Electronic Supplementary Material (ESI) for Green Chemistry.

Organocatalytic Synthesis of Polysubstituted Tetrahydrofurans from Alkenes

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SUPPORTING INFORMATION

Table of Contents	**Page**

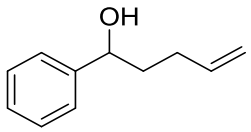
General Remarks

Chromatographic purification of products was accomplished using forced-flow chromatography on Merck® Kieselgel 60 F_{254} 230-400 mesh. Thin-layer chromatography (TLC) was performed on aluminum backed silica plates (0.2 mm, 60 F_{254}). Visualization of the developed chromatogram was performed by fluorescence quenching using phosphomolybdic acid, anisaldehyde or potassium permanganate stains. Melting points were determined on a Buchi® 530 hot stage apparatus and are uncorrected. Mass spectra (ESI) were recorded on a Finningan® Surveyor MSQ LC-MS spectrometer. HRMS spectra were recorded on Bruker® Maxis Impact QTOF spectrometer. ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on Varian® Mercury (200 MHz, 50 MHz and 188 MHz respectively), and are internally referenced to residual solvent signals. Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, bs = broad signal, bs m = broad signal multiplet), coupling constant and assignment. Data for ^{13}C NMR and ^{19}F NMR are reported in terms of chemical shift (δ ppm). Mass spectra and conversions of the reactions were recorded on a Shimadzu® GCMS-QP2010 Plus Gas Chromatograph Mass Spectrometer utilizing a MEGA® column (MEGA-5, F.T : 0.25μm, I.D. : 0.25mm, L : 30m, T_{max} : 350 ºC, Column ID# 11475).

General Procedure for the synthesis of alcohols 1a-1q

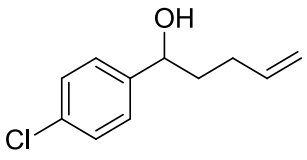
To a stirring solution of the magnesium (84 mg, 7.00 mmol) and I_2 (small crystal), 4-bromo-1-butene (0.71 mL, 7.00 mmol) in anhydrous THF (2 mL) was added slowly at 0 ^oC and the reaction mixture was stirred for 30 min at the same temperature. Aldehyde (2.00 mmol) was dissolved in anhydrous THF (1 mL) and added to the reaction mixture at 0 ^oC. The resulting suspension was stirred for 30 min at 0 ^oC and then overnight at 65 ^oC. The reaction was quenched with saturated aqueous NH_4Cl carefully at 0 ^oC, filtered and extracted with EtOAc (3 x 10 mL). The combined organic layers were washed with brine (30 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. The crude product was purified by column chromatography (5-20% EtOAc in Pet. Ether).

1-Phenylpent-4-en-1-ol (1a)[1]



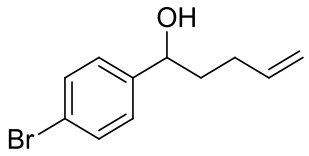
Colorless oil; 71% yield; ^{1}H NMR ($CDCl_3$) δ: 7.37-7.22 (5H, m, ArH), 5.84 (1H, ddt, J = 16.8, 10.2 and 6.5 Hz, =CH), 5.11-4.92 (2H, m, $=CH_2$), 4.71-4.63 (1H, m, CHPh), 2.28-1.67 (5H, m, OH and 2 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 144.5, 138.1, 128.4, 127.5, 125.8, 114.9, 73.9, 38.0, 30.0.

1-(4-Chlorophenyl)pent-4-en-1-ol (1b)[2]



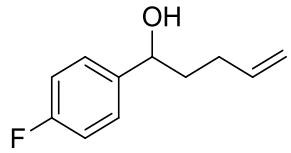
Colorless oil; 76% yield; ^{1}H NMR ($CDCl_3$) δ: 7.27 (2H, d, J = 8.7 Hz, ArH), 7.19 (2H, d, J = 8.7 Hz, ArH), 5.78 (1H, ddt, J = 16.8, 10.2 and 6.5 Hz, =CH), 5.07-4.92 (2H, m, $=CH_2$), 4.63-4.53 (1H, m, CHPh), 2.86 (1H, br s, OH), 2.12-1.92 (2H, m, CH_2) 1.90-1.65 (2H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 142.9, 137.8, 133.0, 128.4, 127.2, 115.1, 73.1, 37.9, 29.8.

1-(4-Bromophenyl)pent-4-en-1-ol (1c)[3]



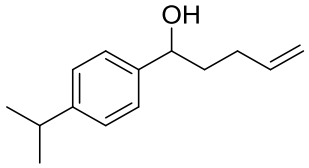
Colorless oil; 70% yield; ^{1}H NMR ($CDCl_3$) δ: 7.46 (2H, d, J = 8.5 Hz, ArH), 7.20 (2H, d, J = 8.5 Hz, ArH), 5.81 (1H, ddt, J = 16.8, 10.2 and 6.5 Hz, =CH), 5.09-4.93 (2H, m, $=CH_2$), 4.70-4.61 (1H, m, CHPh), 2.22-1.64 (5H, m, OH and 2 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 143.4, 137.7, 131.3, 127.5, 121.1, 115.0, 73.0, 37.8, 29.7.

