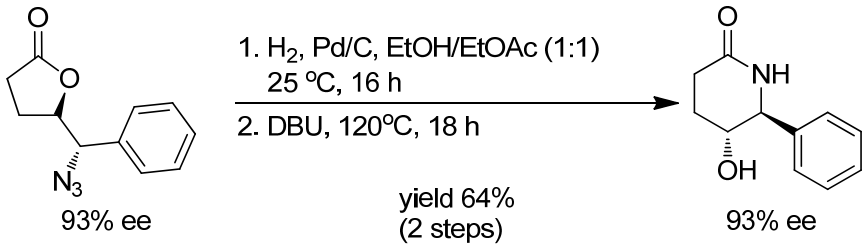
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.495	12101669	547571	49.878	56.322
2	22.533	12160992	424636	50.122	43.678
Total		24262661	972207	100.000	100.000



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.557	500124	24094	3.811	5.181
2	22.514	12621663	440977	96.189	94.819
Total		13121787	465071	100.000	100.000

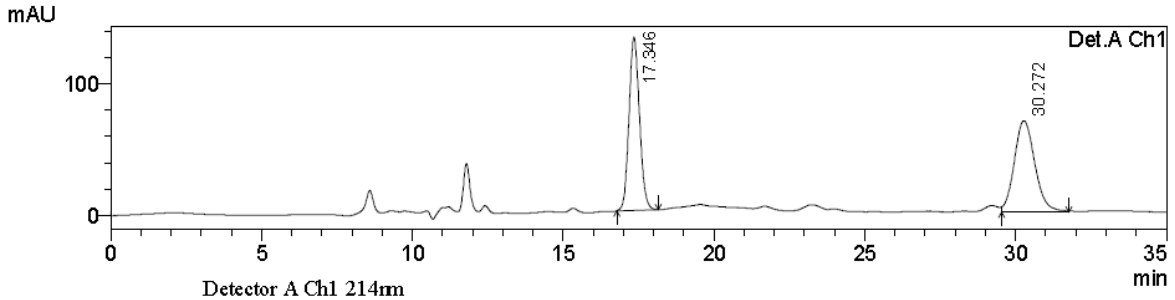


93% ee — **12** — 93% ee — **14**

Synthesis of 5-hydroxy-6-phenylpiperidin-2-one (14)[8]

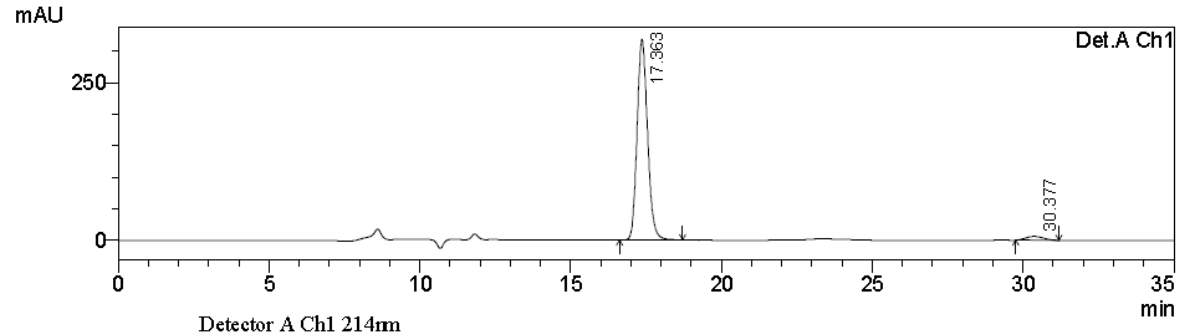
To a solution of azide **12** (154 mg, 0.71 mmol) in 1:1 mixture of EtOH/EtOAc (16 ml), Pd/C (100 mg, 10% on activated carbon) was added. The flask was evacuated and charged with hydrogen, and the mixture was stirred at 25 °C overnight. The mixture was filtered through a pad of Celite, and washed with copious amount of CH_2Cl_2. The filtrate was concentrated under reduced pressure to deliver 5-amino(phenyl)methyl-dihydrofuran-2(3H)-one as an

orange oil. The crude product was then dissolved in DBU (1.5 ml) and heated at 120 °C for 18 hours. The mixture was allowed to cool to room temperature before subjecting to silica gel column chromatography with MeOH:CH_2Cl_2 (2:98) to deliver product **13** as a white solid. White solid, mp 176.5-177.8; $[\alpha]_D^{25}$ -30.1 (*c* 1.0, MeOH, 93% ee); ^{1}H NMR (300 MHz, MeOD): δ 7.41-7.27(m, 5H), 4.46 (d, *J* = 4.8 Hz, 1H), 3.92-3.88 (m, 1H), 2.63-2.52 (m, 1H), 2.47-2.37 (m, 1H), 1.93-1.76 (m, 2H) ; ^{13}C NMR (125 MHz, MeOD): δ 174.8, 142.0, 129.7, 128.9, 128.0, 70.4, 64.5, 28.3, 25.8; HRMS (ESI) calcd for $C_{11}H_{13}NO_2$ $[M - H]^-$: 190.0868; found: 190.0870; HPLC (Daicel Chiralpak IC, *i*-PrOH/*n*-hexane = 50/50, 0.6 mL/min, 214 nm) t_1= 17.4min (major), t_2 = 30.4 min (minor).



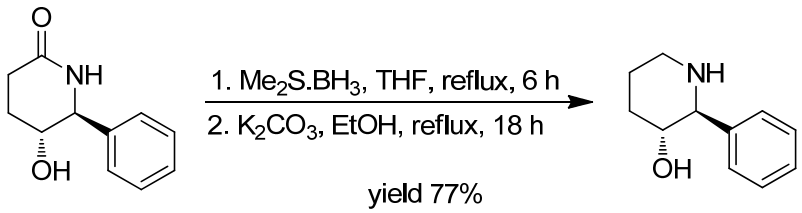
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.346	3187690	131667	49.274	65.513
2	30.272	3281575	69311	50.726	34.487
Total		6469265	200978	100.000	100.000



Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.363	7668551	317767	96.909	98.176
2	30.377	244623	5902	3.091	1.824
Total		7913174	323670	100.000	100.000



13 **15**

Synthesis of 2-phenyl-3-piperidinol (15)

To a solution of **13** (100 mg, 0.52 mmol, 1.0 eq) in THF (3 mL) was added borane-dimethyl sulfide complex (1.30 mL, 2.6 mmol, 2 M in Et_2O, 5.0 eq) under nitrogen atmosphere. The

reaction mixture was heated to reflux for 6 h. After the reaction was cooled to room temperature, MeOH (20 mL) was added cautiously to quench the excess borane-dimethyl sulfide complex. The solvent was removed *in vacuo*. The residue together with powdered K_2CO_3 (215 mg, 1.56 mmol, 3.0 eq) were dissolved in EtOH (6 mL) and. The resulting mixture was heated to reflux for 18 h, cooled, and concentrated *in vacuo*. The residue was then dissolved in CH_2Cl_2 (5 mL) and H_2O (15 mL). The layers were separated and further extraction with CH_2Cl_2 (2 x 5 mL) were performed. The combined organic layer was then washed with brine (15 mL), dried over Na_2SO_4, filtered and concentrated *in vacuo* to yield product **14** was a white solid. White solid, mp 154.4-155.1; $[\alpha]_D^{25}$ -21.4 (*c* 1.0, MeOH);^{1}H NMR (300 MHz, MeOD): δ 7.42-7.26 (m, 5H), 3.63-3.58 (m, 1H), 3.32-3.31 (d, 2H), 3.04-3.01 (d, 1H), 2.68-2.63 (m, 1H), 2.16-2.14 (d, 1H), 1.81-1.79 (d, 1H), 1.75-1.67 (m, 1H), 1.52-1.44 (m, 1H); ^{13}C NMR (125 MHz, MeOD): δ 142.9, 129.4, 129.3, 128.6, 72.7, 70.0, 35.4, 26.5; HRMS (ESI) calcd for $C_{11}H_{13}NO_2$ $[M + H]^+$: 178.1232; found: 178.1223.

F. References

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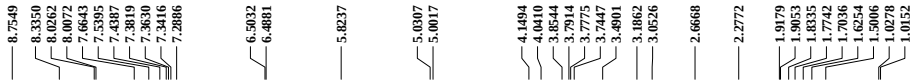

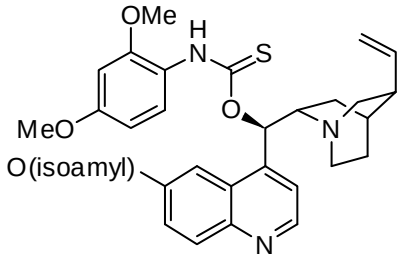

6a

*** Current Data Parameters ***

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PROCNO	:	1

*** Acquisition Parameters ***

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PULPROG	:	zg30
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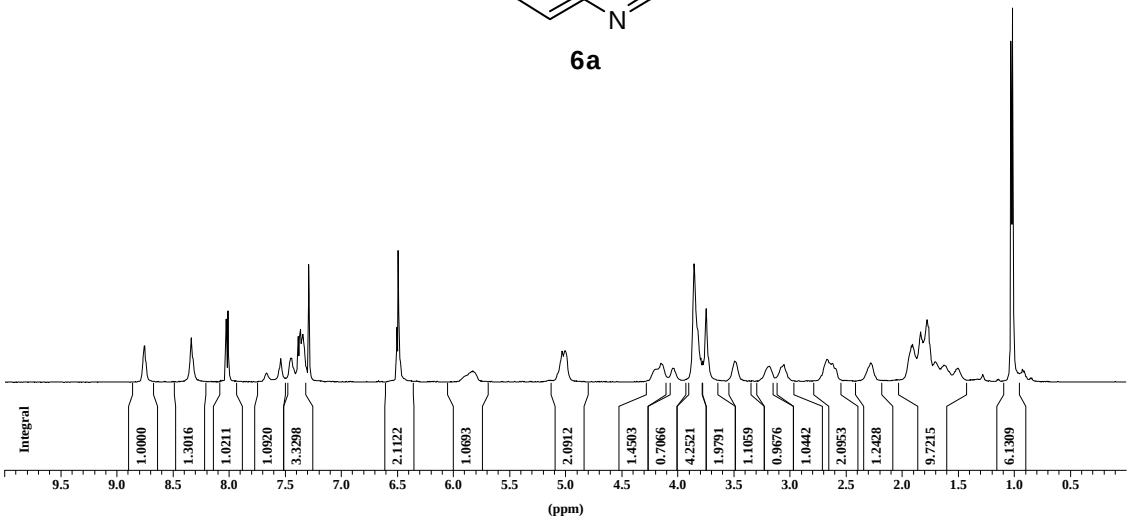
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768
TE	:	297.9 K

*** Processing Parameters ***

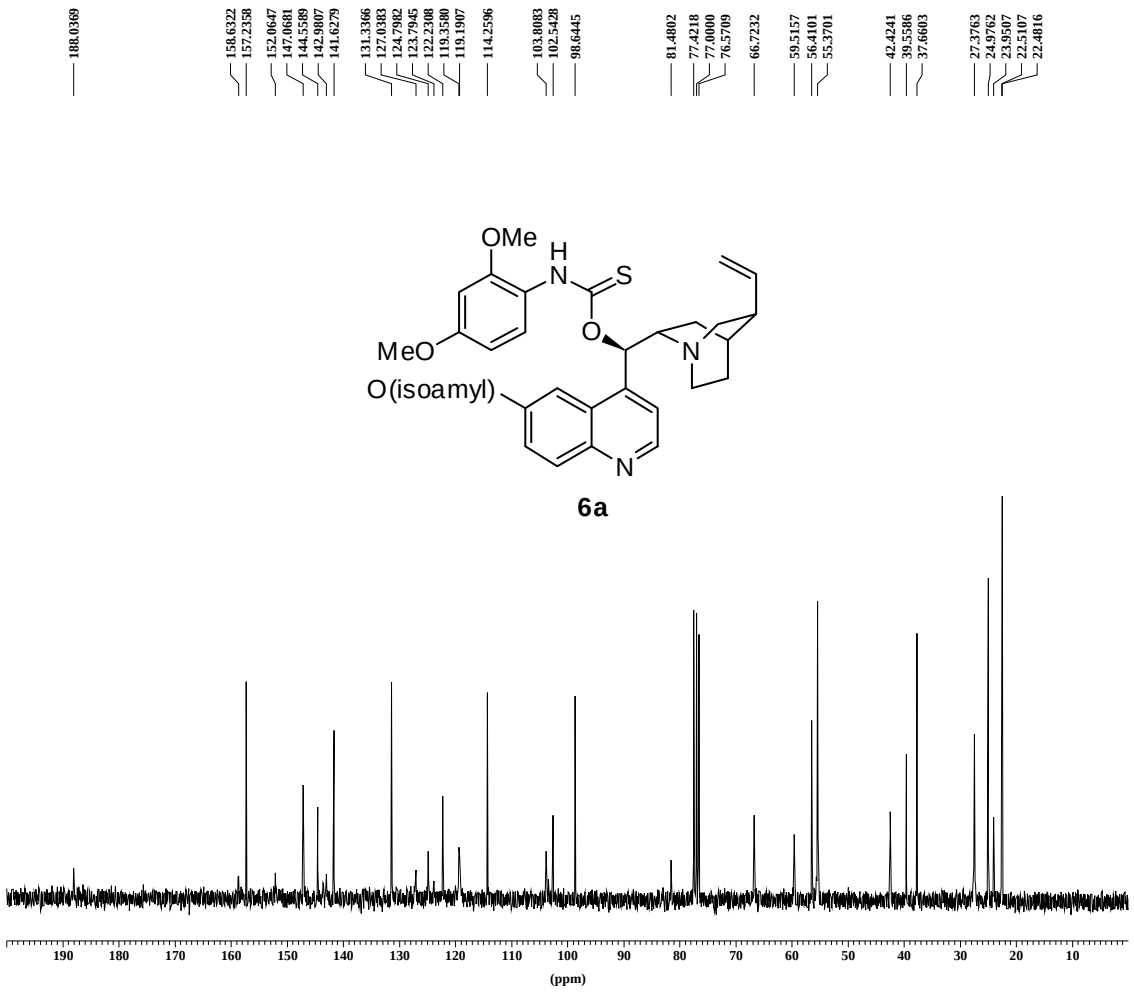
LB	:	0.30 Hz
SF	:	500.1300000 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off



13C Standard AC300 ck-v-157 cat



***** Current Data Parameters *****

NAME	:	fe11ck
EXPNO	:	3
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	207
NUCLEUS	:	off
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm
TD	:	32768
TE	:	297.1 K

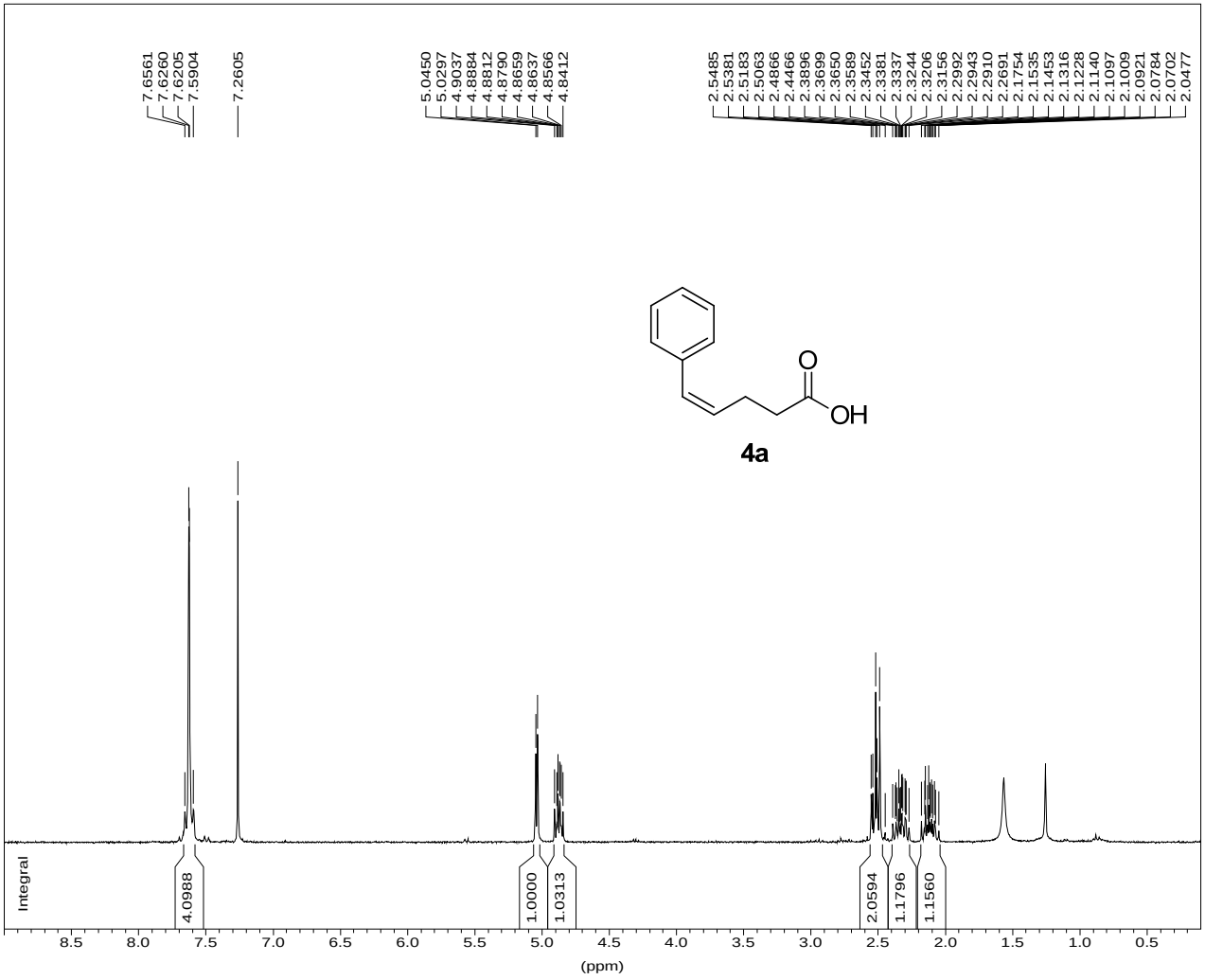
***** Processing Parameters *****

LB	:	1.00 Hz
SF	:	75.4677620 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

1H normal range AC300 cc-112-B



*** Current Data Parameters ***

NAME	:	ja05lcc
EXPNO	:	4
PROCNO	:	1

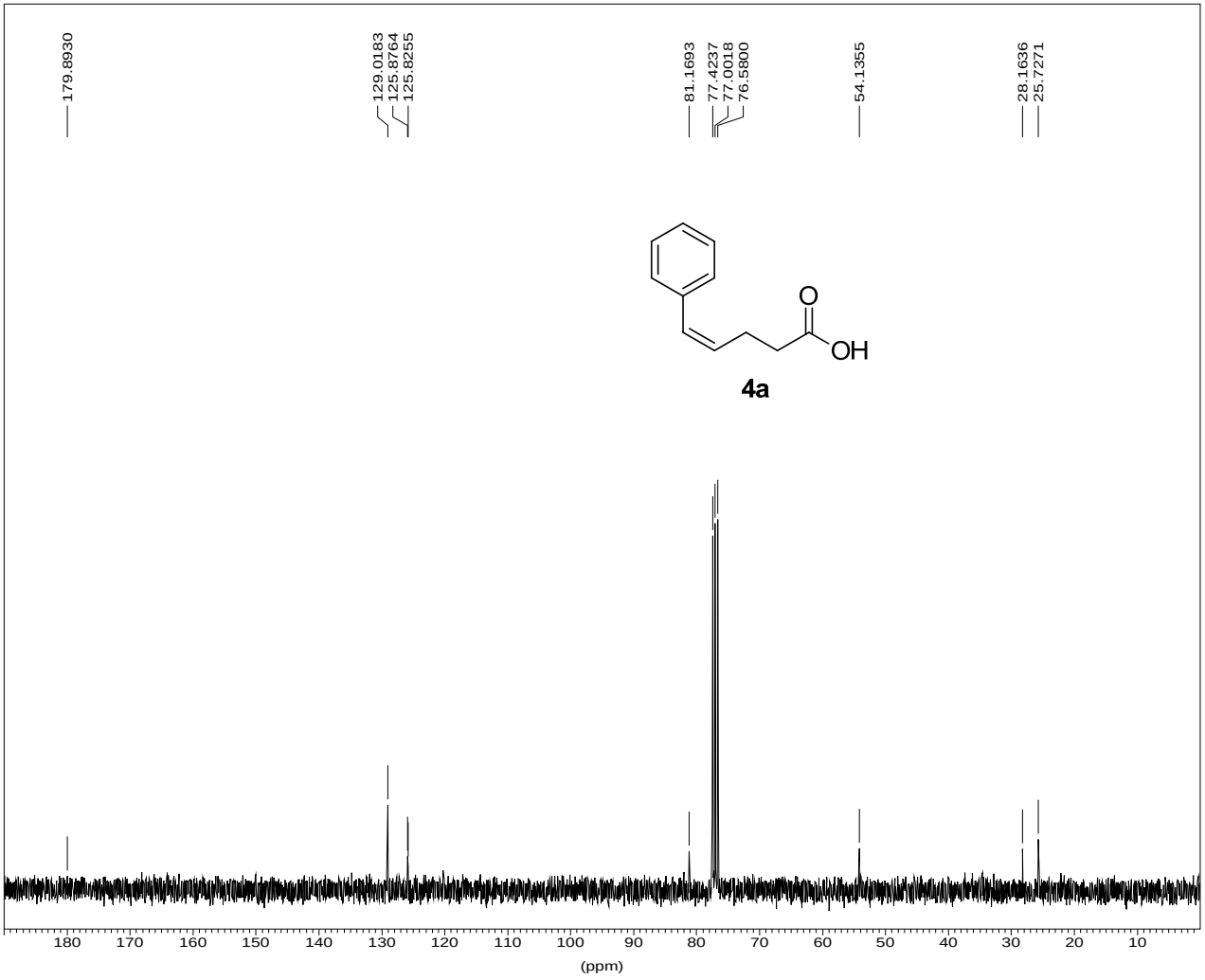
*** Acquisition Parameters ***

BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm

*** Processing Parameters ***

LB	:	0.30 Hz
PHC0	:	33.705 degree
PHC1	:	0.332 degree

13C Standard AC300 cc-112-B



*** Current Data Parameters ***

NAME	:	ja05lcc
EXPNO	:	5
PROCNO	:	1

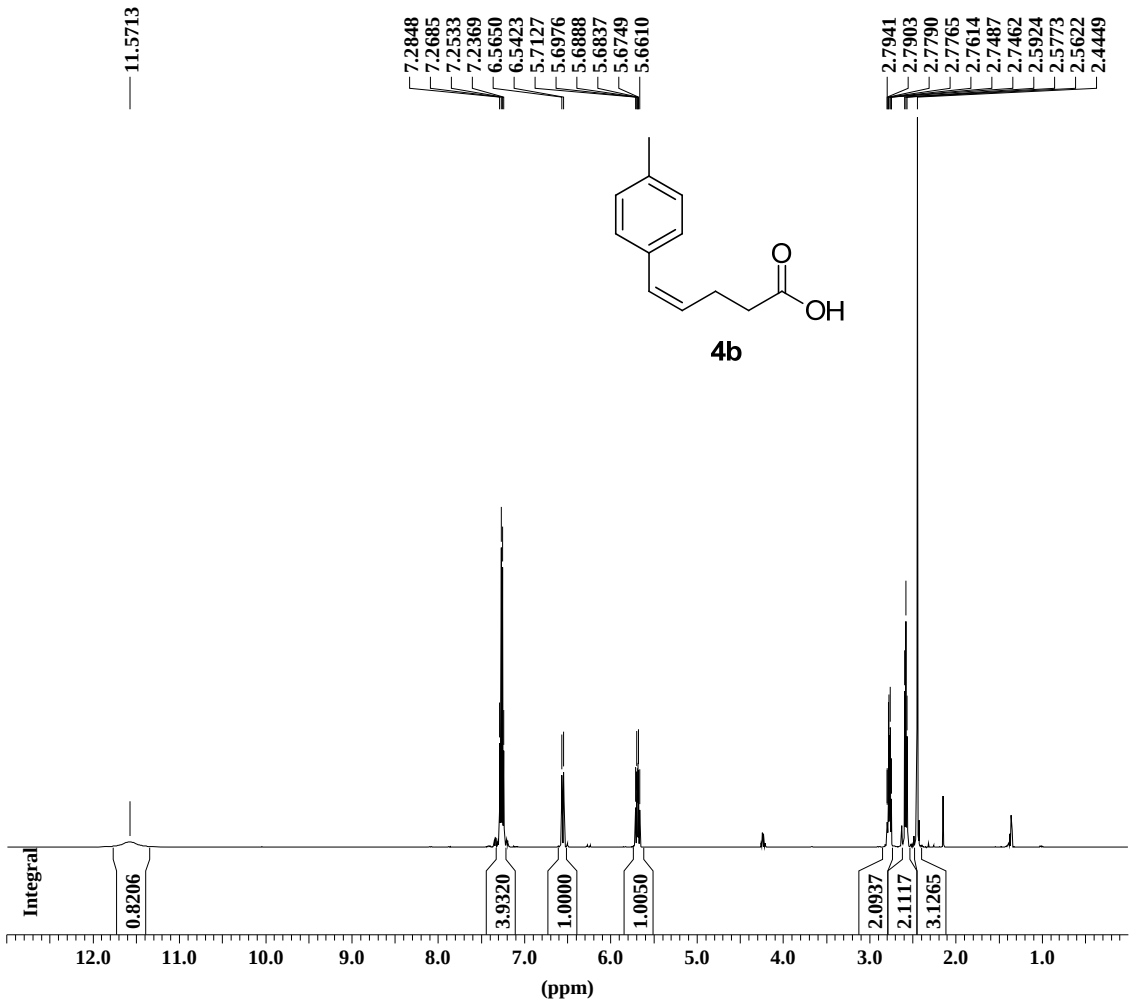
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	202
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	133.257 degree
PHC1	:	7.962 degree

1H AMX500 cc-i-33a



*** Current Data Parameters ***

NAME	:	ck0820
EXPNO	:	1
PROCNO	:	1

*** Acquisition Parameters ***

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	3088.51 Hz
PULPROG	:	zg30
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768
TE	:	300.0 K

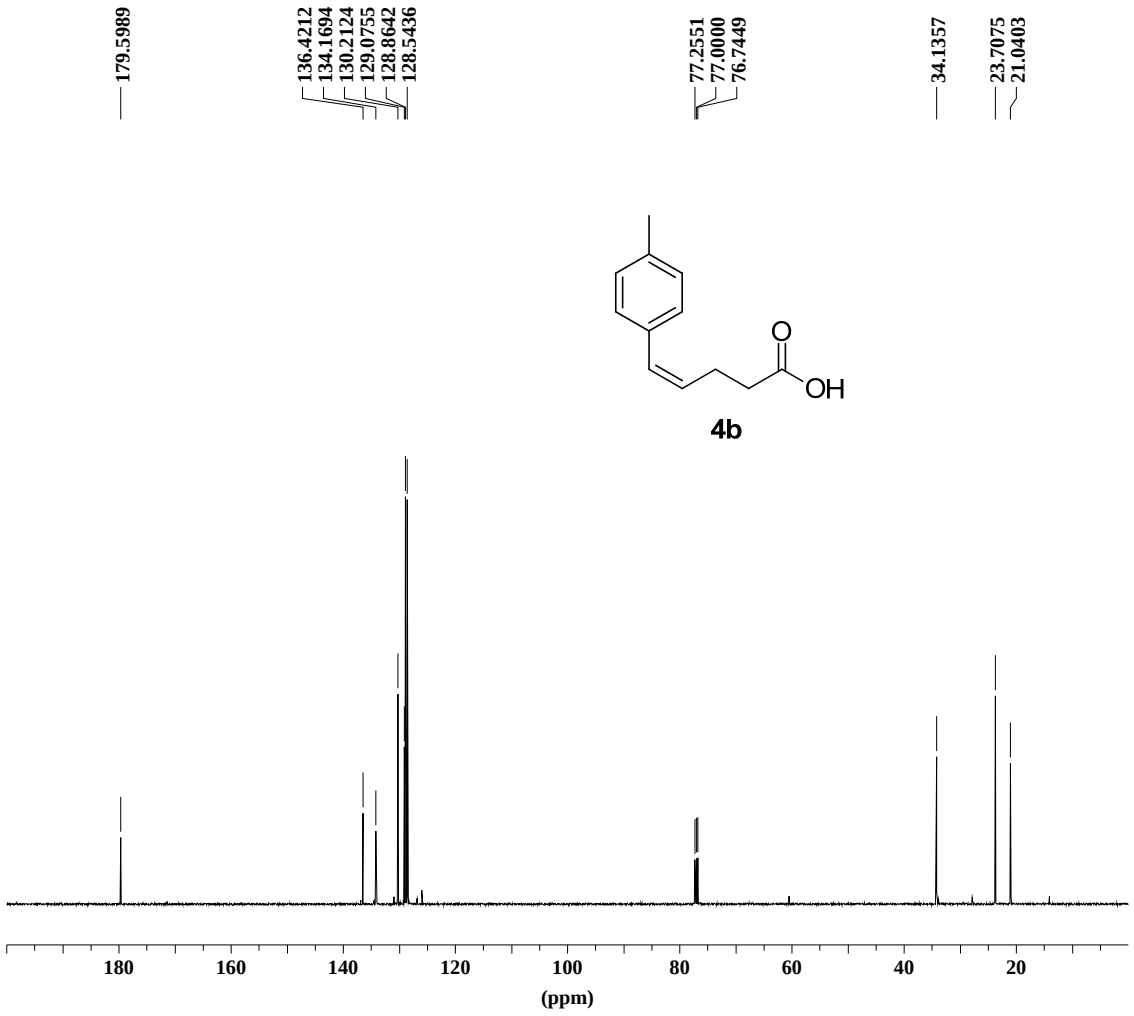
*** Processing Parameters ***

LB	:	0.30 Hz
SF	:	500.1300000 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C AMX500 cc-i-33a



*** Current Data Parameters ***

NAME	:	ck0820
EXPNO	:	2
PROCNO	:	1

*** Acquisition Parameters ***

LOCNUC	:	2H
NS	:	101
NUCLEUS	:	off
O1	:	13204.57 Hz
PULPROG	:	zgpg30
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm
TD	:	65536
TE	:	300.0 K

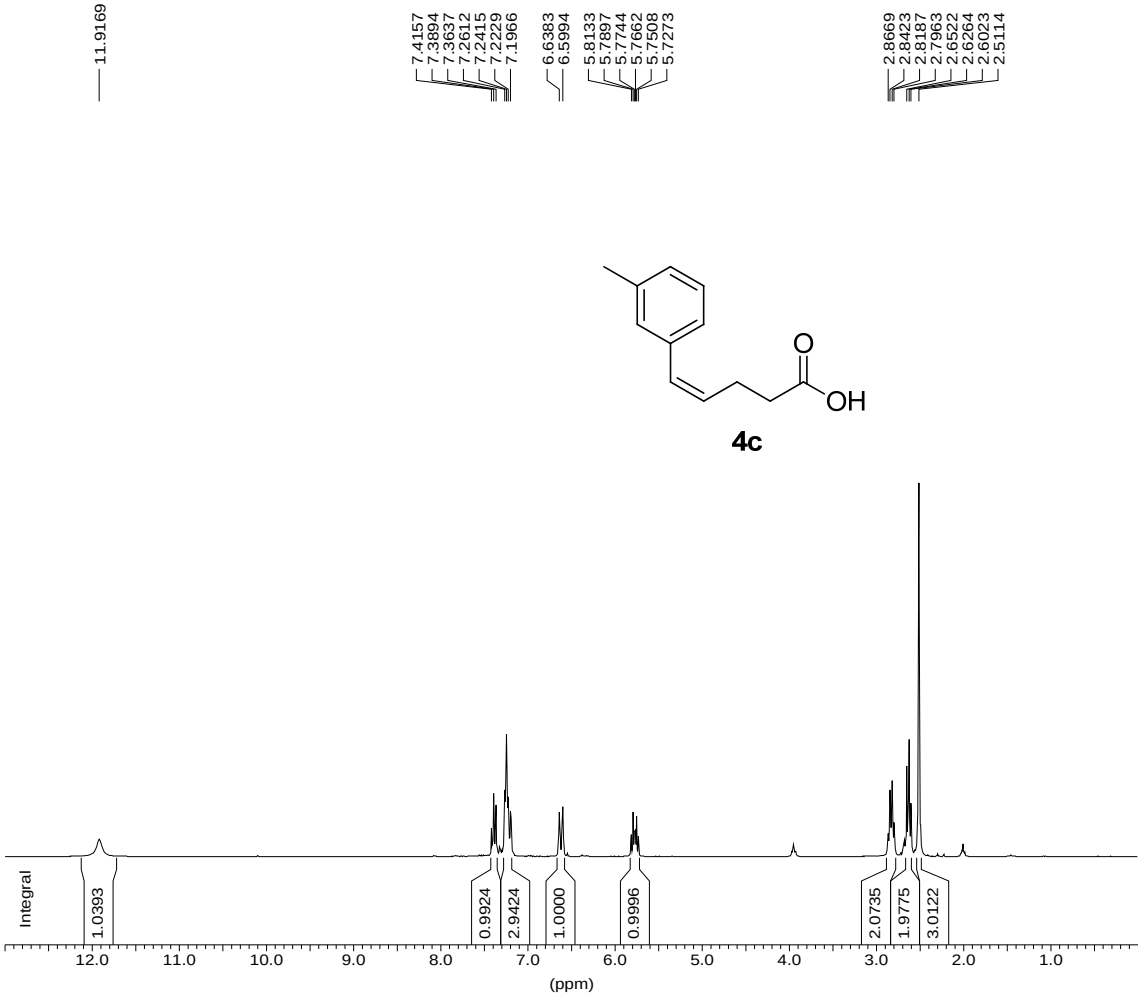
*** Processing Parameters ***

LB	:	1.00 Hz
SF	:	125.7578120 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

1H normal range AC300 cc-i-083-B



*** Current Data Parameters ***

NAME	:	nv01lcc
EXPNO	:	5
PROCNO	:	1

*** Acquisition Parameters ***

NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768

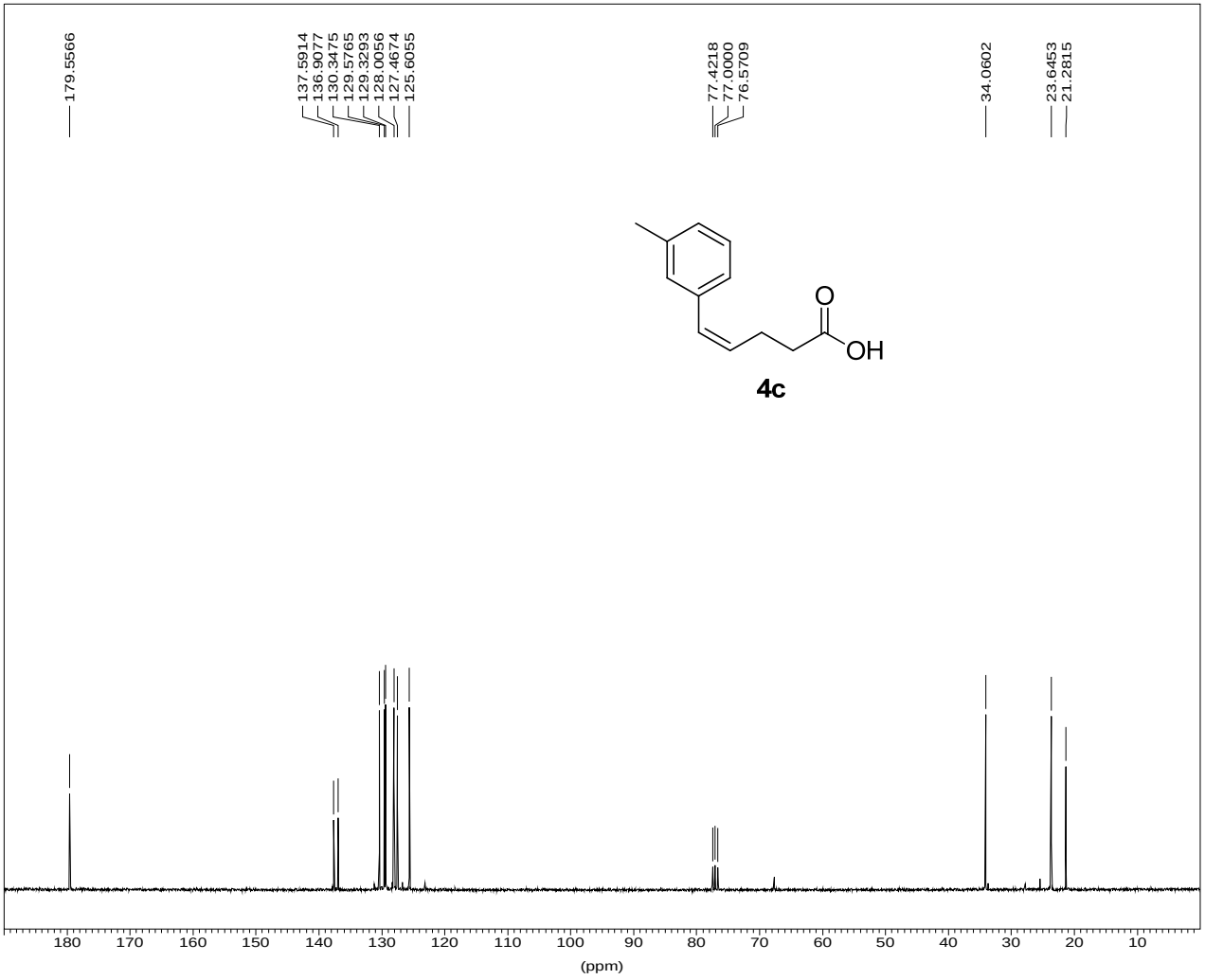
*** Processing Parameters ***

LB	:	0.30 Hz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C Standard AC300 cc-i-083-B



