Supplementary Information

Glycerol conversion to high-value chemicals: Implication of unnatural α-amino acids syntheses using natural resources

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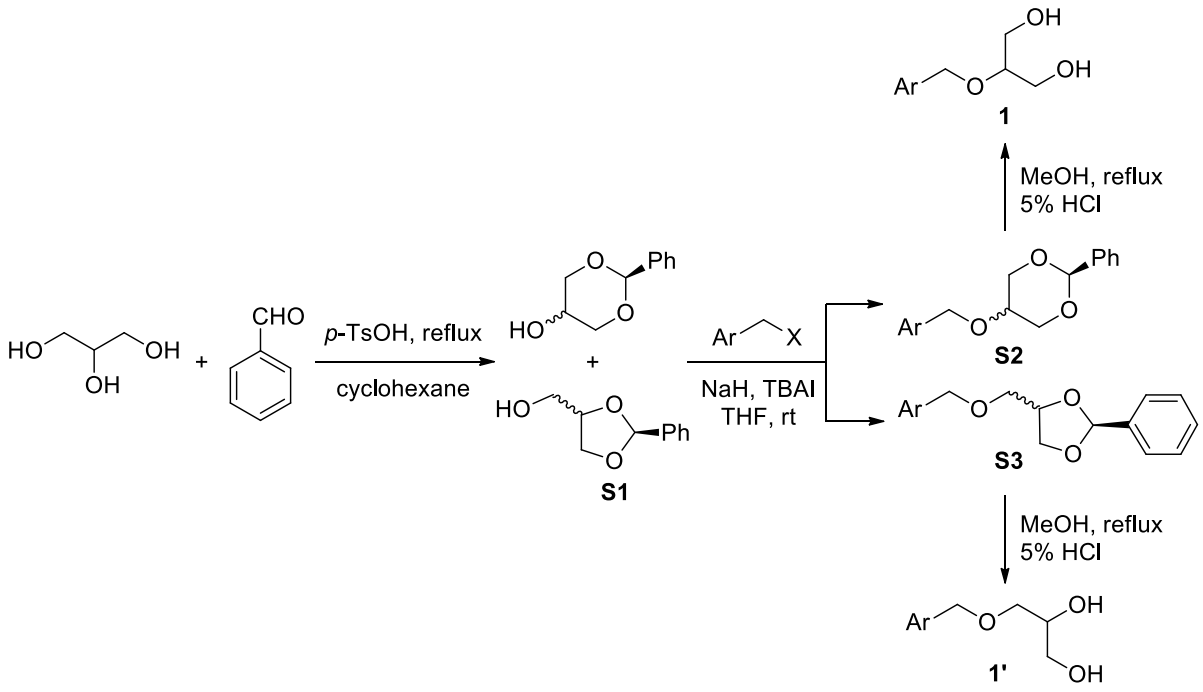
Contents

General methods

Thin-layer chromatography (TLC) was performed on Merck silica gel 60 F254. ^{1}H NMR spectra were recorded on a Varian at 500 MHz in $CDCl_3$ (δ 7.26 ppm) or DMSO-d_6 (δ 2.50 ppm) or CD_3OD-d_4 (δ 2.05 ppm), ^{13}C NMR spectral measurements were performed at 126 MHz using $CDCl_3$ (δ 77.16 ppm) or DMSO-d_6 (δ 39.52 ppm) or CD_3OD-d_4 (δ 49.00 ppm), ^{19}F NMR spectral measurements were performed with $CDCl_3$ or CD_3OD-d_4. The terms m, s, d, t, and q. represent multiplet, singlet, doublet, triplet, and quadruplet, respectively. Commercial grade reagents and solvents were used without further purification. Gas Chromatography High Resolution Mass Spectrometer (GC-HRMS) was obtained on an Agilent 6890 Series and a Bruker Compact mass spectrometer. Infrared (IR) spectra were recorded on a VERTEX 70(Bruker) IR spectrometer in the range of 1000-4000 cm^{-1}. HPLC analyses for the enantiomeric excess (*ee*) were performed with a Varian Pro Star Series HPLC System using chiral columns (Chiralpak AY-H; 250 × 4.6 mm). Melting Point were checked with a M-560(BUCHI). Optical rotation were recorded on PerkinElmer model 343 polarimeter.

General procedure for the synthesis of *O*-benzylglycerol from glycerol [Method A]

All compounds used in the synthesis of *O*-benzylglycerol were prepared following literature procedures.[1-3]



A mixture of benzaldehyde (5.1 mL, 50 mmol), glycerol (8.06 g, 87.5 mmol), and *p*-

toluenesulfonic acid (0.4 g, 2.3 mmol) in THF (16.5 mL) were placed into a 100 mL flask. The reaction mixture was heated under reflux using a Dean Stark trap until no further water was produced. Thereafter, the reaction was further carried out at room temperature for 1 h. The reaction mixture was quenched with $NaHCO_3$ and diethyl ether. The organic layer was washed with brine, dried over Na_2SO_4, and concentrated *in vacuo* to give crude oil. The crude product was purified by column chromatography on silica gel (hexanes:ethyl acetate = 5:1 to 1:1) to give dioxane and dioxolane (**S1**; 5.5 g, 61 %) as opaque white gel.

NaH (60 % in mineral oil, 1.29 g, 32.2 mmol) was washed with pentane (10 mL × 3) in a 100 mL round bottom flask. Dry THF (10 mL) was added dropwise with argon before a solution of compound **S1** (4.85 g, 26.8 mmol) in dry THF was added dropwise over 10 min at 0 ^oC. After 30 min at 0 ^oC, tetrabutylammonium iodide (0.1 g, 0.27 mmol) and a solution of benzyl bromide (3.5 mL, 29.5 mmol) in THF (20 mL) were added and the resulting mixture stirred for 3 h at room temperature. The reaction was quenched with cooled water and ethyl acetate. The organic layer was dried over Na_2SO_4 and concentrated *in vacuo.* The crude product was purified by column chromatography on silica gel (hexanes:ethyl acetate = 10:1 to 7:1) to give dioxane and dioxolane compound **S2** and **S3** as-obtained products.

To compound **S2** (0.52 g, 1.92 mmol) in MeOH (10.3 mL) was added 5% HCl solution (1.7 mL). The reaction mixture was heated under reflux for 20 min. The reaction mixture was extracted with CH_2Cl_2 and the combined organic layers washed with water, dried over Na_2SO_4, and concentrated *in vacuo* to obtain the crude ester. The crude product was purified by flash chromatography on silica gel (hexanes:ethyl acetate = 3:1 to 1:1) to give 2-*O*-benzylglycerol **1** as-obtained product.

1-*O*-Benzylglycerol **1'a** was synthesized by the above-mentioned standard procedure with compound **S3**.

General procedure for the oxidation of *O*-benzylglycerol [Method B]

O-Benzylglycerol (0.5 mmol) and sodium *tert*-butoxide (2.5 mmol) were added to a flame-dried round 10 mL bottom flask equipped with a magnetic stirring bar. The flask was evacuated *in vacuo* until the solvent began to bubble and then refilled with oxygen three times. The reaction mixture was stirred and heated at 60 ^oC for 18 h. 5% HCl solution was slowly added

to acidify (pH 1) the reaction mixture, which was then extracted with ethyl acetate (10 mL × 5). The combined organic layers were dried over Na_2SO_4, concentrated *in vacuo,* and purified by column chromatography on silica gel (hexanes : ethyl acetate : TFA = 3:1:0.2) to give the corresponding benzoic acid and (benzyloxy)acetic acid.

General procedure for the synthesis of aminopropanoate using chiral auxiliary [Method C]

Each step was carried out according to a literature procedure except for the step using a chiral auxiliary. A 100 mL two-neck round bottom flask was purged with argon and charged of lithium diisopropylamine in heptane/THF/ethylbenzene (2.0 M, 10.2 mL, 20.43 mmol) at -78 ^oC. A solution of (*S*)-3-(2-(benzyloxy)acetyl)-4-isopropyloxazolidin-2-one (1.89 g, 6.81 mmol) in THF (16.5 mL) was added dropwise over 5 min to the flask under argon at -78 ^oC. After stirring for 30 min, benzyl bromide (8.1 mL, 68.1 mmol) in THF (24.7 mL) was added dropwise to the reaction mixture over 5 min at -78 ^oC and stirring was continued at -45 °C for 3 h. The reaction mixture was quenched with saturated NH_4Cl solution and ethyl acetate. The organic layer was washed with brine and $NaHCO_3$, dried over Na_2SO_4, and concentrated *in vacuo* to give an oil. The crude product was purified by column chromatography on silica gel (hexanes:ethyl acetate = 3:1 to 1:1) to give aminopropanoate (1.84g, 73.4 %) as transparent brown liquid.

General procedure for the esterification of malonic acid [Method D]

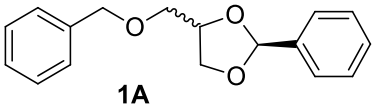
To di-carboxylic acid in MeOH was added carefully 3-4 drops of concentrated sulfuric acid. The reaction mixture was heated under reflux for 20 min. The reaction mixture was extracted in CH_2Cl_2. The combined organic layers were washed with water, dried over Na_2SO_4, and concentrated *in vacuo* to give the corresponding ester. The crude product was purified by flash chromatography on silica gel (hexanes:ethyl acetate = 3:1) to give the ester as a white solid.

General procedure for removal of oxazolidinone auxiliary [Method E]

Compound **23** (1.84 g, 5 mmol) in 25 mL of THF-H_2O (v/v = 4:1) was placed into 100 mL flask equipped with a magnetic stirring bar. To this solution was added 28% H_2O_2 (1.7 mL, 20 mmol) over 5 min under argon atmosphere at 0 ^oC, followed by LiOH (0.24 g, 10 mmol) in 10 mL of water. After stirring at 0 ^oC for 1 h, Na_2SO_3 (0.67 g, 20 mmol) in 15 mL of water was added followed by 50 mL of 5% $NaHCO_3$. After removal of the THF on a rotary evaporation, the resulting mixture was extracted with CH_2Cl_2 (50 mL × 3) to remove the oxazolidinone

auxiliary. The combined organic layers were dried over Na_2SO_4, concentrated in *vacuo* to give the oxazolidinone auxiliary as a white solid. This solid was recrystallized from ethyl acetate-hexanes (1:10) to give pure oxazolidinone auxiliary. 38% HCl (*aq*) was slowly added to acidify (pH 1) the aqueous layer, which was then extracted with ethyl acetate (100 mL × 4). The combined organic layers were dried over Na_2SO_4, concentrated in *vacuo* to give compound **24**.

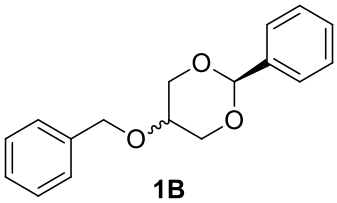
Characterisation data for the products



1A

Yield of product **1A**+**1B** = 75.2% (**1A**: 25.5%, **1B**: 49.7%) **(*2R,4R*)-4-((Benzyloxy)methyl)-2-phenyl-1,3-dioxolane**[4]**:** The title compound was synthesized according to general procedure A. Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (ddd, J = 6.3, 3.7, 1.5 Hz, 2H), 7.39–7.27 (m, 8H), 5.81 (s, 1H), 4.59 (d, J = 1.8 Hz, 2H), 4.43 (dq, J = 6.9, 5.6 Hz, 1H), 4.04 (ddd, J = 13.5, 8.2, 6.2 Hz, 2H), 3.62 (ddd, J = 49.0, 9.8, 5.7 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.91, 137.32, 129.37,128.44, 128.33, 127.77, 126.68, 104.38, 75.41, 73.55, 70.92, 68.02 ppm.

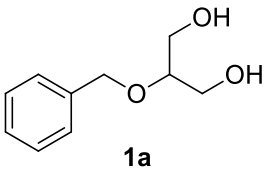
(*2S,4R*)-4-((Benzyloxy)methyl)-2-phenyl-1,3-dioxolane[4]**:** The title compound was synthesized according to general procedure A. Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (dt, J = 4.5, 2.5 Hz, 2H), 7.40–7.28 (m, 8H), 5.94 (s, 1H), 4.61 (s, 2H), 4.45 (tt, J = 6.6, 5.2 Hz, 1H), 4.23 (dd, J = 8.2, 6.7 Hz, 1H), 3.86 (dd, J = 8.3, 6.7 Hz, 1H), 3.64 (ddd, J = 29.9, 10.2, 5.2 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.92, 137,91 129.18, 128.46, 128.33, 127.79, 127.73, 126.44, 103.82, 76.06, 73.58, 70.45, 67.75 ppm.



1B

(*2R,5R*)-5-(Benzyloxy)-2-phenyl-1,3-dioxane[3]**:** The title compound was synthesized according to general procedure A. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50–7.28 (m, 10H), 5.40 (s, 1H), 4.60 (s, 2H), 4.36 (dd, J = 11.1, 4.9 Hz, 2H), 3.80 (ddd, J = 15.0, 9.8, 4.9 Hz, 1H), 3.70–3.61 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.89, 137.64, 128.99, 128.57, 128.29, 128.06, 127.77, 126.05, 101.29, 71.86, 70.21, 67.92 ppm.

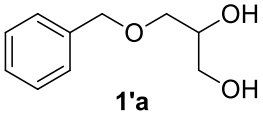
(*2S,5R*)-5-(Benzyloxy)-2-phenyl-1,3-dioxane[3]**:** The title compound was synthesized according to general procedure A. White solid ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (ddd, J = 8.3, 2.8, 1.5 Hz, 2H), 7.42–7.26 (m, 8H), 5.55 (s, 1H), 4.70 (s, 2H), 4.40–4.32 (m, 2H), 4.02 (dq, J = 12.6, 1.4 Hz, 2H), 3.35–3.31 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.13, 138.11, 128.92, 128.47, 128.21, 127.77, 127.72, 126.21, 101.42, 70.32, 69.22, 69.0 ppm.



1a

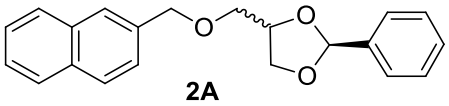
2-(Benzyloxy)propane-1,3-diol (1a)[3]**:** The title compound was synthesized according to general procedure A. White solid; Yield = 75%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.29 (m, 5H), 4.66 (s, 2H), 3.76 (ddd, J = 37.3, 11.7, 4.7 Hz, 4H), 3.60 (p, J =

4.7 Hz, 1H), 2.04 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.99, 128.63, 128.05, 127.85, 79.19, 72.01, 62.38, 62.29 ppm.



1'a

3-(Benzyloxy)propane-1,2-diol (1'a)[5]**:** The title compound was synthesized according to general procedure A. White solid; Yield = 60.2%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.28 (m, 5H), 4.55 (s, 2H), 3.89 (dq, J = 9.9, 5.1 Hz, 1H), 3.73–3.60 (m, 2H), 3.56 (qd, J = 9.7, 5.1 Hz, 2H), 2.74 (s, 1H), 2.26 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.64, 128.54,127.96, 127.81,73.63,71.82, 70.61, 64.10 ppm.

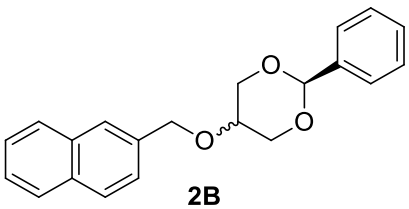


2A

Yield of product **2A**+**2B** = 68.6% (**2A**: 24.6%, **2B**: 44.0%)

(*2R,4R*)-4-((Naphthalen-2-ylmethoxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Slightly yellow-colored semisolid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85–7.76 (m, 4H), 7.51–7.44 (m, 5H), 7.38–7.32 (m, 3H), 5.81 (s, 1H), 4.74 (s, 2H), 4.45 (dq, J = 11.2, 5.6 Hz, 1H), 4.13–4.07 (m, 1H), 3.98 (dd, J = 8.2, 5.3 Hz, 1H), 3.65 (ddd, J = 46.2, 9.9, 5.7 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.30, 135.36, 133.25, 133.04, 129.38, 128.33, 128.27, 127.89, 127.71, 126.69, 126.61, 126.15, 125.95, 125.73, 104.42, 75.48, 73.65, 70.91, 67.98 ppm; HRMS (EI+) m/z: $[M]^+$ calcd for $C_{21}H_{20}O_3$ 320.1412, found 320.1411; IR: 3053, 2868, 1601, 1506, 1458, 1394, 1344, 1267,1218,1095, 1063, 1021 cm^{-1}.

(*2S,4R*)-4-((Naphthalen-2-ylmethoxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Slightly yellow-colored solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87–7.78 (m, 4H), 7.52–7.45 (m, 5H), 7.40–7.33 (m, 3H), 5.95 (s, 1H), 4.77 (s, 2H), 4.49–4.44 (m, 1H), 4.23 (dd, J = 8.2, 6.7 Hz, 1H), 3.86 (dd, J = 8.2, 6.7 Hz, 1H), 3.67 (ddd, J = 30.5, 10.2, 5.2 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.89, 135.37, 133.24, 133.05, 129.18, 128.34, 128.30, 127.88, 127.72, 126.59, 126.45, 126.18, 125.97, 125.70, 103.83, 75.10, 73.69, 70.44, 67.74 ppm. HRMS (EI+) m/z: $[M]^+$ calcd for $C_{21}H_{20}O_3$ 320.1412, found 320.1411; IR: 3053, 2868, 1601, 1506, 1458, 1394, 1344, 1267, 1218, 1095, 1063, 1021 cm^{-1}.

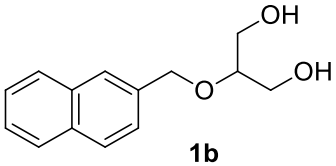


2B

(*2R,5R*)-5-(Naphthalen-2-ylmethoxy)-2-phenyl-1,3-dioxane[6]**:** The title compound was synthesized according to general procedure A. Slightly

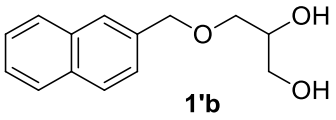
yellow-colored solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (dd, J = 8.8, 5.5 Hz, 3H), 7.76 (s, 1H), 7.49–7.42 (m, 5H), 7.36–7.30 (m, 3H), 5.39 (s, 1H), 4.73 (s, 2H), 4.37 (dd, J = 11.1, 4.9 Hz, 2H), 3.83 (tt, J = 9.8, 5.0 Hz, 1H), 3.66 (dd, J = 15.2, 6.2 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.63, 135.33, 133.24, 133.14, 128.98, 128.43, 128.28, 127.91, 127.72, 126.68, 126.28, 126.12, 126.04, 125.61, 101.30, 72.02, 70.24, 67.95 ppm.

(*2S*,*5R*)-5-(Naphthalen-2-ylmethoxy)-2-phenyl-1,3-dioxane[6]**:** The title compound was synthesized according to general procedure A. Slightly yellow-colored solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (dd, J = 10.9, 5.9 Hz, 4H), 7.55 (dd, J = 8.1, 1.5 Hz, 3H), 7.50–7.44 (m, 2H), 7.40–7.31 (m, 3H), 5.57 (s, 1H), 4.87 (s, 2H), 4.40 (dd, J = 12.5, 1.3 Hz, 2H), 4.04 (dd, J = 12.6, 1.6 Hz, 2H), 3.40–3.36 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.13, 135.62, 133.25, 133.05, 128.92, 128.32, 128.22, 127.86, 127.73, 126.55, 126.22, 126.18, 125.94, 125.77, 101.42, 70.48, 69.21, 69.08 ppm.



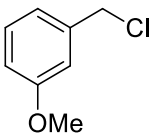
1b

2-(Naphthalen-2-ylmethoxy)propane-1,3-diol (1b): The title compound was synthesized according to general procedure A. White solid; Yield = 60.5%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89–7.78 (m, 4H), 7.53–7.45 (m, 3H), 4.83 (s, 2H), 3.79 (ddd, J = 39.0, 11.7, 4.6 Hz, 4H), 3.68–3.64 (m, 1H), 1.93 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 135.42, 133.27, 133.10, 128.52, 127.90, 127.76, 126.71, 126.32, 126.14, 125.68, 79.15, 72.14, 62.49 ppm.

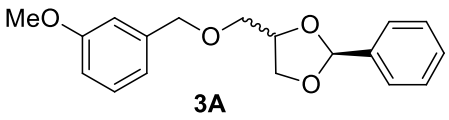


1'b

3-(Naphthalen-2-ylmethoxy)propane-1,2-diol (1'b): The title compound was synthesized according to general procedure A. White solid; Yield = 65.3%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89–7.74 (m, 4H), 7.53–7.42 (m, 3H), 4.72 (s, 2H), 3.92 (d, J = 3.5 Hz, 1H), 3.76–3.56 (m, 4H), 2.62 (d, J = 3.9 Hz, 1H), 2.09 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 135.09, 133.23, 133.08, 128.40, 127.88, 127.73, 126.70, 126.26, 126.08, 125.65, 73.75, 71.85, 70.58, 64.13 ppm.



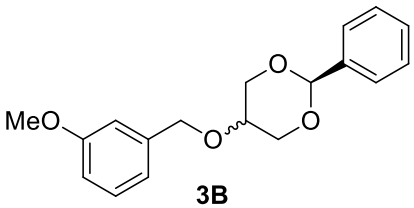
1-(Chloromethyl)-3-methoxybenzene: The title compound was synthesized according to reference[7]. Brown-colored liquid; Yield = 99%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31–7.24 (m, 1H), 7.00–6.92 (m, 2H), 6.89–6.83 (m, 1H), 4.56 (s, 2H), 3.82 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.81, 138.90, 129.77, 120.80, 114.10, 114.01, 55.28, 46.21 ppm.



3A

Yield of product **3A**+**3B** = 80% (**3A**: 27.7%, **3B**: 52.3%)

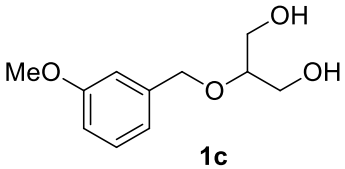
4-(((3-Methoxybenzyl)oxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Colorless oil; No attempt was made to separate the diastereomers mixture; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (dd, J = 5.3, 2.0 Hz, 2H both diastereomers), 7.40–7.35 (m, 3H both diastereomers), 7.29–7.23 (m, 1H both diastereomers), 6.92 (t, J = 7.6 Hz, 2H both diastereomers), 6.86–6.82 (m, 1H both diastereomers), 4.48–4.39 (m, 1H both diastereomers), 3.72–3.54 (m, 2H both diastereomers) ppm; **Cis:** δ 5.95 (s, 1H), 4.59 (s, 2H), 4.24 (dd, J = 8.2, 6.7 Hz, 1H), 3.86 (dd, J = 8.2, 6.7 Hz, 1H), 3.81 (s, 3H) ppm; **Trans:** δ 5.81 (s, 1H), 4.57 (s, 2H), 4.14–4.07 (m, 1H), 3.98 (dd, J = 8.2, 5.3 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) **Cis:** δ 159.79, 139.54, 137.89, 129.48, 129.18, 128.33, 126.44, 119.93, 113.34, 113.05, 103.82, 75.07, 73.42, 70.44, 67.73 ppm; **Trans:** δ 159.79, 139.56, 137.29, 129.46, 129.38, 128.33, 126.69, 119.97, 113.41, 113.02, 104.40, 75.43, 73.40, 70.89, 67.99 ppm; HRMS (EI+) m/z: $[M]^+$ calcd for $C_{18}H_{20}O_4$ 300.1362, found 300.1357; IR: 3041, 2873, 1596, 1486, 1458, 1396, 1265, 1219, 1155, 1096, 1052 cm^{-1}.



3B

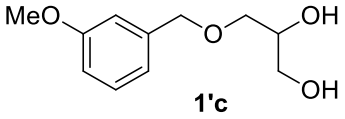
(*2R,5R*)-5-((3-Methoxybenzyl)oxy)-2-phenyl-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; mp = 52.6-53.4 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (dd, J = 7.6, 1.7 Hz, 2H), 7.38–7.32 (m, 3H), 7.27 (dd, J = 12.8, 4.9 Hz, 1H), 6.87 (ddd, J = 11.7, 10.5, 5.0 Hz, 3H), 5.40 (s, 1H), 4.58 (s, 2H), 4.39–4.33 (m, 2H), 3.84–3.76 (m, 4H), 3.69–3.62 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.82, 139.51, 137.64, 129.62, 128.99, 128.29, 126.04, 119.96, 113.50, 113.21, 101.29, 71.73, 70.21, 67.94, 55.24 ppm; HRMS (EI+) m/z: $[M]^+$ calcd for $C_{18}H_{20}O_4$ 300.1362, found 300.1362; IR: 2963, 2914, 2861, 1587, 1490, 1453, 1380, 1268, 1158, 1097, 1035 cm^{-1}.

(*2S,5R*)-5-((3-Methoxybenzyl)oxy)-2-phenyl-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; mp = 77.1-78.0 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54–7.49 (m, 2H), 7.36–7.20 (m, 4H), 6.99–6.91 (m, 2H), 6.80 (dd, J = 8.2, 2.3 Hz, 1H), 5.52 (s, 1H), 4.64 (s, 2H), 4.32 (dd, J = 12.4, 1.0 Hz, 2H), 3.98 (dd, J = 12.5, 1.4 Hz, 2H), 3.75 (s, 3H), 3.31–3.27 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.82, 139.84, 138.11, 129.45, 128.90, 128.19, 126.19, 119.90, 113.42, 112.97, 101.38, 70.20, 69.23, 69.03, 55.23 ppm; HRMS (EI+): m/z calcd for $C_{18}H_{20}O_4$ ($[M]^+$): 300.1362; observed: 300.1362. IR: 3029, 2968, 2903, 1613, 1583, 1489, 1453, 1388, 1336, 1150, 1092, 1045, 1013 cm^{-1}.



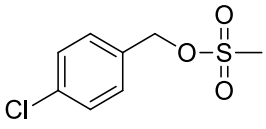
1c

2-((3-Methoxybenzyl)oxy)propane-1,3-diol (1c): The title compound was synthesized according to general procedure A. Colorless oil; Yield = 70.1%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 (dd, J = 10.7, 5.0 Hz, 1H), 6.96–6.89 (m, 2H), 6.85 (dd, J = 8.0, 2.3 Hz, 1H), 4.63 (s, 2H), 3.81 (s, 3H), 3.78 (dd, J = 11.7, 4.5 Hz, 2H), 3.71 (dd, J = 11.7, 4.9 Hz, 2H), 3.58 (p, J = 4.7 Hz, 1H), 2.27 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.83, 139.64, 129.67, 120.01, 113.39, 113.37, 79.21, 71.83, 62.31, 55.25 ppm; HRMS (EI+) m/z: $[M]^+$ calcd for $C_{11}H_{16}O_4$ 212.1049, found 212.1046; IR: 3390, 2936, 2880, 1594, 1490,1345, 1315, 1265, 1158, 1111 cm^{-1}.

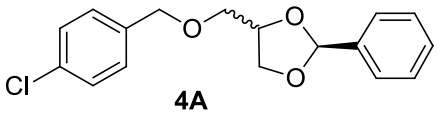


1'c

3-((3-Methoxybenzyl)oxy)propane-1,2-diol (1'c)[8]**:** The title compound was synthesized according to general procedure A. Colorless oil; Yield = 65.5%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 (dd, J = 9.9, 5.7 Hz, 1H), 6.93–6.82 (m, 3H), 4.53 (s, 2H), 3.93–3.87 (m, 1H), 3.81 (s, 3H), 3.74–3.52 (m, 4H), 2.64 (s, 1H), 2.15 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.79, 139.31, 129.56, 119.99, 113.35, 113.24, 73.44, 71.78, 70.66, 64.05, 55.23 ppm.



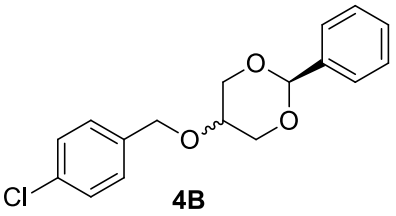
4-Chlorobenzyl methanesulfonate: The title compound was synthesized according to reference [9]. White solid; Yield = 95%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42–7.34 (m, 4H), 5.21 (s, 2H), 2.95 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 135.48, 131.95, 130.19, 129.17. 70.36, 38.41 ppm.



4A

Yield of product **4A**+**4B** = 72% (**4A**: 24.1%, **4B**: 47.9%) **4-((4-Chlorobenzyl)oxy)methyl)-2-phenyl-1,3-dioxolane:** The title compound was synthesized according to general procedure A. White solid; No attempt was made to separate the diastereomers mixture; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (dd, J = 7.5, 2.0 Hz, 2H both diastereomers), 7.40–7.26 (m, 7H both diastereomers), 4.48–4.41 (m, 1H both diastereomers), 3.63 (ddd, J = 25.8, 10.2, 5.1 Hz, 2H both diastereomers) ppm; **Cis:** δ 5.94 (s, 1H), 4.57 (s, 2H), 4.24 (dd, J = 8.3, 6.7 Hz, 1H), 3.85 (dd, J = 8.3, 6.7 Hz, 1H) ppm; **Trans:** δ 5.81 (s, 1H), 4.54 (s, 2H), 4.10 (dd, J = 8.1, 7.1 Hz, 1H), 3.97 (dd, J = 8.2, 5.3 Hz, 1H) ppm; ^{13}C NMR (126

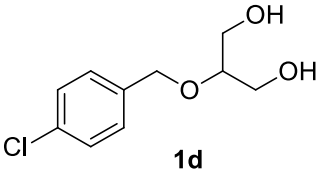
MHz, $CDCl_3$) **Cis:** δ 137.24, 136.42, 133.52, 129.42, 129.04, 128.60, 128.35, 126.66, 104.42, 75.38, 72.74, 71.01, 67.90 ppm; **Trans:** δ 137.82, 136.42, 129.22, 128.99, 128.62, 126.43, 103.87, 75.03,72.78, 70.58,67.64 ppm.



4B

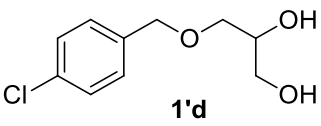
(*2R,5R*)-5-((4-Chlorobenzyl)oxy)-2-phenyl-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; mp = 111.1-111.6 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (dd, J = 7.5, 1.8 Hz, 2H), 7.38–7.25 (m, 7H), 5.40 (s, 1H), 4.56 (s, 2H), 4.35 (dd, J = 11.0, 4.9 Hz, 2H), 3.77 (dt, J = 14.9, 4.8 Hz, 1H), 3.69–3.61 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.56, 136.40, 133.85, 129.03, 129.02, 128.74, 128.31, 126.04, 101.33, 71.03, 71.12, 68.12 ppm; HRMS (EI+) *m*/*z*: $[M]^+$ calcd for $C_{17}H_{17}ClO_3$ 304.0866, found 304.0860; IR: 2976, 2866,1492, 1456, 1386, 1337, 1152, 1090, 1011 cm^{-1}.

(*2S,5R*)-5-((4-Chlorobenzyl)oxy)-2-phenyl-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (dd, J = 7.7, 1.5 Hz, 2H), 7.40–7.29 (m, 7H), 5.55 (s, 1H), 4.64 (s, 2H), 4.34 (dd, J = 12.6, 1.4 Hz, 2H), 4.04 (dd, J = 12.6, 1.6 Hz, 2H), 3.36–3.28 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.05, 136.71, 133.37, 128.96, 128.92, 128.56, 128.20, 126.15, 101.36, 69.68, 69.59, 68.92 ppm.



1d

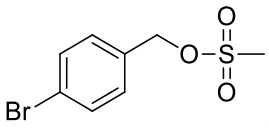
2-((4-Chlorobenzyl)oxy)propane-1,3-diol (1d): The title compound was synthesized according to general procedure A. White solid; Yield = 67.1%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37–7.26 (m, 4H), 4.63 (s, 2H), 3.84–3.70 (m, 4H), 3.59 (p, J = 4.7 Hz, 1H), 2.04 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 136.51, 133.80, 129.11, 128.77, 79.29, 71.19, 62.45 ppm.



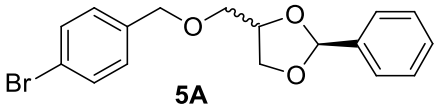
1'd

3-((4-Chlorobenzyl)oxy)propane-1,2-diol (1'd): The title compound was synthesized according to general procedure A. White solid; Yield = 55.8%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.22 (m, 4H), 4.51 (s, 2H), 3.93–3.86 (m, 1H), 3.74–3.59 (m,

2H), 3.54 (qd, *J* = 9.6, 5.2 Hz, 2H), 2.76 (d, *J* = 4.7 Hz, 1H), 2.29 (t, *J* = 5.7 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 136.17, 133.72, 129.08, 128.70, 72.82, 71.89, 70.59, 64.03 ppm.



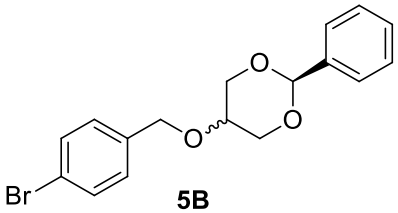
4-Bromobenzyl methanesulfonate: The title compound was synthesized according to reference[10]. White solid; Yield = 90.9%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57–7.53 (m, 2H), 7.32–7.28 (m, 2H), 5.19 (s, 2H), 2.95 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 132.47, 132.15, 130.42, 123.66, 70.36, 38.42 ppm.



5A

Yield of product **5A**+**5B** = 87.7% (**5A**: 20.5%, **5B**: 67.1%)

4-(((4-Bromobenzyl)oxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Opaque white gel type; No attempt was made to separate the diastereomers mixture; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (ddd, *J* = 6.4, 4.7, 2.2 Hz, 4H both diastereomers), 7.39–7.35 (m, 3H both diastereomers), 7.22 (dd, *J* = 11.3, 8.4 Hz, 2H both diastereomers), 4.47–4.38 (m, 2H both diastereomers), 3.62 (dddd, *J* = 15.6, 11.9, 10.0, 6.1 Hz, 2H both diastereomers) ppm; **Cis:** δ 5.94 (s, 1H), 4.56 (s, 2H), 4.24 (dd, *J* = 8.3, 6.7 Hz, 1H), 3.85 (dd, *J* = 8.3, 6.7 Hz, 1H) ppm; **Trans:** δ 5.81 (s, 1H), 4.53 (s, 2H), 4.10 (dd, *J* = 8.1, 7.1 Hz, 1H), 3.97 (dd, *J* = 8.2, 5.3 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 129.42, 129.36, 129.31, 129.22 (aromatic peaks of diastereomers) ppm; **Cis:** δ 137.83, 136.96, 131.58, 128.35,126.43, 121.65, 103.87, 75.03, 72.81, 70.60, 67.64 ppm; **Trans:** δ 137.26, 136.95, 131.56, 128.35, 126.66, 121.64, 104.43, 75.39, 72.77, 71.03, 67.90 ppm; HRMS (EI+) *m*/*z*: $[M]^+$ calcd for Chemical $C_{17}H_{17}BrO_3$ 348.0361, found 348.0356; IR: 2908, 2867, 1592, 1485, 1400, 1221, 1099, 1057 cm^{-1}.

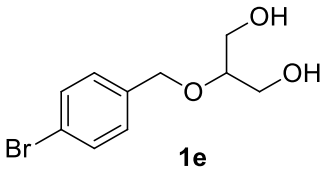


5B

(*2R*,*5R*)-5-((4-Bromobenzyl)oxy)-2-phenyl-1,3-dioxane[11]: The title compound was synthesized according to general procedure A. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51–7.43 (m, 4H), 7.38–7.32 (m, 3H), 7.20 (d, *J* = 8.3 Hz, 2H), 5.39 (s, 1H), 4.54 (s, 2H), 4.35 (dd, *J* = 11.0, 4.9 Hz, 2H), 3.77 (ddd, *J* = 14.9, 9.7, 4.9 Hz, 1H), 3.64 (t, *J* = 10.7 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.56, 136.92, 131.67, 129.31, 129.02, 128.29, 126.04, 121.93, 101.31, 71.03,70.09, 68.13 ppm.

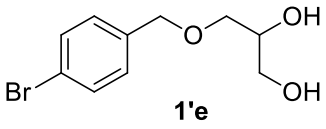
(*2S*,*5R*)-5-((4-Bromobenzyl)oxy)-2-phenyl-1,3-dioxane[11]: The title compound was

synthesized according to general procedure A. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (ddt, J = 10.7, 8.9, 2.0 Hz, 4H), 7.39–7.27 (m, 5H), 5.56 (s, 1H), 4.65 (s, 2H), 4.35 (dd, J = 12.6, 1.4 Hz, 2H), 4.10–4.02 (m, 2H), 3.37–3.32 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.04, 137.24, 131.55, 129.30, 128.95, 128.23, 126.16, 121.54, 101.41, 69.70, 69.65, 68.95 ppm.



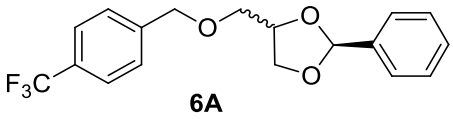
1e

2-((4-Bromobenzyl)oxy)propane-1,3-diol (1e)[11]**:** The title compound was synthesized according to general procedure A. White solid; Yield = 99%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, J = 8.2 Hz, 2H), 7.24 (d, J = 8.1 Hz, 2H), 4.62 (s, 2H), 3.77 (td, J = 11.6, 7.2 Hz, 2H), 3.62–3.56 (m, 1H), 1.94 (s, 2H) ppm; ^{13}CNMR (126 MHz, $CDCl_3$) δ 137.05, 131.72, 129.41, 121.90, 79.33, 71.22, 62.47 ppm.



1'e

3-((4-Bromobenzyl)oxy)propane-1,2-diol (1'e): The title compound was synthesized according to general procedure A. White solid; Yield = 95%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, J = 8.3 Hz, 2H), 7.17 (d, J = 8.2 Hz, 2H), 4.46 (s, 2H), 3.91–3.82 (m, 1H), 3.69–3.43 (m,5H), 3.20 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 136.74, 131.58, 129.37, 121.72, 72.71, 71.69, 70.81, 63.91 ppm.



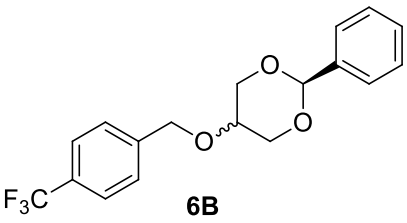
6A

Yield of product **6A**+**6B** = 90.6% (**6A**: 30.1%, **6B**: 60.5%)

(*2R,4R*)-2-Phenyl-4-(((4-(trifluoromethyl)benzyl)oxy)methyl)-1,3-dioxolane: The title compound was synthesized according to general procedure A. White solid; mp = 57.2-58.1 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (d, J = 8.1 Hz, 2H), 7.52–7.42 (m, 4H), 7.40–7.34 (m, 3H), 5.82 (s, 1H), 4.64 (s, 2H), 4.45 (dq, J = 6.9, 5.5 Hz, 1H), 4.12 (dd, J = 8.1, 7.1 Hz, 1H), 4.00 (dd, J = 8.2, 5.3 Hz, 1H), 3.65 (ddd, J = 33.2, 9.9, 5.6 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.08 (s), 137.25 (s), 129.90 (q, J = 32.4 Hz), 129.44 (s), 128.36 (s), 127.61 (s), 126.66 (s), 125.38 (q, J = 3.8 Hz), 124.16 (q, J = 272.0 Hz), 104.46 (s), 75.39 (s), 72.69 (s), 71.29 (s), 67.83 (s) ppm; ; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.50 ppm; HRMS (EI+) m/z: $[M]^+$ calcd for $C_{18}H_{17}F_3O_3$ 338.1130, found 338.1125; IR: 2878, 1460, 1402, 1322, 1232, 1153, 1107, 1057, 1023 cm^{-1}.

(*2S,4R*)-2-Phenyl-4-(((4-(trifluoromethyl)benzyl)oxy)methyl)-1,3-dioxolane: The title compound was synthesized according to general procedure A. White solid; mp = 53.9-54.4 °C;

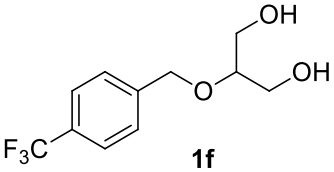
H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, J = 8.1 Hz, 2H), 7.52–7.44 (m, 4H), 7.42–7.35 (m, 3H), 5.96 (s, 1H), 4.67 (s, 2H), 4.50–4.44 (m, 1H), 4.29–4.23 (m, 1H), 3.87 (dd, J = 8.1, 6.9 Hz, 1H), 3.68 (qd, J = 10.2, 5.0 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.07, 137.80, 129.94 (dd, J = 64.7, 32.4 Hz), 129.26, 128.38, 127.57, 126.44, 125.41 (q, J = 3.7 Hz), 124.15 (dd, J = 544.0, 272.0 Hz), 103.93, 75.04, 72.75, 70.89, 67.57 (s) ppm; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.49 ppm; HRMS (EI+) m/z: $[M]^+$ calcd for $C_{18}H_{17}F_3O_3$ 338.1130, found 338.1125; IR: 2878, 1463, 1410, 1330, 1221, 1158, 1101, 1062, 1021 cm^{-1}.



6B

(*2R*,*5R*)-2-Phenyl-5-((4-(trifluoromethyl)benzyl)oxy)-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; mp = 118.5-119.4 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, J = 8.1 Hz, 2H), 7.49–7.43 (m, 4H), 7.39–7.33 (m, 3H), 5.41 (s, 1H), 4.66 (s, 2H), 4.39 (dd, J = 11.0, 4.9 Hz, 2H), 3.81 (ddd, J = 14.9, 9.7, 4.9 Hz, 1H), 3.68 (dd, J = 11.2, 10.1 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.95, 137.52, 130.21 (q, J = 32.4 Hz), 129.06, 128.32, 127.61, 125.50 (q, J = 3.8 Hz), 124.07 (dd, J = 544.1, 272.0 Hz), 70.93, 70.04, 68.41 ppm; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.58 ppm; HRMS (EI+) m/z: $[M]^+$ calcd for $C_{18}H_{17}F_3O_3$ 338.1130, found 338.1136; IR: 2978, 2868, 1457, 1400, 1330, 1218, 1158, 1108, 1066, 1019 cm^{-1}.

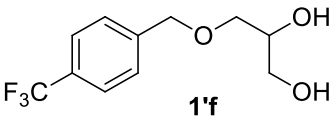
(*2S*,*5R*)-2-Phenyl-5-((4-(trifluoromethyl)benzyl)oxy)-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; mp = 92.9-93.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (dd, J = 39.1, 8.3 Hz, 6H), 7.41–7.34 (m, 3H), 5.58 (s, 1H), 4.75 (s, 2H), 4.38 (dd, J = 12.6, 1.2 Hz, 2H), 4.08 (dd, J = 12.5, 1.0 Hz, 2H), 3.37 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.40, 138.03, 129.85 (q, J = 32.4 Hz), 128.99, 128.26, 127.57, 126.16, 125.38 (q, J = 3.8 Hz), 124.17 (q, J = 271.9 Hz), 101.43, 70.14, 69.65, 68.91 ppm; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.48 ppm; HRMS (EI+) m/z: $[M]^+$ calcd for $C_{18}H_{17}F_3O_3$ 338.1130, found 338.1136; IR: 2982, 2869, 1618, 1453, 1394, 1323, 1280, 1217, 1146, 1097, 1065, 1014 cm^{-1}.



1f

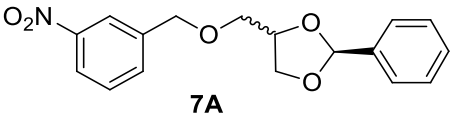
2-((4-(Trifluoromethyl)benzyl)oxy)propane-1,3-diol (1f)[13]: The title compound was synthesized according to general procedure A. White solid; Yield = 84.1%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, J = 8.1 Hz, 2H), 7.47 (d, J = 8.0 Hz, 2H), 4.72 (s, 2H), 3.86–3.72 (m, 4H), 3.60 (p, J = 4.7 Hz, 1H), 2.18 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ

142.10, 130.12 (dd, *J* = 64.8, 32.3 Hz), 127.71, 125.51 (q, *J* = 3.7 Hz), 124.08 (dd, *J* = 544.0, 272.0 Hz), 79.56, 71.12, 62.45 ppm; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.55 ppm.



1'f

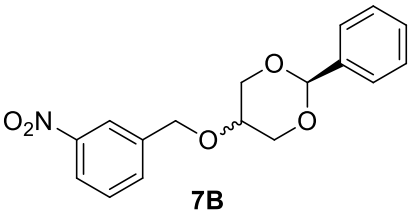
3-((4-(Trifluoromethyl)benzyl)oxy)propane-1,2-diol (1'f): The title compound was synthesized according to general procedure A. Colorless oil; Yield = 83.1%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.1 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 4.62 (s, 2H), 3.97–3.90 (m, 1H), 3.77–3.55 (m, 4H), 2.67 (d, *J* = 4.4 Hz, 1H), 2.17 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.81, 130.08 (q, *J* = 32.4 Hz), 127.66, 125.47 (q, *J* = 3.7 Hz), 124.10 (q, *J* = 272.0 Hz), 72.76, 72.09, 70.69, 63.97 ppm; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.56 ppm; HRMS (EI+) *m*/*z*: $[M]^+$ calcd for $C_{11}H_{13}F_3O_3$ 250.0817, found 250.0817; IR: 3377, 2926, 2873, 1718, 1621, 1456,1417, 1325, 1163, 1118, 1065, 1020 cm^{-1}.



7A

Yield of product **7A**+**7B** = 82% (**7A**: 19.3%, **7B**: 62.7%)

4-(((3-Nitrobenzyl)oxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Slightly orange-colored liquid; Yield = 19.3%; No attempt was made to separate the diastereomers mixture: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.28–8.08 (m, 2H both diastereomers), 7.74–7.62 (m, 1H both diastereomers), 7.57–7.45 (m, 3H both diastereomers), 7.42–7.31 (m, 3H, both diastereomers), 4.52–4.42 (m, 1H both diastereomers), 3.76–3.62 (m, 2H both diastereomers) ppm; **Cis:** δ 5.96 (s, 1H), 4.70 (s, 2H), 4.26 (dd, *J* = 8.2, 6.9 Hz, 1H), 3.87 (dd, *J* = 8.3, 6.8 Hz, 1H) ppm; **Trans:** δ 5.82 (s, 1H), 4.67 (s, 2H), 4.16–4.10 (m, 1H), 4.00 (dd, *J* = 8.2, 5.3 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 29.42, 129.46, 128.39, 129.28 (aromatic peaks of diastereomers); **Cis:** δ 148.37, 137.80, 133.32, 126.47, 122.70, 122.23, 103.97, 75.05, 72.28, 71.13, 67.52 ppm; **Trans:** δ 140.27, 137.38, 133.38, 126.66, 122.69, 122.26, 104.48, 75.39, 72.25, 71.49, 67.76 ppm; HRMS (FAB+) *m*/*z*: $[M+H]^+$ calcd for $C_{17}H_{17}NO_5$ 316.1185, found 316.1179; IR: 2874, 1734, 1528, 1528, 1395, 1349, 1216, 1096, 1022 cm^{-1}.

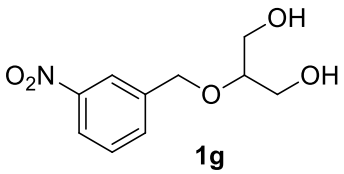


7B

(*2R*,*5R*)-5-((3-Nitrobenzyl)oxy)-2-phenyl-1,3-dioxane: The title compound was synthesized according to general procedure A. Light yellow-colored solid; mp = 87.8-88.6 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.27–8.14 (m, 2H), 7.65 (d, *J* = 7.6 Hz, 1H),

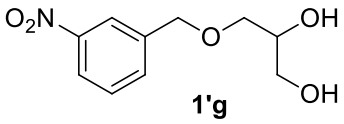
7.55–7.33 (m, 6H), 5.43 (s, 1H), 4.69 (s, 2H), 4.41 (dd, *J* = 11.0, 4.9 Hz, 2H), 3.87 – 3.79 (m, 1H), 3.70 (t, *J* = 10.6 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.41, 140.11, 137.47, 133.19, 129.50, 129.07, 128.32, 126.06, 122.90, 122.19, 101.36, 70.36, 69.93, 68.62 ppm; HRMS (FAB+) *m*/*z*: $[M+H]^+$ calcd for $C_{17}H_{17}NO_5$ 316.1185, found 316.1181; IR: 2979, 2914, 2875, 1619, 1583, 1527,1478, 1452, 1397, 1349, 1311, 1269, 1219, 1105 cm^{-1}.

(*2S*,*5R*)-5-((3-Nitrobenzyl)oxy)-2-phenyl-1,3-dioxane: The title compound was synthesized according to general procedure A. Thick yellow-colored solid; mp = 113.1-113.9 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.28 (s, 1H), 8.14 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.77 (d, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.9 Hz, 3H), 7.41–7.33 (m, 3H), 5.59 (s, 1H), 4.78 (s, 2H), 4.40 (dd, *J* = 12.7, 1.3 Hz, 2H), 4.11 (dd, *J* = 12.7, 1.5 Hz, 2H), 3.45–3.41 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.33, 140.59, 137.98, 133.36, 129.40, 129.00, 128.26, 126.15, 122.61, 122.18, 101.45, 70.63, 69.25, 68.86 ppm; HRMS (FAB+) *m*/*z*: $[M+H]^+$ calcd for $C_{17}H_{17}NO_5$ 316.1185, found 316.1181; IR: 2979, 2918, 2860, 1526, 1456, 1389, 1345, 1152, 1090 cm^{-1}.



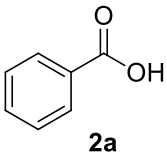
1g

2-((3-Nitrobenzyl)oxy)propane-1,3-diol (1g): The title compound was synthesized according to general procedure A. Orange-colored solid; mp = 61.2-62.0 ºC; Yield = 85.2%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 (s, 1H), 8.16 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.70 (dd, *J* = 7.6, 0.5 Hz, 1H), 7.54 (t, *J* = 7.9 Hz, 1H), 4.78 (s, 2H), 3.83 (ddd, *J* = 34.4, 11.7, 4.5 Hz, 4H), 3.65 (p, *J* = 4.7 Hz, 1H), 2.14 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.41, 140.35, 133.39, 129.49, 122.82, 122.32, 79.87, 70.69, 62.56 ppm; HRMS (CI+) *m*/*z*: $[M]^+$ calcd for $C_{10}H_{13}NO_5$ 228.0872, found 228.0879; IR: 3353, 2949, 2888, 1518, 1482, 1447, 1349, 1246, 1212, 1086, 1042 cm^{-1}.



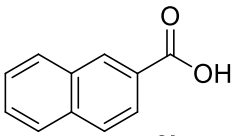
1'g

3-((3-Nitrobenzyl)oxy)propane-1,2-diol (1'g): The title compound was synthesized according to general procedure A. Pale yellow-colored solid; Yield = 76.5%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25–8.13 (m, 2H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.54 (t, *J* = 7.9 Hz, 1H), 4.66 (s, 2H), 3.99–3.93 (m, 1H), 3.79–3.59 (m, 4H), 2.57 (s, 1H), 2.04 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.41, 140.00, 133.36, 129.50, 122.83, 122.30, 72.30, 72.21, 70.67,63.87 ppm.



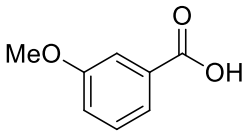
2a

Benzoic acid (2a)[11]**:** The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 12.74 (s, 1H), 8.27–8.06 (m, 2H), 7.71–7.43 (m, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.55, 133.85, 130.24, 129.35, 128.50 ppm.



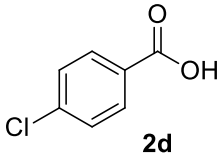
2b

2-Naphthoic acid (2b)[14]**:** The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.73 (s, 1H), 8.13 (dd, J = 8.6, 1.5 Hz, 1H), 8.04–7.87 (m, 3H), 7.67–7.54 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.73, 135.99, 132.47,132.18, 129.56, 128.69, 128.36, 127.84, 126.81, 126.46, 125.41 ppm.



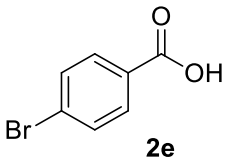
2c

3-Methoxybenzoic acid (2c)[15]**:** The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73 (d, J = 7.8 Hz, 1H), 7.63 (dd, J = 2.5, 1.5 Hz, 1H), 7.39 (t, J = 8.0 Hz, 1H), 7.17 (ddd, J = 8.3, 2.6, 0.8 Hz, 1H), 3.87 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.85, 159.64, 130.53, 129.55, 122.71, 120.52, 114.41, 55.48 ppm.



2d

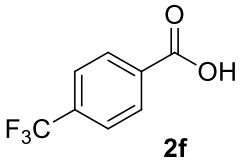
4-Chlorobenzoic acid (2d)[15]**:** The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, DMSO-d_6) δ 13.22 (s, 1H), 7.97 (d, J = 8.5 Hz, 2H), 7.58 (d, J = 8.5 Hz, 2H) ppm; ^{13}C NMR (126 MHz, DMSO-d_6) δ 166.93, 138.25, 131.59, 130.14, 129.16 ppm.



2e

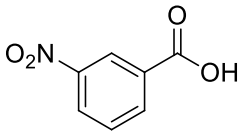
4-Bromobenzoic acid (2e)[16]**:** The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, Acetone-d_6) δ 11.45 (s,

1H), 8.00–7.95 (m, 2H), 7.75–7.69 (m, 2H) ppm; ^{13}C NMR (126 MHz, DMSO-d_6) δ 166.52, 131.61, 131.20, 129.92, 126.79 ppm.



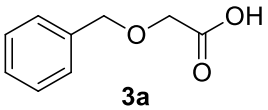
2f

4-(Trifluoromethyl)benzoic acid (2f)[16]**:** The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, Acetone) δ 11.72 (s, 1H), 8.30–8.22 (m, 2H), 7.89 (d, J = 8.1 Hz, 2H) ppm; ^{13}C NMR (126 MHz, DMSO-d_6) δ 166.68, 135.14, 132.92 (q, J = 31.8 Hz), 130.57, 126.07 (q, J = 3.7 Hz), 124.29 (q, J = 272.7 Hz) ppm; ^{19}F NMR (471 MHz, MeOD) δ -63.62 ppm.



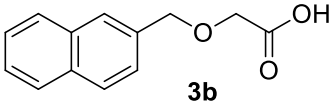
2g

3-Nitrobenzoic acid (2g)[16]**:** The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, DMSO-d_6) δ 13.70 (s, 1H), 8.65 – 8.59 (m, 1H), 8.47 (ddd, J = 8.2, 2.3, 0.9 Hz, 1H), 8.38 – 8.33 (m, 1H), 7.82 (t, J = 8.0 Hz, 1H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 165.44, 147.82, 135.29, 132.39, 130.46, 127.26, 123.61 ppm.



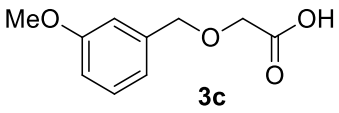
3a

2-(Benzyloxy)acetic acid (3a)[17]**:** The title compound was synthesized according to general procedure B. Slightly yellow-colored oil; Yield = 92%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42–7.29 (m, 5H), 4.65 (s, 2H), 4.15 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.55, 136.49, 128.64, 128.32, 128.14, 73.53, 66.59 ppm.



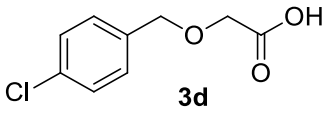
3b

2-(Naphthalen-2-ylmethoxy)acetic acid (3b): The title compound was synthesized according to general procedure B. Yellow-colored solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89–7.76 (m, 4H), 7.54–7.45 (m, 3H), 4.81 (s, 2H), 4.18 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.79, 133.96, 133.21, 133.19, 128.53, 127.94, 127.74, 127.16, 126.32, 126.23, 125.76, 73.60, 66.59 ppm.



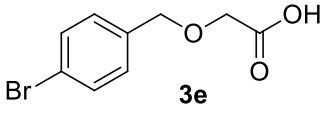
3c

2-((3-Methoxybenzyl)oxy)acetic acid (3c): The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (dd, J = 13.4, 5.6 Hz, 1H), 6.98–6.84 (m, 3H), 4.63 (s, 2H), 4.14 (s, 2H), 3.82 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.24, 159.89, 138.04, 129.70, 120.31, 113.95, 113.40, 73.40, 66.56, 55.27 ppm.



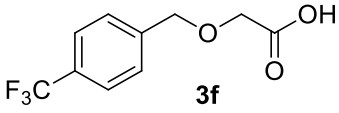
3d

2-((4-Chlorobenzyl)oxy)acetic acid (3d): The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.28 (m, 4H), 4.62 (s, 2H), 4.15 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.76, 135.03,134.17, 129.39, 128.83, 72.75, 66.70 ppm.



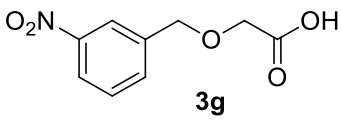
3e

2-((4-Bromobenzyl)oxy)acetic acid (3e): The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54–7.47 (m, 2H), 7.29–7.22 (m, 2H), 4.60 (s, 2H), 4.16 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.89, 135.61, 131.76, 129.67, 122.25, 72.75, 66.71 ppm.



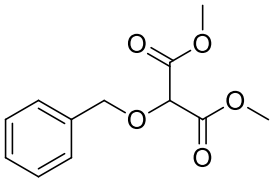
3f

2-((4-(Trifluoromethyl)benzyl)oxy)acetic acid (3f)[18]**:** The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, J = 8.1 Hz, 2H), 7.49 (d, J = 8.0 Hz, 2H), 4.71 (s, 2H), 4.20 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.11, 140.78, 130.40 (q, J = 32.4 Hz), 127.94, 125.56 (q, J = 3.8 Hz), 124.05 (dd, J = 544.1, 272.1 Hz), 72.69, 66.99 ppm; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.61 ppm.

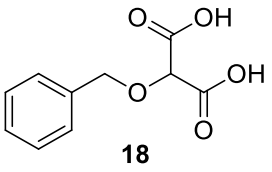


3g

2-((3-Nitrobenzyl)oxy)acetic acid (3g): The title compound was synthesized according to general procedure B. Yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25 (s, 1H), 8.18 (dd, J = 8.2, 1.3 Hz, 1H), 7.72 (d, J = 7.9 Hz, 1H), 7.56 (t, J = 7.9 Hz, 1H) , 4.75 (s, 2H), 4.26 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.14, 148.42, 139.05, 133.61, 129.60, 123.11, 122.57, 72.24, 67.20 ppm.

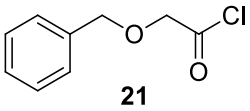


Dimethyl 2-(benzyloxy)malonate[19]: The title compound was synthesized according to general procedure D. Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41–7.30 (m, 5H), 4.71 (s, 2H), 4.57 (s, 1H), 3.80 (s, 6H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.92, 135.95, 128.58, 128.42, 128.41, 77.51, 72.89, 52.92 ppm.



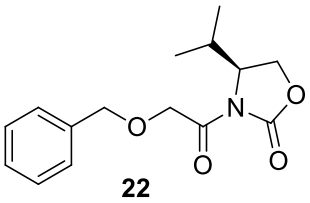
18

2-(Benzyloxy)malonic acid (18): The title compound was synthesized according to general procedure B. White solid; ^{1}H NMR (500 MHz, MeOD) δ 11.62 (s, 2H), 7.48 – 7.29 (m, 5H), 4.73 (s, 2H), 4.69 (s, 1H) ppm; ^{13}C NMR (126 MHz, MeOD) δ 170.15, 138.15, 129.52, 129.47, 129.25, 79.01, 73.52 ppm.



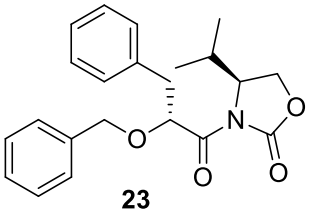
21

2-(Benzyloxy)acetyl chloride (21): The title compound was synthesized according to reference[17]. Transparent brown-colored liquid; Yield = 99%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.29 (m, 5H), 4.66 (s, 2H), 4.43 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.88, 136.07, 128.68, 128.46, 128.15, 74.82, 73.59 ppm.



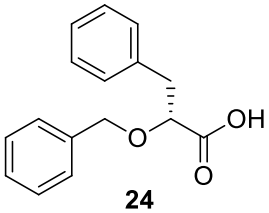
22

(*S*)-3-(2-(Benzyloxy)acetyl)-4-isopropyloxazolidin-2-one (22): The title compound was synthesized according to reference[20]. White solid; Yield = 70%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45–7.25 (m, 5H), 4.70 (s, 2H), 4.66 (s, 2H), 4.47–4.42 (m, 1H), 4.32 (t, J = 8.8 Hz, 1H), 4.25 (dd, J = 9.2, 3.1 Hz, 1H), 2.48–2.38 (m, 1H), 0.90 (dd, J = 24.6, 7.0 Hz, 6H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.10, 154.02, 137.22,128.50, 128.10, 128.01, 73.51, 69.63, 64.44, 58.24, 28.28, 17.92, 14.67 ppm.



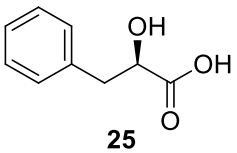
23

(*S*)-3-((*R*)-2-(Benzyloxy)-3-phenylpropanoyl)-4-isopropyl-oxazolidin-2-one (23): The title compound was synthesized according to general procedure C. Transparent brown-colored liquid; Yield = 73.4%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (d, J = 7.2 Hz, 2H), 7.31–7.20 (m, 6H), 7.12 (dd, J = 6.6, 2.9 Hz, 2H), 5.32 (dd, J = 9.2, 3.6 Hz, 1H), 4.50 (d, J = 11.7 Hz, 1H), 4.36 (td, J = 8.5, 4.2 Hz, 2H), 4.23–4.14 (m, 2H), 3.17 (dd, J = 13.5, 3.6 Hz, 1H), 2.94 (dd, J = 13.5, 9.2 Hz, 1H), 2.29–2.21 (m, 1H), 0.87 (d, J = 7.0 Hz, 3H), 0.76 (d, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.45, 153.65, 137.42, 137.09, 129.76, 128.24, 128.18, 128.07, 127.66, 126.63, 78.45, 72.81, 64.02, 58.27, 39.52, 28.42, 17.86, 14.76 ppm; HRMS (FAB+) m/z: $[M+H]^+$ calcd for $C_{22}H_{25}NO_4$ 368.1862, found 368.1863; IR: 2964, 2875, 1776, 1709, 1495, 1454, 1388, 1300, 1241, 1204, 1110 cm^{-1}.



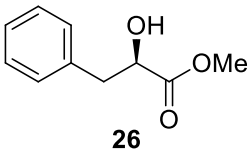
24

(*R*)-2-(Benzyloxy)-3-phenylpropanoic acid (24): The title compound was synthesized according to reference[21]. White solid; $[\alpha]_D^{20}$ +78.25° (*c* 0.8, EtOH). (lit. $[\alpha]_D^{49}$ +72.5° (*c* 0.8, EtOH)) [22]; Yield = 83.4%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51–7.10 (m, 10H), 4.63 (dd, J = 11.6, 4.5 Hz, 1H), 4.43 (d, J = 11.7 Hz, 1H), 4.20 (dd, J = 8.3, 4.1 Hz, 1H), 3.12 (ddd, J = 22.4, 14.1, 6.2 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.16, 136.79,136.68, 129.53,128.40, 128.39, 127.95, 127.90, 126.84, 78.61, 72.77, 38.96 ppm.



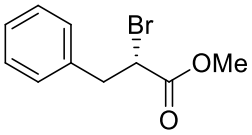
25

(*R*)-2-Hydroxy-3-phenylpropanoic acid (25): The title compound was synthesized according to reference[23]. White solid; $[\alpha]_D^{20}$ +26.7° (*c* 1.0, $(CH_3)_2CO$). (lit. $[\alpha]_D^{20}$ +26.95° (*c* 1.0, $(CH_3)_2CO$)) [24]; Yield = 83.4%; ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.43–7.00 (m, 5H), 4.13 (dd, J = 7.8, 4.2 Hz, 1H), 2.86 (ddd, J = 22.0, 13.7, 6.2 Hz, 2H) ppm; ^{13}C NMR (126 MHz, DMSO-d_6) δ 175.01, 138.03, 129.28,127.85, 125.99, 70.94, 39.89 ppm.

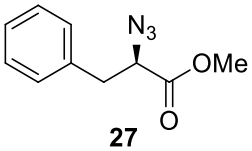


26

(*R*)-Methyl 2-hydroxy-3-phenylpropanoate (26): The title compound was synthesized according to reference[25]. White solid; $[\alpha]_D^{20}$ +7.2° (*c* 1.0, $CHCl_3$). (lit. $[\alpha]_D^{22}$ +6.1° (*c* 1.0, $CHCl_3$) [26]; Yield = 86.1%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.18 (m, 5H), 4.45 (td, *J* = 6.5, 4.5 Hz, 1H), 3.77 (s, 3H), 3.12 (dd, *J* = 13.9, 4.4 Hz, 1H), 2.96 (dd, *J* = 13.9, 6.8 Hz, 1H), 2.72 (dd, *J* = 11.4, 6.2 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.58, 136.42, 129.47, 128.41, 126.87, 71.34, 52.39, 40.55 ppm.

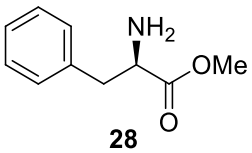


(*S*)-methyl 2-bromo-3-phenylpropanoate: The title compound was synthesized according to reference[27]. Colorless oil; $[\alpha]_D^{20}$ -21.6° (*c* 1.28, $CHCl_3$). (lit. $[\alpha]_D^{24}$ -21.8° (*c* 1.28, $CHCl_3$) [27]; Yield = 90%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34–7.18 (m, 5H), 4.40 (dd, *J* = 8.4, 7.1 Hz, 1H), 3.72 (s, 3H), 3.46 (dd, *J* = 14.1, 8.4 Hz, 1H) , 3.24 (dd, *J* = 14.1, 7.1 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.89 , 136.73, 129.17 , 128.69 , 127.37 , 52.93, 45.10, 41.14 ppm.



27

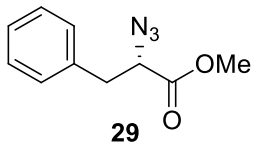
(*R*)-Methyl 2-azido-3-phenylpropanoate (27): The title compound was synthesized according to reference[28]. Colorless oil; $[\alpha]_D^{20}$ +45.4° (*c* 1.0, $CHCl_3$). (lit. $[\alpha]_D^{28}$ +47.8° (*c* 1.0, $CHCl_3$) [29]; Yield = 92%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.21 (m, 5H), 4.07 (dd, *J* = 8.8, 5.4 Hz, 1H), 3.77 (s, 3H), 3.18 (dd, *J* = 14.0, 5.4 Hz, 1H), 3.01 (dd, *J* = 14.0, 8.8 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.40, 135.93, 129.18, 128.71, 127.29, 63.28, 52.64, 37.67 ppm.



28

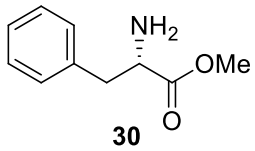
(*R*)-Methyl 2-amino-3-phenylpropanoate (28): The title compound was synthesized according to reference[30]. Colorless oil; $[\alpha]_D^{20}$ -26.2° (*c* 4.0, EtOH). (lit. $[\alpha]_D^{23}$ -25.0° (*c* 4.04, EtOH) [31]; Yield = 85%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.16 (m, 5H), 3.80–3.68 (m, 4H), 3.09 (dd, *J* = 13.5, 5.2 Hz, 1H), 2.86 (dd, *J* = 13.5, 7.9 Hz, 1H), 1.52 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.44, 137.24, 129.27, 128.57, 126.84, 55.84, 51.96, 41.11 ppm; HPLC

analysis: Chiralpak AY-H, (hexane/EtOH/2-aminoethanol = 1/1/0.1 %, wavelength = 257 nm, flow rate = 1.0 mL/min), t_R = 6.2 min (major), t_R = 7.5 min (minor).



29

(*S*)-Methyl 2-azido-3-phenylpropanoate (29): The title compound was synthesized according to reference[32]. Colorless oil; $[\alpha]_D^{20}$ -45.2° (*c* 1.0, $CHCl_3$). (lit. $[\alpha]_D^{28}$ -47.8° (*c* 1.0, $CHCl_3$) [29]; Yield = 88.6%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.21 (m, 5H), 4.07 (dd, *J* = 8.8, 5.4 Hz, 1H), 3.77 (s, 3H), 3.18 (dd, *J* = 14.0, 5.4 Hz, 1H), 3.01 (dd, *J* = 14.0, 8.8 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.40, 135.93, 129.18, 128.71, 127.29, 63.28, 52.64, 37.67 ppm.



30

(*S*)-Methyl 2-amino-3-phenylpropanoate (30): The title compound was synthesized according to reference[30]. Colorless oil; $[\alpha]_D^{20}$ +26.5° (*c* 4.0, EtOH). (lit. $[\alpha]_D^{23}$ +25.0° (*c* 4.04, EtOH) [31]; Yield = 85%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.16 (m, 5H), 3.80–3.68 (m, 4H), 3.09 (dd, *J* = 13.5, 5.2 Hz, 1H), 2.86 (dd, *J* = 13.5, 7.9 Hz, 1H), 1.52 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.44, 137.24, 129.27, 128.57, 126.84, 55.84, 51.96, 41.11 ppm; HPLC analysis: Chiralpak AY-H (hexane/EtOH/2-aminoethanol = 1/1/0.1 %, wavelength = 257 nm, flow rate = 1.0 mL/min), t_R = 6.2 min (minor), t_R = 7.3 min (major).