*** Current Data Parameters ***

NAME	:	nv01lcc
EXPNO	:	6
PROCNO	:	1

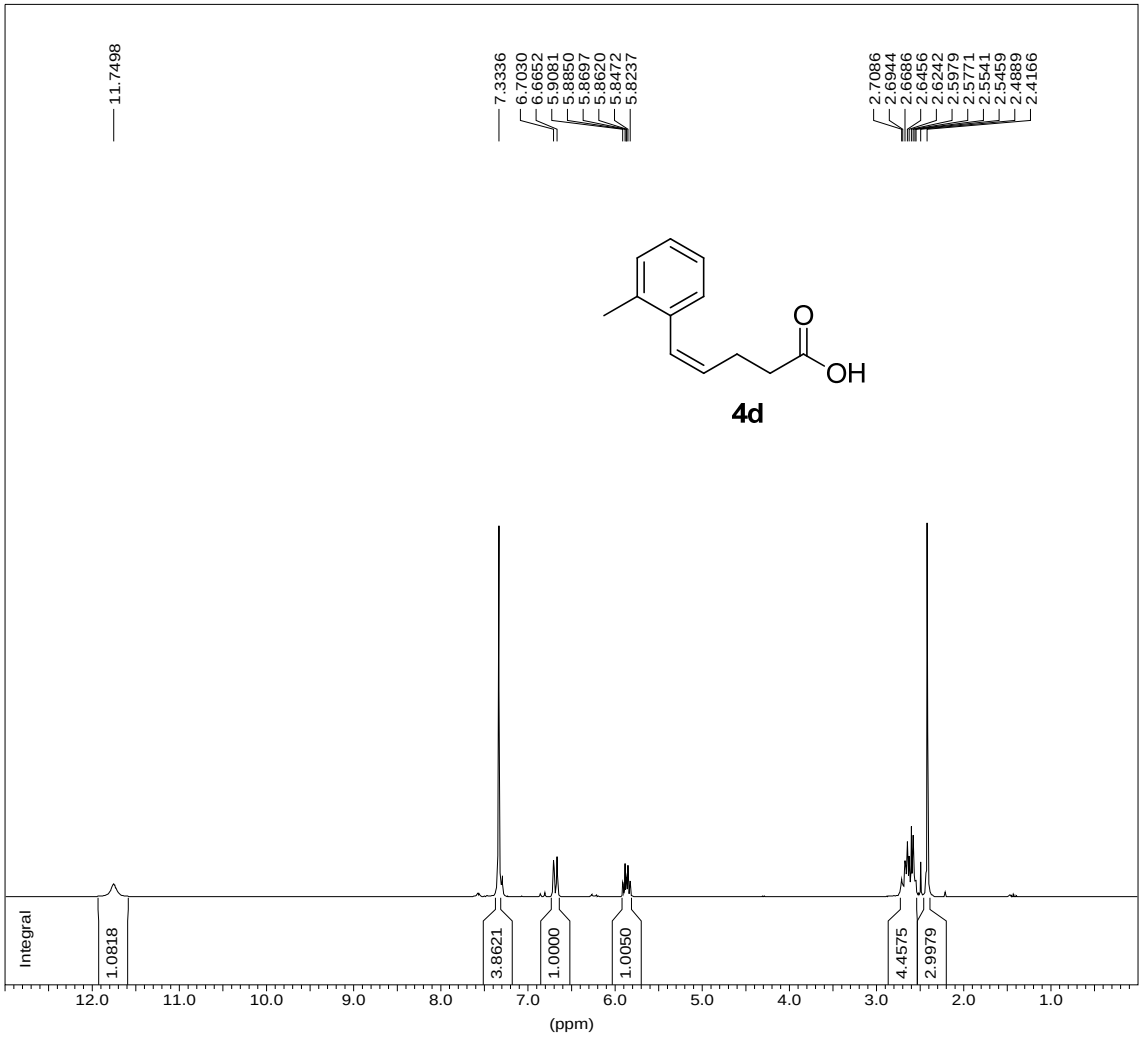
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	172.629 degree
PHC1	:	-3.689 degree

1H normal range AC300 cc-i-076-A



*** Current Data Parameters ***

NAME	:	oc21lcc
EXPNO	:	4
PROCNO	:	1

*** Acquisition Parameters ***

NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768

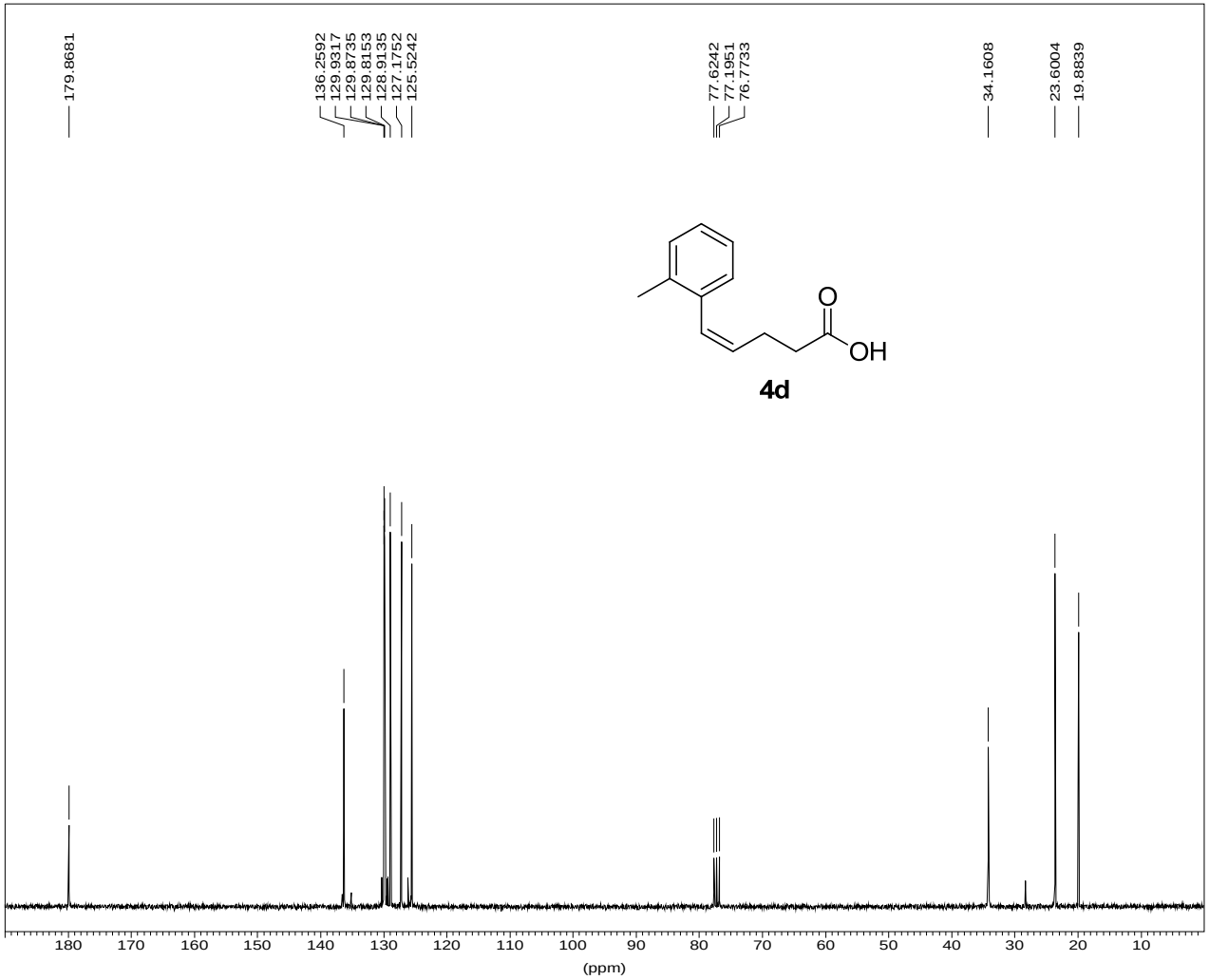
*** Processing Parameters ***

LB	:	0.30 Hz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C Standard AC300 cc-i-076-A



*** Current Data Parameters ***

NAME	:	oc21lcc
EXPNO	:	5
PROCNO	:	1

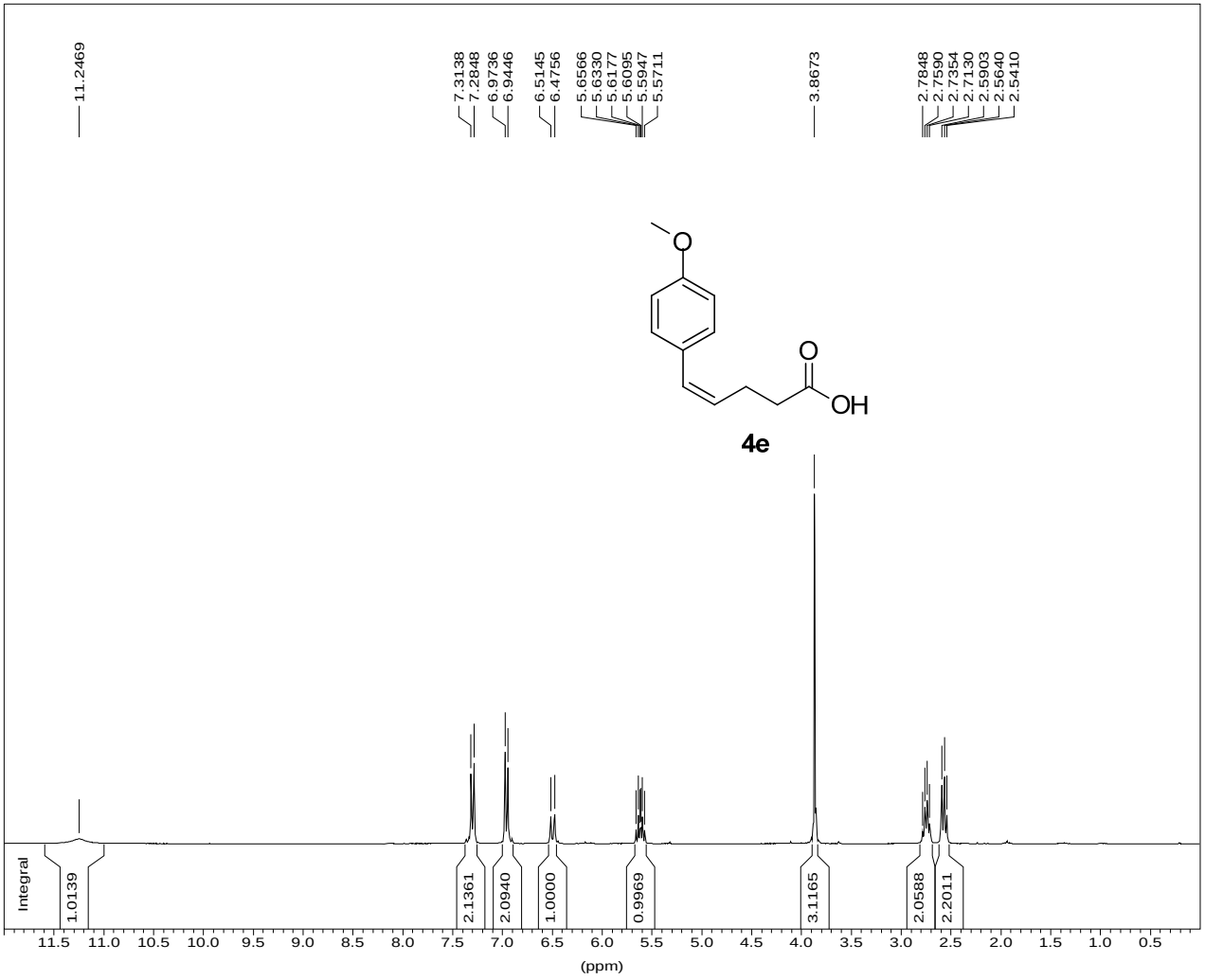
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	111
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	172.766 degree
PHC1	:	1.067 degree

1H normal range AC300 cc-i-103-C



*** Current Data Parameters ***

NAME	:	de02lcc
EXPNO	:	5
PROCNO	:	1

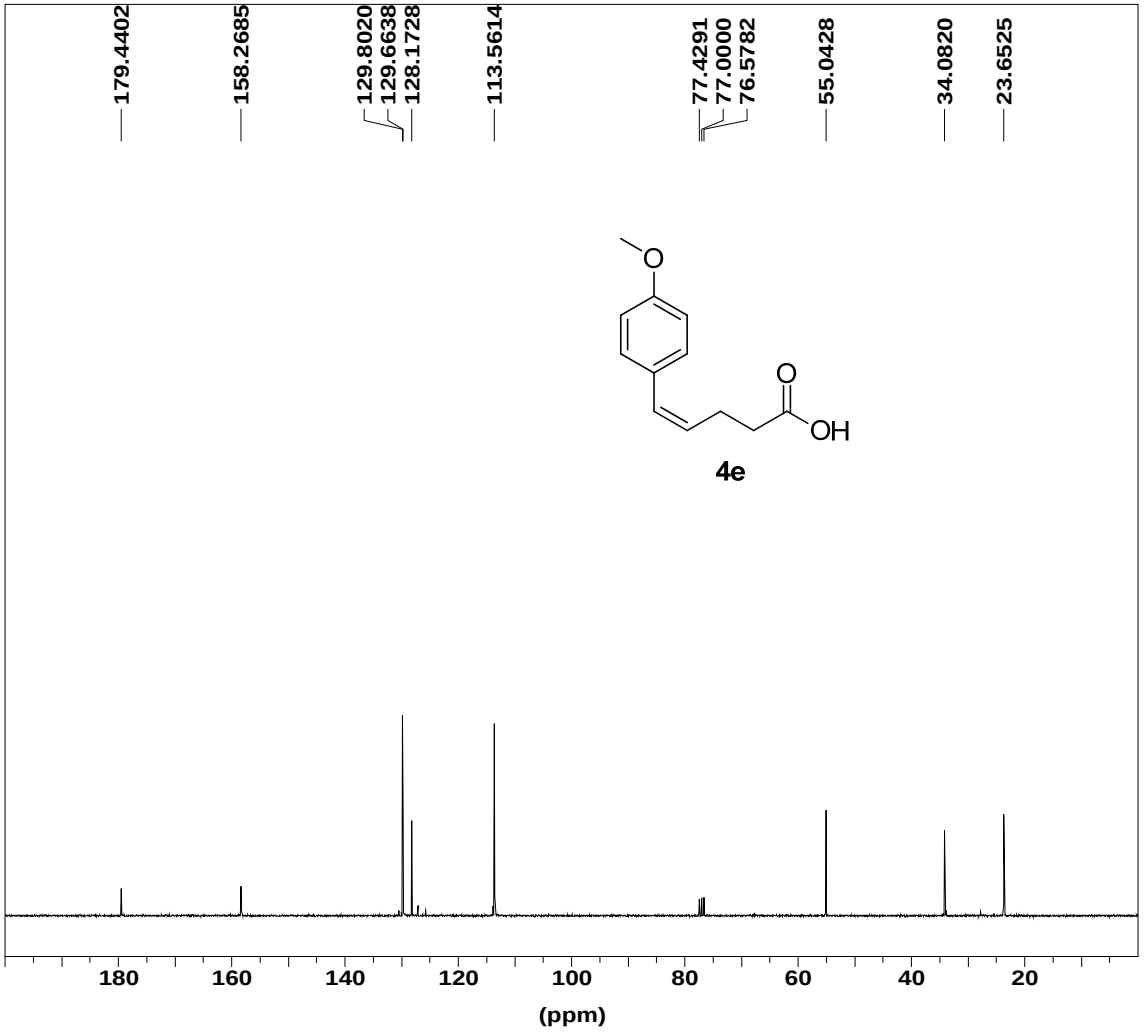
*** Acquisition Parameters ***

BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm

*** Processing Parameters ***

LB	:	0.30 Hz
PHC0	:	126.073 degree
PHC1	:	-0.278 degree

13C Standard AC300 cc-i-103-C



*** Current Data Parameters ***

NAME	:	de02lcc
EXPNO	:	6
PROCNO	:	1

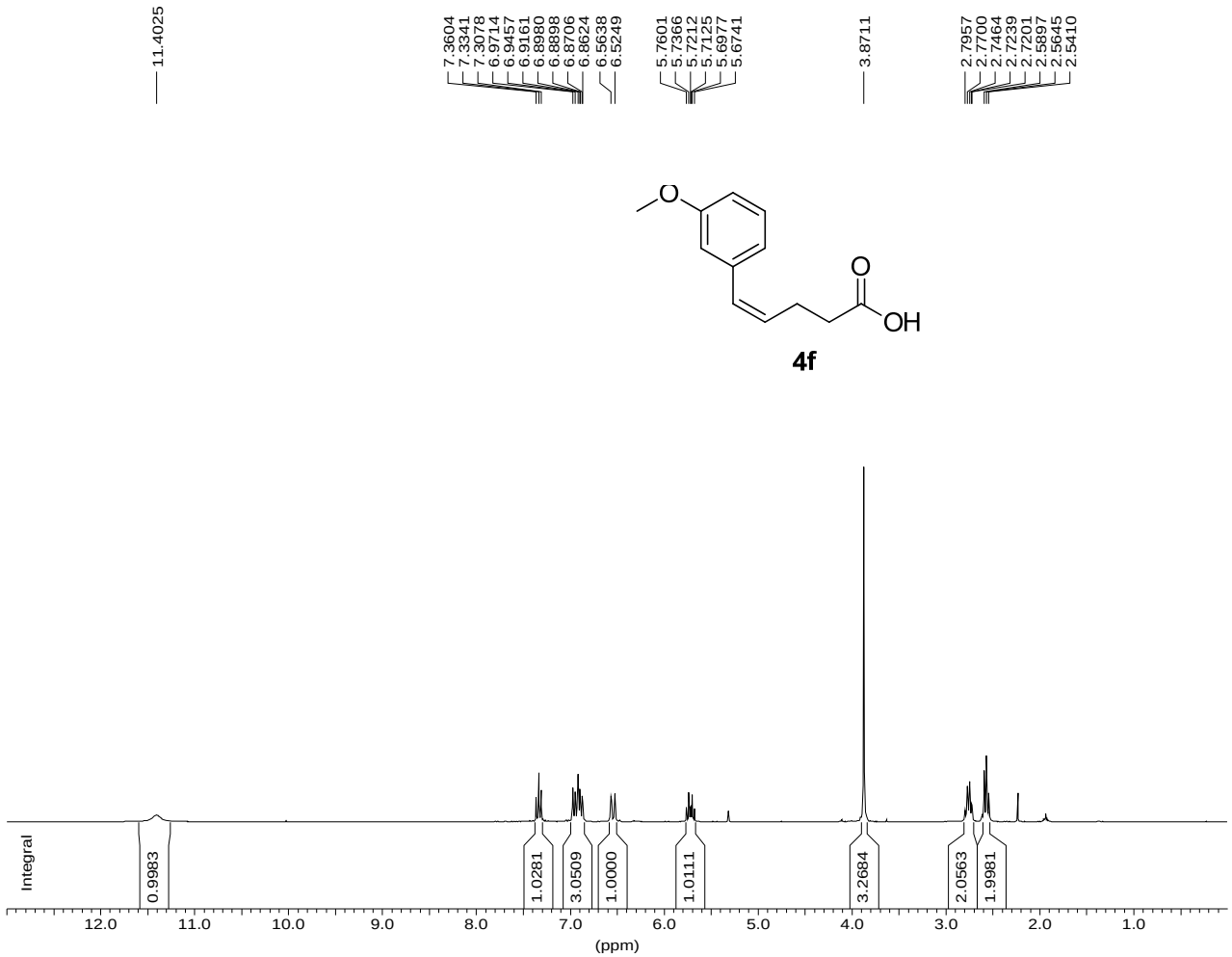
*** Acquisition Parameters ***

NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm
TD	:	32768

*** Processing Parameters ***

LB	:	1.00 Hz

1H normal range AC300 cc-090-B



*** Current Data Parameters ***

NAME	:	de05lcc
EXPNO	:	8
PROCNO	:	1

*** Acquisition Parameters ***

NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768

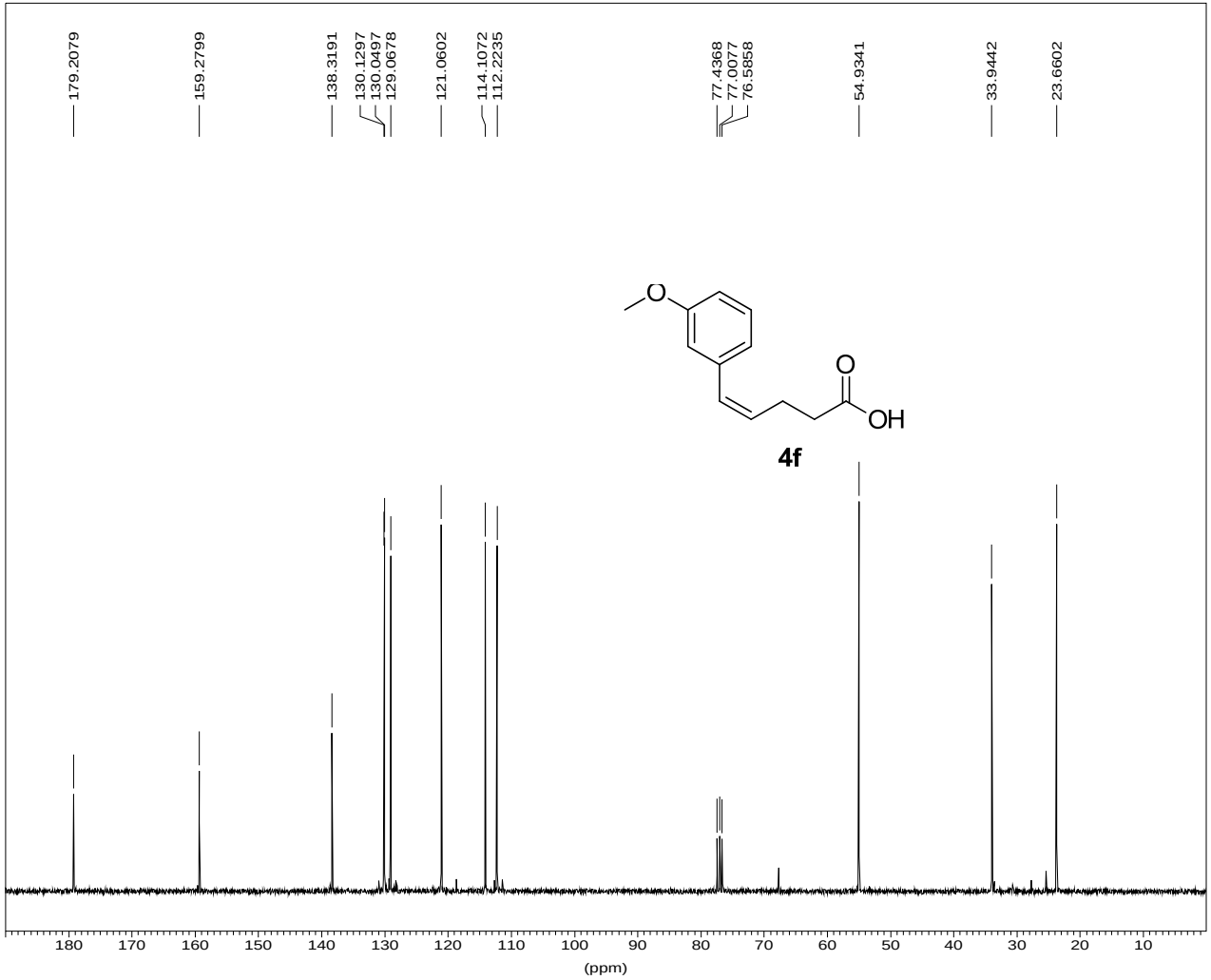
*** Processing Parameters ***

LB	:	0.30 Hz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C Standard AC300 cc-i-090-B



*** Current Data Parameters ***

NAME	:	de05lcc
EXPNO	:	9
PROCNO	:	1

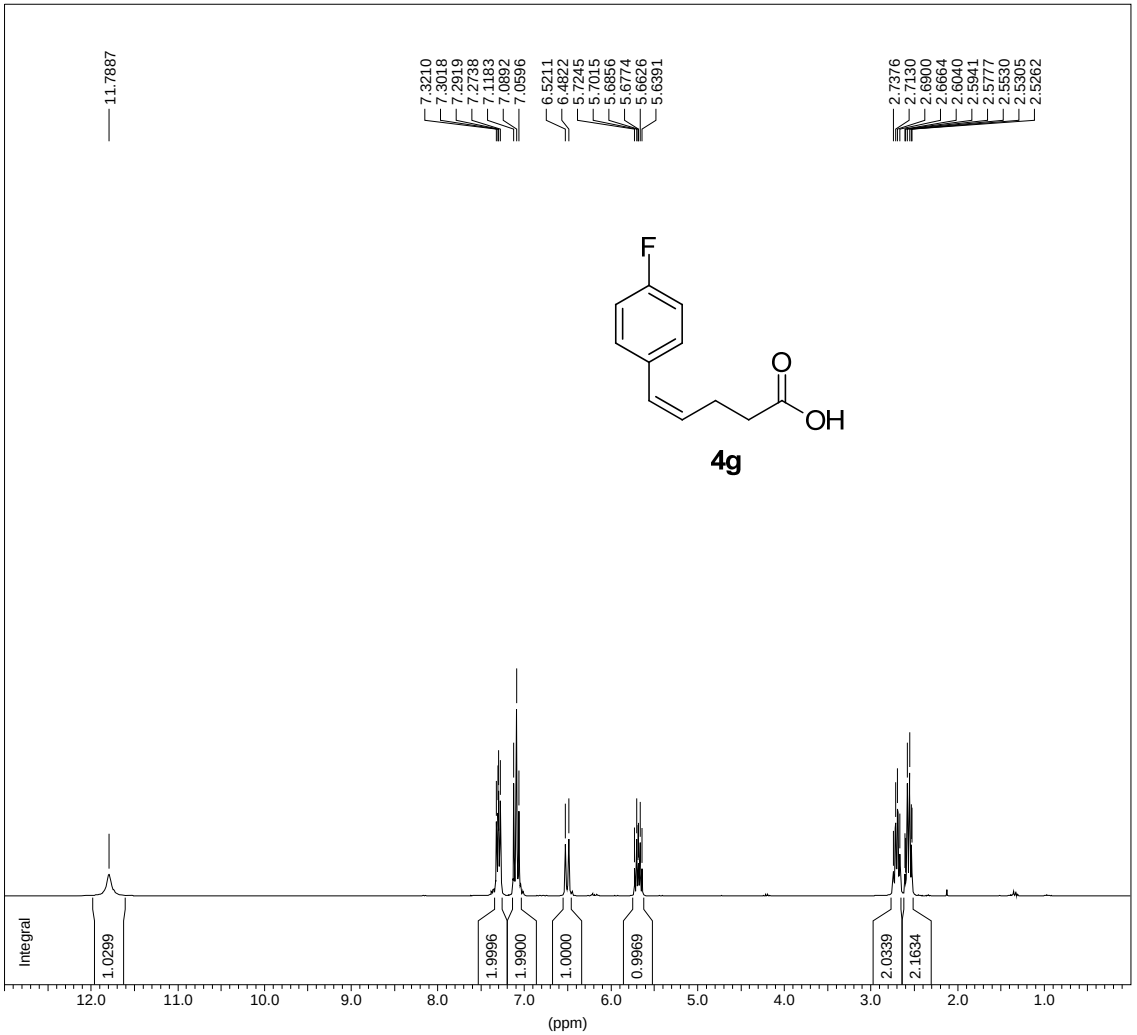
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	129.443 degree
PHC1	:	4.161 degree

1H normal range AC300 cc-i-073-B



*** Current Data Parameters ***

NAME	:	oc17lcc
EXPNO	:	2
PROCNO	:	1

*** Acquisition Parameters ***

INSTRUM	:	spect
LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768
TE	:	298.3 K

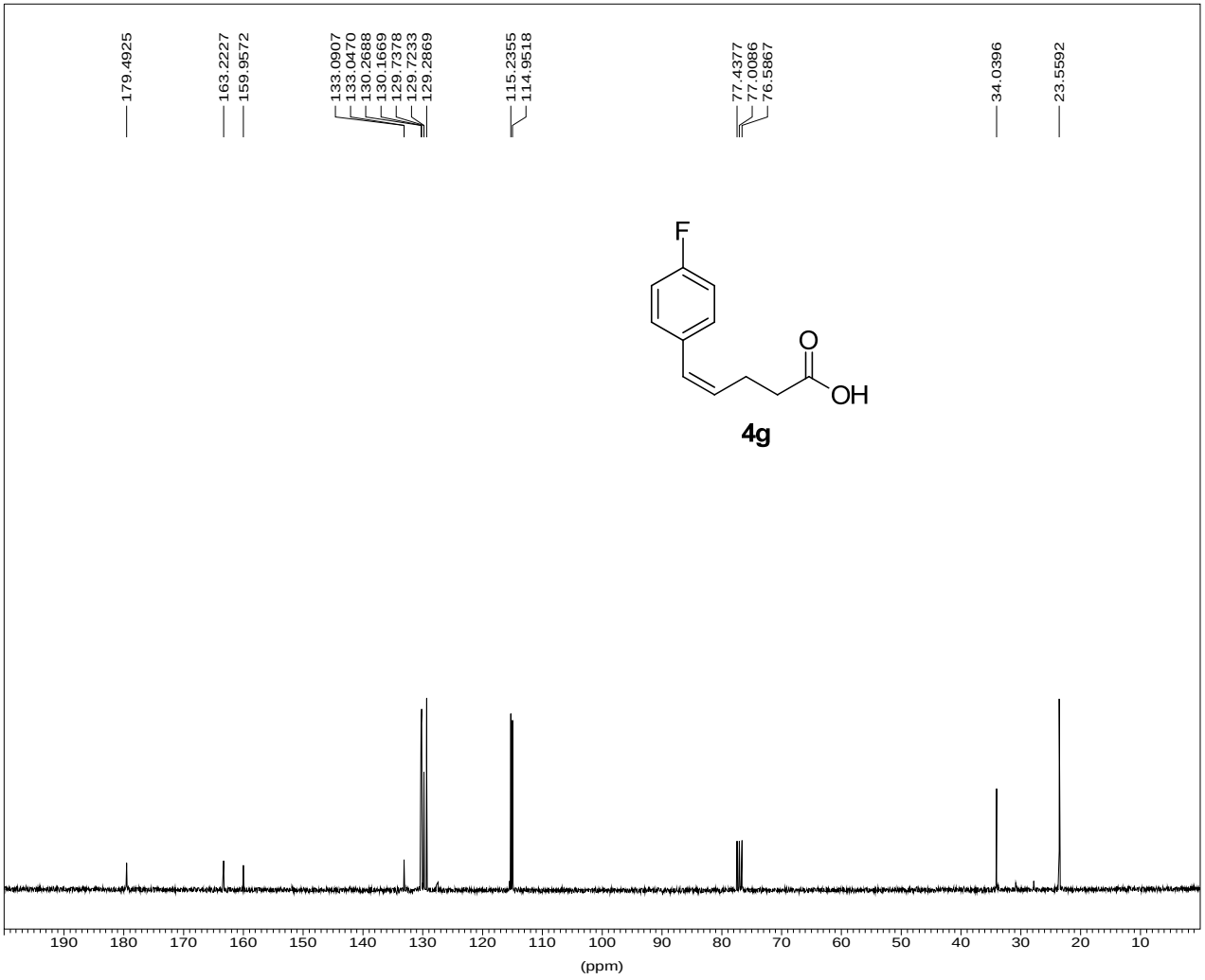
*** Processing Parameters ***

LB	:	0.30 Hz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C Standard AC300 cc-039-A



*** Current Data Parameters ***

NAME	:	de05lcc
EXPNO	:	7
PROCNO	:	1

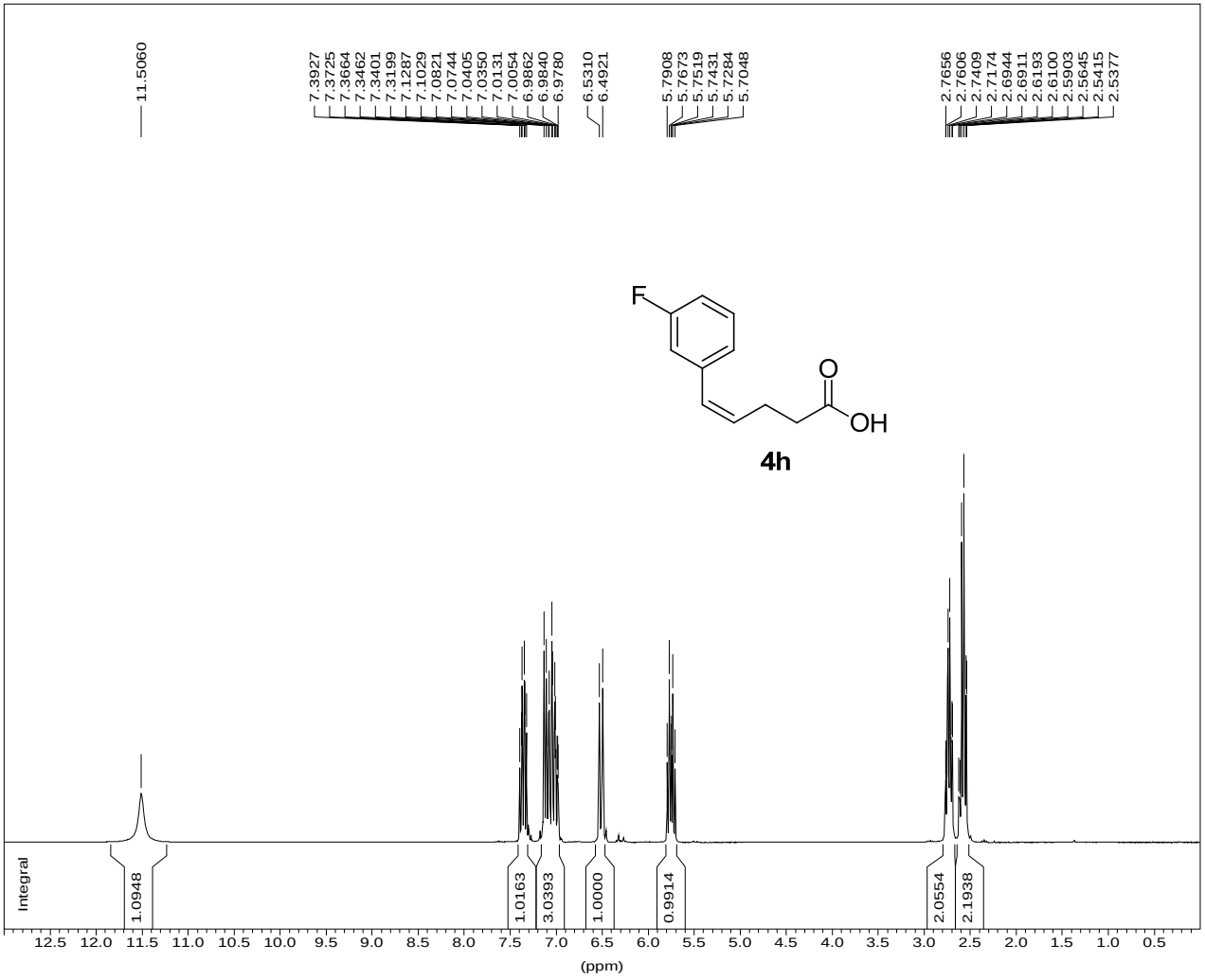
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	60.894 degree
PHC1	:	6.282 degree

1H normal range AC300 cc-i-103-A



*** Current Data Parameters ***

NAME	:	de02lcc
EXPNO	:	1
PROCNO	:	1

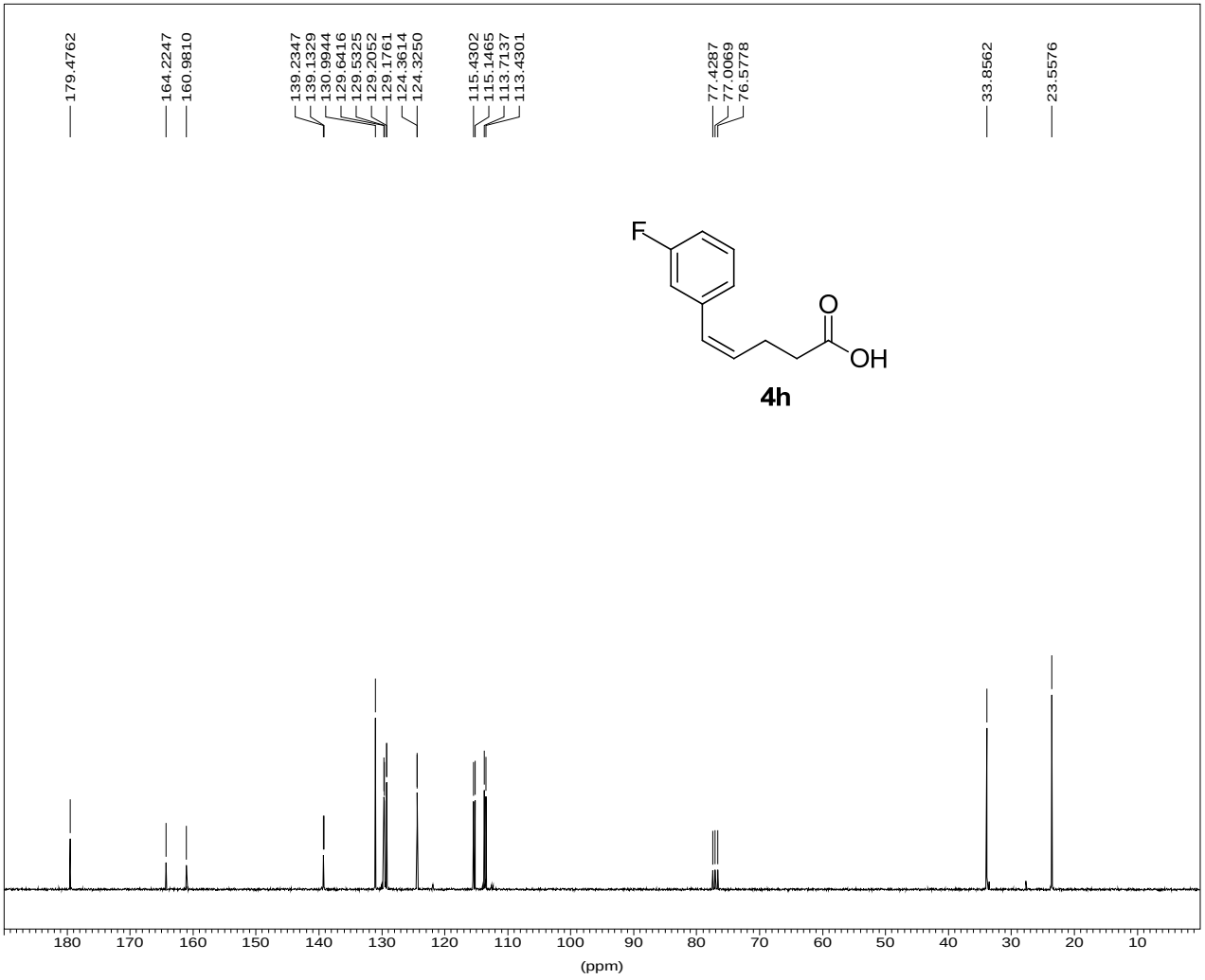
*** Acquisition Parameters ***

BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm

*** Processing Parameters ***

LB	:	0.30 Hz
PHC0	:	123.186 degree
PHC1	:	1.190 degree

13C Standard AC300 cc-i-103-A



*** Current Data Parameters ***

NAME	:	de02lcc
EXPNO	:	2
PROCNO	:	1

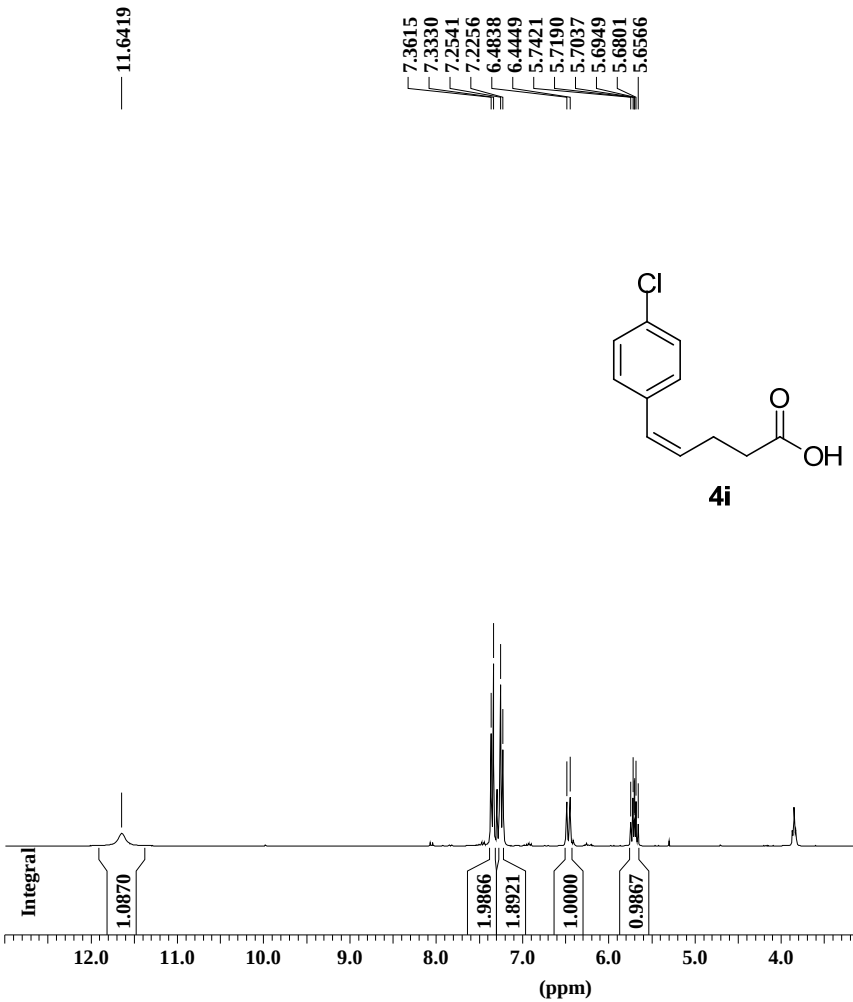
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	123.730 degree
PHC1	:	8.448 degree

1H normal range AC300 cc-i-087-A



***** Current Data Parameters *****

NAME	:	nv04lcc
EXPNO	:	1
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768
TE	:	297.1 K

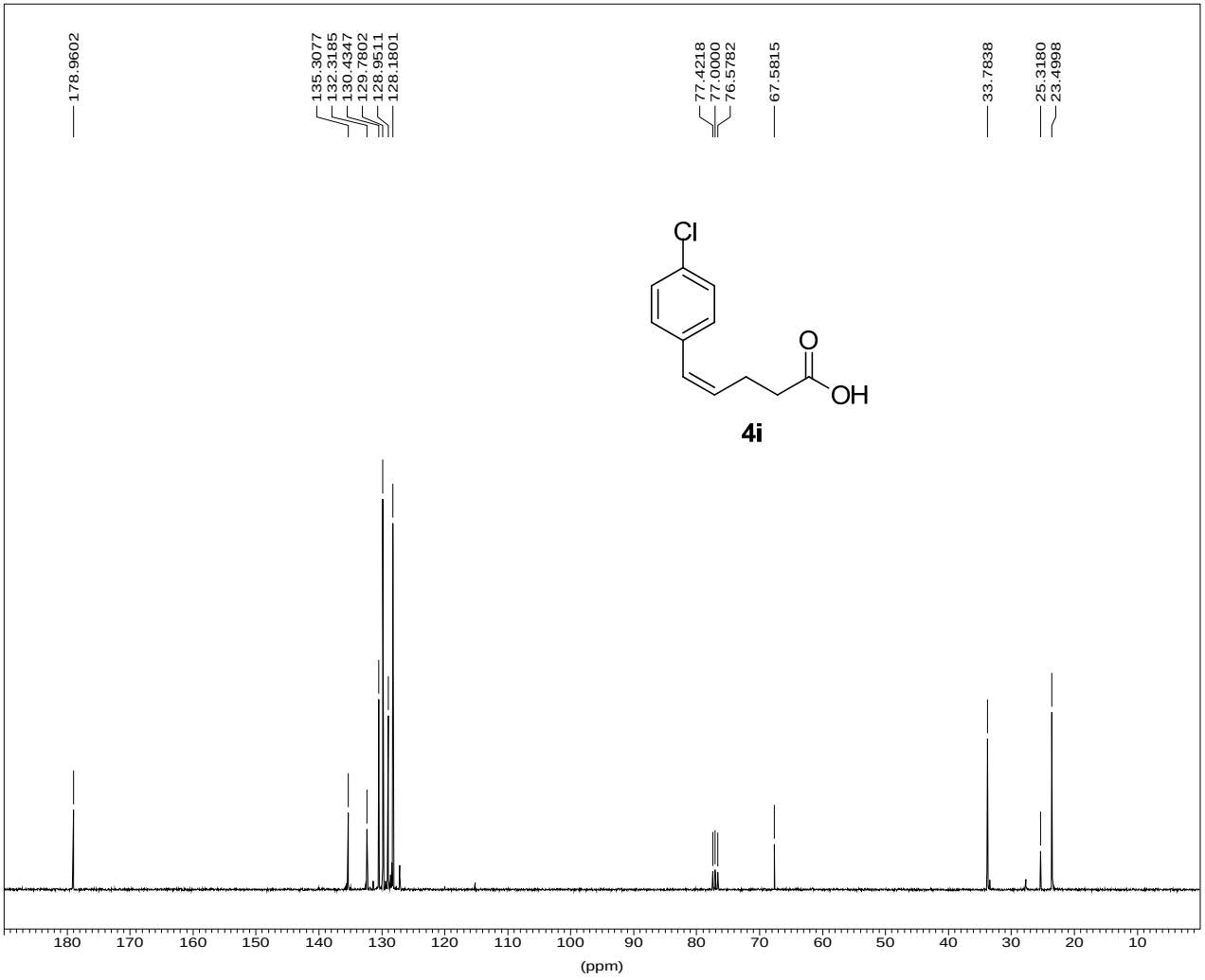
***** Processing Parameters *****

LB	:	0.30 Hz
SF	:	300.1300000 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

13C Standard AC300 cc-i-087-A



*** Current Data Parameters ***

NAME	:	nv04lcc
EXPNO	:	2
PROCNO	:	1

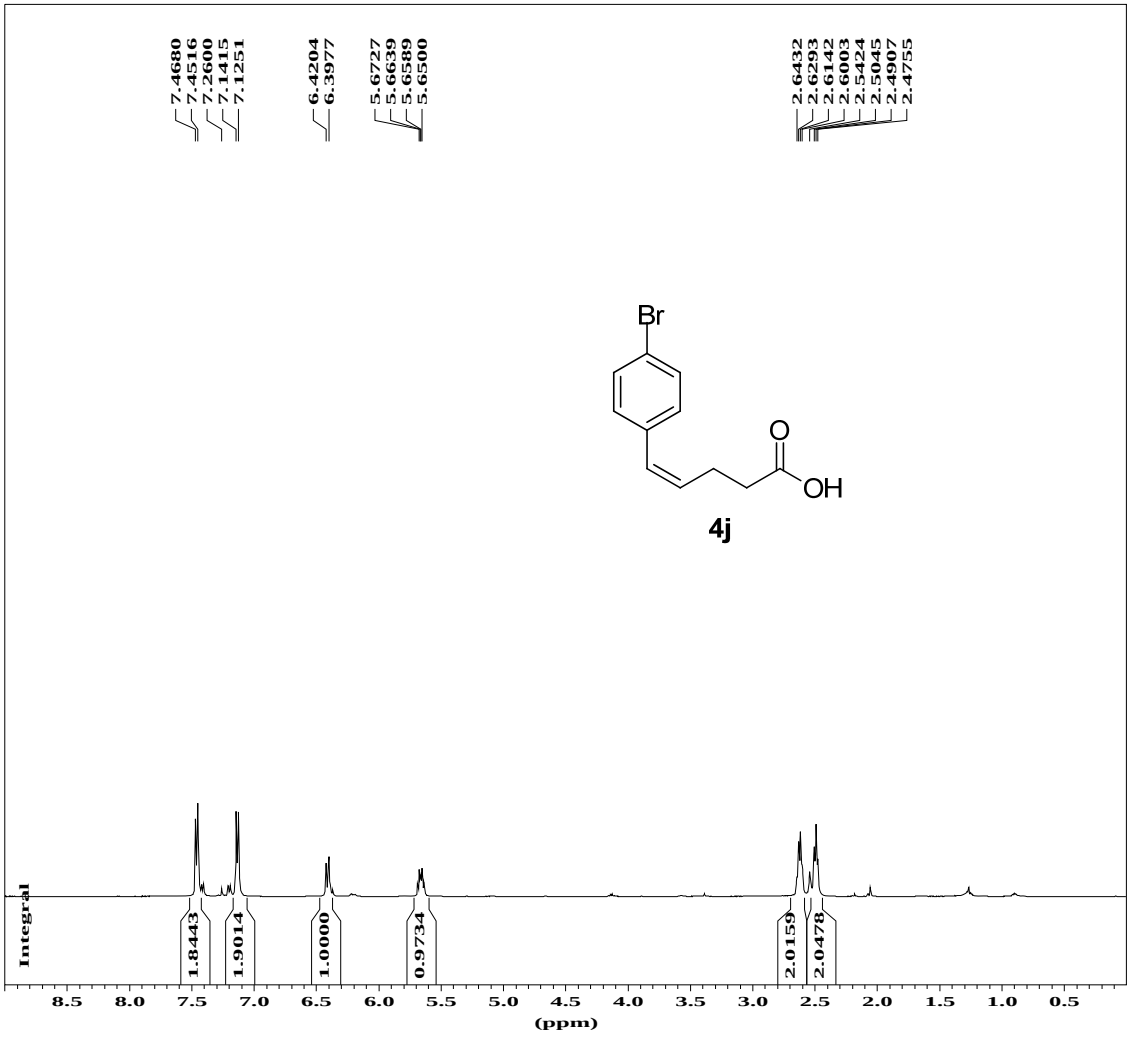
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	229.915 degree
PHC1	:	15.443 degree

1H AMX500 4-Br acid



***** Current Data Parameters *****

NAME	:	ck0214
EXPNO	:	5
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	3088.51 Hz
PULPROG	:	zg30
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768
TE	:	298.0 K

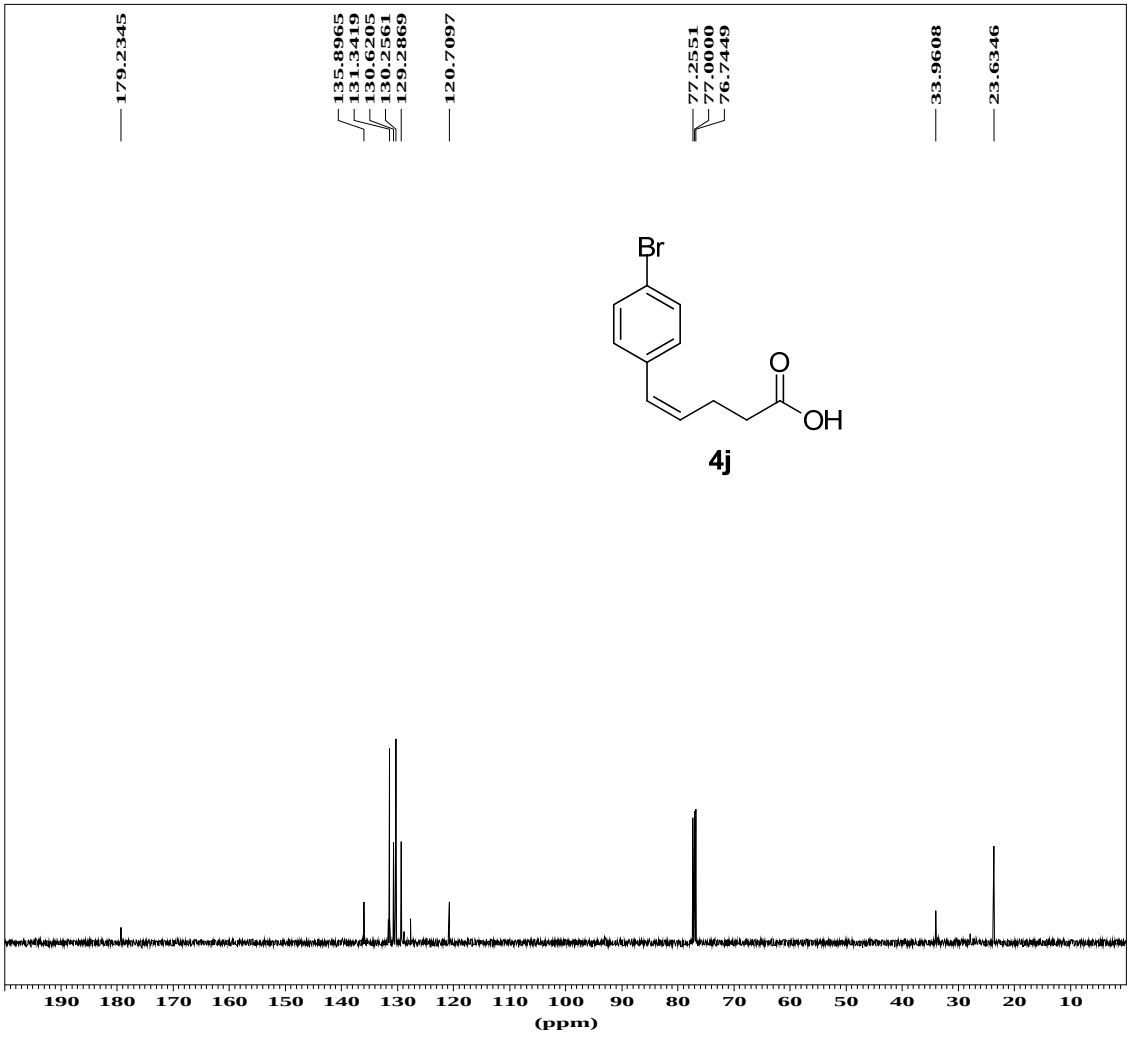
***** Processing Parameters *****

LB	:	0.30 Hz
SF	:	500.1300140 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

13C AMX500 4-Br acid



***** Current Data Parameters *****

NAME	:	ck0214
EXPNO	:	6
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	30
NUCLEUS	:	off
O1	:	13204.57 Hz
PULPROG	:	zgpg30
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm
TD	:	65536
TE	:	298.3 K

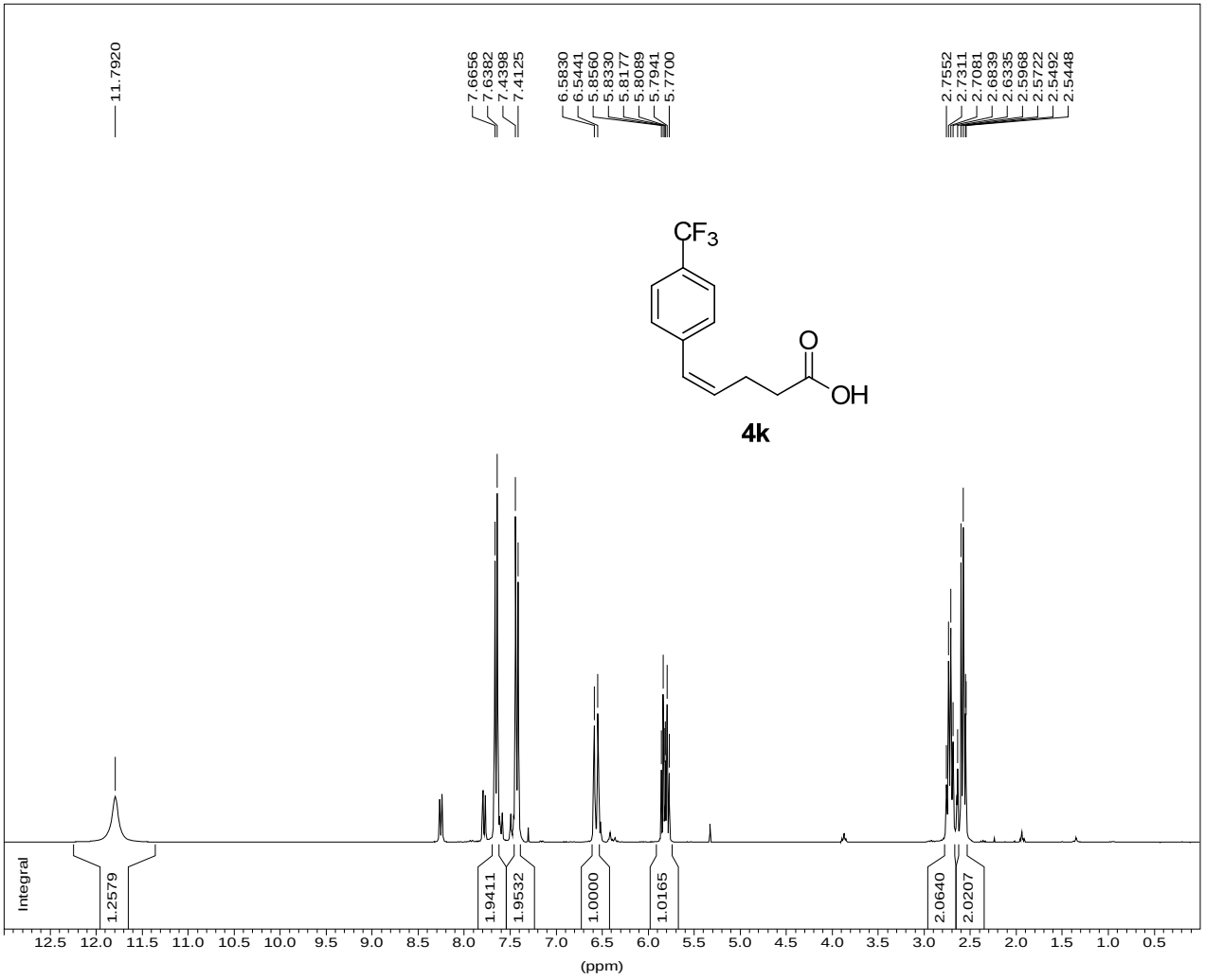
***** Processing Parameters *****

LB	:	1.00 Hz
SF	:	125.7577970 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

1H normal range AC300 cc-093-A



*** Current Data Parameters ***

NAME	:	nv25lcc
EXPNO	:	1
PROCNO	:	1

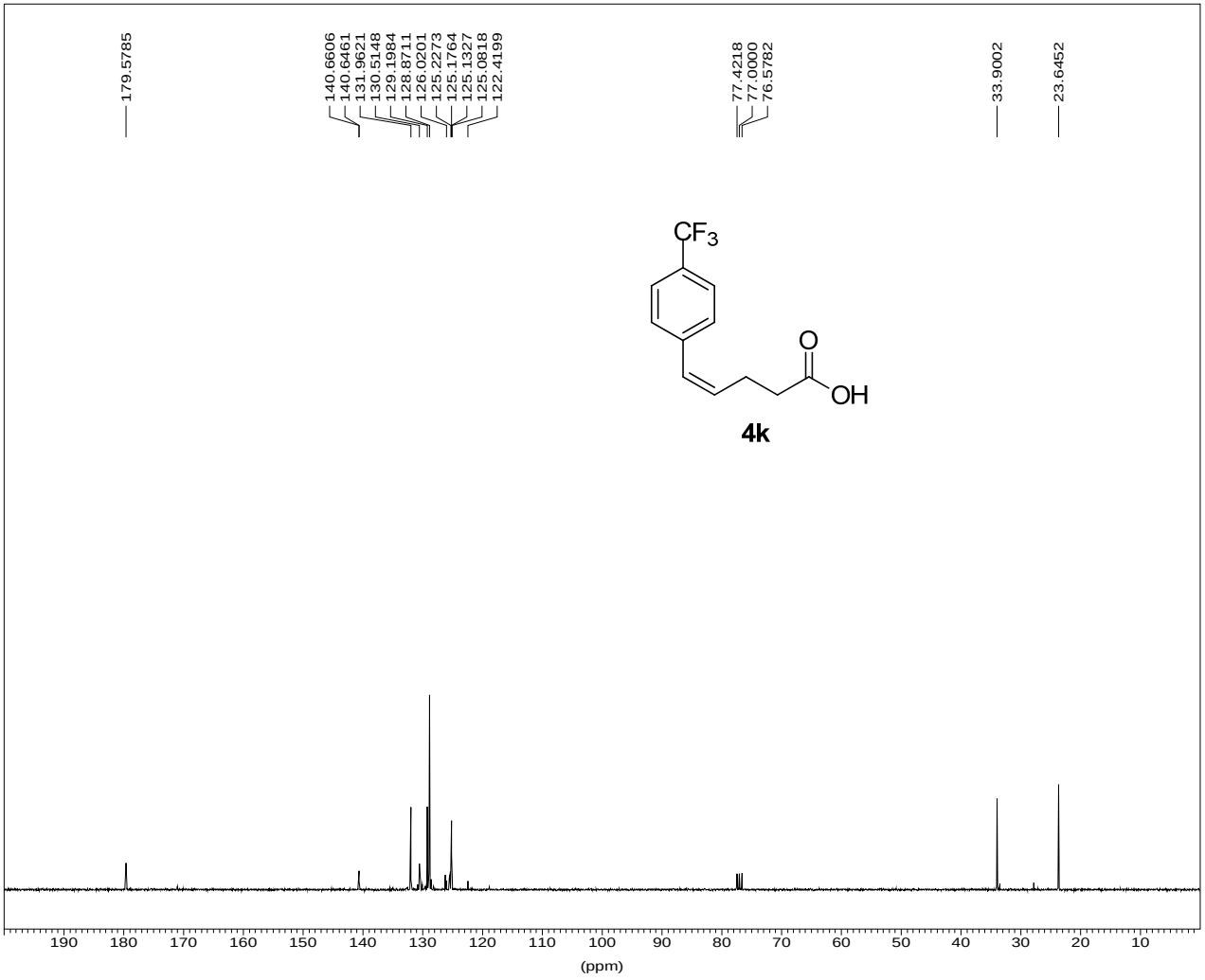
*** Acquisition Parameters ***

BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm

*** Processing Parameters ***

LB	:	0.30 Hz
PHC0	:	126.640 degree
PHC1	:	0.468 degree

13C Standard AC300 cc-093-A



*** Current Data Parameters ***

NAME	:	nv25lcc
EXPNO	:	2
PROCNO	:	1

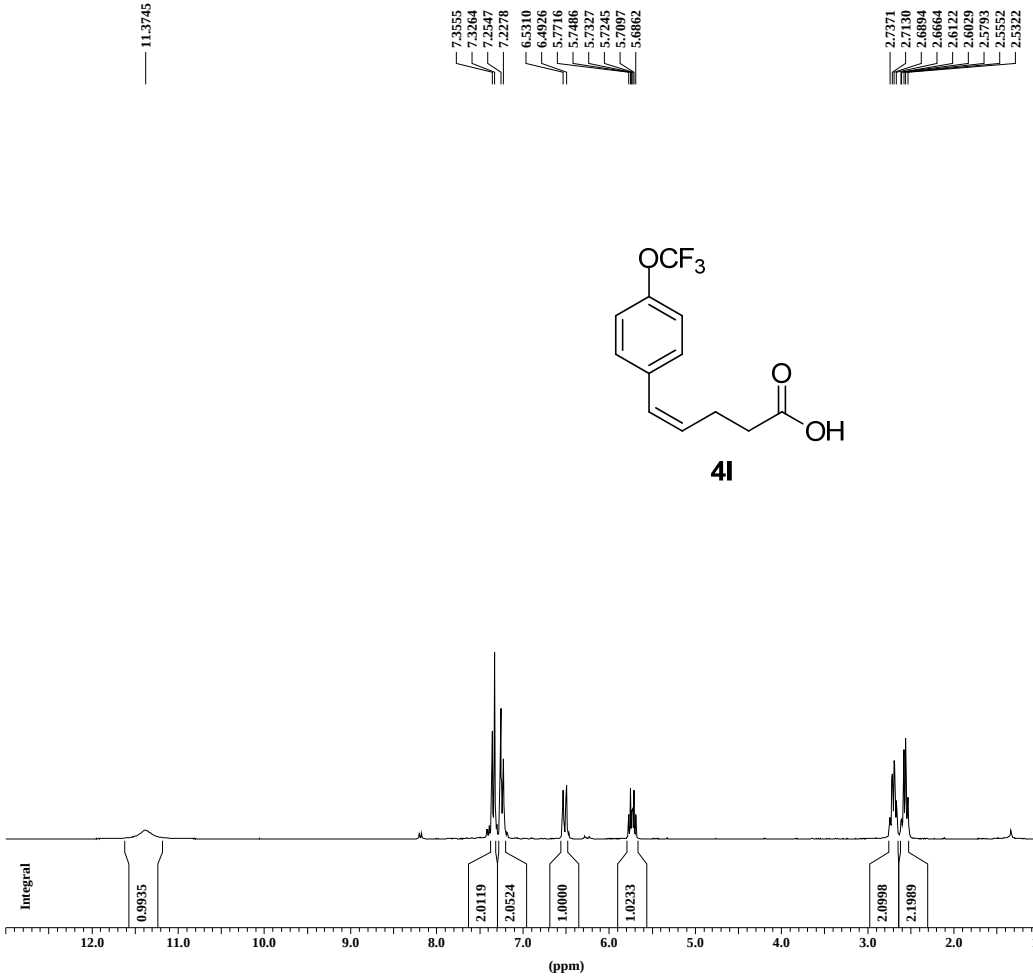
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	132.986 degree
PHC1	:	-2.942 degree

1H normal range AC300 cc-079-A



***** Current Data Parameters *****

NAME	:	oc25lcc
EXPNO	:	1
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768
TE	:	297.4 K

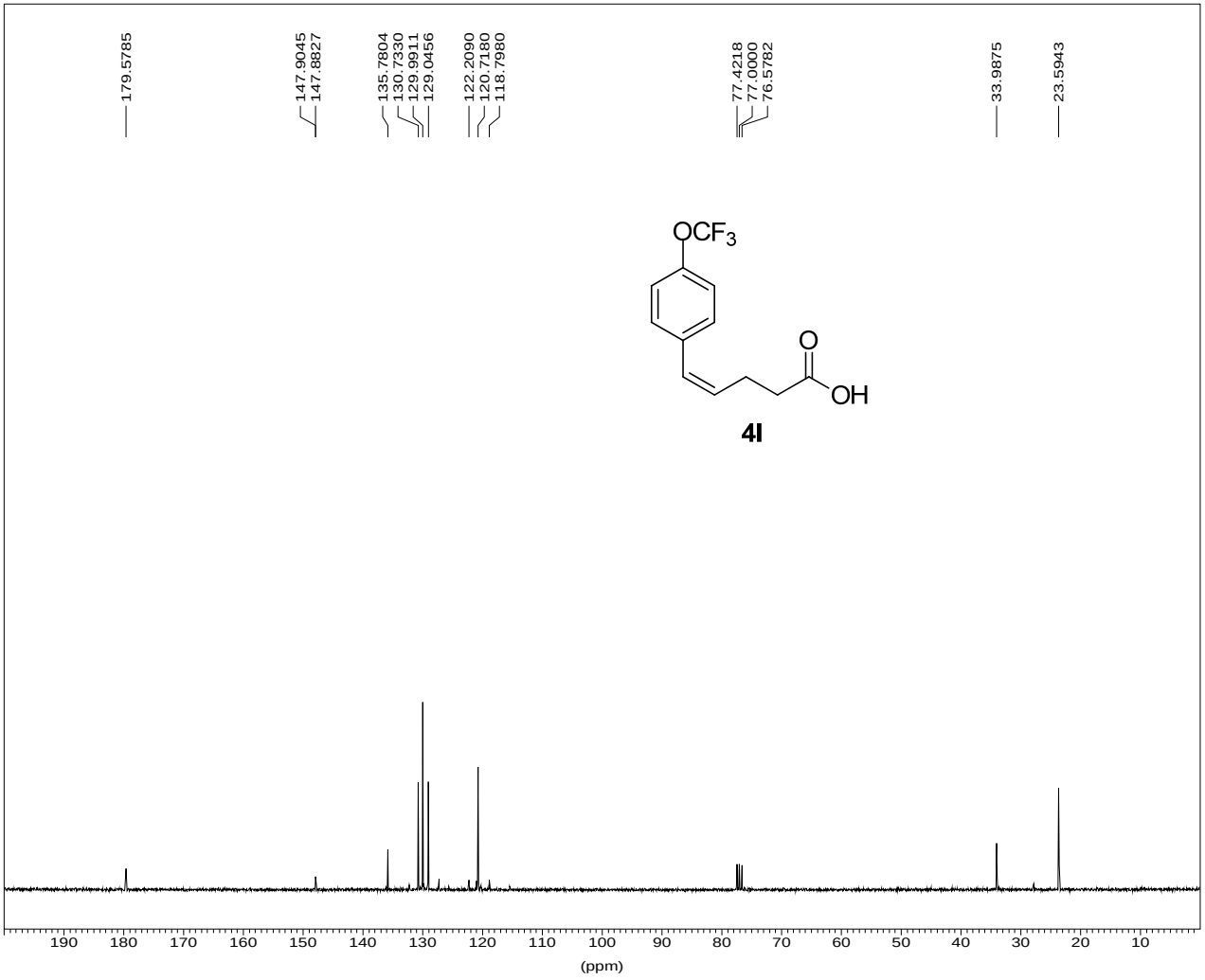
***** Processing Parameters *****

LB	:	0.30 Hz
SF	:	300.1300000 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