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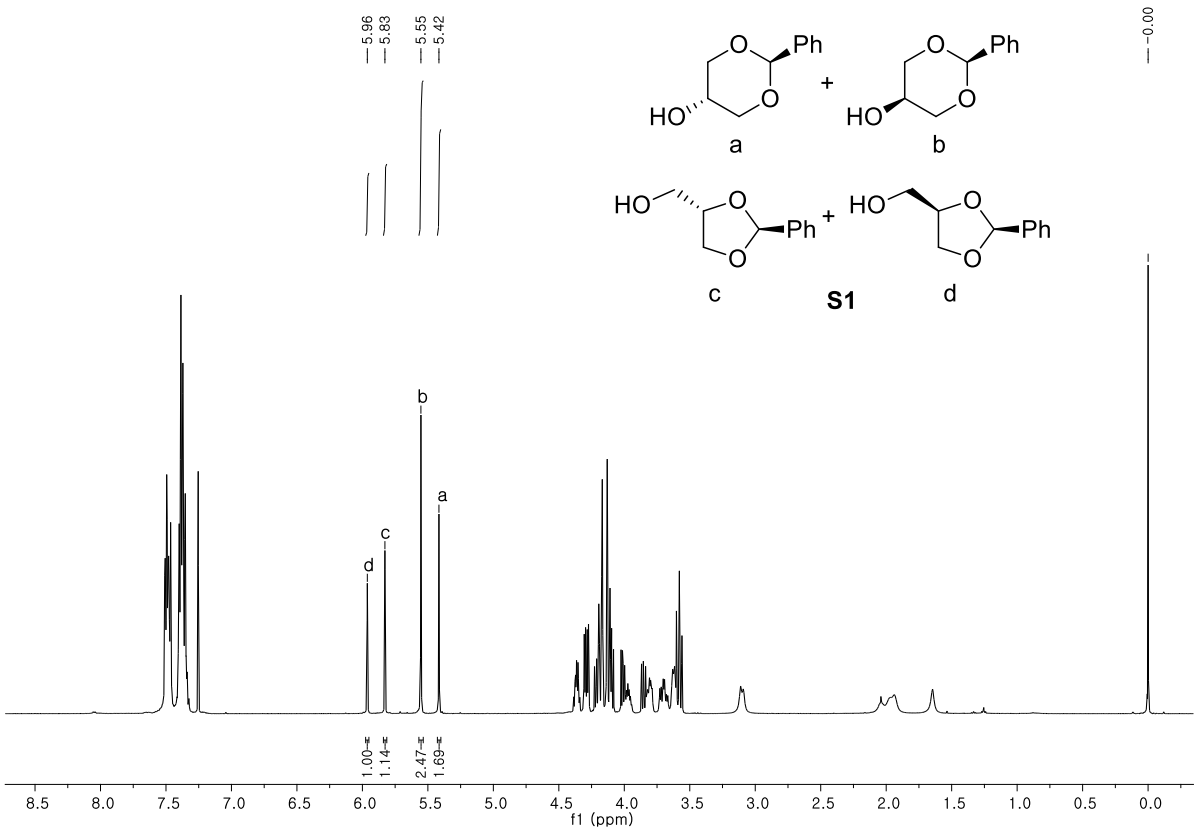
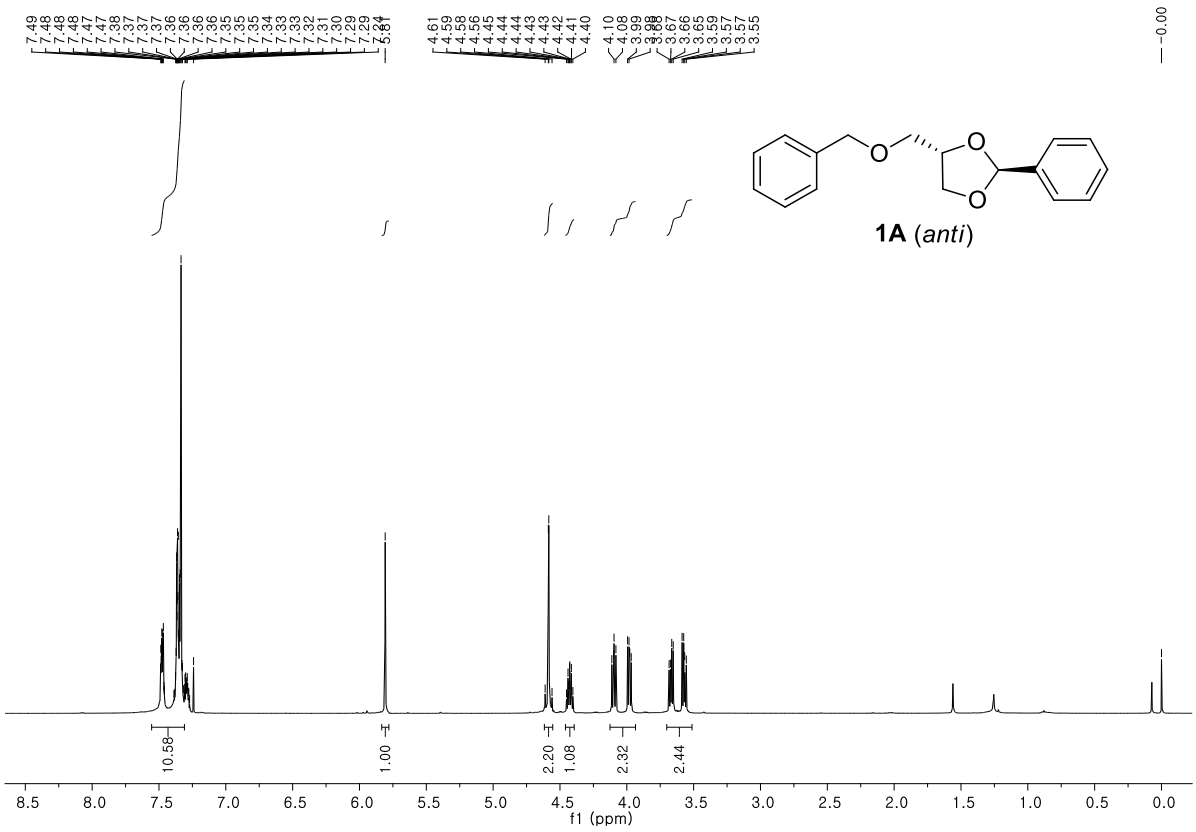
^{1}H NMR and ^{13}C NMR spectra of the products

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4.61 4.59 4.58 4.56 4.45 4.44 4.44 4.43 4.42 4.41 4.40
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0.00
O
O
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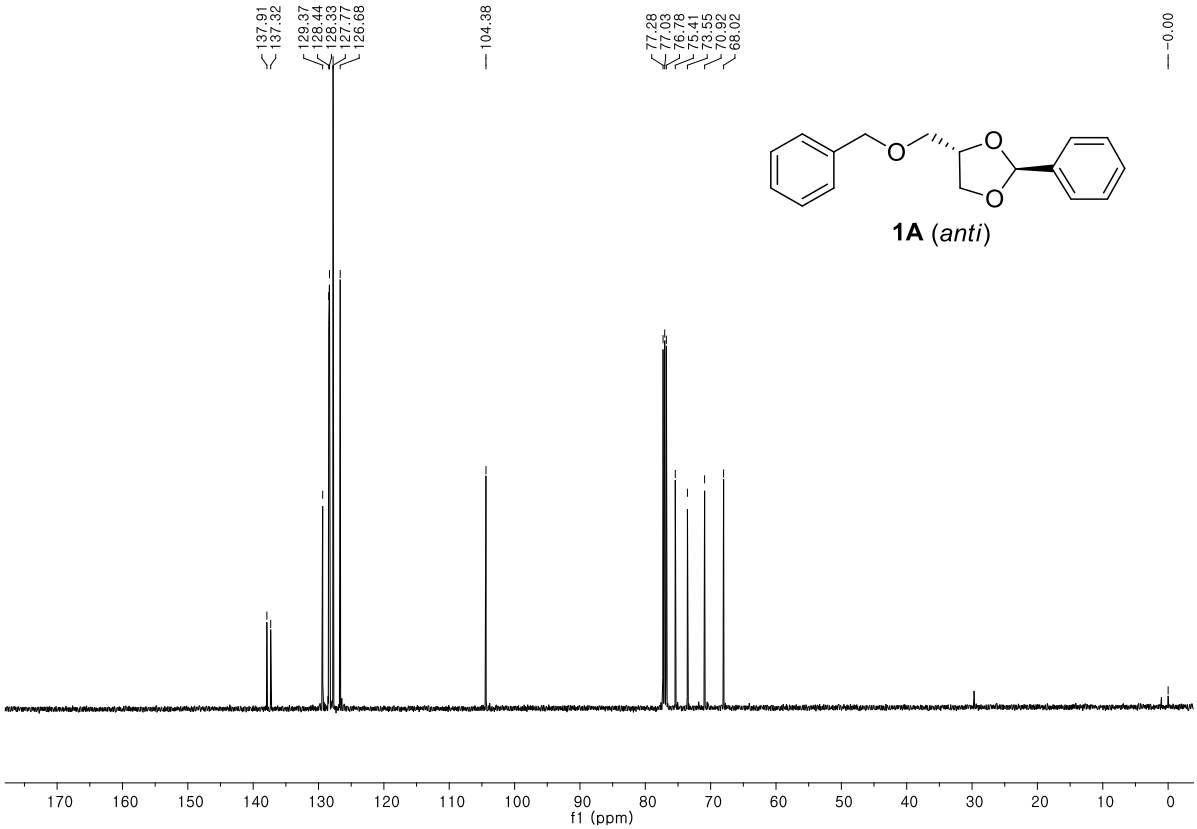
1A (anti)
10.58
1.00
2.20
1.08
2.32
2.44
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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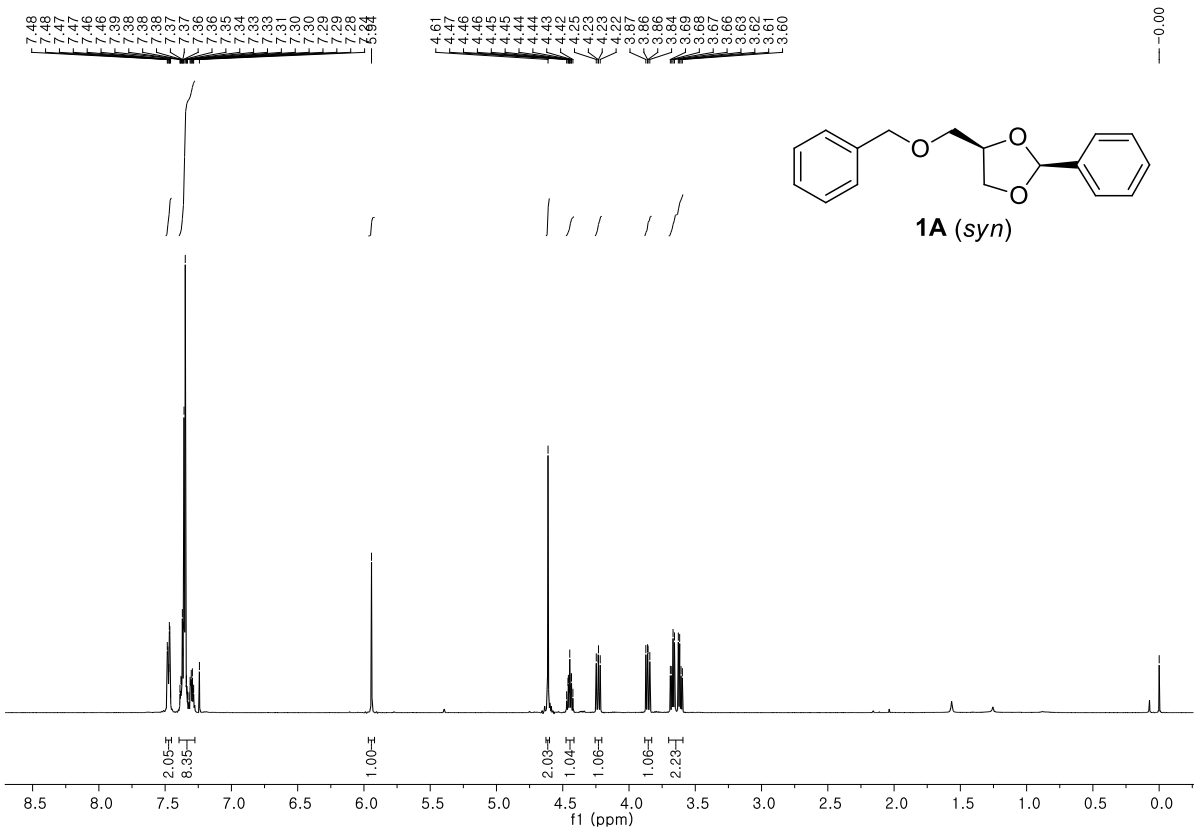
f1 (ppm)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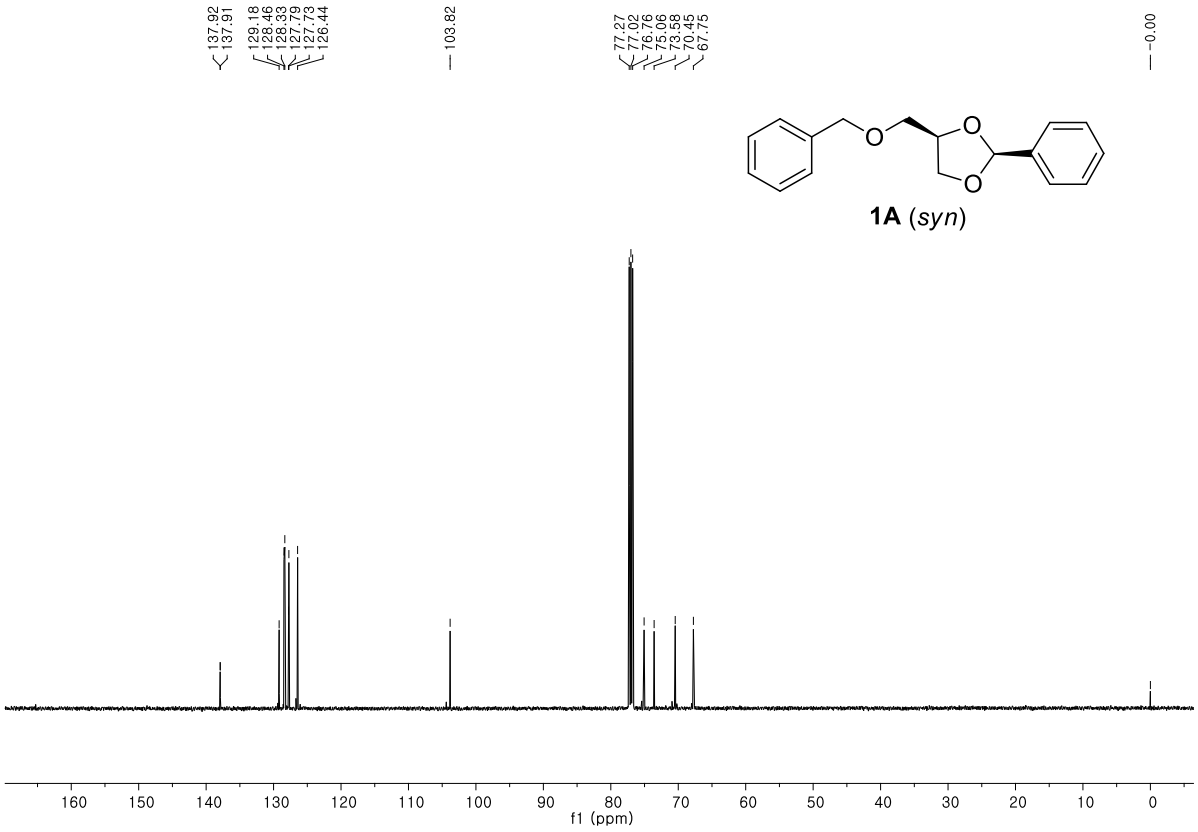
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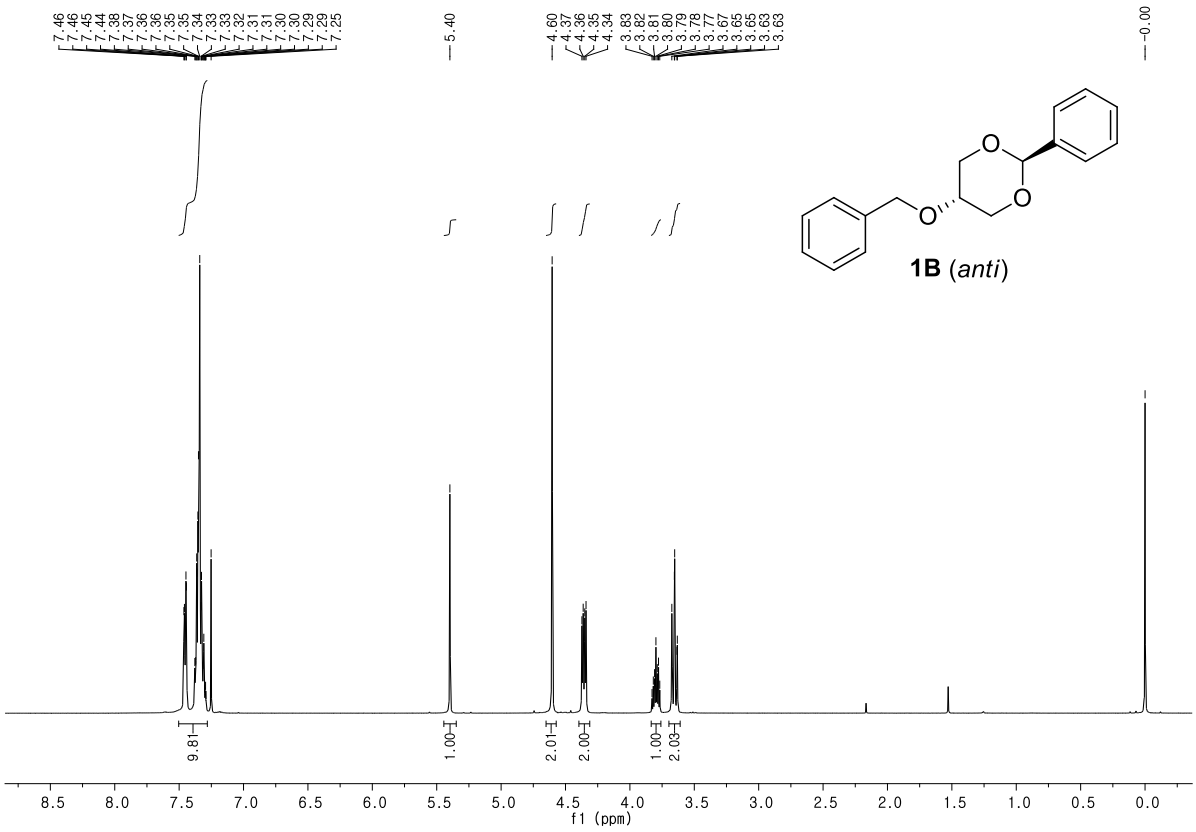
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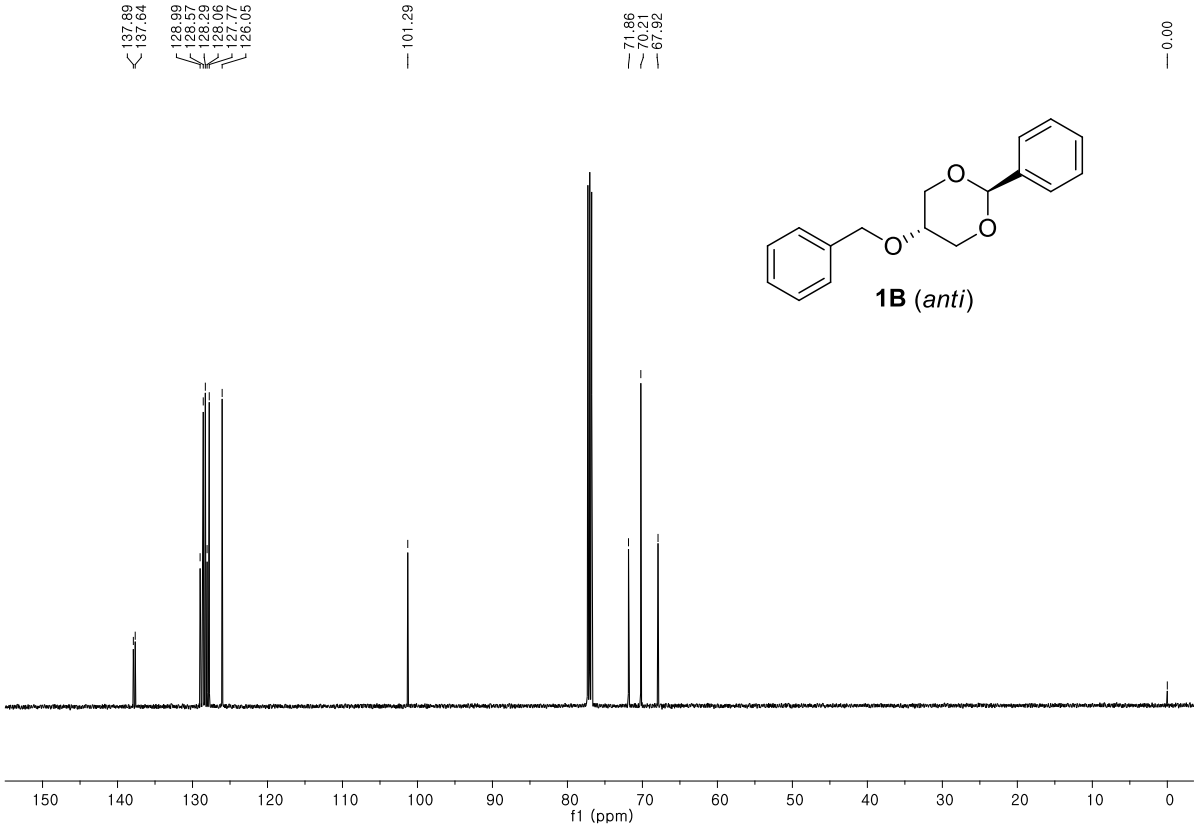
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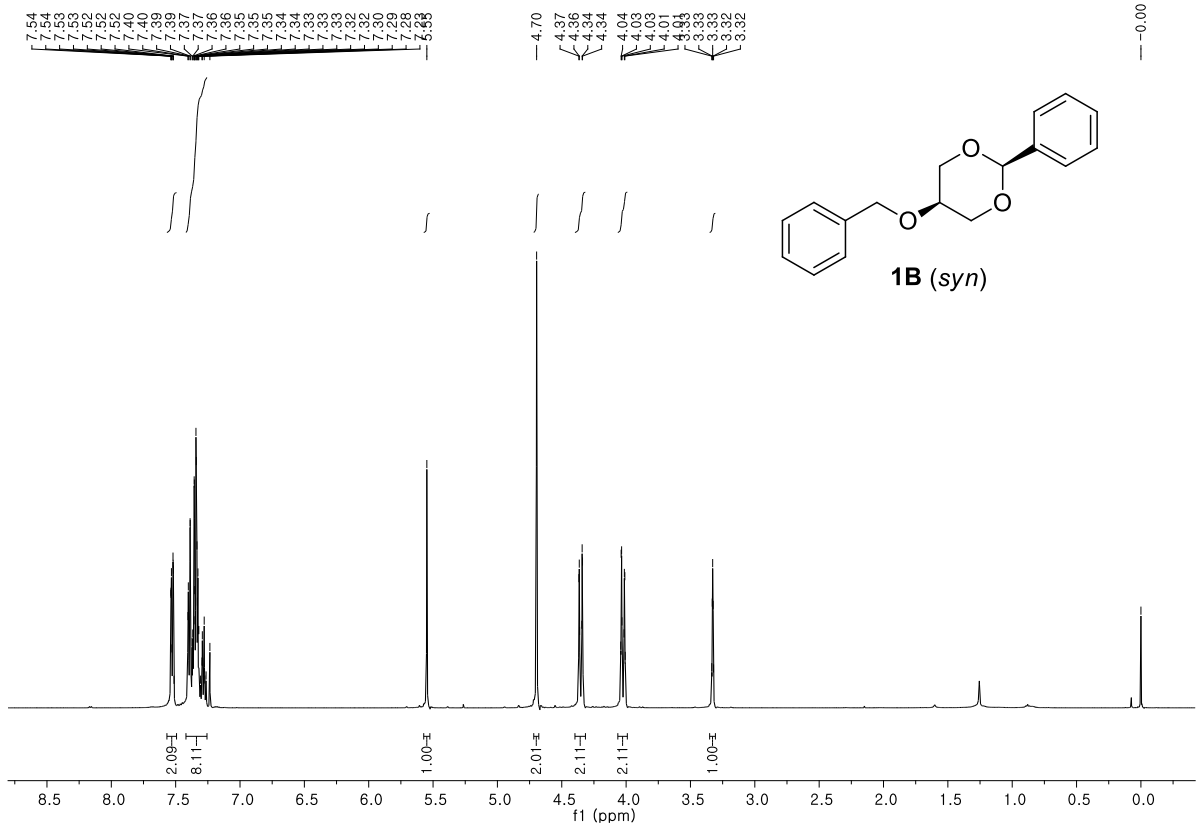
f1 (ppm)
1B (anti)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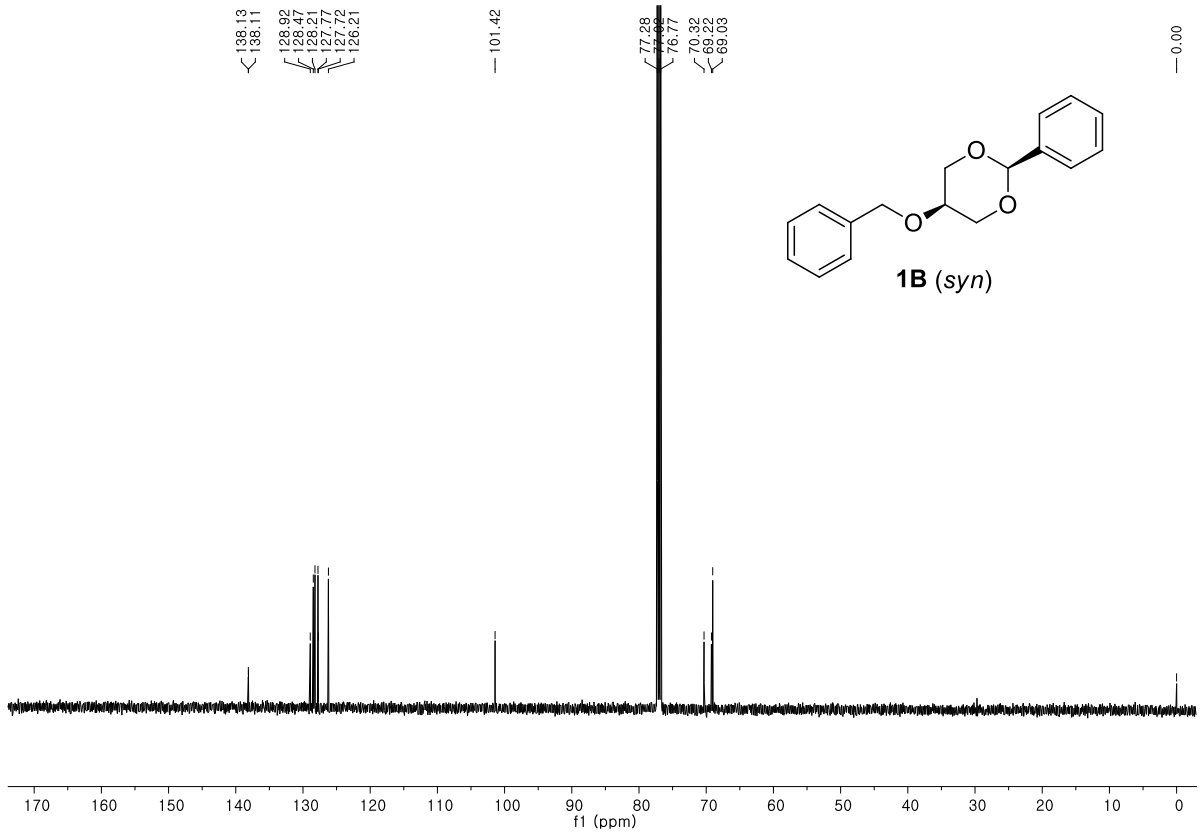
7.53
7.53
7.52
7.52
7.52
7.40
7.40
7.39
7.39
7.37
7.37
7.36
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.30
7.29
7.28
5.33
4.70
4.37
4.36
4.34
4.34
4.04
4.03
4.03
4.01
3.33
3.33
3.32
3.32
0.00
O
O
O
1B (syn)
2.09
8.11
1.00
2.01
2.11
2.11
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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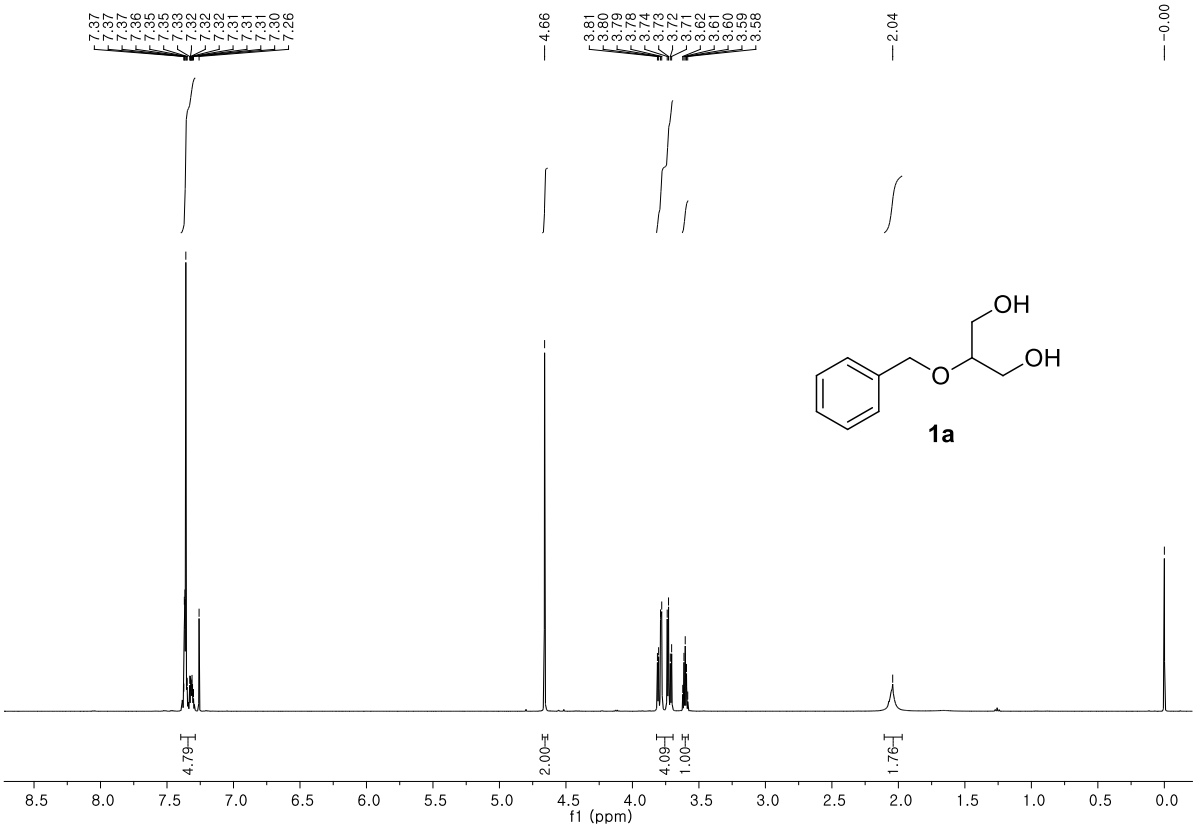
128.47
128.21
127.77
127.72
126.21
101.42
77.28
77.02
76.77
70.32
69.22
69.03
0.00
O
O
O
1B (syn)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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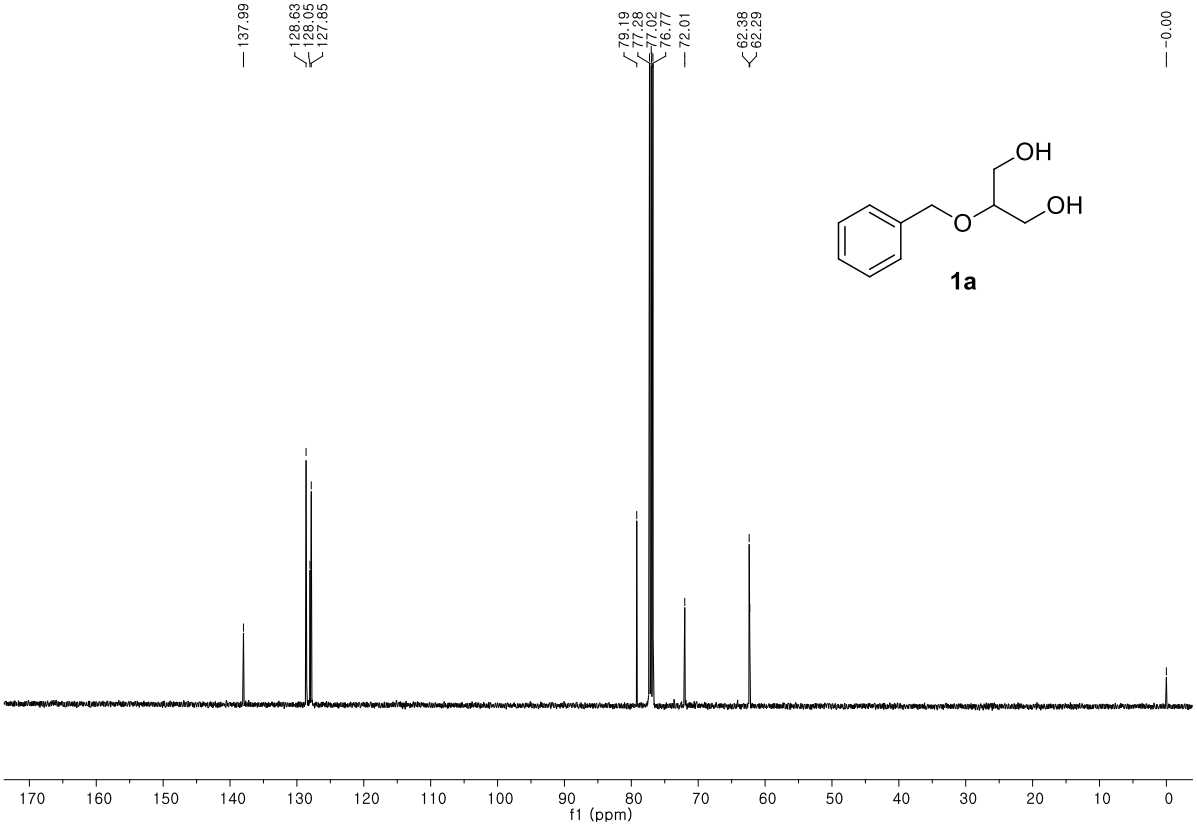
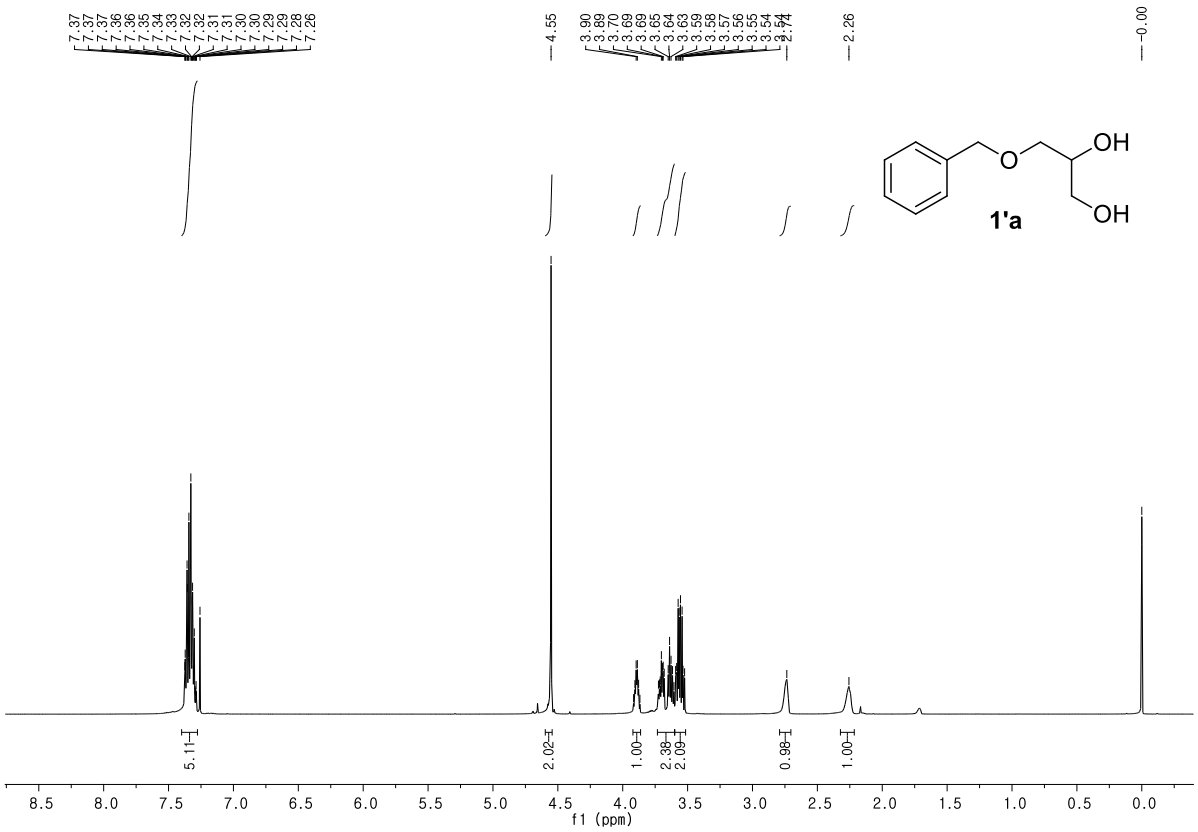
7.31
7.31
7.30
7.26
4.66
3.81
3.80
3.79
3.78
3.74
3.73
3.72
3.71
3.62
3.61
3.60
3.59
3.58
2.04
0.00
OH
OH
O
1a
4.79
2.00
4.09
1.00
1.76
f1 (ppm)


137.99
128.63
128.05
127.85
79.19
77.28
77.02
76.77
72.01
62.38
62.29
0.00
OH
OH
O
1a
f1 (ppm)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7.37
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7.33
7.32
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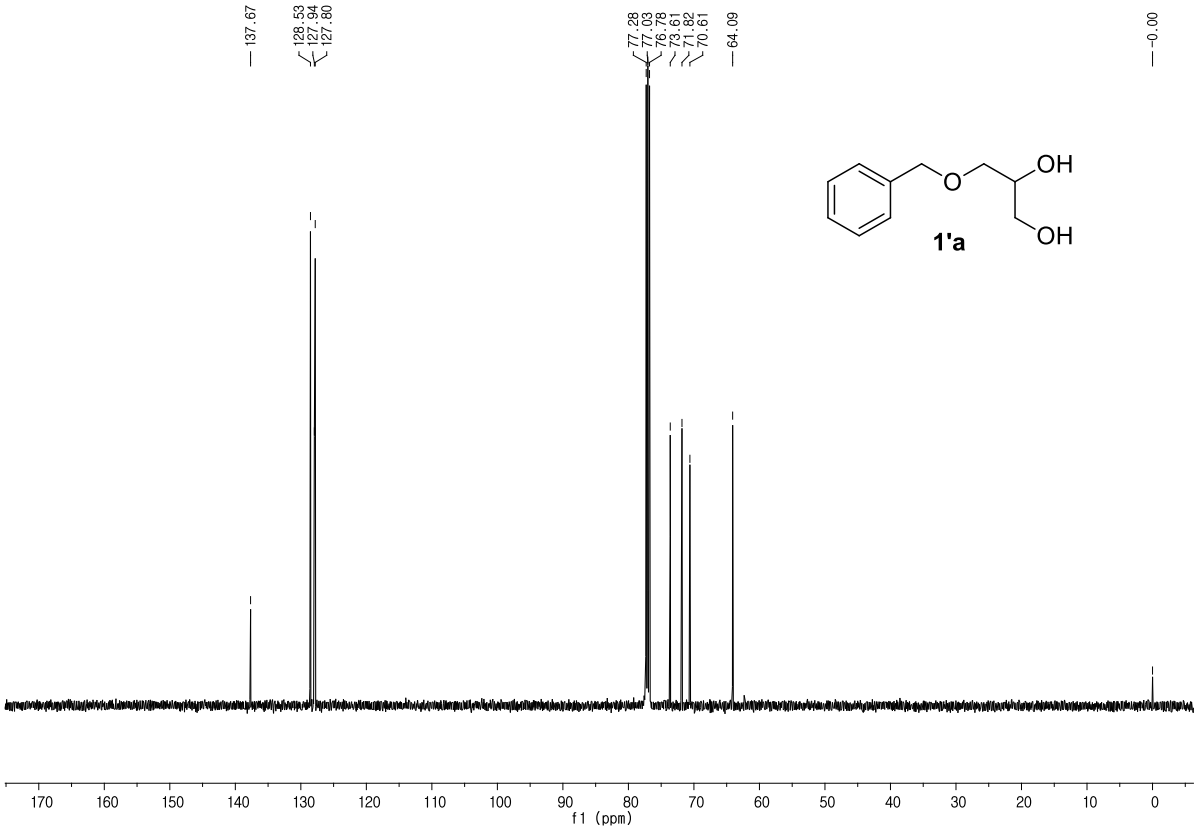
7.31
7.31
7.30
7.30
7.29
7.29
7.28
7.26
4.55
3.90
3.89
3.70
3.69
3.69
3.65
3.64
3.63
3.58
3.57
3.56
3.55
3.54
2.54
2.26
0.00
OH
O
OH
1'a
5.11
2.02
1.00
2.38
2.09
0.98
1.00
f1 (ppm)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128.53
127.94
127.80
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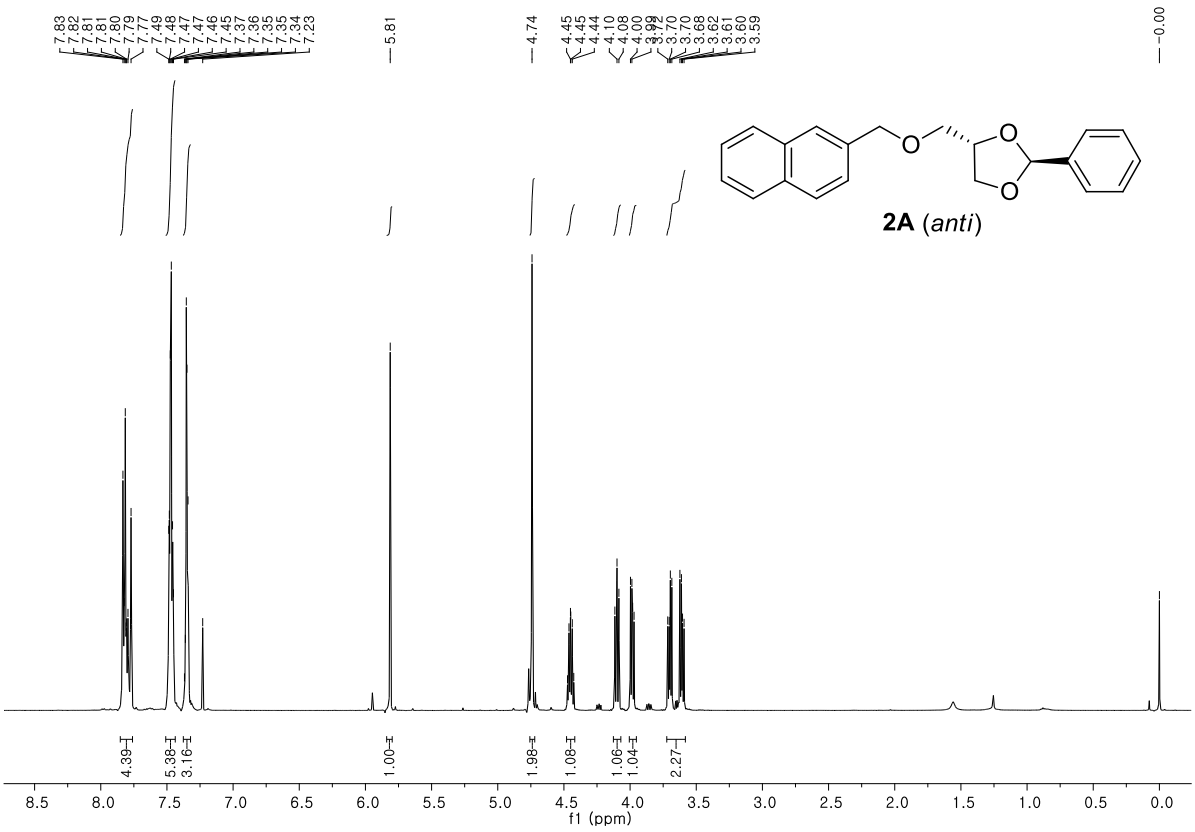
76.78
73.61
71.82
70.61
64.09
0.00
OH
O
OH
1'a
f1 (ppm)


7.83
7.82
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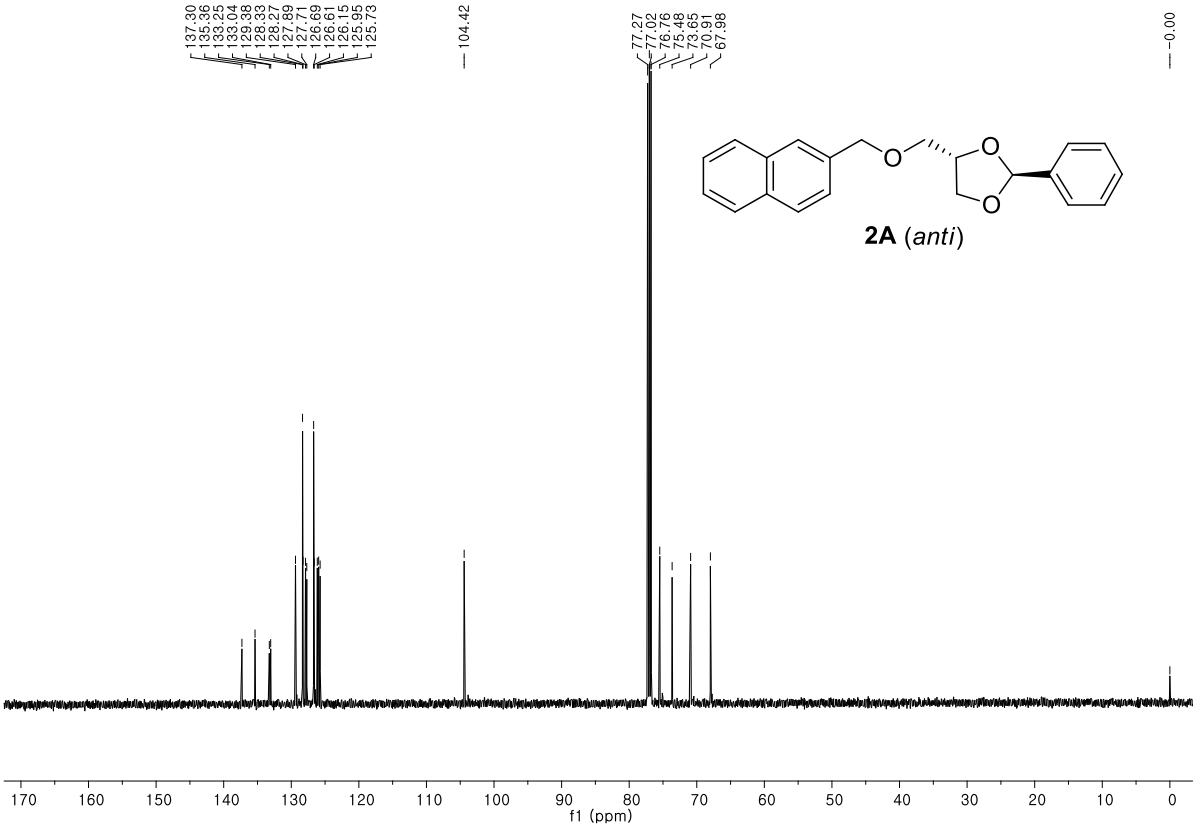
7.81
7.80
7.79
7.77
7.49
7.48
7.47
7.47
7.46
7.45
7.37
7.36
7.35
7.35
7.34
7.23
5.81
4.74
4.45
4.45
4.44
4.10
4.08
4.00
3.72
3.70
3.70
3.68
3.62
3.61
3.60
3.59
0.00
2A (anti)
4.39
5.38
3.16
1.00
1.98
1.08
1.06
1.04
2.27
f1 (ppm)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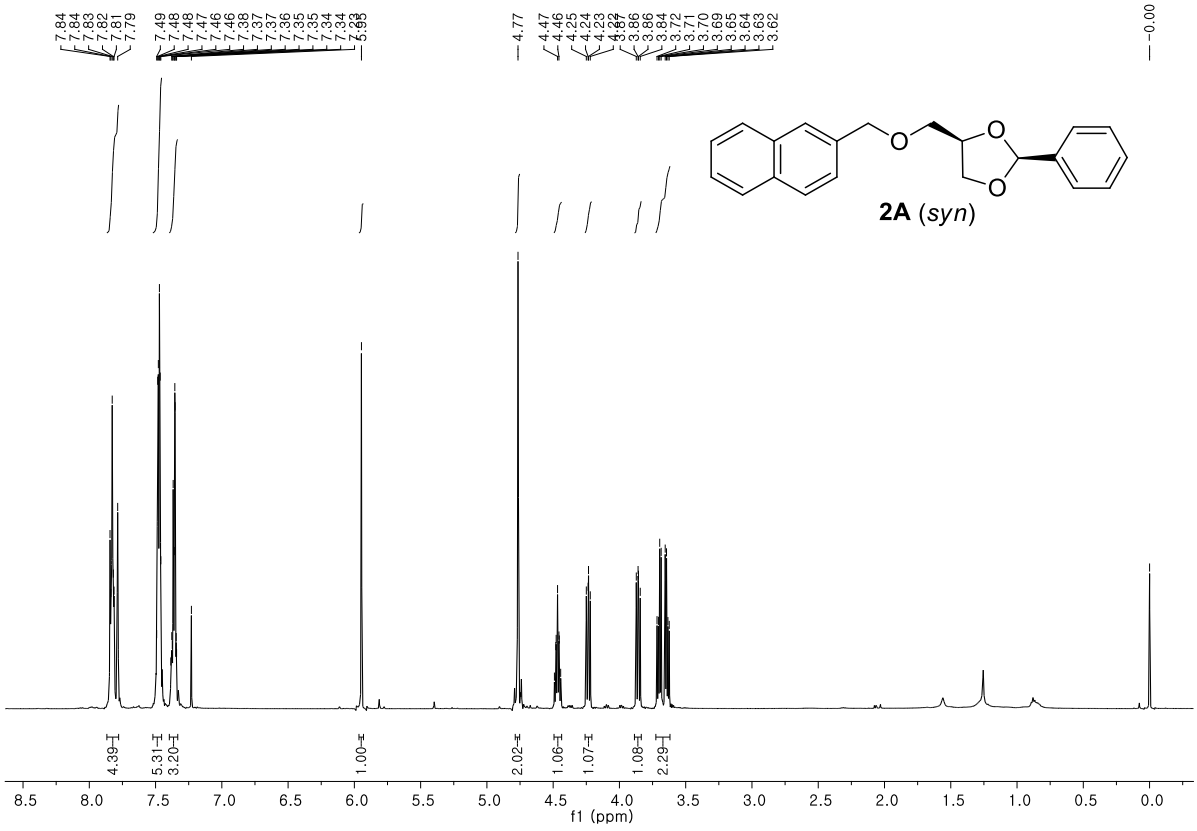
135.36
133.25
133.04
129.38
128.33
128.27
127.89
127.71
126.69
126.61
126.15
125.95
125.73
104.42
77.27
77.02
76.76
75.48
73.65
70.91
67.98
0.00
2A (anti)
f1 (ppm)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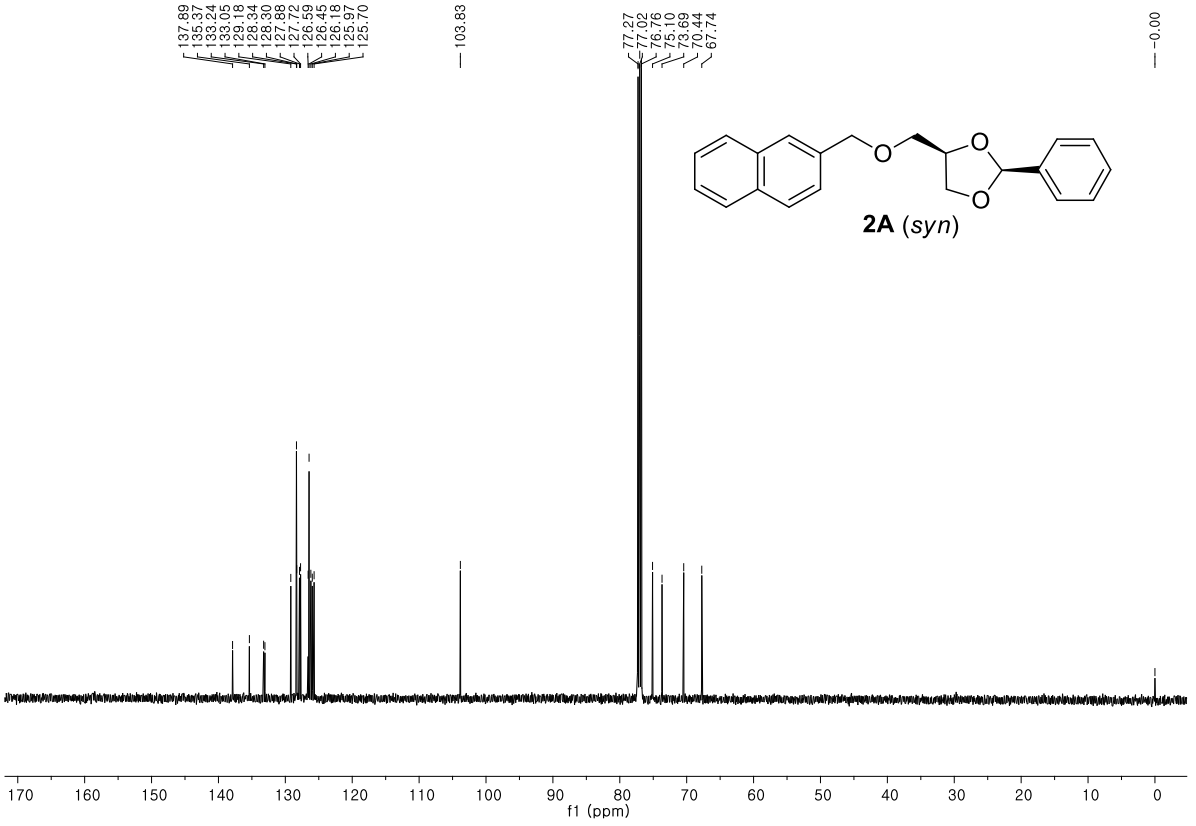
7.84
7.83
7.82
7.81
7.79
7.49
7.48
7.48
7.47
7.46
7.46
7.38
7.37
7.37
7.36
7.35
7.35
7.34
7.34
5.95
4.77
4.47
4.46
4.25
4.24
4.23
3.87
3.86
3.86
3.84
3.72
3.71
3.70
3.69
3.65
3.64
3.63
3.62
0.00
2A (syn)
4.39
5.31
3.20
1.00
2.02
1.06
1.07
1.08
2.29
f1 (ppm)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135.37
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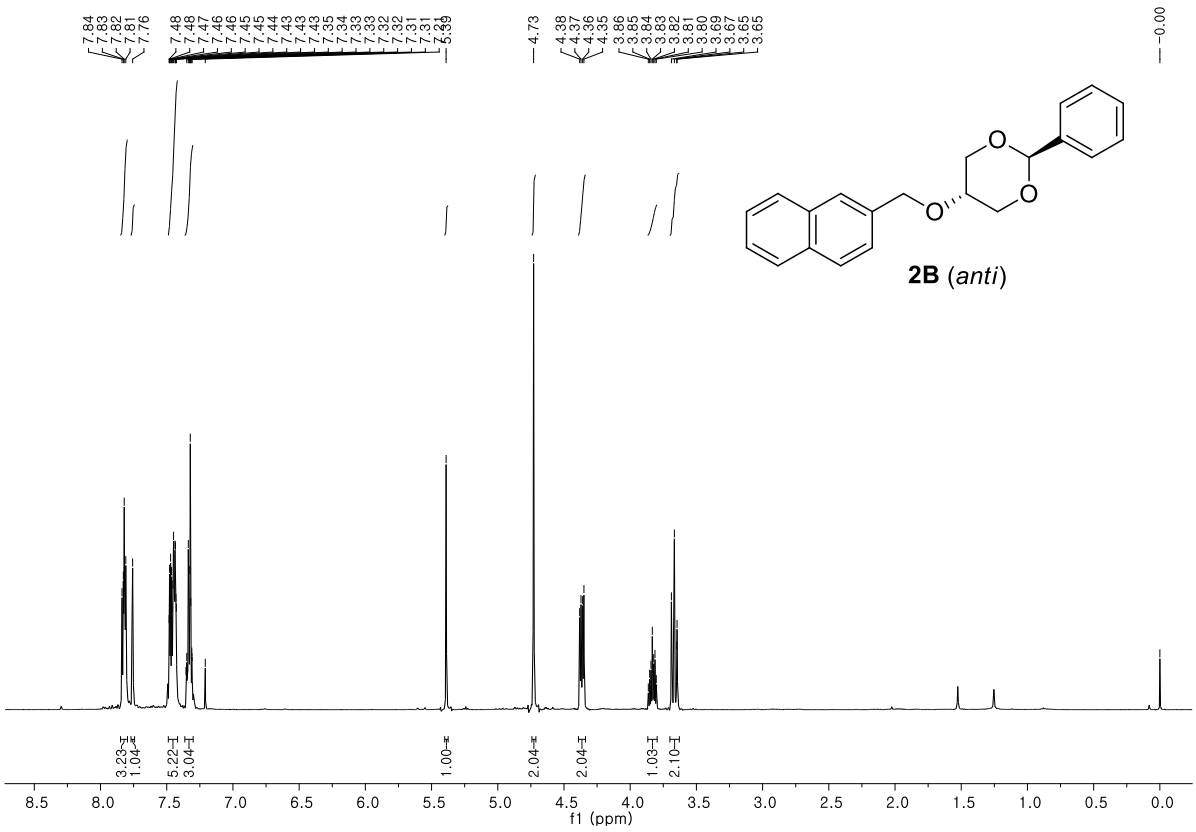
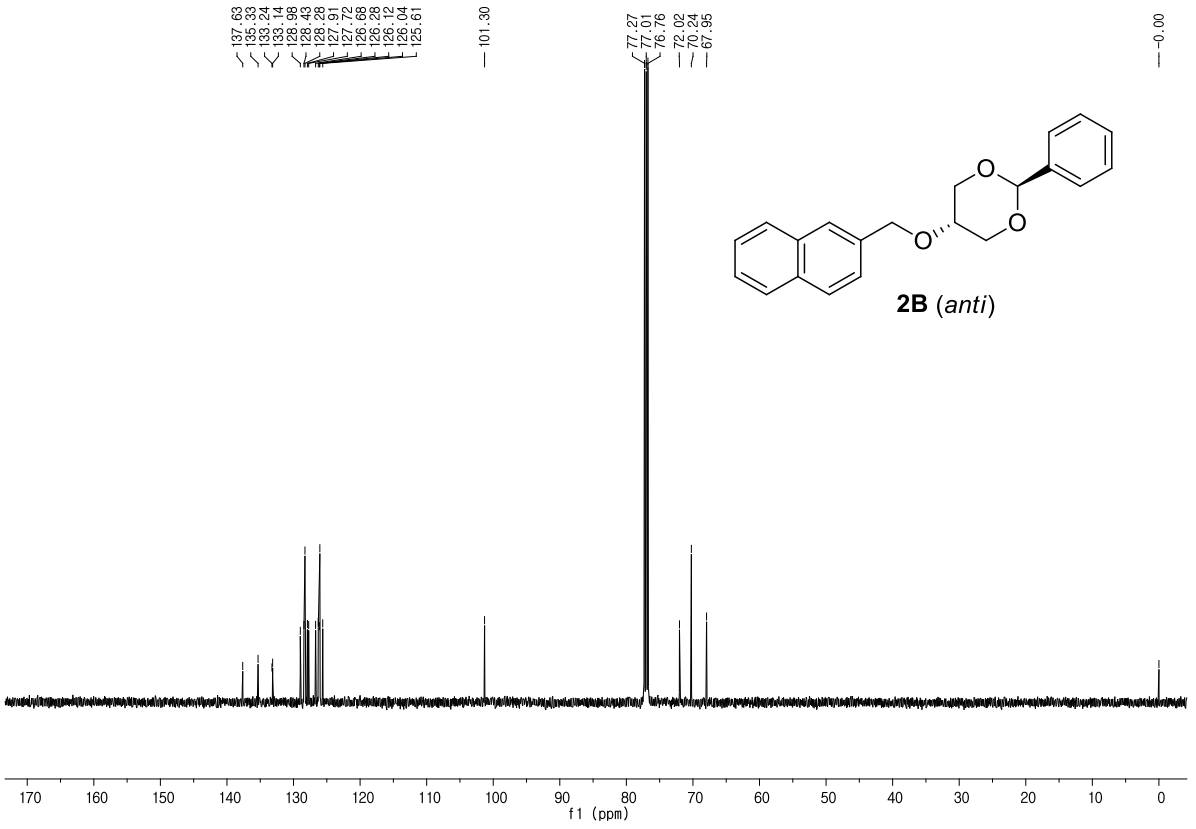
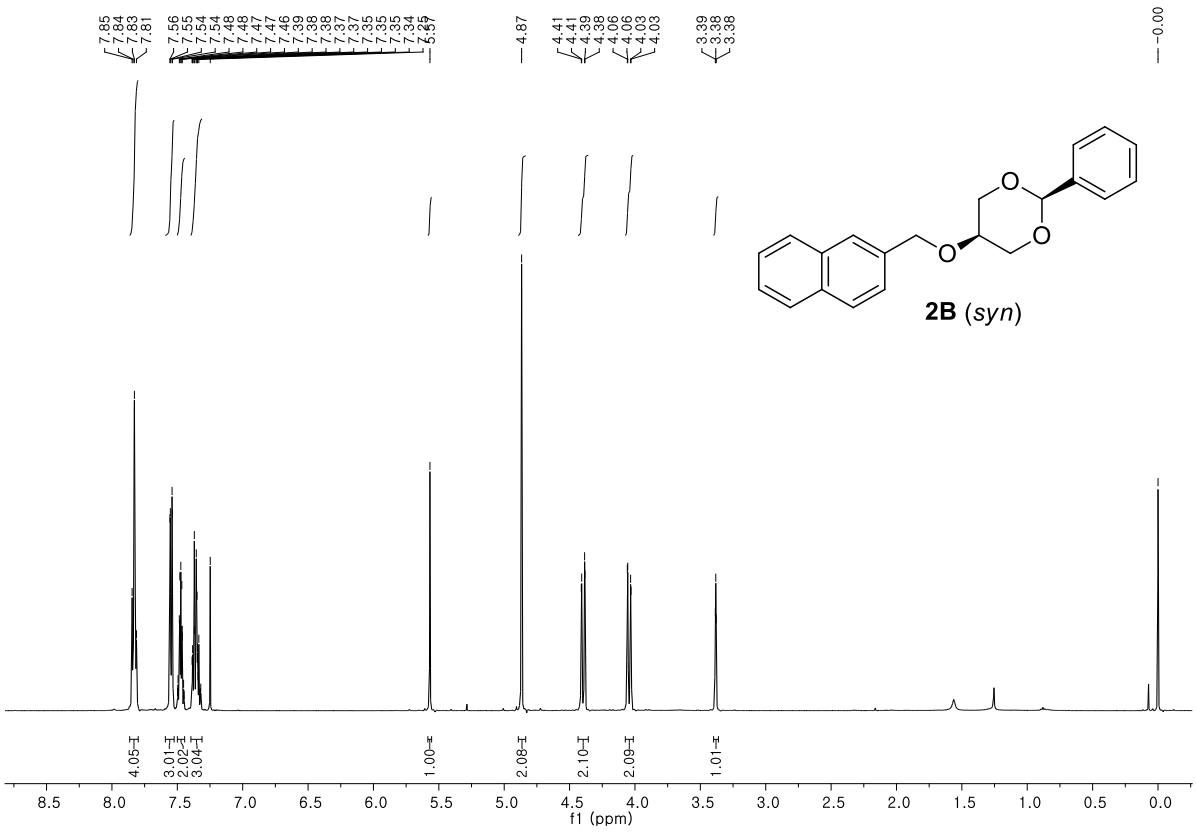
133.05
129.18
128.34
128.30
127.88
127.72
126.59
126.45
126.18
125.97
125.70
103.83
77.27
77.02
76.76
75.10
73.69
70.44
67.74
0.00
2A (syn)
f1 (ppm)



2B (*anti*)