13C Standard AC300 cc-079-A



*** Current Data Parameters ***

NAME	:	oc25lcc
EXPNO	:	2
PROCNO	:	1

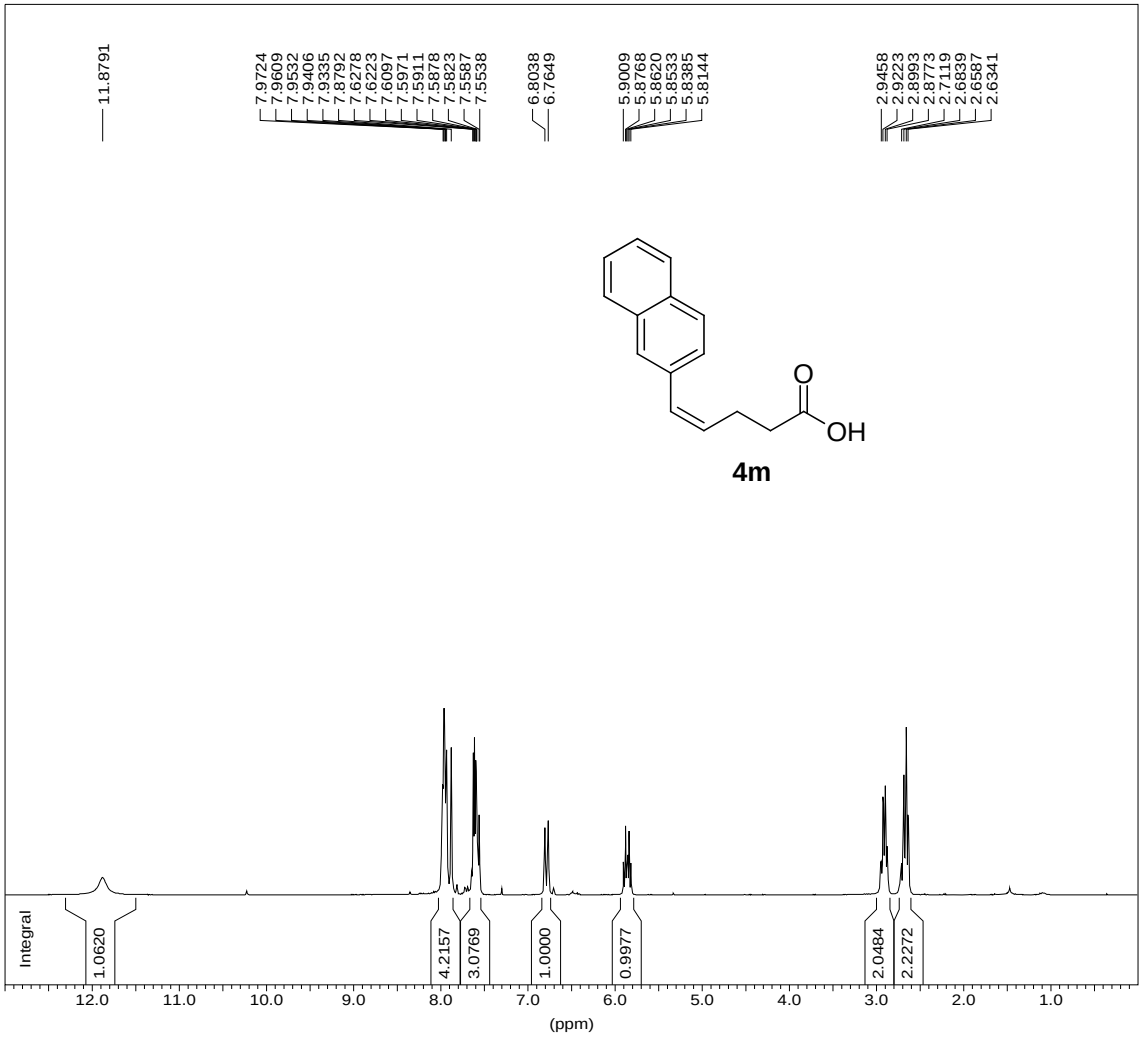
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	165.849 degree
PHC1	:	2.377 degree

1H normal range AC300 cc-i-076-B



*** Current Data Parameters ***

NAME	:	oc21lcc
EXPNO	:	6
PROCNO	:	1

*** Acquisition Parameters ***

NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768

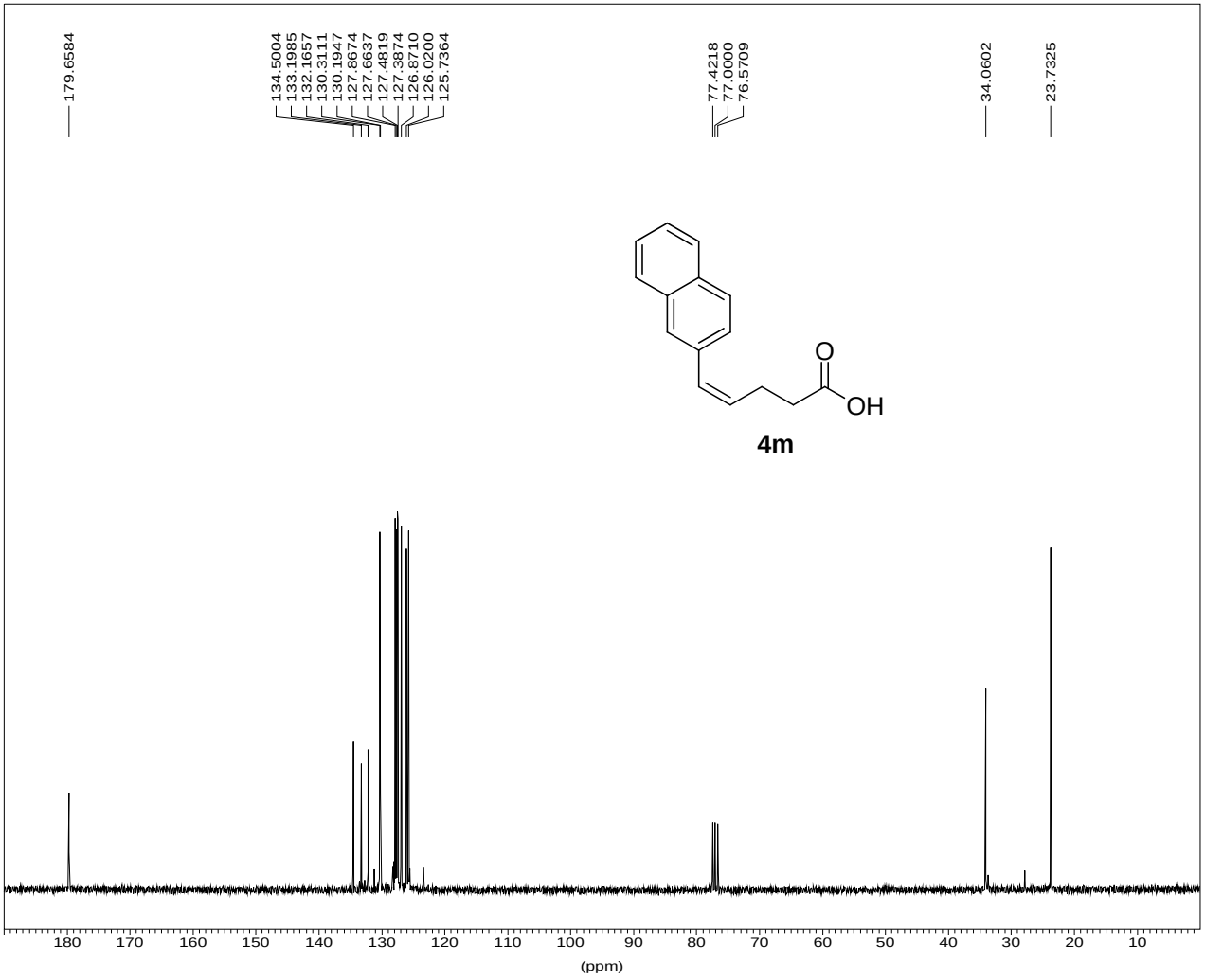
*** Processing Parameters ***

LB	:	0.30 Hz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C Standard AC300 cc-i-076-B



*** Current Data Parameters ***

NAME	:	oc21lcc
EXPNO	:	7
PROCNO	:	1

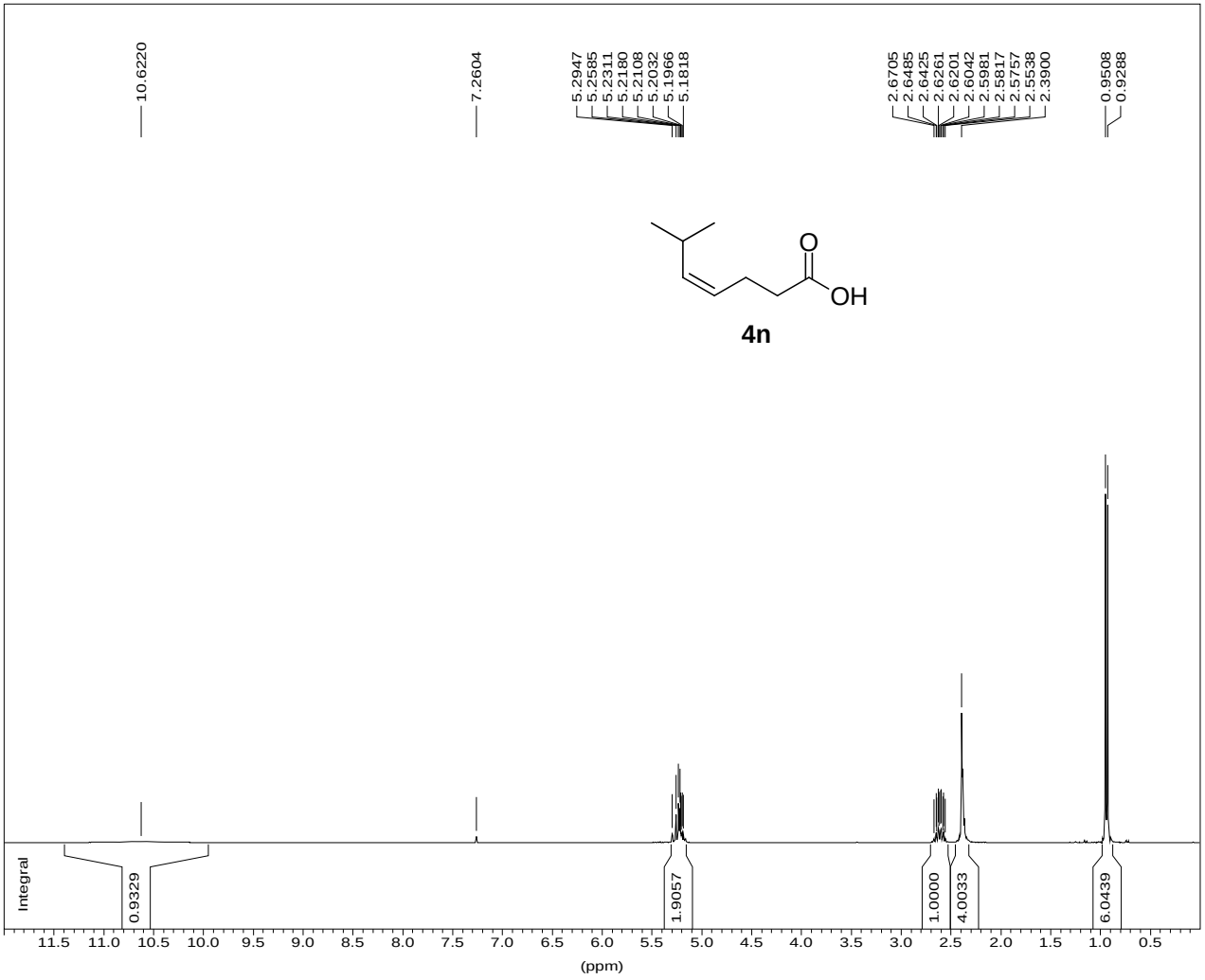
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	173.391 degree
PHC1	:	-0.418 degree

1H normal range AC300 cc-i-103-B



*** Current Data Parameters ***

NAME	:	de02lcc
EXPNO	:	3
PROCNO	:	1

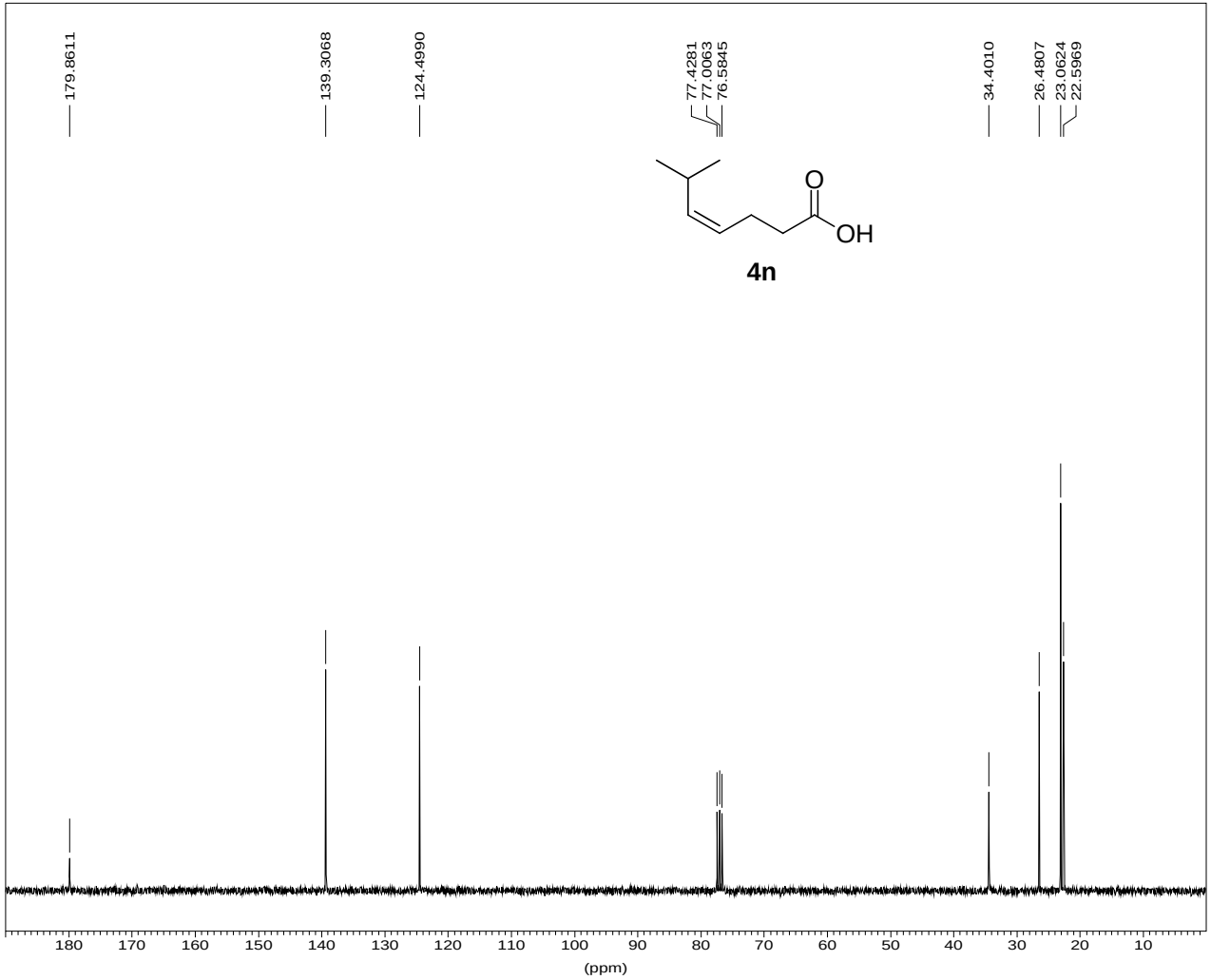
*** Acquisition Parameters ***

BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm

*** Processing Parameters ***

LB	:	0.30 Hz
PHC0	:	236.844 degree
PHC1	:	2.939 degree

13C Standard AC300 cc-i-103-B



*** Current Data Parameters ***

NAME	:	de02lcc
EXPNO	:	4
PROCNO	:	1

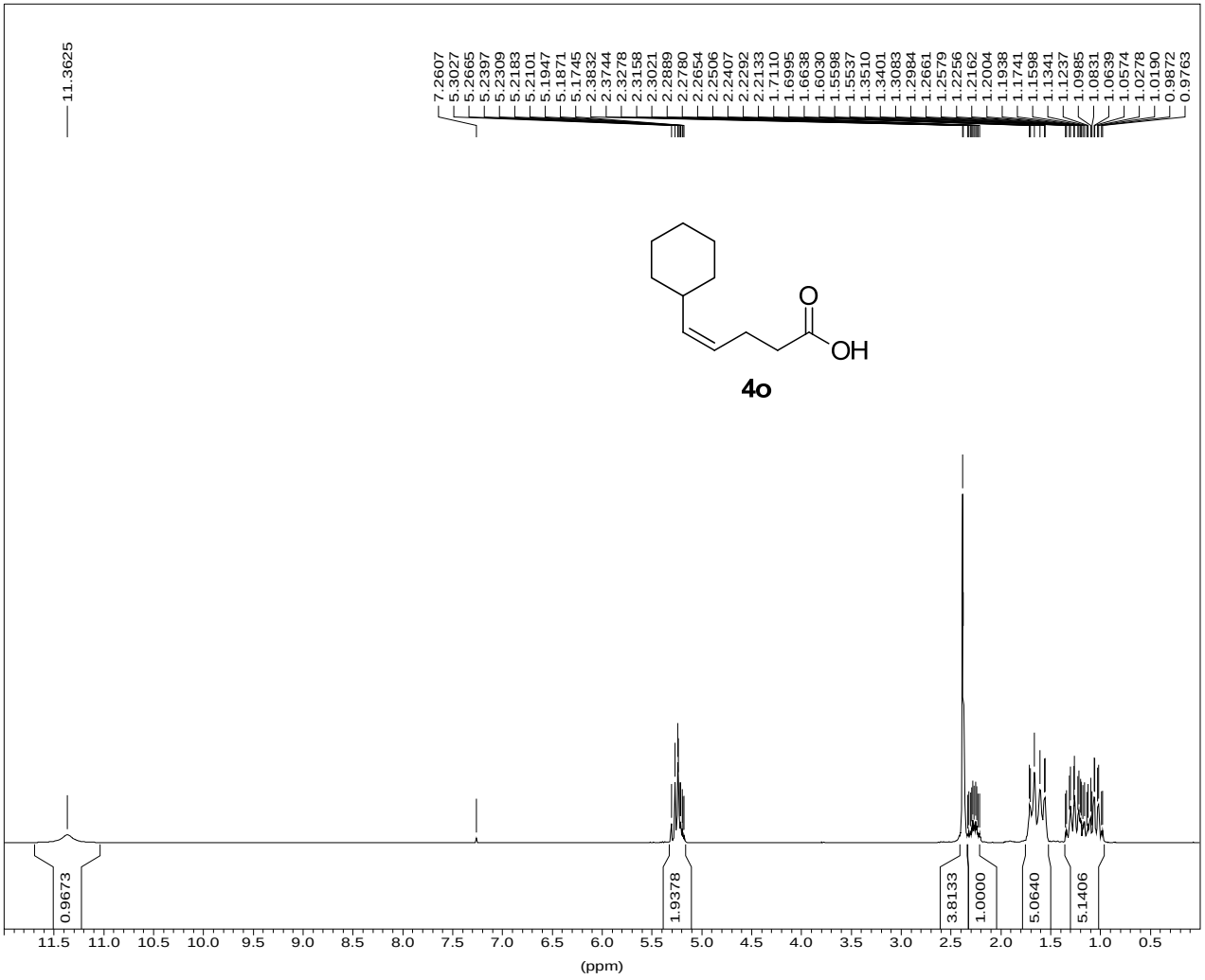
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	108
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	125.135 degree
PHC1	:	8.434 degree

1H normal range AC300 cc-i-103-D

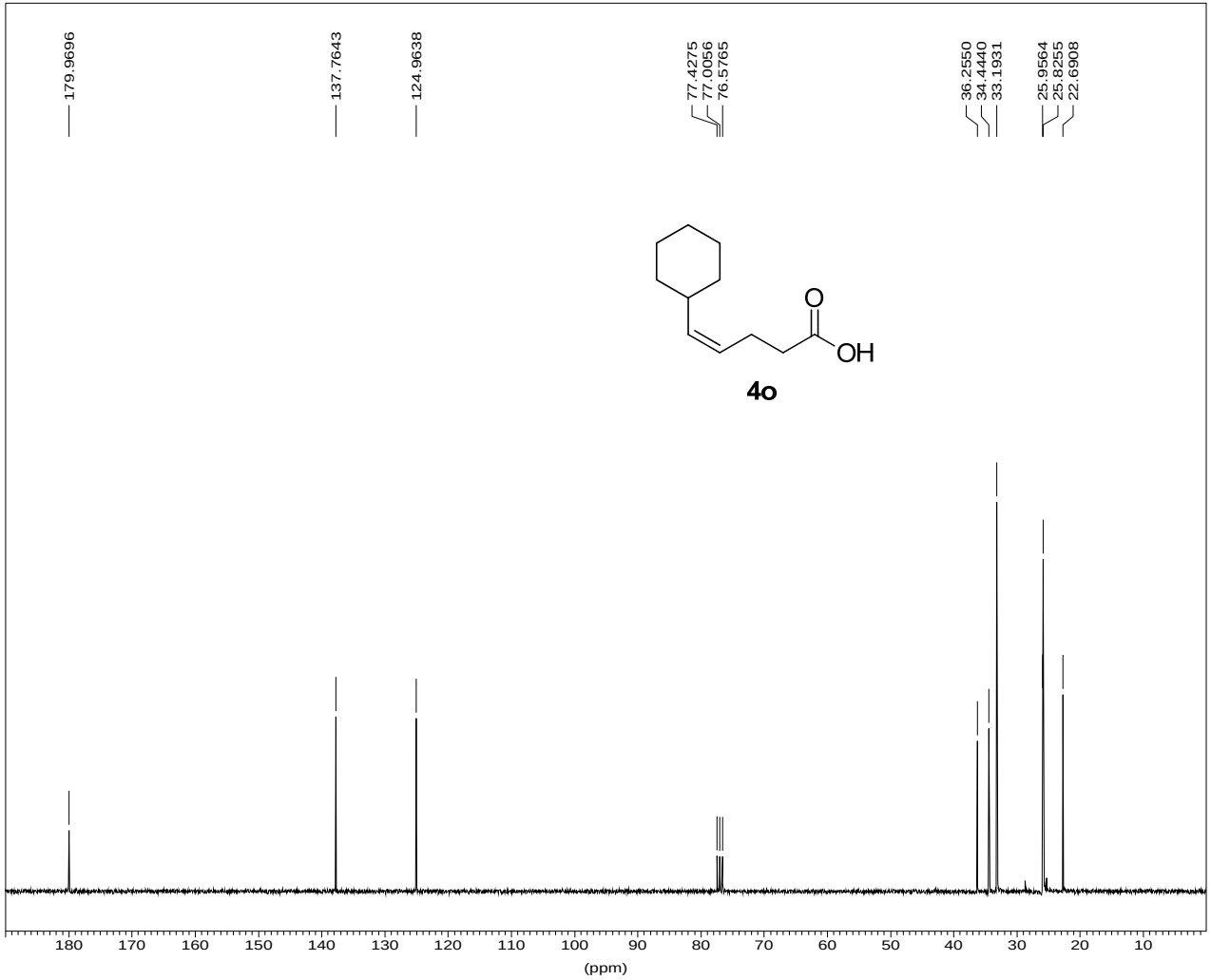


4o

*** Current Data Parameters ***

NAME	:	de02lcc
EXPNO	:	7
PROCNO	:	1
*** Acquisition Parameters ***		
BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
*** Processing Parameters ***		
LB	:	0.30 Hz
PHC0	:	126.063 degree
PHC1	:	1.329 degree

13C Standard AC300 cc-i-103-C



*** Current Data Parameters ***

NAME	:	de02lcc
EXPNO	:	8
PROCNO	:	1

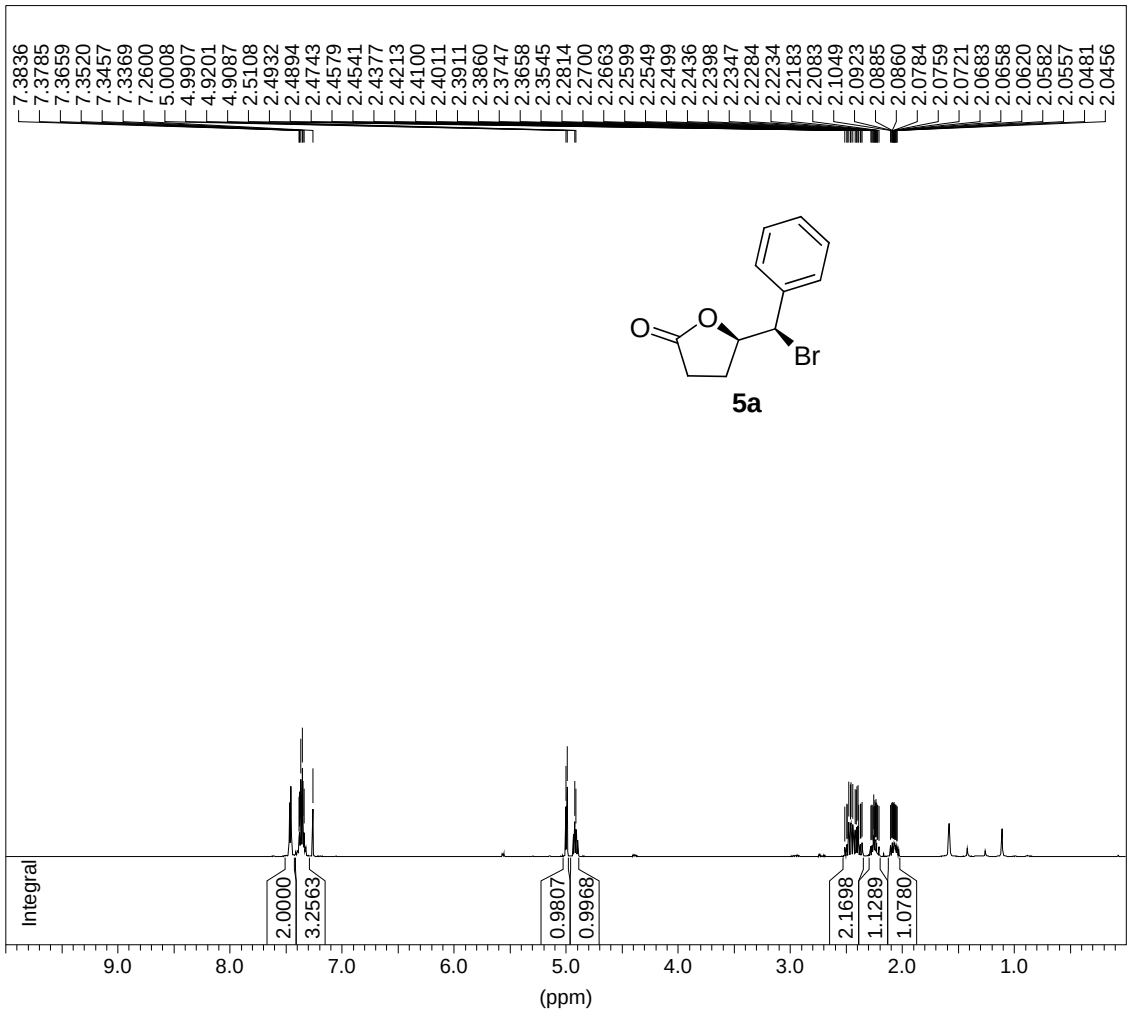
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	57
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	114.104 degree
PHC1	:	31.913 degree

1H AMX500 cis lactone 1



*** Current Data Parameters ***

NAME	:	ck0415
EXPNO	:	3
PROCNO	:	1

*** Acquisition Parameters ***

INSTRUM	:	av500
LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	3088.51 Hz
PULPROG	:	zg30
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768

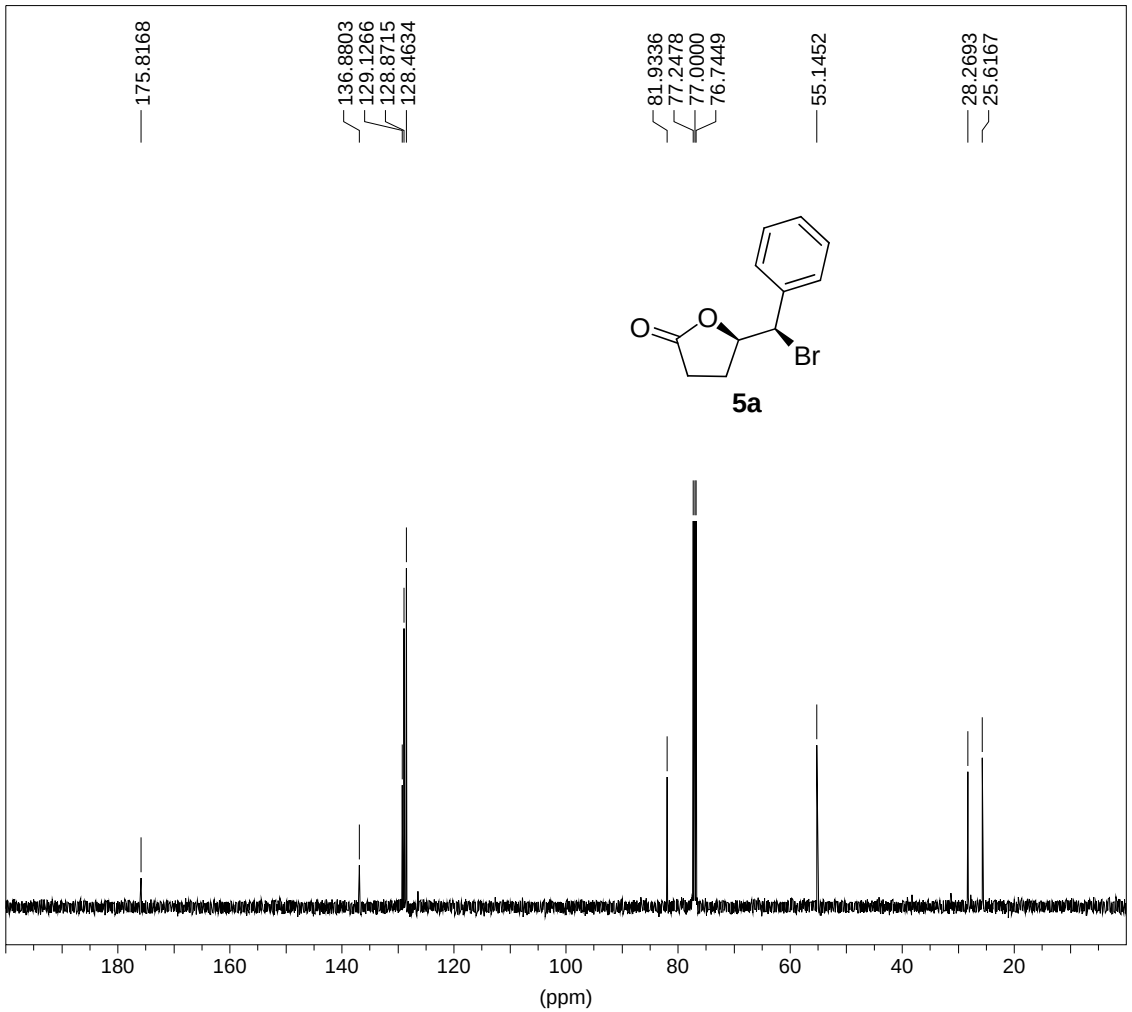
*** Processing Parameters ***

LB	:	0.30 Hz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C AMX500 cis lactone 1



Bruker

*** Current Data Parameters ***

NAME	:	ck0415
EXPNO	:	4
PROCNO	:	1

*** Acquisition Parameters ***

INSTRUM	:	av500
LOCNUC	:	2H
NS	:	201
NUCLEUS	:	off
O1	:	13204.57 Hz
PULPROG	:	zgpg30
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm
TD	:	65536