2B (*anti*)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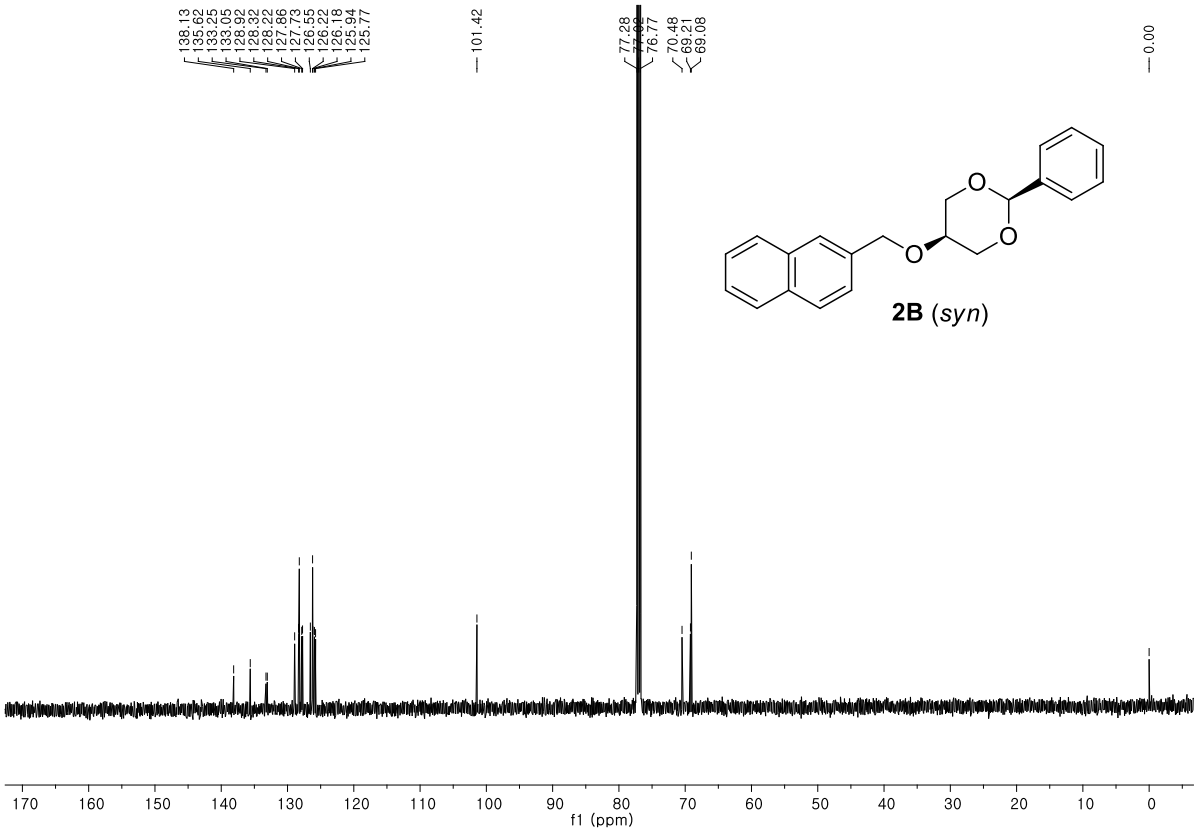
7.84
7.83
7.81
7.56
7.55
7.54
7.54
7.48
7.48
7.47
7.47
7.46
7.39
7.38
7.38
7.37
7.37
7.35
7.35
7.34
5.37
4.87
4.41
4.41
4.39
4.38
4.06
4.06
4.03
4.03
3.39
3.38
3.38
0.00
O
O
O
2B (syn)
4.05
3.01
2.02
3.04
1.00
2.08
2.10
2.09
1.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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135.62
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133.05
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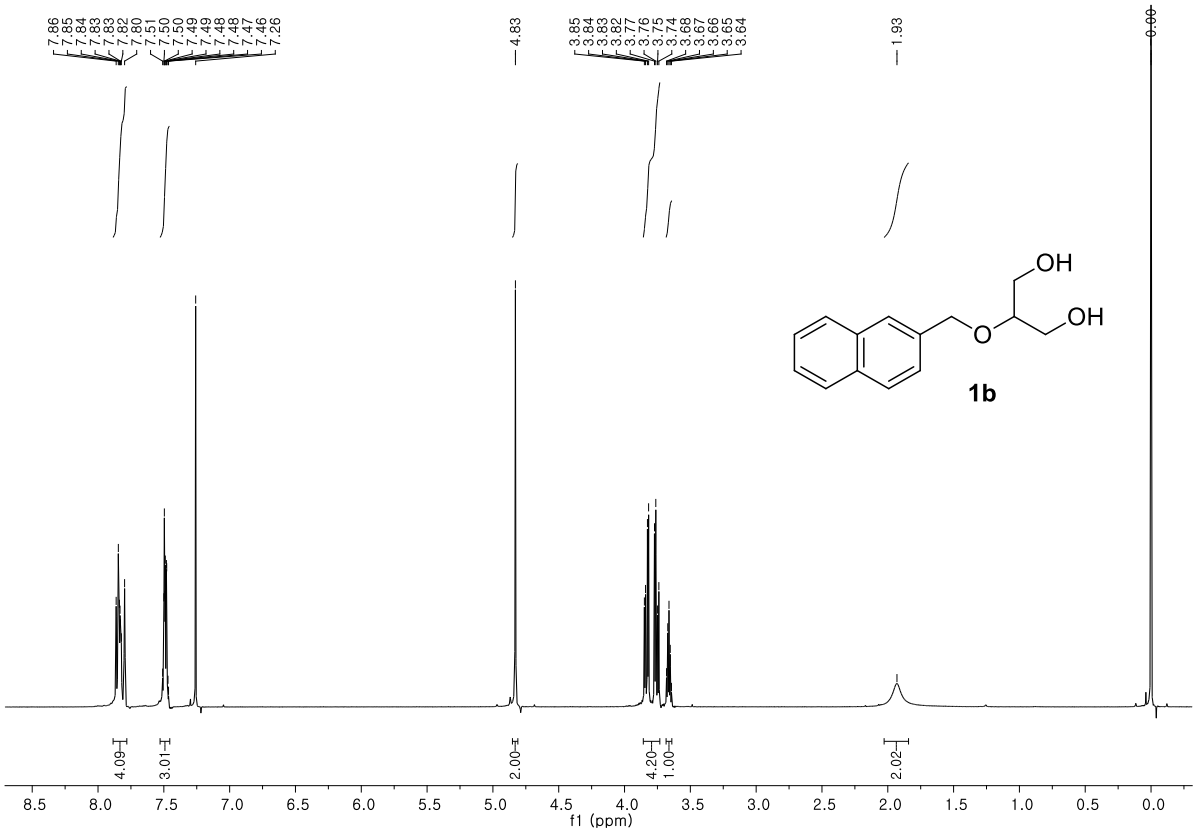
128.32
128.22
127.86
127.73
126.55
126.22
126.18
125.94
125.77
101.42
77.28
77.03
76.77
70.48
69.21
69.08
0.00
O
O
O
2B (syn)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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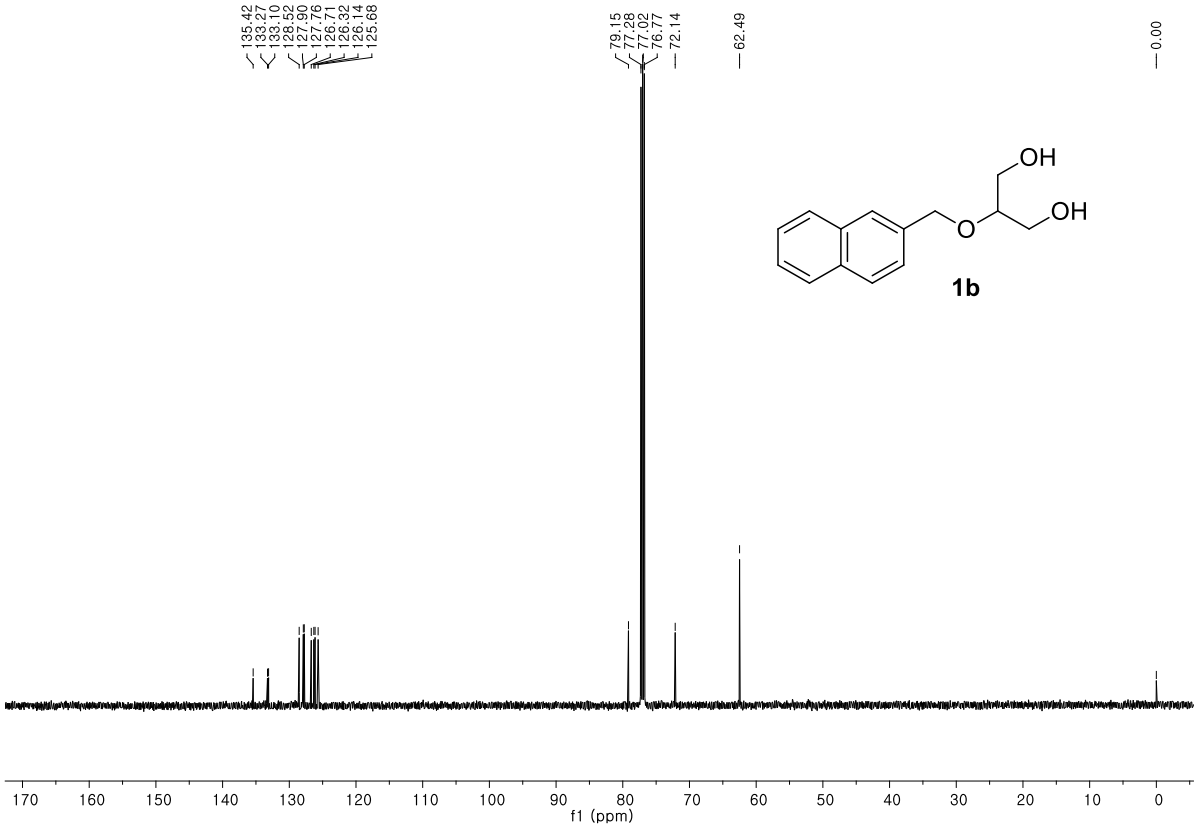
7.85
7.84
7.83
7.83
7.82
7.80
7.51
7.50
7.50
7.49
7.49
7.48
7.48
7.47
7.46
7.26
4.83
3.85
3.84
3.83
3.82
3.77
3.76
3.75
3.74
3.68
3.67
3.66
3.65
3.64
1.93
0.00
OH
OH
O
1b
4.09
3.01
2.00
4.20
1.00
2.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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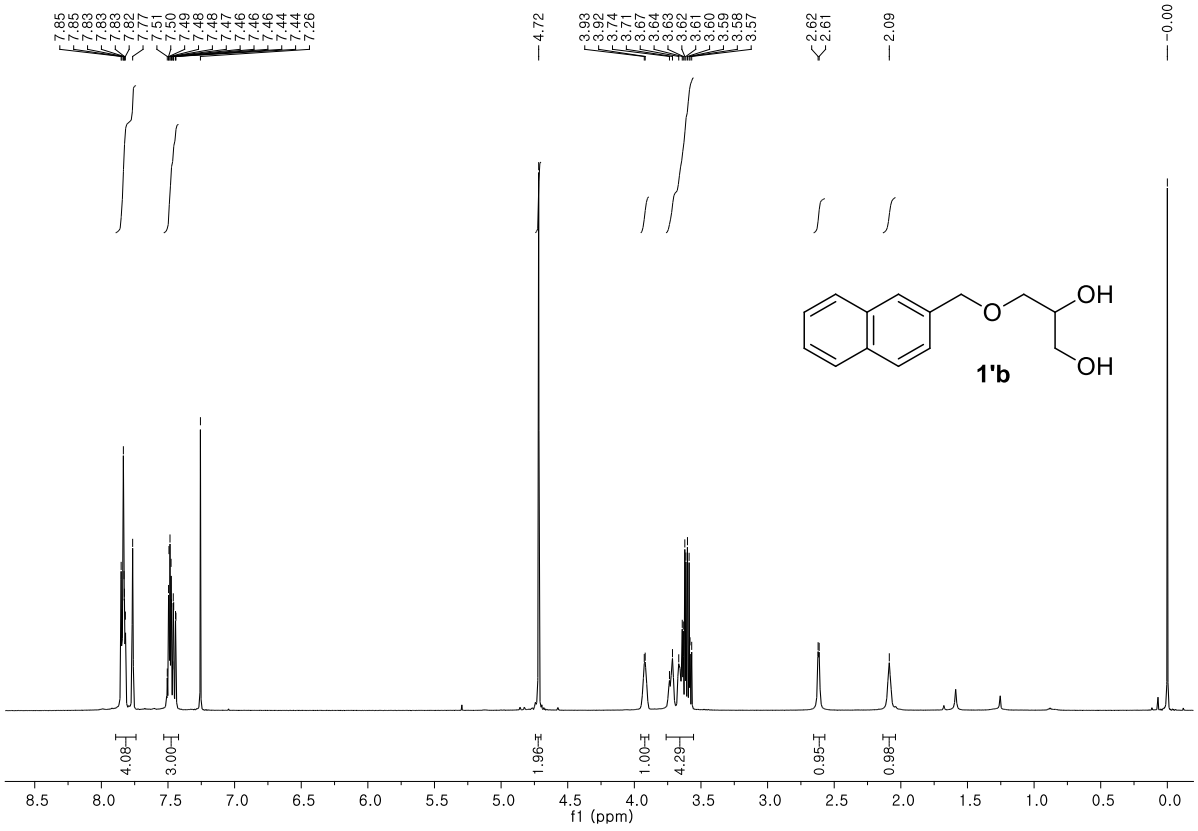
133.27
133.10
128.52
127.90
127.76
126.71
126.32
126.14
125.68
79.15
77.28
77.02
76.77
72.14
62.49
0.00
OH
OH
O
1b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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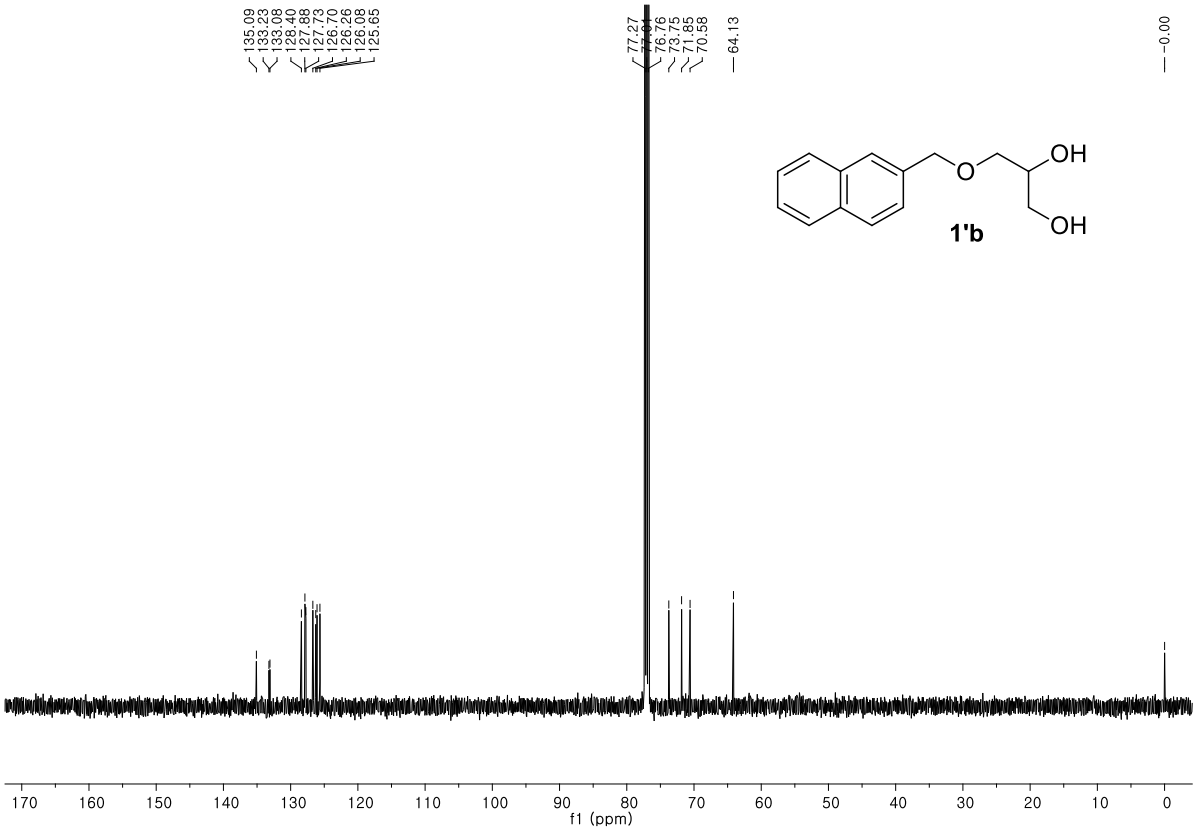
7.85
7.83
7.83
7.83
7.82
7.77
7.51
7.50
7.49
7.48
7.48
7.47
7.46
7.46
7.46
7.44
7.44
7.26
4.72
3.93
3.92
3.74
3.71
3.67
3.64
3.63
3.62
3.61
3.60
3.59
3.58
3.57
2.62
2.61
2.09
0.00
OH
O
OH
1'b
4.08
3.00
1.96
1.00
4.29
0.95
0.98
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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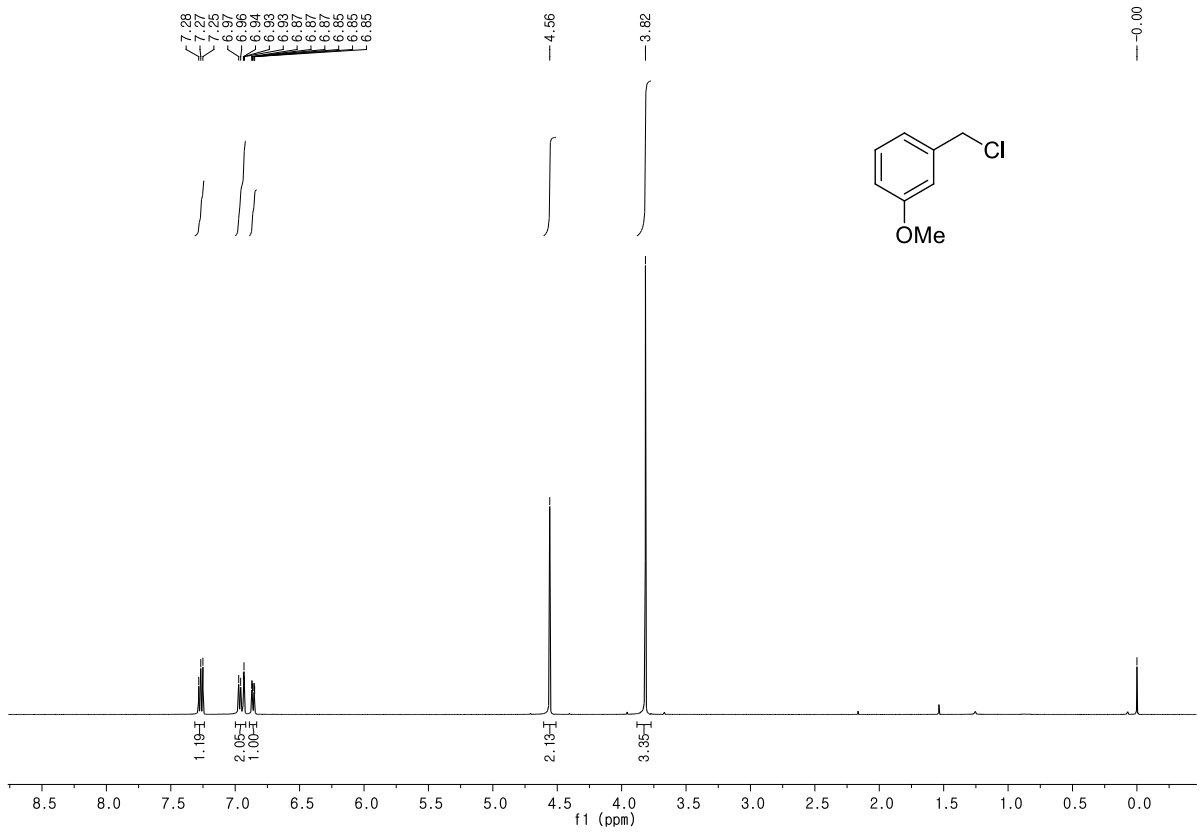
128.40
127.88
127.73
126.70
126.26
126.08
125.65
77.27
77.01
76.76
73.75
71.85
70.58
64.13
0.00
OH
O
OH
1'b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.27
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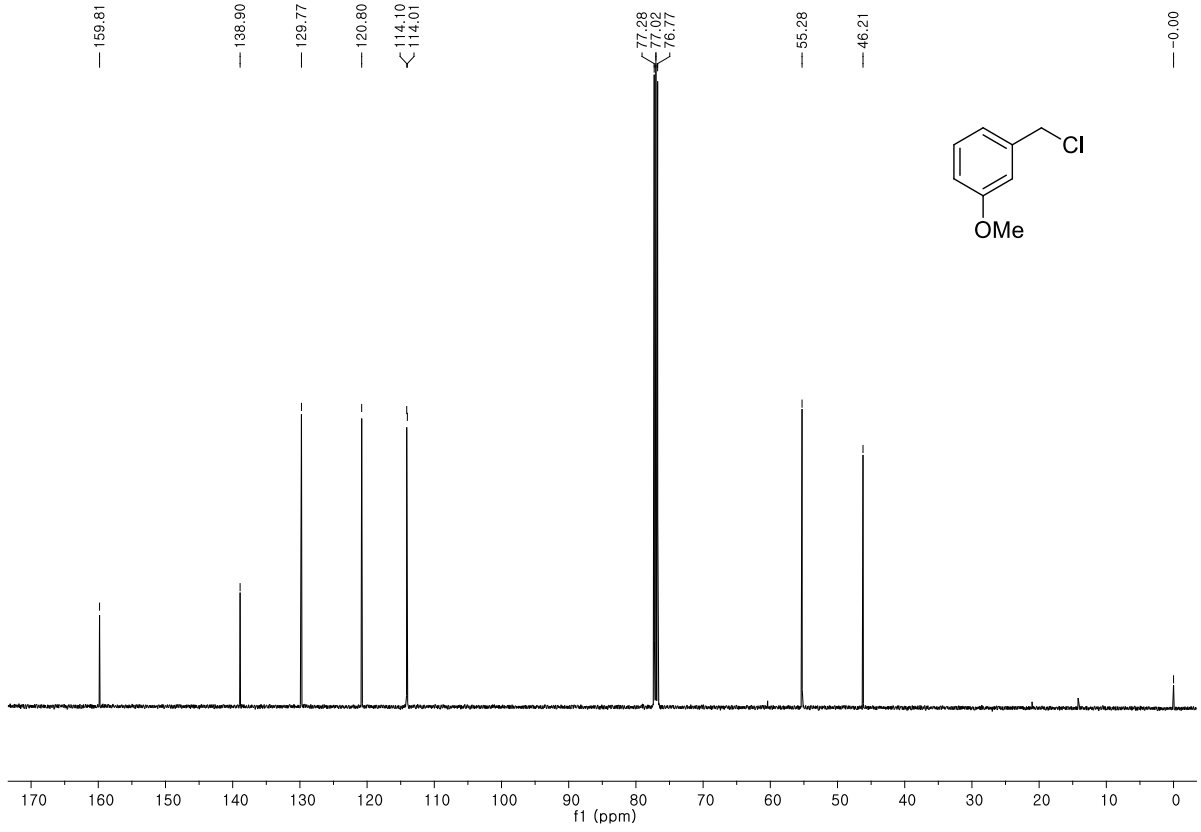
6.97
6.96
6.94
6.93
6.87
6.87
6.87
6.85
6.85
4.56
3.82
0.00
Cl
OMe
1.19
2.05
1.00
2.13
3.35
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


159.81
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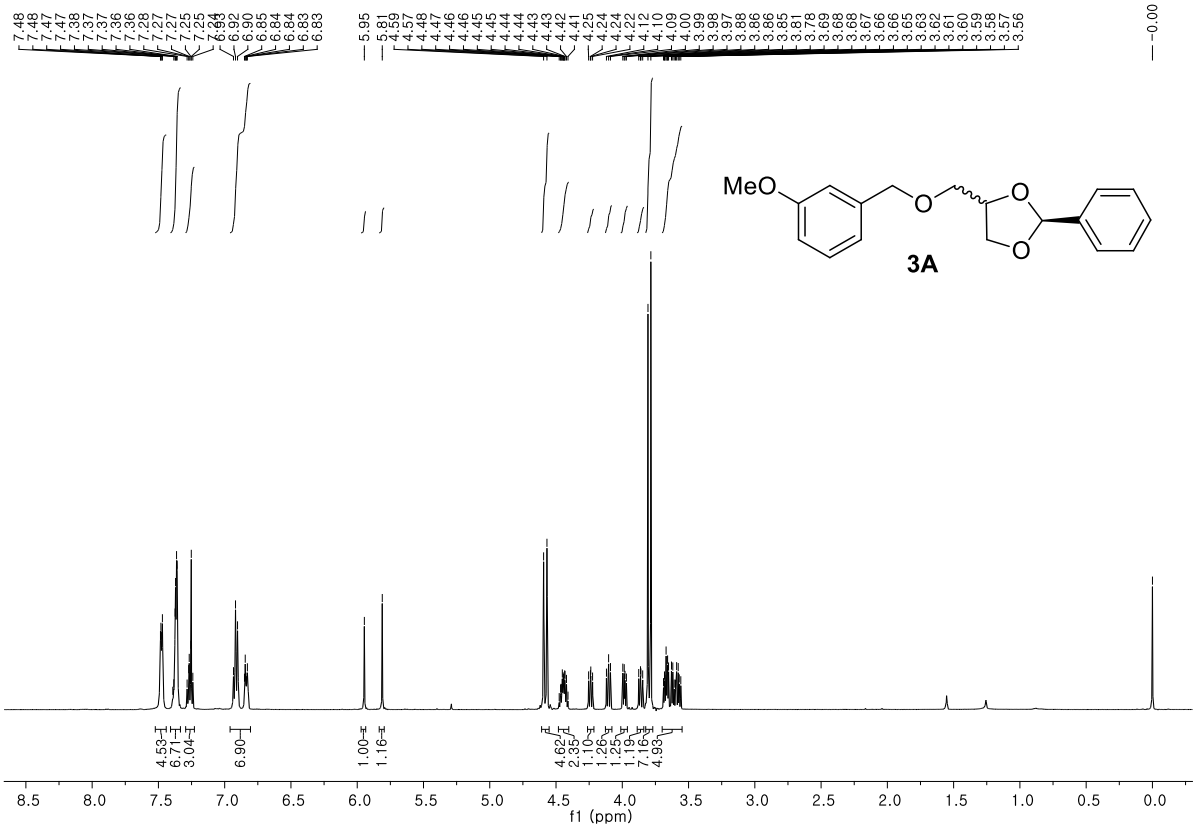
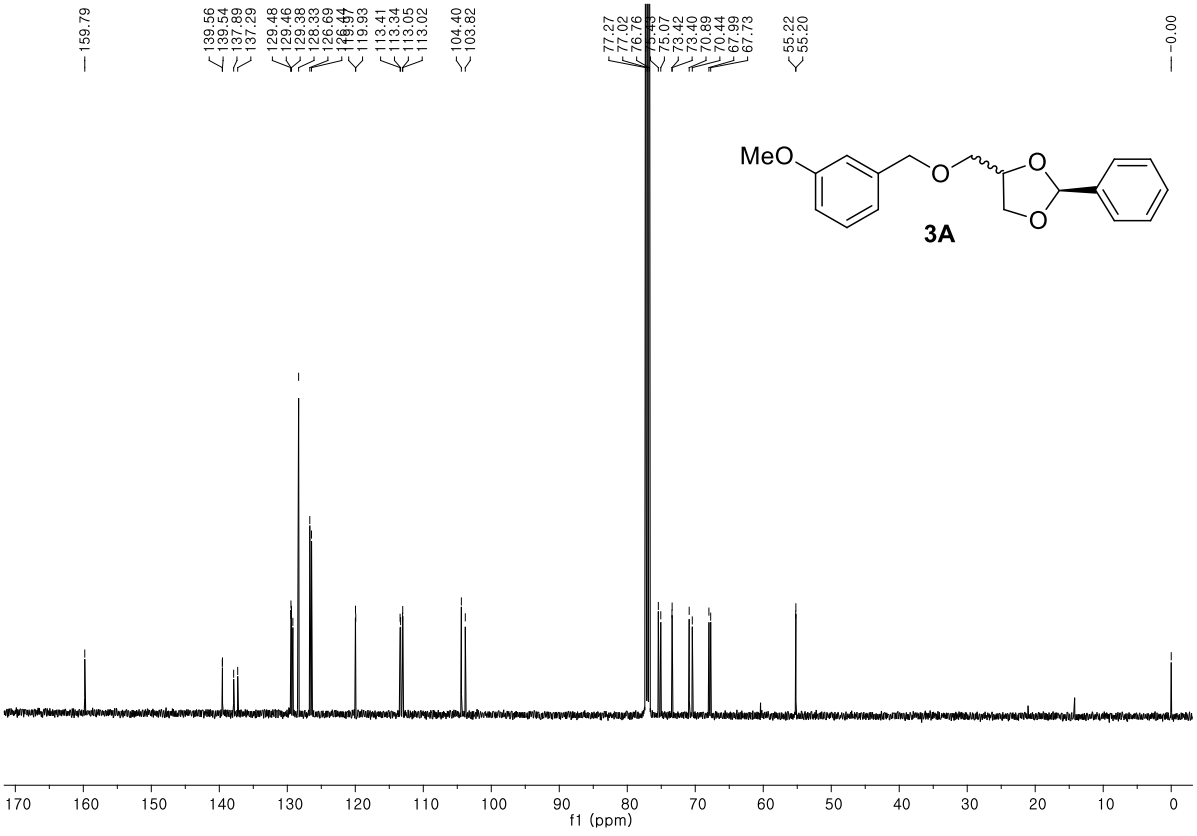
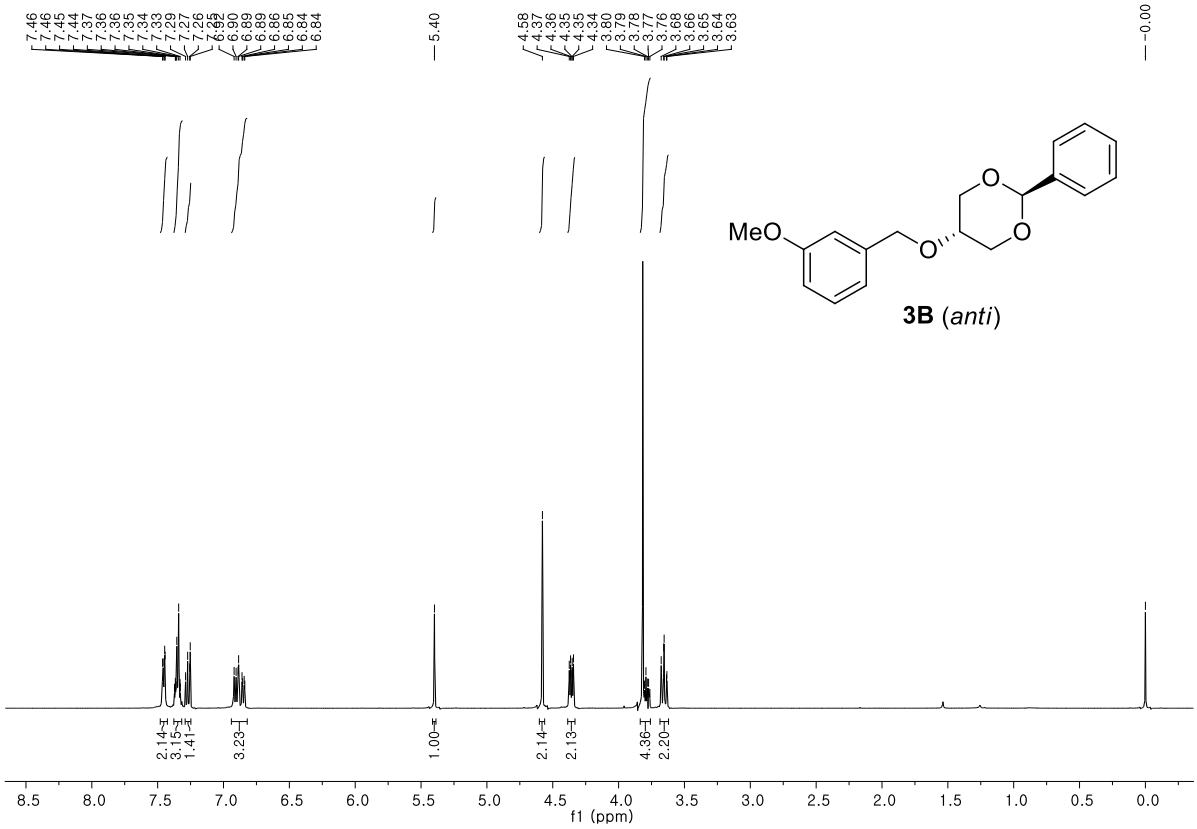
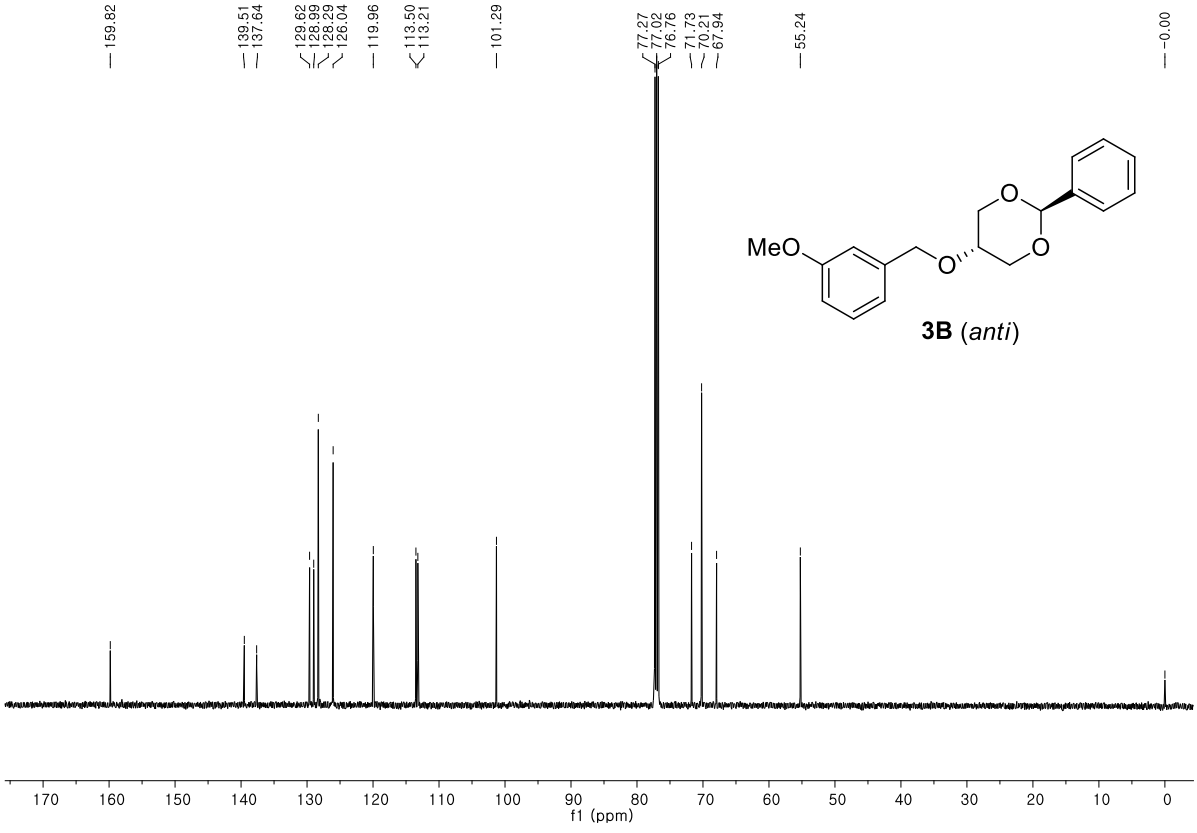
120.80
114.10
114.01
77.28
77.02
76.77
55.28
46.21
0.00
Cl
OMe
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MeO
O
O
O
3A
f1 (ppm)

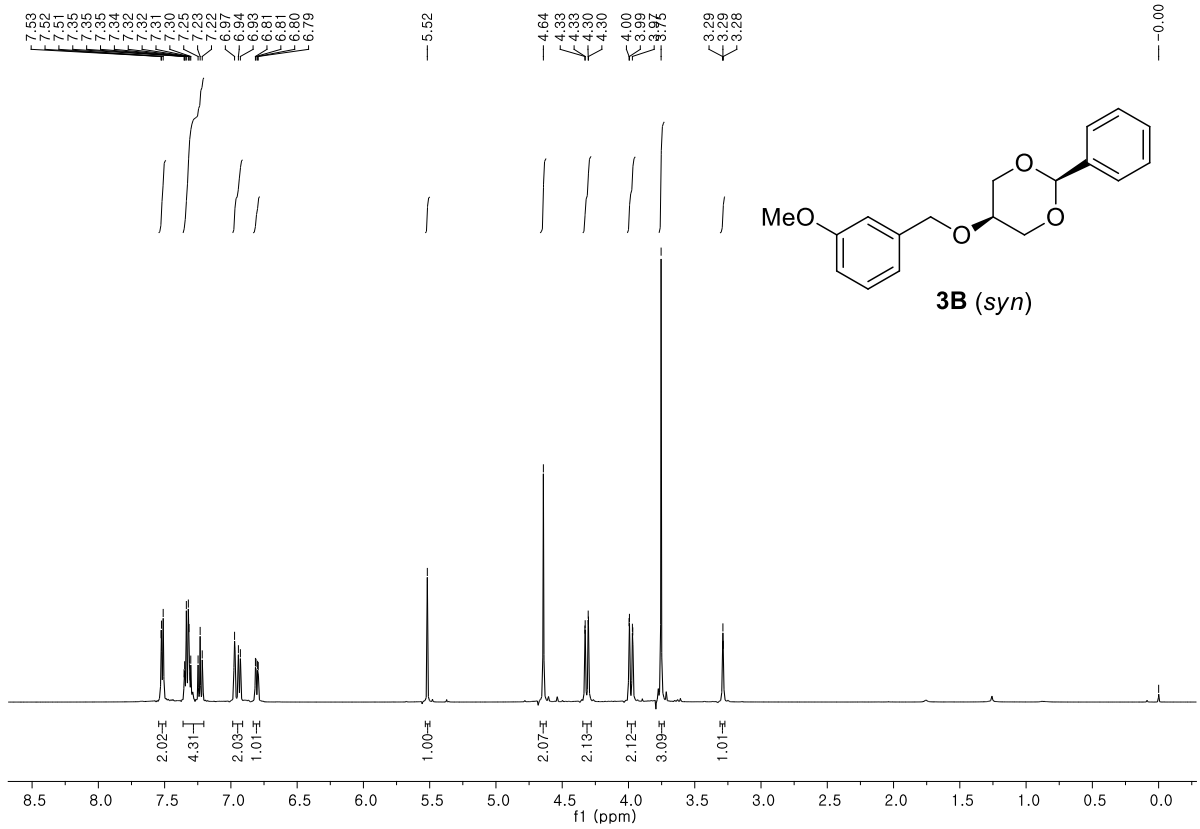

MeO
O
O
O
3A
f1 (ppm)



3B (*anti*)



3B (*anti*)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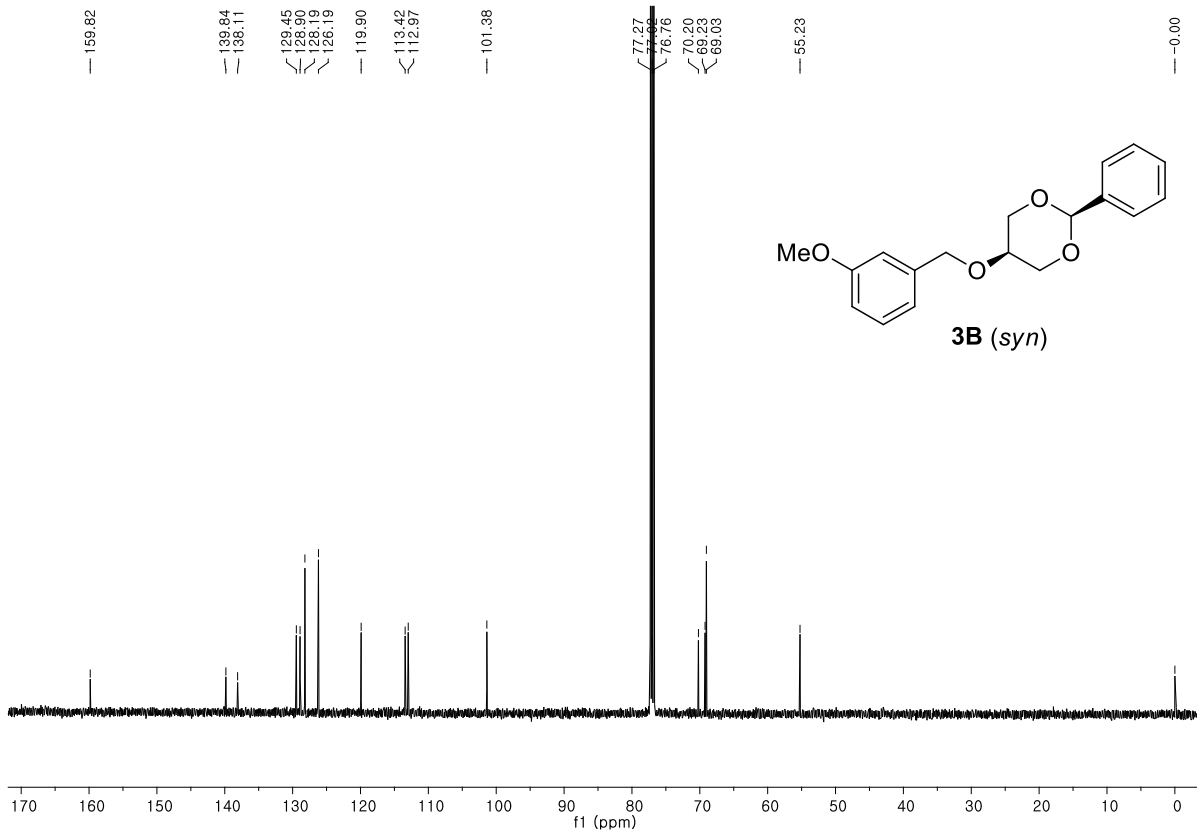
7.35
7.34
7.32
7.32
7.31
7.30
7.25
7.23
7.22
6.97
6.94
6.93
6.81
6.81
6.80
6.79
5.52
4.64
4.33
4.33
4.30
4.30
4.00
3.99
3.97
3.75
3.29
3.29
3.28
0.00
MeO
O
O
O
3B (syn)
2.02
4.31
2.03
1.01
1.00
2.07
2.13
2.12
3.09
1.01
f1 (ppm)


159.82
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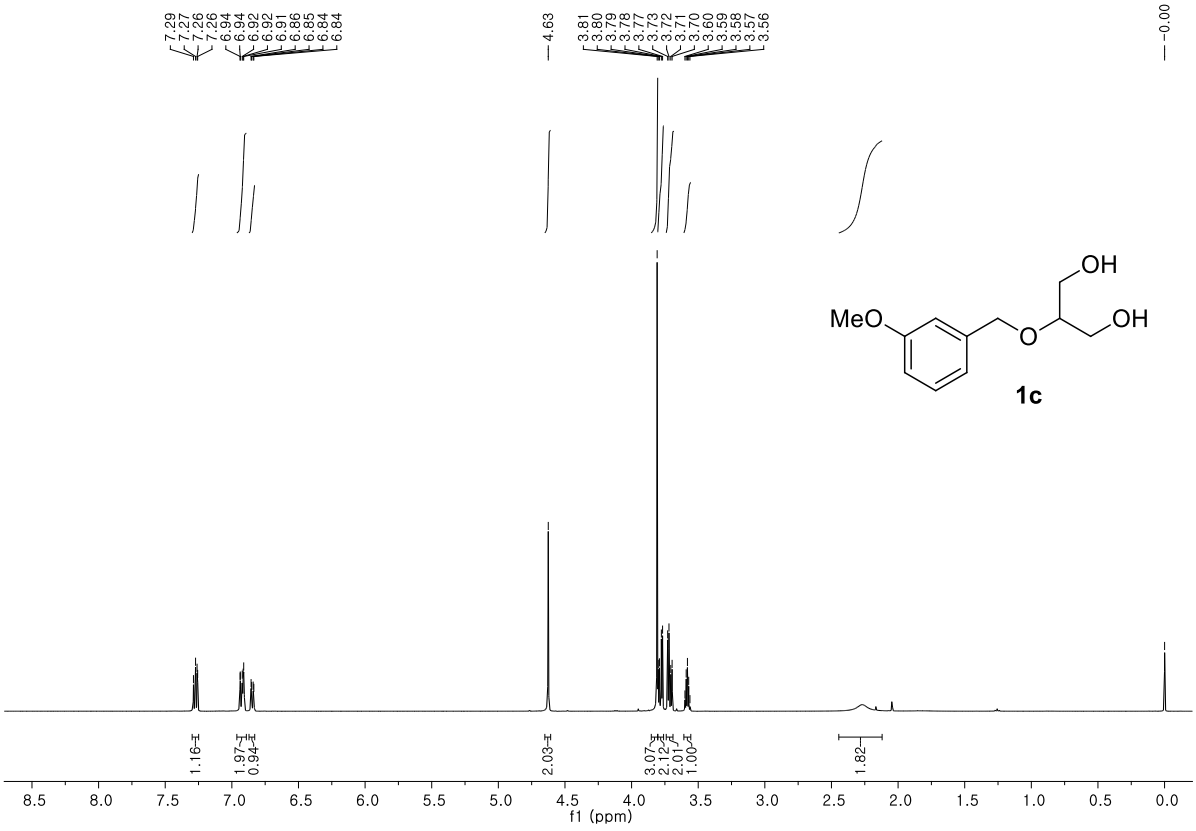
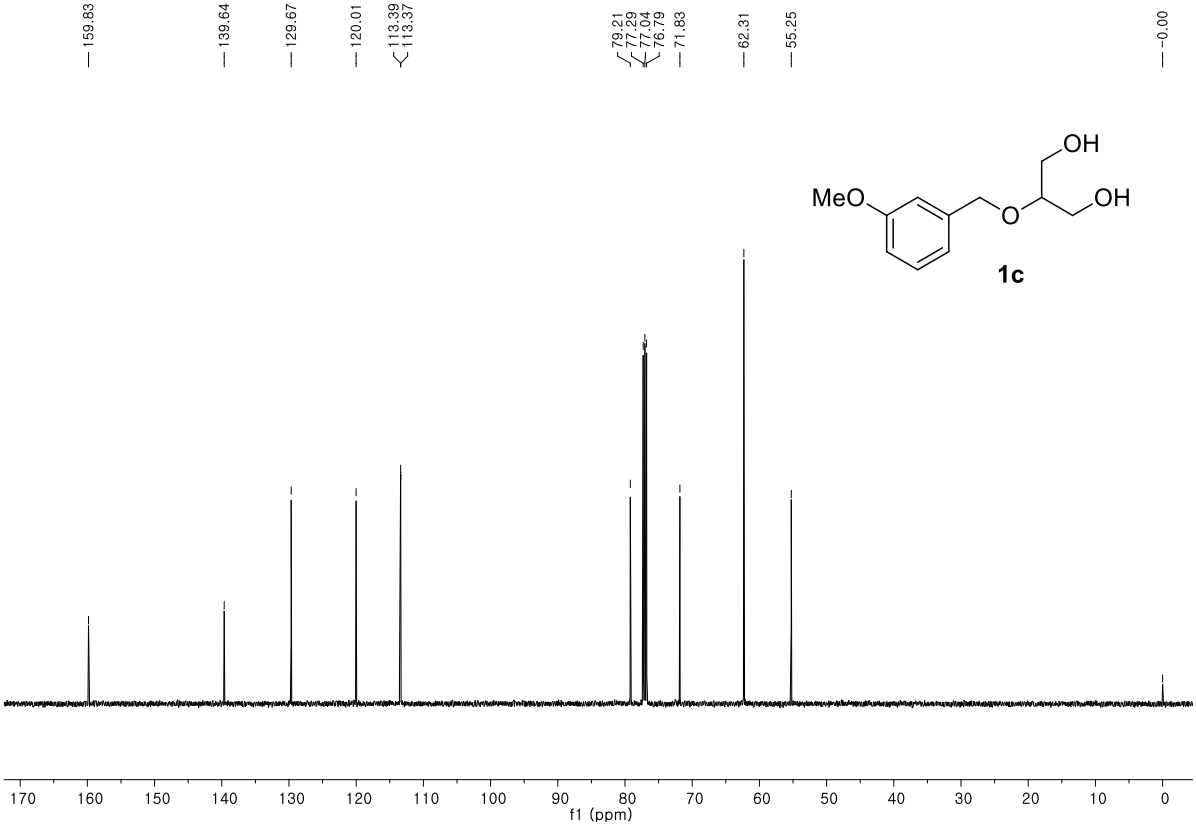
129.45
128.90
128.19
126.19
119.90
113.42
112.97
101.38
77.27
77.02
76.76
70.20
69.23
69.03
55.23
0.00
MeO
O
O
O
3B (syn)
f1 (ppm)