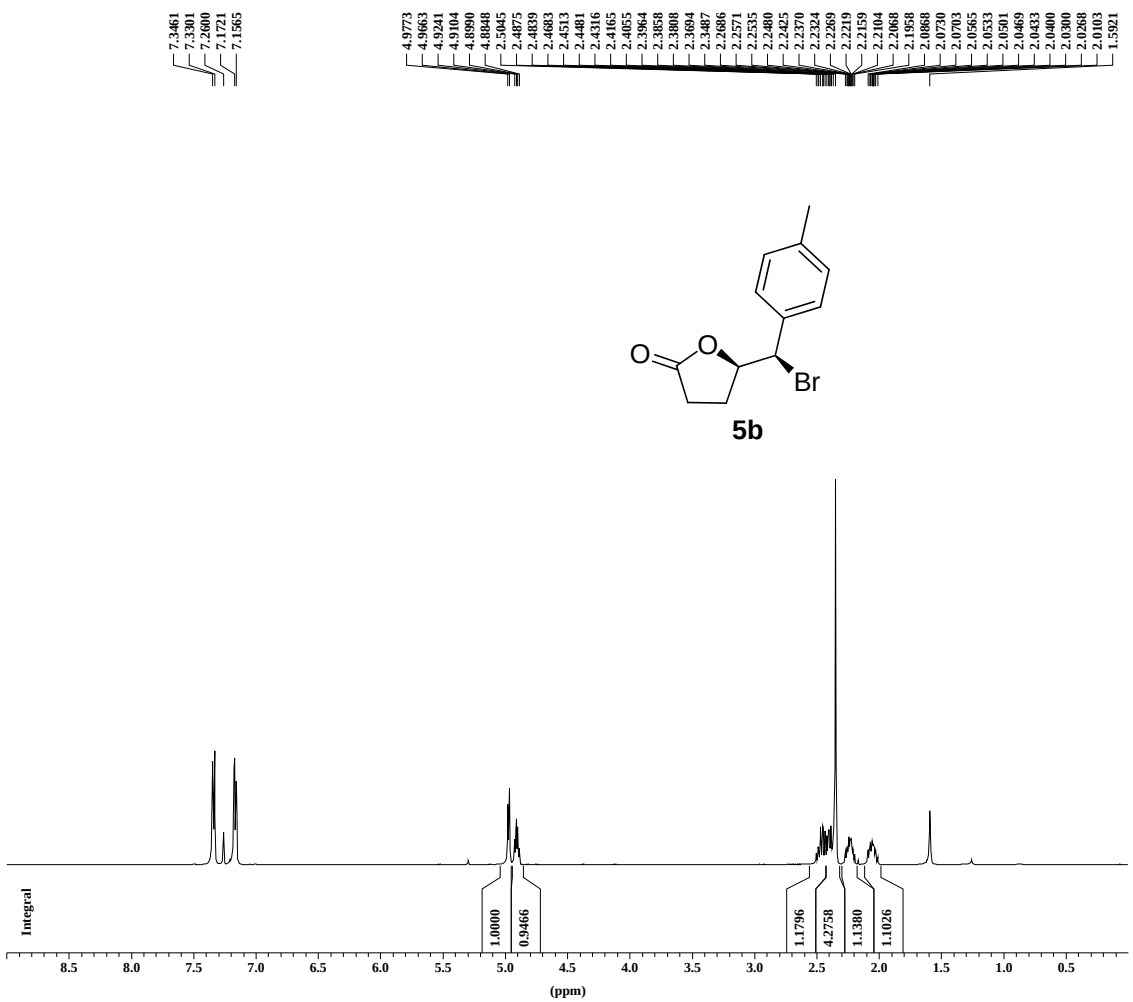
*** Processing Parameters ***

LB	:	1.00 Hz

*** 1D NMR Plot Parameters ***

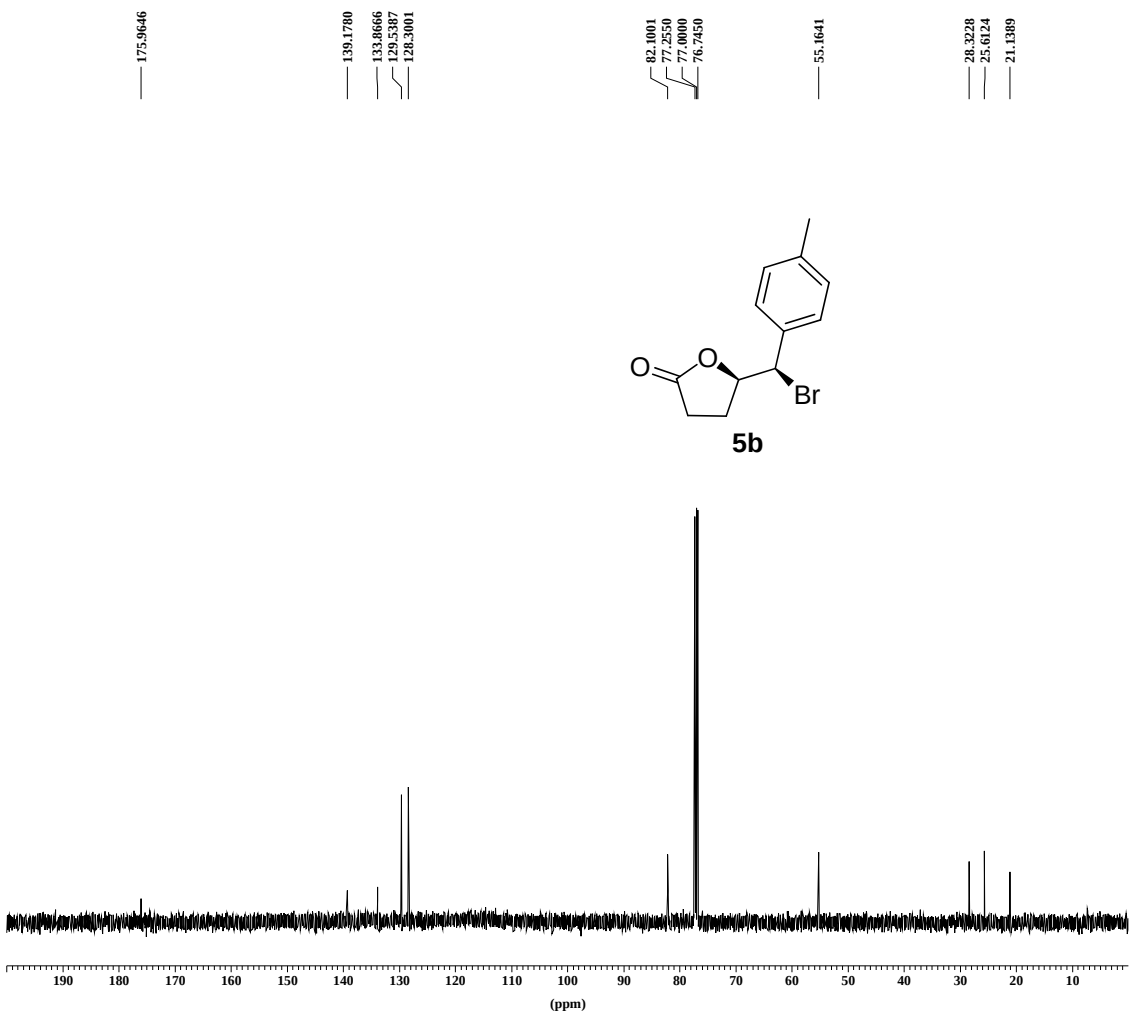
NUCLEUS	:	off

cc-i-167a



*** Current Data Parameters ***
NAME : ck0214
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
LOCNUC : 2H
NS : 8
NUCLEUS : off
O1 : 2751.27 Hz
PULPROG : zg
SFO1 : 500.2327513 MHz
SOLVENT : CDCl3
SW : 15.0080 ppm
TD : 32768
TE : 300.0 K
*** Processing Parameters ***
LB : 0.10 Hz
SF : 500.2300090 MHz
*** 1D NMR Plot Parameters ***
NUCLEUS : off

cc-i-167a



*** Current Data Parameters ***

NAME : ck0214

EXPNO : 2

PROCNO : 1

*** Acquisition Parameters ***

LOCNUC : 2H

NS : 210

NUCLEUS : off

O1 : 12577.84 Hz

PULPROG : zgpg30

SFO1 : 125.7955118 MHz

SOLVENT : CDCl3

SW : 238.7210 ppm

TD : 65536

TE : 300.0 K

*** Processing Parameters ***

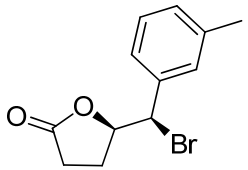
LB : 1.00 Hz

SF : 125.7829370 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS : off

1H normal range AC300 cc-i-112-D



5c

***** Current Data Parameters *****

NAME	:	ja05lcc
EXPNO	:	6
PROCNO	:	1

***** Acquisition Parameters *****

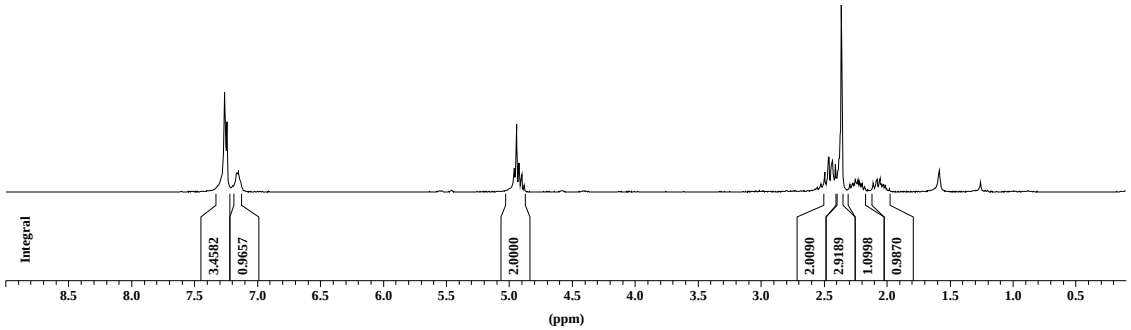
LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768
TE	:	678.6 K

***** Processing Parameters *****

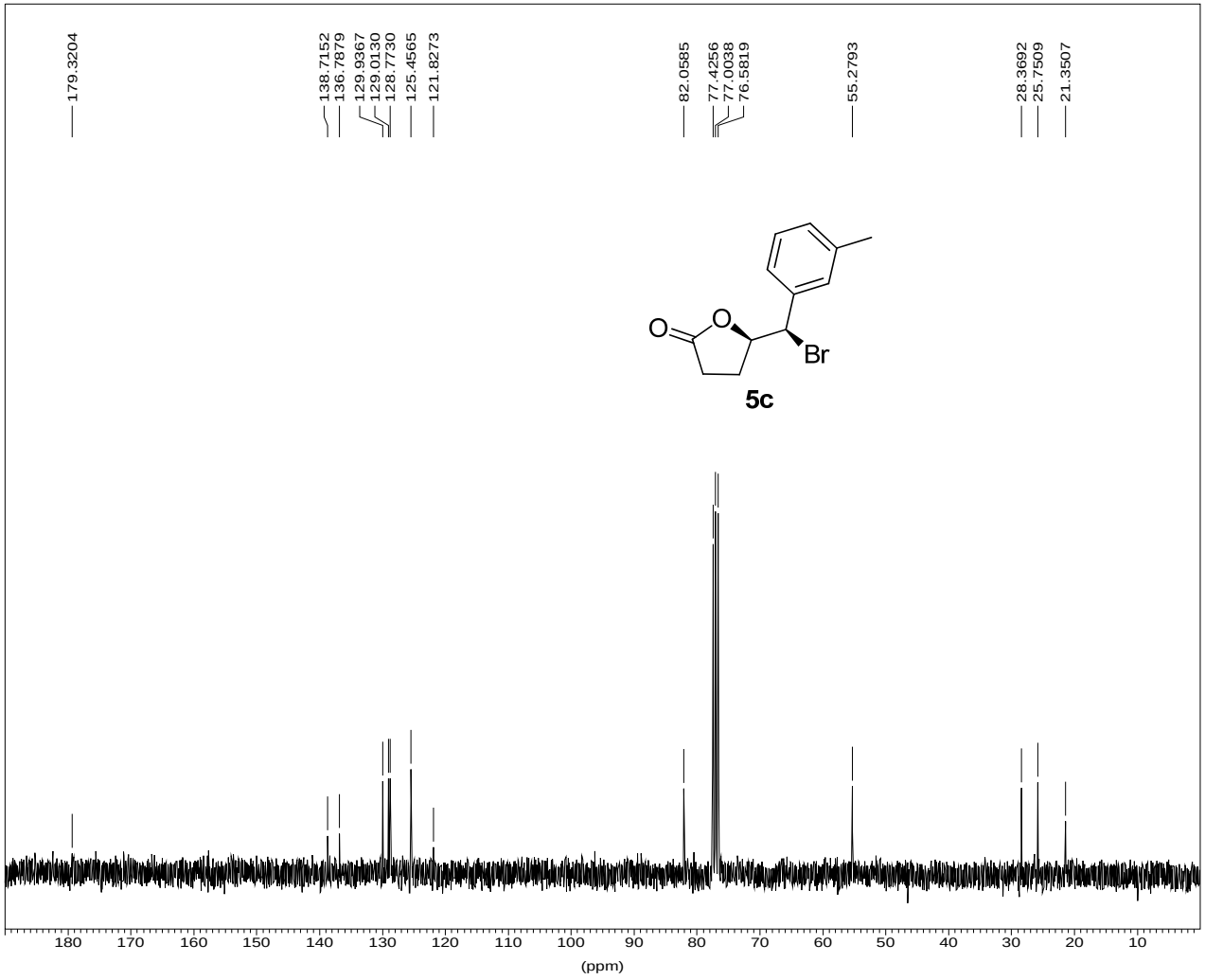
LB	:	0.30 Hz
SF	:	300.1300115 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off



13C Standard AC300 cc-i-112-D



*** Current Data Parameters ***

NAME	:	ja05lcc
EXPNO	:	7
PROCNO	:	1

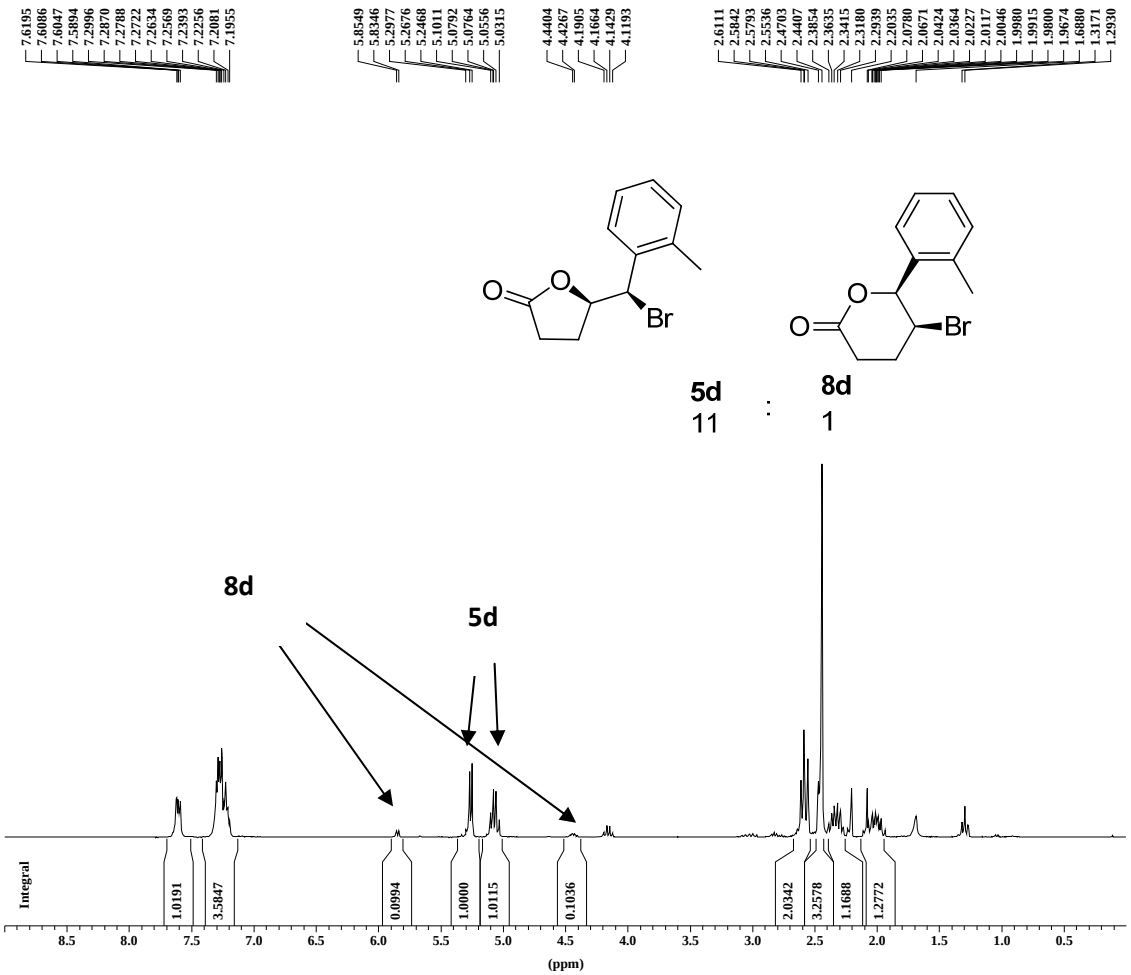
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	222
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	139.602 degree
PHC1	:	-3.288 degree

1H normal range AC300 cc-i-162b



***** Current Data Parameters *****

NAME	:	fe11ck
EXPNO	:	1
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768
TE	:	297.1 K

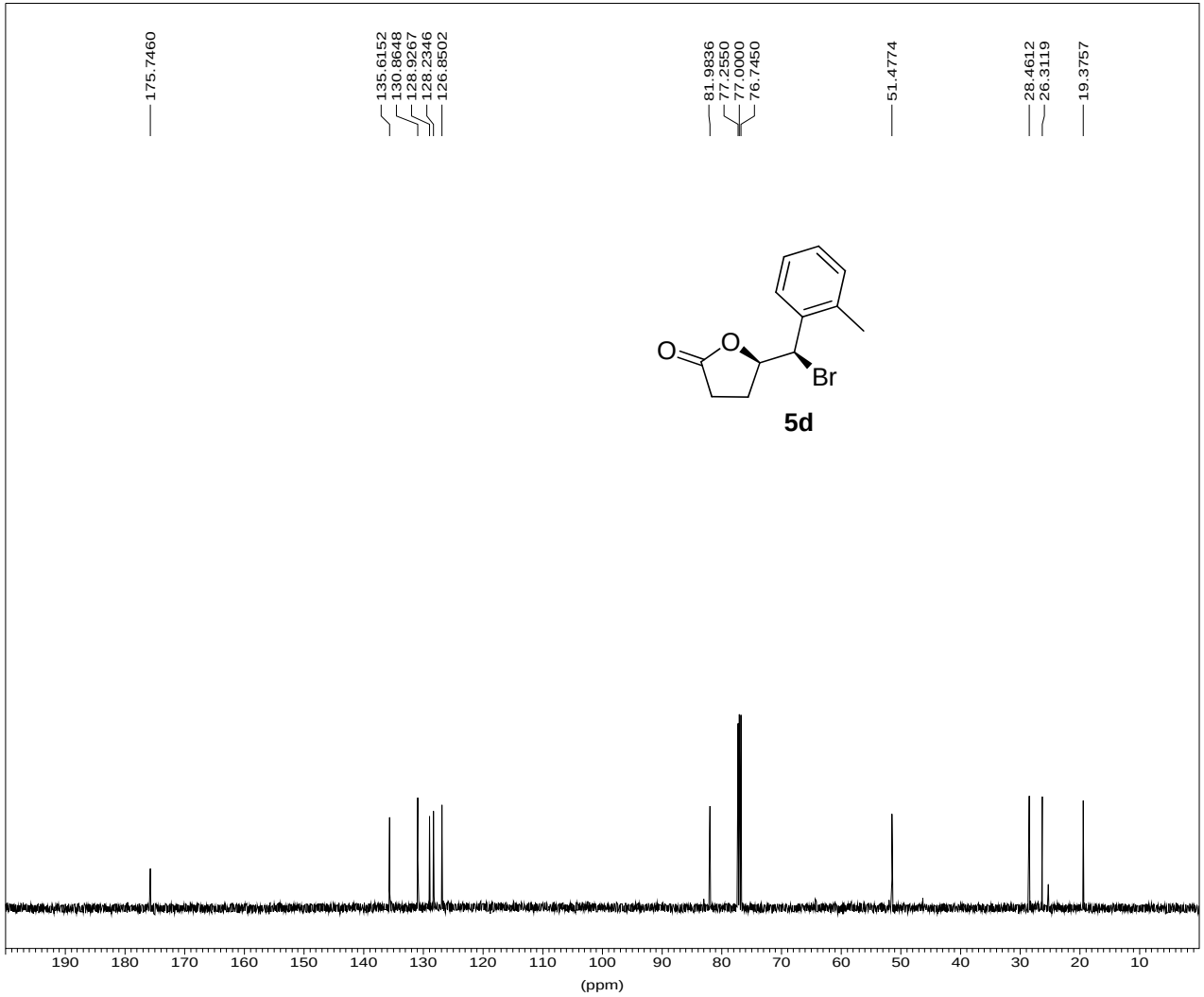
***** Processing Parameters *****

LB	:	0.30 Hz
SF	:	300.1300000 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

ck-v-119



*** Current Data Parameters ***

NAME	:	ck0131
EXPNO	:	4
PROCNO	:	1

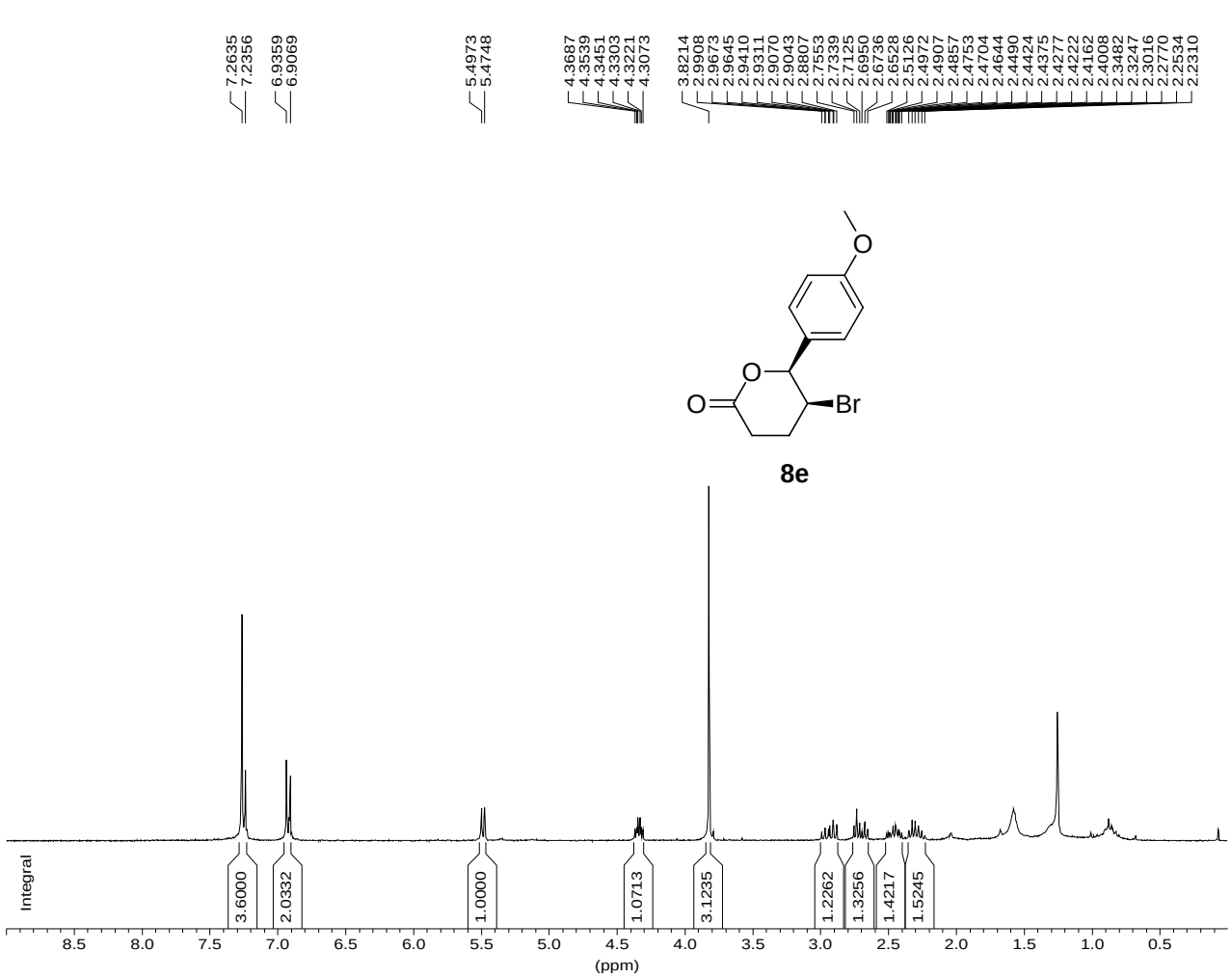
*** Acquisition Parameters ***

BF1	:	125.7829340 MHz
LOCNUC	:	2H
NS	:	140
O1	:	12577.84 Hz
PULPROG	:	zgpg30
SFO1	:	125.7955118 MHz
SOLVENT	:	CDCl3
SW	:	238.7210 ppm

*** Processing Parameters ***

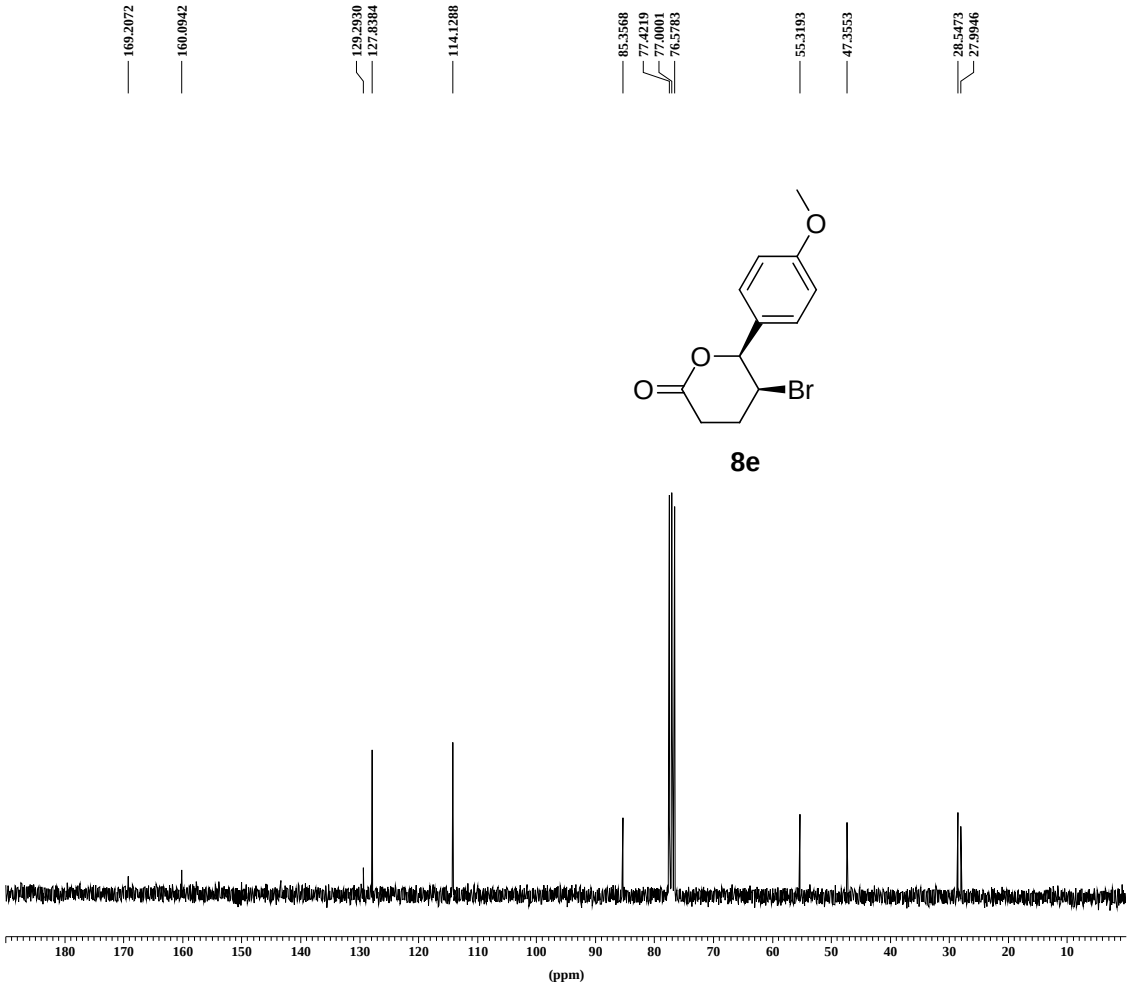
LB	:	1.00 Hz
PHC0	:	188.334 degree
PHC1	:	11.705 degree

1H normal range AC300 cc-i-104-A



*** Current Data Parameters ***
NAME : de06lcc
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
NS : 8
NUCLEUS : off
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm
TD : 32768
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
NUCLEUS : off

13C Standard AC300 cc-i-166-C



***** Current Data Parameters *****

NAME	:	fe11lcc
EXPNO	:	6
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	657
NUCLEUS	:	off
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm
TD	:	32768
TE	:	297.1 K

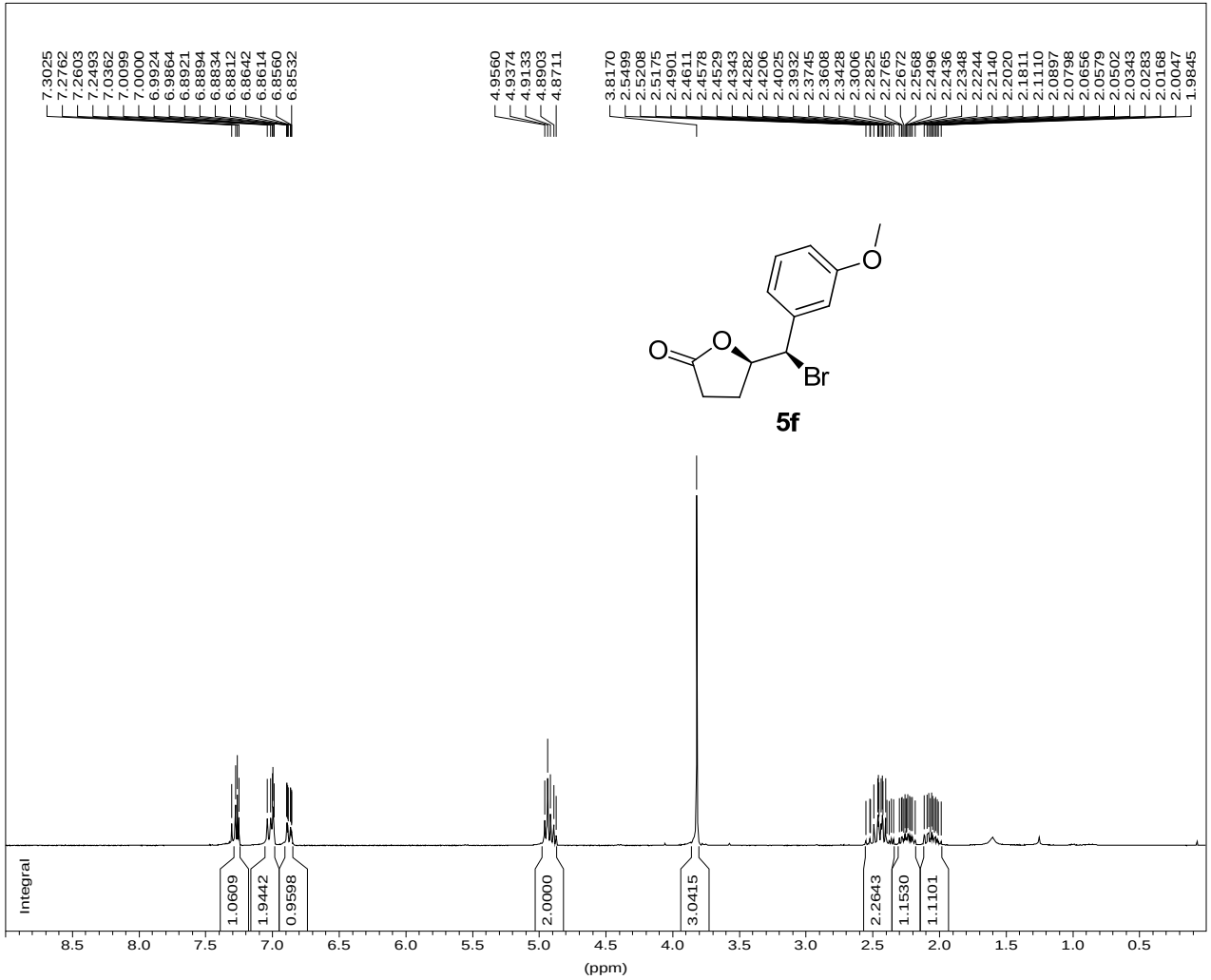
***** Processing Parameters *****

LB	:	1.00 Hz
SF	:	75.4677517 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

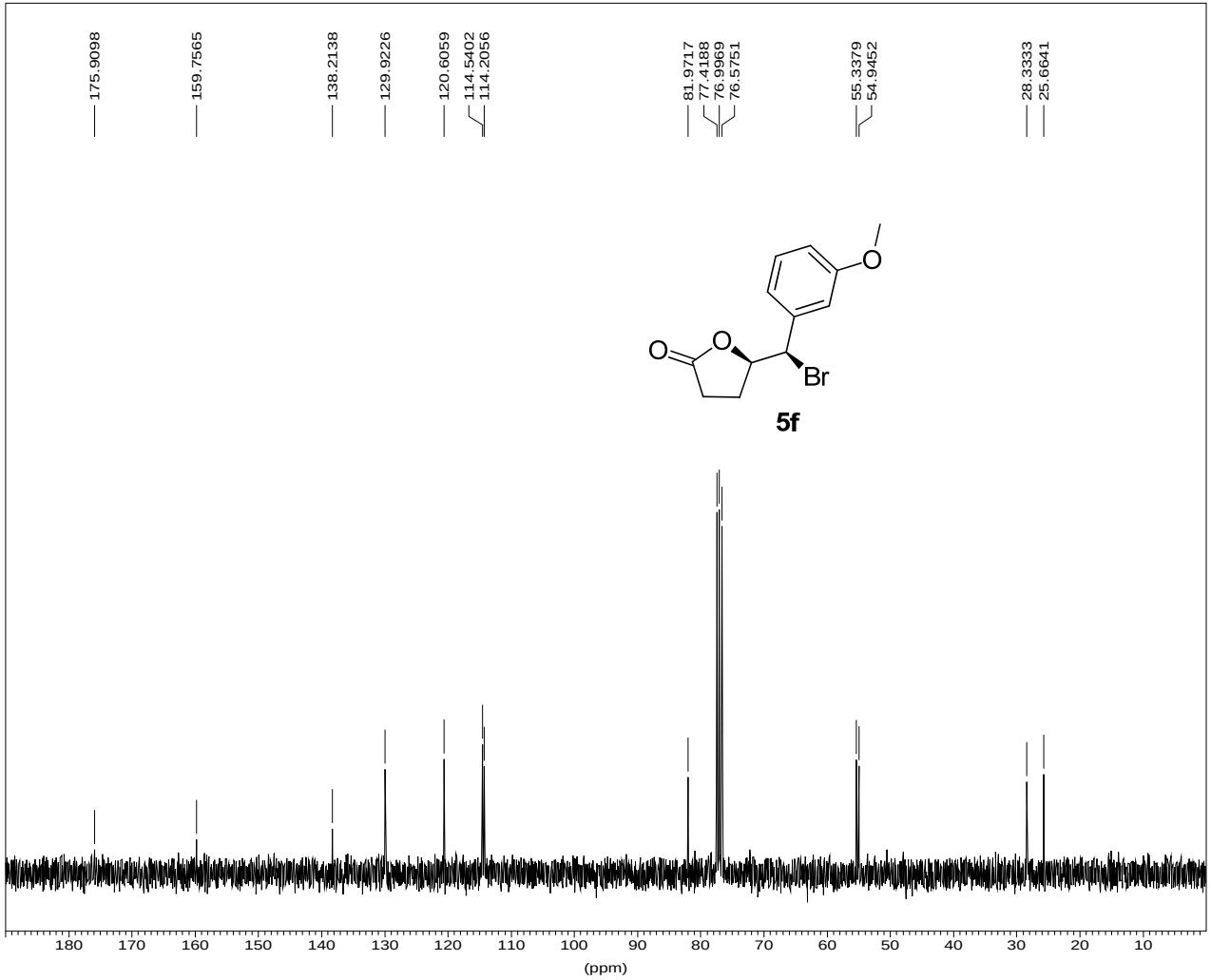
1H normal range AC300 cc-112-A



*** Current Data Parameters ***

NAME	:	ja11cc
EXPNO	:	1
PROCNO	:	1
*** Acquisition Parameters ***		
BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
*** Processing Parameters ***		
LB	:	0.30 Hz
PHC0	:	17.566 degree
PHC1	:	19.220 degree

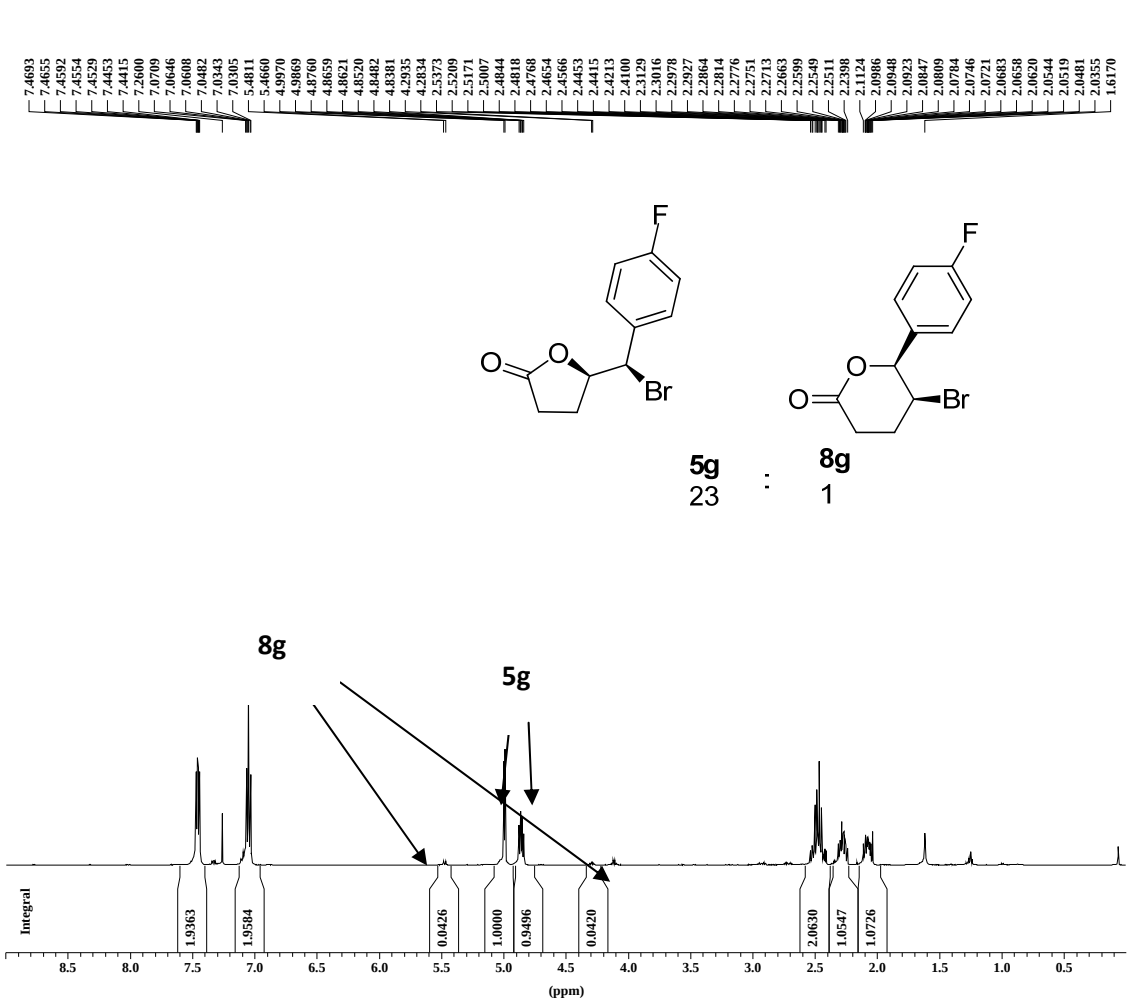
13C Standard AC300 cc-112-A



*** Current Data Parameters ***

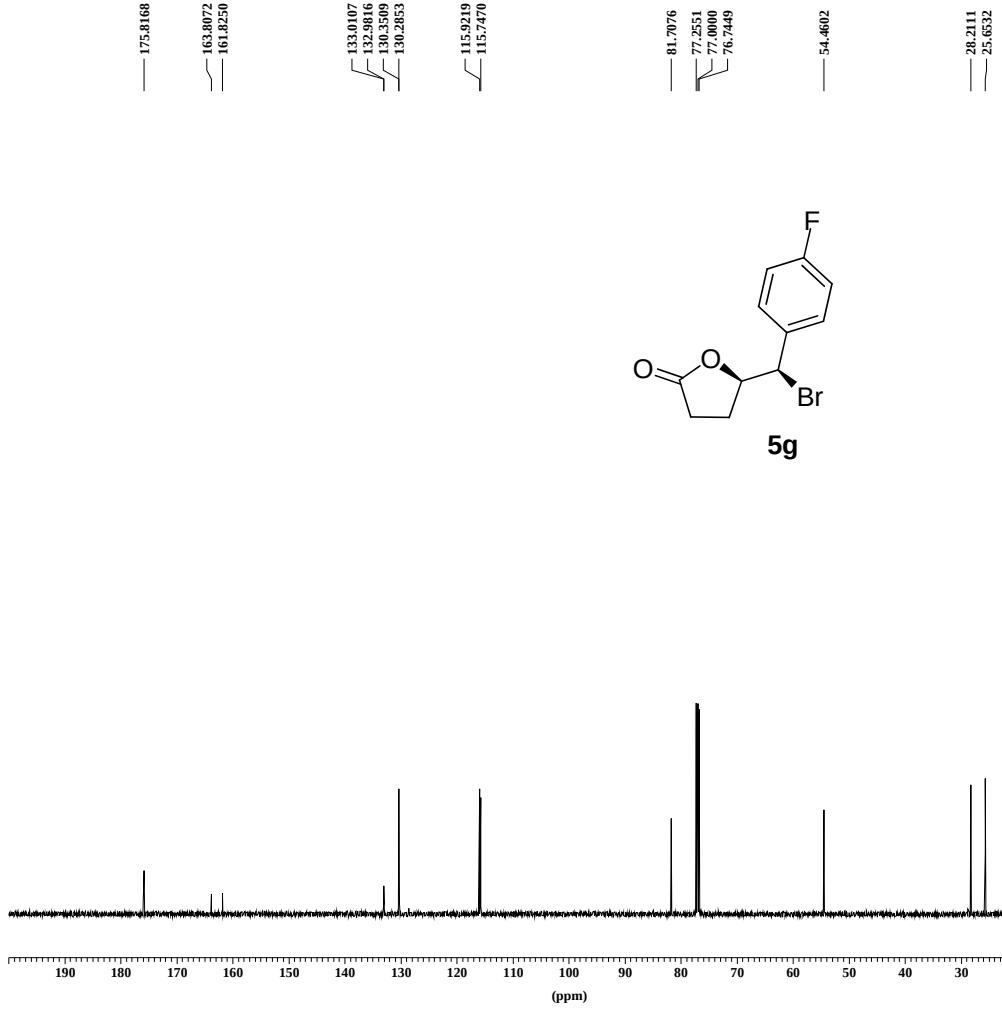
NAME	:	ja11cc
EXPNO	:	2
PROCNO	:	1
*** Acquisition Parameters ***		
BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	131
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm
*** Processing Parameters ***		
LB	:	1.00 Hz
PHC0	:	112.052 degree
PHC1	:	36.544 degree

1H AMX500 cc-i-165



*** Current Data Parameters ***
NAME : ck0213
EXPNO : 2
PROCNO : 1
*** Acquisition Parameters ***
LOCNUC : 2H
NS : 8
NUCLEUS : off
O1 : 3088.51 Hz
PULPROG : zg30
SFO1 : 500.1330885 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm
TD : 32768
TE : 297.9 K
*** Processing Parameters ***
LB : 0.30 Hz
SF : 500.1300140 MHz
*** 1D NMR Plot Parameters ***
NUCLEUS : off

13C AMX500 cc-i-165



*** Current Data Parameters ***

NAME : ck0213

EXPNO : 3

PROCNO : 1

*** Acquisition Parameters ***

LOCNUC : 2H

NS : 227

NUCLEUS : off

O1 : 13204.57 Hz

PULPROG : zgpg30

SFO1 : 125.7709936 MHz

SOLVENT : CDCl3

SW : 238.7675 ppm

TD : 65536

TE : 298.3 K

*** Processing Parameters ***

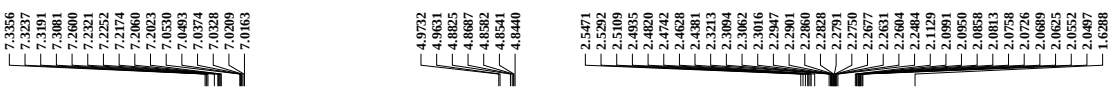
LB : 1.00 Hz

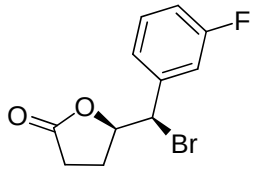
SF : 125.7577950 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS : off

cc-i-167b





5h

*** Current Data Parameters ***

NAME	:	ck0214
EXPNO	:	3
PROCNO	:	1

*** Acquisition Parameters ***

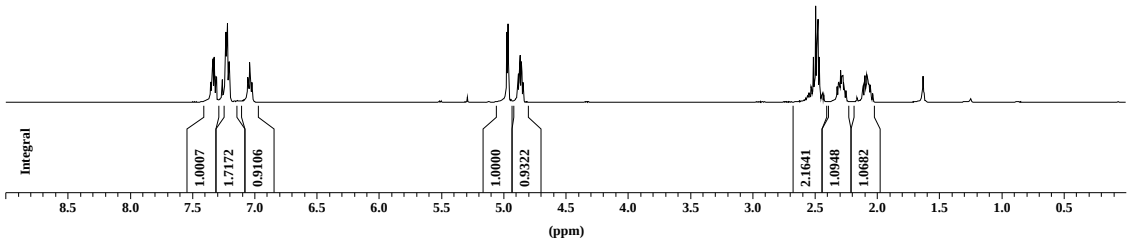
LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	2751.27 Hz
PULPROG	:	zg
SFO1	:	500.2327513 MHz
SOLVENT	:	CDCl3
SW	:	15.0080 ppm
TD	:	32768
TE	:	300.0 K

*** Processing Parameters ***

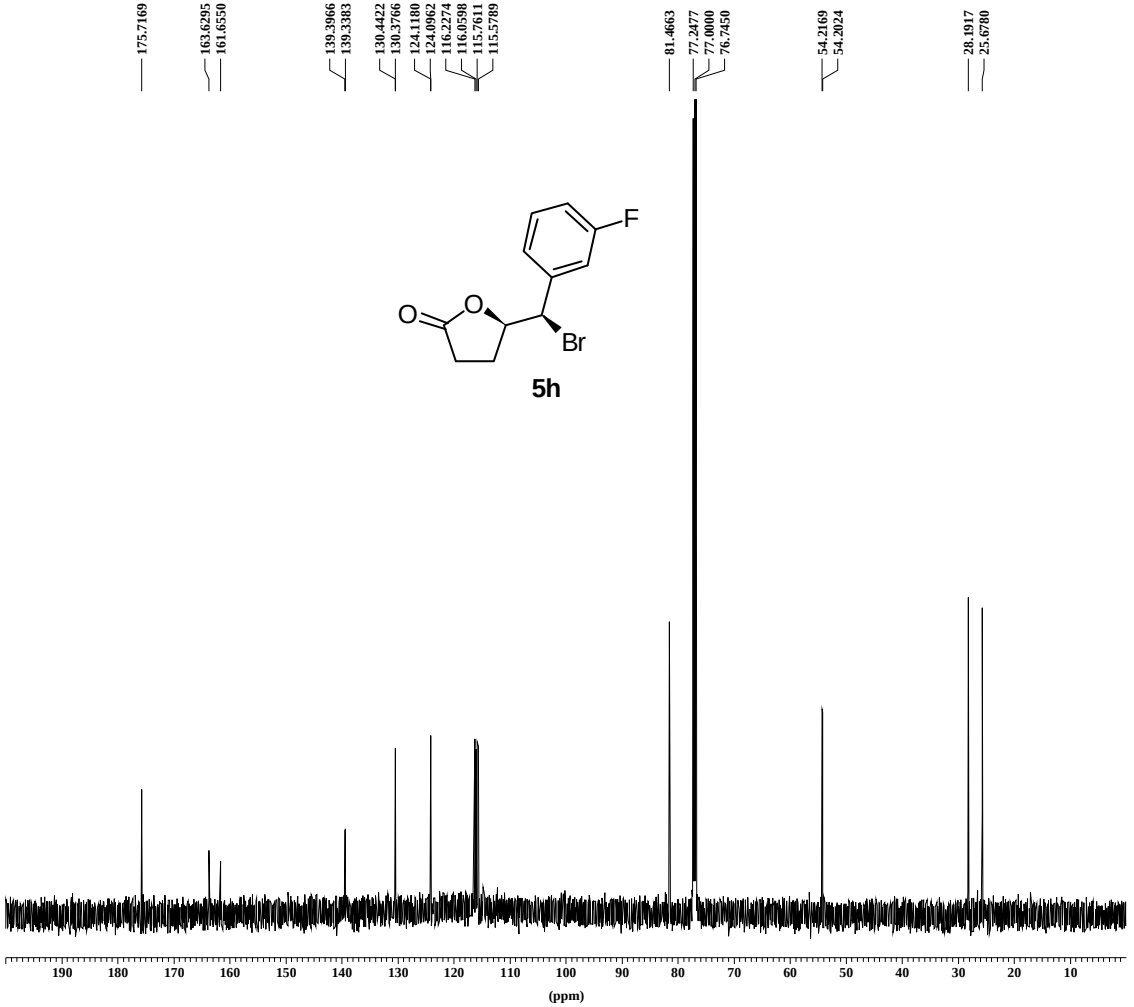
LB	:	0.10 Hz
SF	:	500.2300090 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off



cc-i-167b



*** Current Data Parameters ***

NAME : ck0214

EXPNO : 4

PROCNO : 1

*** Acquisition Parameters ***

LOCNUC : 2H

NS : 309

NUCLEUS : off

O1 : 12577.84 Hz

PULPROG : zgpg30

SFO1 : 125.7955118 MHz

SOLVENT : CDCl3

SW : 238.7210 ppm

TD : 65536

TE : 300.0 K

*** Processing Parameters ***

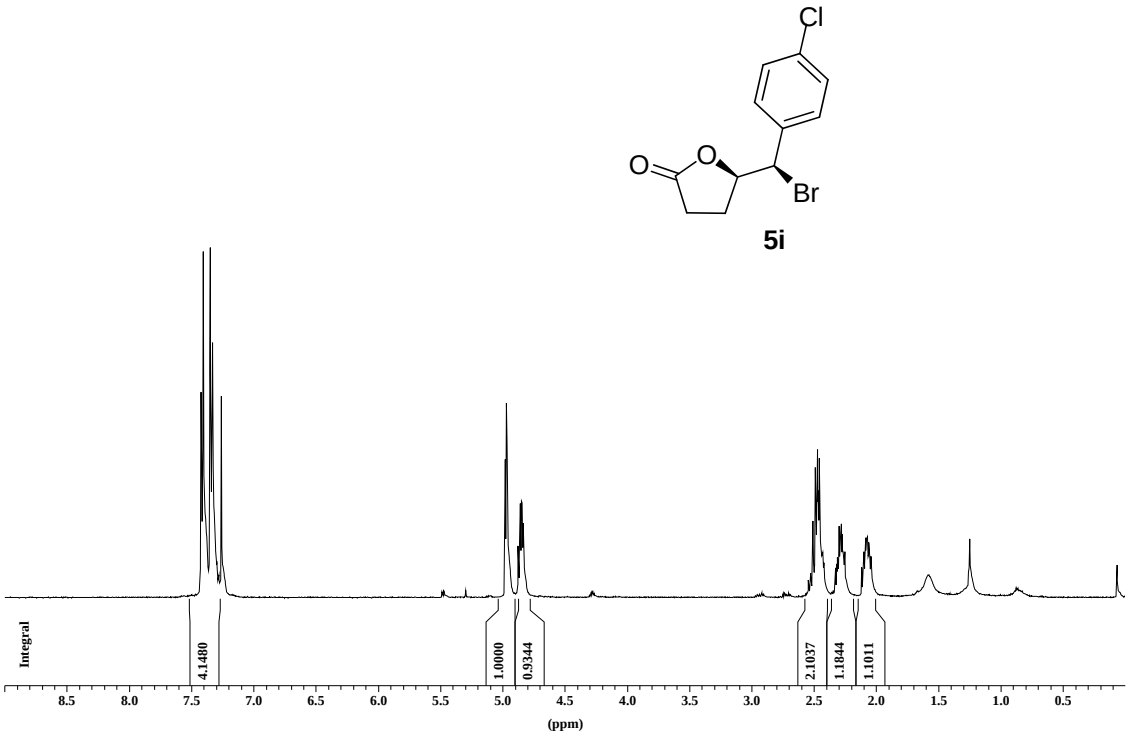
LB : 1.00 Hz

SF : 125.7829400 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS : off

1H AMX500 cc-i-104c



***** Current Data Parameters *****

NAME	:	ck0208
EXPNO	:	5
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	3088.51 Hz
PULPROG	:	zg30
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768
TE	:	295.3 K

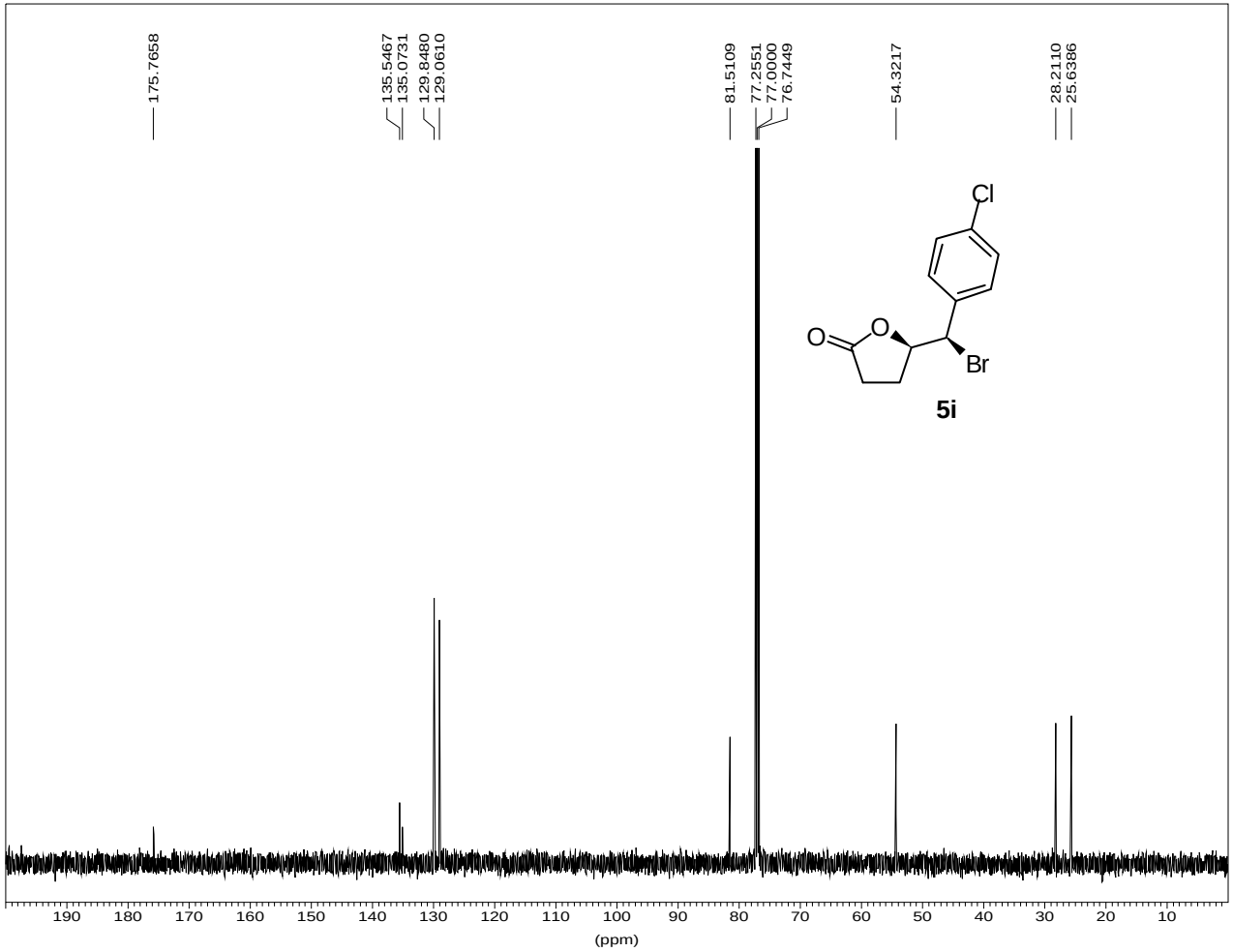
***** Processing Parameters *****

LB	:	0.30 Hz
SF	:	500.1300140 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

13C AMX500 cc-i-104c



*** Current Data Parameters ***

NAME	:	ck0208
EXPNO	:	6
PROCNO	:	1

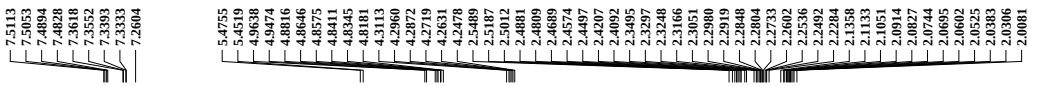
*** Acquisition Parameters ***

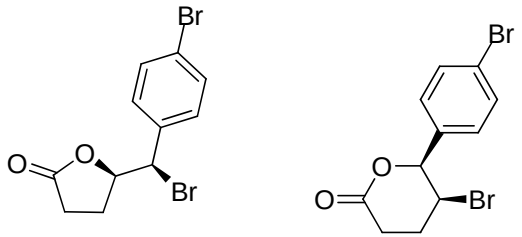
BF1	:	125.7577890 MHz
LOCNUC	:	2H
NS	:	210
O1	:	13204.57 Hz
PULPROG	:	zgpg30
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	-46.560 degree
PHC1	:	17.922 degree

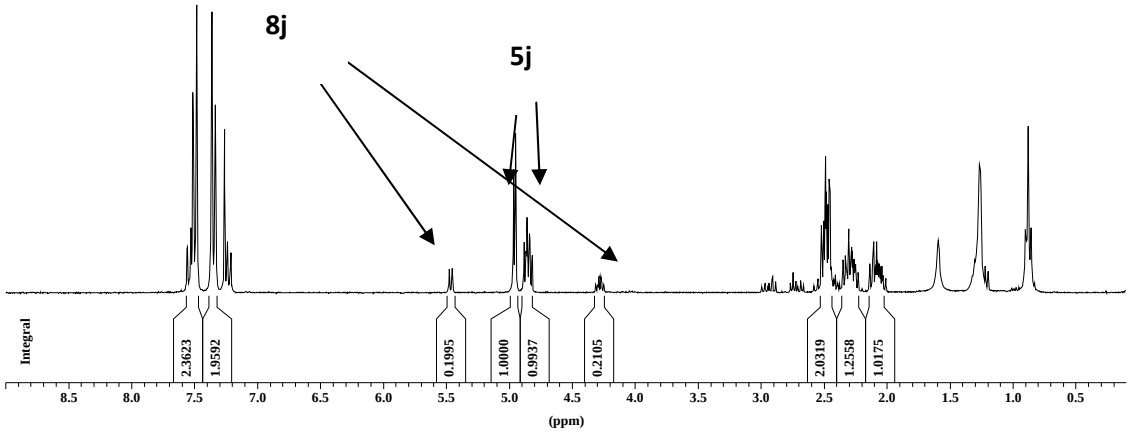
1H normal range AC300 cc-i-111





5j : **8j**

5 : 1



***** Current Data Parameters *****

NAME	:	de30lcc
EXPNO	:	8
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768
TE	:	678.6 K

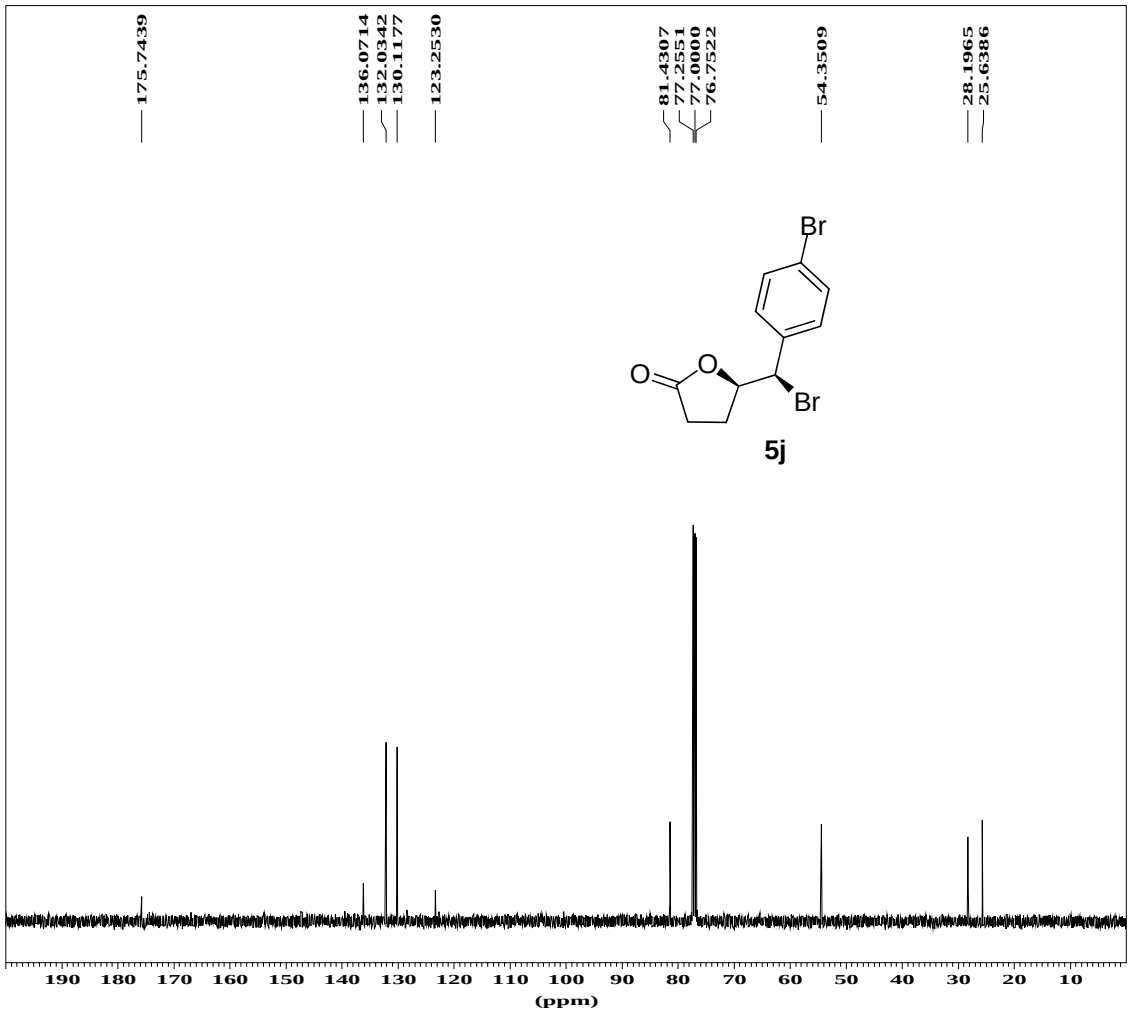
***** Processing Parameters *****

LB	:	0.30 Hz
SF	:	300.1300123 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

13C AMX500 4-Br lactone



***** Current Data Parameters *****

NAME	:	ck0214
EXPNO	:	8
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	136
NUCLEUS	:	off
O1	:	13204.57 Hz
PULPROG	:	zgpg30
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm
TD	:	65536
TE	:	298.3 K

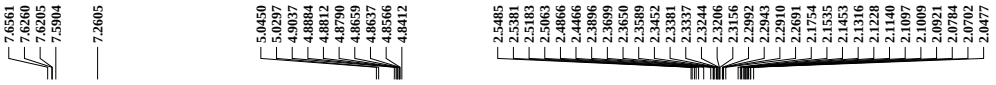
***** Processing Parameters *****

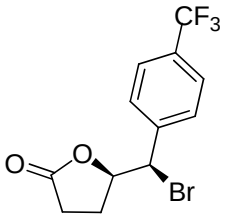
LB	:	1.00 Hz
SF	:	125.7577930 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

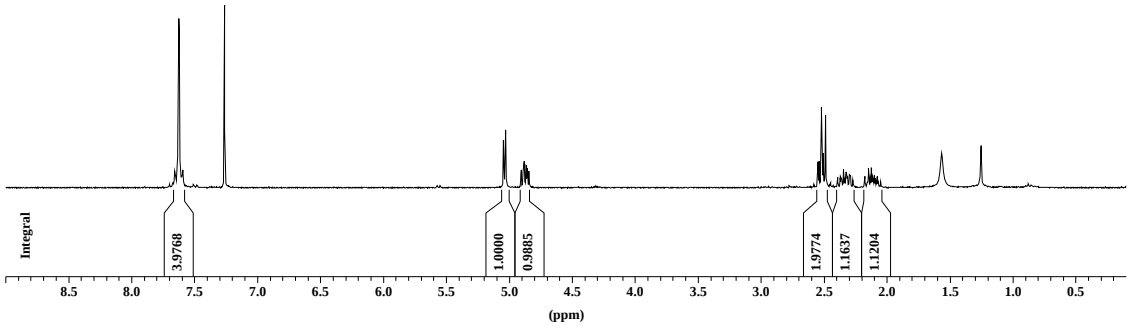
1H normal range AC300 cc-112-B



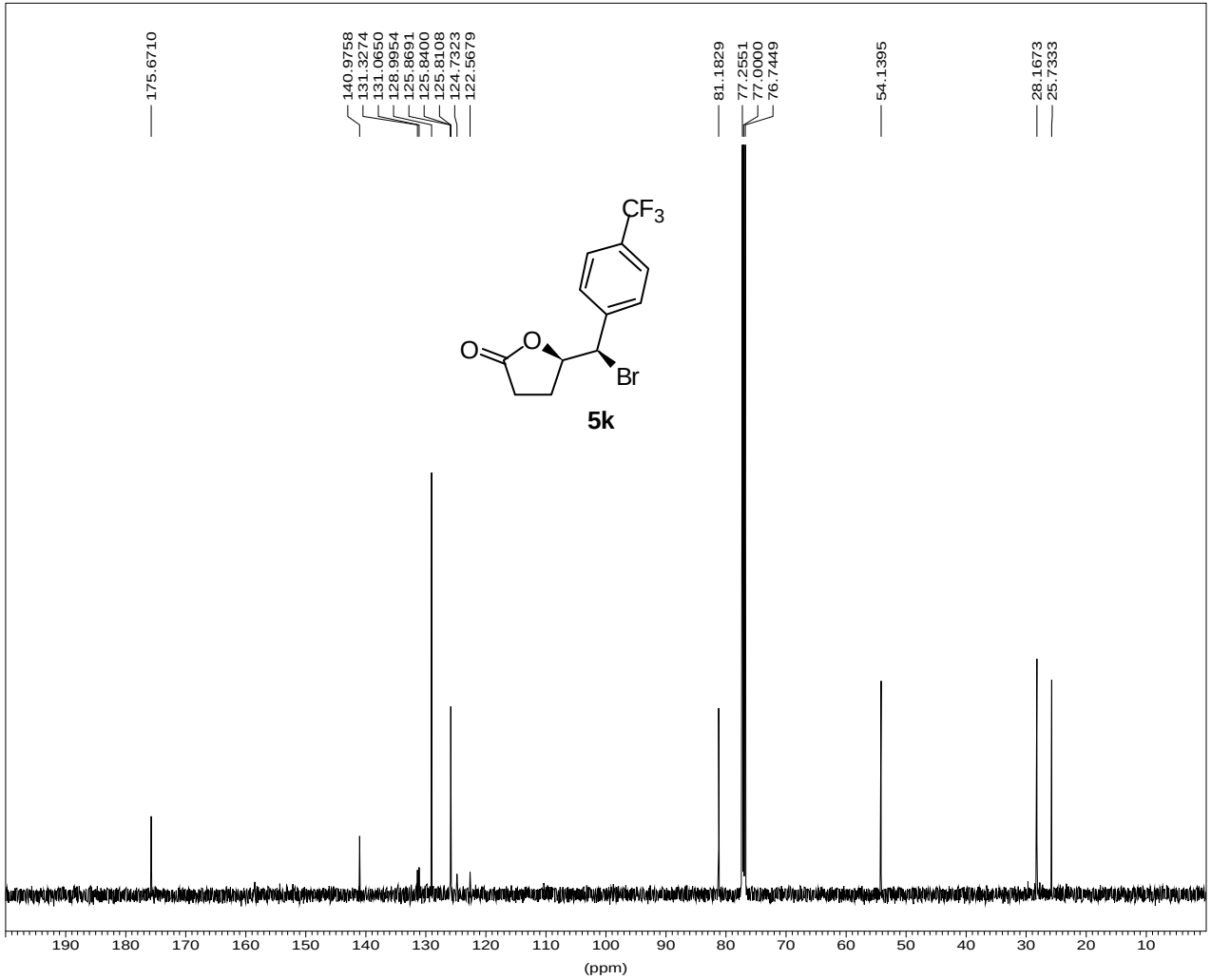


5k

*** Current Data Parameters ***
NAME : ja05lcc
EXPNO : 4
PROCNO : 1
*** Acquisition Parameters ***
LOCNUC : 2H
NS : 8
NUCLEUS : off
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm
TD : 32768
TE : 678.6 K
*** Processing Parameters ***
LB : 0.30 Hz
SF : 300.1300122 MHz
*** 1D NMR Plot Parameters ***
NUCLEUS : off



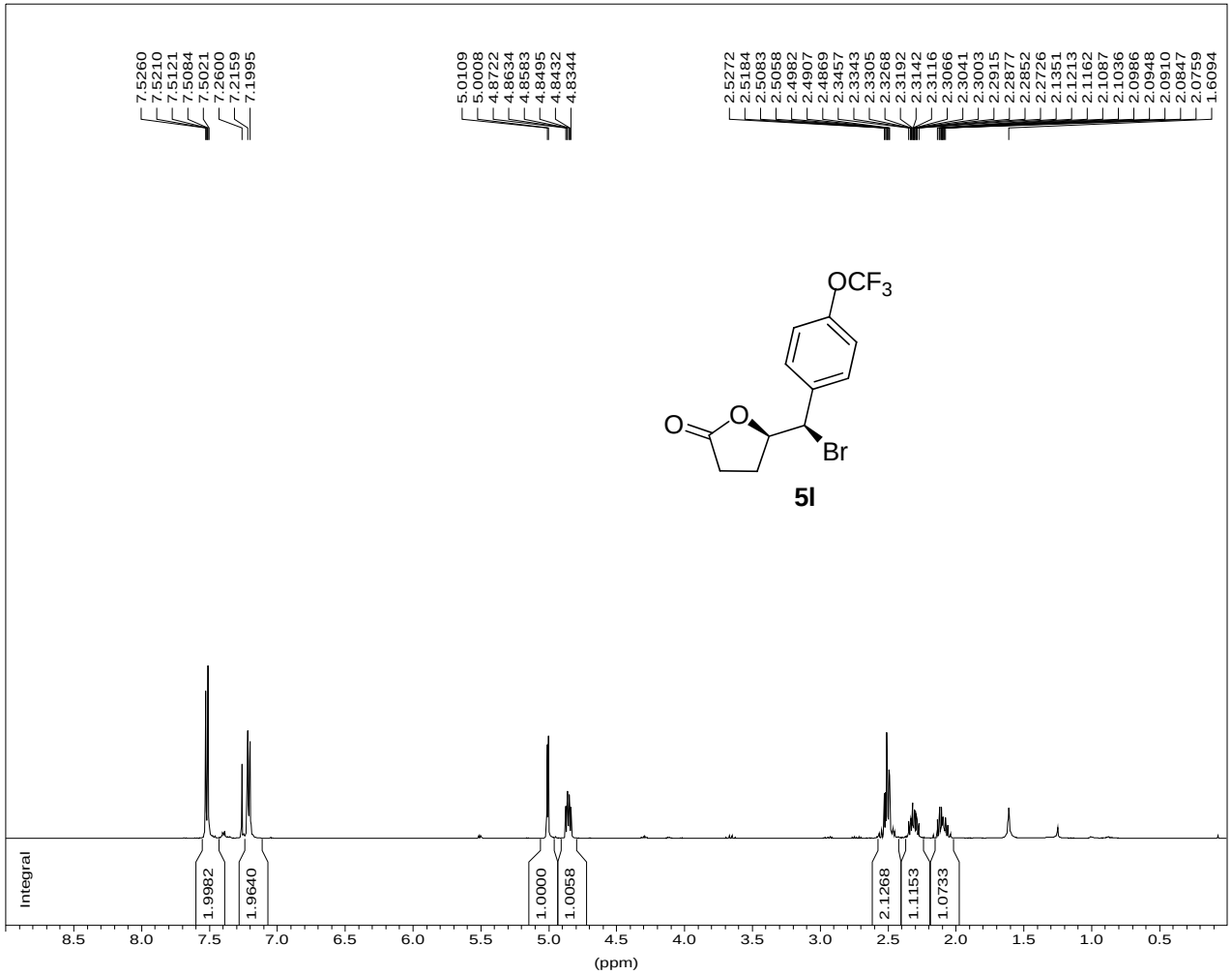
13C AMX500 cc-i-166a



*** Current Data Parameters ***

NAME	:	ck0210
EXPNO	:	2
PROCNO	:	1
*** Acquisition Parameters ***		
BF1	:	125.7577890 MHz
LOCNUC	:	2H
NS	:	499
O1	:	13204.57 Hz
PULPROG	:	zgpg30
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm
*** Processing Parameters ***		
LB	:	1.00 Hz
PHC0	:	305.067 degree
PHC1	:	41.844 degree