7.29
7.27
7.26
7.26
6.94
6.94
6.92
6.92
6.91
6.86
6.85
6.84
6.84
4.63
3.81
3.80
3.79
3.78
3.77
3.73
3.72
3.71
3.70
3.60
3.59
3.58
3.57
3.56
0.00
OH
MeO
O
OH
1c
1.16
1.97
0.94
2.03
3.07
2.12
2.01
1.00
1.82
f1 (ppm)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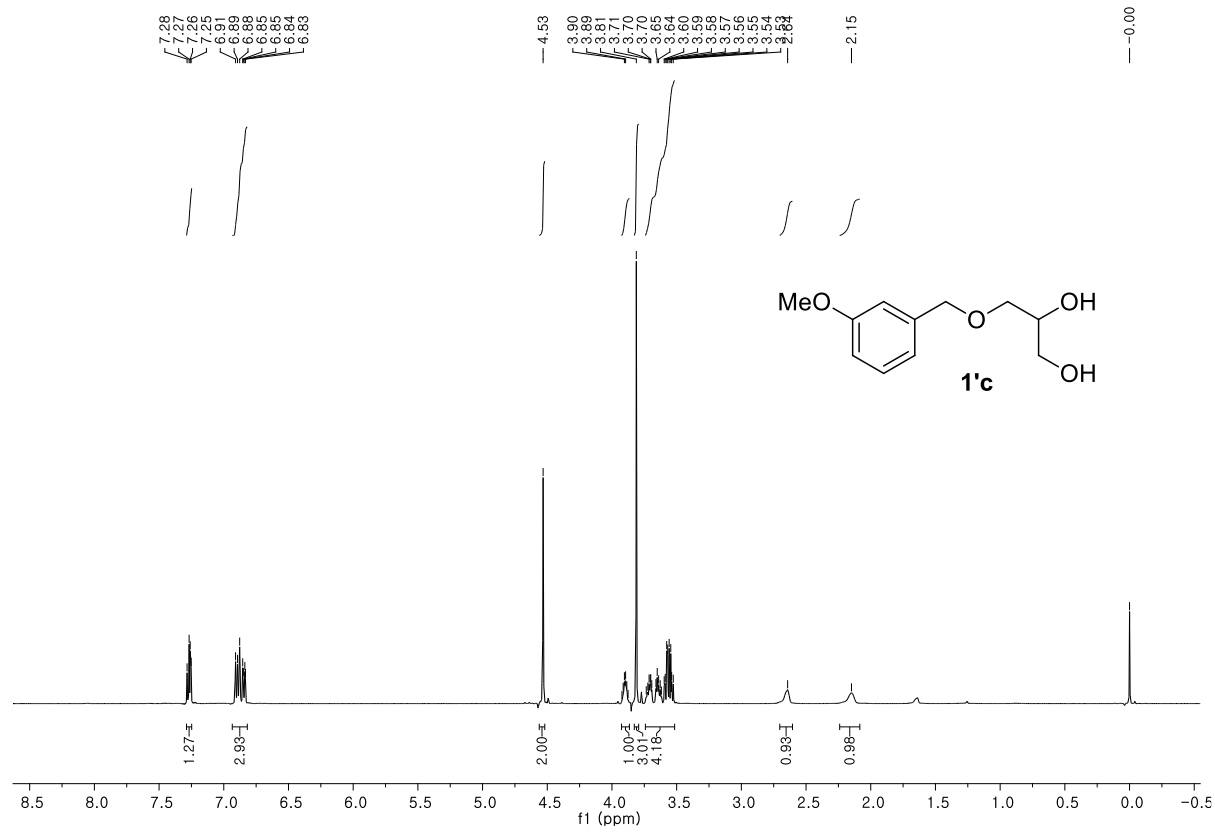
129.67
120.01
113.39
113.37
79.21
77.29
77.04
76.79
71.83
62.31
55.25
0.00
OH
MeO
O
OH
1c
f1 (ppm)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7.26
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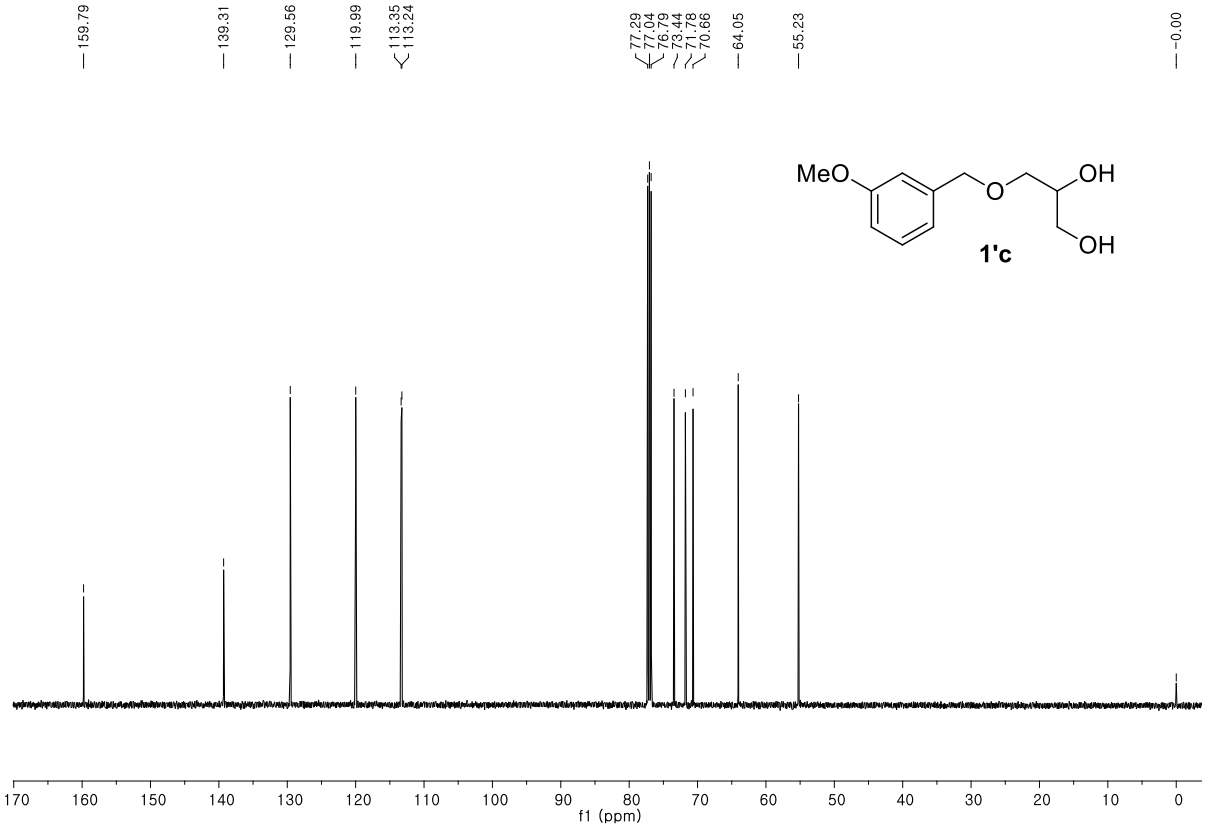
6.89
6.88
6.85
6.85
6.84
6.83
4.53
3.90
3.89
3.81
3.71
3.70
3.70
3.65
3.64
3.60
3.59
3.58
3.57
3.56
3.55
3.54
2.84
2.15
0.00
MeO
O
OH
OH
1'c
1.27
2.93
2.00
1.00
3.01
4.18
0.93
0.98
f1 (ppm)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113.24
77.29
77.04
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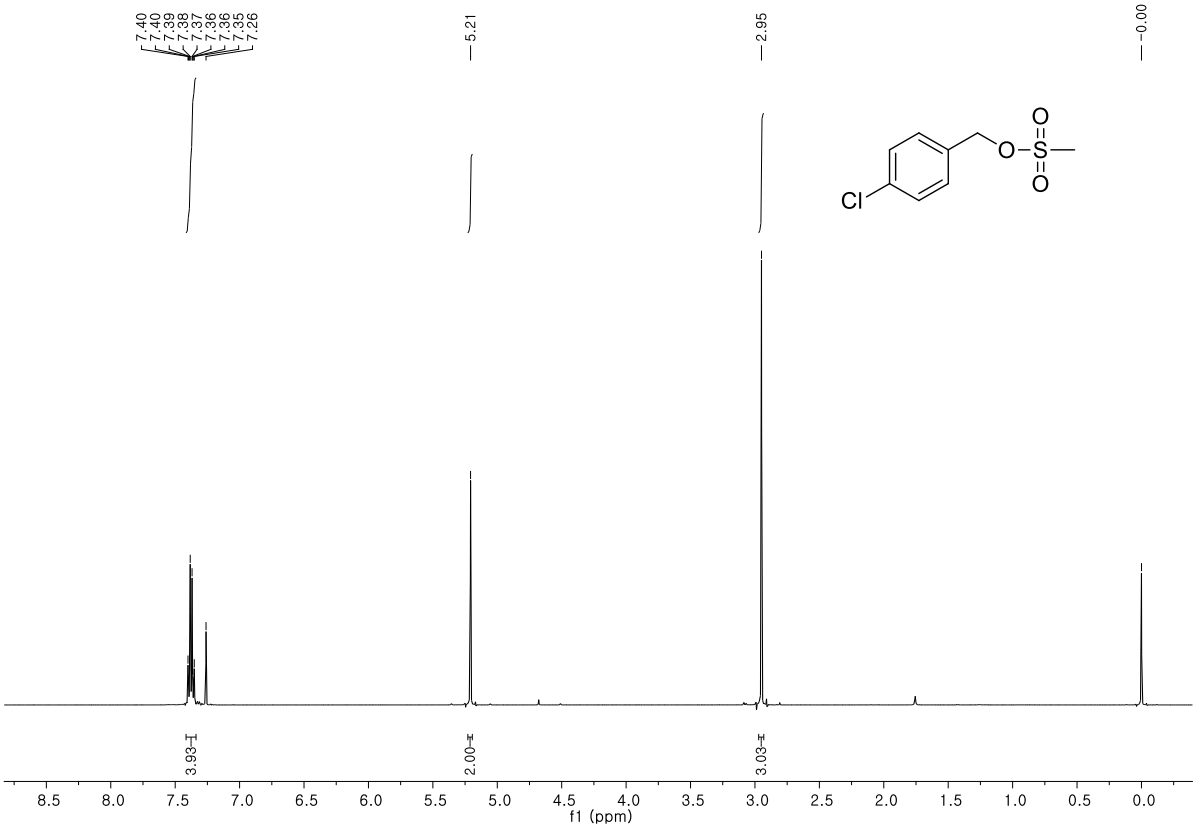
71.78
70.66
64.05
55.23
0.00
MeO
O
OH
OH
1'c
f1 (ppm)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7.36
7.35
7.26
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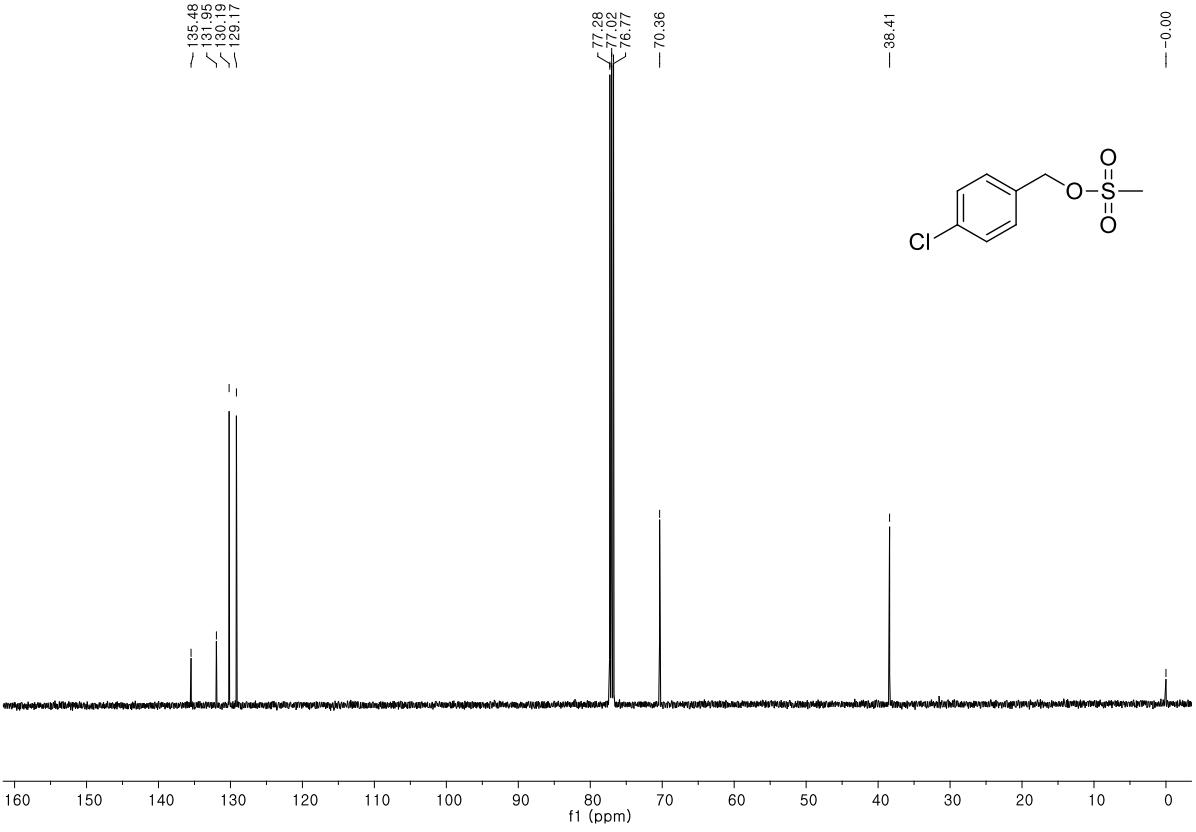
2.95
0.00
Cl
O
S
O
O
3.93
2.00
3.03
f1 (ppm)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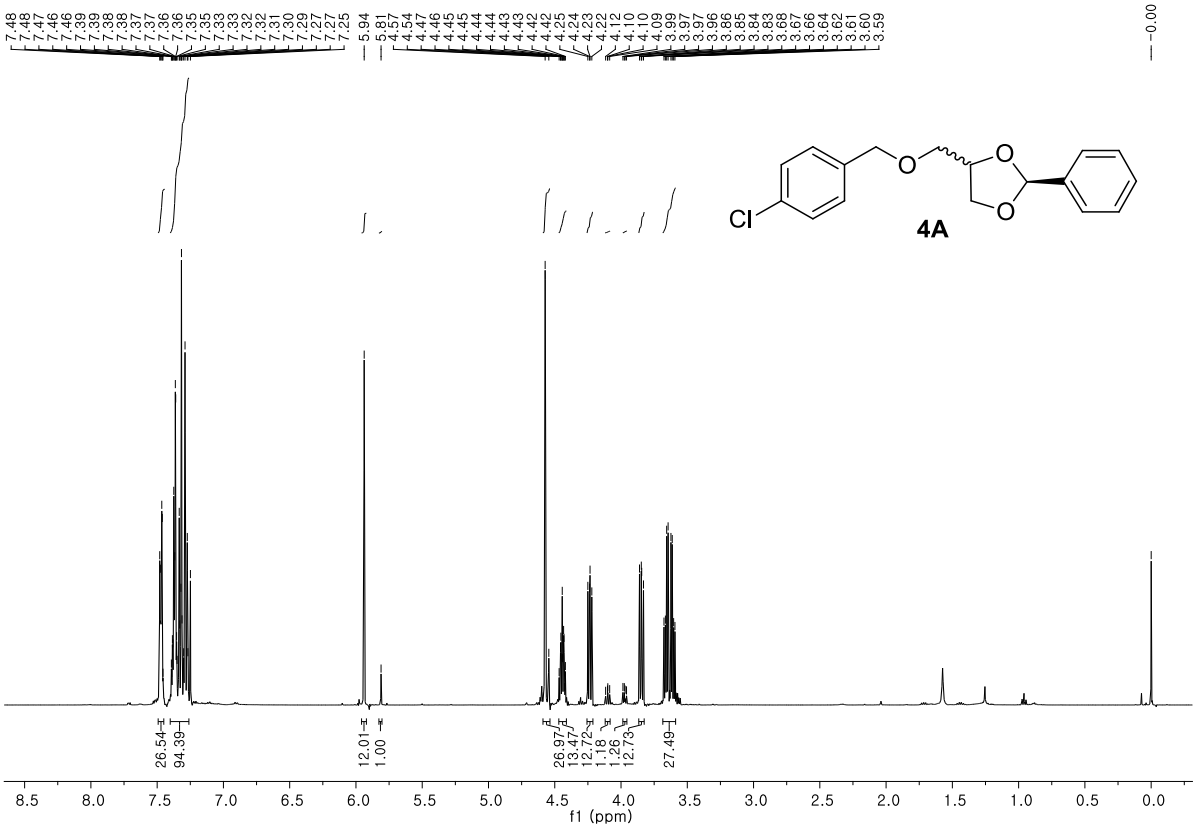
76.77
70.36
38.41
0.00
Cl
O
S
O
O
f1 (ppm)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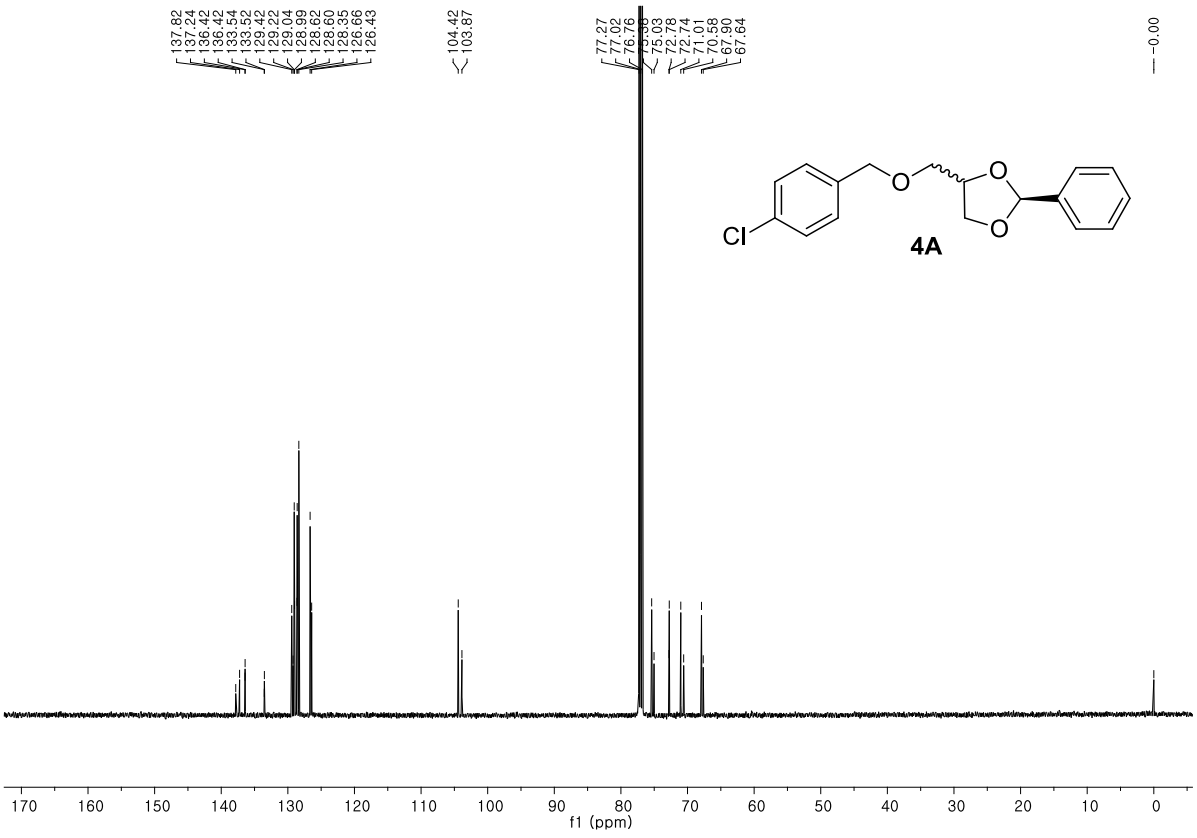
7.35
7.35
7.33
7.33
7.32
7.32
7.31
7.30
7.29
7.27
7.27
7.25
5.94
5.81
4.57
4.54
4.47
4.46
4.45
4.45
4.44
4.44
4.43
4.43
4.42
4.42
4.25
4.24
4.23
4.22
4.12
4.10
4.10
4.09
3.99
3.97
3.97
3.96
3.86
3.85
3.84
3.68
3.67
3.66
3.64
3.62
3.61
3.60
3.59
0.00
Cl
O
O
O
4A
26.54
94.39
12.01
1.00
26.97
13.47
12.72
1.18
1.26
12.73
27.49
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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129.22
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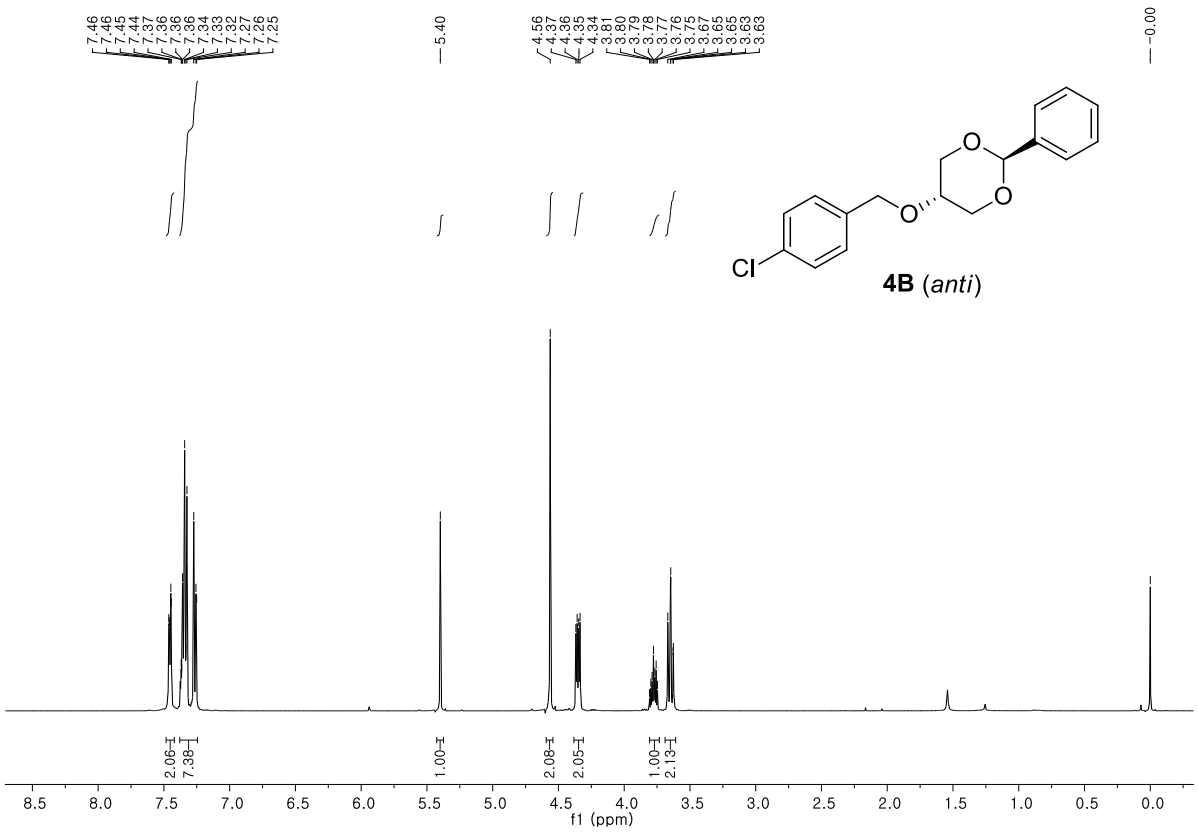
128.62
128.60
128.35
126.66
126.43
104.42
103.87
77.27
77.02
76.76
75.36
75.03
72.78
72.74
71.01
70.58
67.90
67.64
0.00
Cl
O
O
O
4A
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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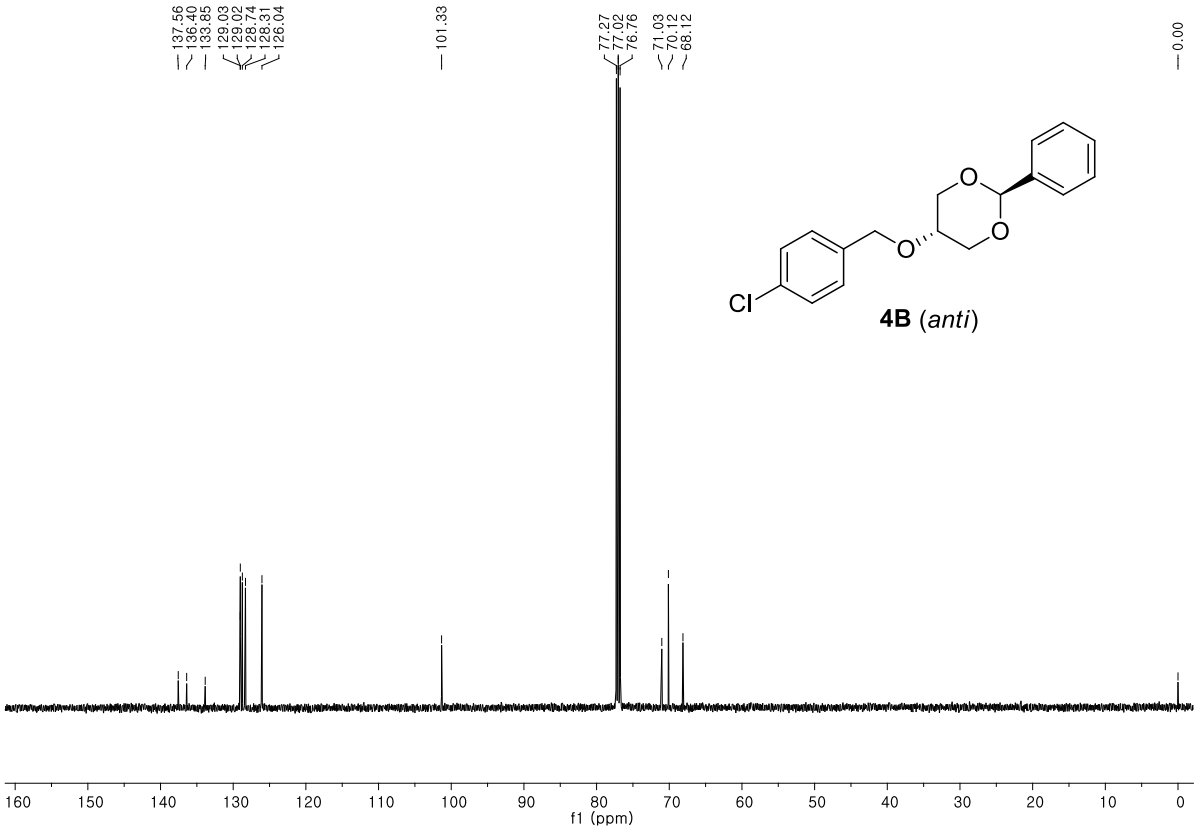
7.36
7.36
7.36
7.34
7.33
7.32
7.27
7.26
7.25
5.40
4.56
4.37
4.36
4.35
4.34
3.81
3.80
3.79
3.78
3.77
3.76
3.75
3.67
3.65
3.65
3.63
3.63
0.00
Cl
4B (anti)
2.06
7.38
1.00
2.08
2.05
1.00
2.13
f1 (ppm)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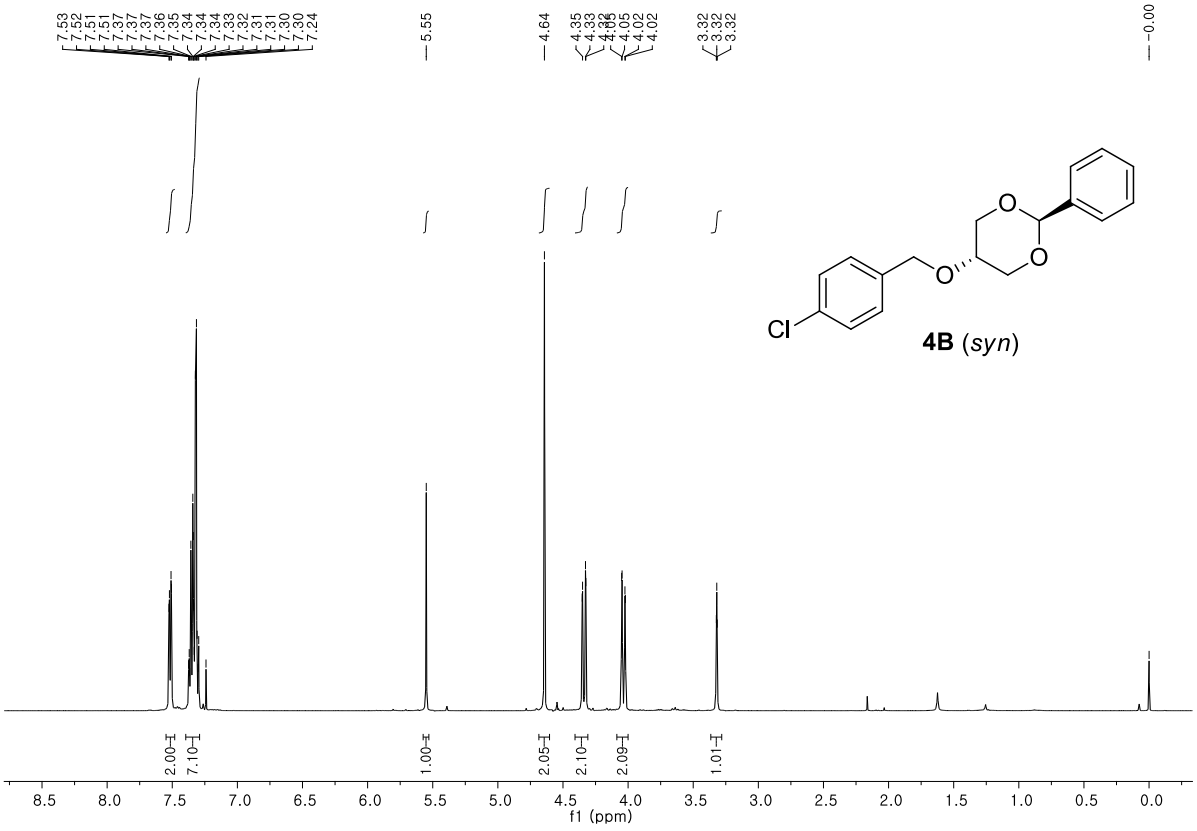
136.40
133.85
129.03
129.02
128.74
128.31
126.04
101.33
77.27
77.02
76.76
71.03
70.12
68.12
0.00
Cl
4B (anti)
f1 (ppm)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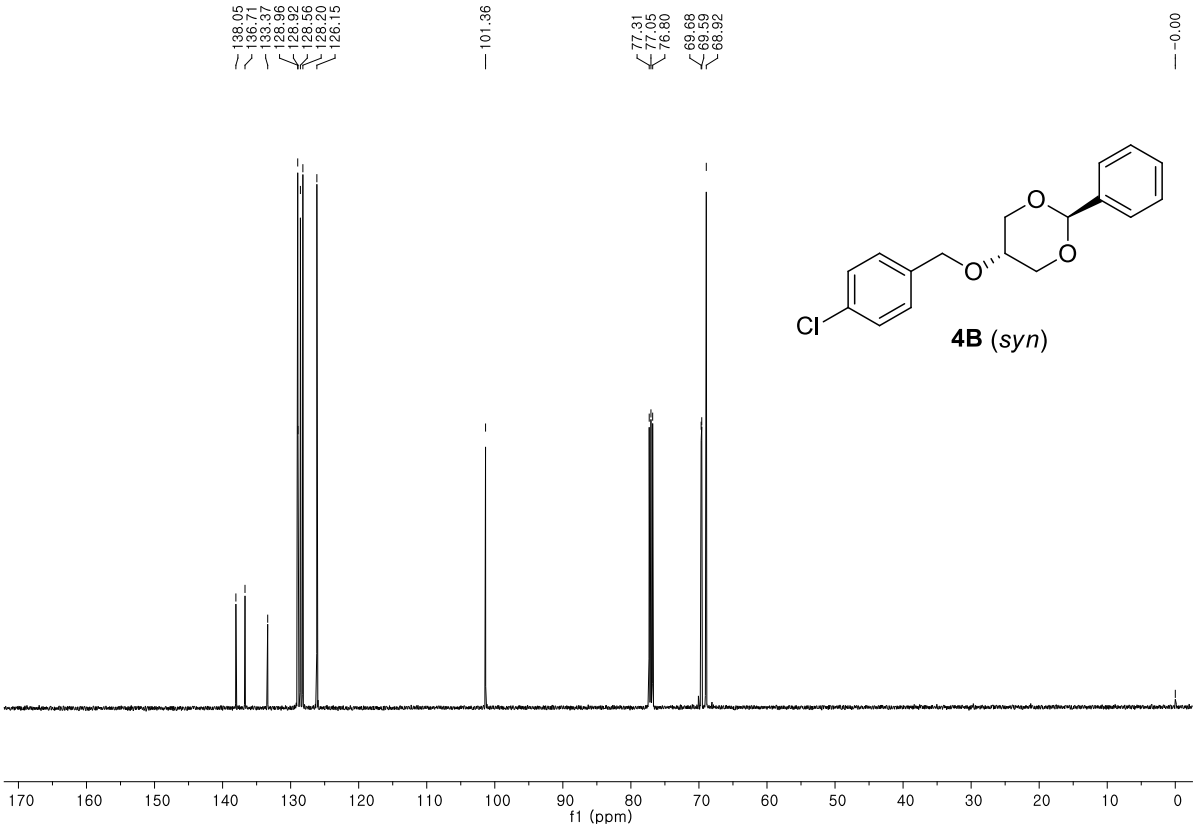
7.36
7.35
7.34
7.34
7.34
7.33
7.32
7.31
7.31
7.30
7.30
7.24
5.55
4.64
4.35
4.33
4.05
4.02
4.02
3.32
3.32
3.32
0.00
2.00
7.10
1.00
2.05
2.10
2.09
1.01
f1 (ppm)
Cl
4B (syn)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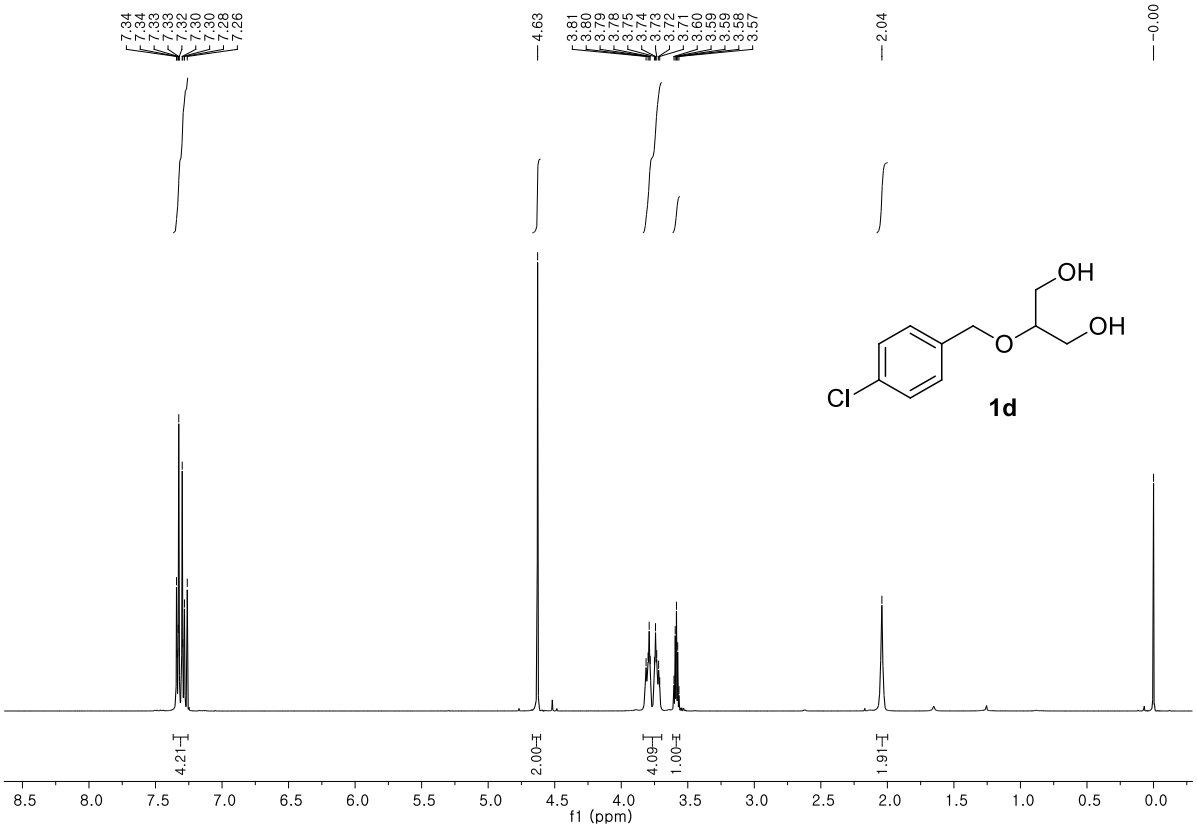
136.71
133.37
128.96
128.92
128.56
128.20
126.15
101.36
77.31
77.05
76.80
69.68
69.59
68.92
0.00
f1 (ppm)
Cl
4B (syn)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7.34
7.33
7.33
7.32
7.30
7.30
7.28
7.26
4.63
3.81
3.80
3.79
3.78
3.75
3.74
3.73
3.72
3.71
3.60
3.59
3.59
3.58
3.57
2.04
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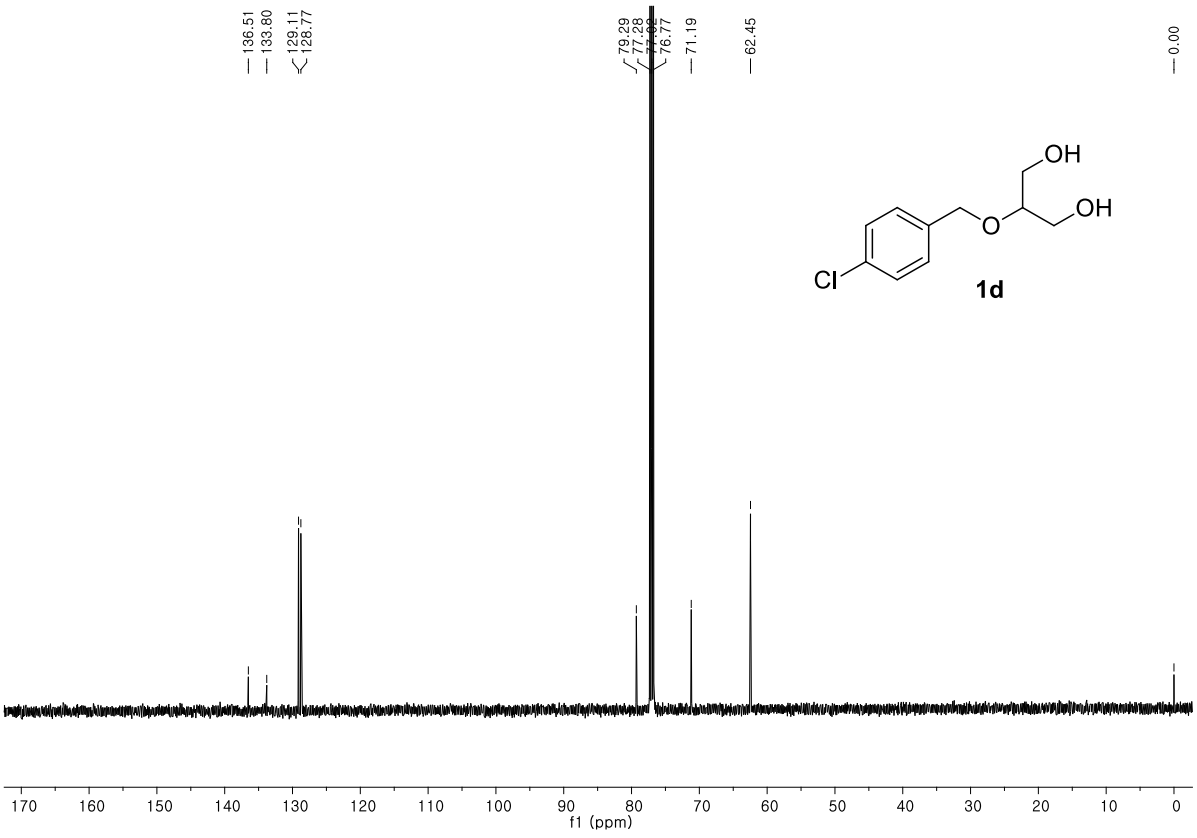
OH
OH
O
Cl
1d
4.21
2.00
4.09
1.00
1.91
f1 (ppm)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133.80
129.11
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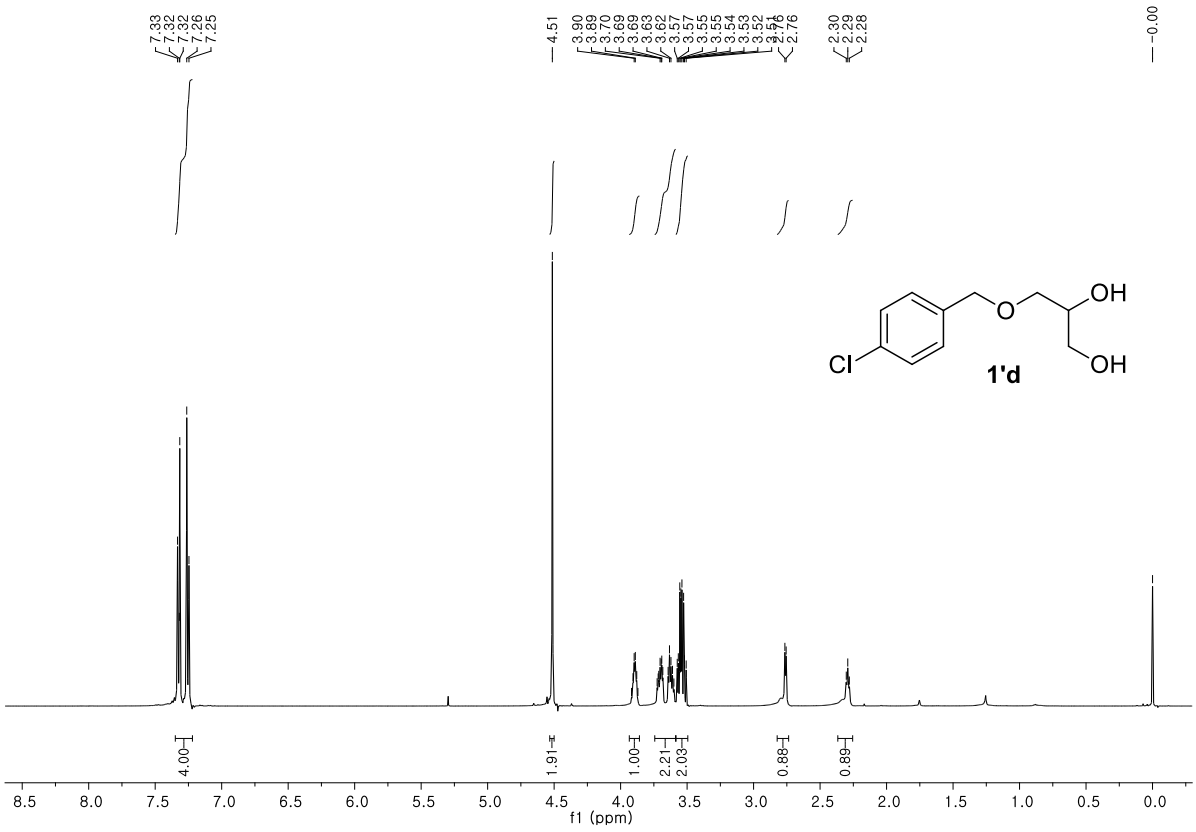
77.03
76.77
71.19
62.45
0.00
OH
OH
O
Cl
1d
f1 (ppm)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7.32
7.26
7.25
4.51
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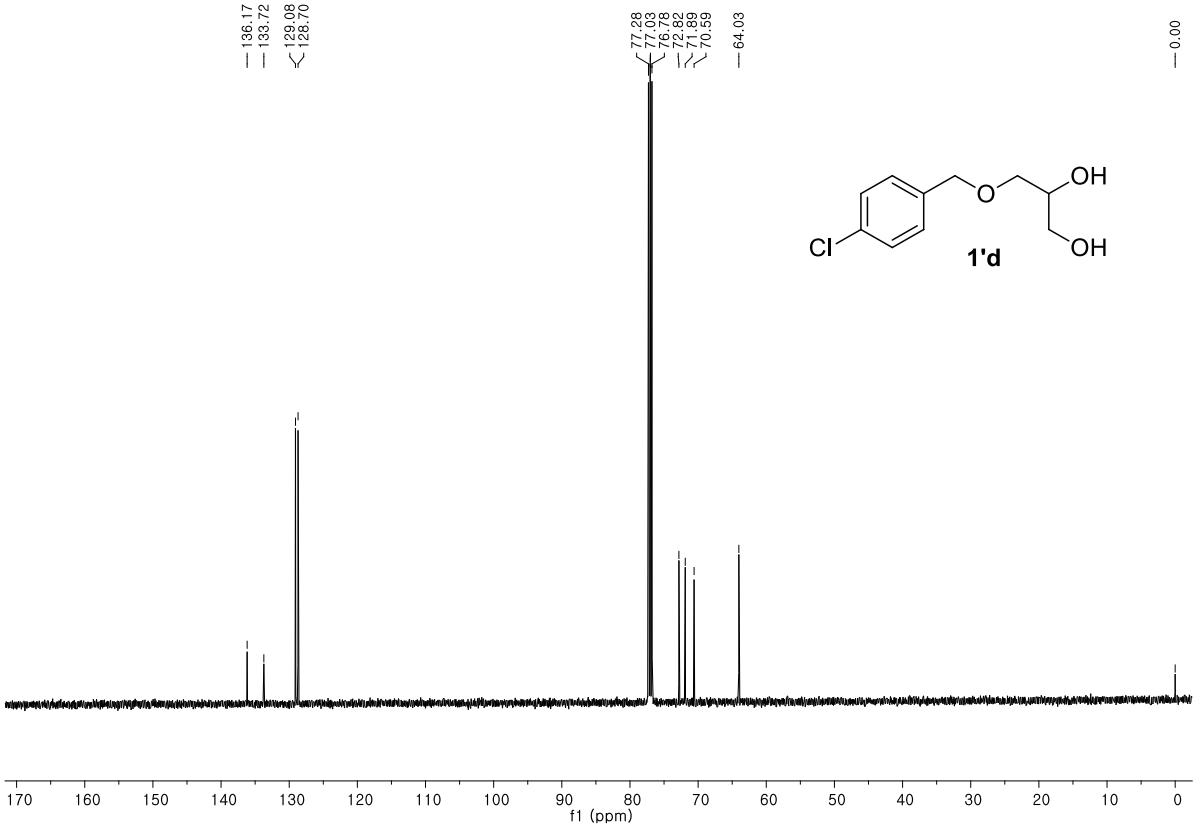
3.89
3.70
3.69
3.69
3.63
3.62
3.57
3.57
3.55
3.55
3.54
3.53
3.52
2.76
2.30
2.29
2.28
0.00
OH
O
OH
Cl
1'd
4.00
1.91
1.00
2.21
2.03
0.88
0.89
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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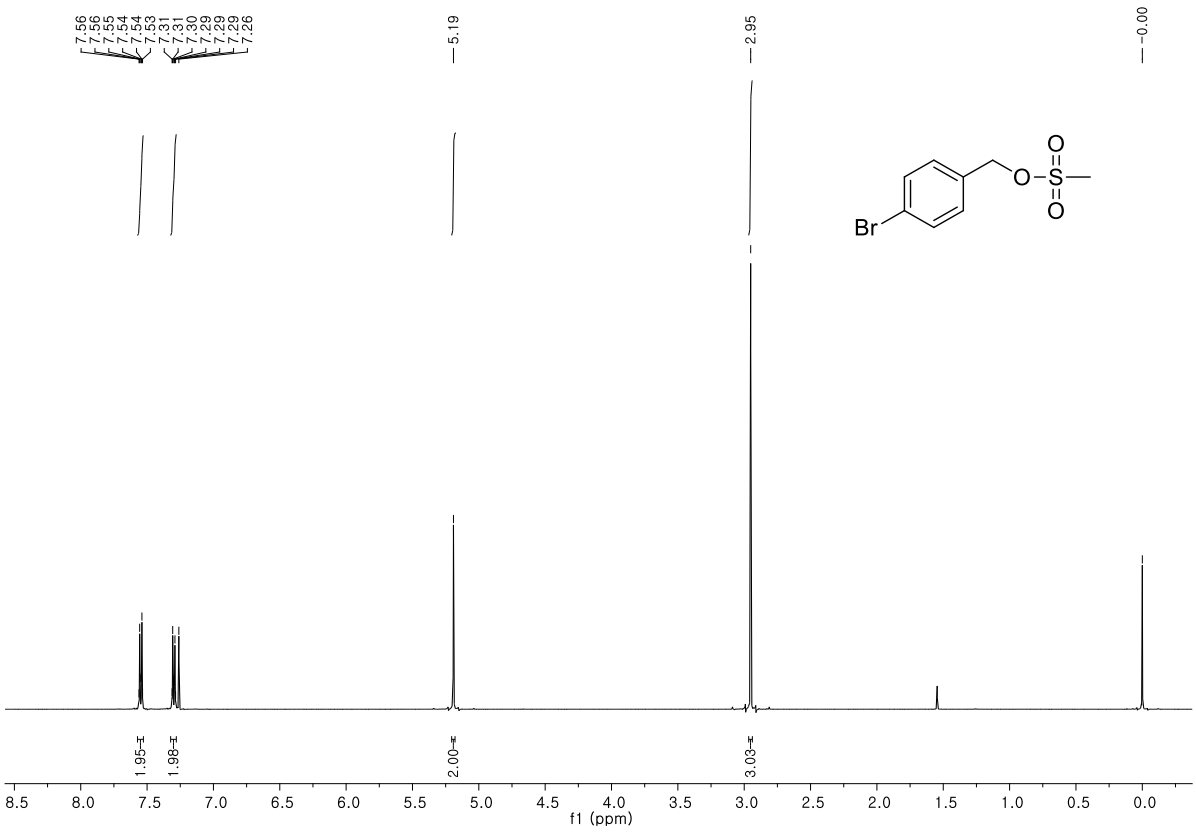
129.08
128.70
77.28
77.03
76.78
72.82
71.89
70.59
64.03
0.00
OH
O
OH
Cl
1'd
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.55
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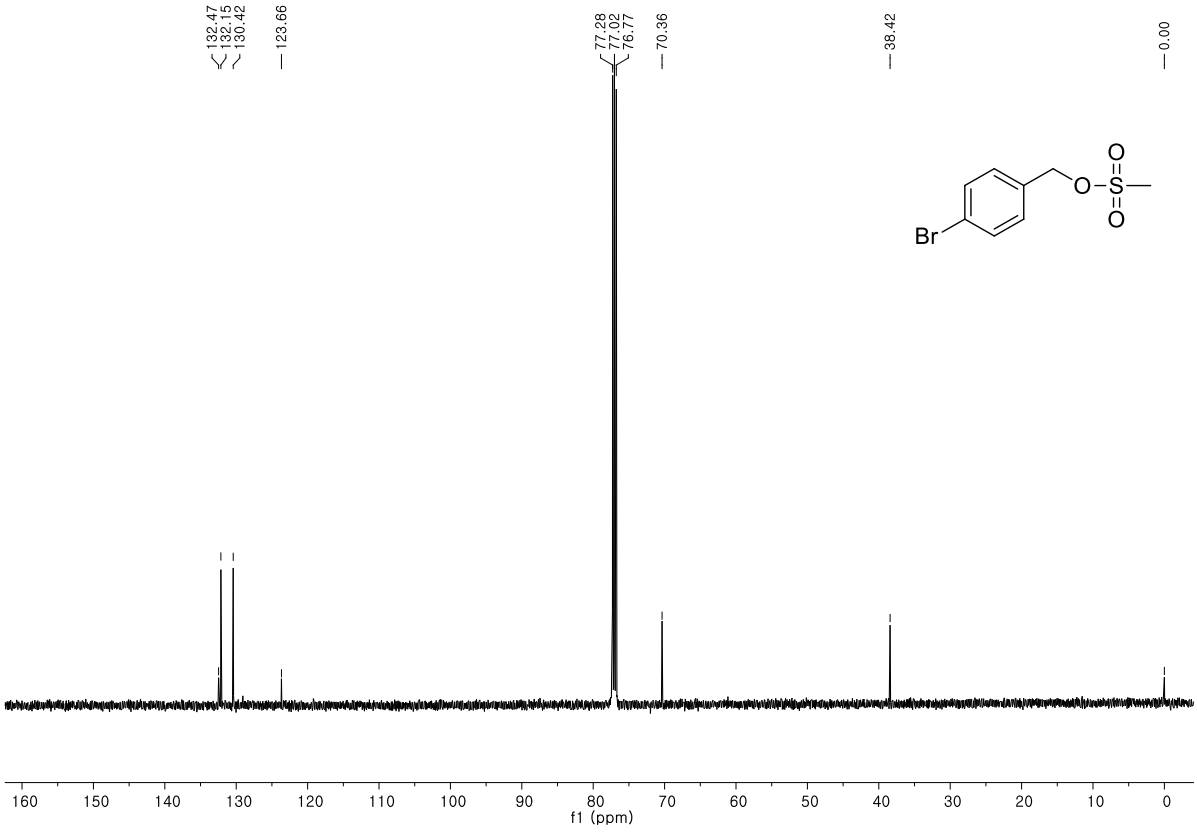
7.54
7.53
7.31
7.31
7.30
7.29
7.29
7.29
7.26
5.19
2.95
0.00
Br
O
S
O
O
1.95
1.98
2.00
3.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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132.15
130.42
123.66
77.28
77.02
76.77
70.36
38.42
0.00
Br
O
S
O
O
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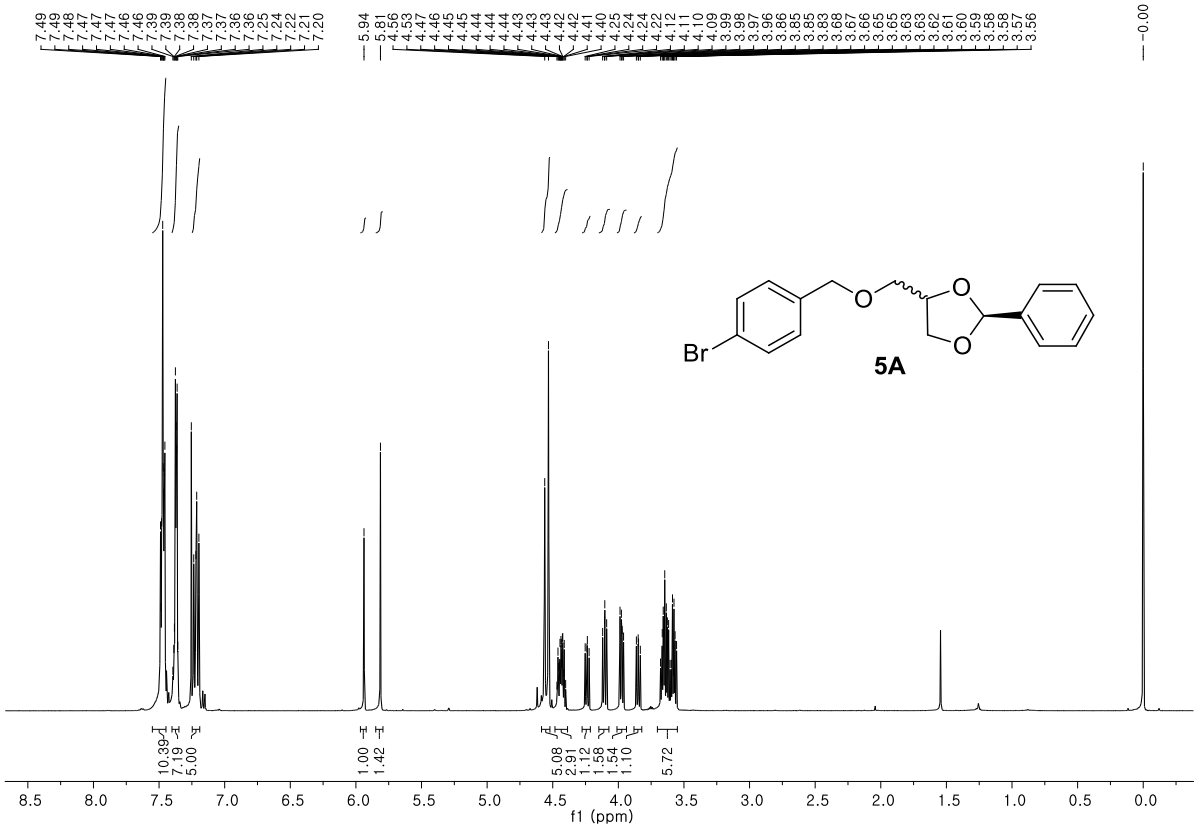
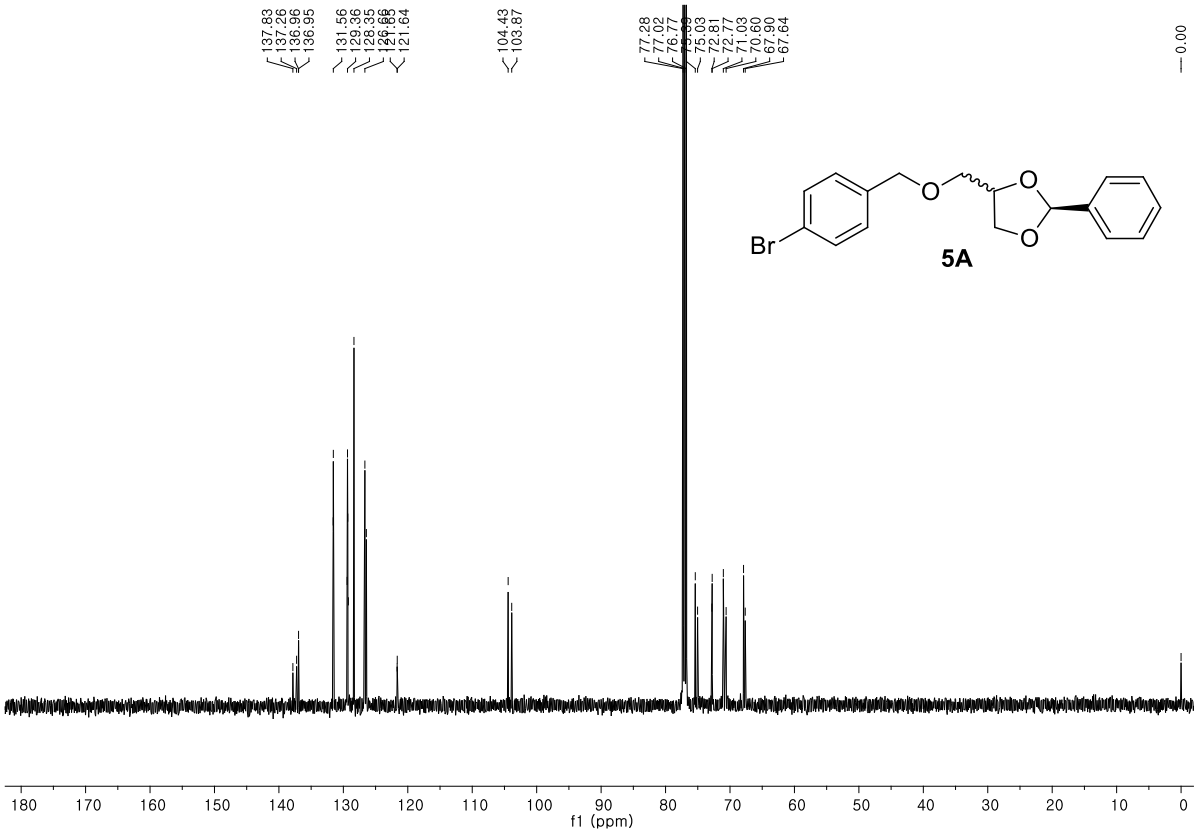
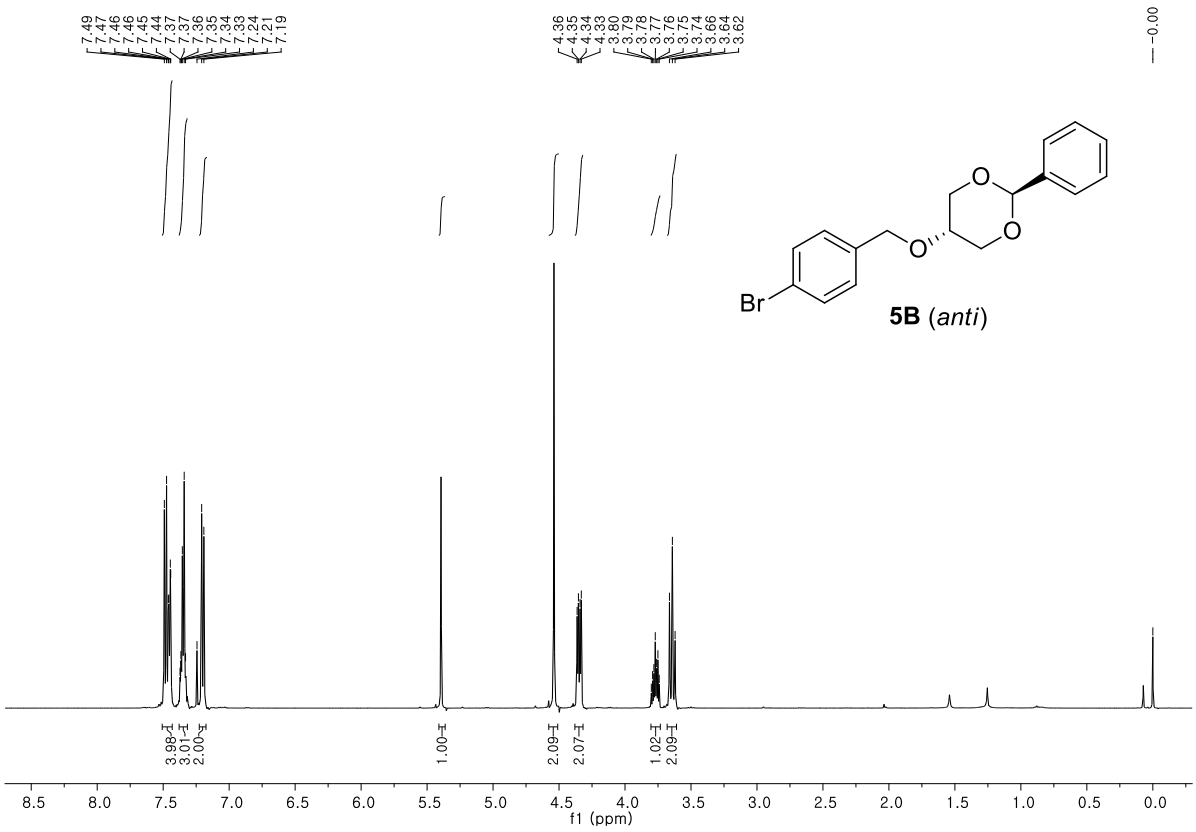
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Br
O
O
O
5A
f1 (ppm)


Br
O
O
O
5A
f1 (ppm)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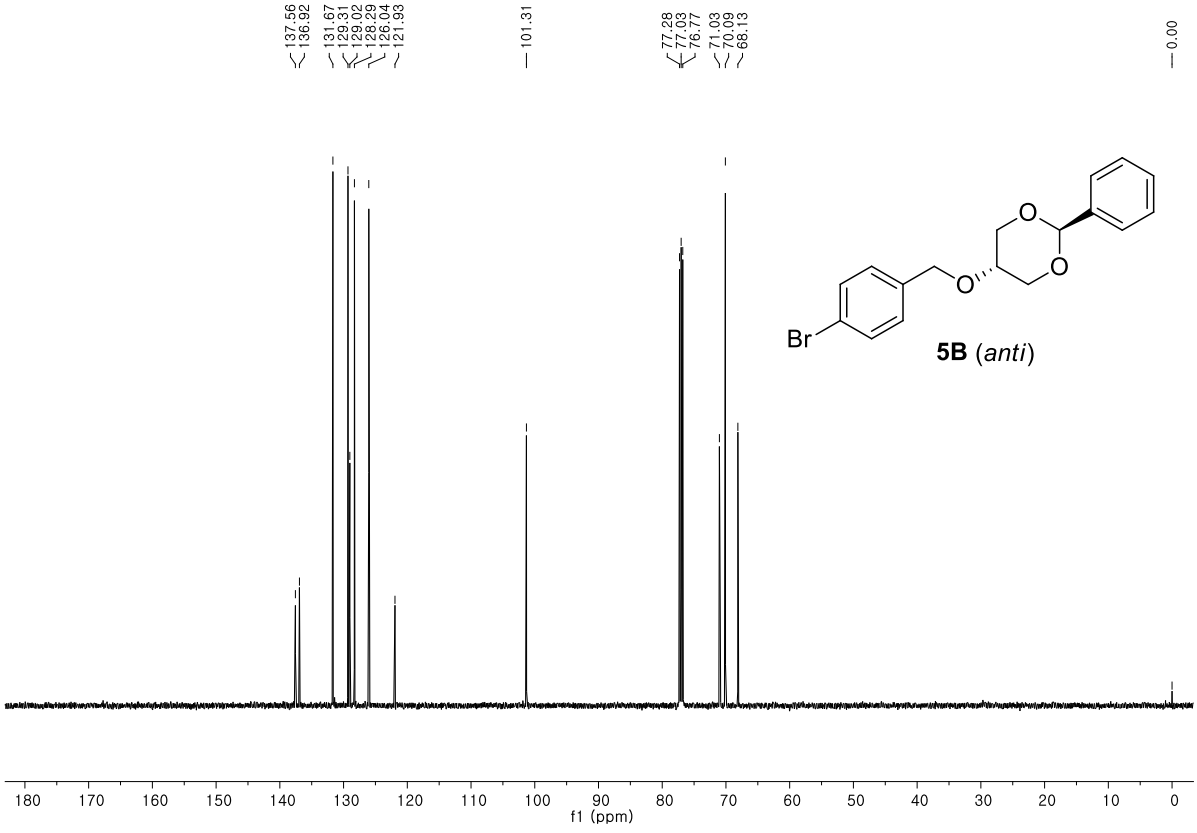
7.33
7.24
7.21
7.19
4.36
4.35
4.34
4.33
3.80
3.79
3.78
3.77
3.76
3.75
3.74
3.66
3.64
3.62
0.00
3.98
3.01
2.00
1.00
2.09
2.07
1.02
2.09
f1 (ppm)
Br
5B (anti)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101.31
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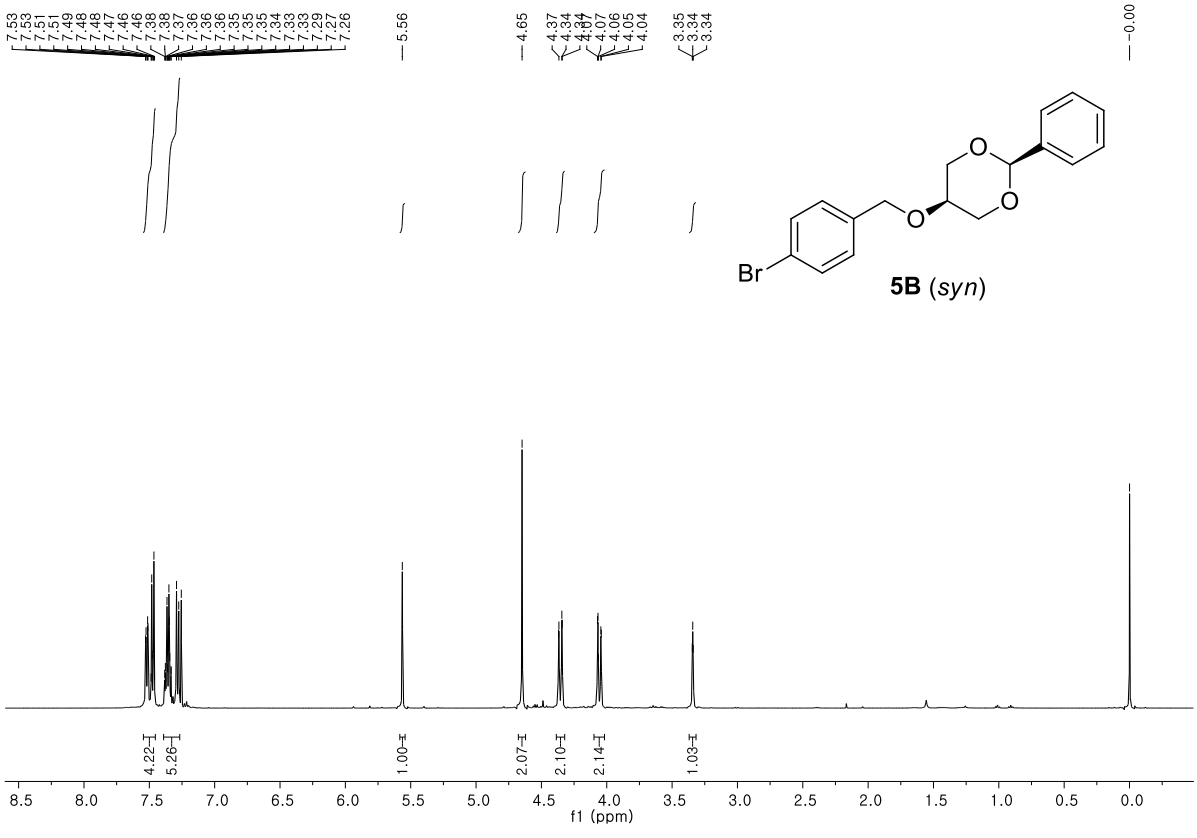
77.03
76.77
71.03
70.09
68.13
0.00
f1 (ppm)
Br
5B (anti)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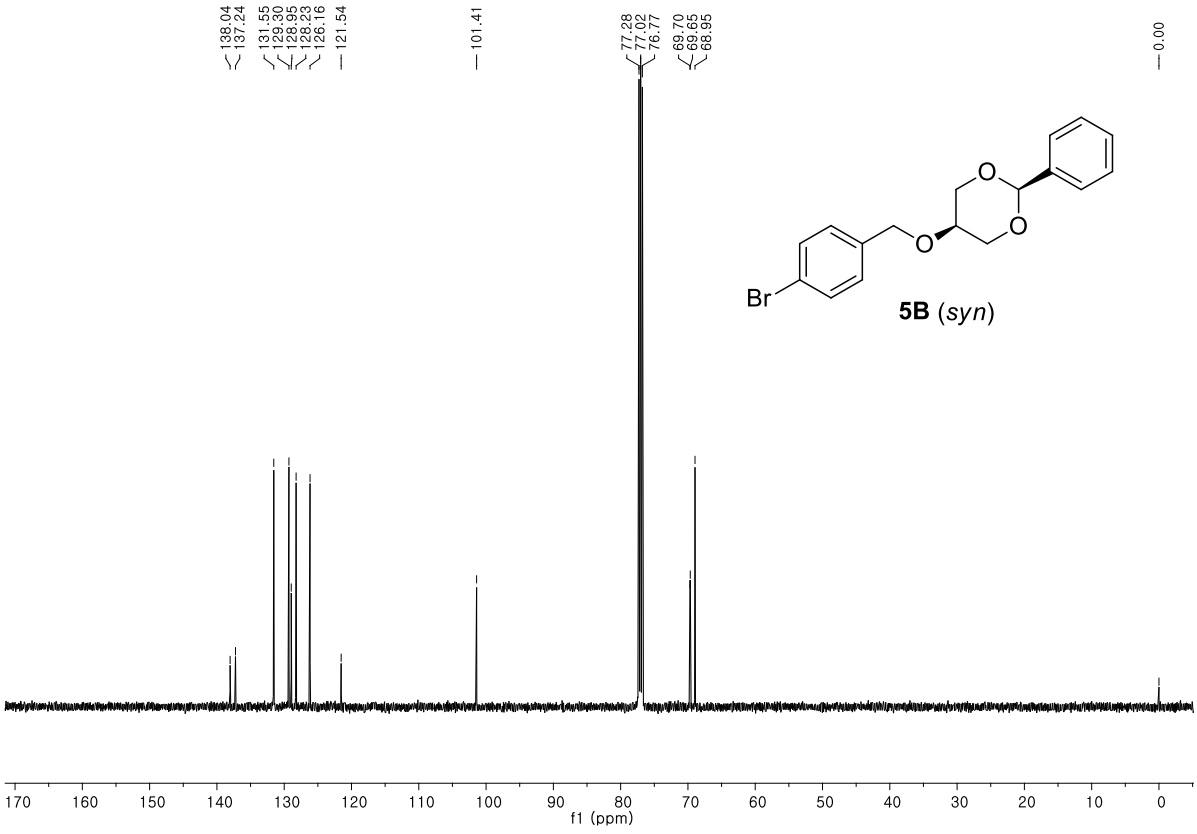
7.38
7.37
7.36
7.36
7.35
7.35
7.34
7.33
7.29
7.27
7.26
5.56
4.65
4.37
4.34
4.07
4.06
4.05
4.04
3.35
3.34
3.34
0.00
O
O
O
Br
5B (syn)
4.22
5.26
1.00
2.07
2.10
2.14
1.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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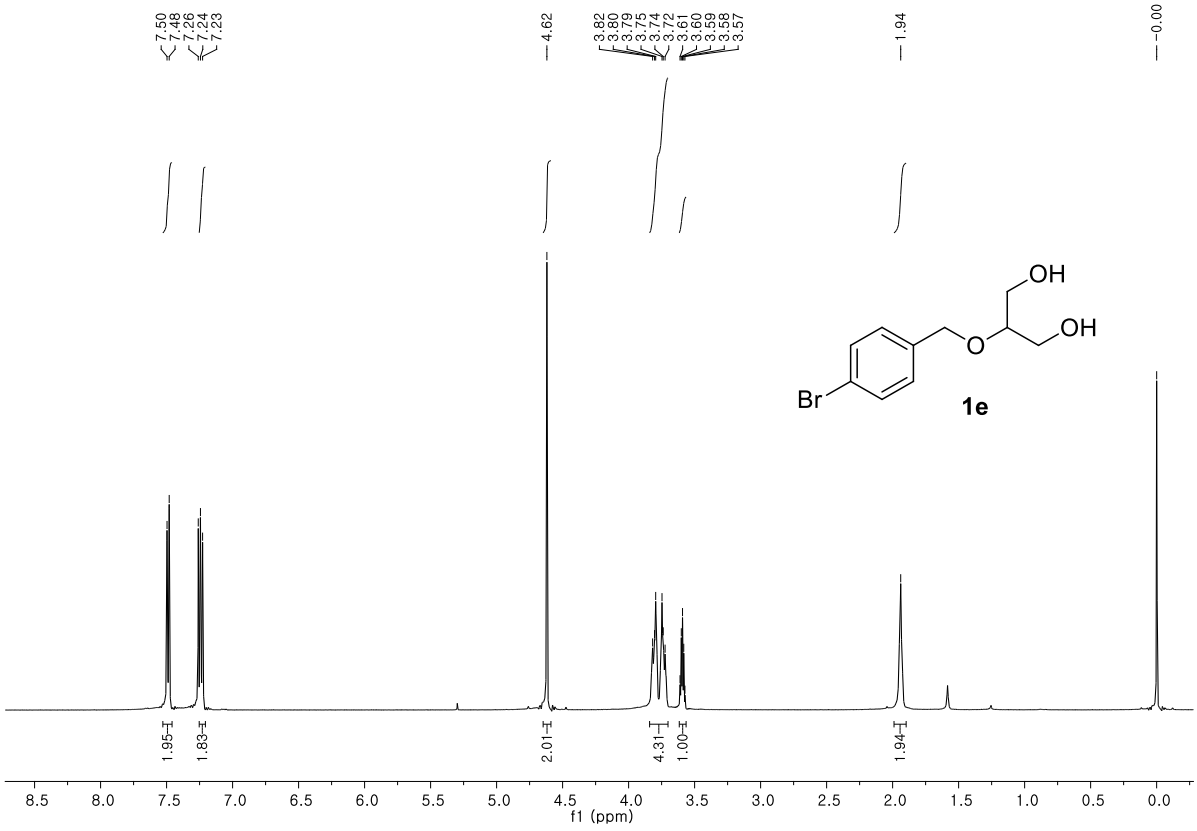
129.30
128.95
128.23
126.16
121.54
101.41
77.28
77.02
76.77
69.70
69.65
68.95
0.00
O
O
O
Br
5B (syn)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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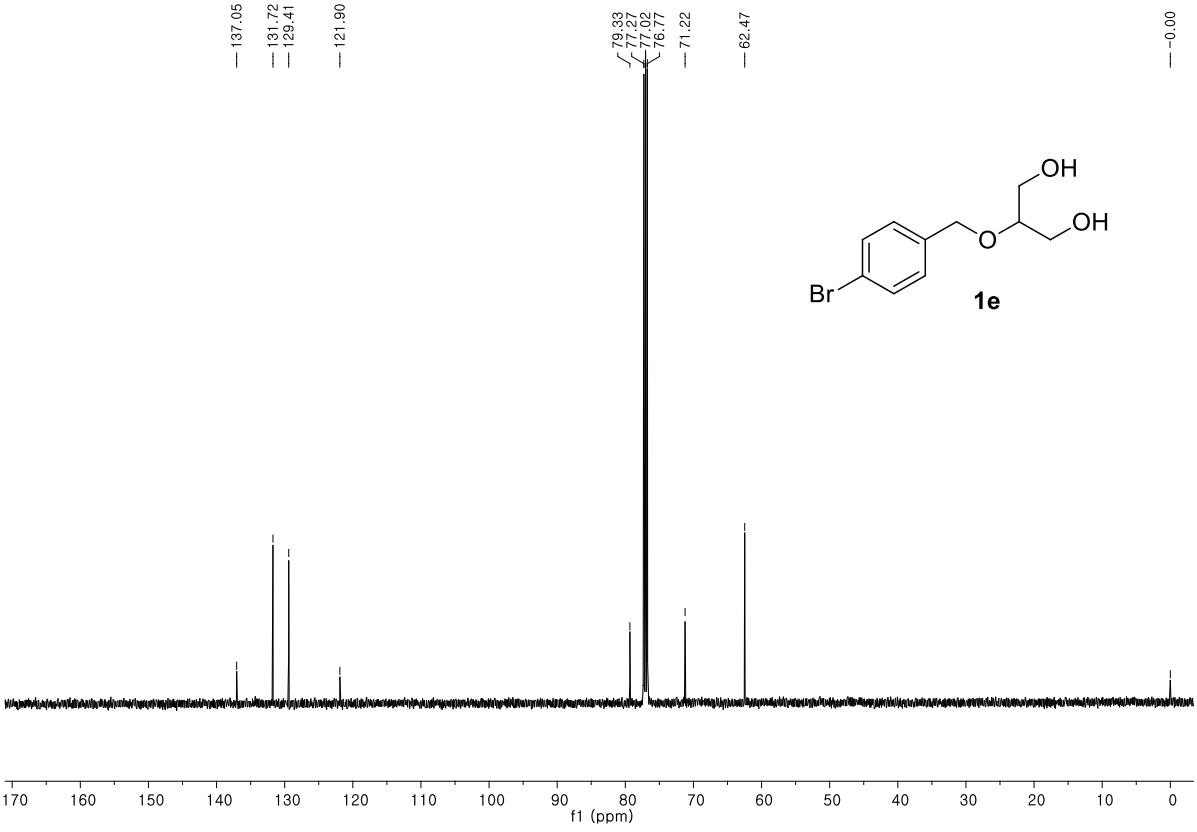
7.48
7.26
7.24
7.23
4.62
3.82
3.80
3.79
3.75
3.74
3.72
3.61
3.60
3.59
3.58
3.57
1.94
0.00
OH
OH
O
Br
1e
1.95
1.83
2.01
4.31
1.00
1.94
f1 (ppm)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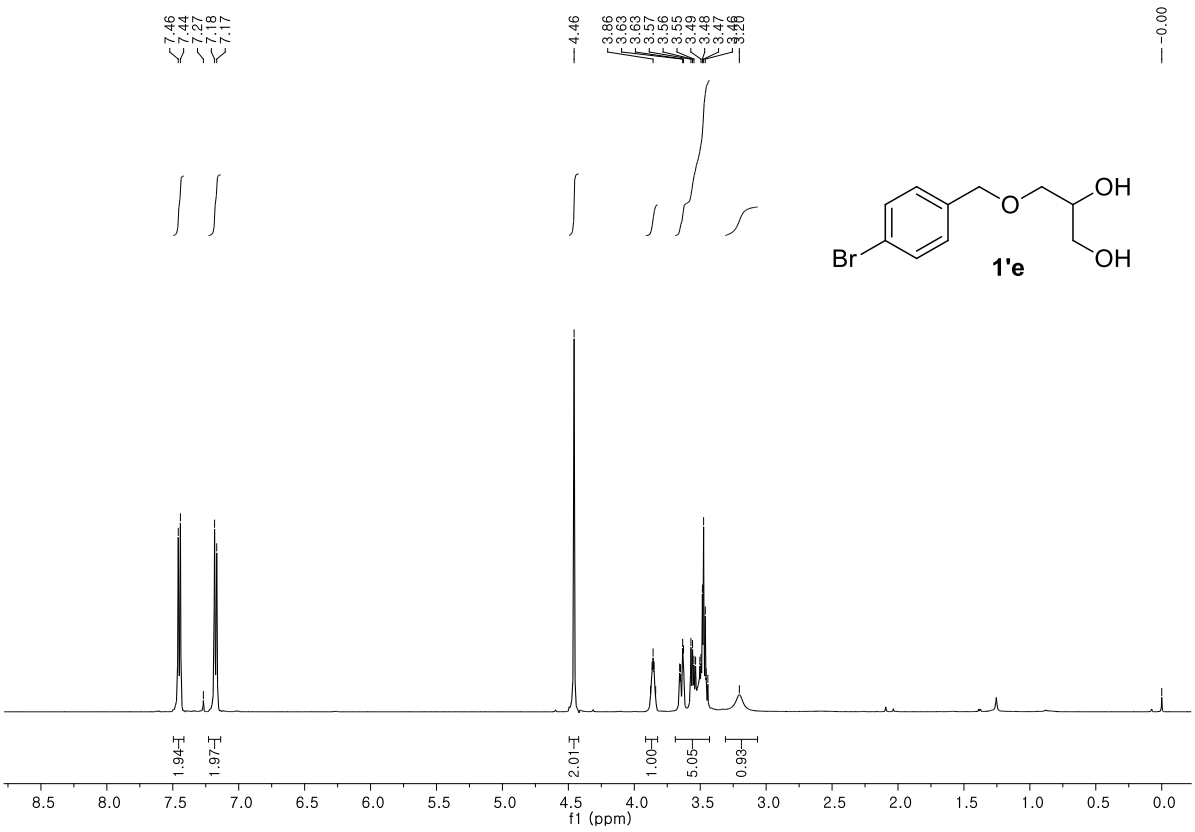
131.72
129.41
121.90
79.33
77.27
77.02
76.77
71.22
62.47
0.00
OH
OH
O
Br
1e
f1 (ppm)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7.44
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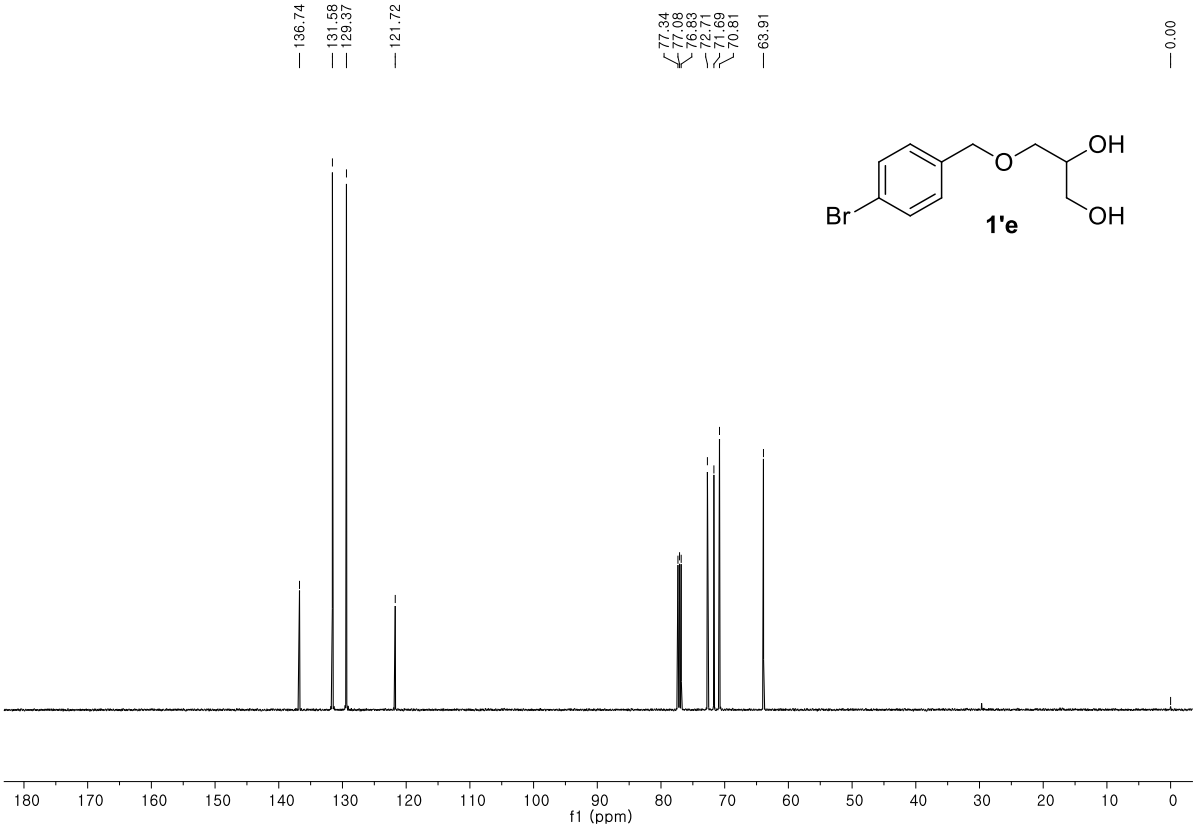
3.86
3.63
3.63
3.57
3.56
3.55
3.49
3.48
3.47
3.46
3.20
0.00
Br
O
OH
OH
1'e
1.94
1.97
2.01
1.00
5.05
0.93
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


136.74
131.58
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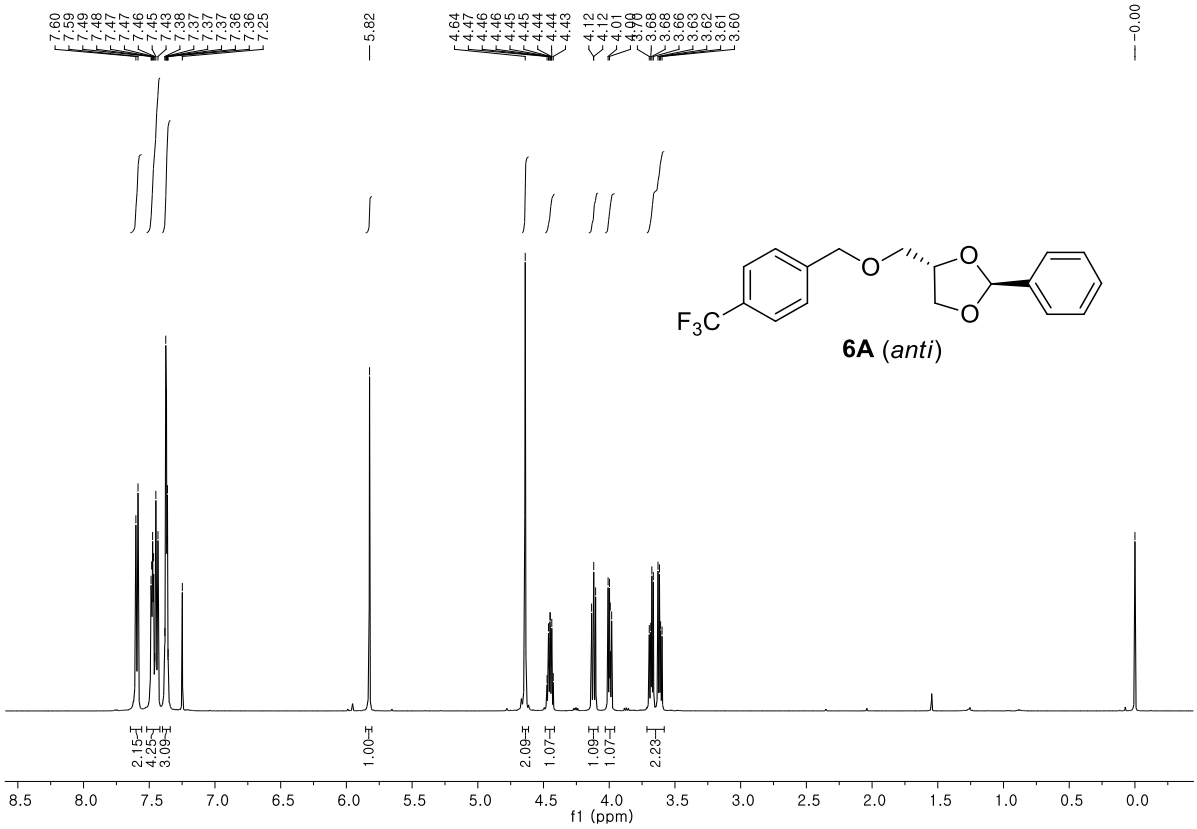
121.72
77.34
77.08
76.83
72.71
71.69
70.81
63.91
0.00
Br
O
OH
OH
1'e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.45
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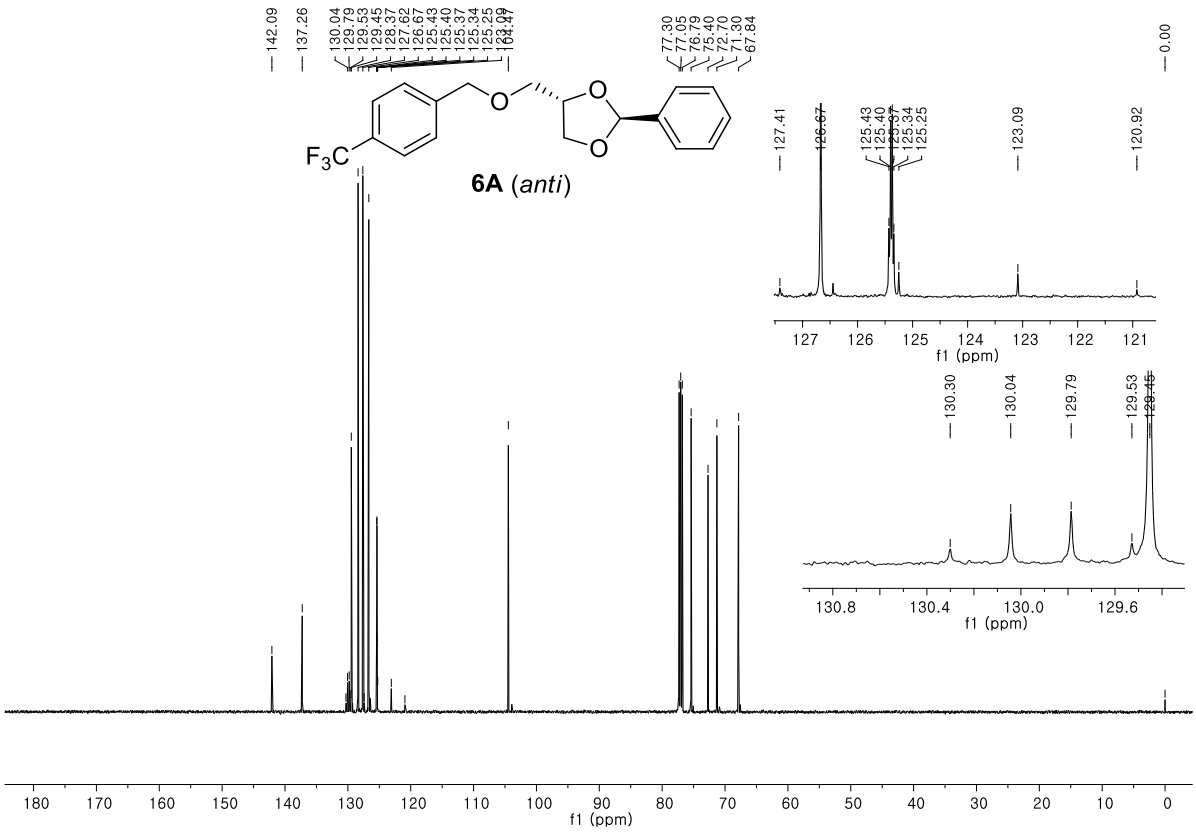
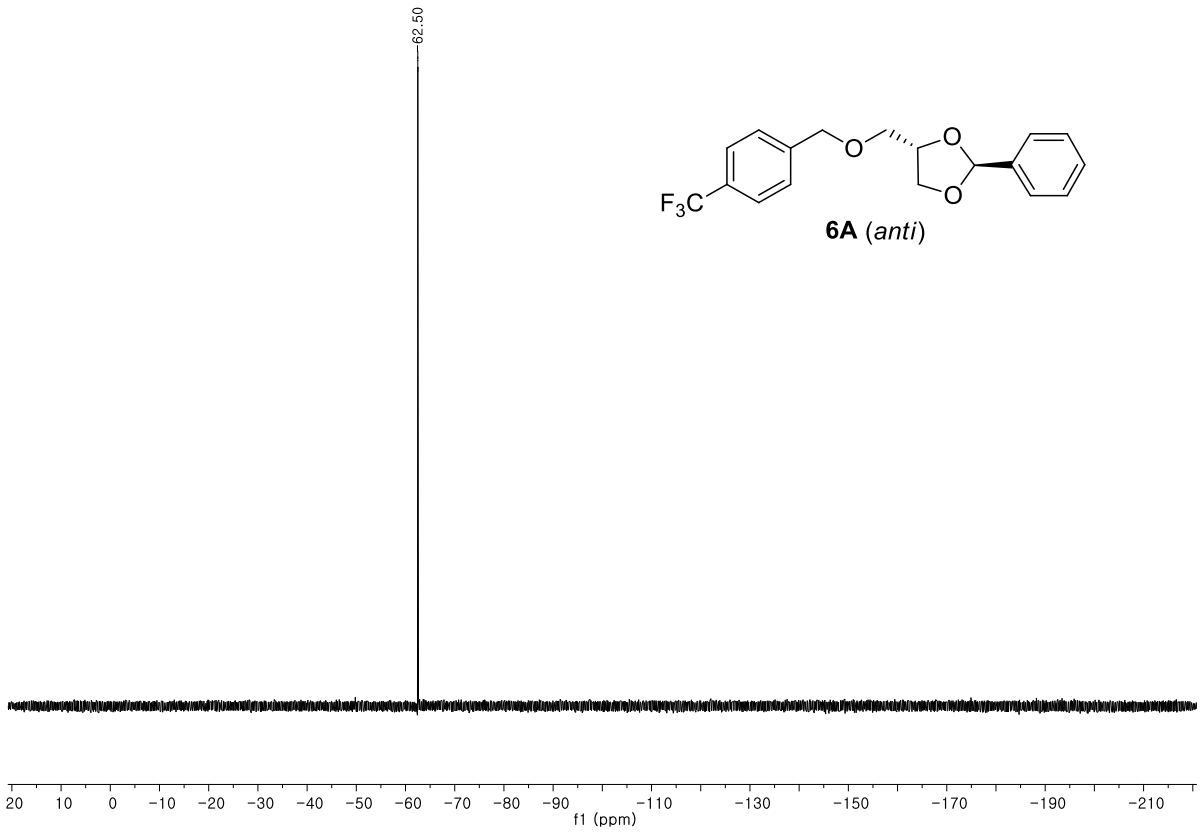
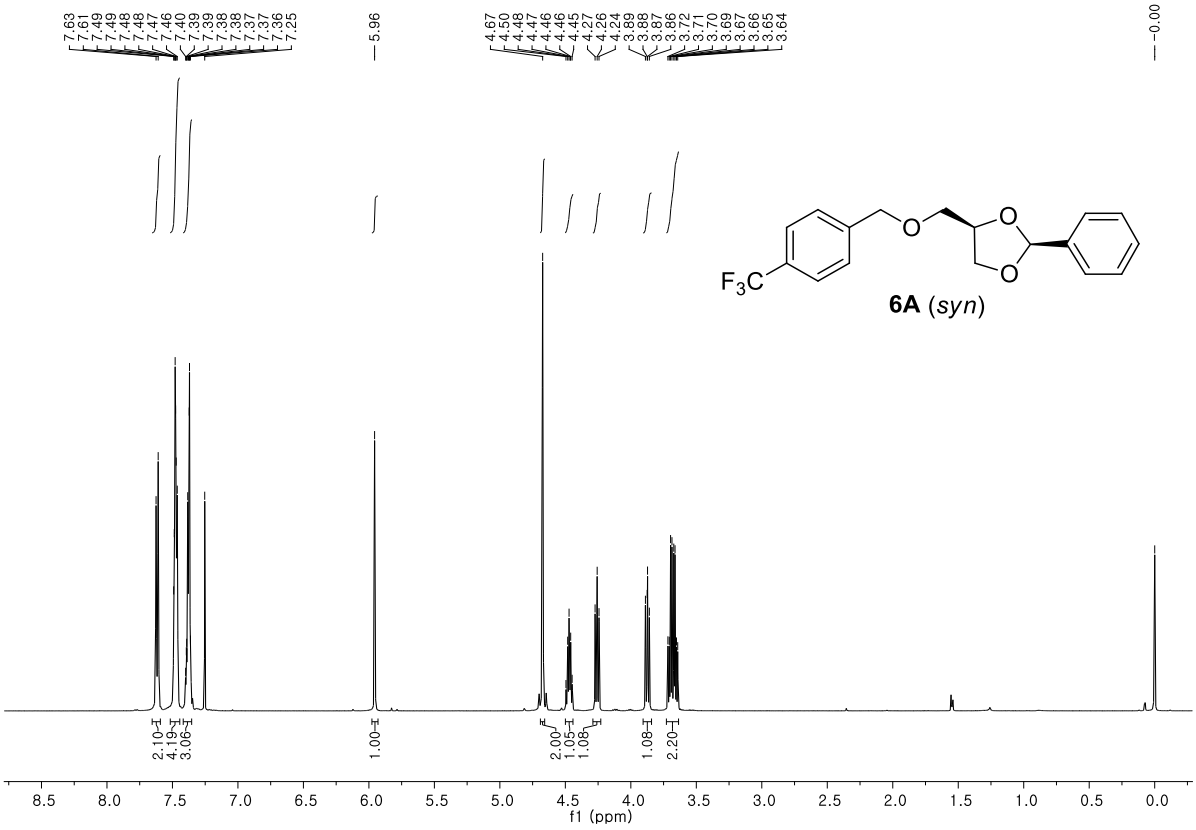
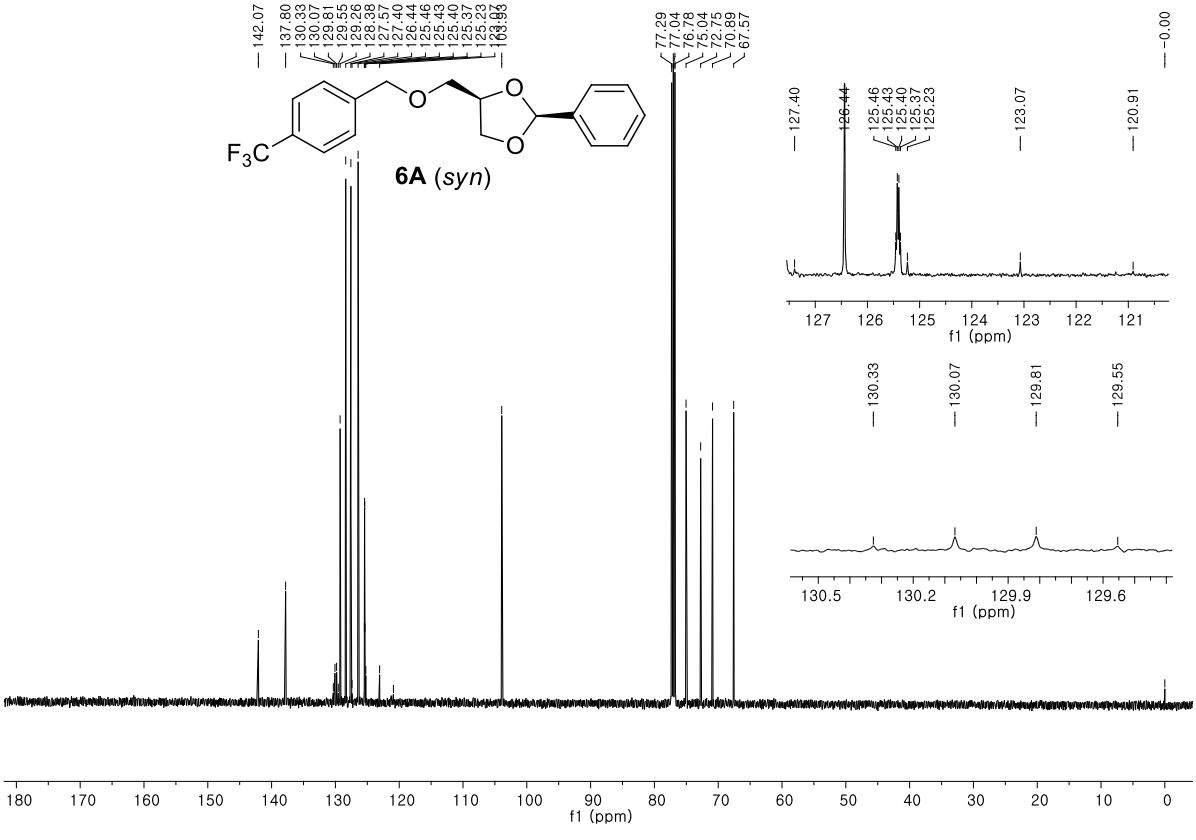
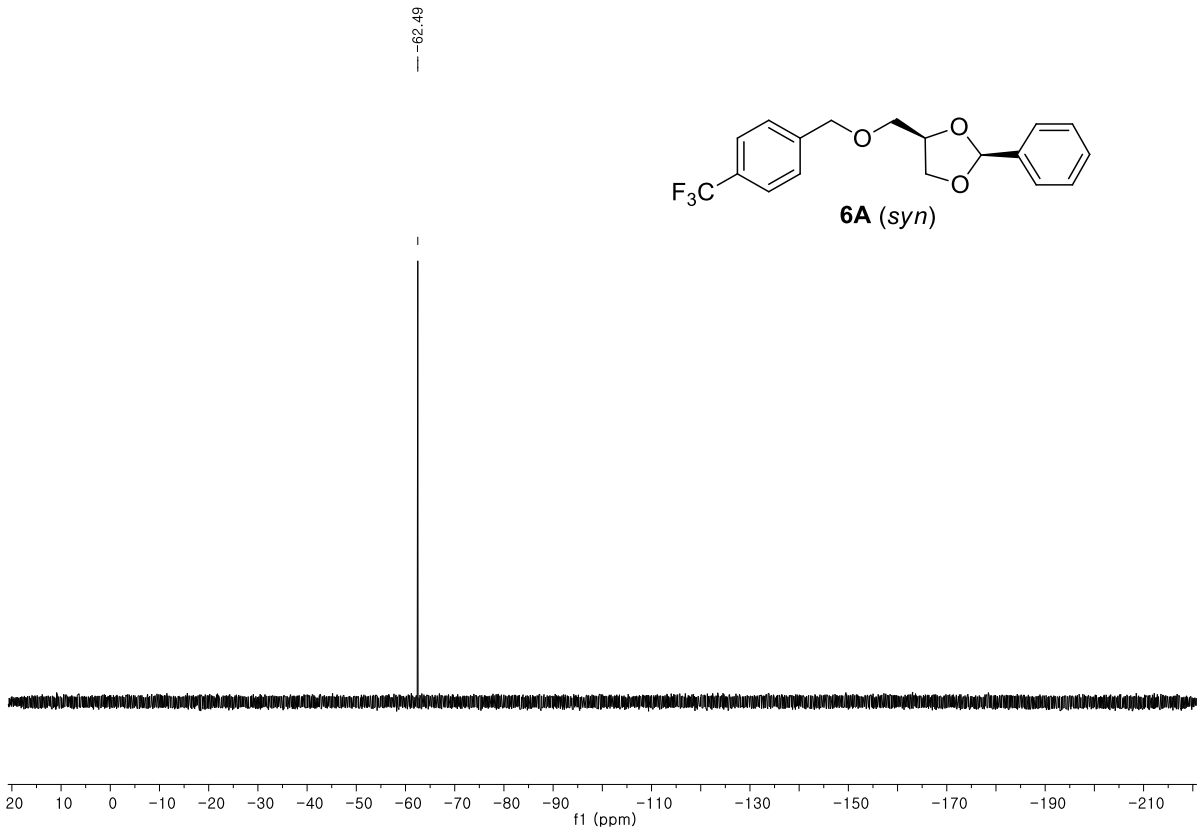
7.37
7.37
7.36
7.36
7.25
5.82
4.64
4.47
4.46
4.46
4.45
4.45
4.44
4.44
4.43
4.12
4.12
4.01
3.98
3.68
3.68
3.66
3.63
3.62
3.61
3.60
0.00
F3C
6A (anti)
2.15
4.25
3.09
1.00
2.09
1.07
1.09
1.07
2.23
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


142.09
137.26
130.04
129.79
129.53
129.45
128.37
127.62
126.67
125.43
125.40
125.37
125.34
125.25
123.09
120.92
77.30
77.05
76.79
75.40
72.70
71.30
67.84
0.00
F3C
6A (anti)
127.41
126.67
125.43
125.40
125.37
125.34
125.25
123.09
120.92
127 126 125 124 123 122 121
f1 (ppm)
130.30
130.04
129.79
129.53
129.46
130.8 130.4 130.0 129.6
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

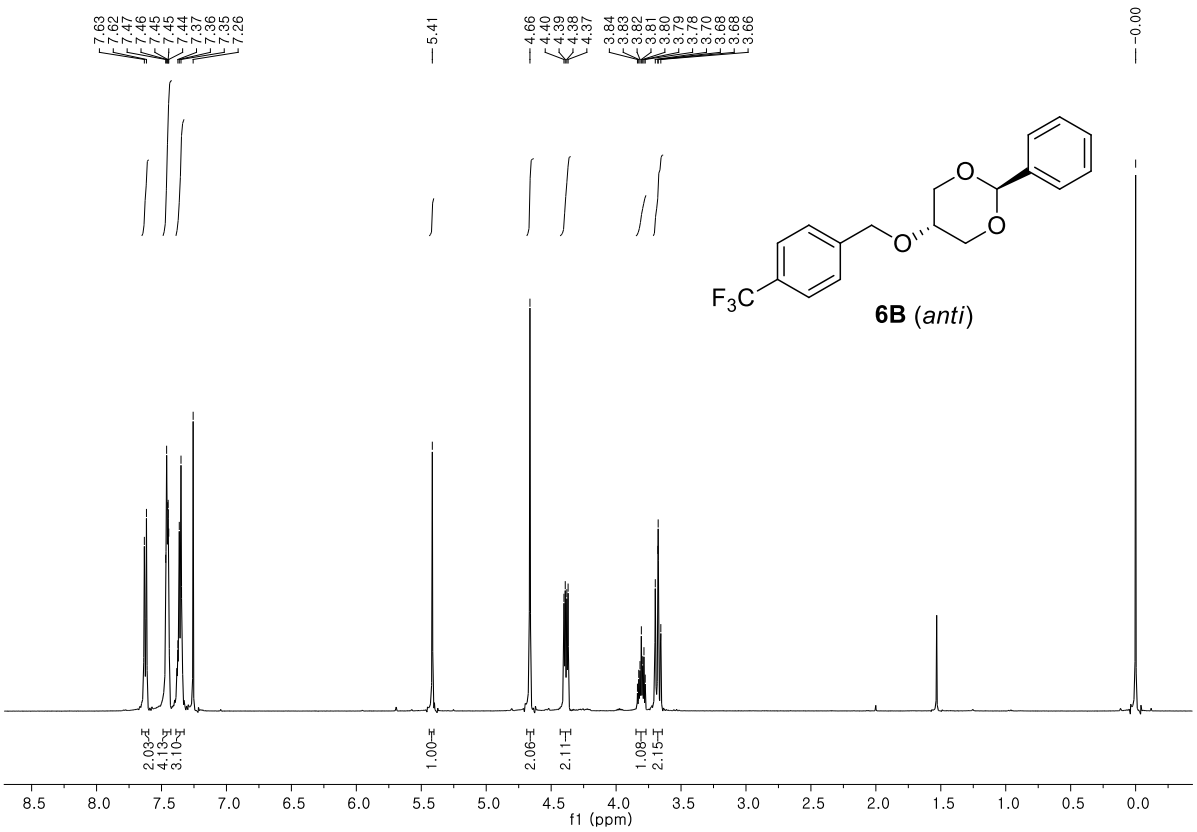

62.50
O
O
O
F3C
6A (anti)
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

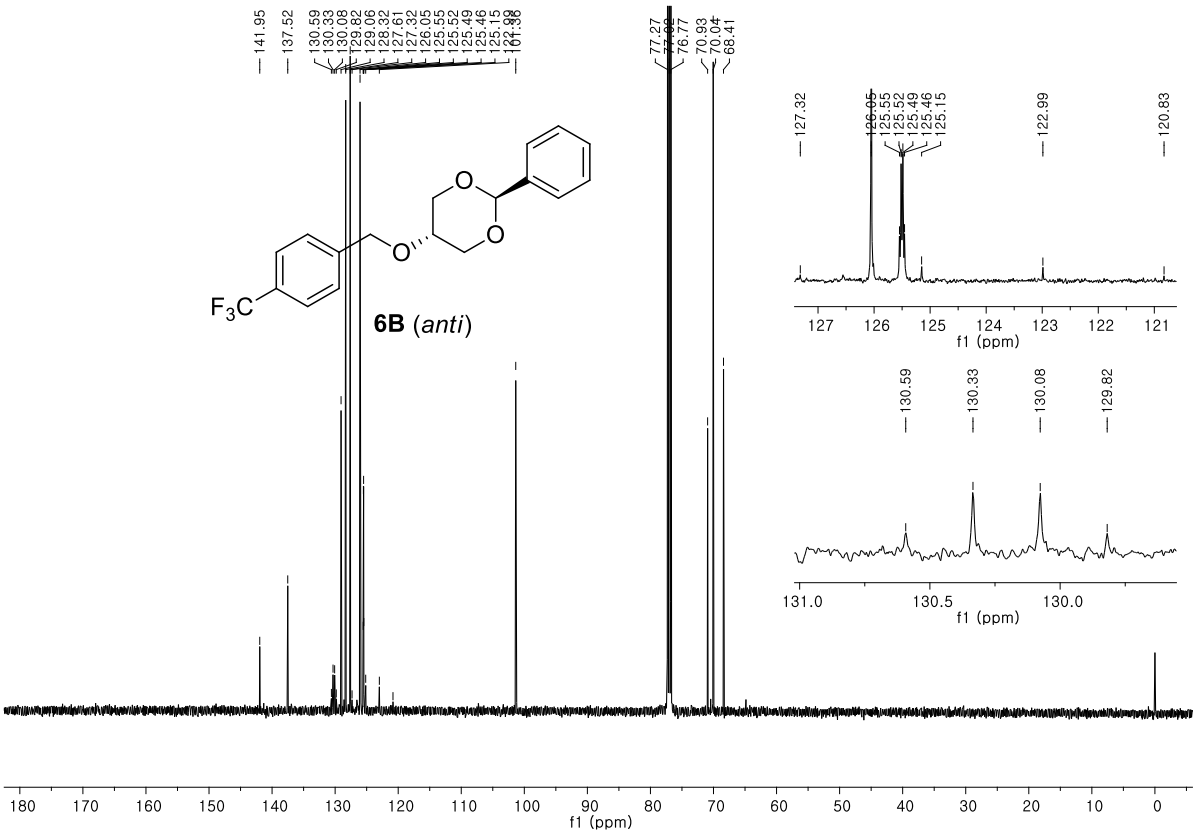

7.63
7.61
7.49
7.49
7.48
7.48
7.47
7.46
7.40
7.39
7.39
7.38
7.38
7.37
7.37
7.36
7.25
5.96
4.67
4.50
4.48
4.47
4.46
4.46
4.45
4.27
4.26
4.24
3.89
3.88
3.87
3.86
3.72
3.71
3.70
3.69
3.67
3.66
3.65
3.64
0.00
F3C
6A (syn)
2.10
4.19
3.06
1.00
2.00
1.05
1.08
1.08
2.20
f1 (ppm)

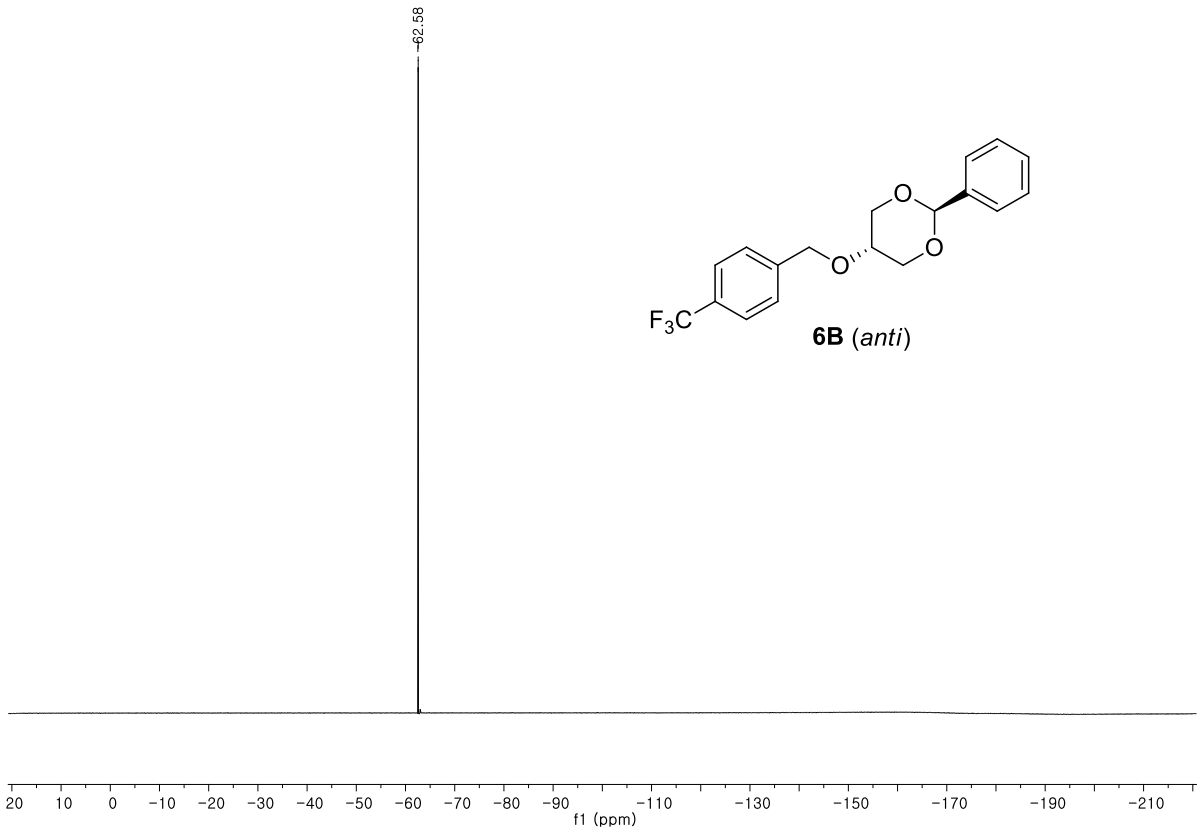

142.07
137.80
130.33
130.07
129.81
129.55
129.26
128.38
127.57
127.40
126.44
125.46
125.43
125.40
125.37
125.23
123.07
120.91
77.29
77.04
76.78
75.04
72.75
70.89
67.57
0.00
F3C
6A (syn)
127.40
126.44
125.46
125.43
125.40
125.37
125.23
123.07
120.91
f1 (ppm)
130.33
130.07
129.81
129.55
f1 (ppm)
f1 (ppm)


-62.49
O
O
O
F3C
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

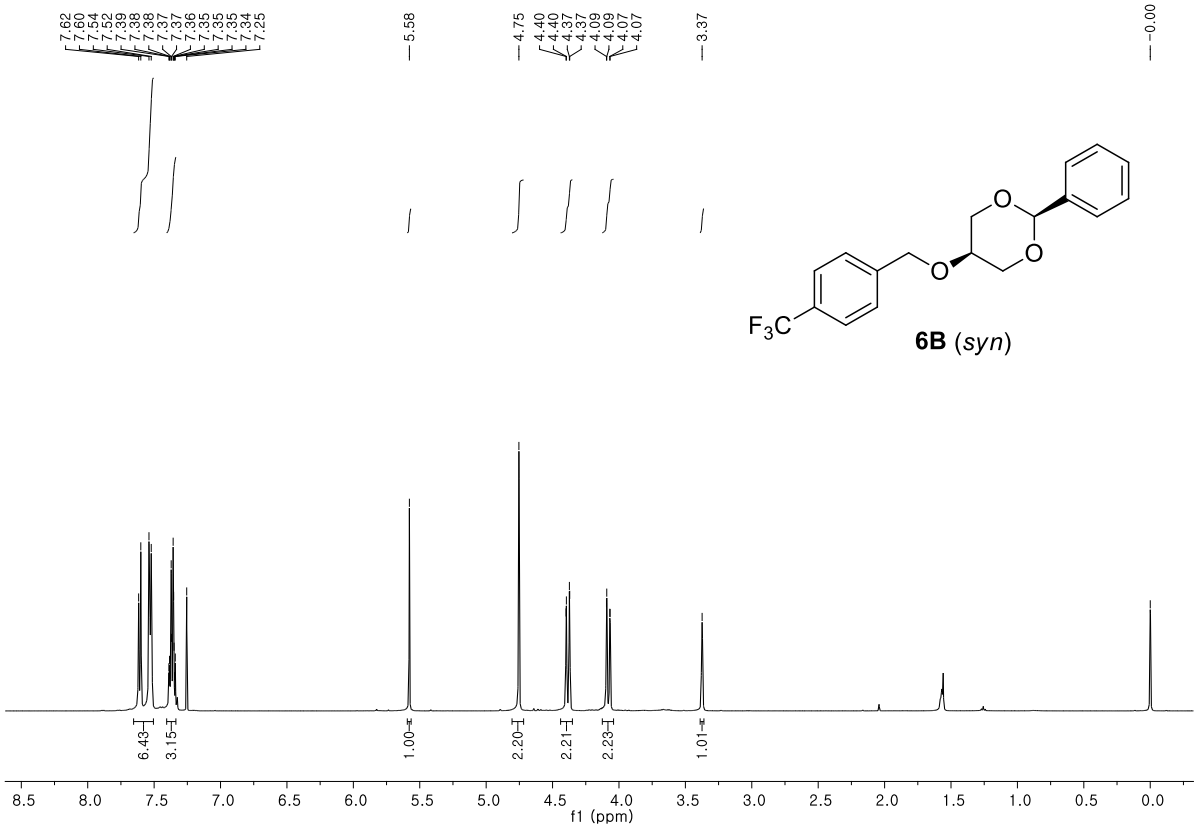
6A (*syn*)

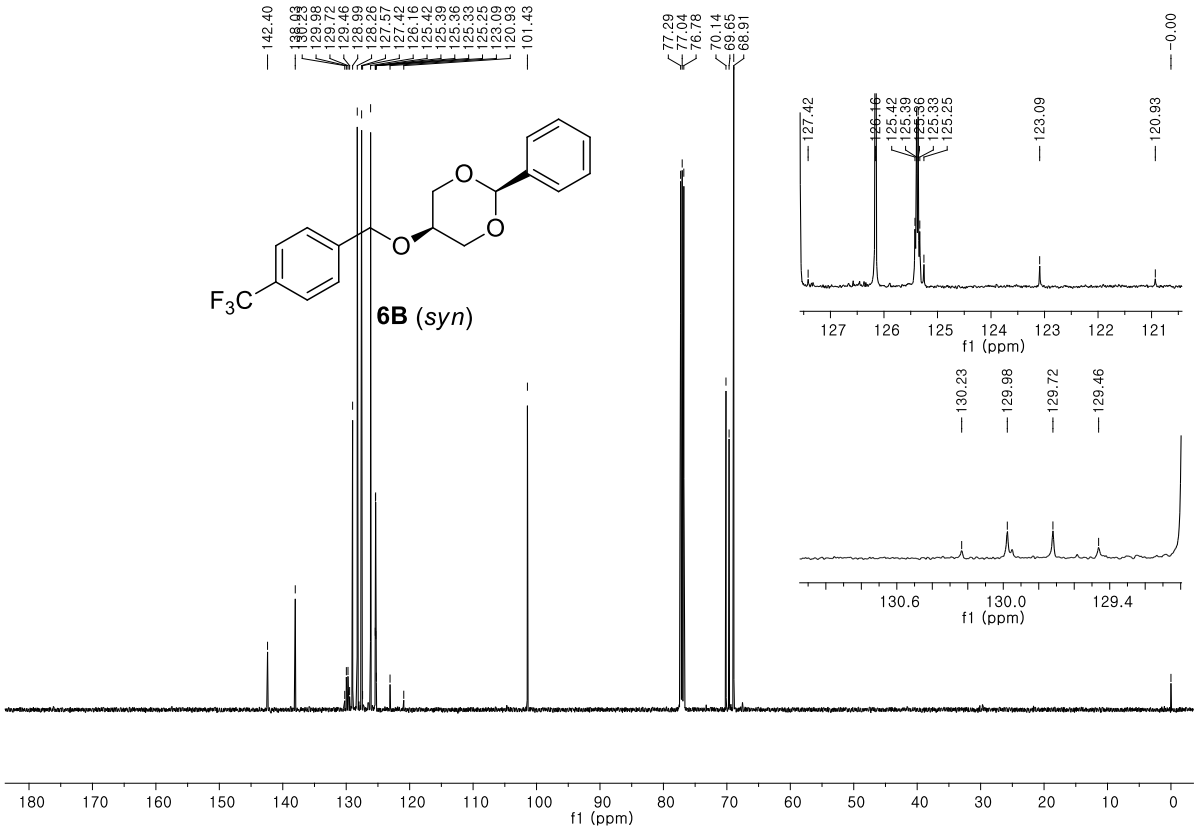

7.63
7.62
7.47
7.46
7.45
7.44
7.37
7.36
7.35
7.26
5.41
4.66
4.40
4.39
4.38
4.37
3.84
3.83
3.82
3.81
3.80
3.79
3.78
3.70
3.68
3.68
3.66
0.00
2.03
4.13
3.10
1.00
2.06
2.11
1.08
2.15
F3C
6B (anti)
f1 (ppm)

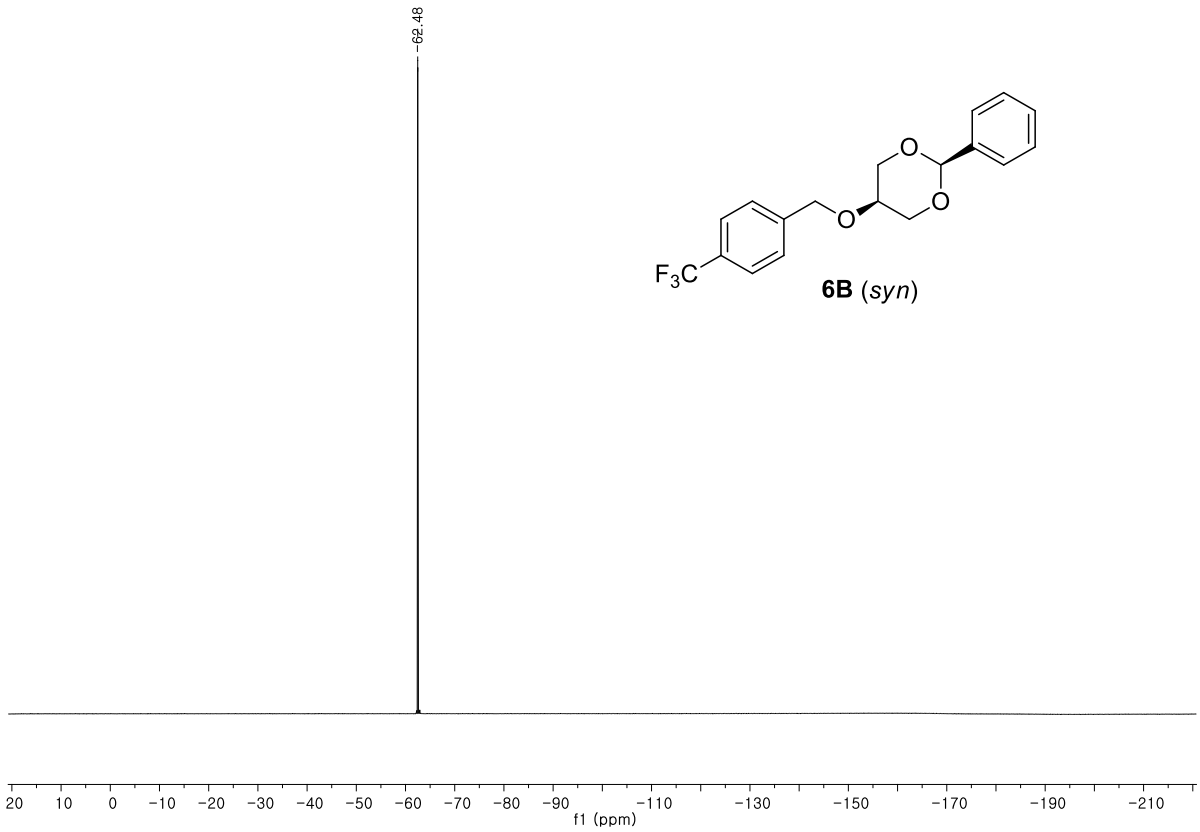

141.95
137.52
130.59
130.33
130.08
129.82
129.06
128.32
127.61
127.32
126.05
125.55
125.52
125.49
125.46
125.15
77.27
76.77
70.93
70.04
68.41
F3C
6B (anti)
127.32
126.05
125.55
125.52
125.49
125.46
125.15
122.99
120.83
130.59
130.33
130.08
129.82
f1 (ppm)

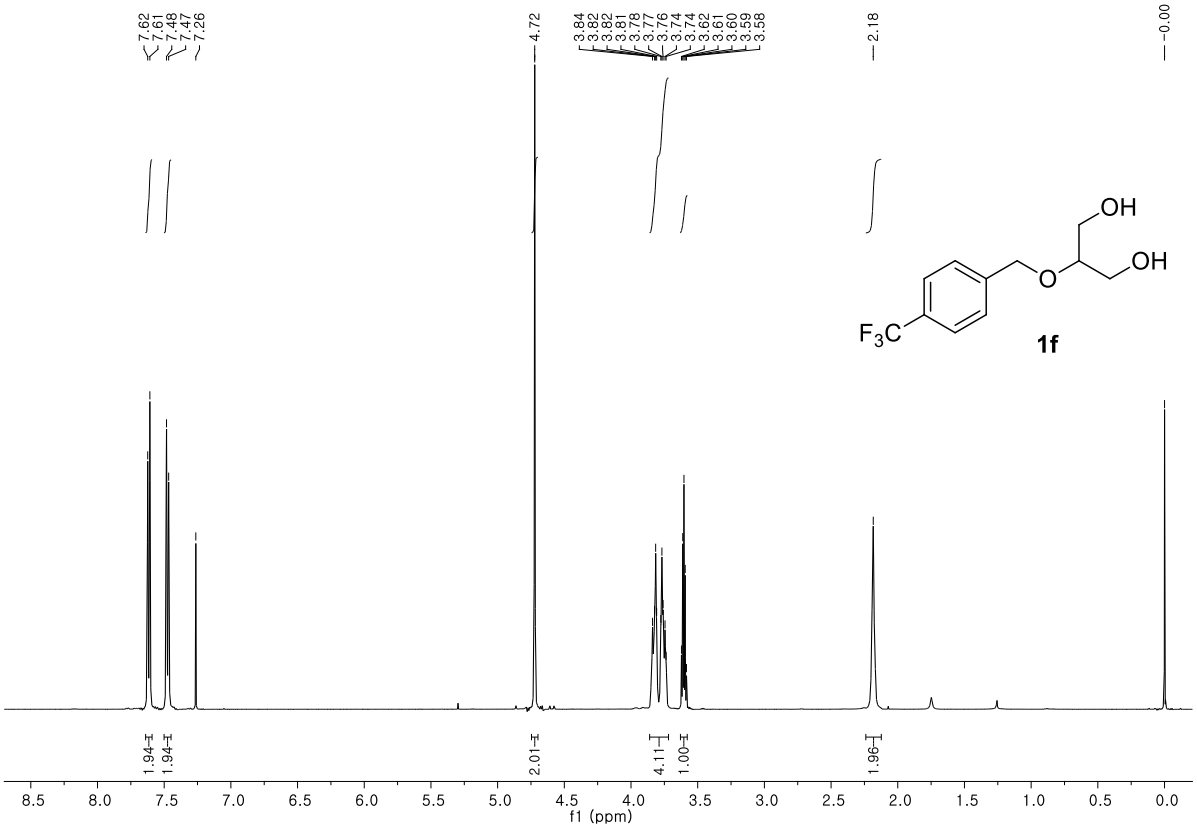

-62.58
O
O
O
F3C
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

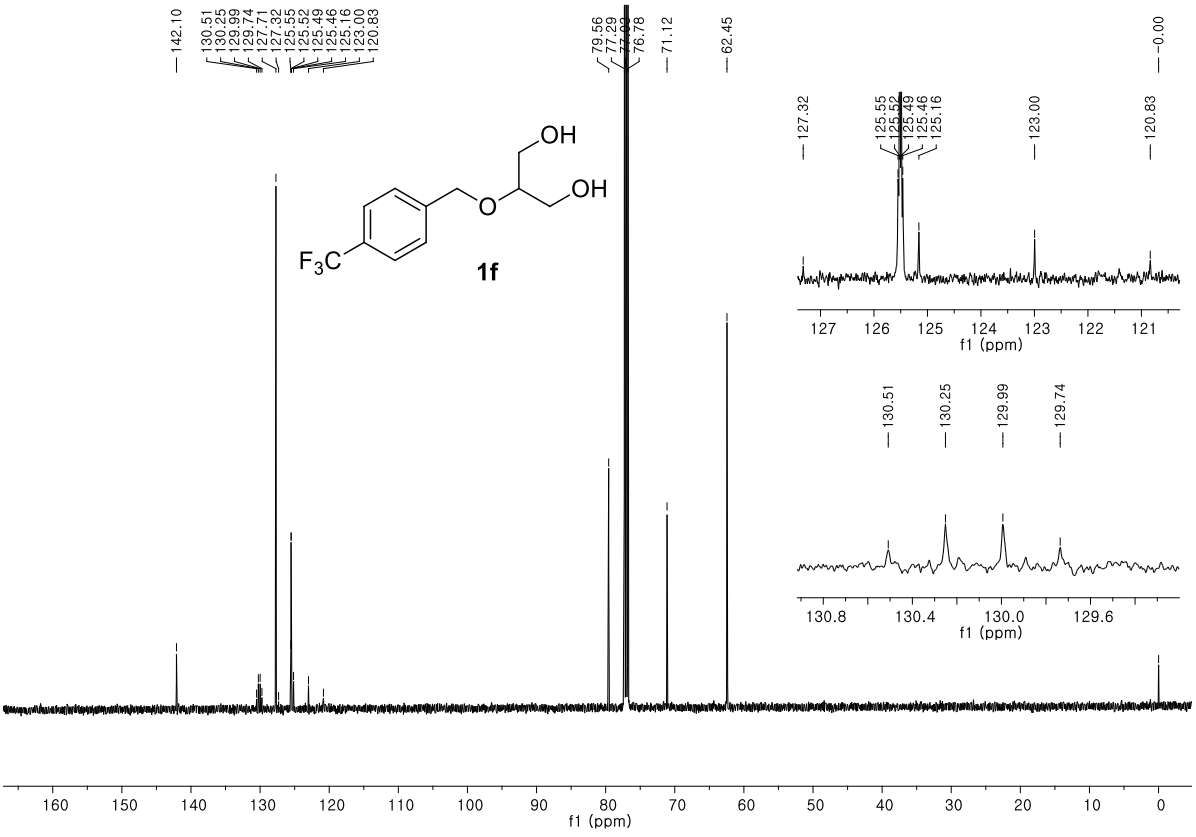
6B (*anti*)

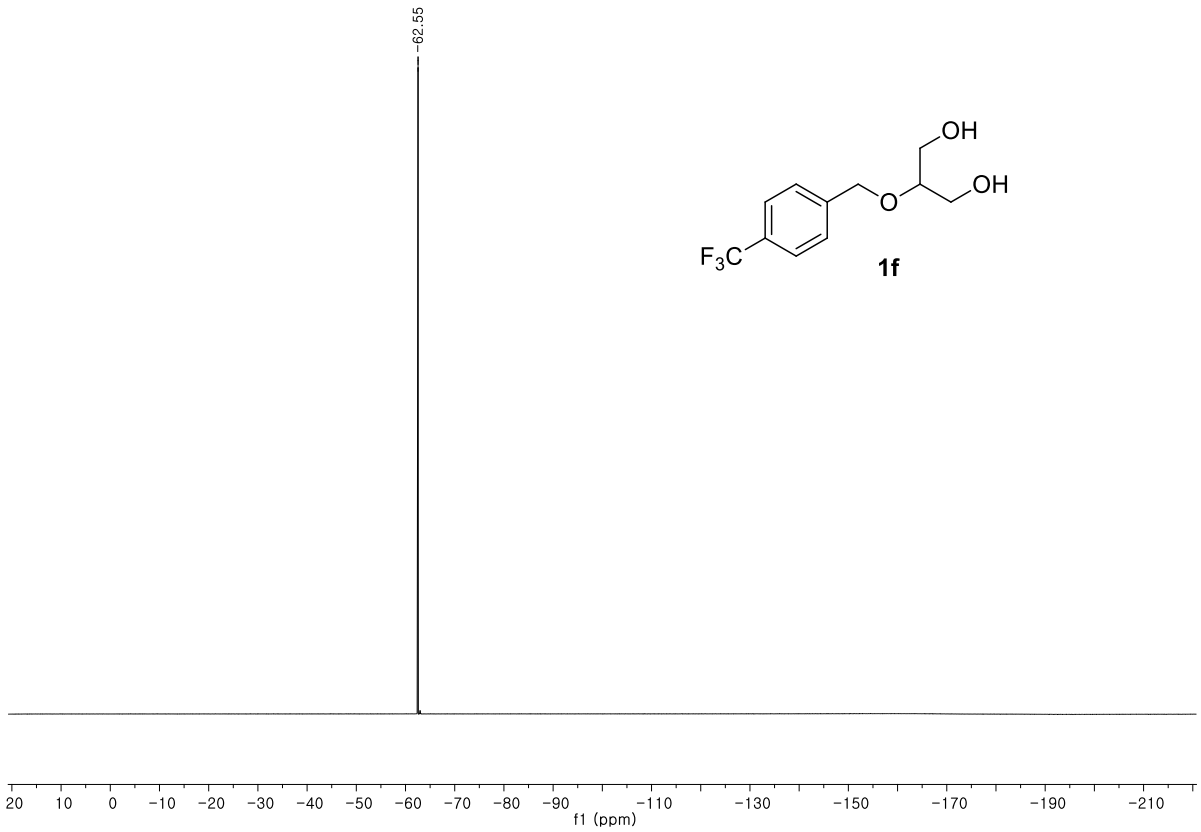

7.62
7.60
7.54
7.52
7.39
7.38
7.38
7.37
7.37
7.36
7.35
7.35
7.35
7.34
7.25
5.58
4.75
4.40
4.40
4.37
4.37
4.09
4.09
4.07
4.07
3.37
0.00
F3C
6B (syn)
6.43
3.15
1.00
2.20
2.21
2.23
1.01
f1 (ppm)

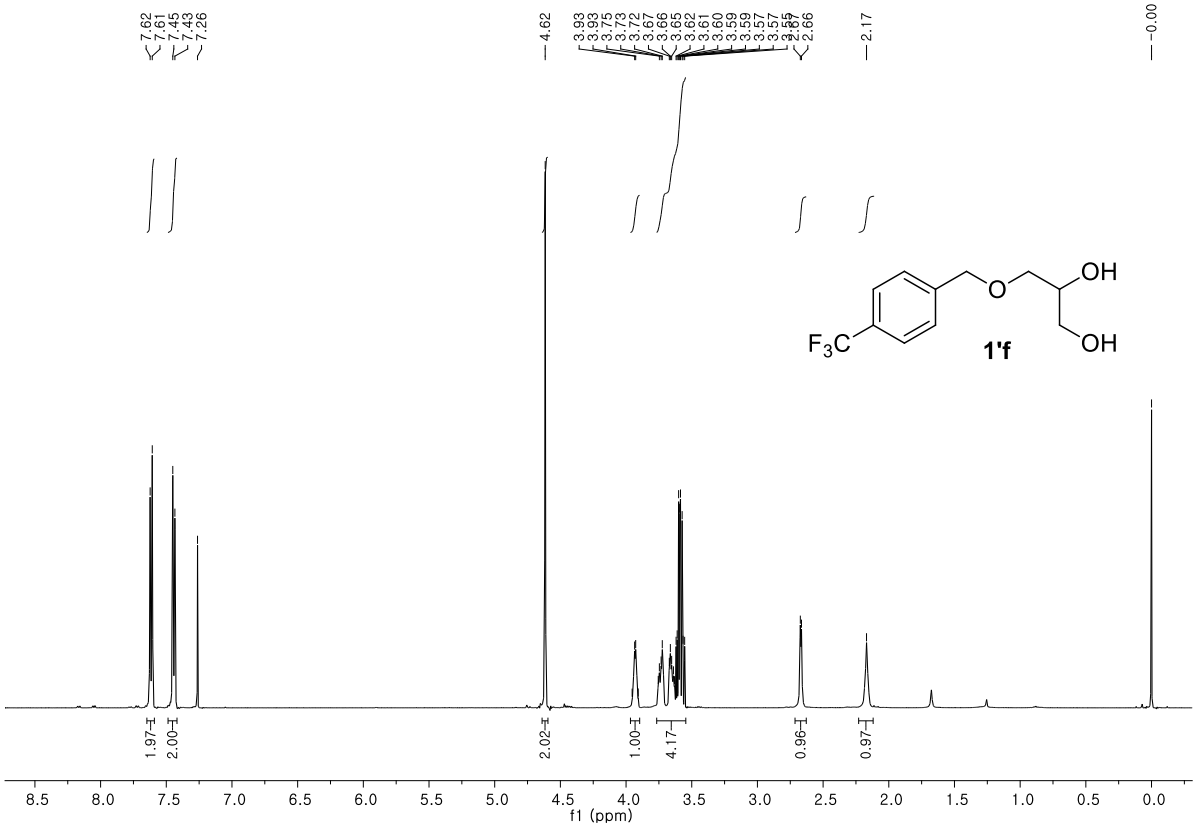

142.40
138.03
138.03
129.98
129.72
129.46
128.99
128.26
127.57
127.42
126.16
125.42
125.39
125.36
125.33
125.25
123.09
120.93
101.43
77.29
77.04
76.78
70.14
69.65
68.91
0.00
F3C
6B (syn)
127.42
126.16
125.42
125.39
125.36
125.33
125.25
123.09
120.93
f1 (ppm)
130.23
129.98
129.72
129.46
f1 (ppm)
f1 (ppm)

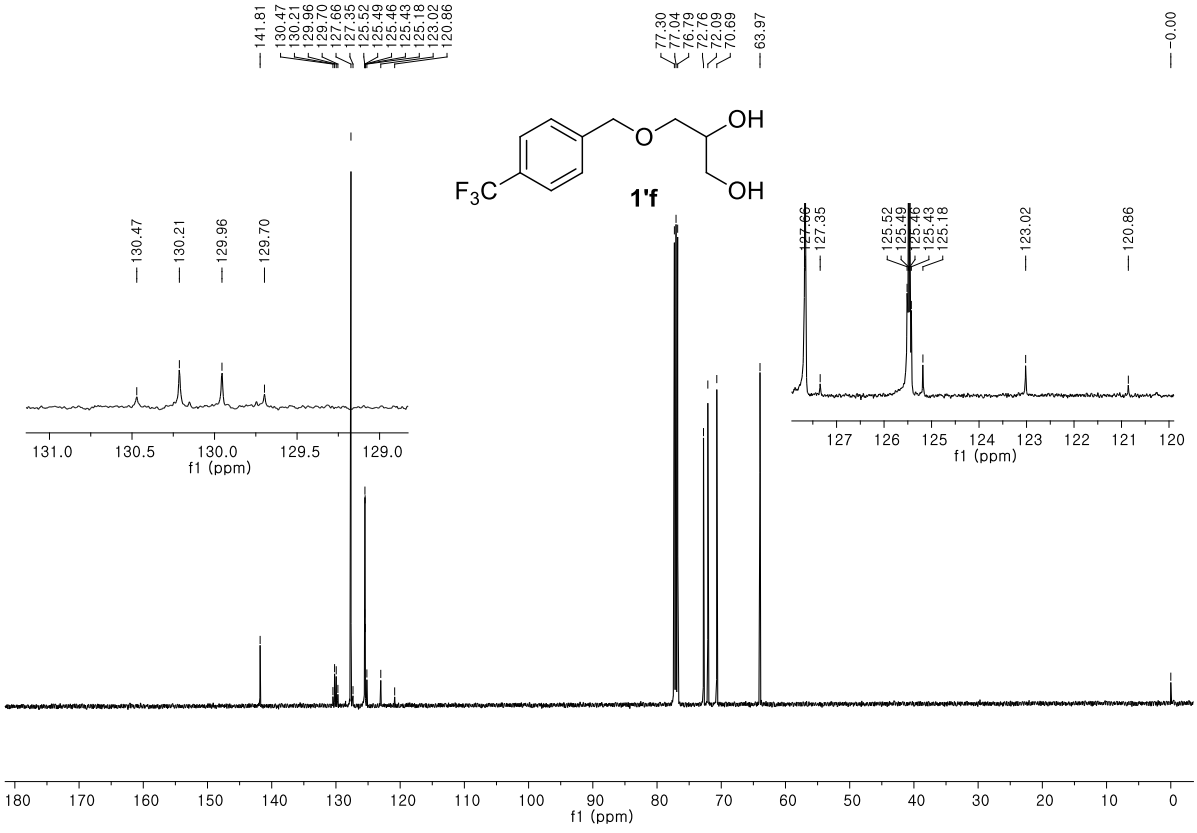

-62.48
O
O
O
F3C
6B (syn)
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

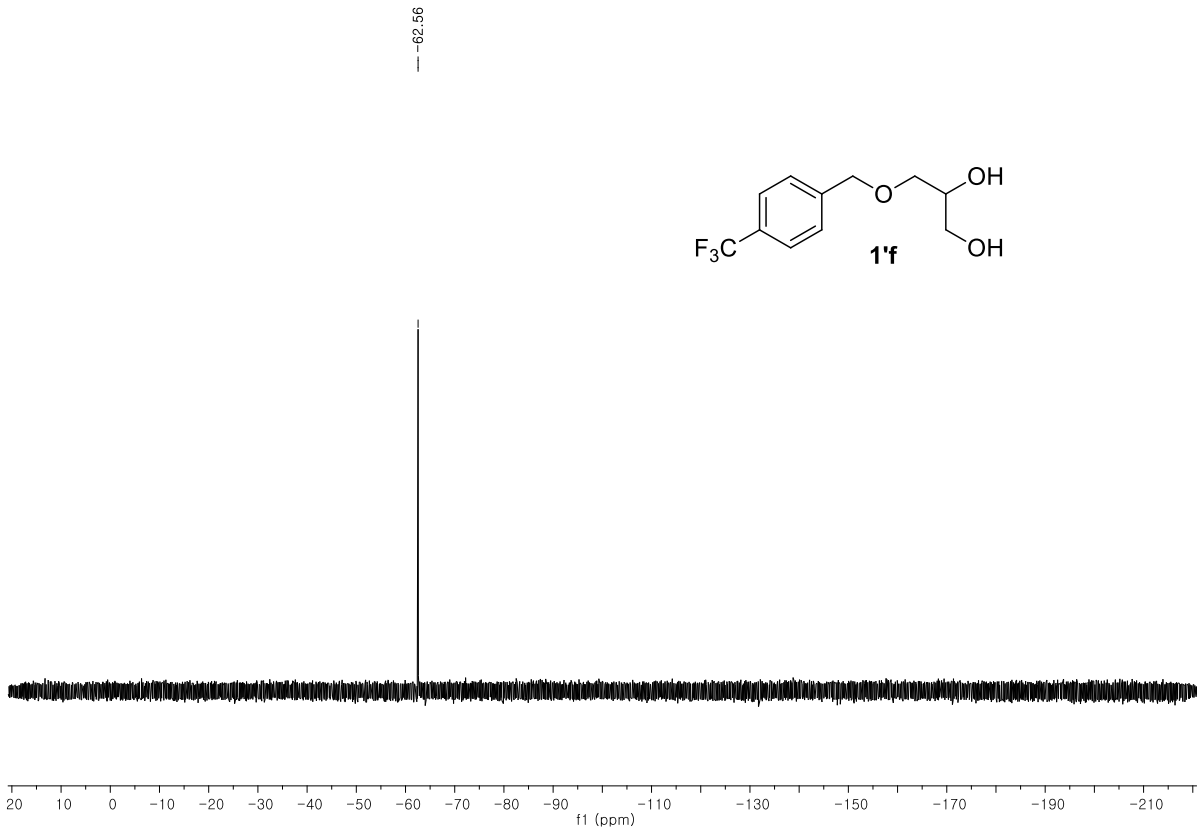

7.62
7.61
7.48
7.47
7.26
4.72
3.84
3.82
3.82
3.81
3.78
3.77
3.76
3.74
3.74
3.62
3.61
3.60
3.59
3.58
2.18
0.00
OH
OH
O
F3C
1f
1.94
1.94
2.01
4.11
1.00
1.96
f1 (ppm)

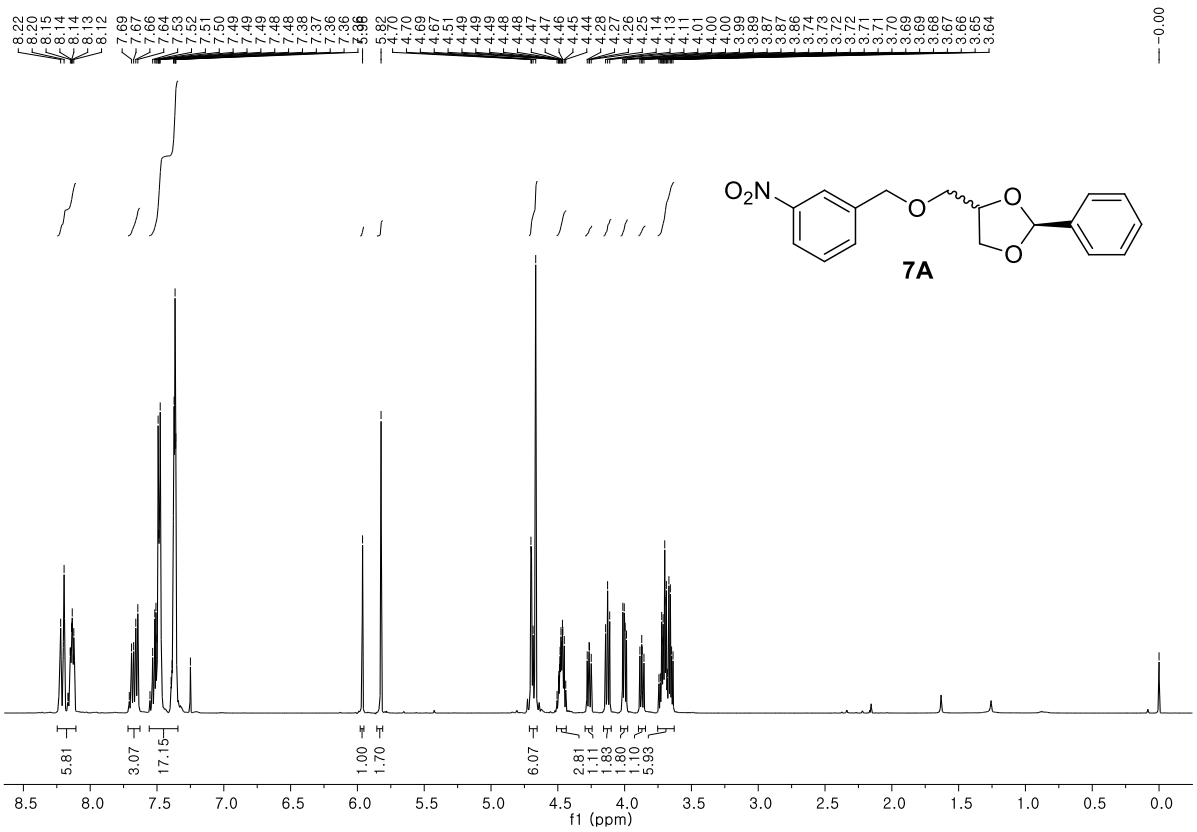

142.10
130.51
130.25
129.99
129.74
127.71
127.32
125.55
125.52
125.49
125.46
125.16
123.00
120.83
79.56
77.29
77.03
76.78
71.12
62.45
0.00
OH
OH
O
F3C
1f
127.32
125.55
125.52
125.49
125.46
125.16
123.00
120.83
f1 (ppm)
130.51
130.25
129.99
129.74
f1 (ppm)
f1 (ppm)

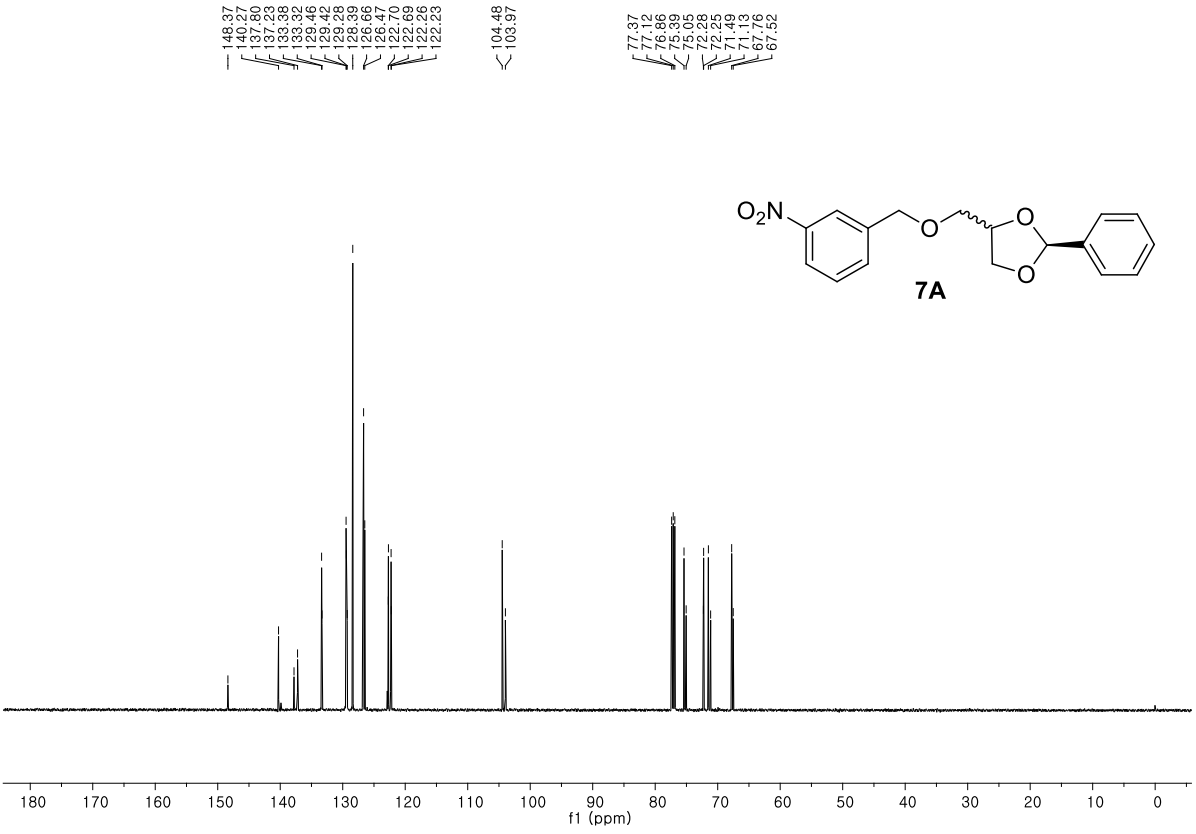

-62.55
OH
OH
O
F3C
1f
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

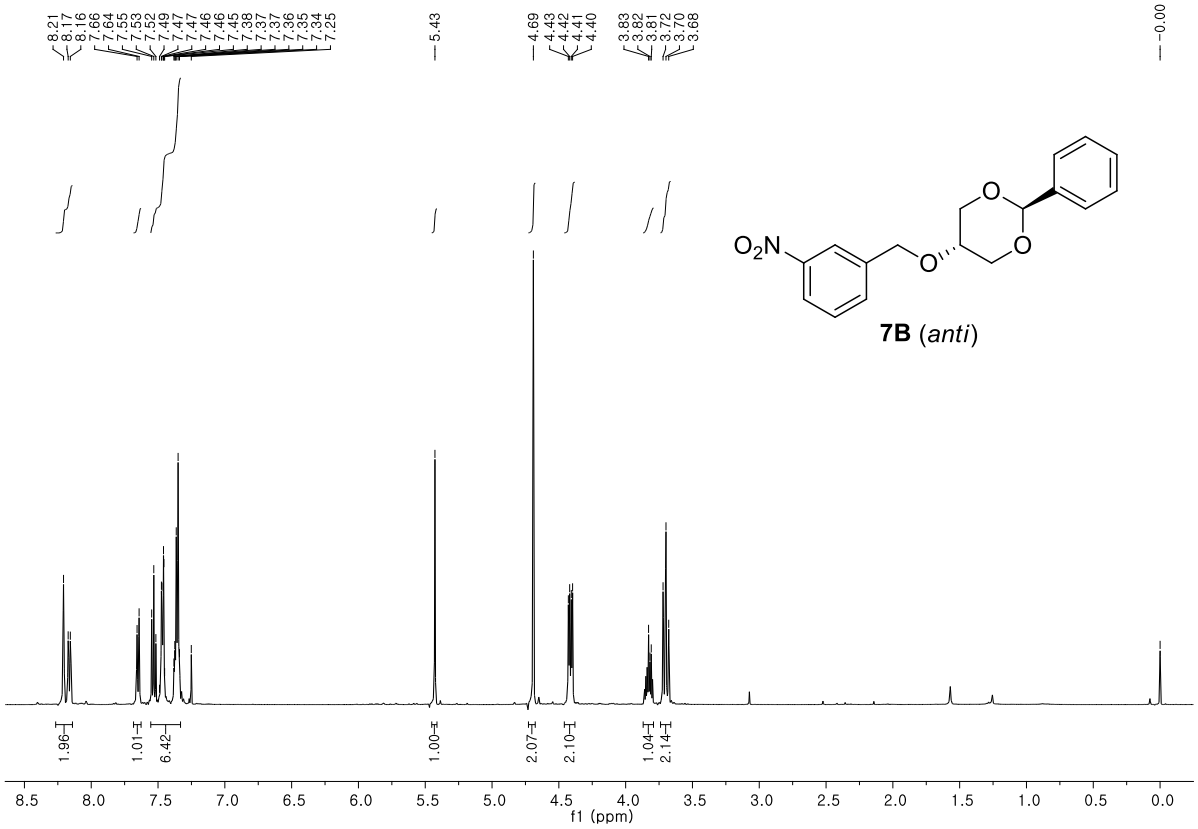

7.62
7.61
7.45
7.43
7.26
4.62
3.93
3.93
3.75
3.73
3.72
3.67
3.66
3.65
3.62
3.61
3.60
3.59
3.59
3.57
3.57
2.66
2.17
0.00
OH
O
OH
F3C
1'f
1.97
2.00
2.02
1.00
4.17
0.96
0.97
f1 (ppm)

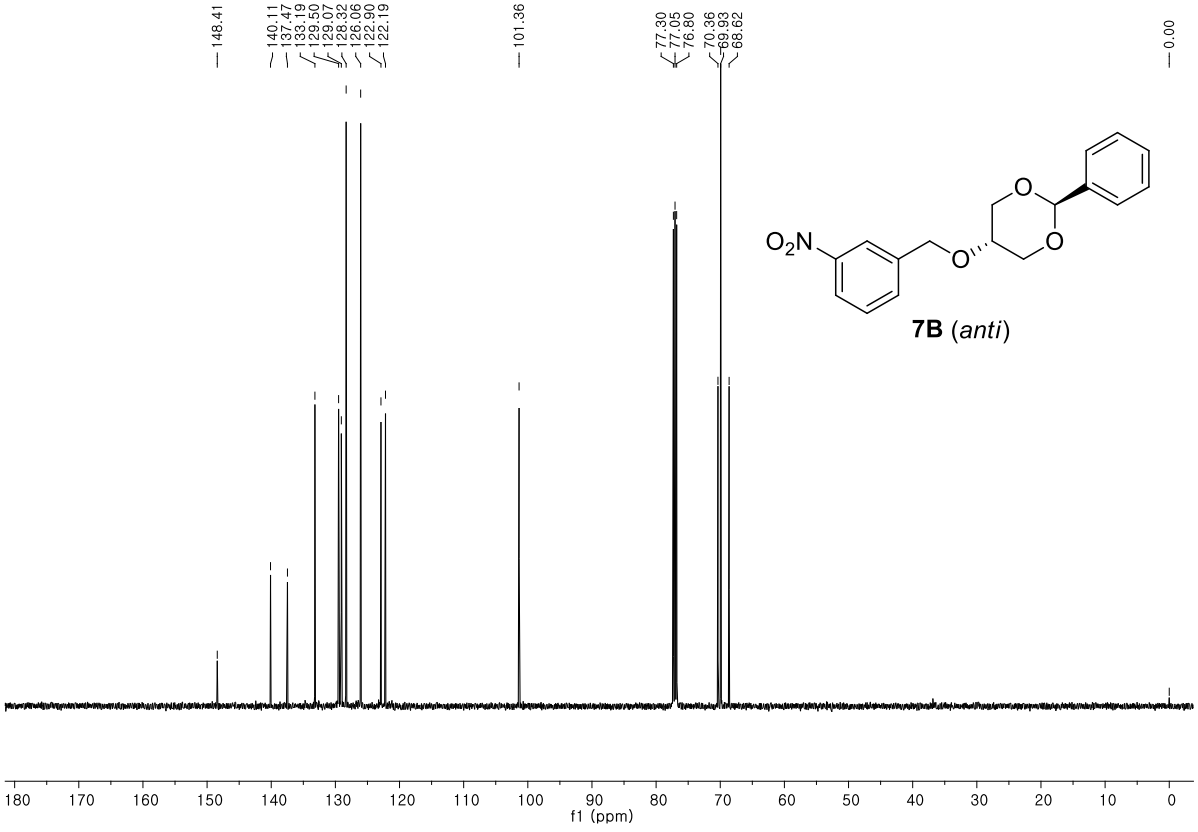

141.81
130.47
130.21
129.96
129.70
127.66
127.35
125.52
125.49
125.46
125.43
125.18
123.02
120.86
77.30
77.04
76.79
72.76
72.09
70.69
63.97
0.00
OH
O
OH
F3C
1'f
130.47
130.21
129.96
129.70
131.0
130.5
130.0
129.5
129.0
f1 (ppm)
127.66
127.35
125.52
125.49
125.46
125.43
125.18
123.02
120.86
127
126
125
124
123
122
121
120
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