1H AMX500 cc-i-166b



*** Current Data Parameters ***

NAME	:	ck0210
EXPNO	:	3
PROCNO	:	1

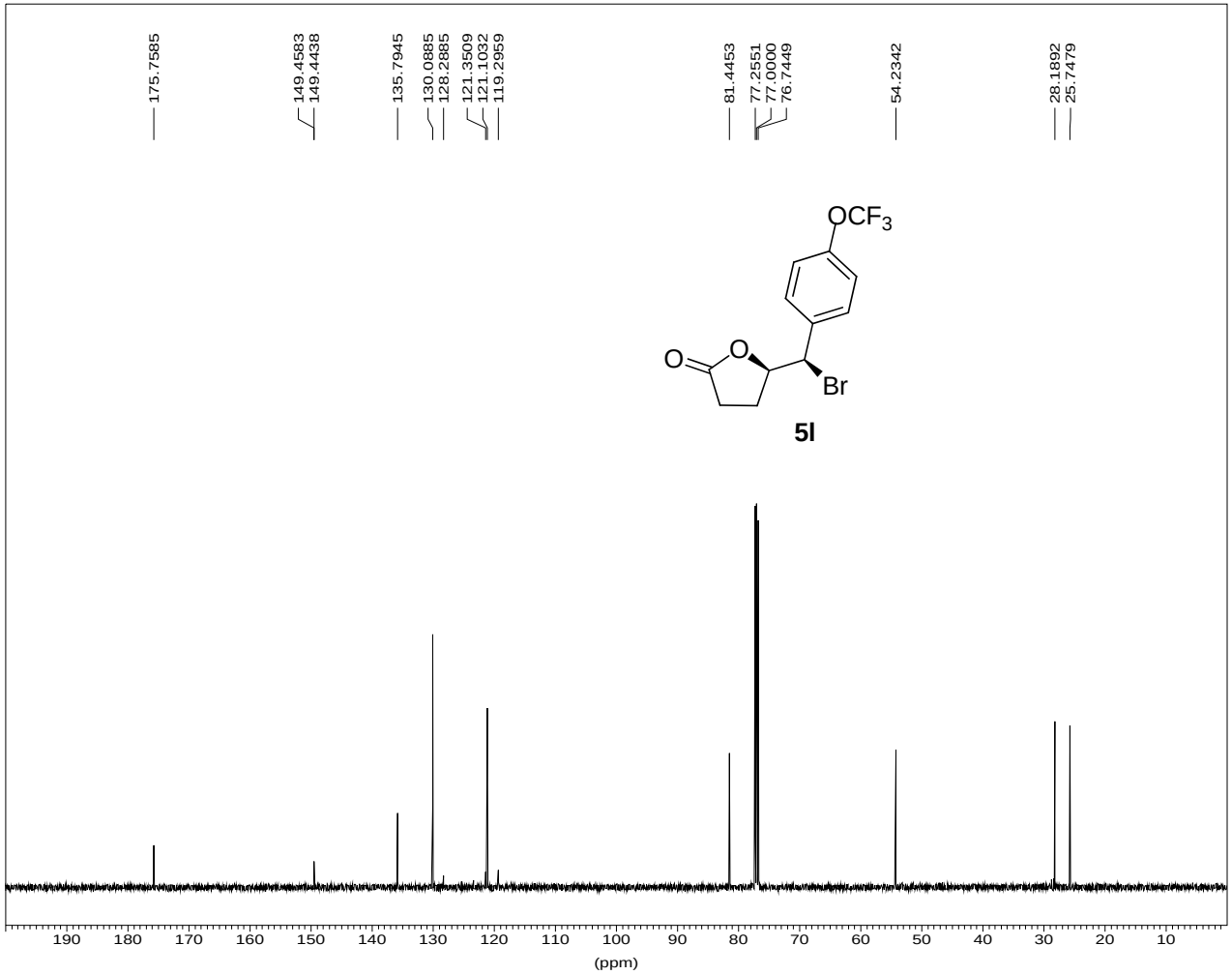
*** Acquisition Parameters ***

BF1	:	500.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	3088.51 Hz
PULPROG	:	zg30
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm

*** Processing Parameters ***

LB	:	0.30 Hz
PHC0	:	274.235 degree
PHC1	:	1.980 degree

13C AMX500 cc-i-166b



*** Current Data Parameters ***

NAME	:	ck0210
EXPNO	:	4
PROCNO	:	1

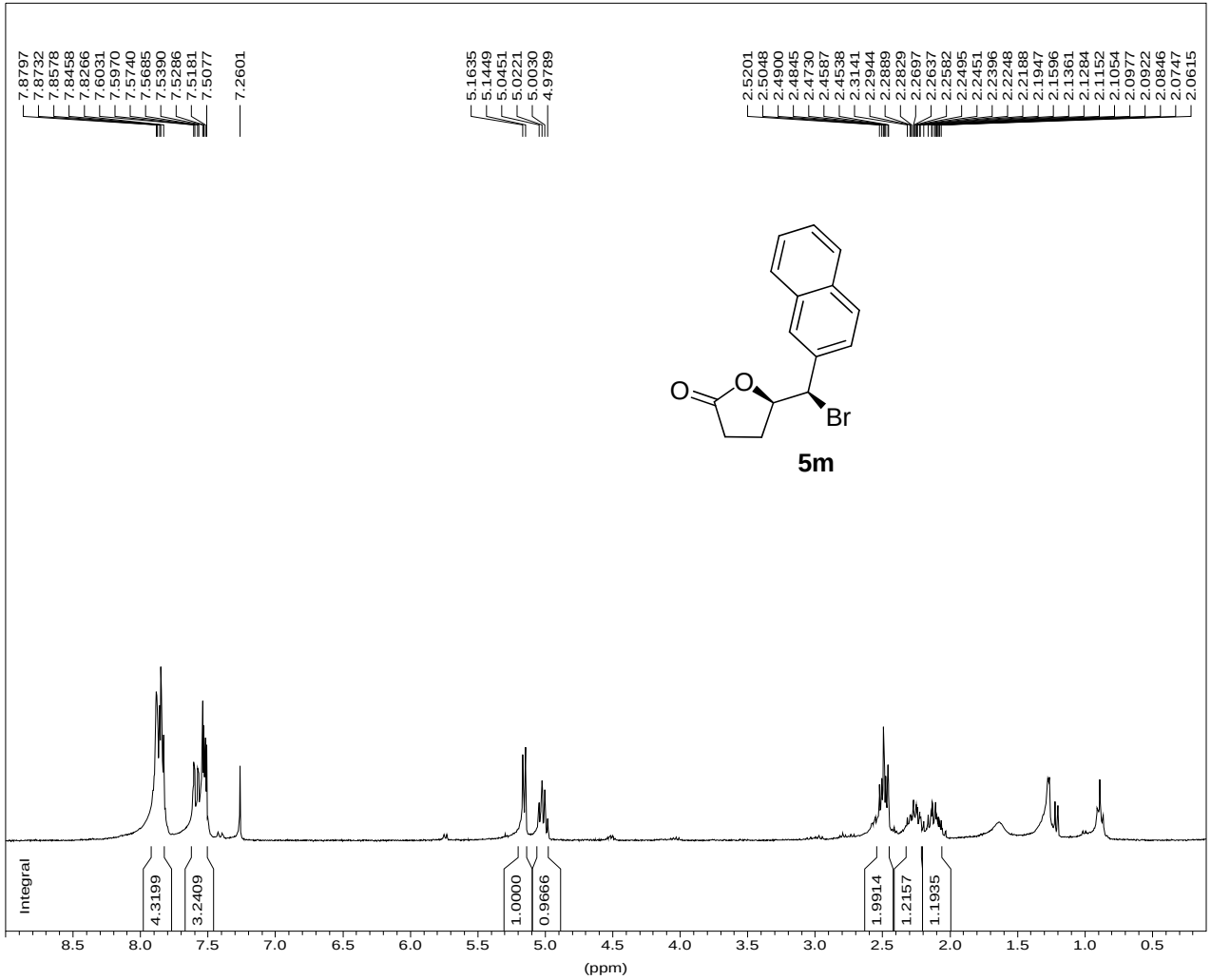
*** Acquisition Parameters ***

BF1	:	125.7577890 MHz
LOCNUC	:	2H
NS	:	382
O1	:	13204.57 Hz
PULPROG	:	zgpg30
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	315.176 degree
PHC1	:	24.463 degree

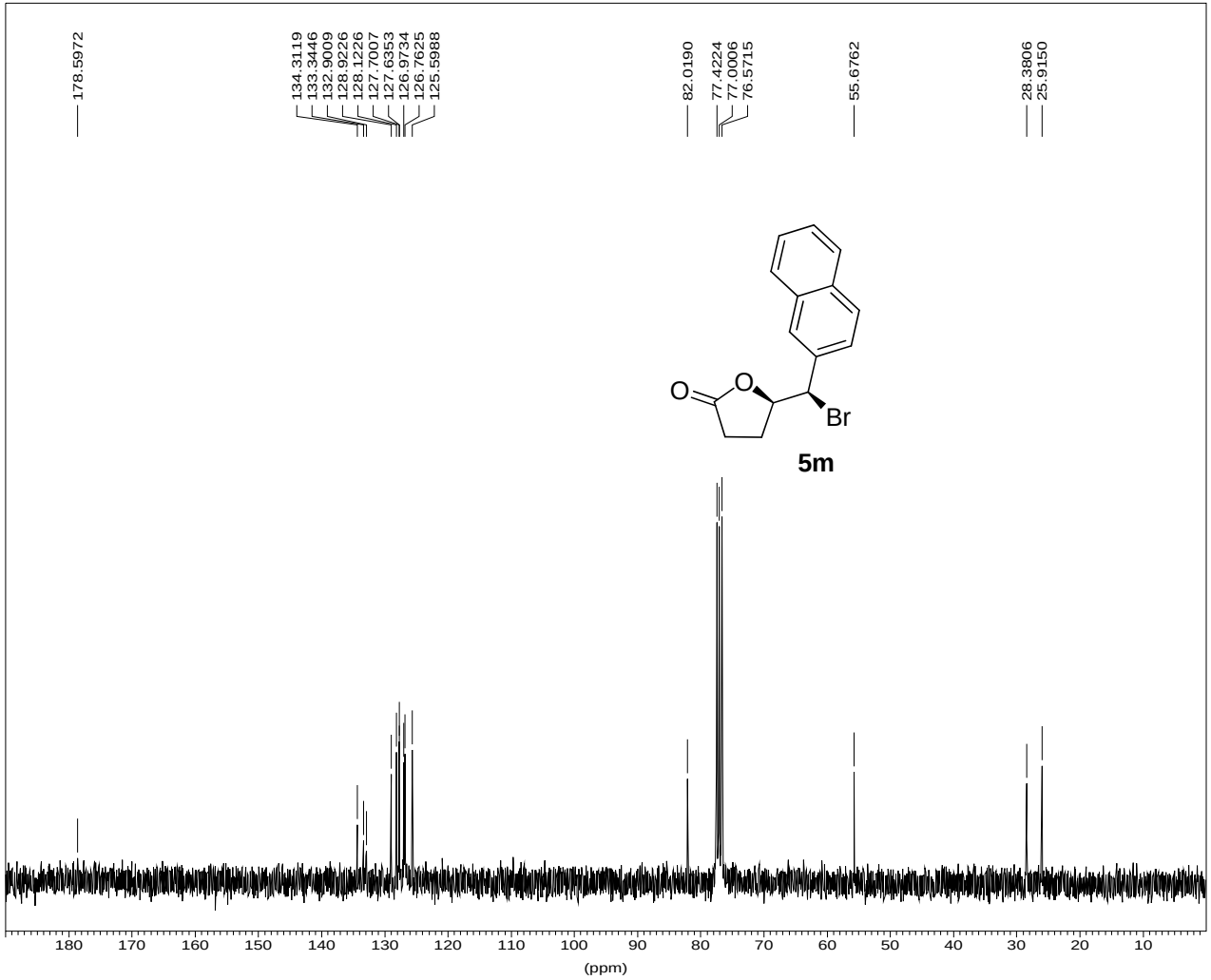
1H normal range AC300 cc-i-112-c



*** Current Data Parameters ***

NAME	:	ja06lcc
EXPNO	:	1
PROCNO	:	1
*** Acquisition Parameters ***		
BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
*** Processing Parameters ***		
LB	:	0.30 Hz
PHC0	:	29.363 degree
PHC1	:	3.159 degree

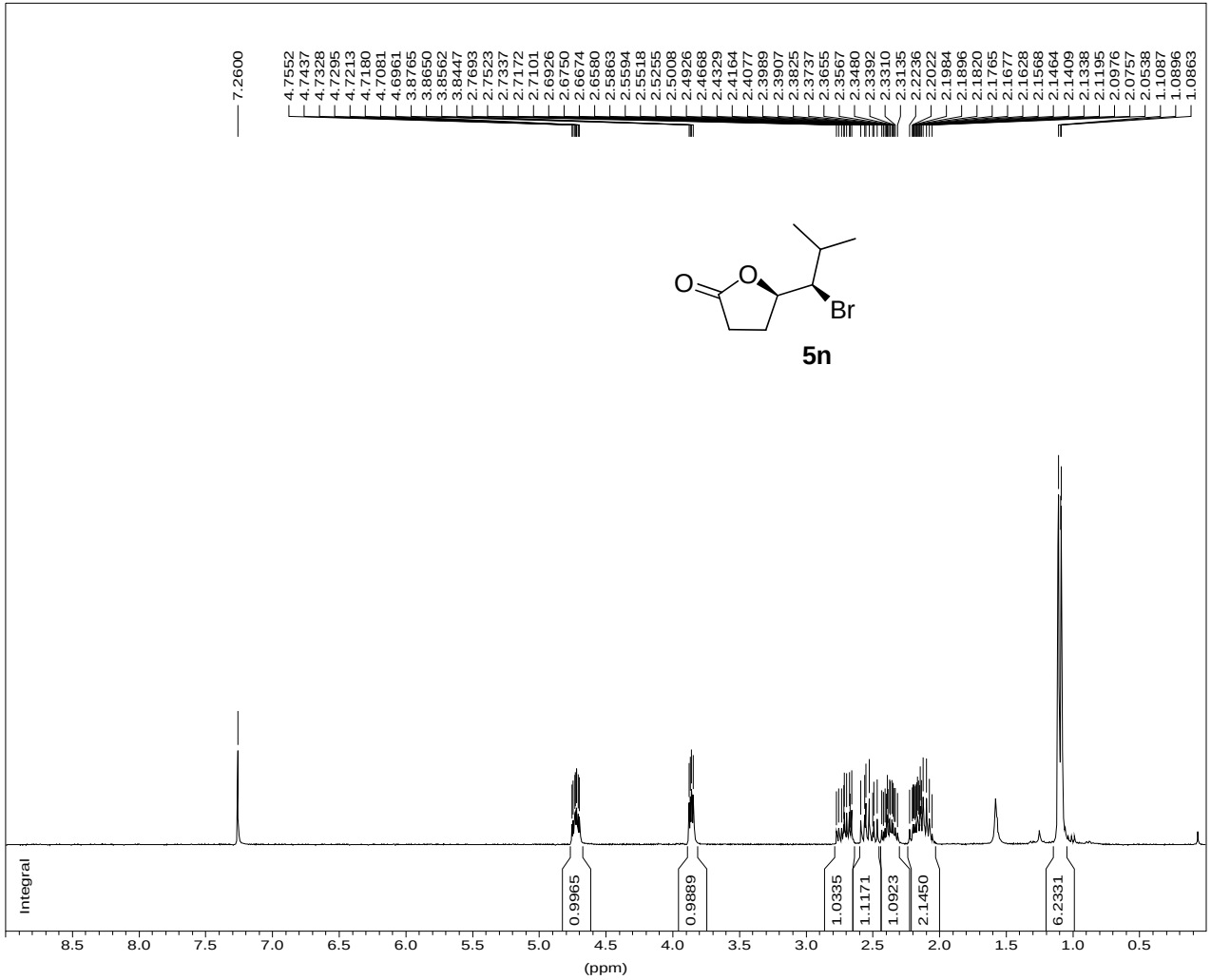
13C Standard AC300 cc-i-112-c



*** Current Data Parameters ***

NAME	:	ja06lcc
EXPNO	:	2
PROCNO	:	1
*** Acquisition Parameters ***		
BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	355
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm
*** Processing Parameters ***		
LB	:	1.00 Hz
PHC0	:	142.329 degree
PHC1	:	-8.112 degree

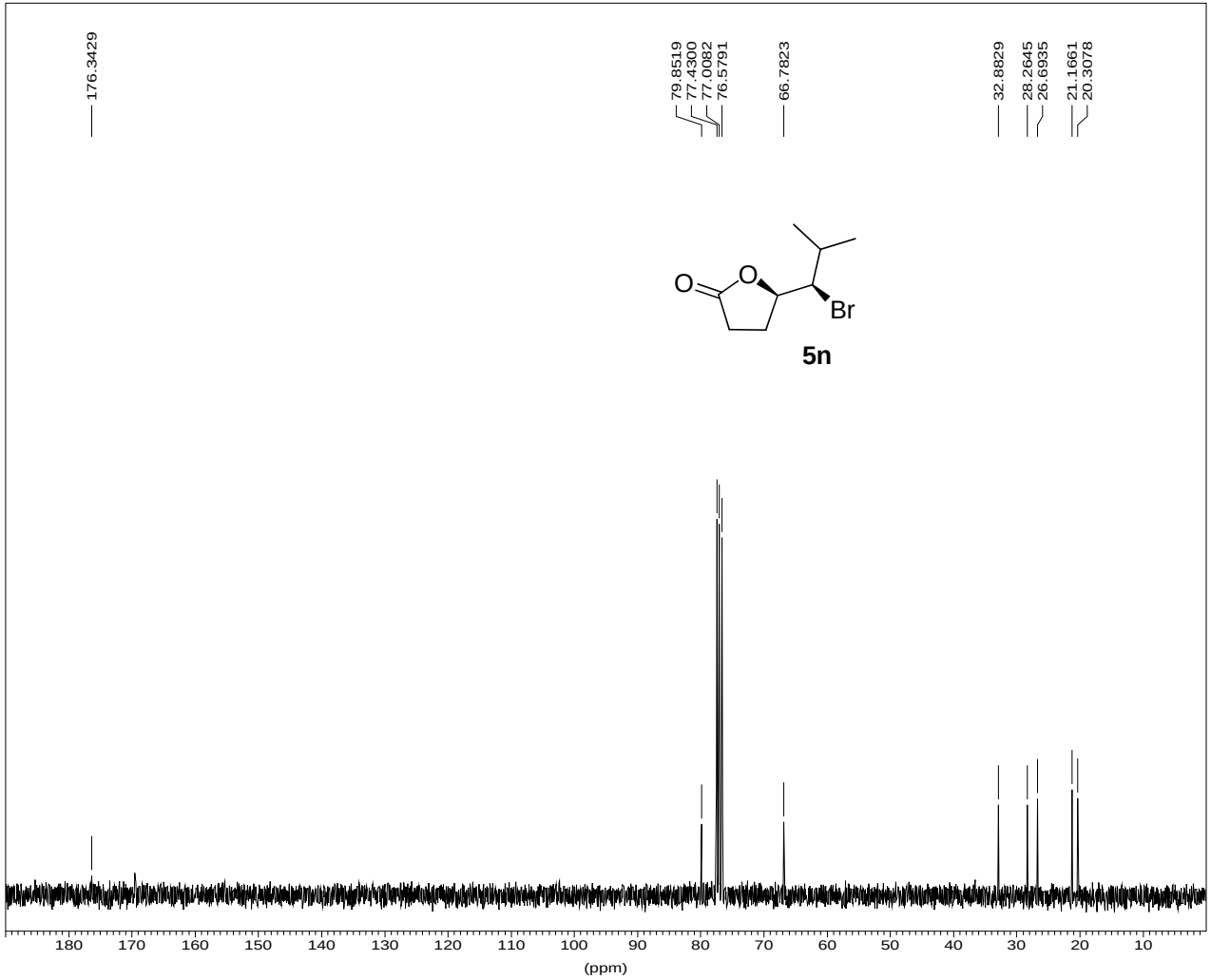
1H normal range AC300 cc-118-C



*** Current Data Parameters ***

NAME	:	ja11cc
EXPNO	:	5
PROCNO	:	1
*** Acquisition Parameters ***		
BF1	:	300.1300000 MHz
LOCNUC	:	2H
NS	:	8
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
*** Processing Parameters ***		
LB	:	0.30 Hz
PHC0	:	39.341 degree
PHC1	:	-10.890 degree

13C Standard AC300 cc-118-C



*** Current Data Parameters ***

NAME	:	ja11cc
EXPNO	:	6
PROCNO	:	1

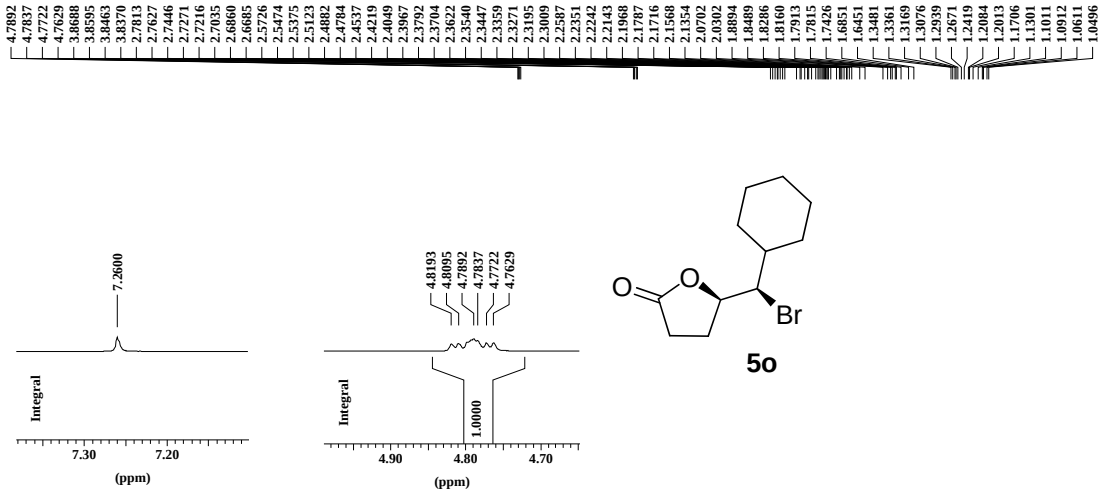
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	301
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	106.061 degree
PHC1	:	31.741 degree

1H normal range AC300 cc-i-118-D



*** Current Data Parameters ***

NAME	:	ja11cc
EXPNO	:	3
PROCNO	:	1

*** Acquisition Parameters ***

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	CDCl3
SW	:	17.9519 ppm
TD	:	32768
TE	:	297.0 K

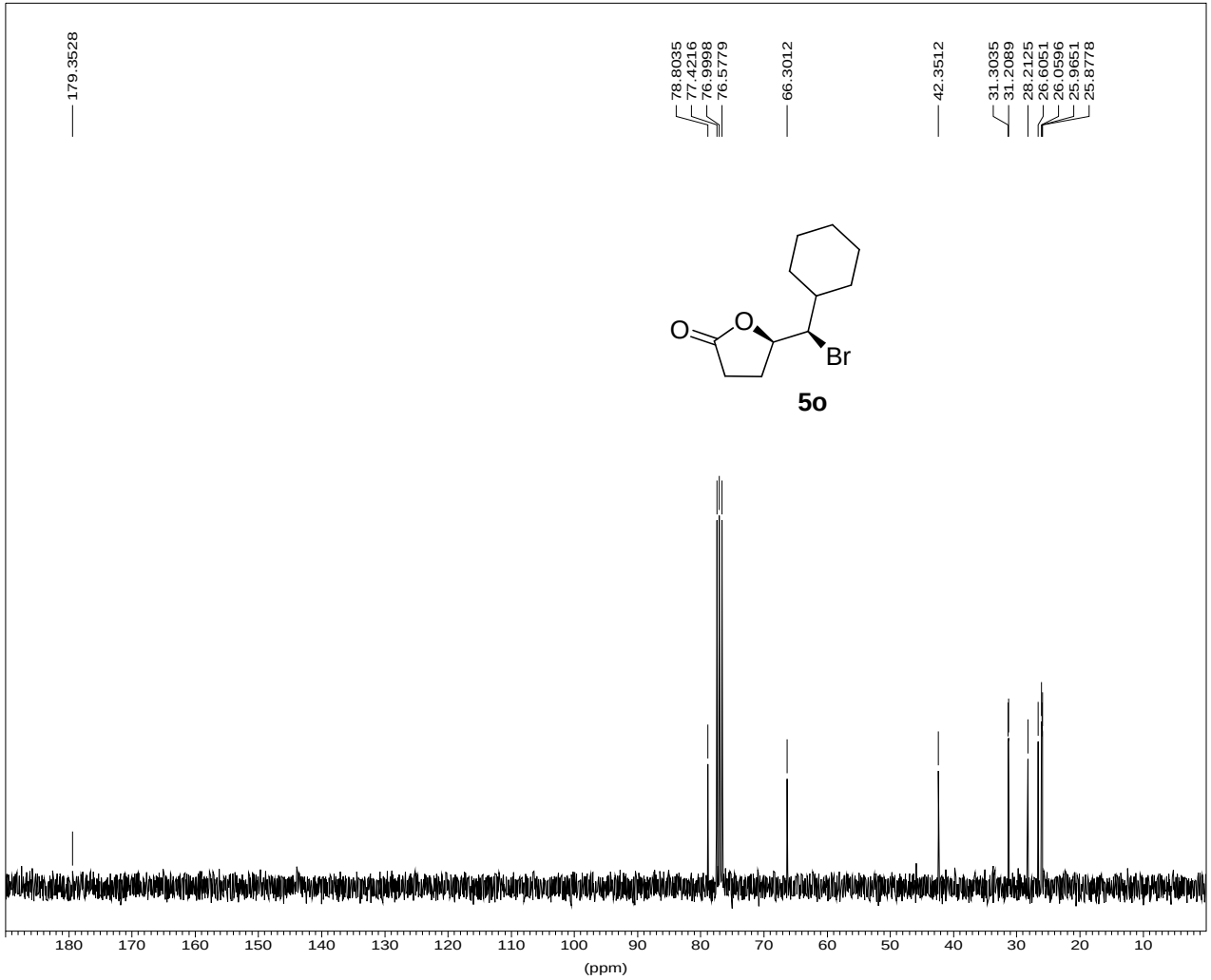
*** Processing Parameters ***

LB	:	0.30 Hz
SF	:	300.1300130 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C Standard AC300 cc-118-C



*** Current Data Parameters ***

NAME	:	ja11cc
EXPNO	:	4
PROCNO	:	1

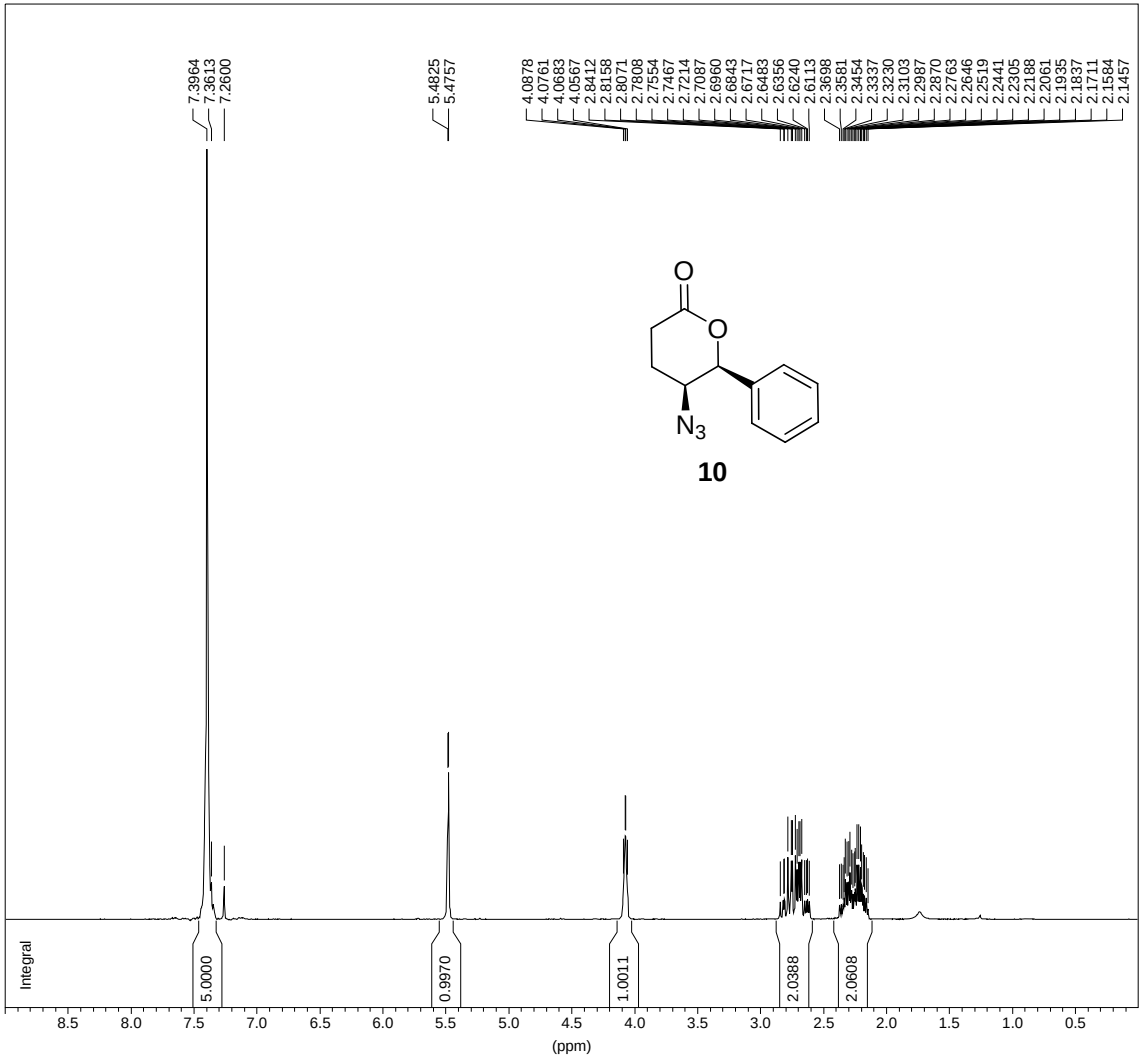
*** Acquisition Parameters ***

BF1	:	75.4677490 MHz
LOCNUC	:	2H
NS	:	158
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	CDCl3
SW	:	238.2968 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
PHC0	:	108.472 degree
PHC1	:	38.307 degree

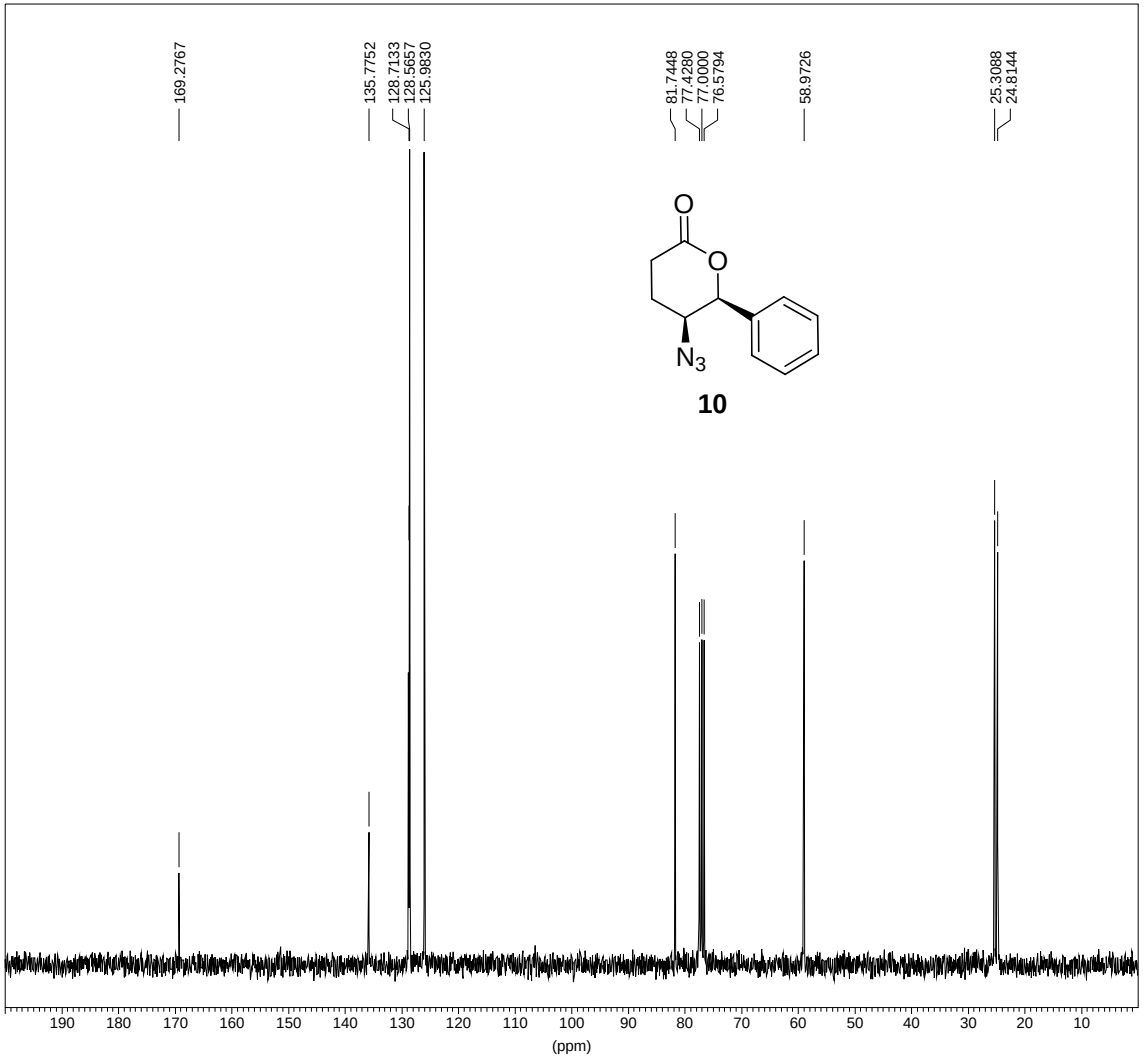
ck-ii-134



*** Current Data Parameters ***

NAME	:	se10ck
EXPNO	:	1
PROCNO	:	1
*** Acquisition Parameters ***		
INSTRUM	:	spect
LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1250.00 Hz
PULPROG	:	zg30
SFO1	:	300.1312500 MHz
SOLVENT	:	D2O
SW	:	15.9573 ppm
TD	:	32768
TE	:	299.2 K
*** Processing Parameters ***		
LB	:	0.10 Hz
*** 1D NMR Plot Parameters ***		
NUCLEUS	:	off

ck-ii-134



*** Current Data Parameters ***

NAME	:	se10ck
EXPNO	:	5
PROCNO	:	1

*** Acquisition Parameters ***

INSTRUM	:	spect
LOCNUC	:	2H
NS	:	52
NUCLEUS	:	off
O1	:	8365.03 Hz
PULPROG	:	zgdc
SFO1	:	75.4760840 MHz
SOLVENT	:	CDCl3
SW	:	241.7743 ppm
TD	:	32768
TE	:	299.2 K