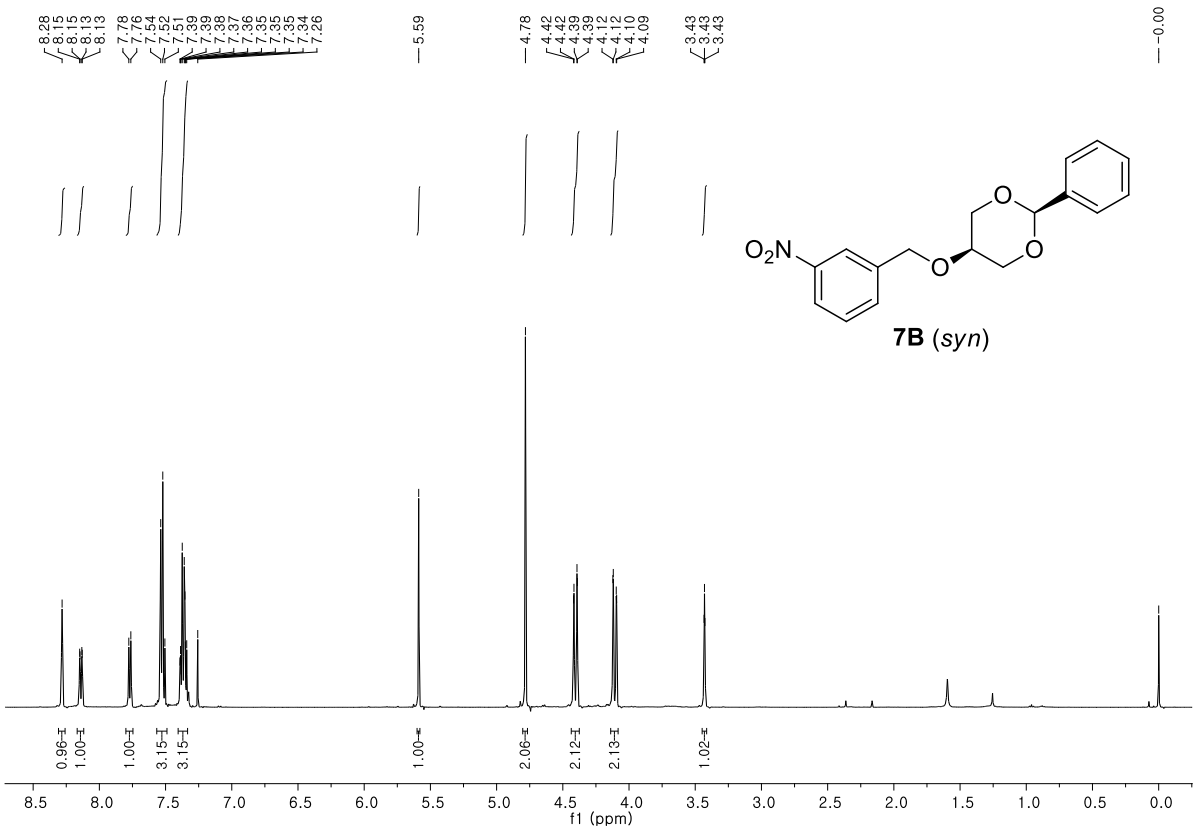

-62.56
OH
O
OH
F3C
1'f
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

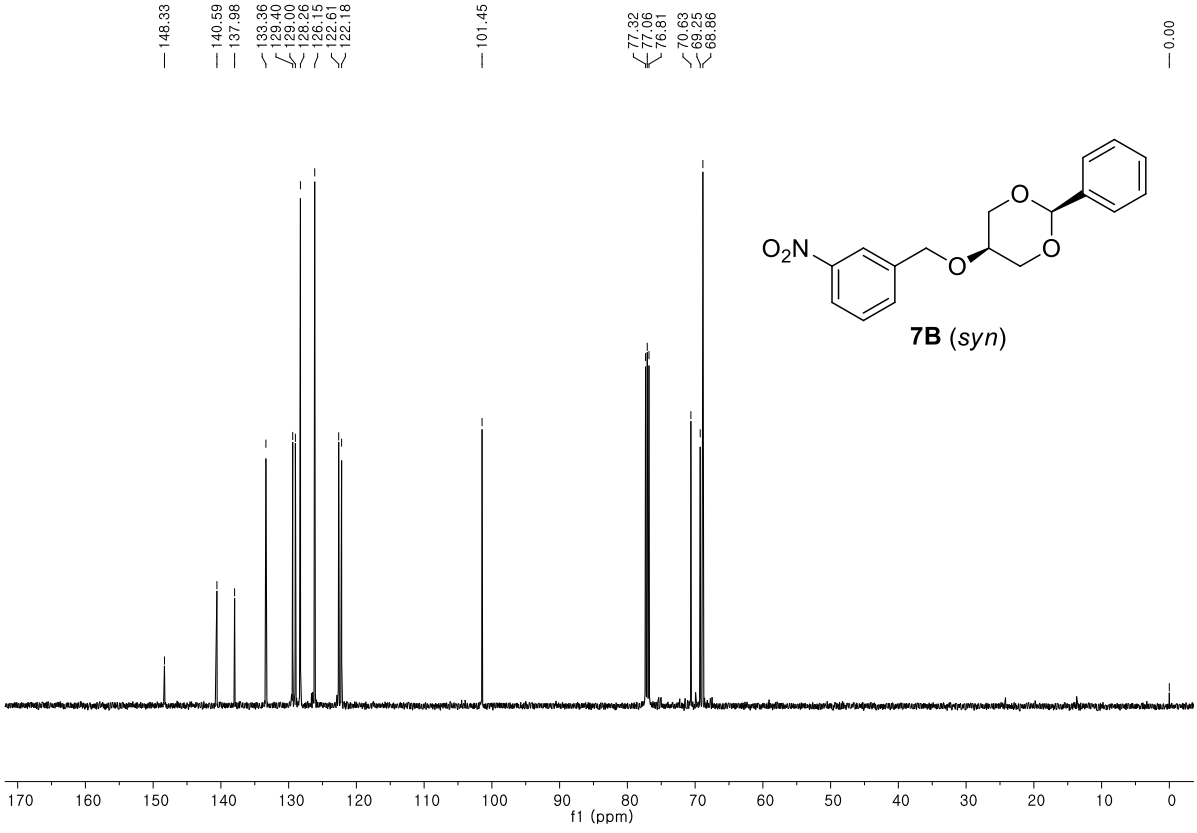

8.22
8.20
8.15
8.14
8.14
8.13
8.12
7.69
7.67
7.66
7.64
7.53
7.52
7.51
7.50
7.49
7.49
7.48
7.48
7.38
7.37
7.36
7.36
5.98
5.96
5.82
4.70
4.70
4.69
4.67
4.51
4.49
4.49
4.49
4.48
4.48
4.47
4.47
4.46
4.45
4.44
4.28
4.27
4.26
4.25
4.14
4.13
4.11
4.01
4.00
4.00
3.99
3.89
3.87
3.87
3.74
3.73
3.72
3.71
3.71
3.70
3.69
3.69
3.68
3.67
3.66
3.65
3.64
0.00
O2N
O
O
O
7A
5.81
3.07
17.15
1.00
1.70
6.07
2.81
1.11
1.83
1.80
1.10
5.93
f1 (ppm)

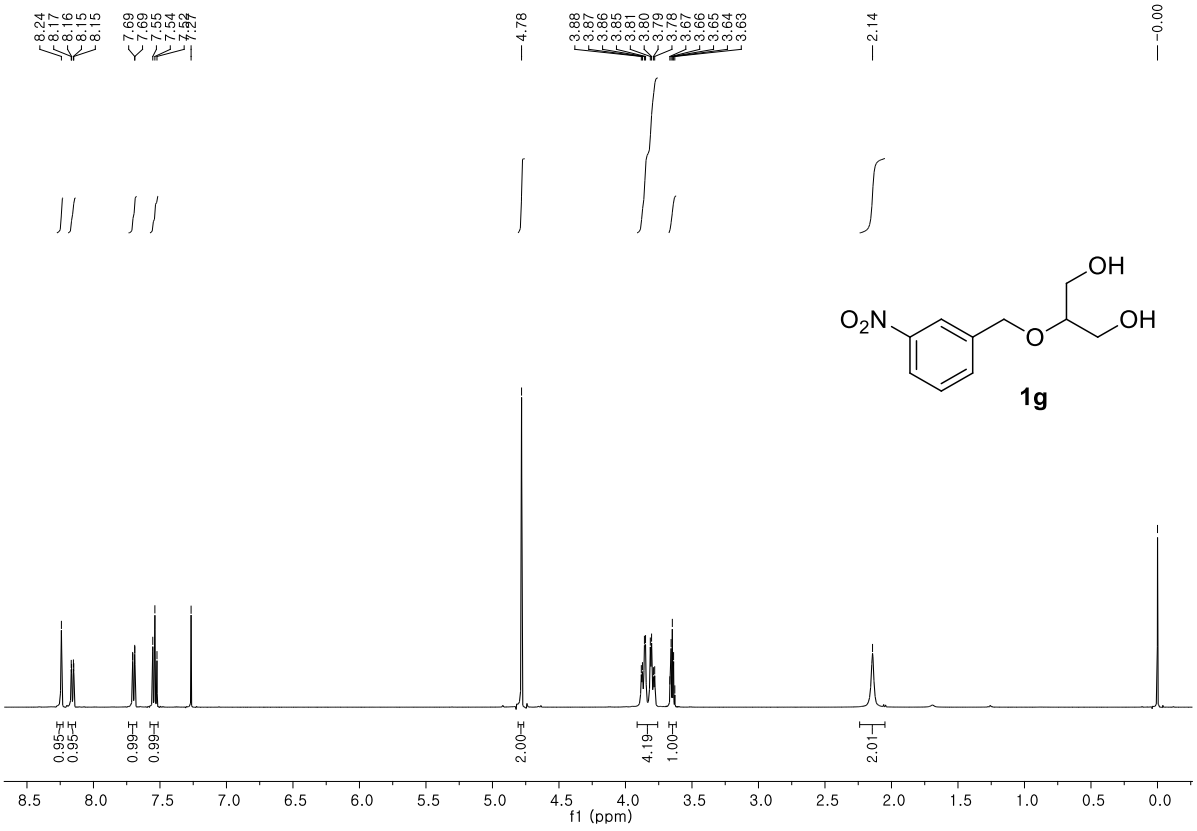

148.37
140.27
137.80
137.23
133.38
133.32
129.46
129.42
129.28
128.39
126.66
126.47
122.70
122.69
122.26
122.23
104.48
103.97
77.37
77.12
76.86
75.39
75.05
72.28
72.25
71.49
71.13
67.76
67.52
O2N
O
O
O
7A
f1 (ppm)

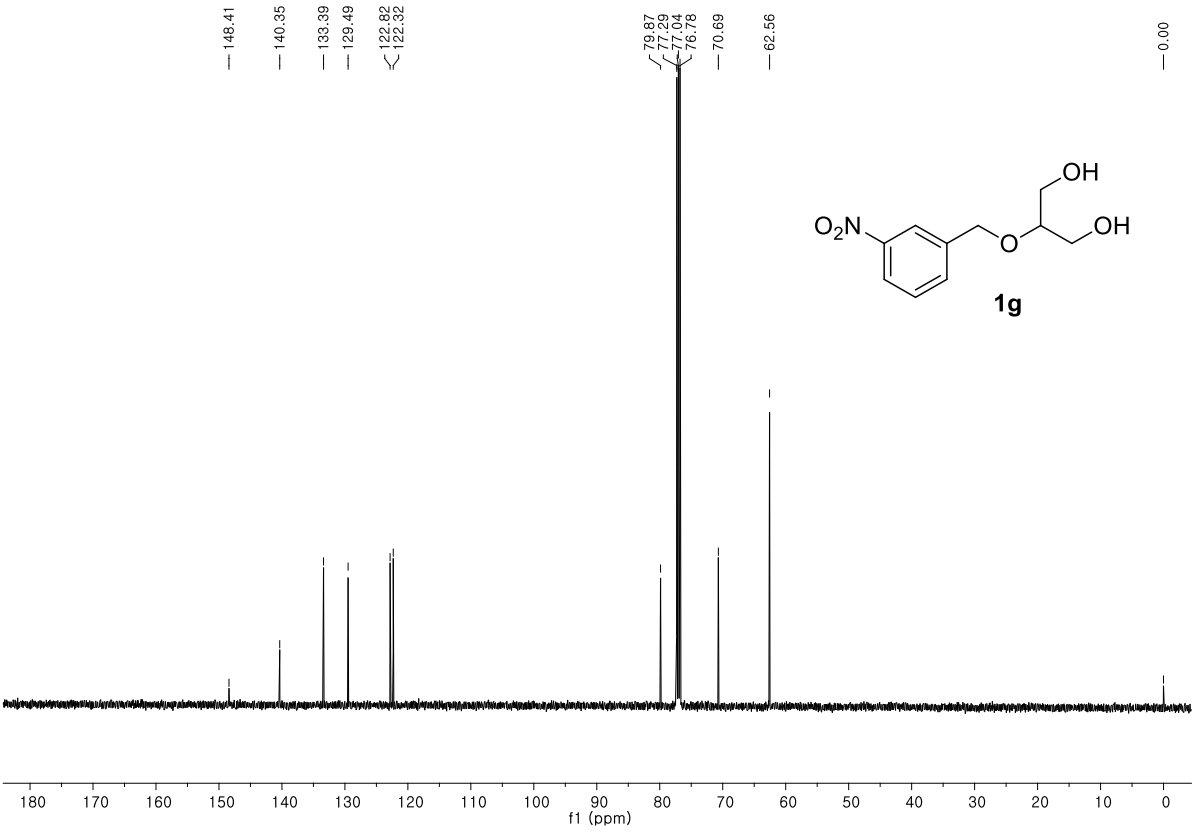

8.21
8.17
8.16
7.66
7.64
7.55
7.53
7.52
7.49
7.47
7.47
7.46
7.46
7.45
7.38
7.37
7.37
7.36
7.35
7.34
7.25
5.43
4.69
4.43
4.42
4.41
4.40
3.83
3.82
3.81
3.72
3.70
3.68
0.00
1.96
1.01
6.42
1.00
2.07
2.10
1.04
2.14
f1 (ppm)
O2N
O
O
O
7B (anti)

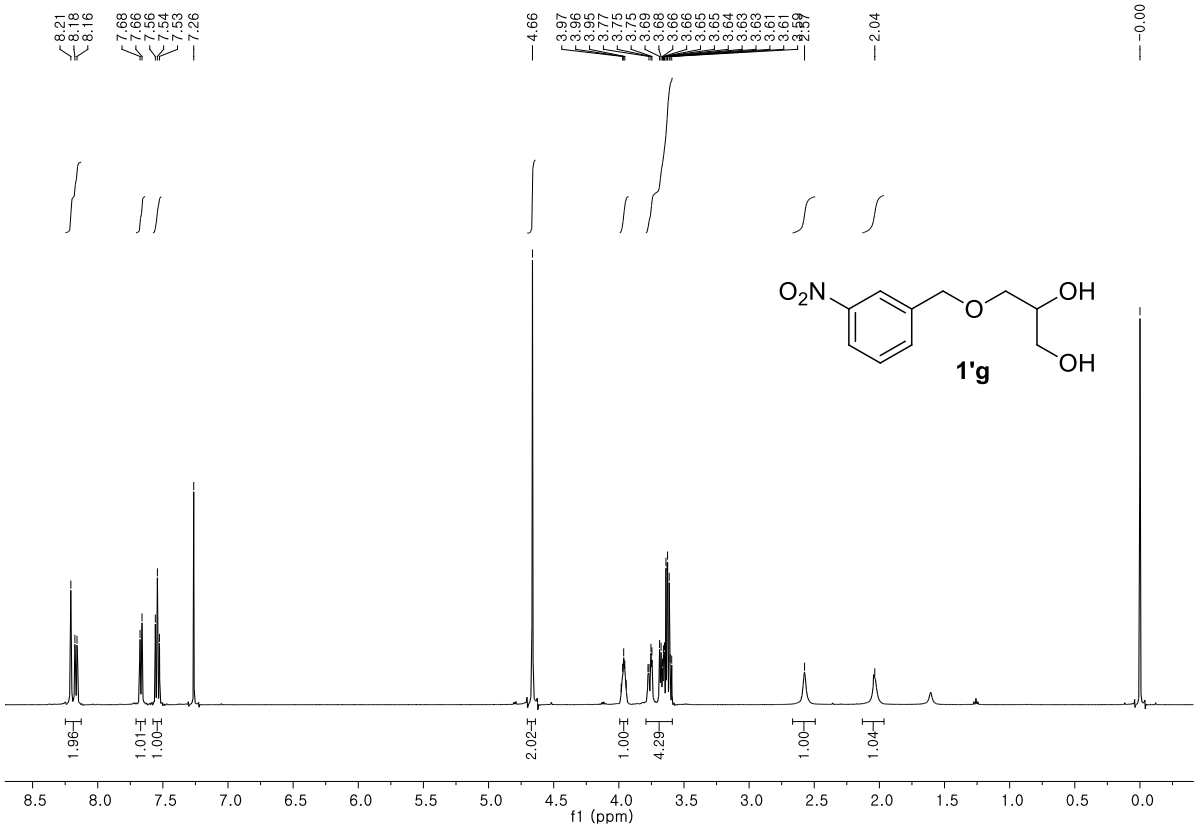

148.41
140.11
137.47
133.19
129.50
129.07
128.32
126.06
122.90
122.19
101.36
77.30
77.05
76.80
70.36
69.93
68.62
0.00
f1 (ppm)
O2N
O
O
O
7B (anti)

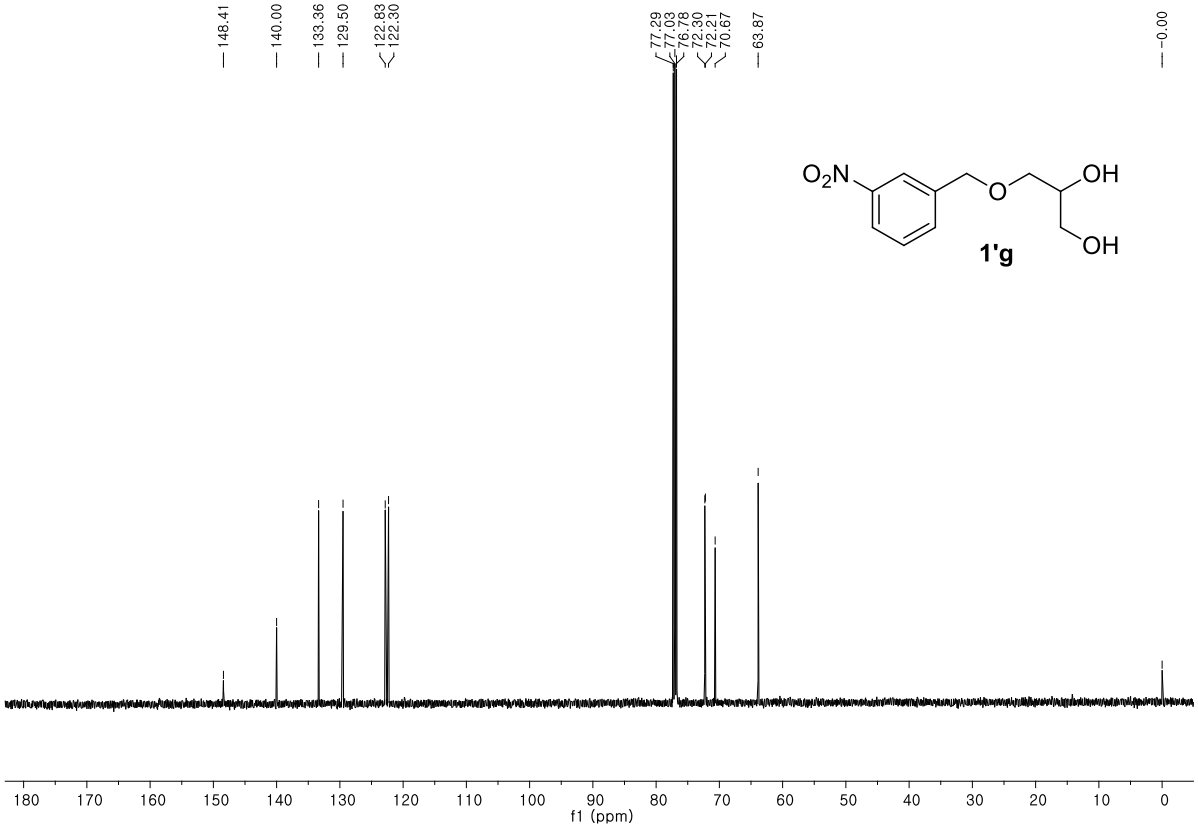

8.28
8.15
8.15
8.13
8.13
7.78
7.76
7.54
7.52
7.51
7.39
7.38
7.37
7.36
7.35
7.35
7.34
7.26
5.59
4.78
4.42
4.42
4.39
4.39
4.12
4.12
4.10
4.09
3.43
3.43
3.43
0.00
O2N
O
O
O
7B (syn)
0.96
1.00
1.00
3.15
3.15
1.00
2.06
2.12
2.13
1.02
f1 (ppm)

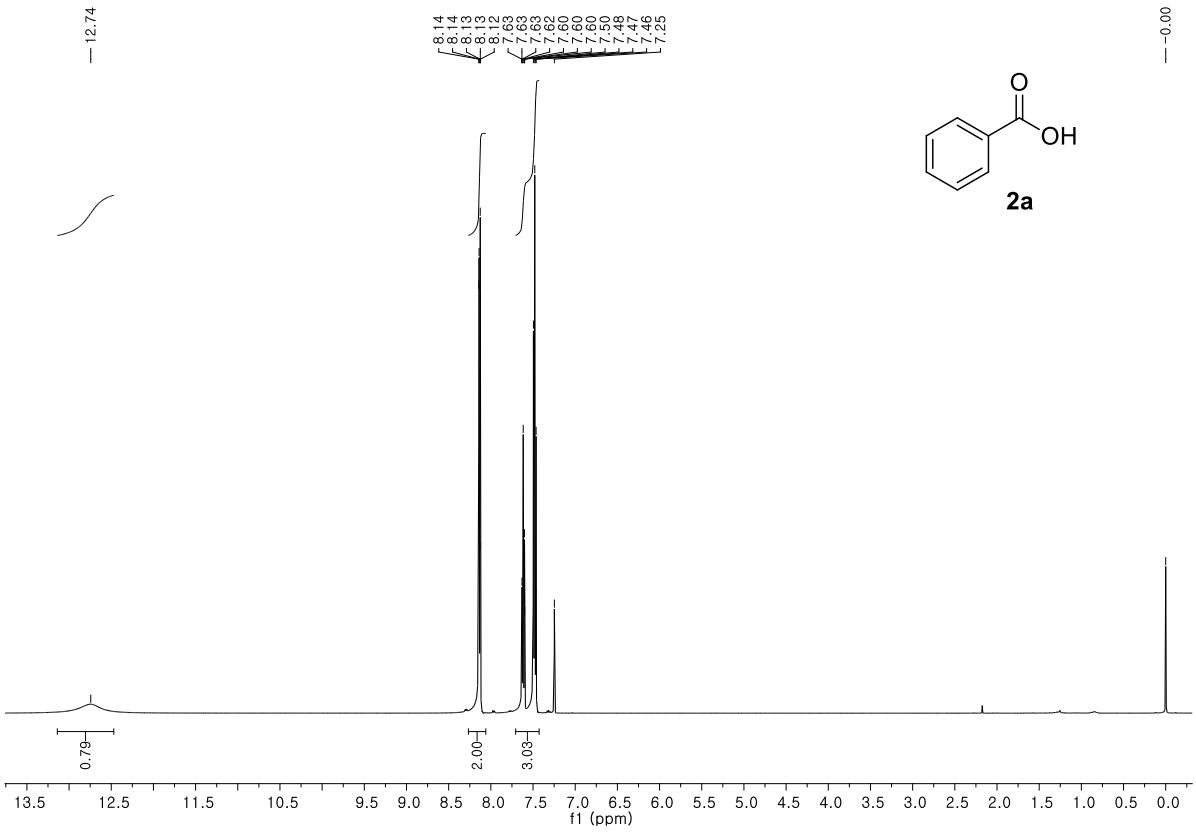

148.33
140.59
137.98
133.36
129.40
129.00
128.26
126.15
122.61
122.18
101.45
77.32
77.06
76.81
70.63
69.25
68.86
0.00
O2N
O
O
O
7B (syn)
f1 (ppm)

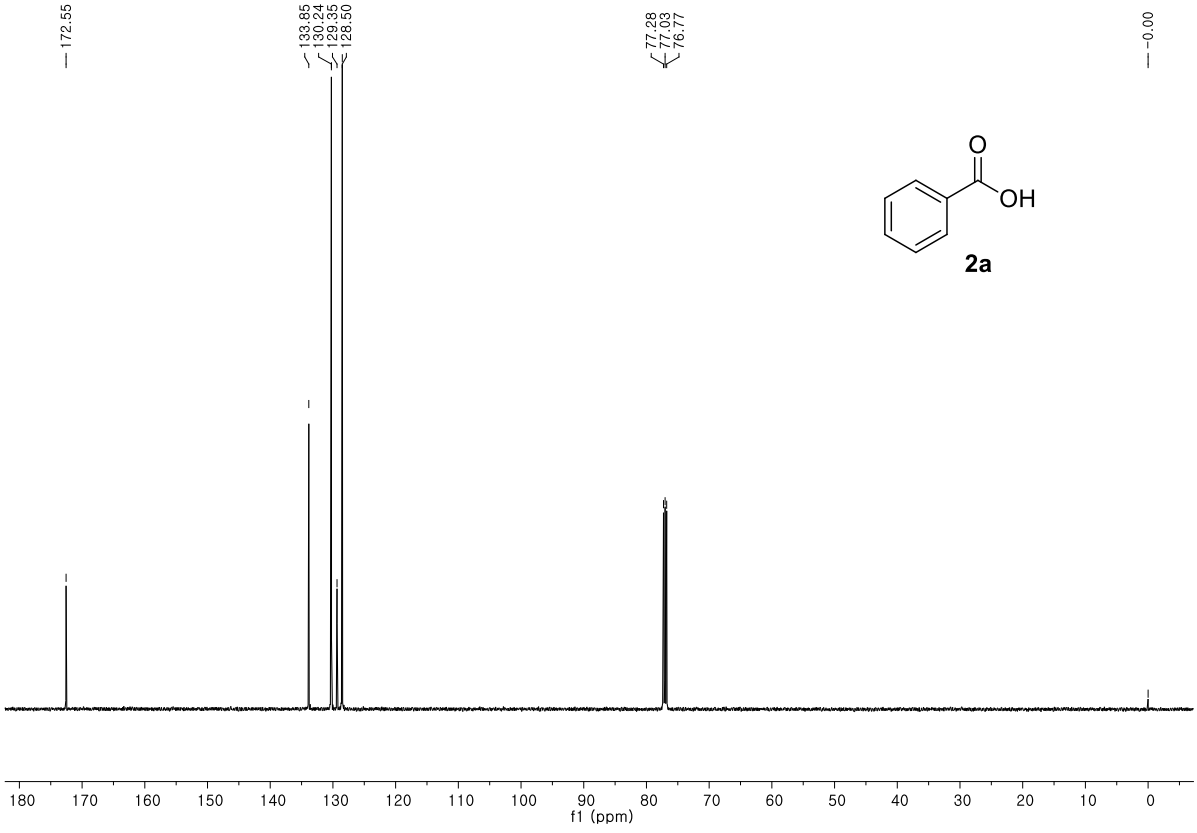

8.24
8.17
8.16
8.15
8.15
7.69
7.69
7.55
7.54
7.27
4.78
3.88
3.87
3.86
3.85
3.81
3.80
3.79
3.78
3.67
3.66
3.65
3.64
3.63
2.14
0.00
OH
O2N
O
OH
1g
0.95
0.95
0.99
0.99
2.00
4.19
1.00
2.01
f1 (ppm)


148.41
140.35
133.39
129.49
122.82
122.32
79.87
77.29
77.04
76.78
70.69
62.56
0.00
OH
O2N
O
OH
1g
f1 (ppm)


8.21
8.18
8.16
7.68
7.66
7.56
7.54
7.53
7.28
4.66
3.97
3.96
3.95
3.77
3.75
3.75
3.69
3.68
3.66
3.66
3.65
3.65
3.64
3.63
3.63
3.61
3.61
2.04
0.00
O2N
O
OH
OH
1'g
1.96
1.01
1.00
2.02
1.00
4.29
1.00
1.04
f1 (ppm)


148.41
140.00
133.36
129.50
122.83
122.30
77.29
77.03
76.78
72.30
72.21
70.67
63.87
0.00
O2N
O
OH
OH
1'g
f1 (ppm)


12.74
8.14
8.14
8.13
8.13
8.12
7.63
7.63
7.63
7.62
7.60
7.60
7.60
7.50
7.48
7.47
7.46
7.25
0.00
O
OH
2a
0.79
2.00
3.03
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


172.55
133.85
130.24
129.35
128.50
77.28
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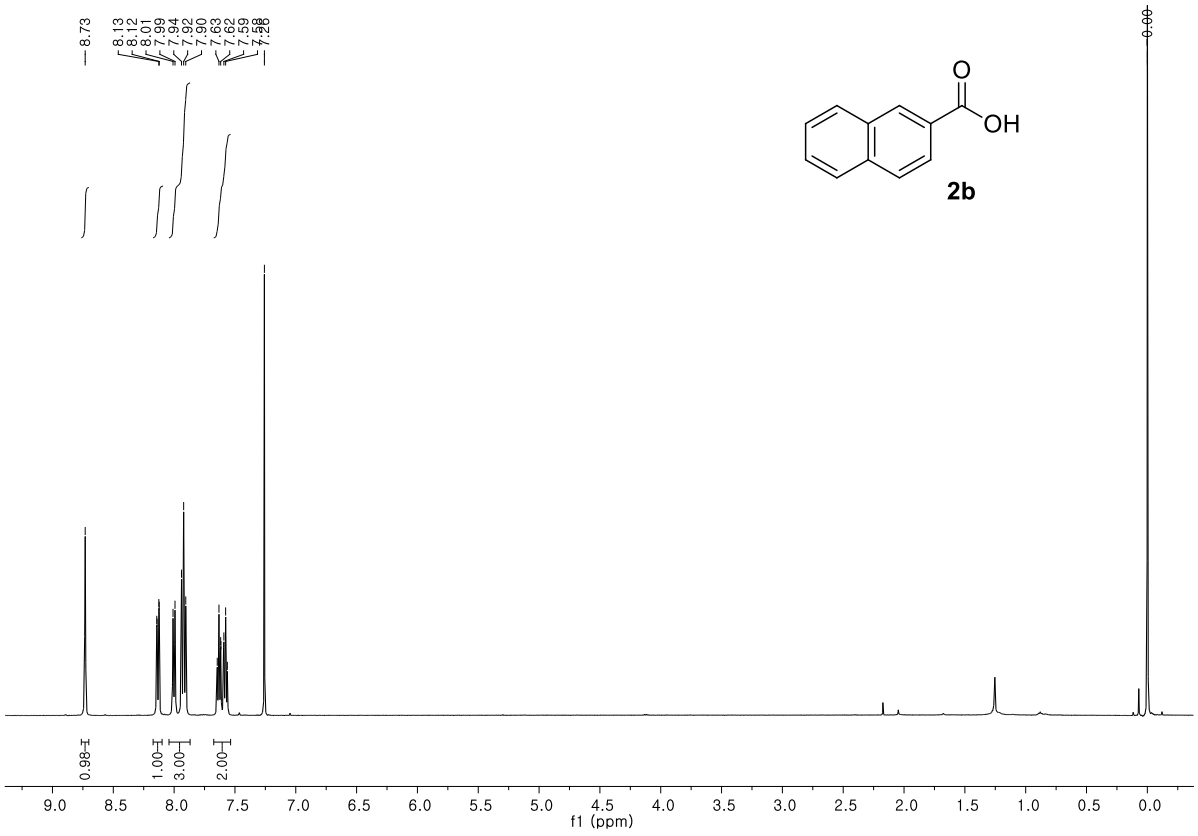
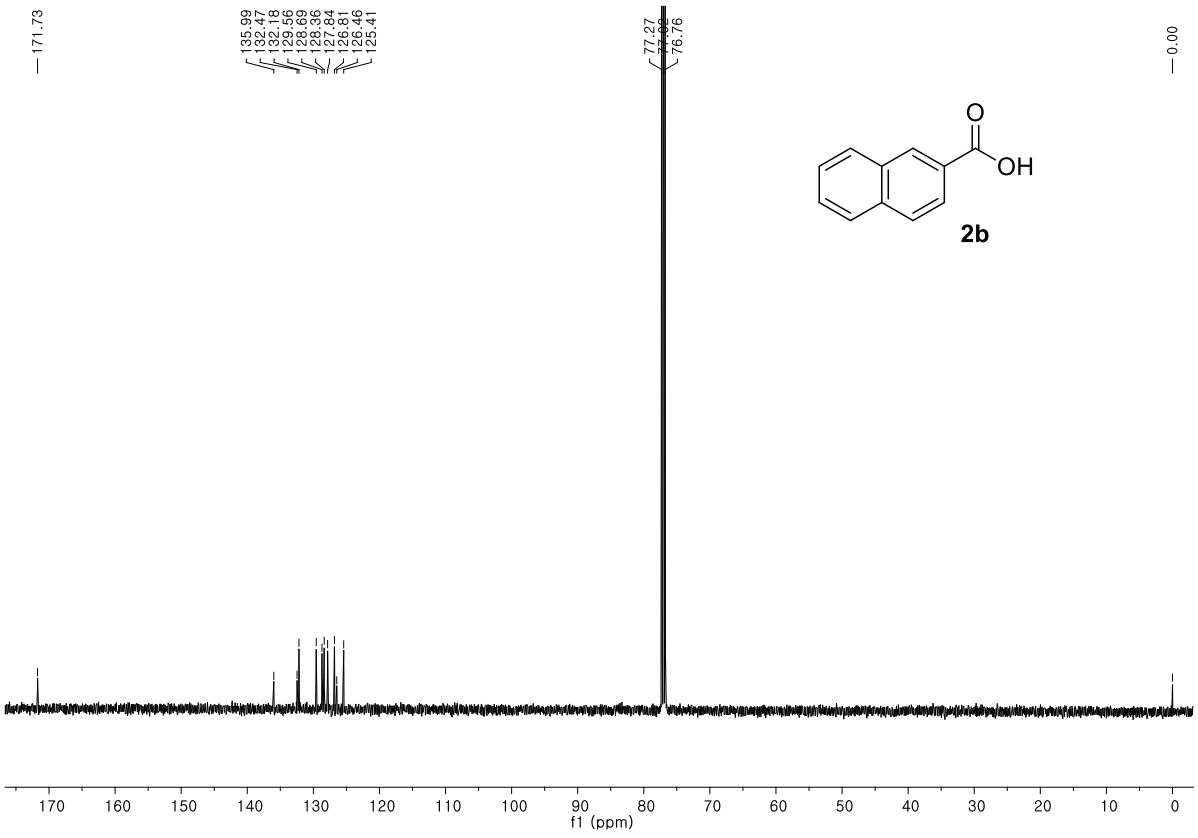
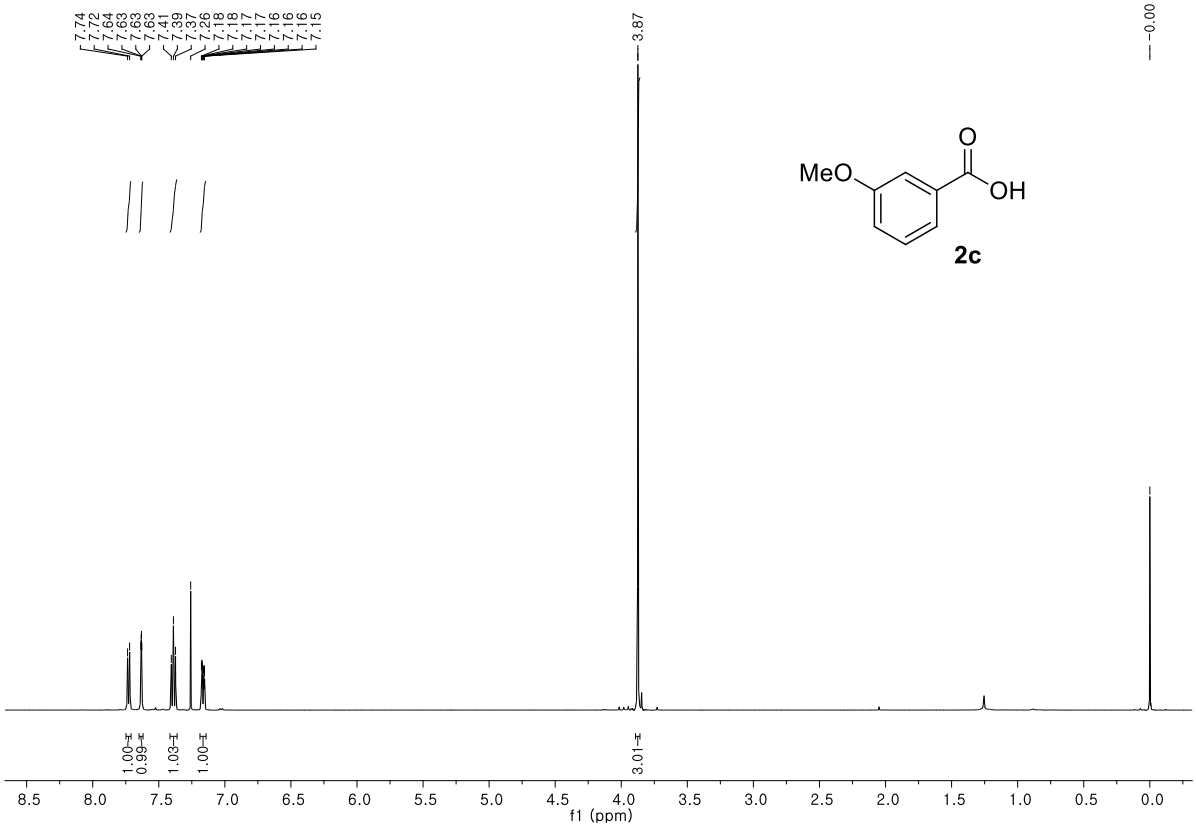
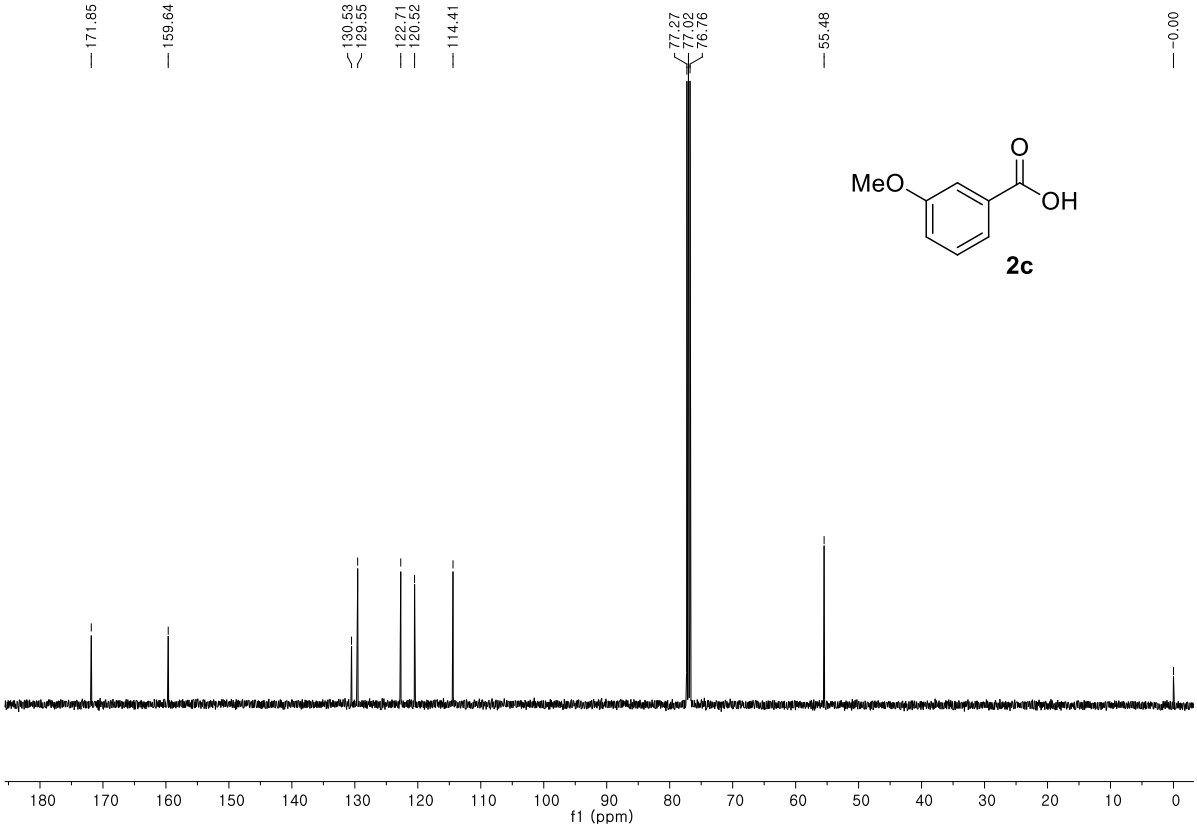
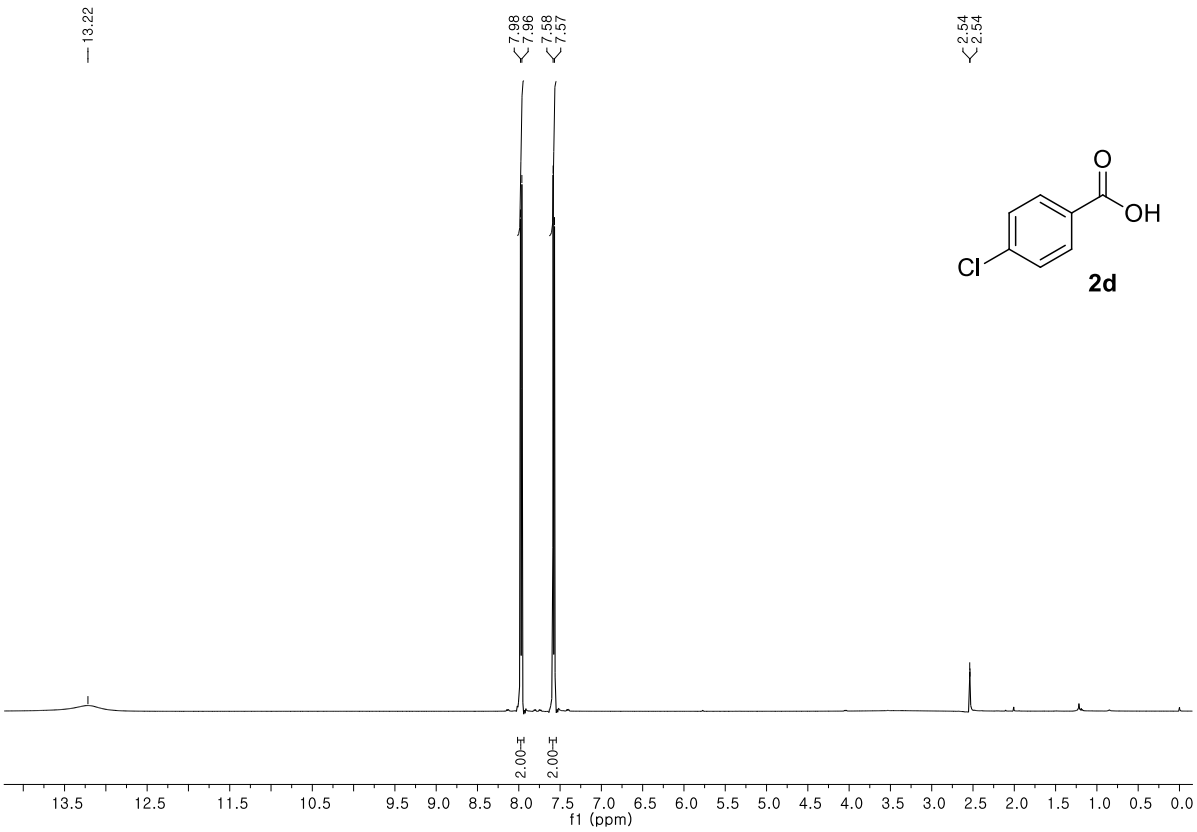
76.77
0.00
O
OH
2a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

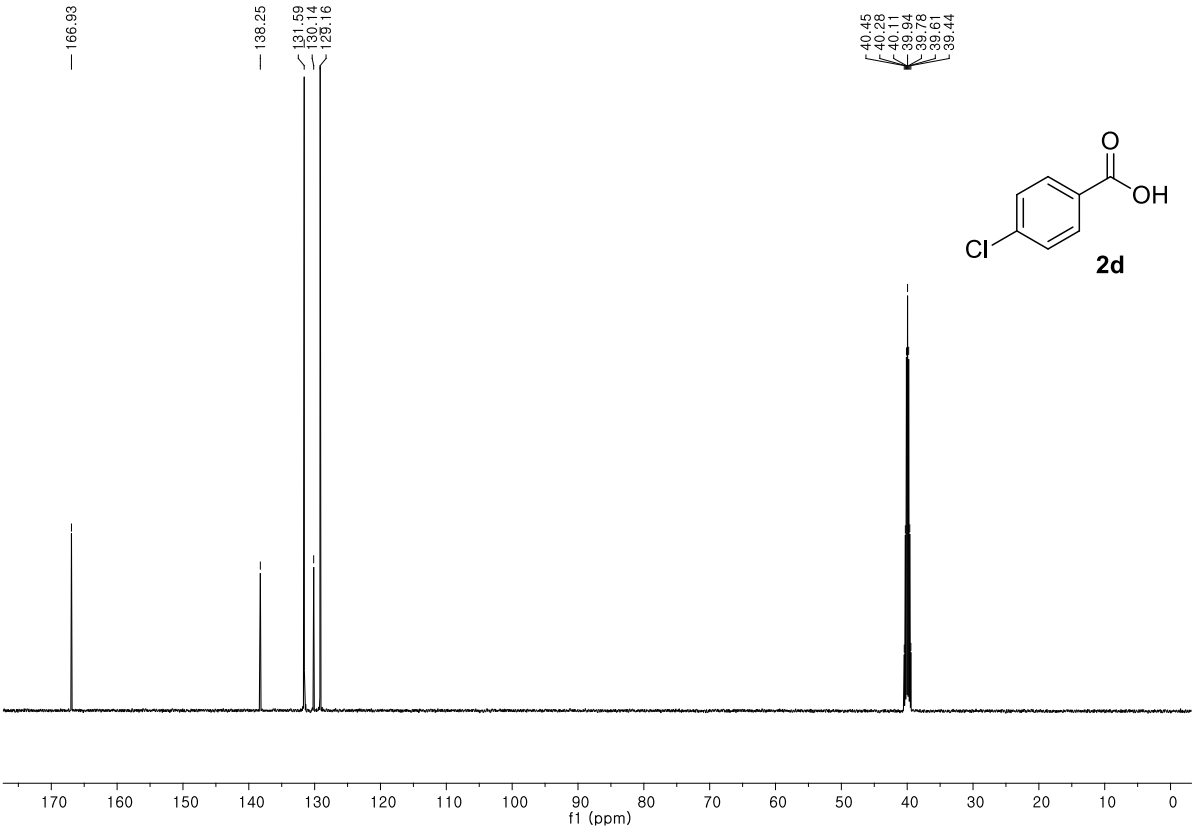

8.73
8.13
8.12
8.01
7.99
7.94
7.92
7.90
7.63
7.62
7.59
7.58
7.28
0.00
O
OH
2b
0.98
1.00
3.00
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

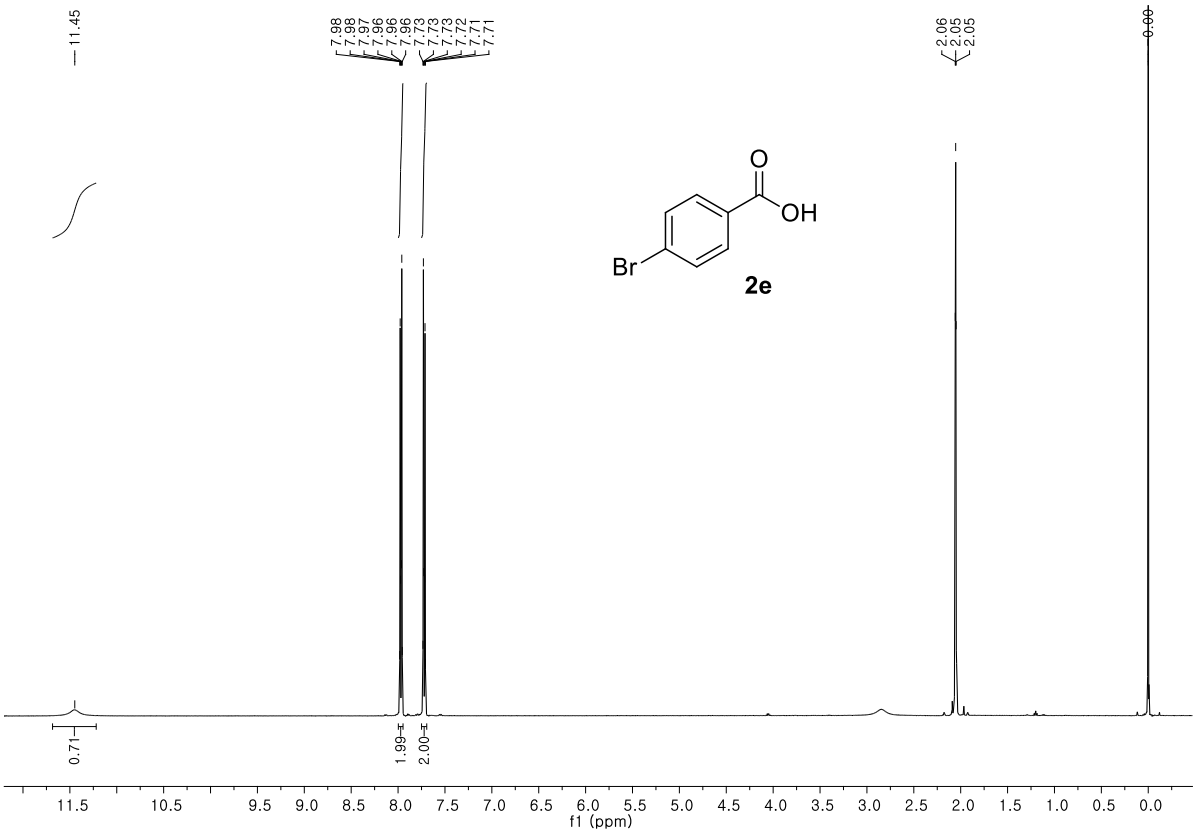

171.73
135.99
132.47
132.18
129.56
128.69
128.36
127.84
126.81
126.46
125.41
77.27
77.02
76.76
0.00
O
OH
2b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

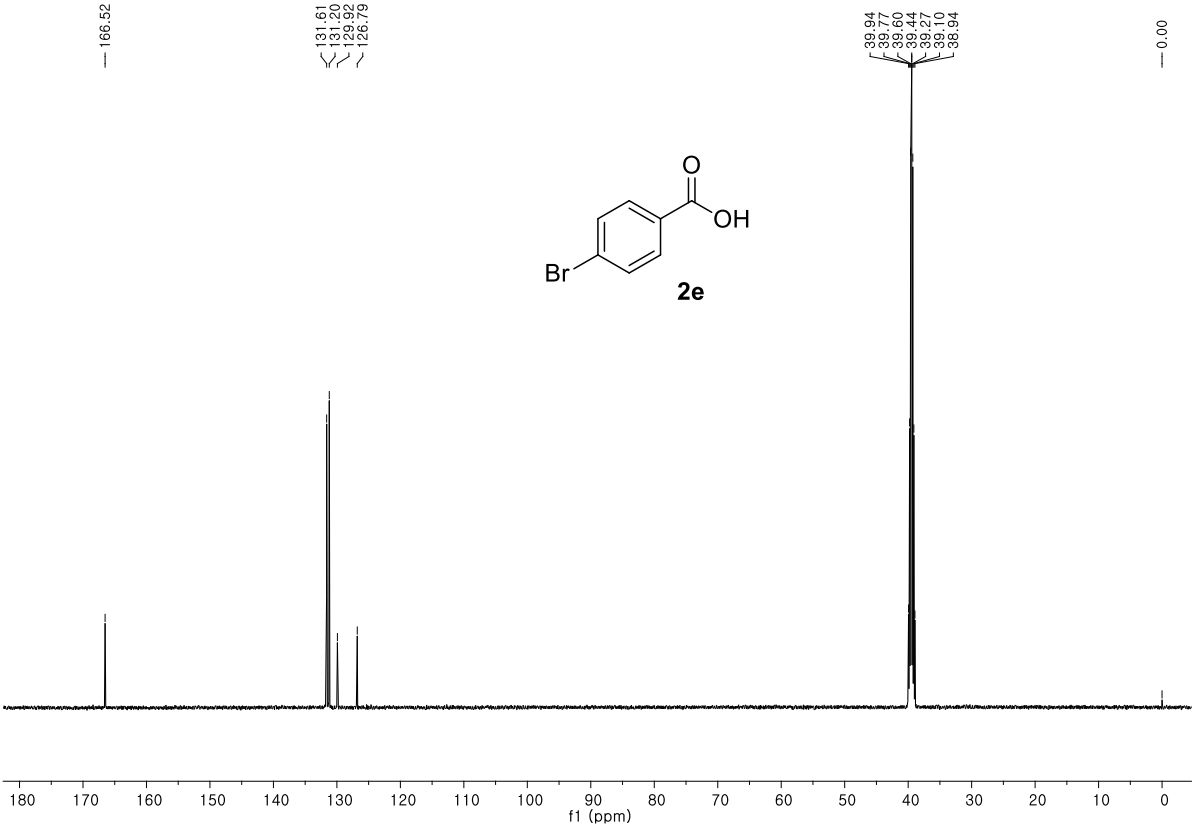

7.74
7.72
7.64
7.63
7.63
7.63
7.41
7.39
7.37
7.26
7.18
7.18
7.17
7.17
7.16
7.16
7.16
7.15
3.87
0.00
MeO
O
OH
2c
1.00
0.99
1.03
1.00
3.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

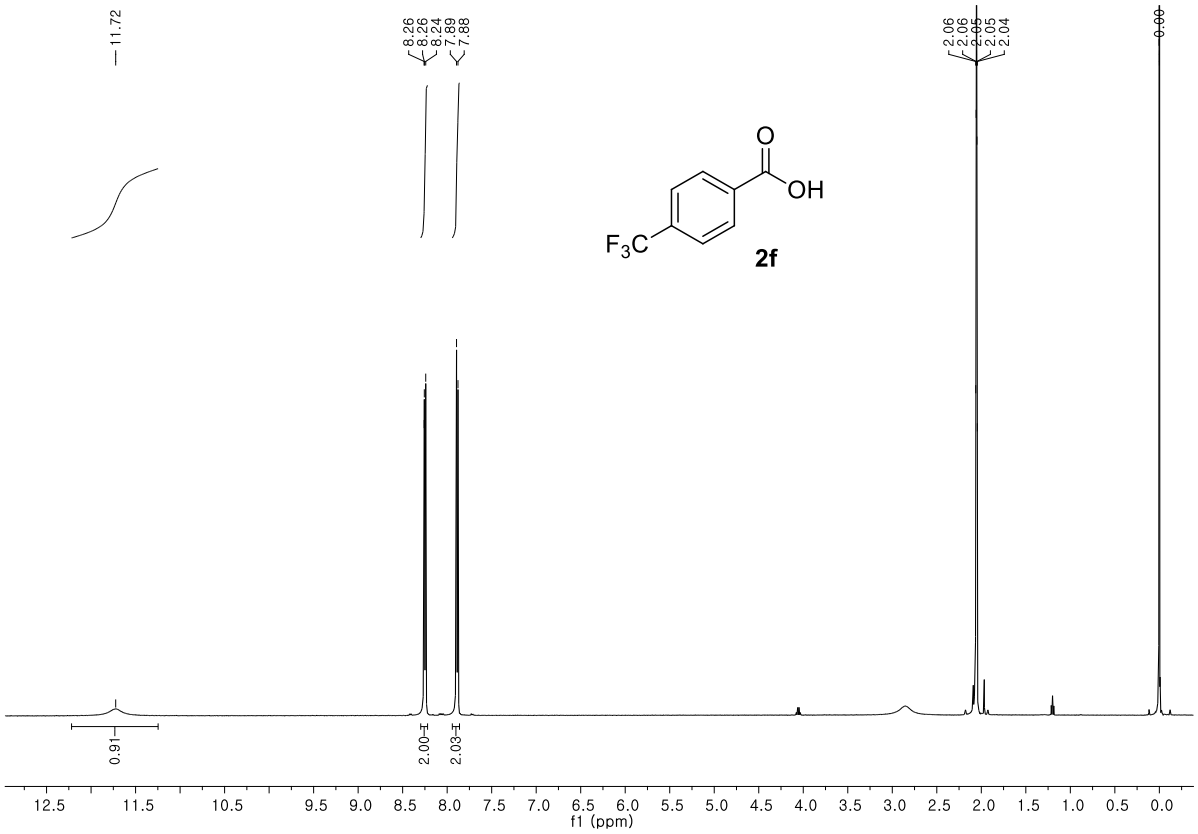

171.85
159.64
130.53
129.55
122.71
120.52
114.41
77.27
77.02
76.76
55.48
0.00
MeO
O
OH
2c
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

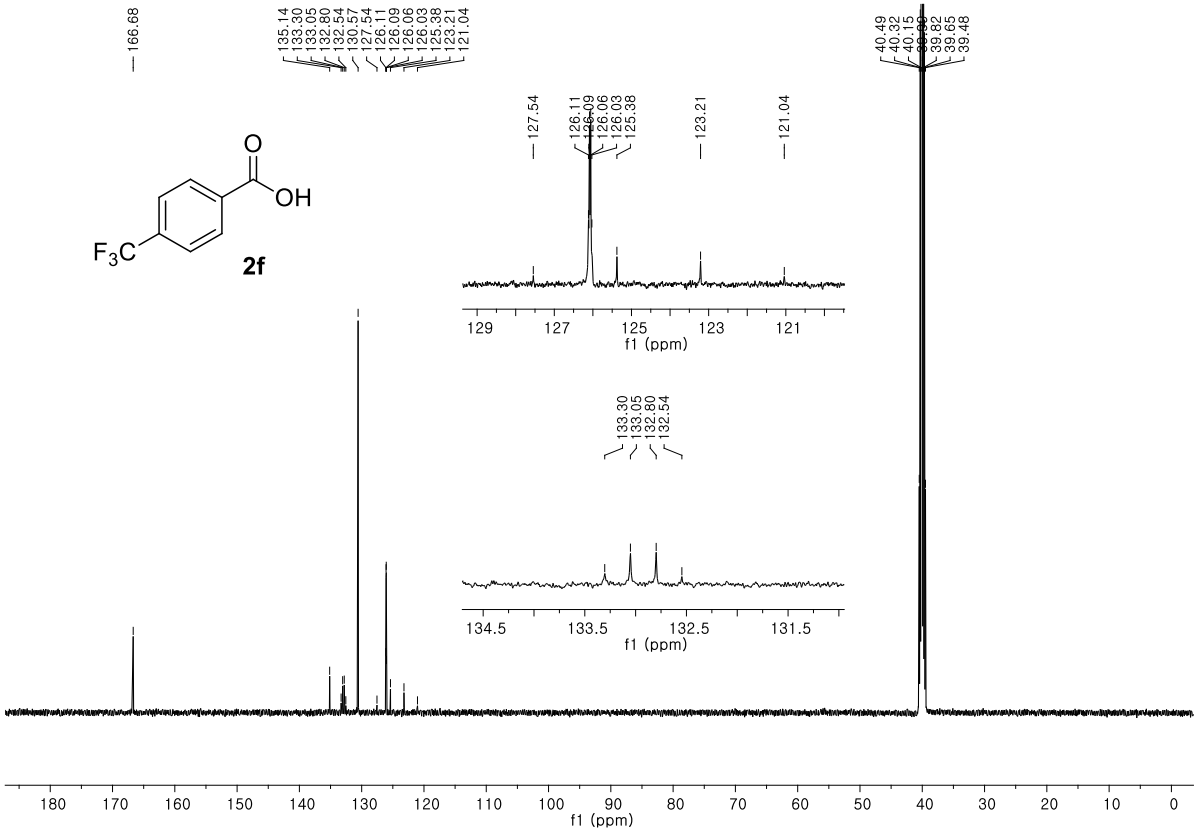

13.22
7.98
7.96
7.58
7.57
2.54
2.54
2.00
2.00
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
O
OH
Cl
2d

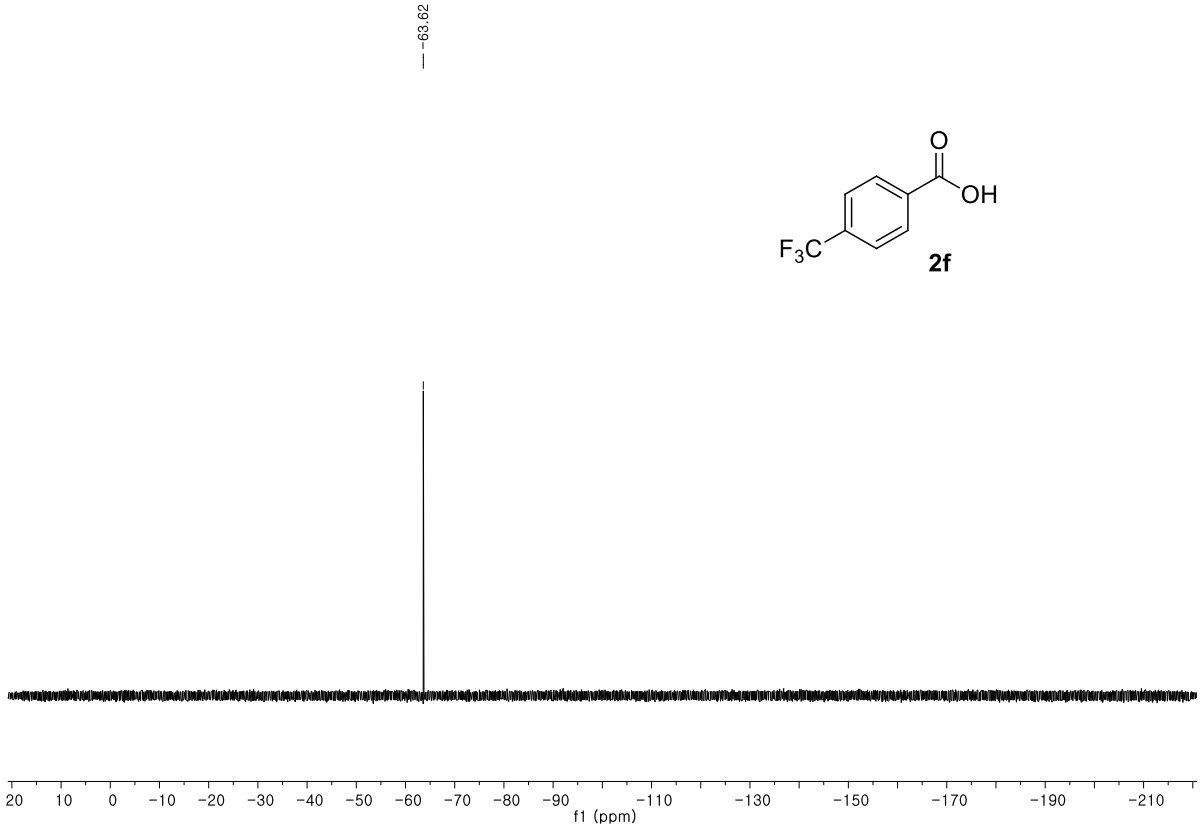

166.93
138.25
131.59
130.14
129.16
40.45
40.28
40.11
39.94
39.78
39.61
39.44
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
O
OH
Cl
2d

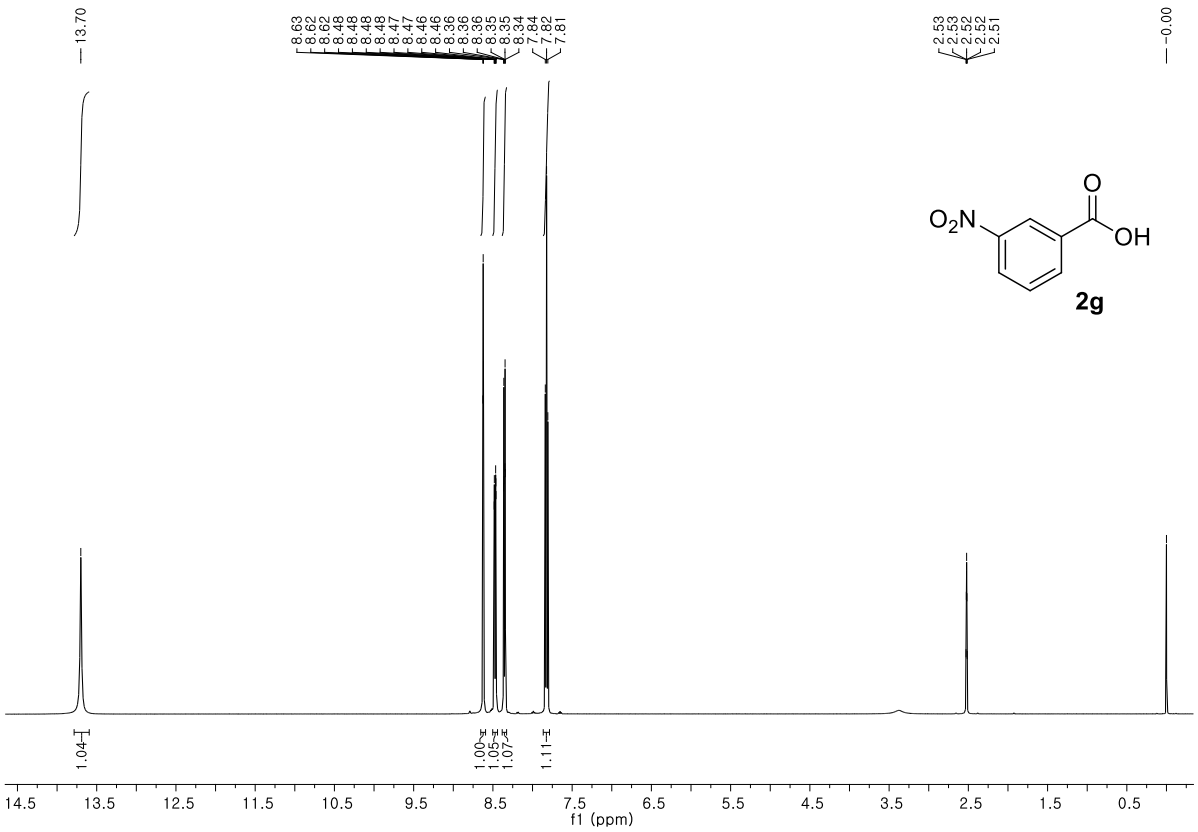

11.45
7.98
7.98
7.97
7.96
7.96
7.96
7.73
7.73
7.73
7.72
7.71
7.71
2.06
2.05
2.05
0.00
O
OH
Br
2e
0.71
1.99
2.00
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

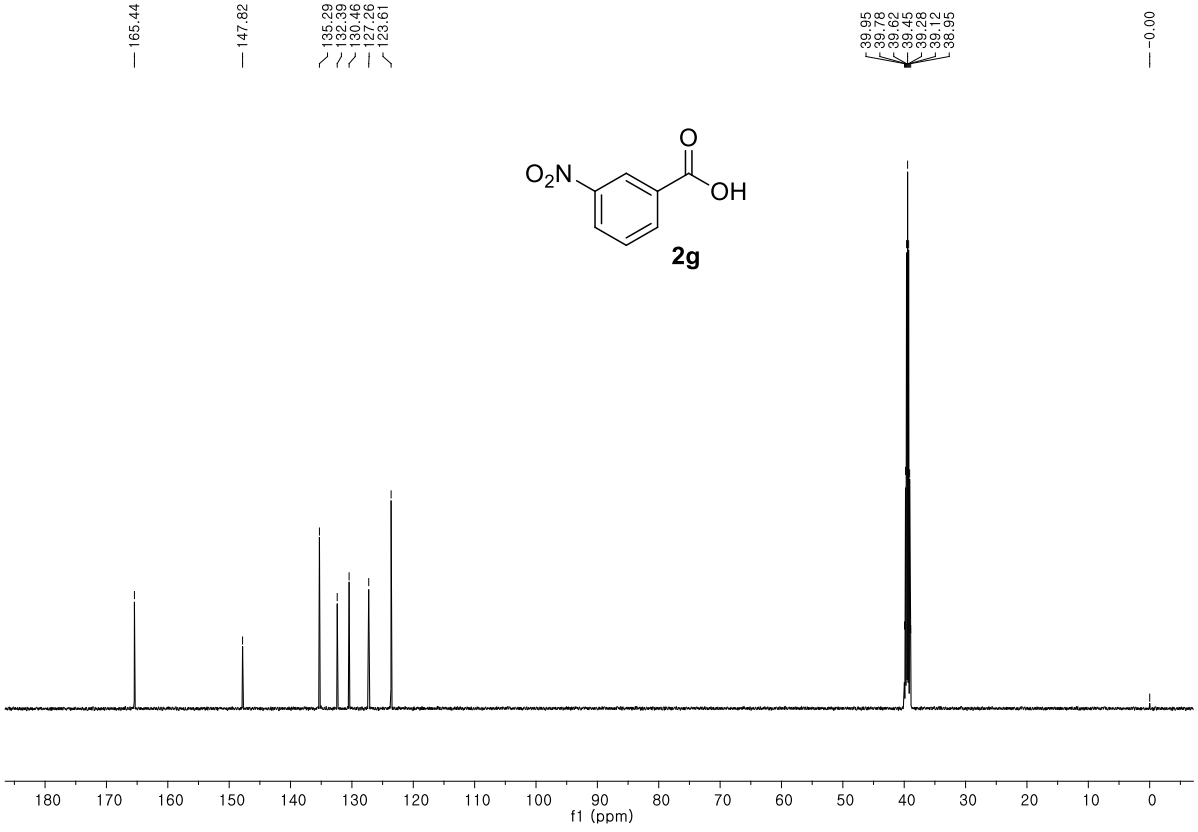

166.52
131.61
131.20
129.92
126.79
39.94
39.77
39.60
39.44
39.27
39.10
38.94
0.00
O
OH
Br
2e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

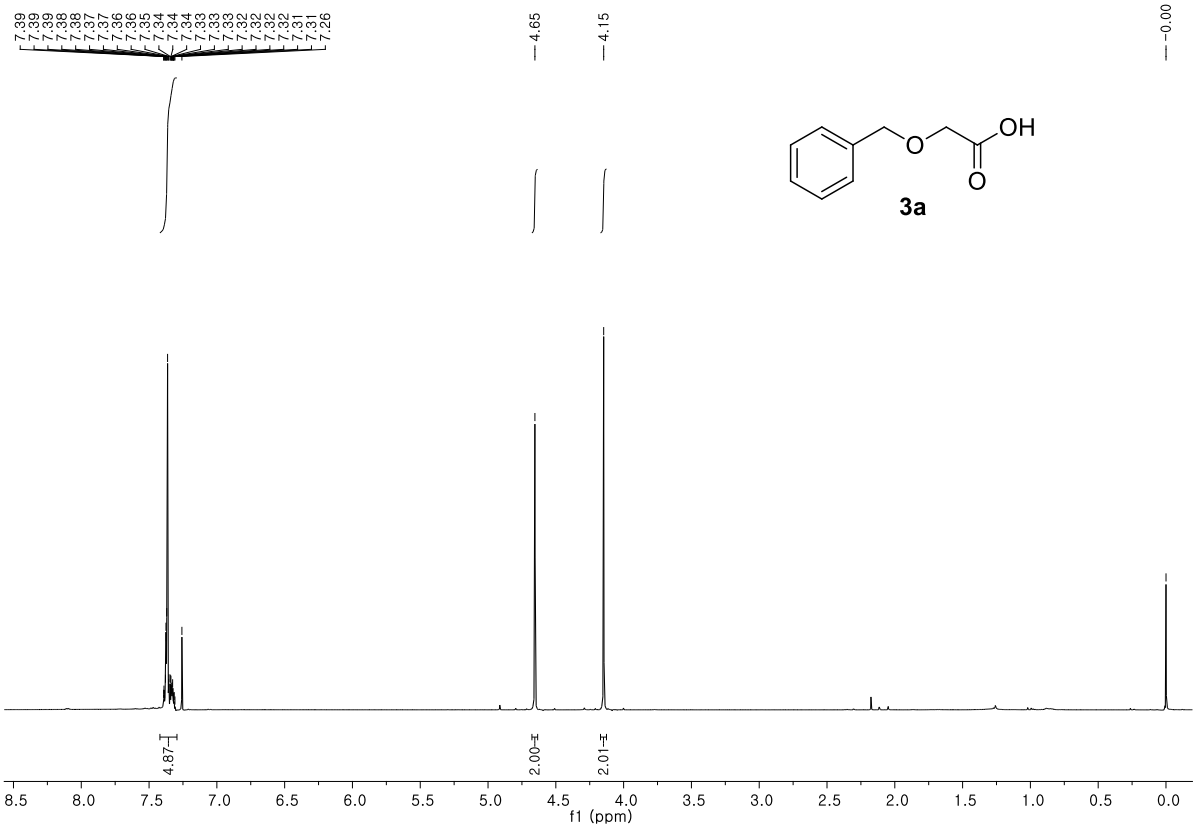

11.72
8.26
8.26
8.24
7.89
7.88
2.06
2.06
2.05
2.05
2.04
0.00
O
OH
F3C
2f
0.91
2.00
2.03
f1 (ppm)

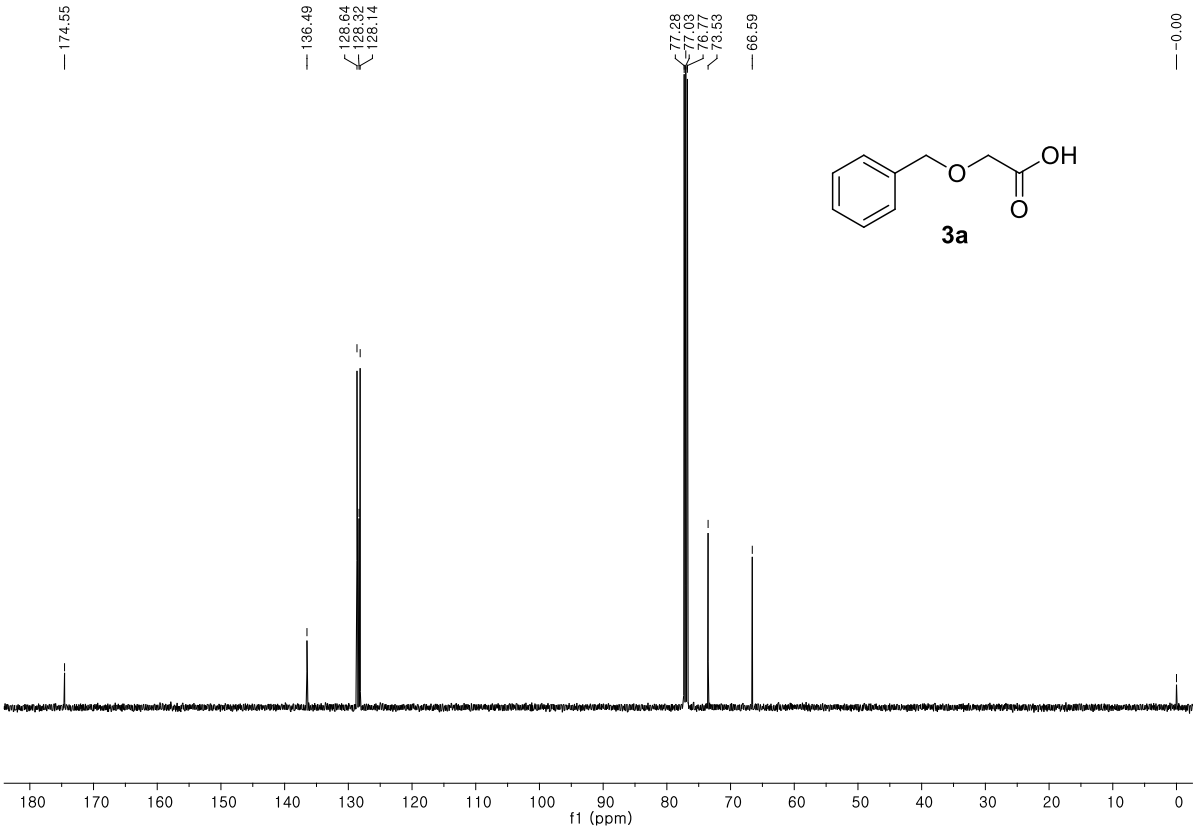

166.68
135.14
133.30
133.05
132.80
132.54
130.57
127.54
126.11
126.09
126.06
126.03
125.38
123.21
121.04
40.49
40.32
40.15
39.98
39.82
39.65
39.48
O
OH
F3C
2f
f1 (ppm)

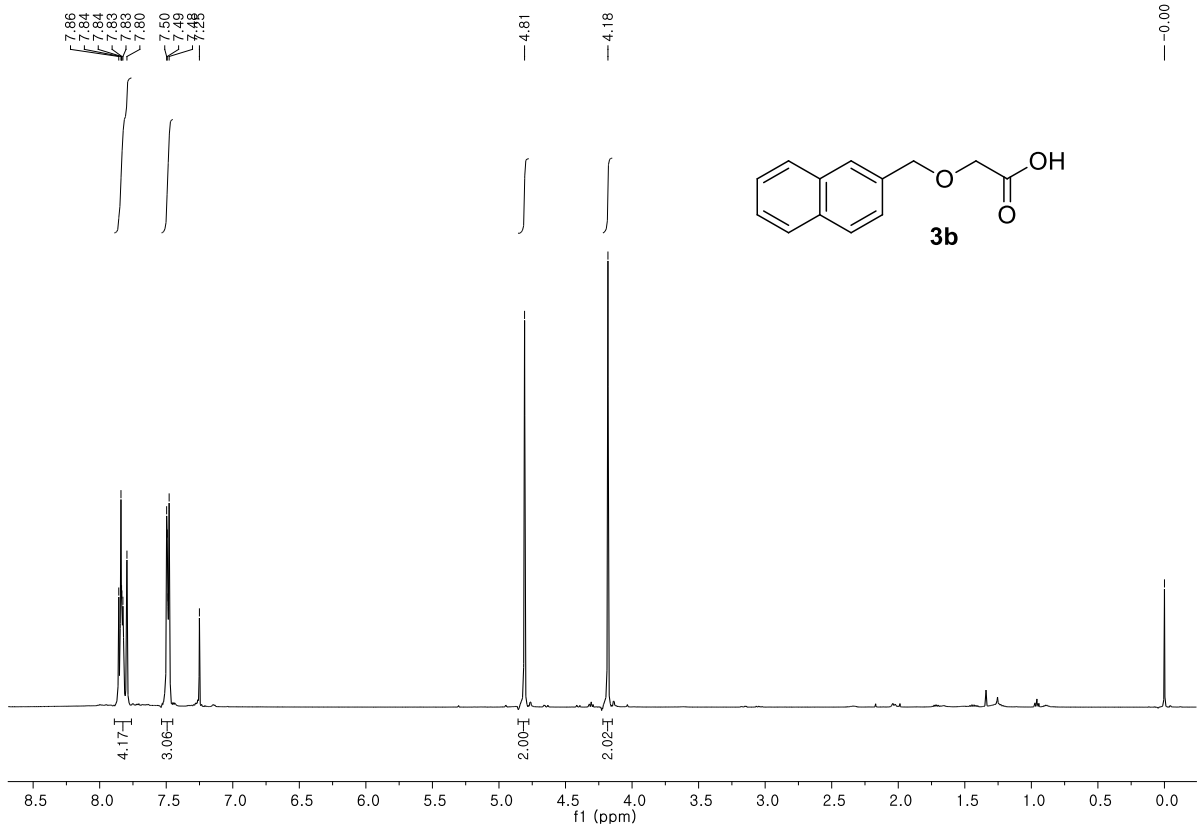

−63.62
O
OH
F3C
2f
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−110
−130
−150
−170
−190
−210
f1 (ppm)

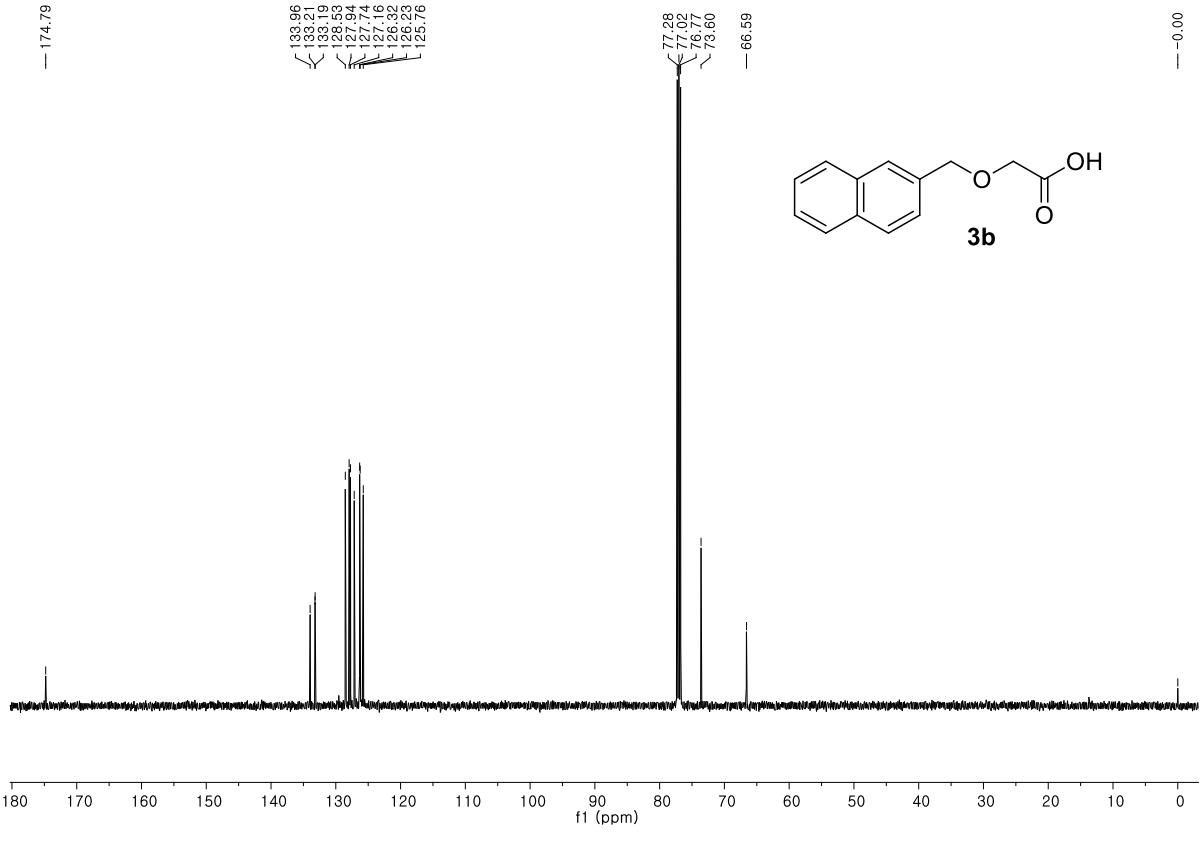

13.70
8.63
8.62
8.62
8.48
8.48
8.48
8.48
8.47
8.47
8.46
8.46
8.36
8.36
8.36
8.35
8.35
8.34
7.84
7.82
7.81
2.53
2.53
2.52
2.52
2.51
0.00
O
O2N
OH
2g
1.04
1.00
1.05
1.07
1.11
14.5
13.5
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

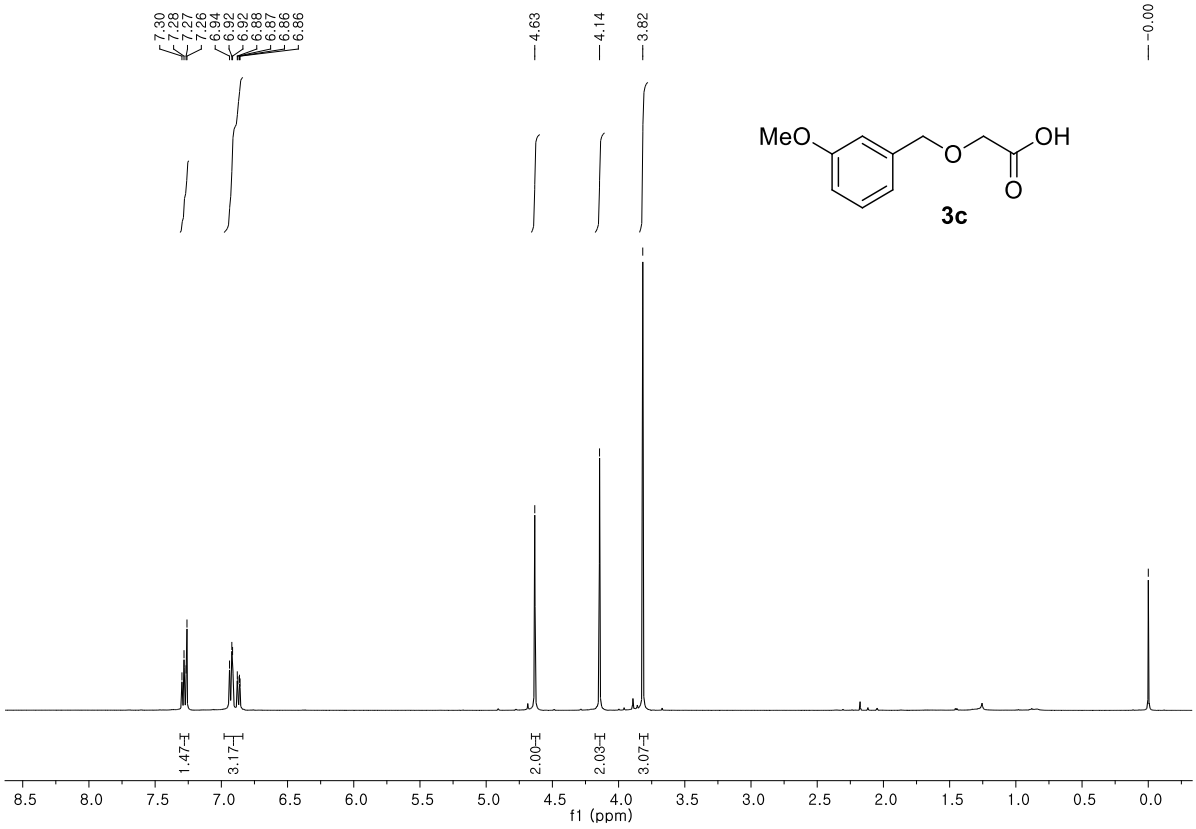

165.44
147.82
135.29
132.39
130.46
127.26
123.61
39.95
39.78
39.62
39.45
39.28
39.12
38.95
0.00
O
O2N
OH
2g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

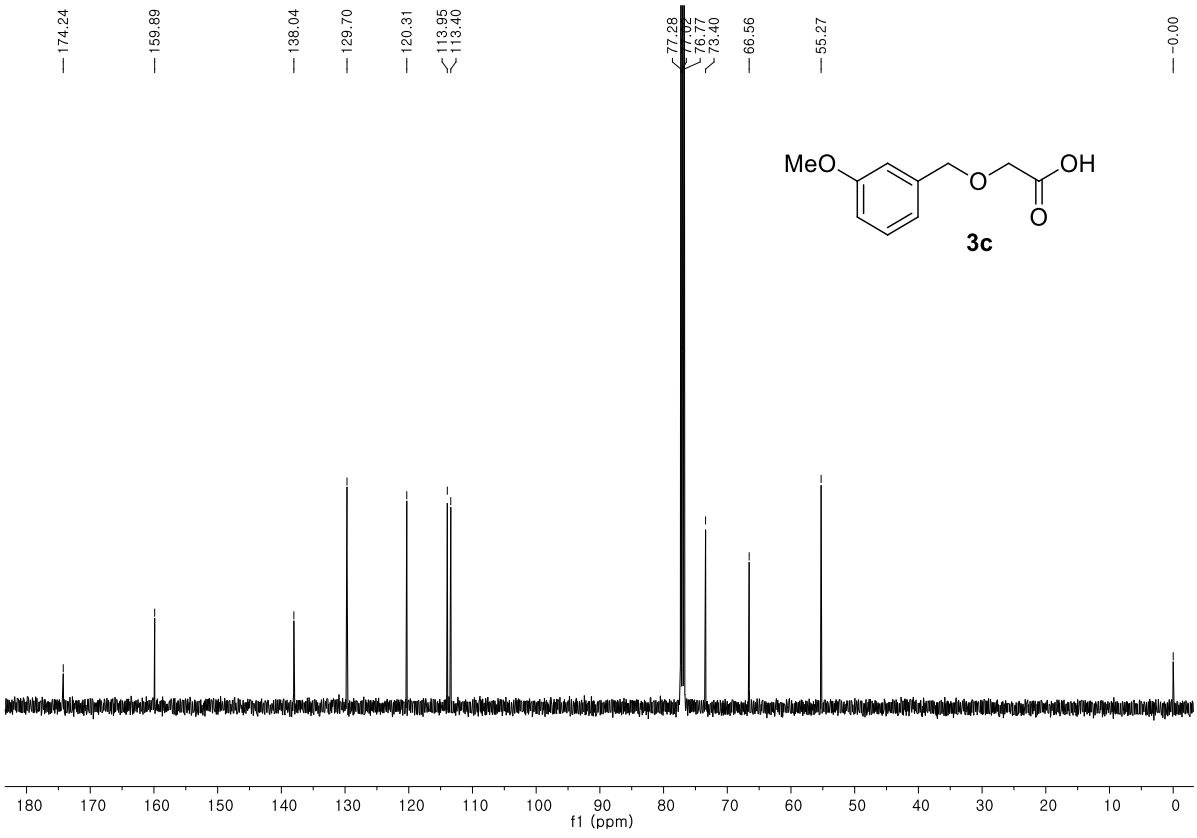

7.39
7.39
7.39
7.38
7.38
7.37
7.37
7.36
7.36
7.35
7.34
7.34
7.34
7.33
7.33
7.32
7.32
7.32
7.32
7.31
7.31
7.26
4.65
4.15
0.00
OH
O
O
3a
4.87
2.00
2.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


174.55
136.49
128.64
128.32
128.14
77.28
77.03
76.77
73.53
66.59
0.00
OH
O
O
3a
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.86
7.84
7.84
7.83
7.83
7.80
7.50
7.49
7.48
7.25
4.81
4.18
0.00
OH
O
O
3b
4.17
3.06
2.00
2.02
f1 (ppm)


174.79
133.96
133.21
133.19
128.53
127.94
127.74
127.16
126.32
126.23
125.76
77.28
77.02
76.77
73.60
66.59
0.00
OH
O
O
3b
f1 (ppm)


7.30
7.28
7.27
7.26
6.94
6.92
6.92
6.88
6.87
6.86
6.86
4.63
4.14
3.82
0.00
MeO
O
OH
O
3c
1.47
3.17
2.00
2.03
3.07
f1 (ppm)


174.24
159.89
138.04
129.70
120.31
113.95
113.40
77.28
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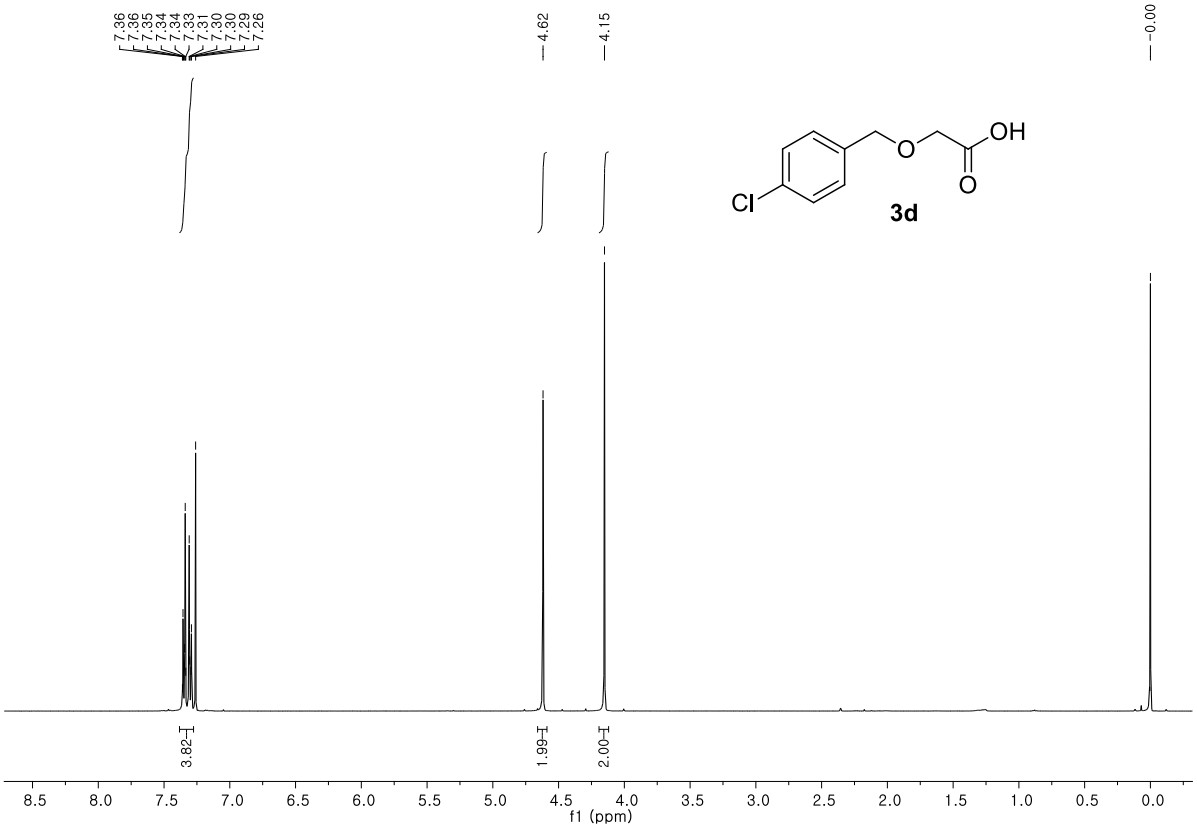
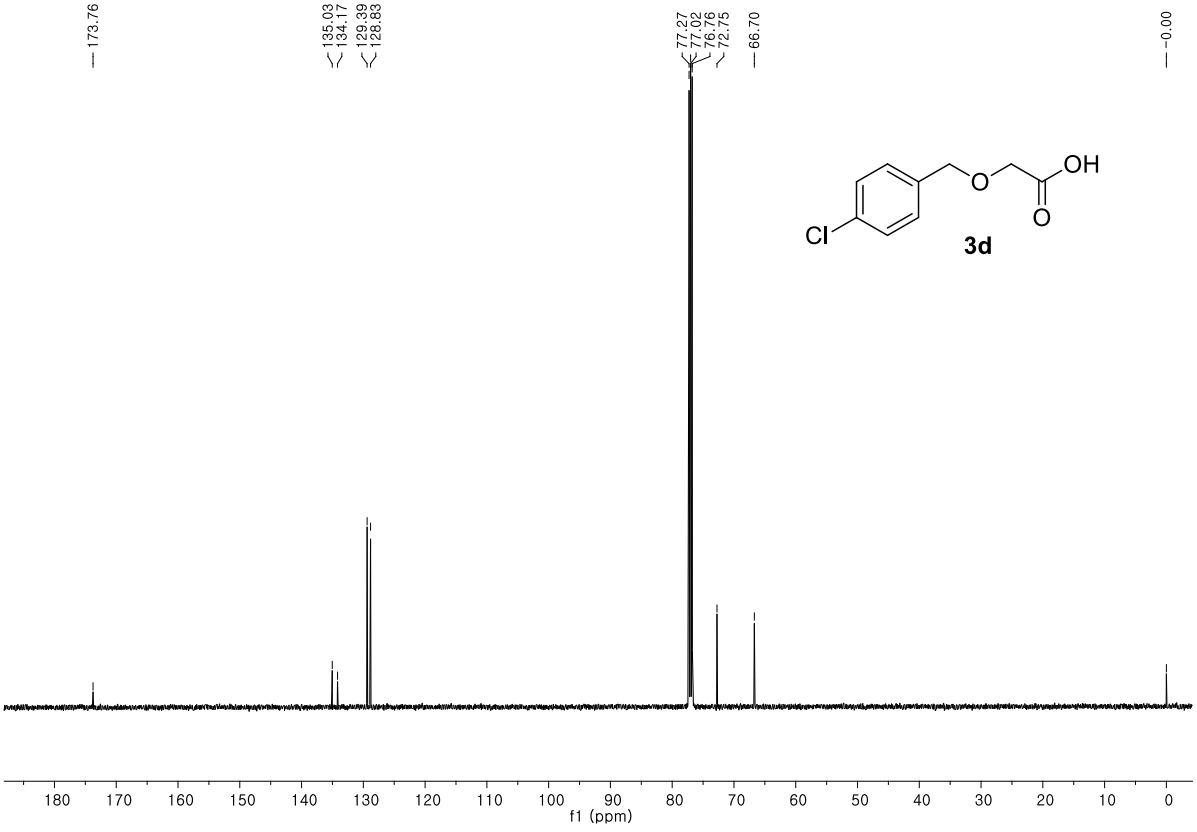
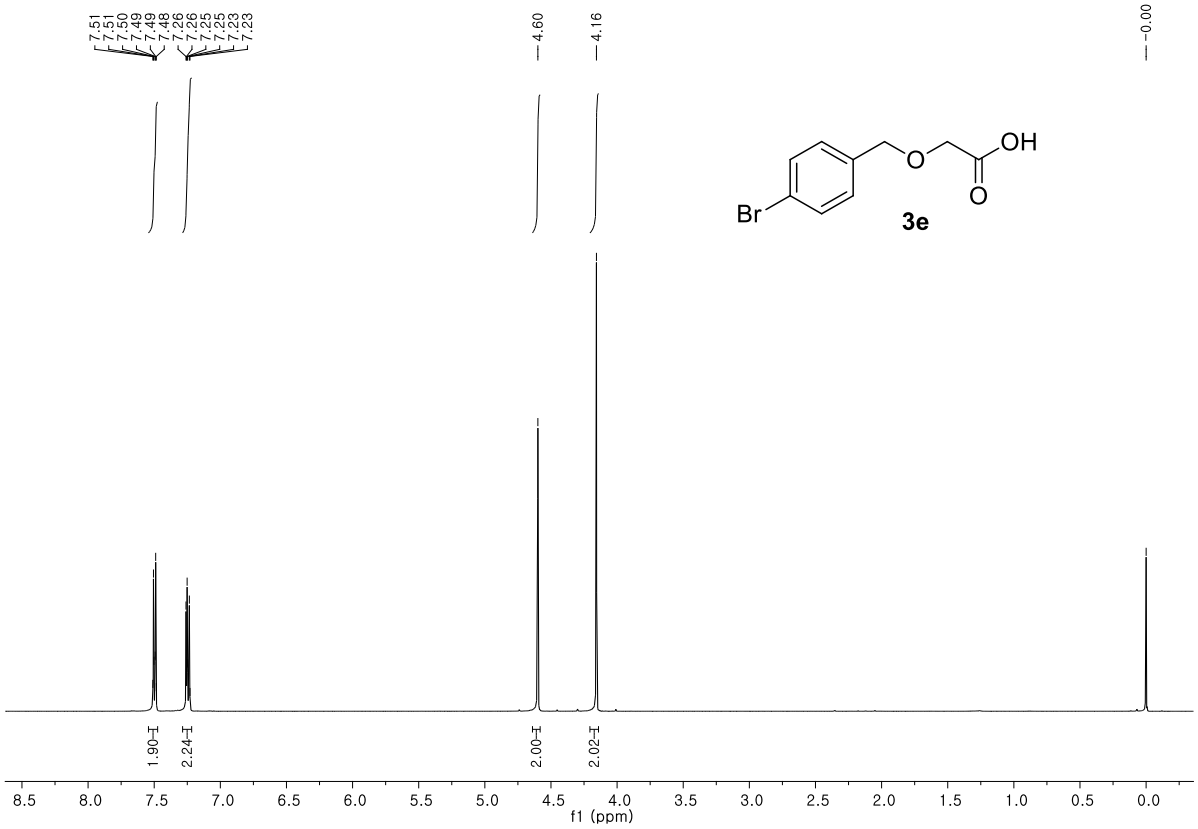
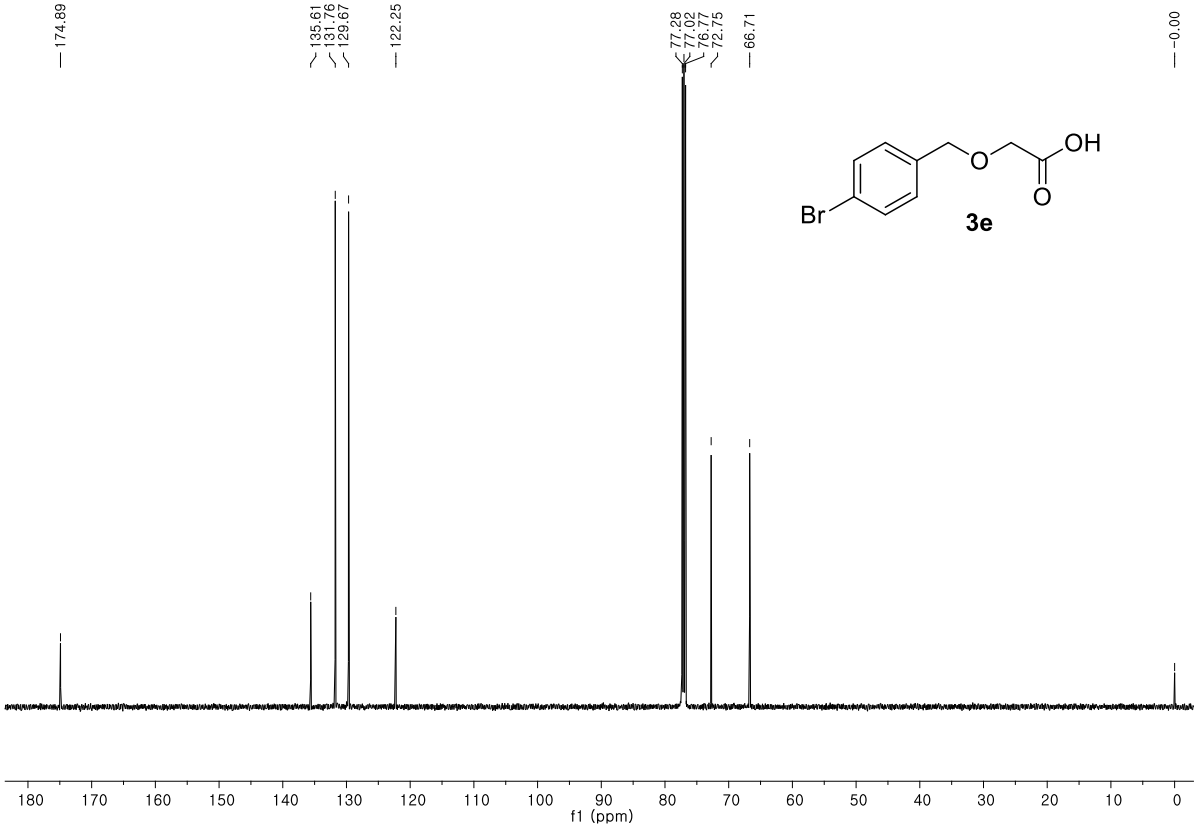
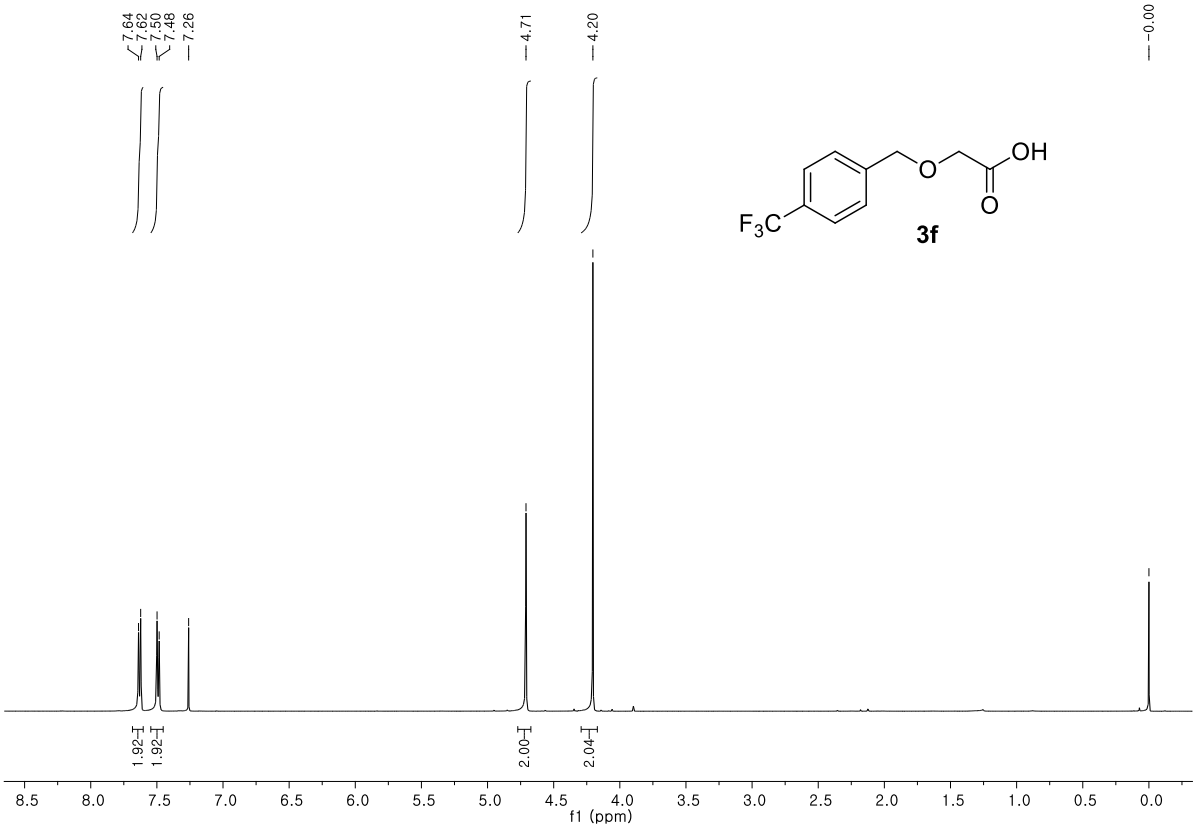
76.77
73.40
66.56
55.27
0.00
MeO
O
OH
O
3c
f1 (ppm)

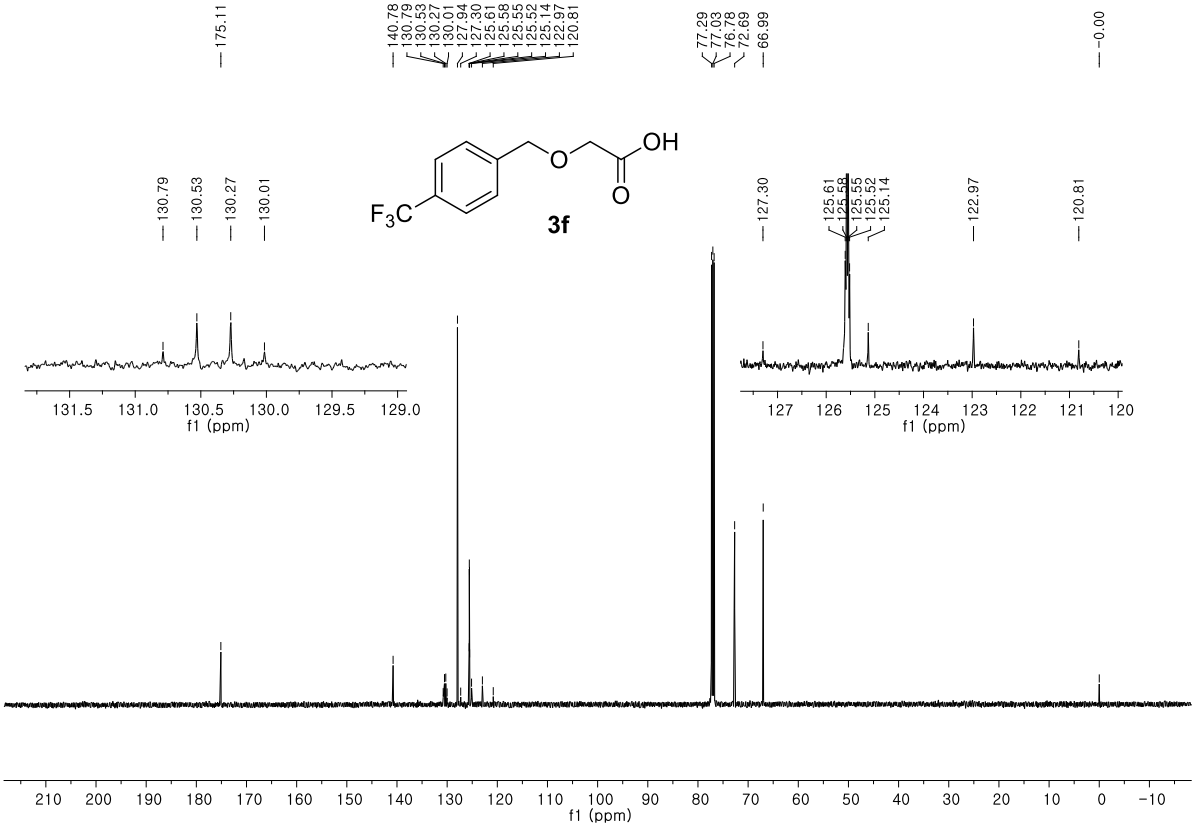

7.36
7.36
7.35
7.34
7.34
7.33
7.31
7.30
7.30
7.29
7.26
4.62
4.15
0.00
OH
O
O
Cl
3d
3.82
1.99
2.00
f1 (ppm)

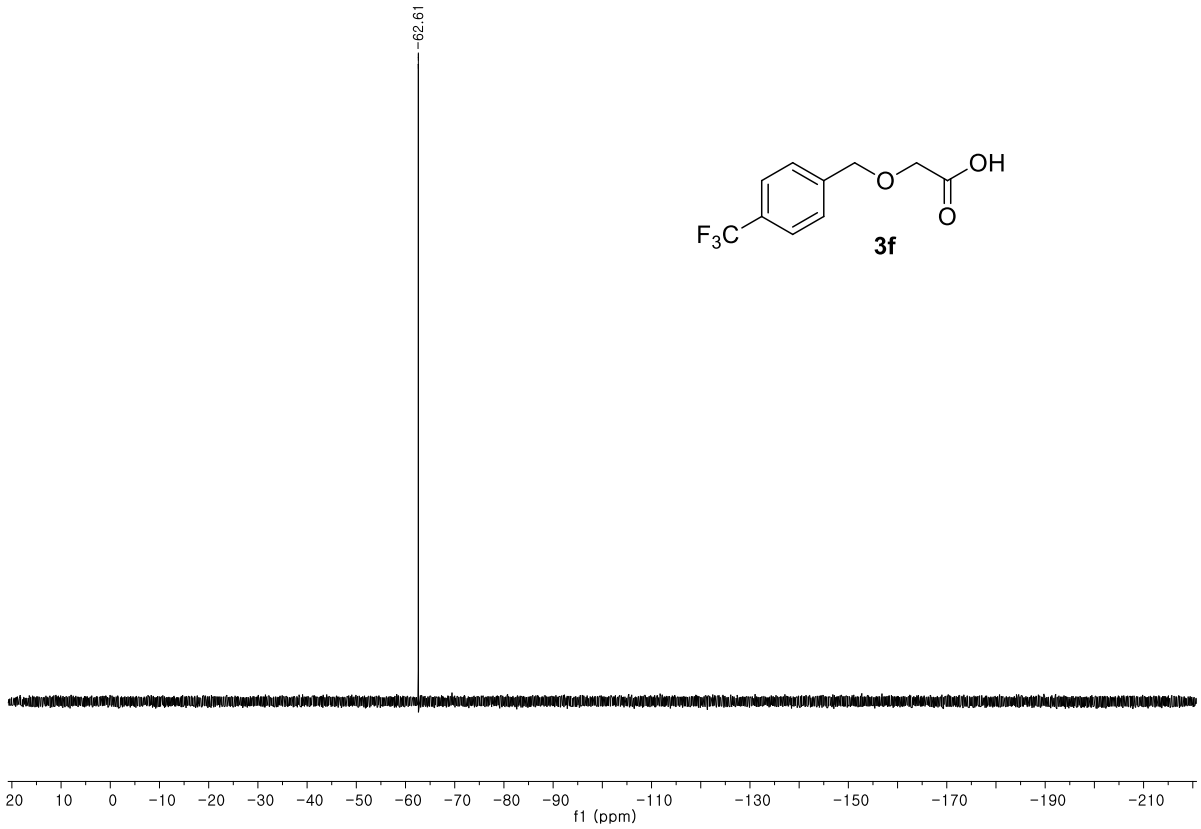

173.76
135.03
134.17
129.39
128.83
77.27
77.02
76.76
72.75
66.70
0.00
OH
O
O
Cl
3d
f1 (ppm)

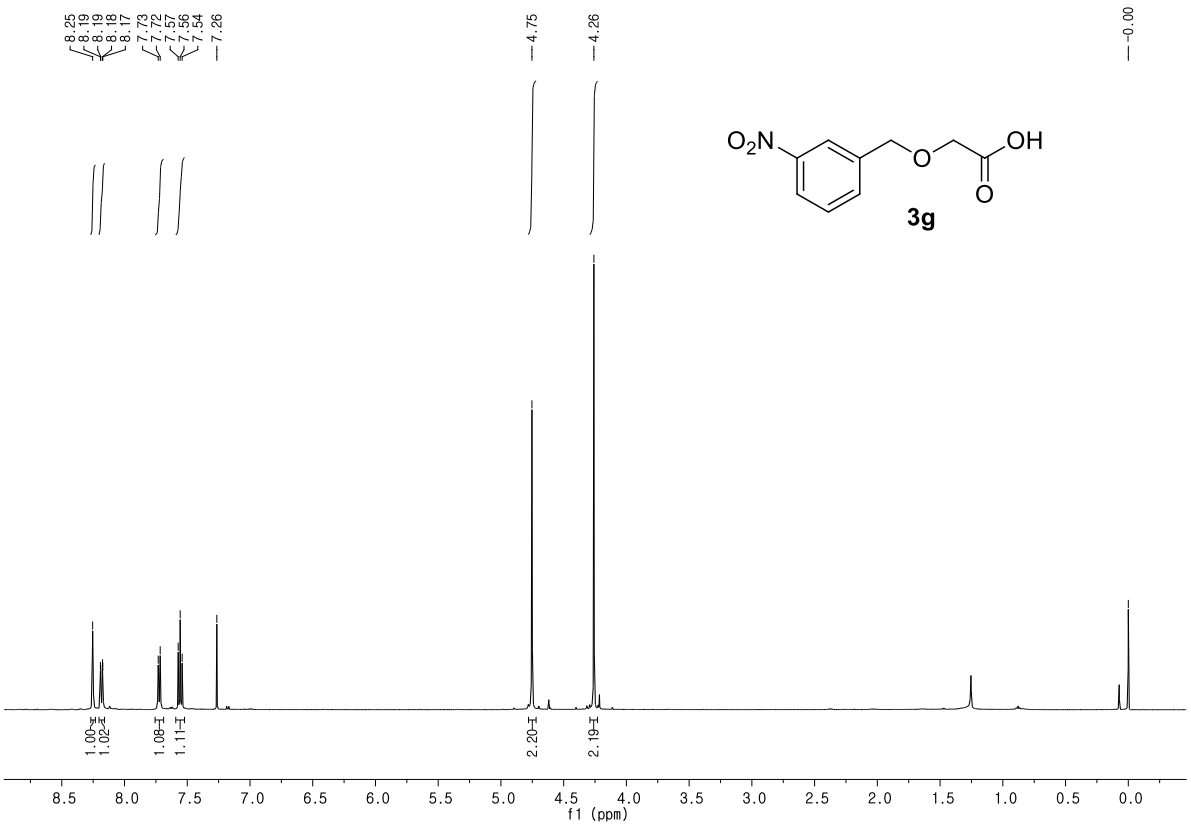

7.51
7.51
7.50
7.49
7.49
7.48
7.26
7.26
7.25
7.25
7.23
7.23
4.60
4.16
0.00
OH
O
O
Br
3e
1.90
2.24
2.00
2.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

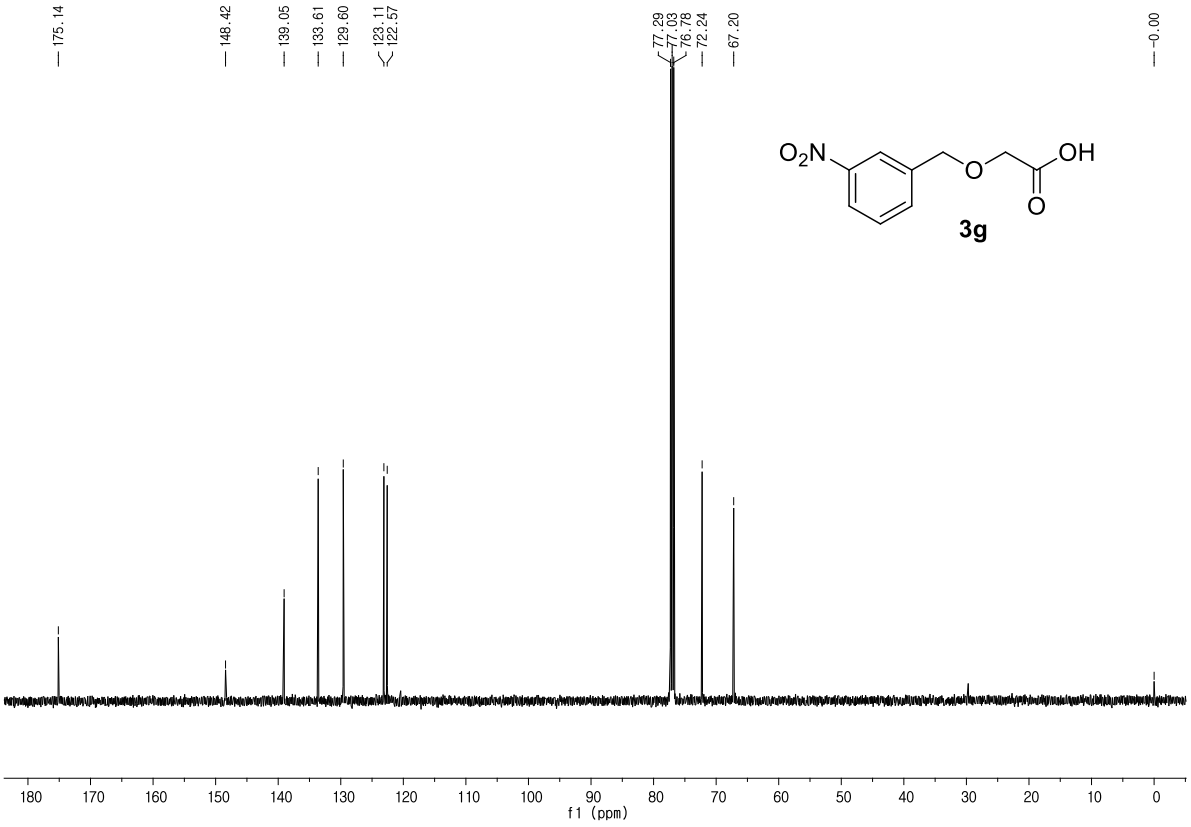

174.89
135.61
131.76
129.67
122.25
77.28
77.02
76.77
72.75
66.71
0.00
OH
O
O
Br
3e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

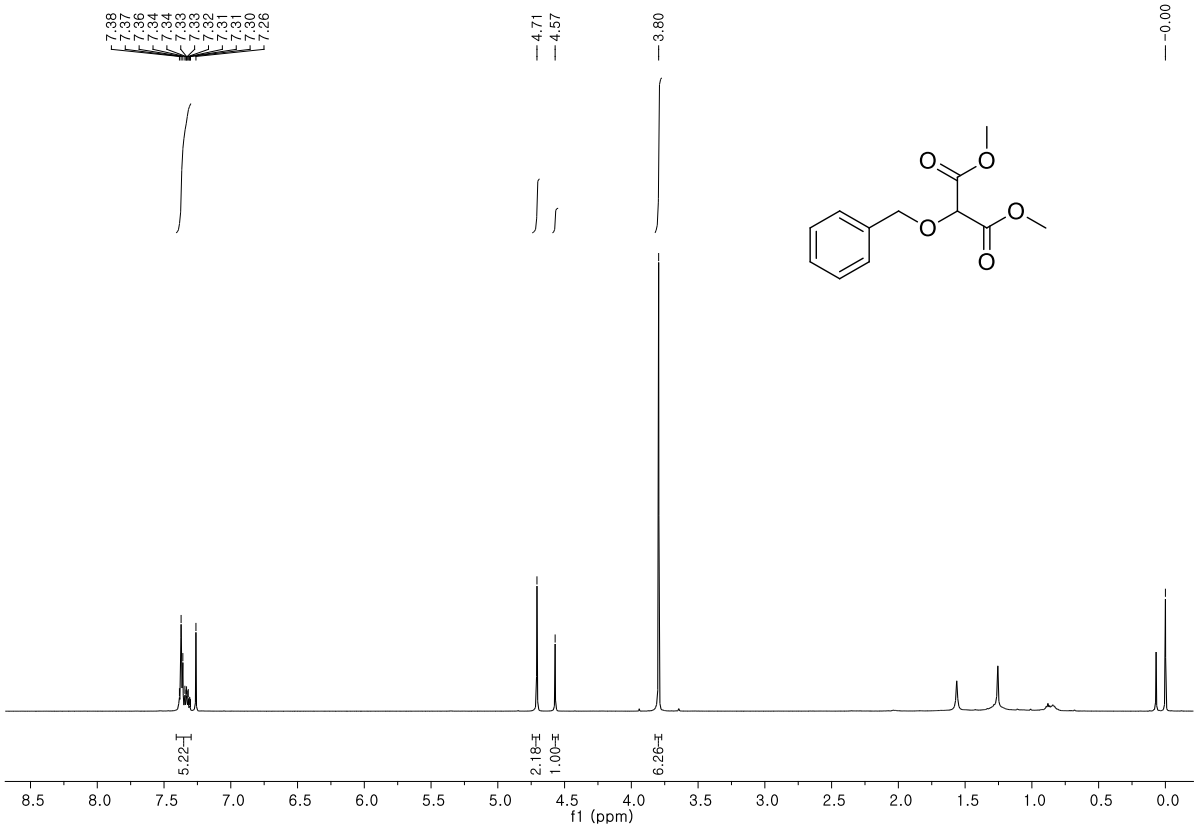

7.64
7.62
7.50
7.48
7.26
4.71
4.20
0.00
OH
O
O
F3C
3f
1.92
1.92
2.00
2.04
f1 (ppm)

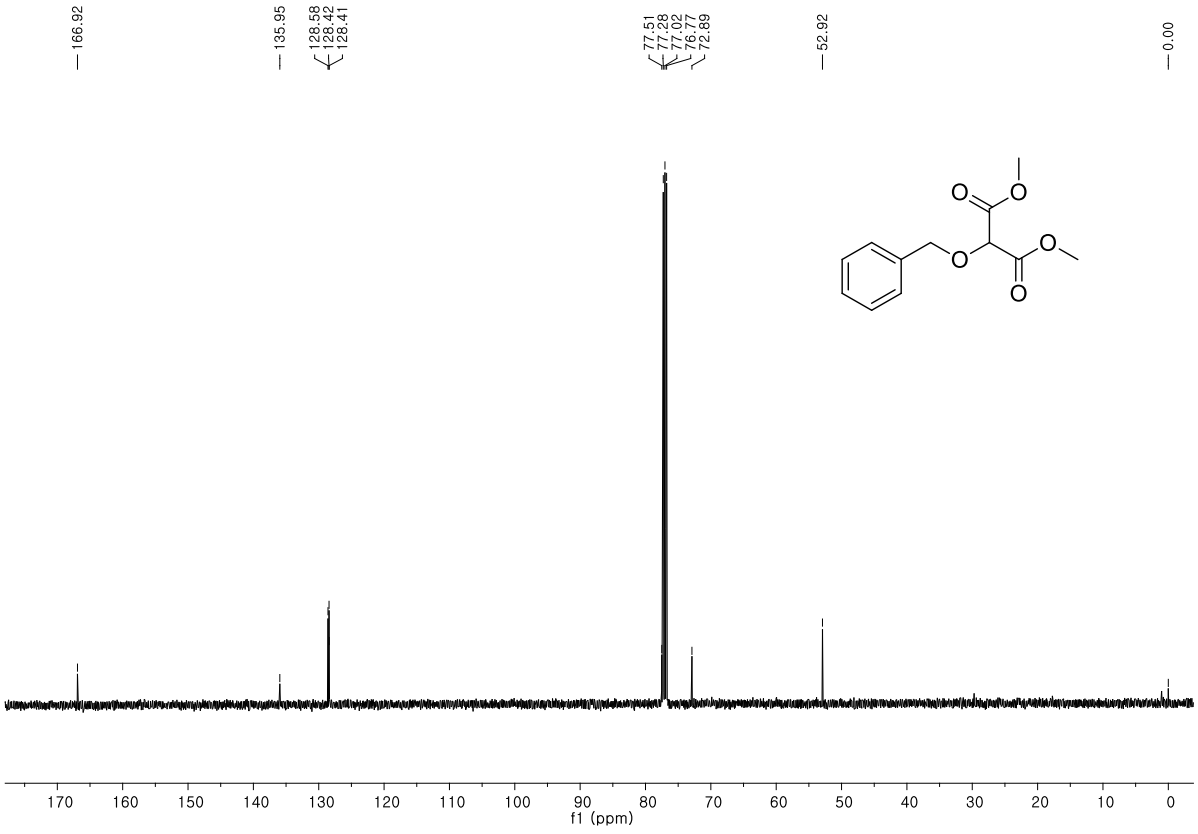

175.11
140.78
130.79
130.53
130.27
130.01
127.94
127.30
125.61
125.58
125.55
125.52
125.14
122.97
120.81
77.29
77.03
76.78
72.69
66.99
0.00
OH
O
O
F3C
3f
130.79
130.53
130.27
130.01
127.30
125.61
125.58
125.55
125.52
125.14
122.97
120.81
f1 (ppm)
f1 (ppm)
f1 (ppm)


-62.61
OH
O
O
F3C
3f
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)


8.25
8.19
8.19
8.18
8.17
7.73
7.72
7.57
7.56
7.54
7.26
4.75
4.26
0.00
O2N
O
OH
O
3g
1.00
1.02
1.08
1.11
2.20
2.19
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


175.14
148.42
139.05
133.61
129.60
123.11
122.57
77.29
77.03
76.78
72.24
67.20
0.00
O2N
O
OH
O
3g
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.38
7.37
7.36
7.34
7.34
7.33
7.33
7.32
7.31
7.31
7.30
7.26
4.71
4.57
3.80
0.00
5.22
2.18
1.00
6.26
f1 (ppm)


166.92
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128.58
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77.02
76.77
72.89
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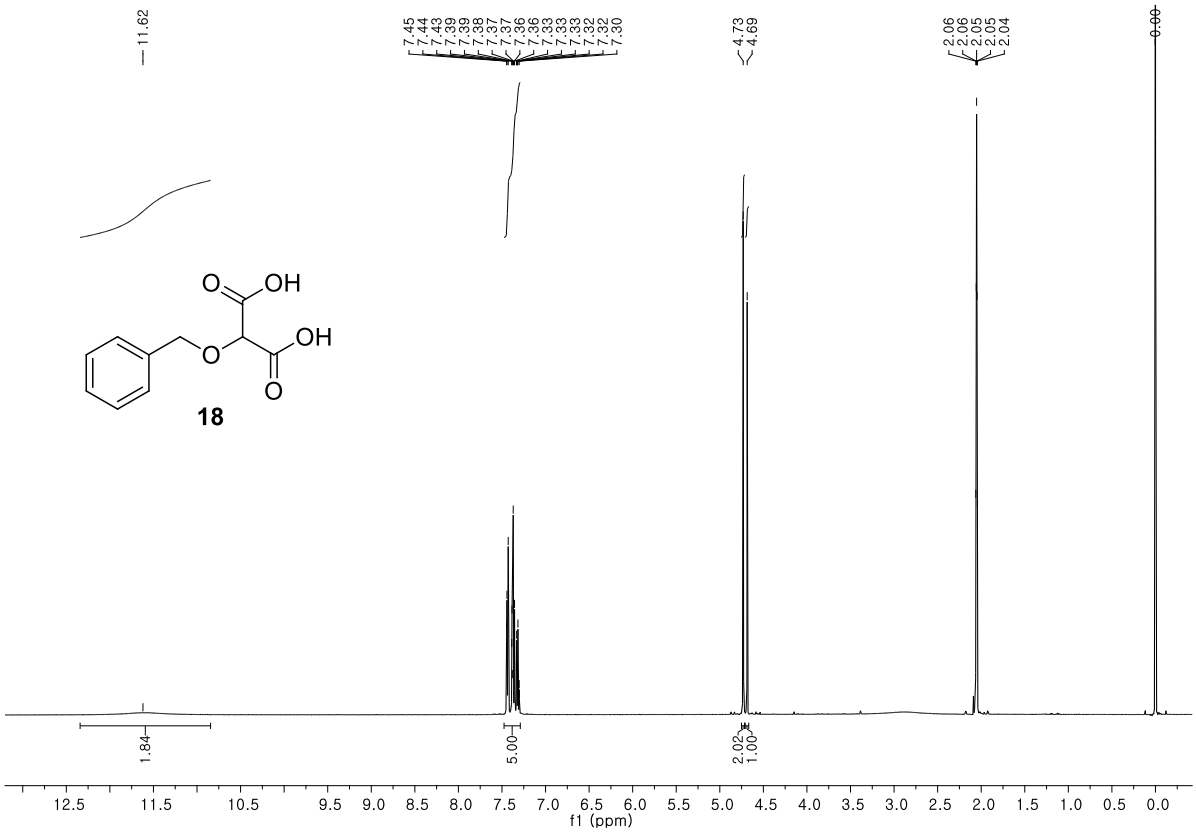
0.00
f1 (ppm)


11.62
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7.43
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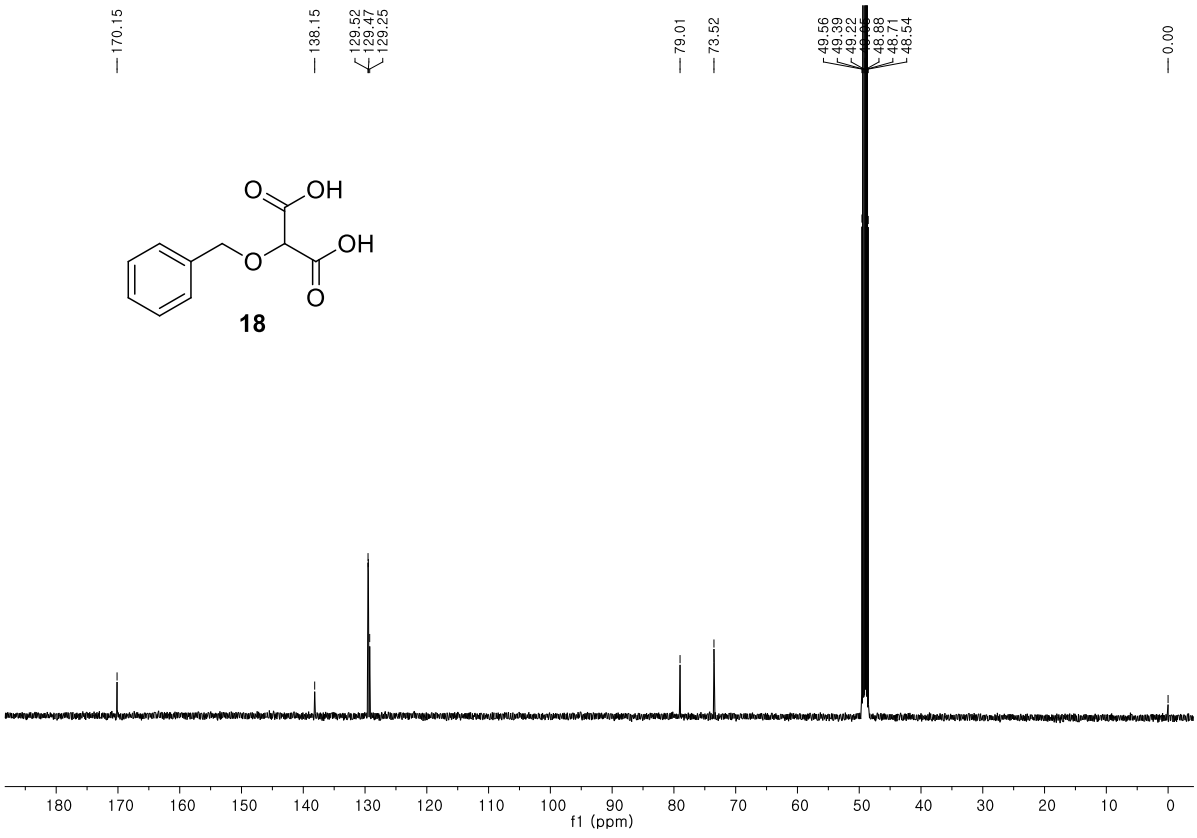
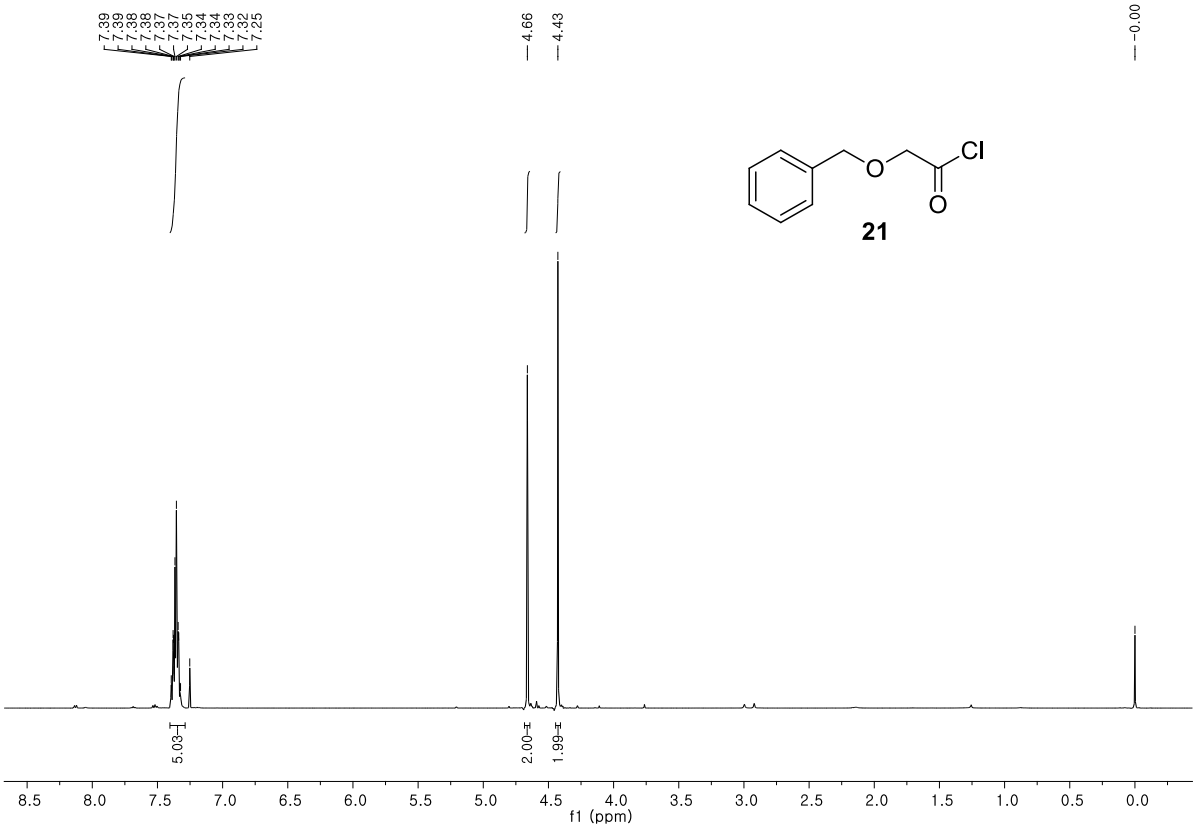
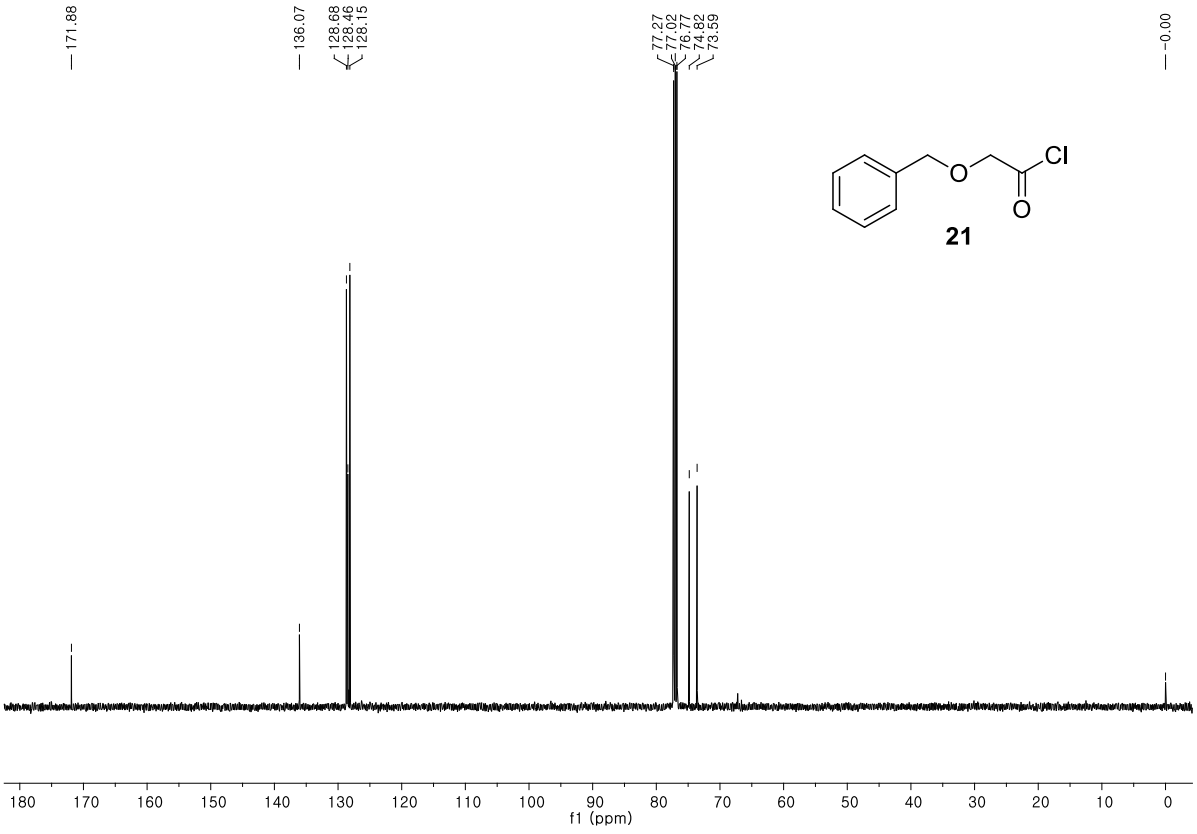
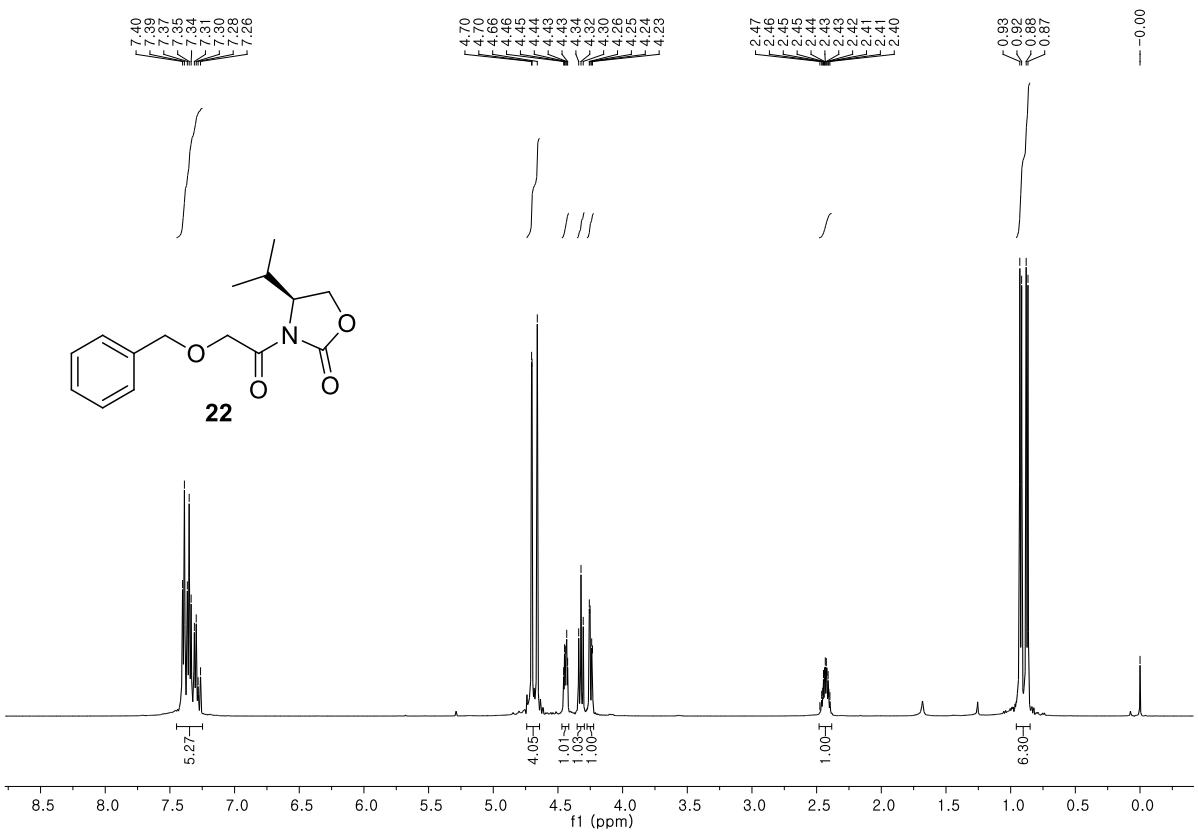
7.39
7.38
7.37
7.37
7.36
7.36
7.33
7.33
7.32
7.32
7.30
4.73
4.69
2.06
2.06
2.05
2.05
2.04
0.00
O
OH
OH
O
O
18
1.84
5.00
2.02
1.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.15
138.15
129.52
129.47
129.25
79.01
73.52
49.56
49.39
49.22
48.88
48.71
48.54
0.00
O
OH
OH
O
O
18
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.39
7.39
7.38
7.38
7.37
7.37
7.35
7.34
7.34
7.33
7.32
7.25
4.66
4.43
0.00
Cl
O
O
21
5.03
2.00
1.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