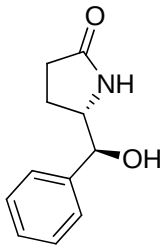
*** Processing Parameters ***

LB	:	2.00 Hz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

1H normal range AC300 ck-ii-215



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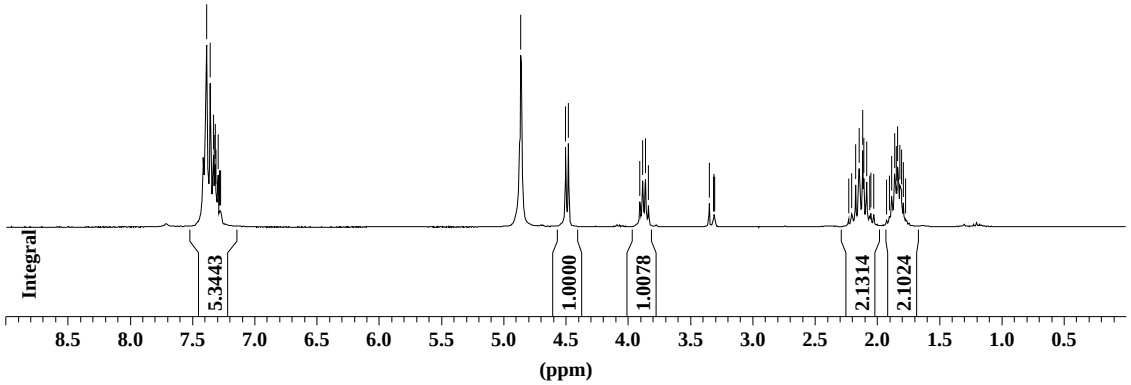
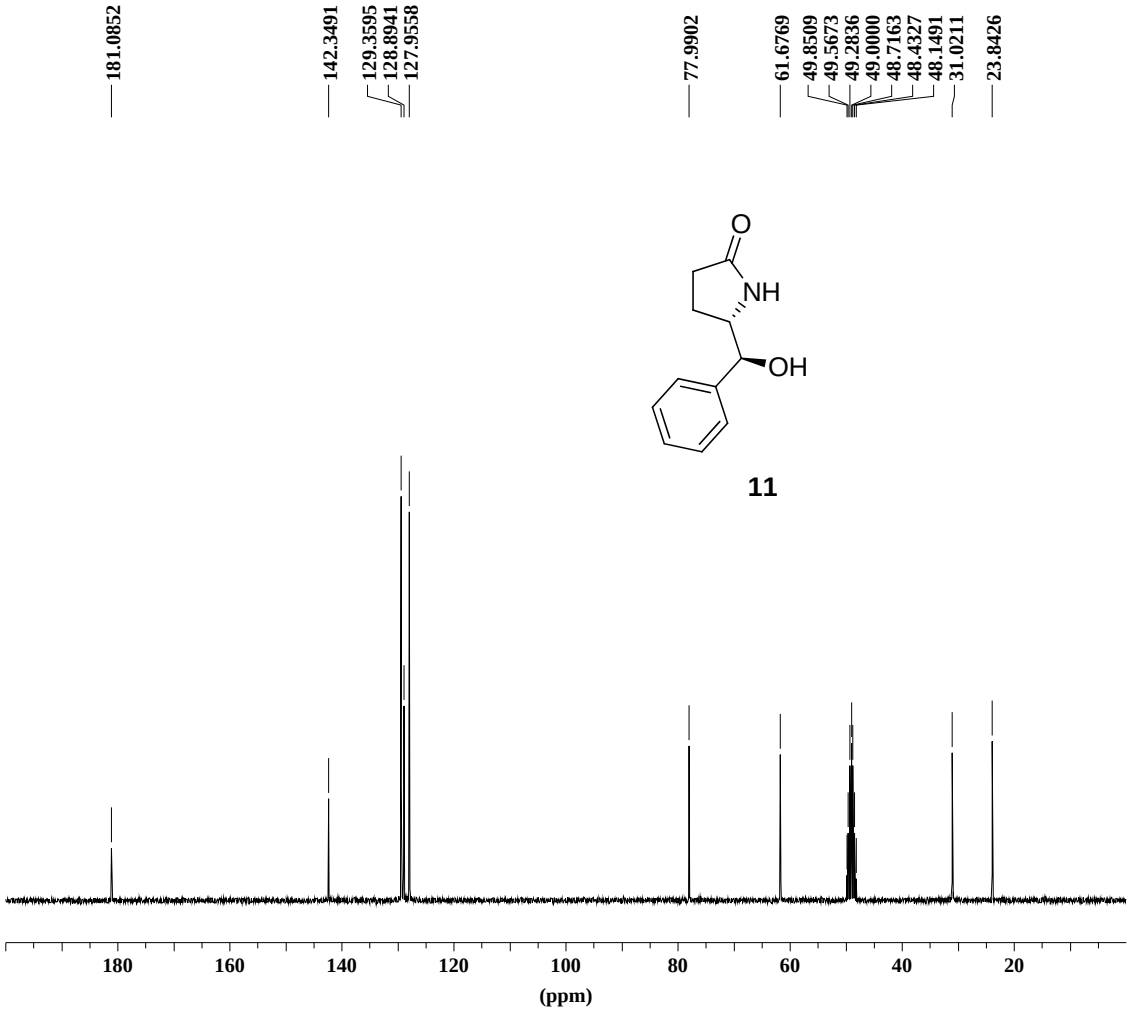
*** Current Data Parameters ***
NAME : oc23ck
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
LOCNUC : 2H
NS : 8
NUCLEUS : off
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : MeOD
SW : 17.9519 ppm
TD : 32768
TE : 297.4 K
*** Processing Parameters ***
LB : 0.30 Hz
SF : 300.1300050 MHz
*** 1D NMR Plot Parameters ***
NUCLEUS : off



13C Standard AC300 ck-ii-215



*** Current Data Parameters ***

NAME	:	oc23ck
EXPNO	:	2
PROCNO	:	1

*** Acquisition Parameters ***

LOCNUC	:	2H
NS	:	202
NUCLEUS	:	off
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	MeOD
SW	:	238.2968 ppm
TD	:	32768
TE	:	297.5 K

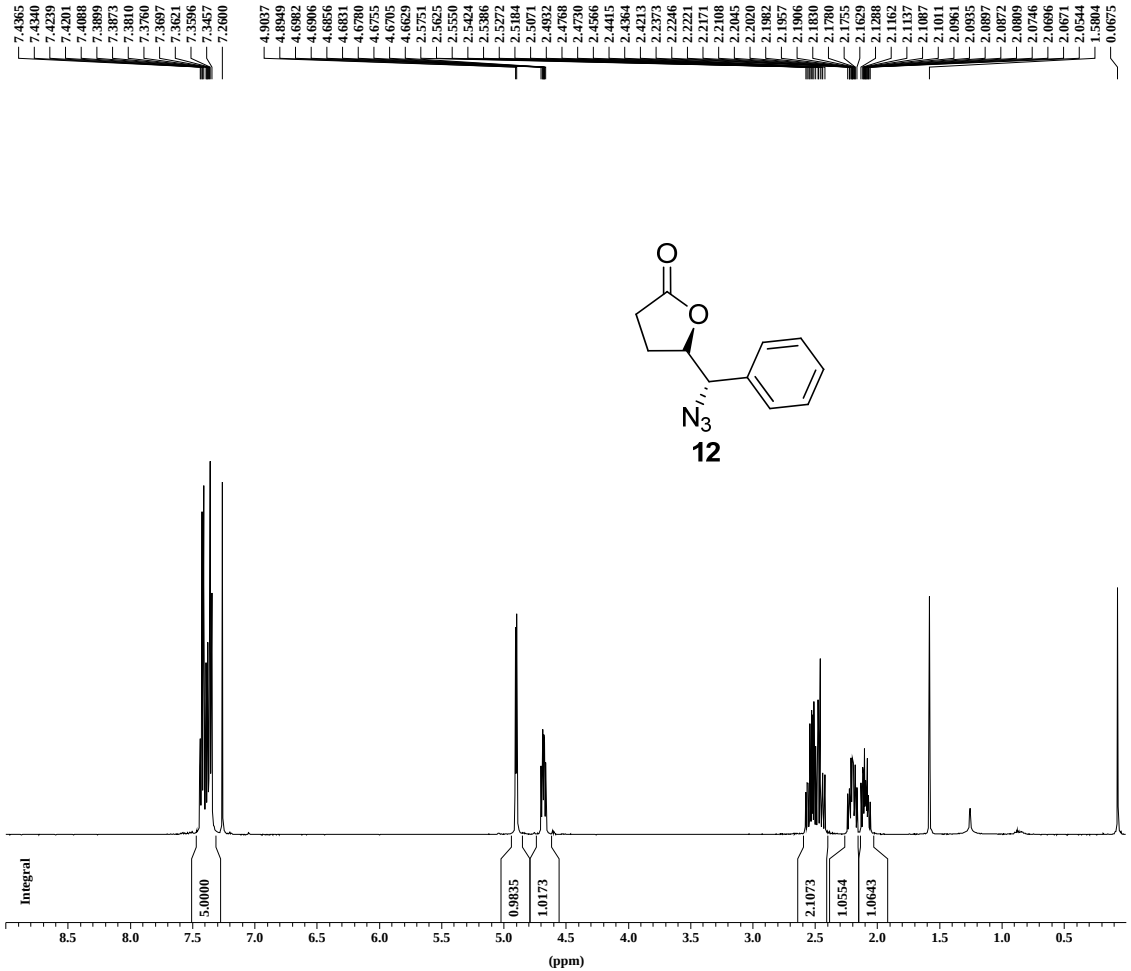
*** Processing Parameters ***

LB	:	1.00 Hz
SF	:	75.4676520 MHz

*** 1D NMR Plot Parameters ***

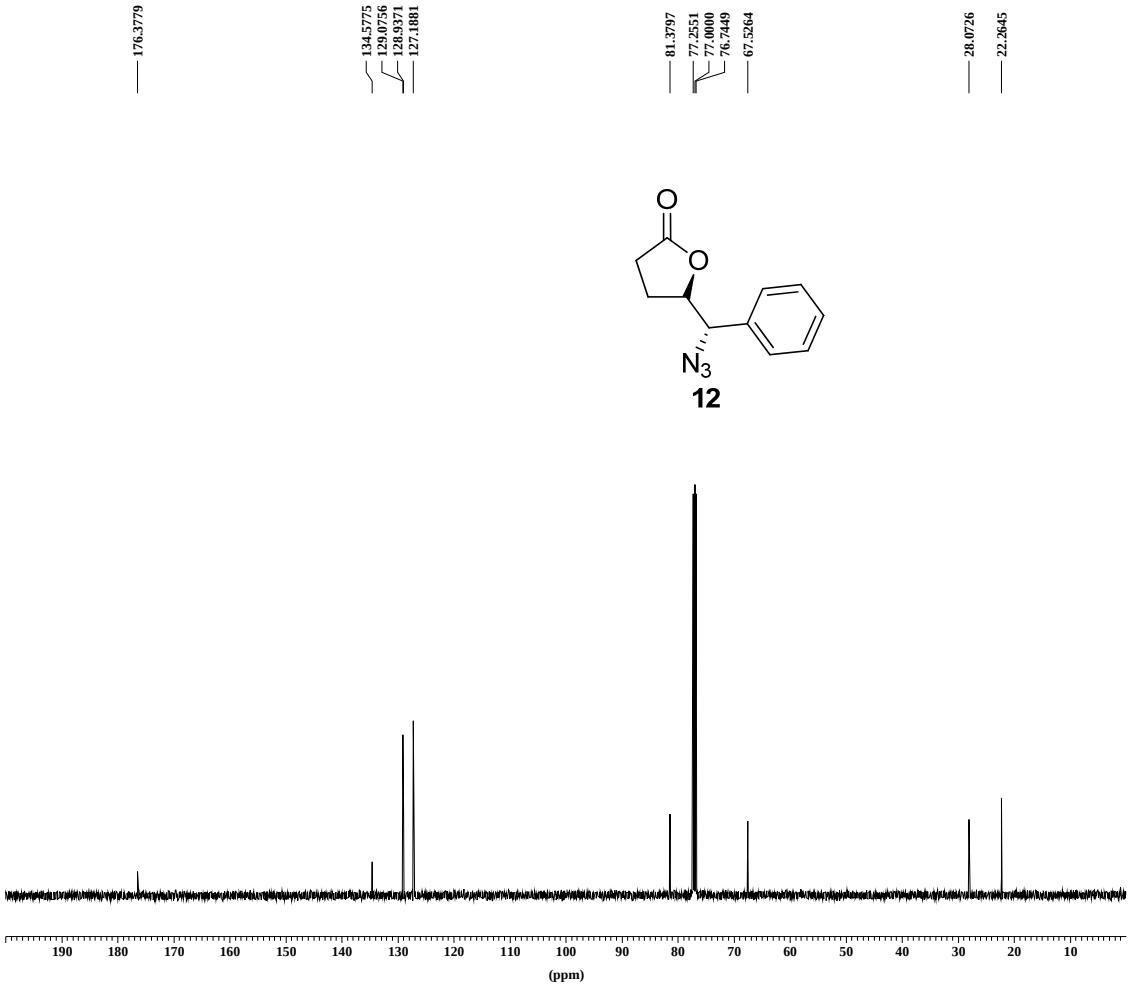
NUCLEUS	:	off

1H AMX500 cc-i-azide



*** Current Data Parameters ***
NAME : ck0209
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
LOCNUC : 2H
NS : 8
NUCLEUS : off
O1 : 3088.51 Hz
PULPROG : zg30
SFO1 : 500.1330885 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm
TD : 32768
TE : 294.7 K
*** Processing Parameters ***
LB : 0.30 Hz
SF : 500.1300140 MHz
*** 1D NMR Plot Parameters ***
NUCLEUS : off

13C AMX500 cc-i-azide



*** Current Data Parameters ***

NAME : ck0209

EXPNO : 2

PROCNO : 1

*** Acquisition Parameters ***

LOCNUC : 2H

NS : 201

NUCLEUS : off

O1 : 13204.57 Hz

PULPROG : zgpg30

SFO1 : 125.7709936 MHz

SOLVENT : CDCl3

SW : 238.7675 ppm

TD : 65536

TE : 295.6 K

*** Processing Parameters ***

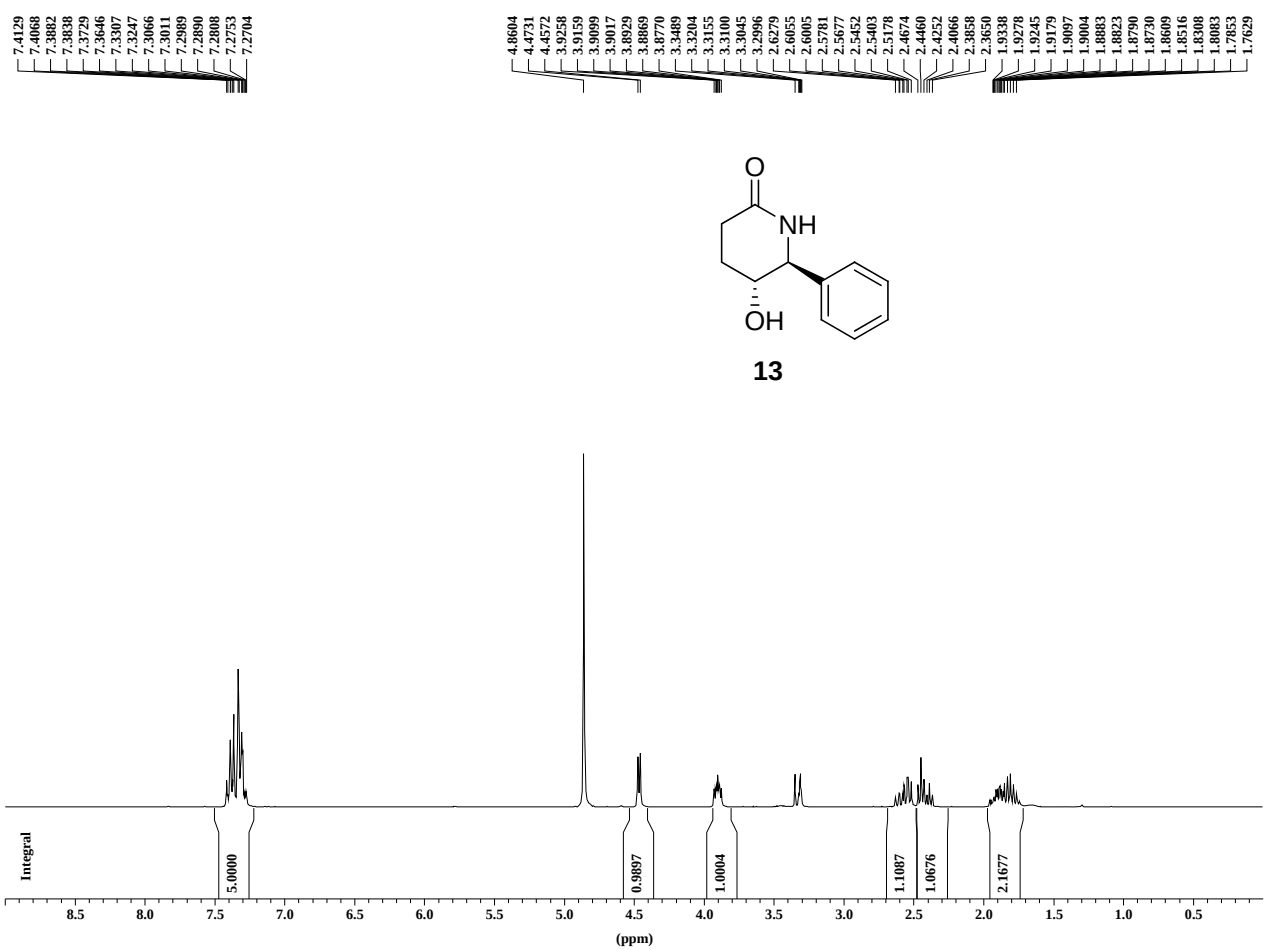
LB : 1.00 Hz

SF : 125.7577940 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS : off

1H normal range AC300 ck-v-93



***** Current Data Parameters *****

NAME	:	ja12ck
EXPNO	:	3
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	1853.43 Hz
PULPROG	:	zg30
SFO1	:	300.1318534 MHz
SOLVENT	:	MeOD
SW	:	17.9519 ppm
TD	:	32768
TE	:	296.9 K

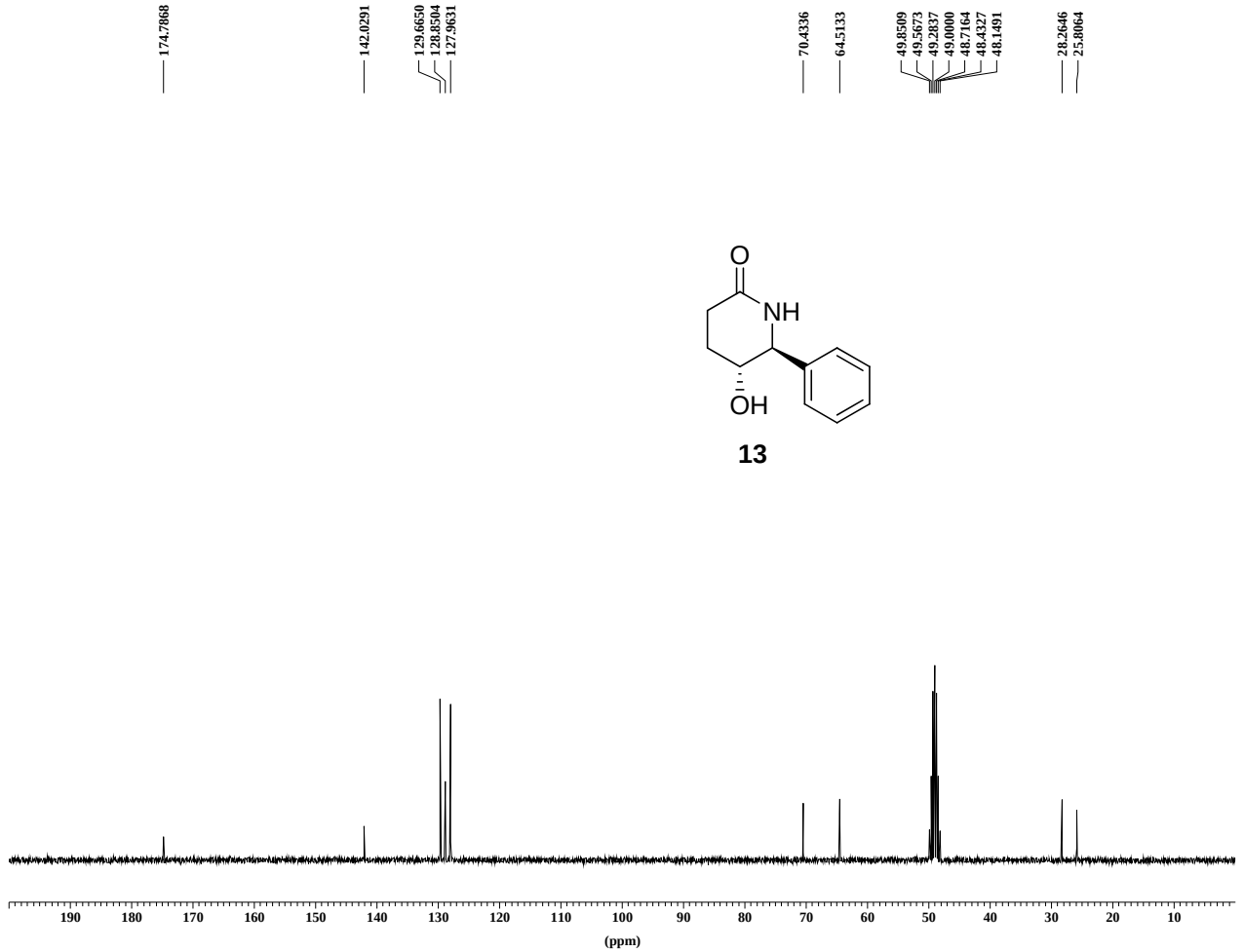
***** Processing Parameters *****

LB	:	0.30 Hz
SF	:	300.1300050 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

13C Standard AC300 ck-v-93



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***** Current Data Parameters *****

NAME	:	ja12ck
EXPNO	:	4
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	203
NUCLEUS	:	off
O1	:	7924.11 Hz
PULPROG	:	zgpg30
SFO1	:	75.4756731 MHz
SOLVENT	:	MeOD
SW	:	238.2968 ppm
TD	:	32768
TE	:	297.1 K

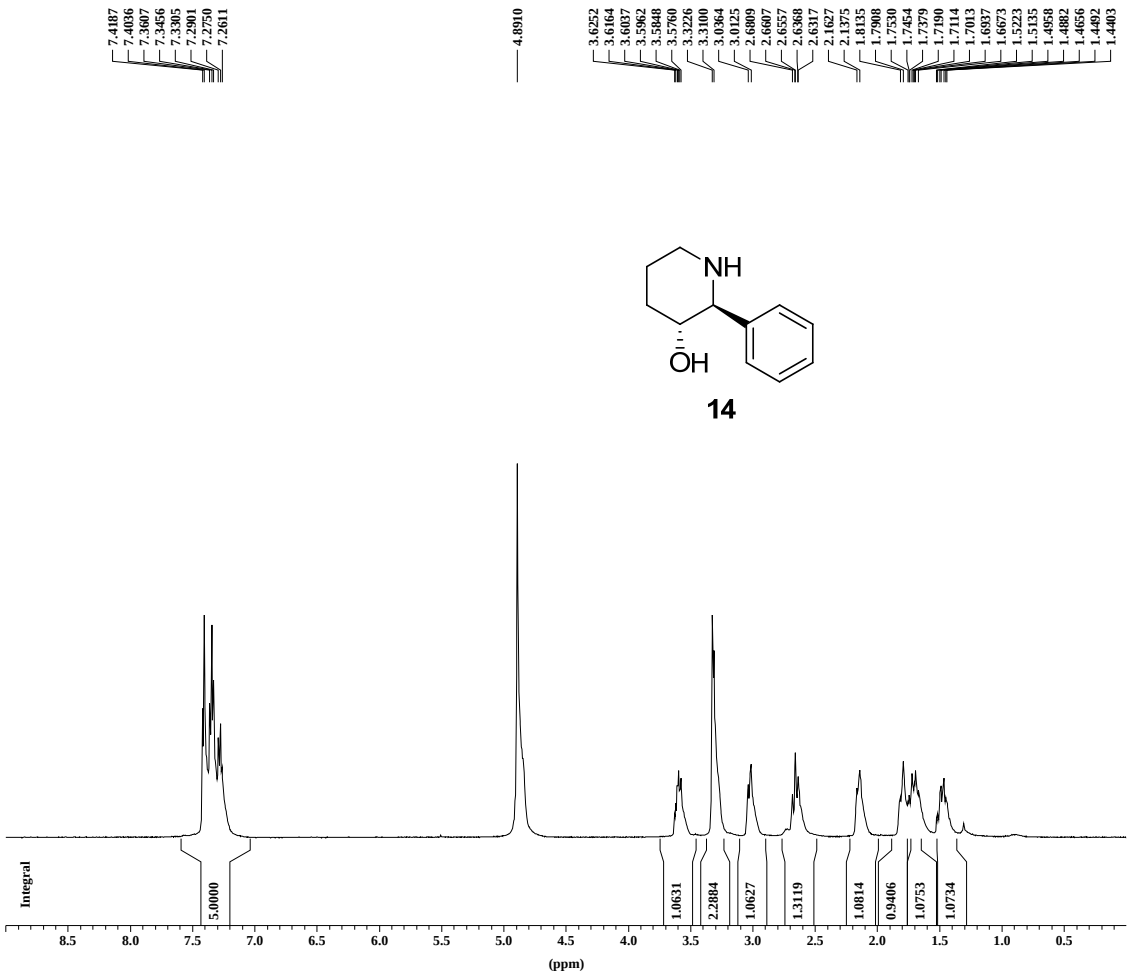
***** Processing Parameters *****

LB	:	1.00 Hz
SF	:	75.4676440 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

1H AMX500 cc-i-159



***** Current Data Parameters *****

NAME	:	ck0208
EXPNO	:	1
PROCNO	:	1

***** Acquisition Parameters *****

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	3088.51 Hz
PULPROG	:	zg30
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768
TE	:	295.2 K

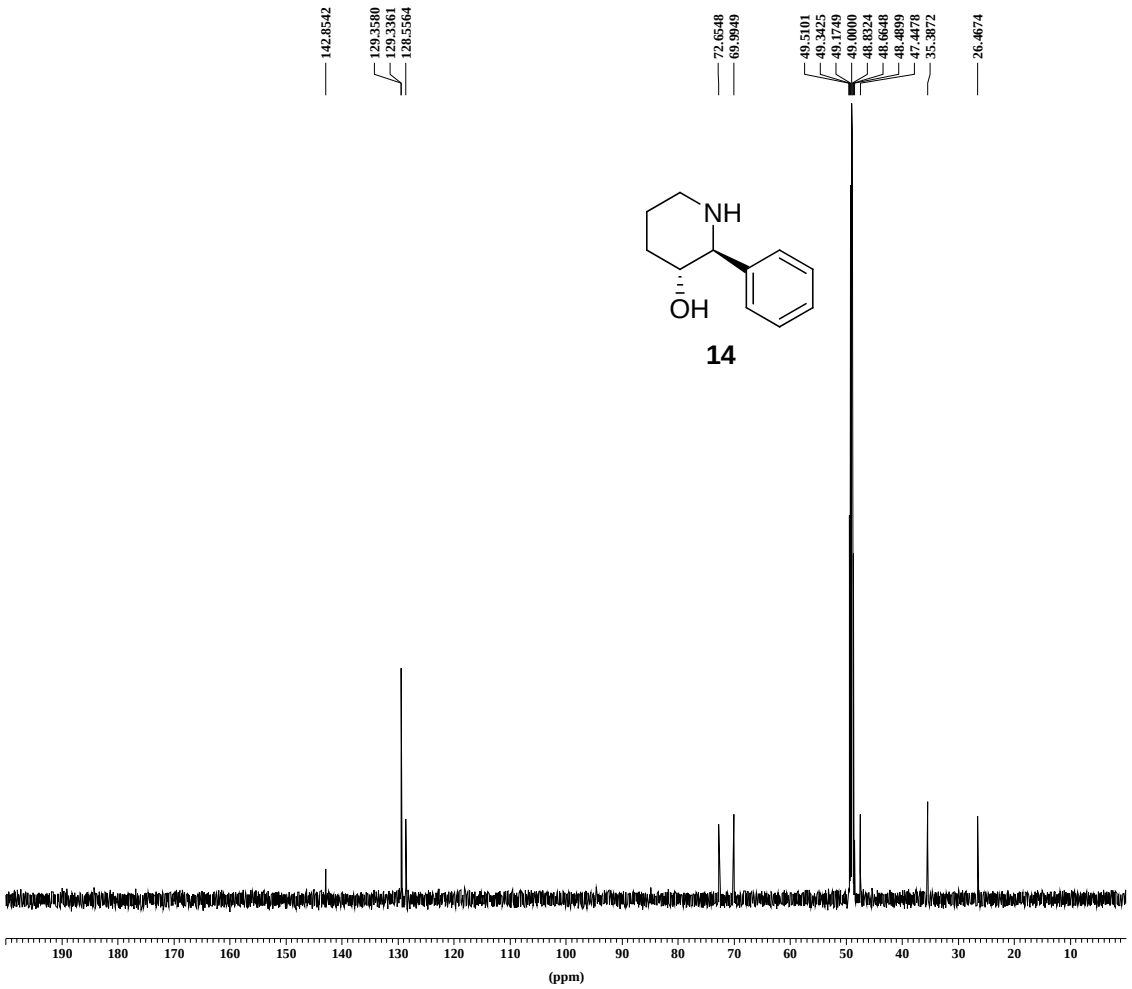
***** Processing Parameters *****

LB	:	0.30 Hz
SF	:	500.1300060 MHz

***** 1D NMR Plot Parameters *****

NUCLEUS	:	off

13C AMX500 cc-i-159



*** Current Data Parameters ***

NAME : ck0208

EXPNO : 2

PROCNO : 1

*** Acquisition Parameters ***

LOCNUC : 2H

NS : 165

NUCLEUS : off

O1 : 13204.57 Hz

PULPROG : zgpg30

SFO1 : 125.7709936 MHz

SOLVENT : MeOD

SW : 238.7675 ppm

TD : 65536

TE : 295.9 K

*** Processing Parameters ***

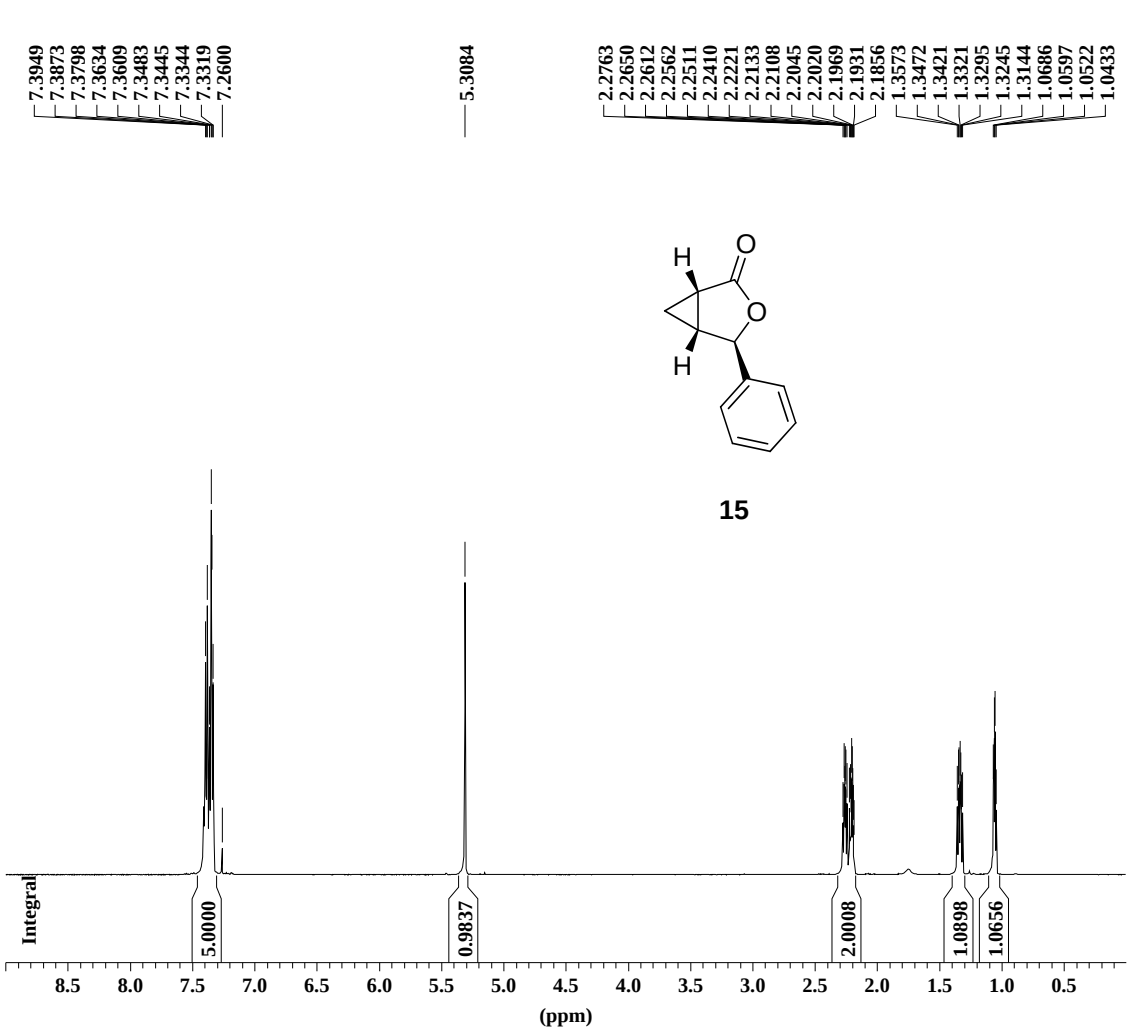
LB : 1.00 Hz

SF : 125.7576130 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS : off

1H AMX500 ck-ii-019



*** Current Data Parameters ***

NAME	:	ck0208
EXPNO	:	1
PROCNO	:	1

*** Acquisition Parameters ***

LOCNUC	:	2H
NS	:	8
NUCLEUS	:	off
O1	:	3088.51 Hz
PULPROG	:	zg30
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768
TE	:	303.1 K

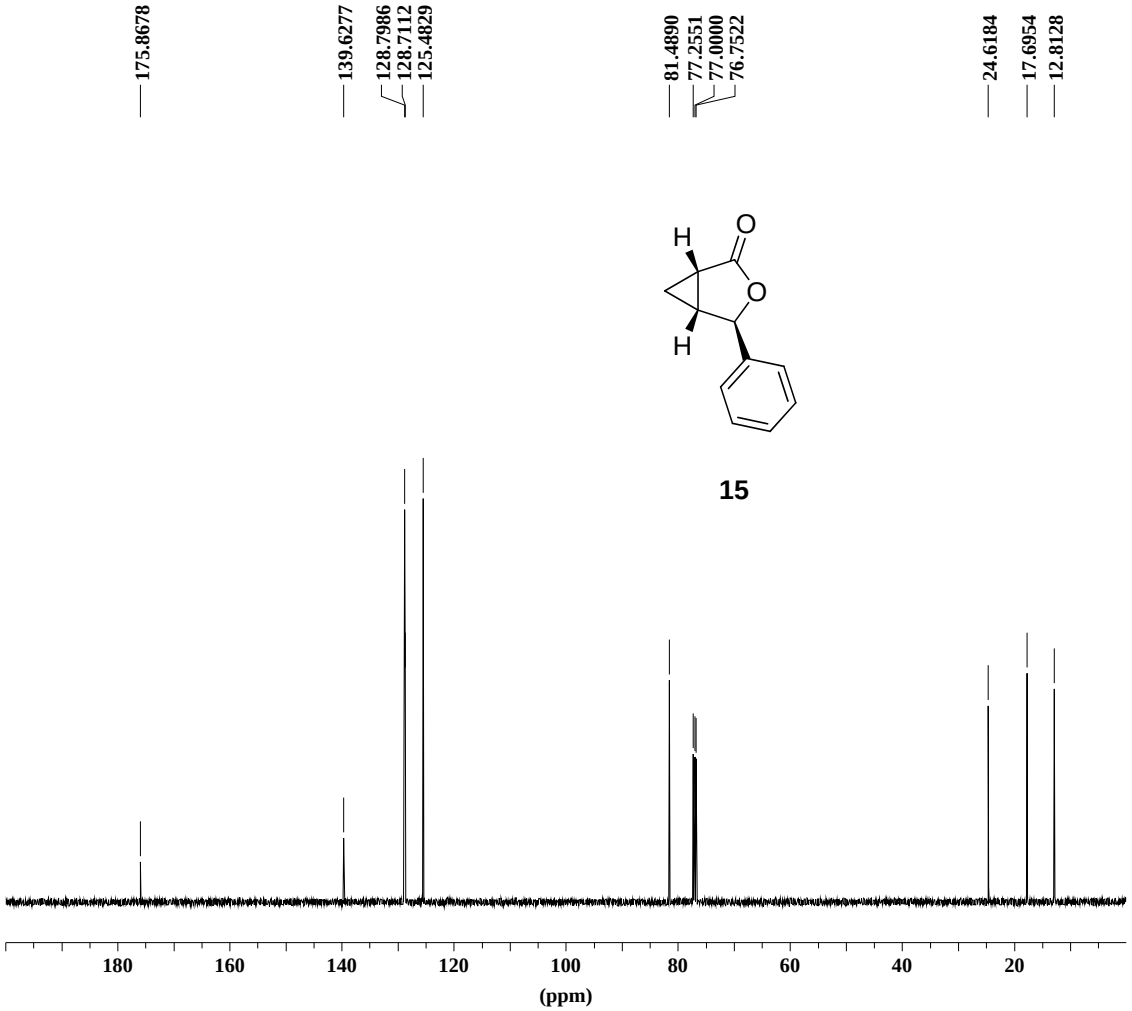
*** Processing Parameters ***

LB	:	0.30 Hz
SF	:	500.1300120 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

13C AMX500 ck-iii-019



*** Current Data Parameters ***

NAME	:	ck0208
EXPNO	:	2
PROCNO	:	1

*** Acquisition Parameters ***

LOCNUC	:	2H
NS	:	55
NUCLEUS	:	off
O1	:	13204.57 Hz
PULPROG	:	zgpg30
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm
TD	:	65536
TE	:	301.0 K

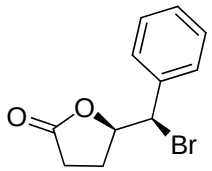
*** Processing Parameters ***

LB	:	1.00 Hz
SF	:	125.7578040 MHz

*** 1D NMR Plot Parameters ***

NUCLEUS	:	off

X-ray Structure of **5a**



5a