171.88
136.07
128.68
128.46
128.15
77.27
77.02
76.77
74.82
73.59
0.00
Cl
O
O
21
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.39
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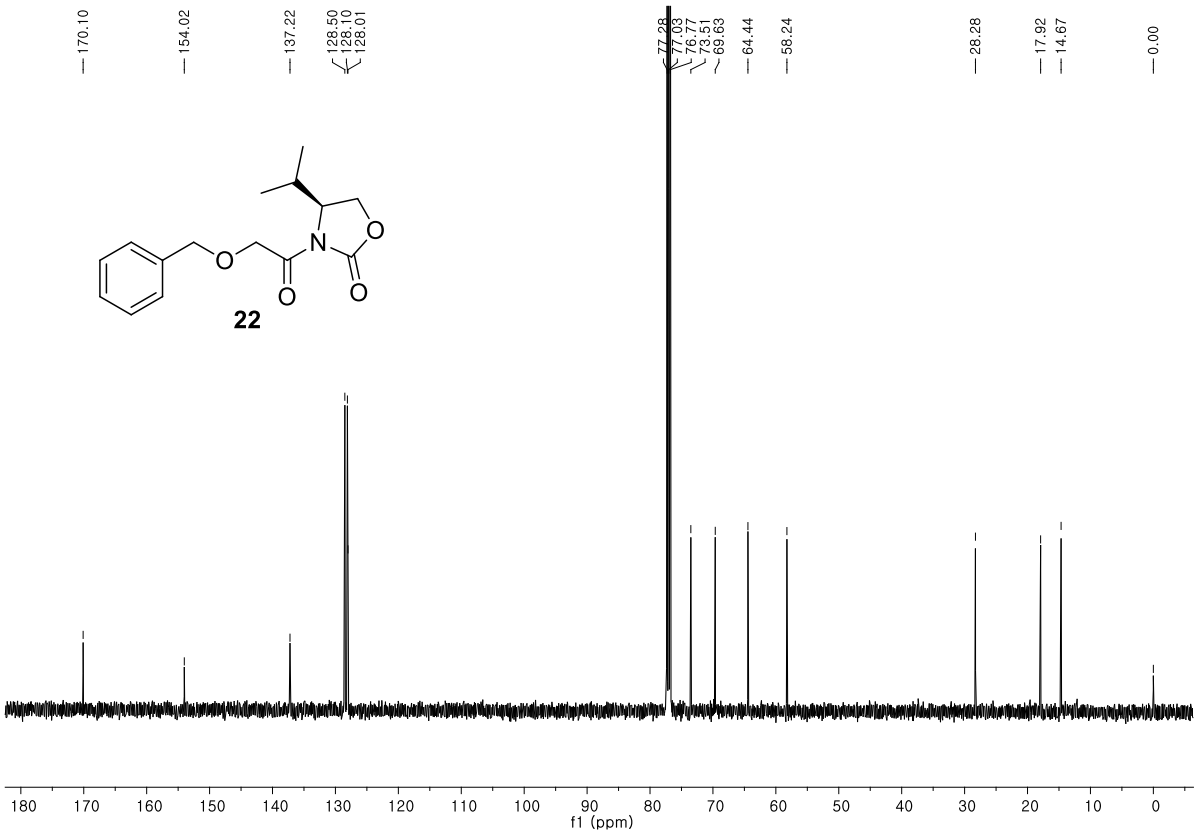
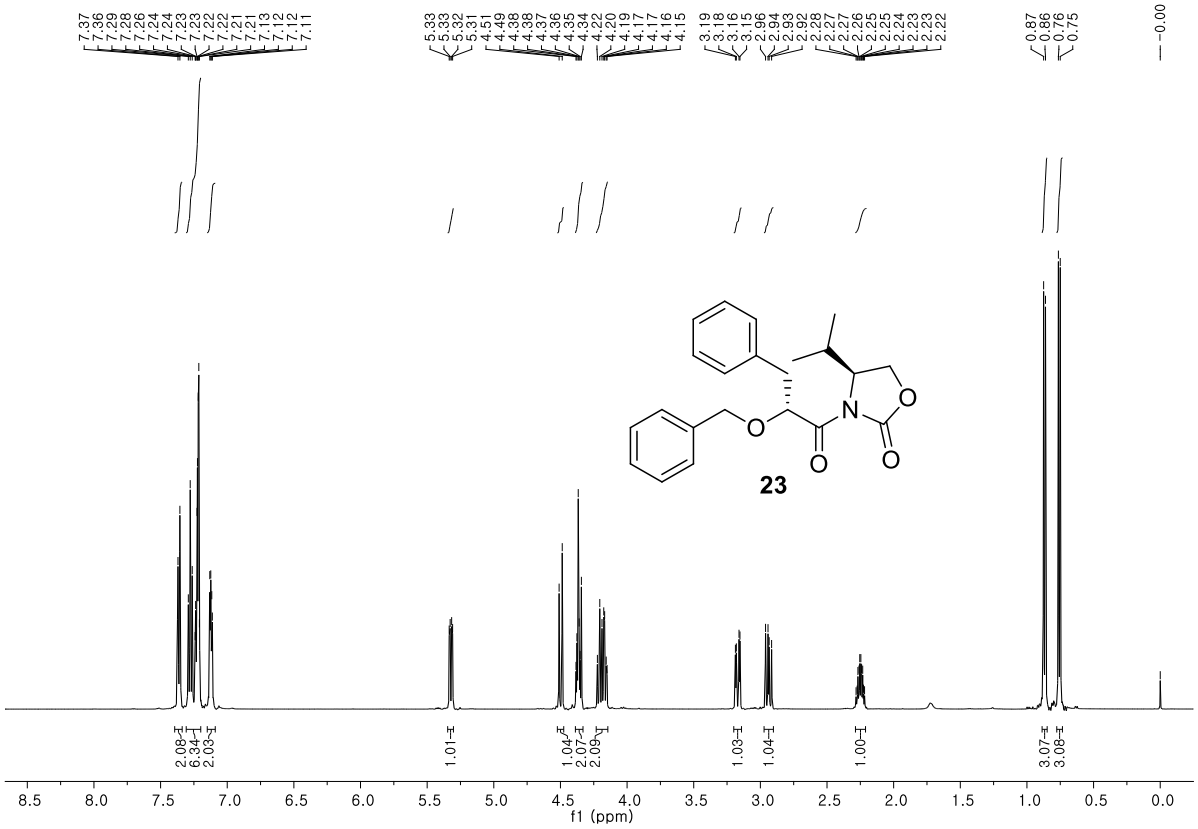
7.34
7.31
7.30
7.28
7.26
4.70
4.70
4.66
4.46
4.45
4.44
4.43
4.43
4.34
4.32
4.30
4.26
4.25
4.24
4.23
2.47
2.46
2.45
2.45
2.44
2.43
2.43
2.42
2.41
2.41
2.40
0.93
0.92
0.88
0.87
0.00
22
5.27
4.05
1.01
1.03
1.00
1.00
6.30
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.10
154.02
137.22
128.50
128.10
128.01
77.28
77.03
76.77
73.51
69.63
64.44
58.24
28.28
17.92
14.67
0.00
22
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.37
7.36
7.29
7.28
7.26
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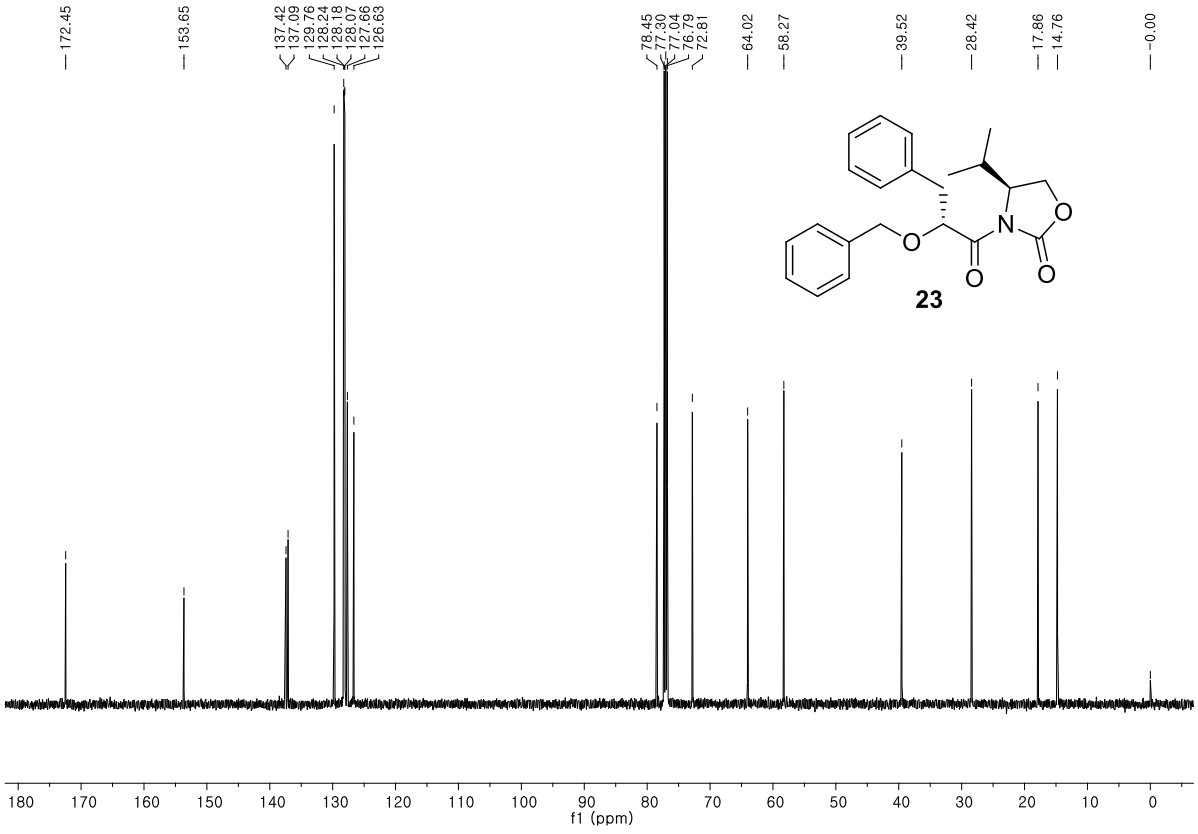
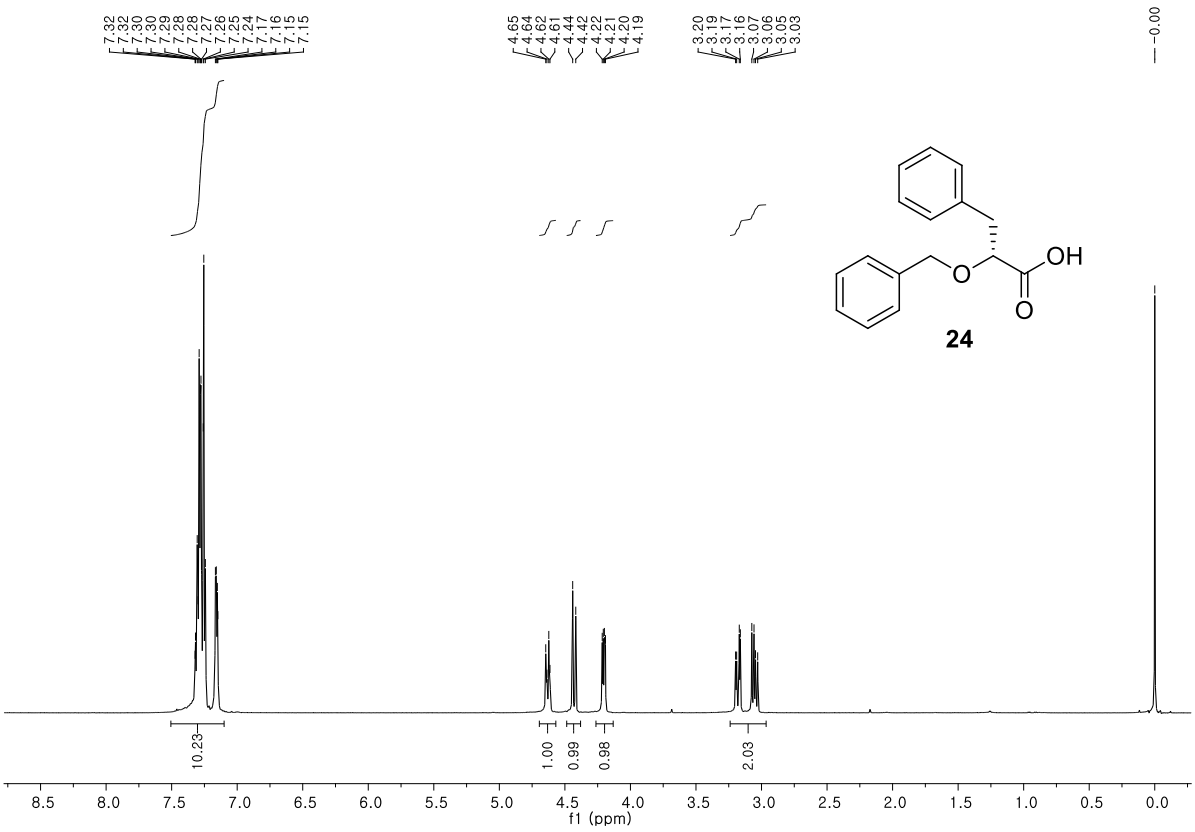
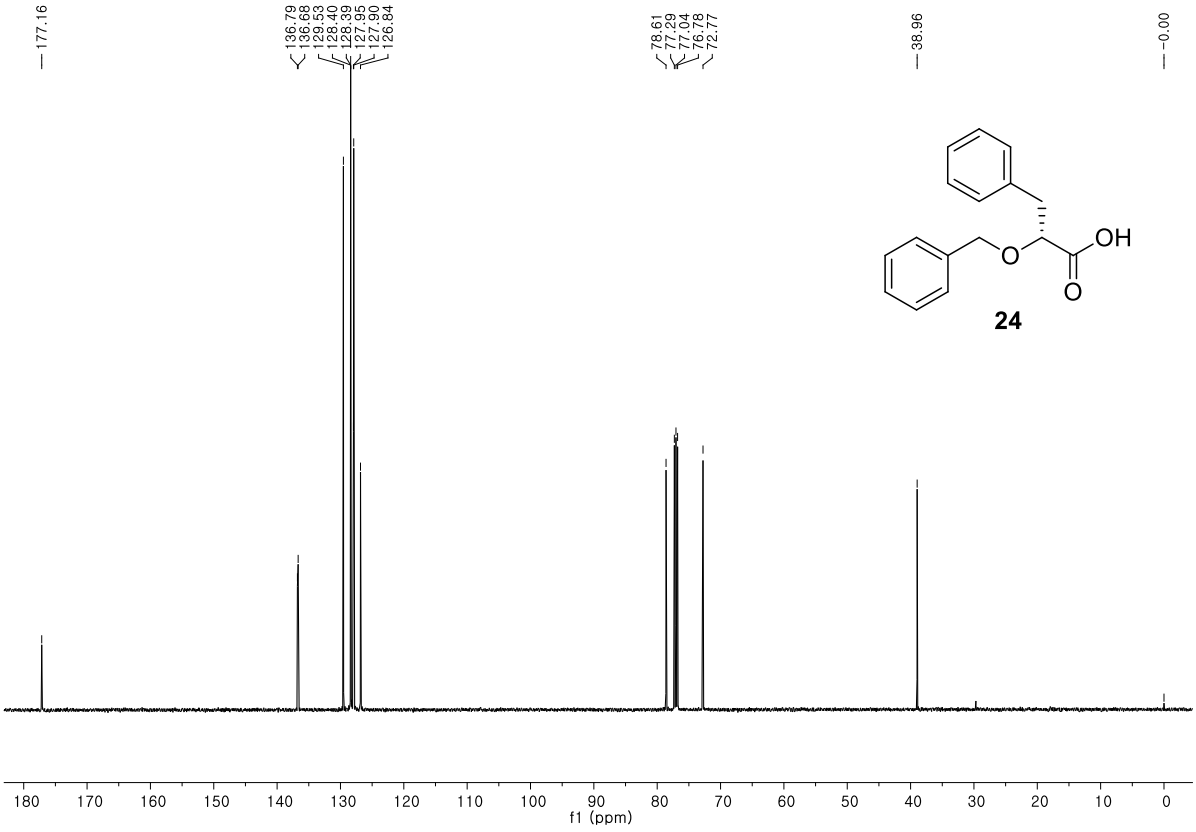
7.24
7.23
7.23
7.22
7.22
7.21
7.21
7.13
7.12
7.12
7.11
5.33
5.33
5.32
5.31
4.51
4.49
4.38
4.38
4.37
4.36
4.35
4.34
4.22
4.20
4.19
4.17
4.17
4.16
4.15
3.19
3.18
3.16
3.15
2.96
2.94
2.93
2.92
2.28
2.27
2.27
2.26
2.25
2.25
2.24
2.23
2.23
2.22
0.87
0.86
0.76
0.75
0.00
2.08
6.34
2.03
1.01
1.04
2.07
2.09
1.03
1.04
1.00
3.07
3.08
23
f1 (ppm)


172.45
153.65
137.42
137.09
129.76
128.24
128.18
128.07
127.66
126.63
78.45
77.30
77.04
76.79
72.81
64.02
58.27
39.52
28.42
17.86
14.76
0.00
23
f1 (ppm)


7.32
7.32
7.30
7.30
7.29
7.28
7.28
7.27
7.26
7.25
7.24
7.17
7.16
7.15
7.15
4.65
4.64
4.62
4.61
4.44
4.42
4.22
4.21
4.20
4.19
3.20
3.19
3.17
3.16
3.07
3.06
3.05
3.03
0.00
OH
O
O
24
10.23
1.00
0.99
0.98
2.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


177.16
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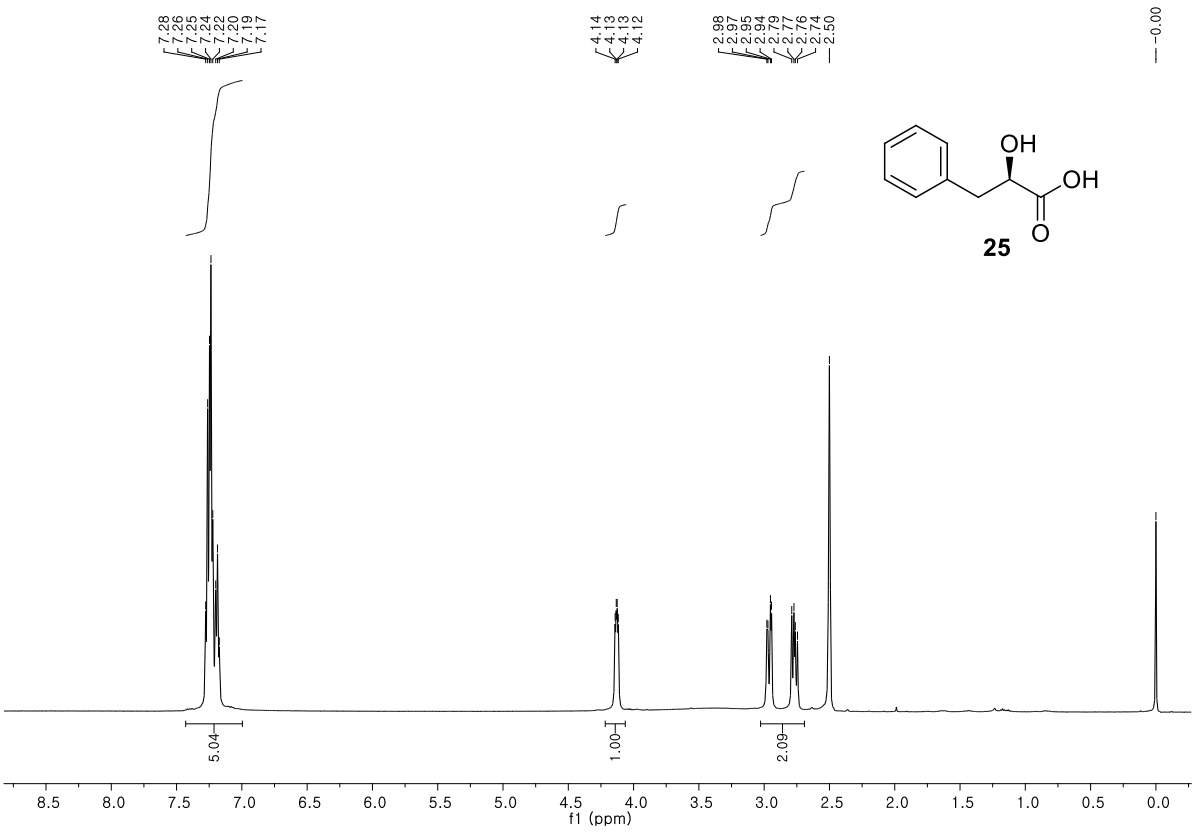
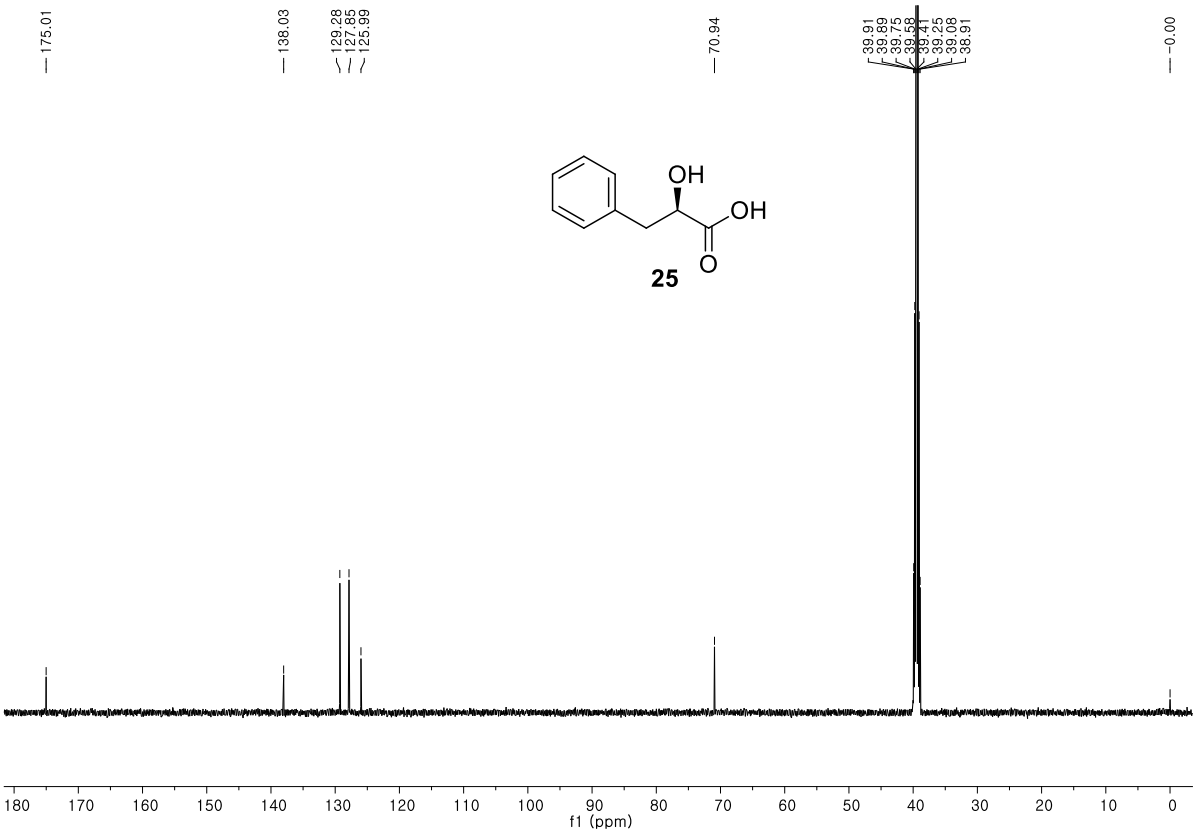
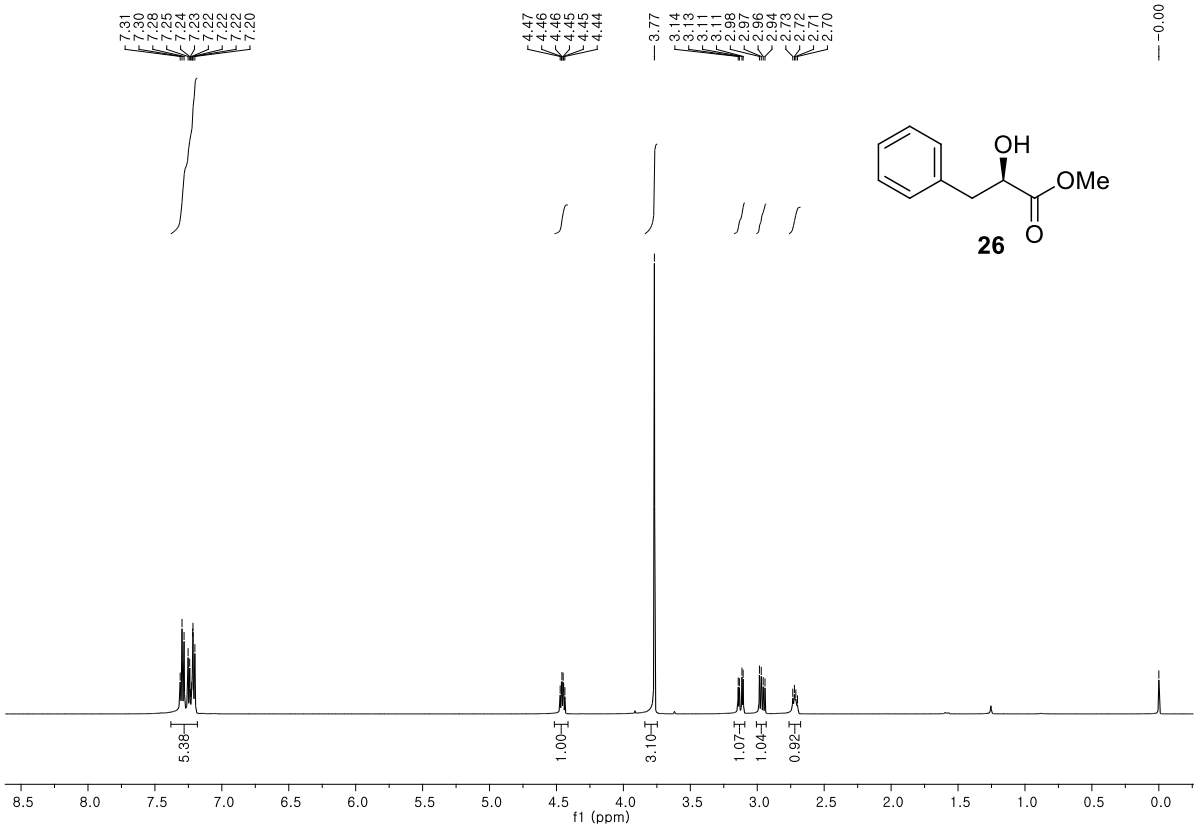
128.53
128.40
128.39
127.95
127.90
126.84
78.61
77.29
77.04
76.78
72.77
38.96
0.00
OH
O
O
24
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.28
7.26
7.25
7.24
7.22
7.20
7.19
7.17
4.14
4.13
4.13
4.12
2.98
2.97
2.95
2.94
2.79
2.77
2.76
2.74
2.50
0.00
OH
OH
O
25
5.04
1.00
2.09
f1 (ppm)


175.01
138.03
129.28
127.85
125.99
70.94
39.91
39.89
39.75
39.58
39.41
39.25
39.08
38.91
0.00
OH
OH
O
25
f1 (ppm)


7.31
7.30
7.28
7.25
7.24
7.23
7.22
7.22
7.22
7.20
4.47
4.46
4.46
4.45
4.45
4.44
3.77
3.14
3.13
3.11
3.11
2.98
2.97
2.96
2.94
2.73
2.72
2.71
2.70
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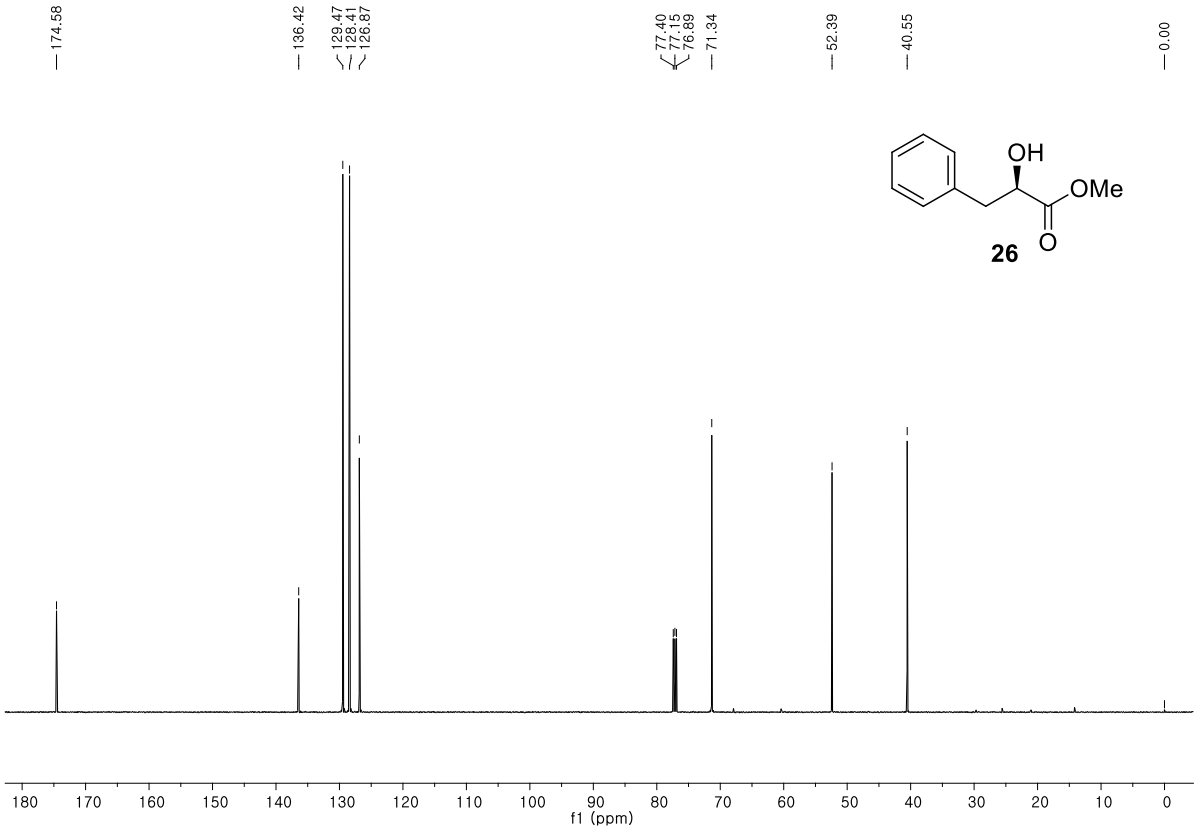
OH
OMe
O
26
5.38
1.00
3.10
1.07
1.04
0.92
f1 (ppm)


174.58
136.42
129.47
128.41
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77.15
76.89
71.34
52.39
40.55
0.00
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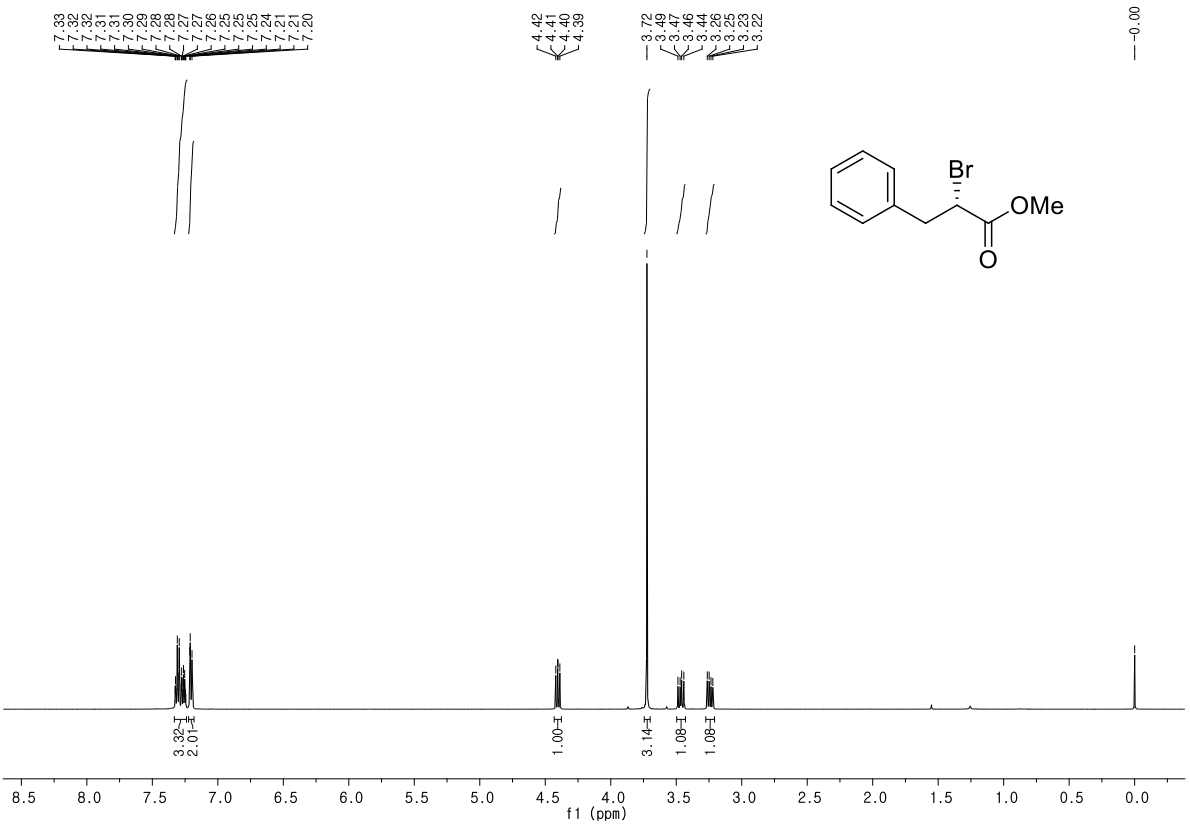
OMe
O
26
f1 (ppm)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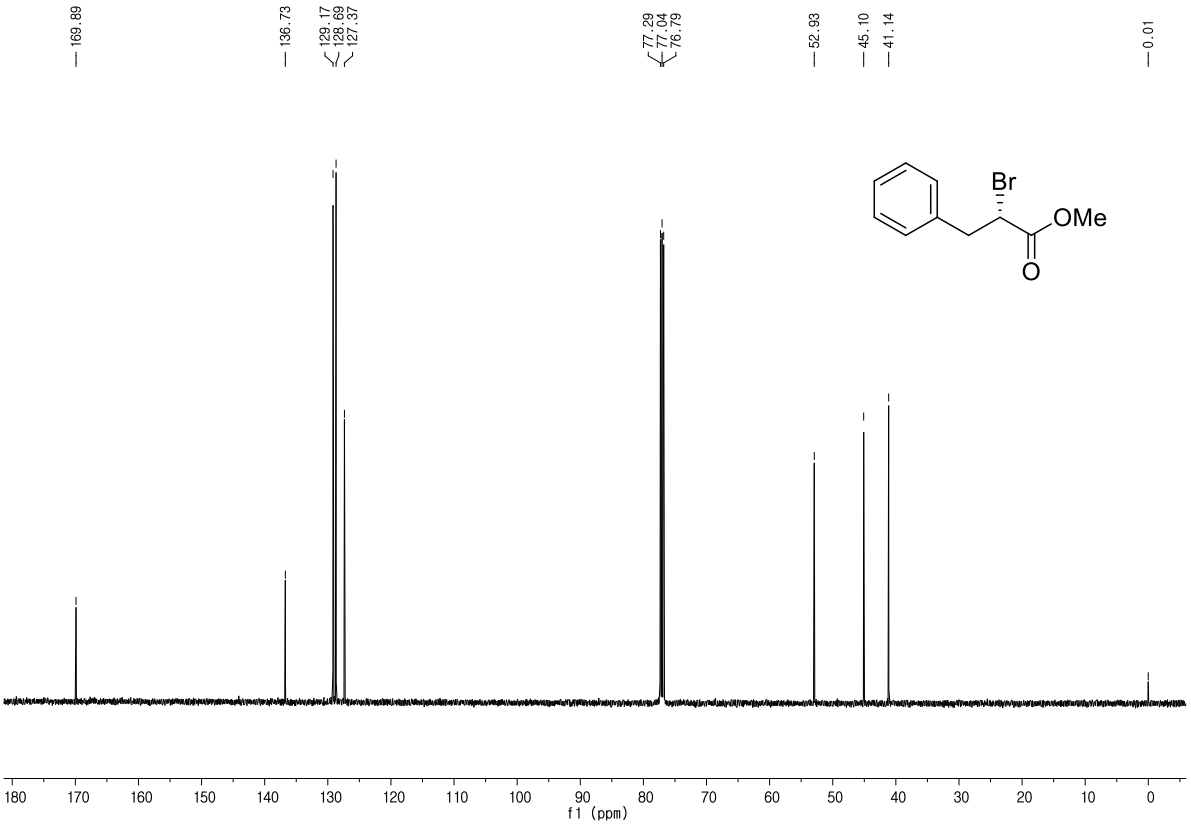
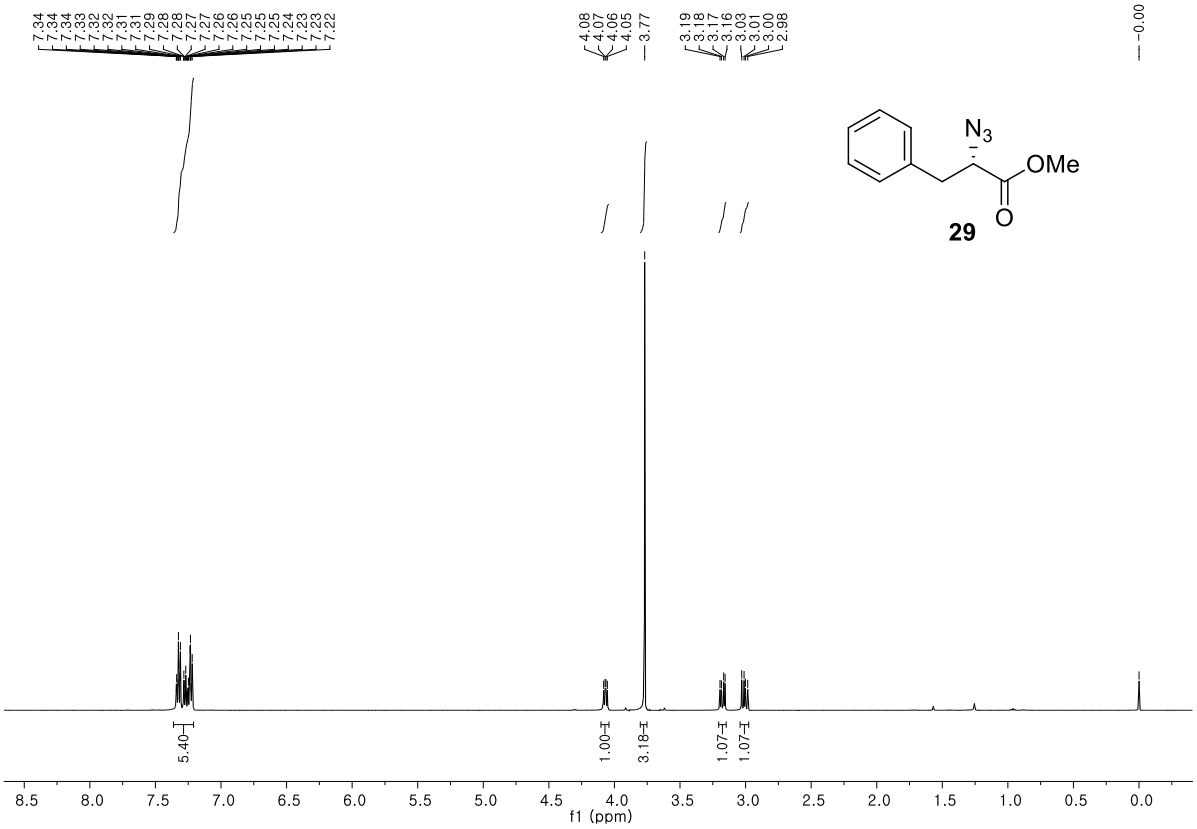
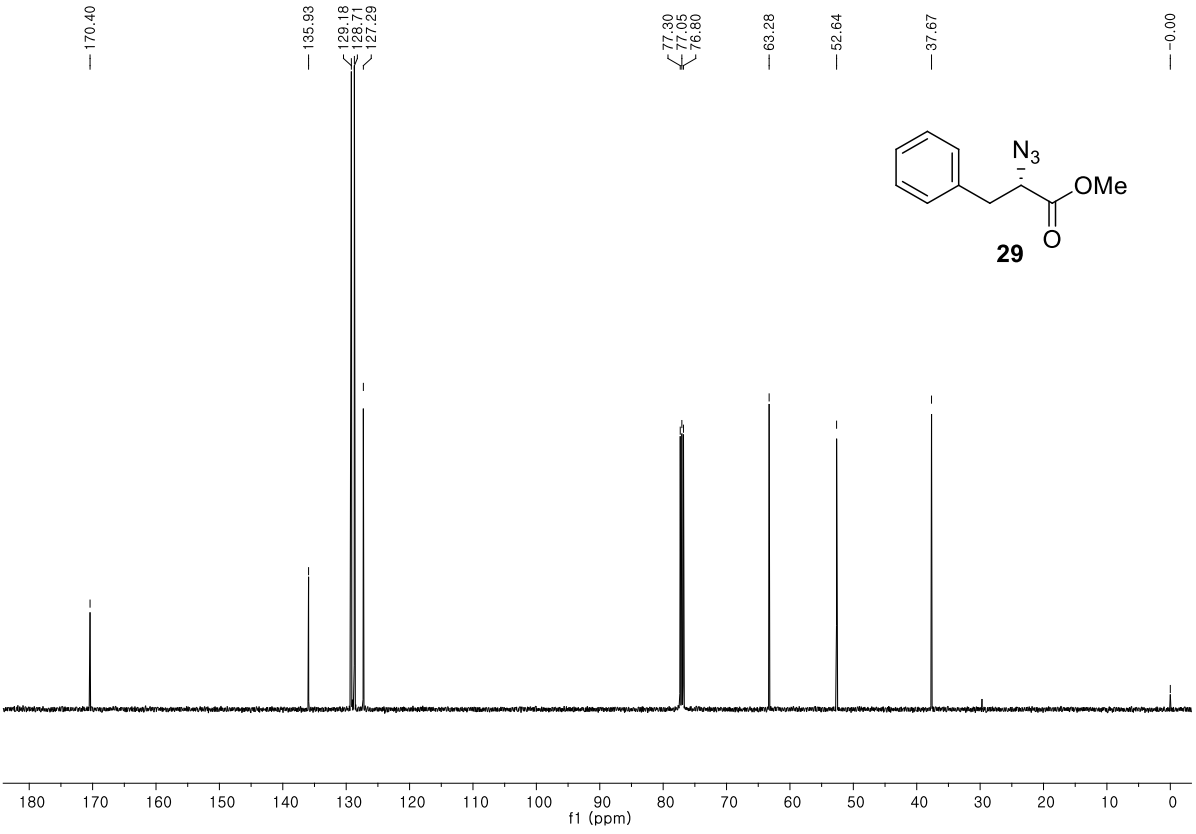
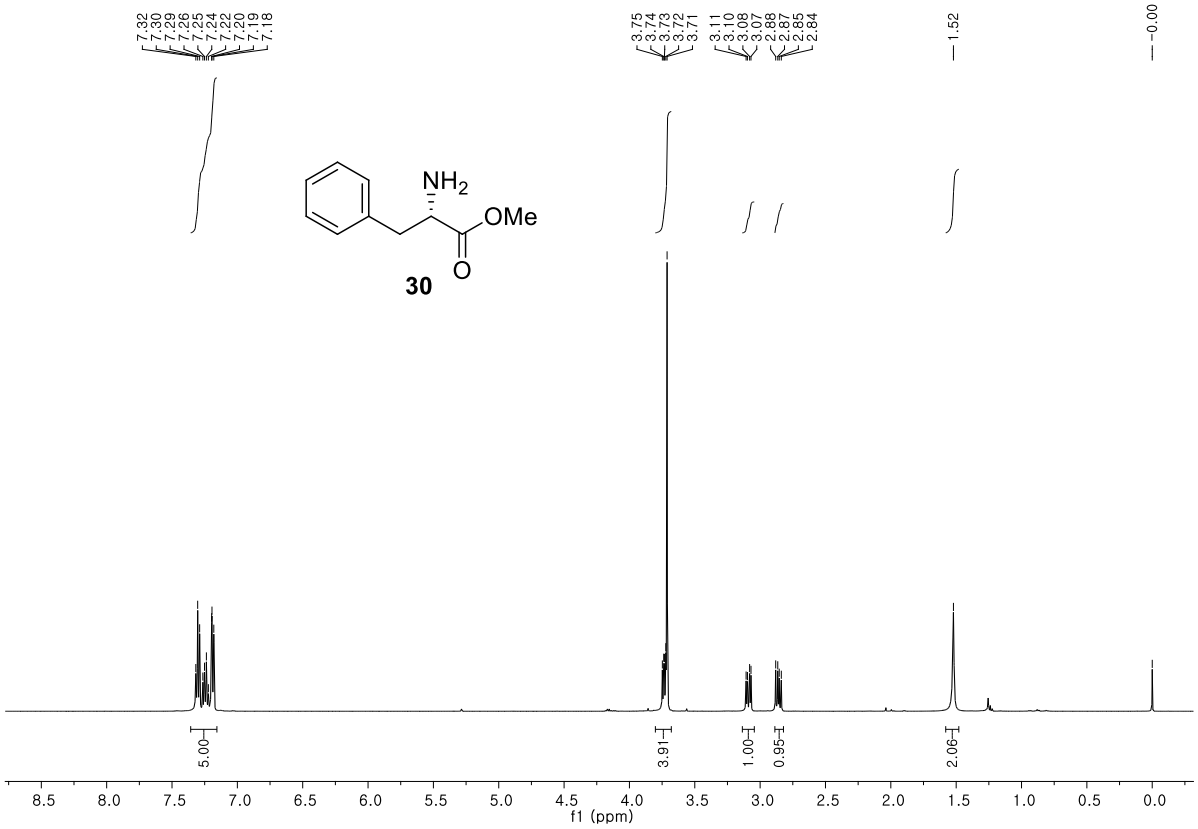
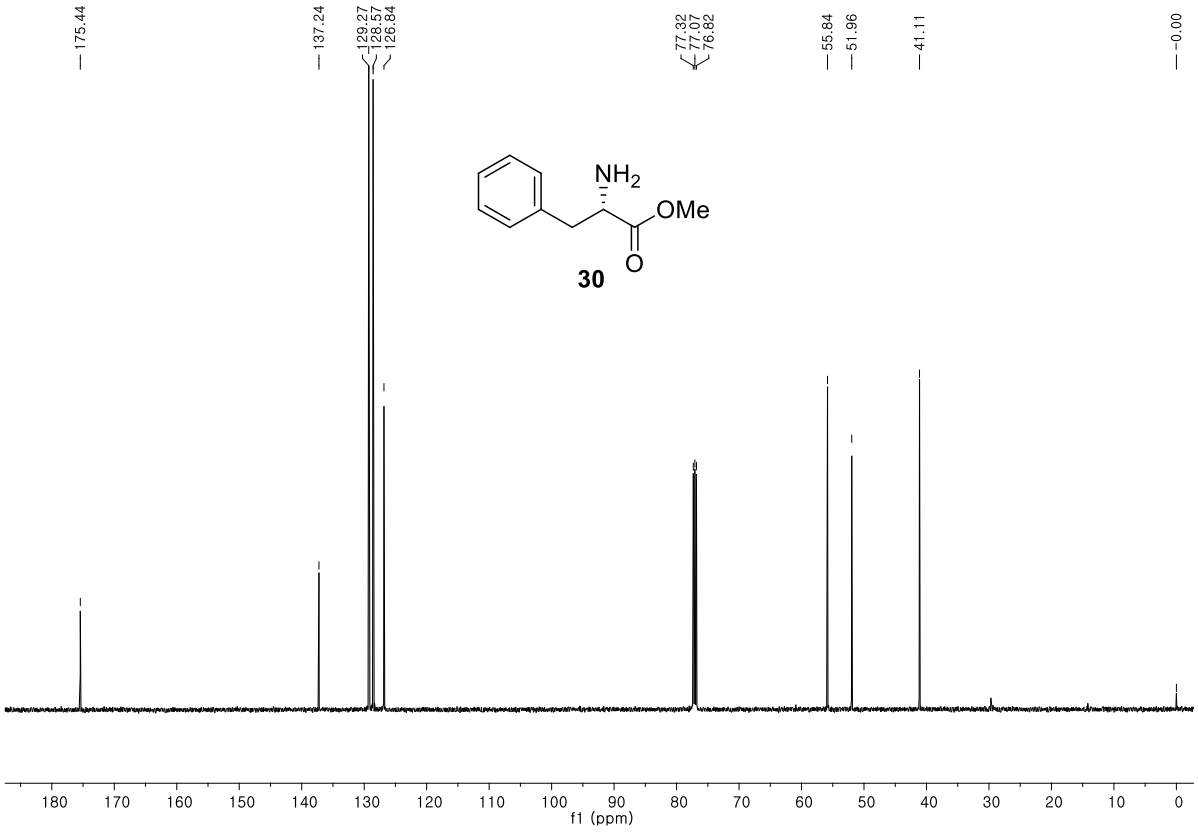
7.32
7.32
7.31
7.31
7.30
7.29
7.28
7.28
7.27
7.27
7.26
7.25
7.25
7.25
7.24
7.21
7.21
7.20
4.42
4.41
4.40
4.39
3.72
3.49
3.47
3.46
3.44
3.26
3.25
3.23
3.22
0.00
Br
OMe
O
3.32
2.01
1.00
3.14
1.08
1.08
f1 (ppm)


169.89
136.73
129.17
128.69
127.37
77.29
77.04
76.79
52.93
45.10
41.14
0.01
Br
OMe
O
f1 (ppm)

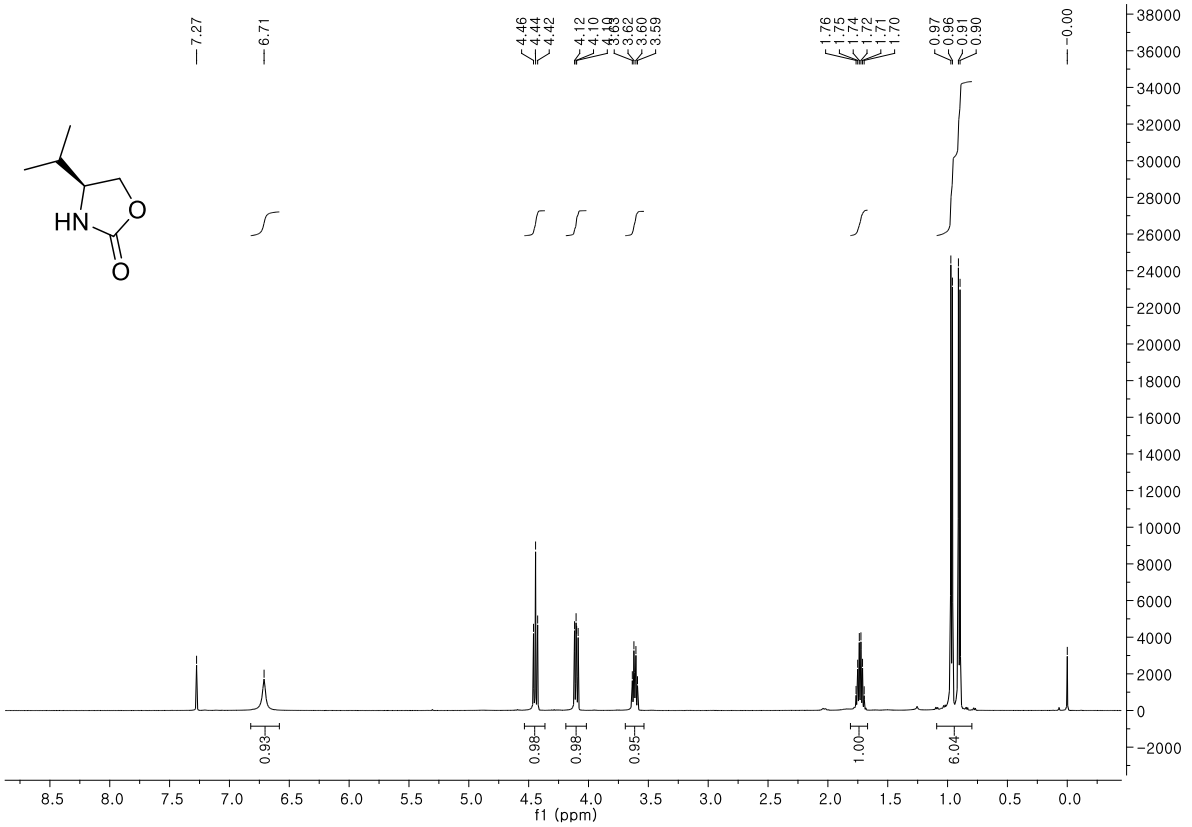

7.34
7.34
7.34
7.33
7.32
7.32
7.31
7.31
7.29
7.28
7.28
7.27
7.27
7.26
7.26
7.25
7.25
7.25
7.24
7.23
7.23
7.22
4.08
4.07
4.06
4.05
3.77
3.19
3.18
3.17
3.16
3.03
3.01
3.00
2.98
0.00
N3
OMe
O
29
5.40
1.00
3.18
1.07
1.07
f1 (ppm)

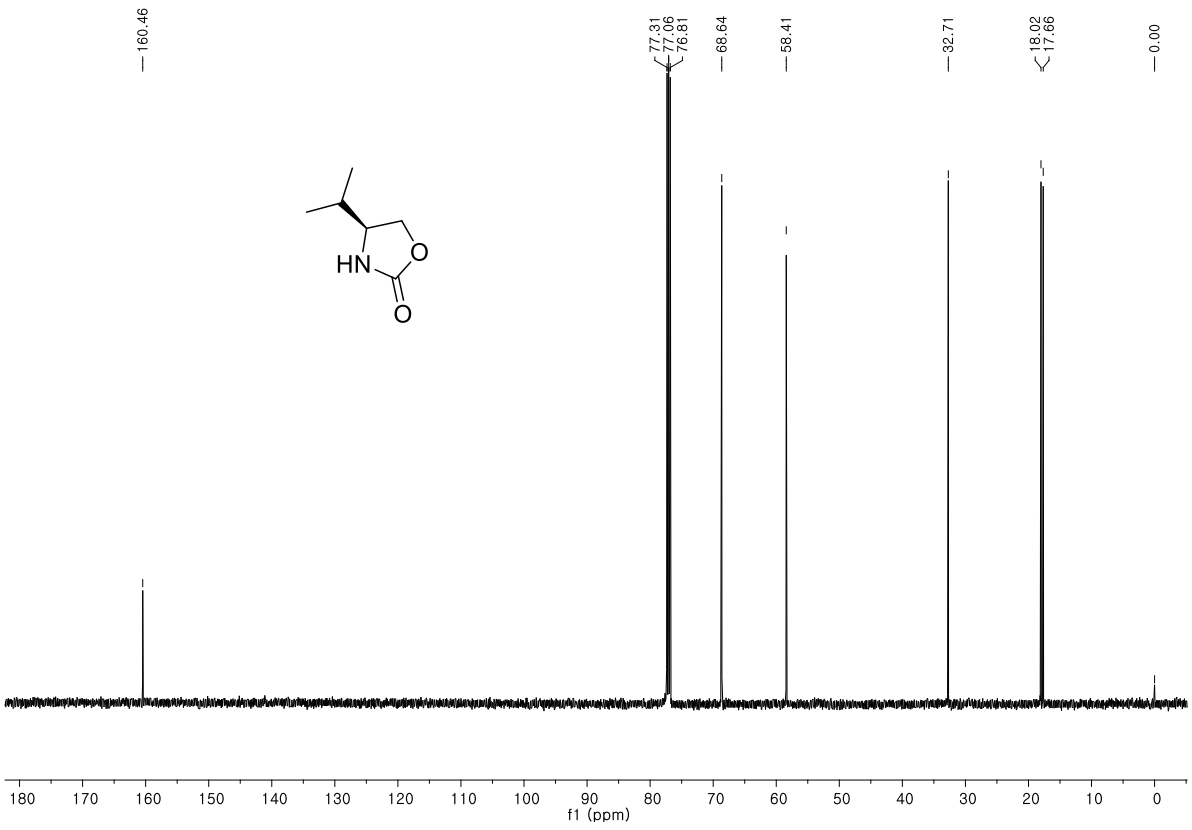

170.40
135.93
129.18
128.71
127.29
77.30
77.05
76.80
63.28
52.64
37.67
0.00
N3
OMe
O
29
f1 (ppm)


7.32
7.30
7.29
7.26
7.25
7.24
7.22
7.20
7.19
7.18
3.75
3.74
3.73
3.72
3.71
3.11
3.10
3.08
3.07
2.88
2.87
2.85
2.84
1.52
0.00
NH2
OMe
O
30
5.00
3.91
1.00
0.95
2.06
f1 (ppm)


175.44
137.24
129.27
128.57
126.84
77.32
77.07
76.82
55.84
51.96
41.11
0.00
NH2
OMe
O
30
f1 (ppm)

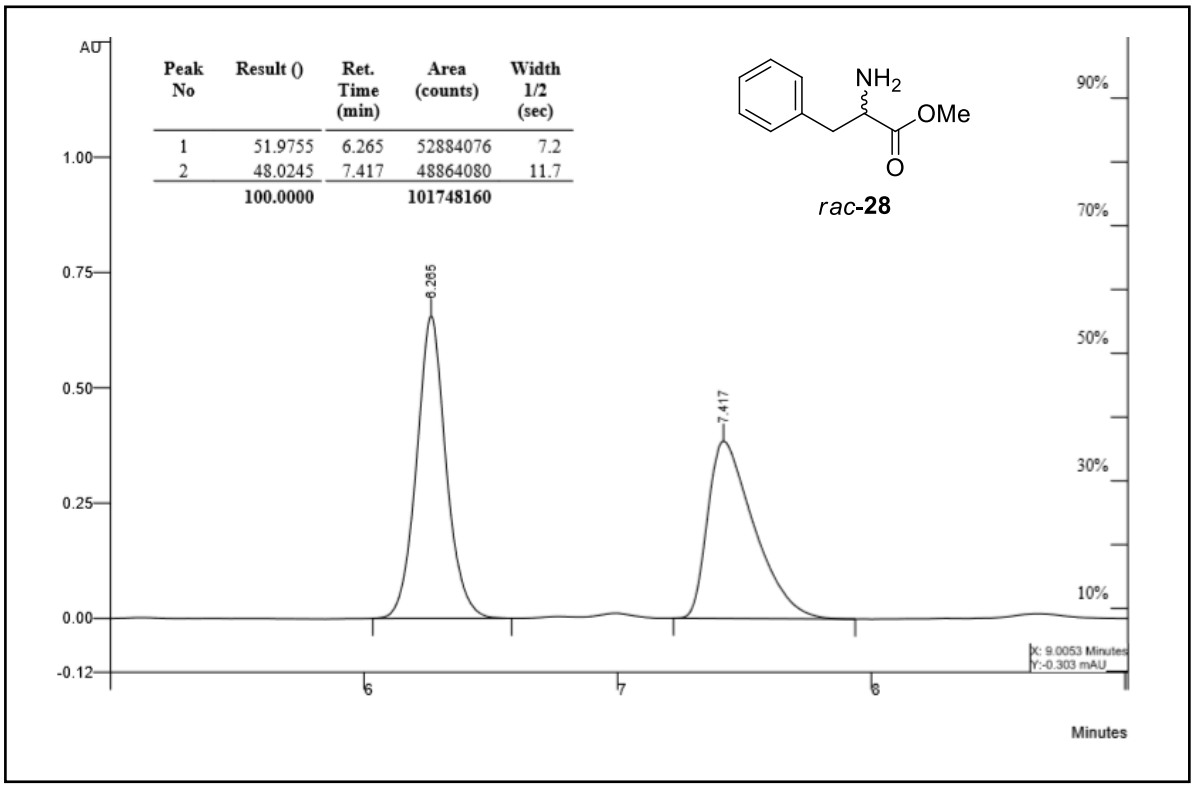
NMR spectra of recovering chiral auxiliary





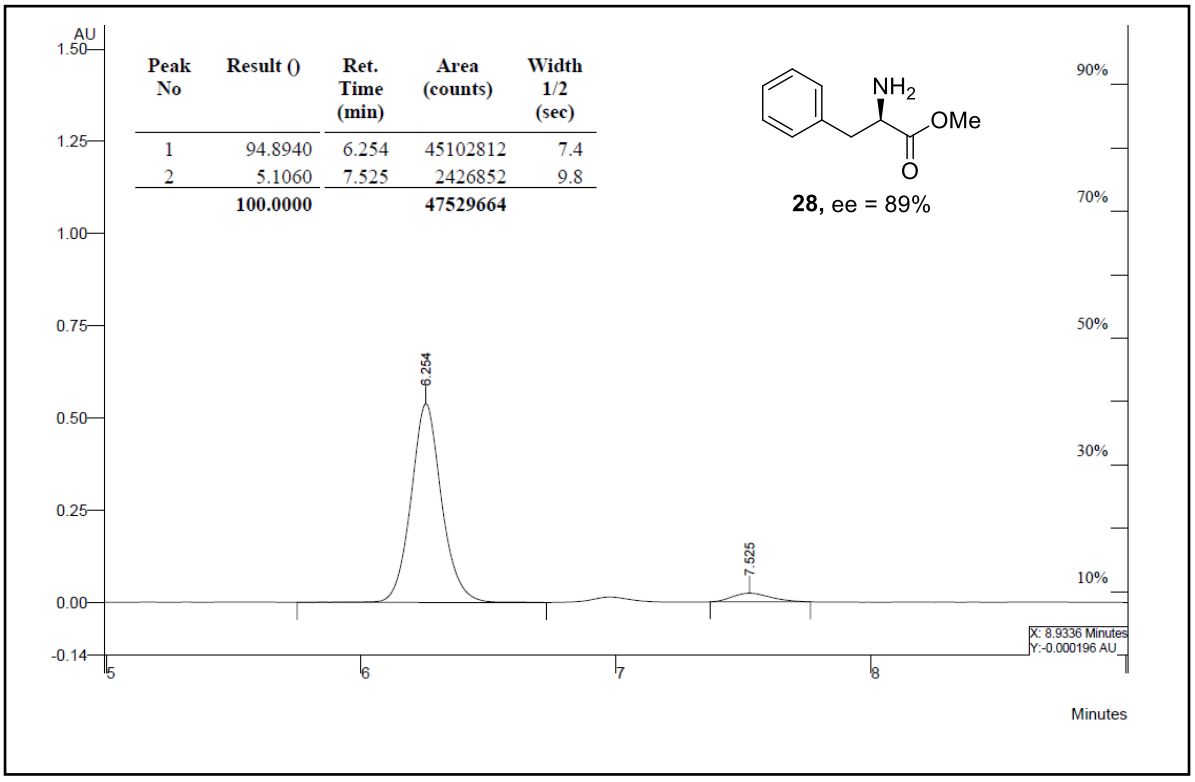
HPLC spectra of chiral products

HPLC spectrum of **racemic 28**



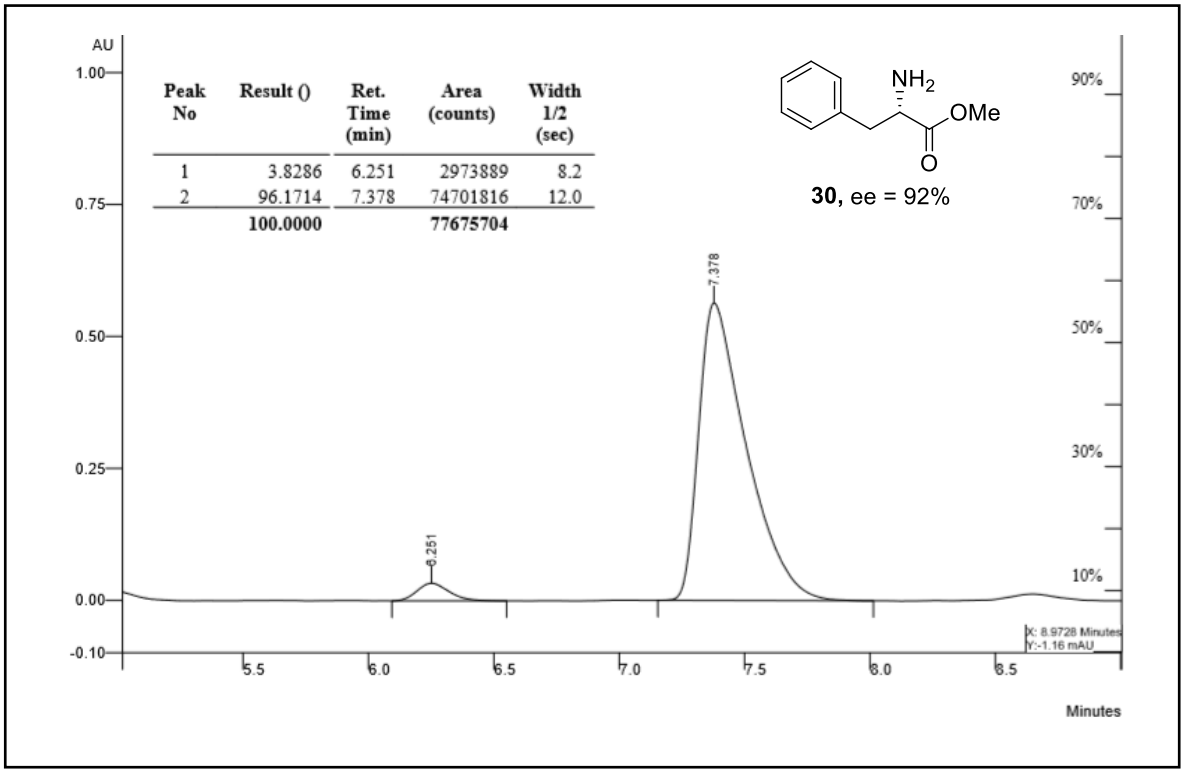
Peak No	Result ()	Ret. Time (min)	Area (counts)	Width 1/2 (sec)
1	51.9755	6.265	52884076	7.2
2	48.0245	7.417	48864080	11.7
	100.0000		**101748160**	

HPLC spectrum of **chiral product 28**



Peak No	Result ()	Ret. Time (min)	Area (counts)	Width 1/2 (sec)
1	94.8940	6.254	45102812	7.4
2	5.1060	7.525	2426852	9.8
	100.0000		**47529664**	

HPLC spectrum of **chiral product 30**



Peak No	Result ()	Ret. Time (min)	Area (counts)	Width 1/2 (sec)
1	3.8286	6.251	2973889	8.2
2	96.1714	7.378	74701816	12.0
	100.0000		**77675704**	