1-(4-Fluorophenyl)pent-4-en-1-ol (1d)[4]



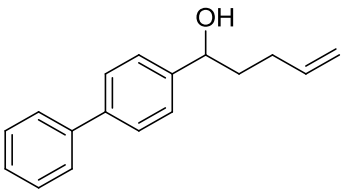
Colorless oil; 68% yield; ^{1}H NMR ($CDCl_3$) δ: 7.30-7.18 (2H, m, ArH), 7.05-6.93 (2H, m, ArH), 5.79 (1H, ddt, J = 16.8, 10.1 and 6.5 Hz, =CH), 5.07-4.92 (2H, m, $=CH_2$), 4.64-4.52 (1H, m, CHPh), 2.68 (1H, br s, OH), 2.17-1.60 (4H, m, 2 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 162.0 (d, J = 244.9 Hz), 140.19 (d, J = 2.8 Hz), 137.9, 127.4 (d, J = 8.0 Hz), 115.1 (d, J = 21.3 Hz), 115.0, 73.1, 38.0, 29.9; ^{19}F NMR ($CDCl_3$) δ: -35.8.

1-(4-Isopropylphenyl)pent-4-en-1-ol (1e)



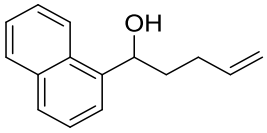
Colorless oil; 68% yield; ^{1}H NMR ($CDCl_3$) δ: 7.27 (2H, d, J = 8.5 Hz, ArH), 7.20 (2H, d, J = 8.5 Hz, ArH), 5.84 (1H, ddt, J = 16.8, 10.1 and 6.4 Hz, =CH), 5.10-4.93 (2H, m, $=CH_2$), 4.71-4.61 (1H, m, CHPh), 2.90 [1H, sept, J = 6.9 Hz, CH(CH_3)$_2$], 2.23-1.98 (2H, m, CH_2), 1.96-1.71 (3H, m, OH and CH_2), 1.24 (6H, d, J = 6.9 Hz, 2 x CH_3); ^{13}C NMR ($CDCl_3$) δ: 148.0, 141.9, 138.2, 126.3, 125.8, 114.7, 73.6, 37.8, 33.7, 30.0, 23.9.

1-([1,1'-Biphenyl]-4-yl)pent-4-en-1-ol (1f)[5]



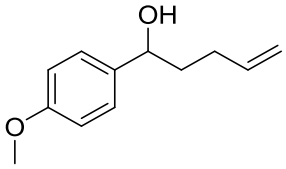
White solid; mp 68-70 ^{o}C; 58% yield; ^{1}H NMR ($CDCl_3$) δ: 7.67-7.52 (4H, m, ArH), 7.50-7.29 (5H, m, ArH), 5.85 (1H, ddt, J = 16.8, 10.1 and 6.5, =CH), 5.15-4.94 (2H, m, $=CH_2$), 4.81-4.68 (1H, m, CHPh), 2.28-2.07 (2H, m, CH_2), 2.02-1.76 (3H, m, OH and 1 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 143.5, 140.7, 140.5, 138.1, 128.7, 127.2, 127.2, 127.0, 126.3, 115.0, 73.7, 38.0, 30.1.

1-(Naphthalen-2-yl)pent-4-en-1-ol (1g)[3]



Colorless oil; 65% yield; ^{1}H NMR ($CDCl_3$) δ: 8.12-8.03 (1H, m, ArH), 7.92-7.81 (1H, m, ArH), 7.79 (1H, d, J = 8.1 Hz, ArH), 7.63 (1H, d, J = 6.7 Hz, ArH), 7.55-7.42 (3H, m, ArH), 5.91 (1H, ddt, J = 16.7, 10.1 and 6.5 Hz, =CH), 5.45 (1H, t, J = 5.5 Hz, CHPh), 5.17-4.96 (2H, m, $=CH_2$), 2.36-2.20 (3H, m, OH and 1 x CH_2), 2.08-1.92 (2H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 140.3, 138.1, 133.7, 130.2, 128.8, 127.8, 125.9, 125.4, 125.4, 123.1, 122.8, 115.1, 70.4, 37.2, 30.3.

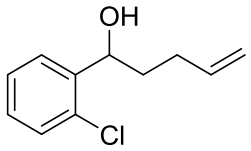
1-(4-Methoxyphenyl)pent-4-en-1-ol (1h)[3]



Colorless oil; 85% yield; ^{1}H NMR ($CDCl_3$) δ: 7.20 (2H, d, J = 8.7 Hz, ArH), 6.83 (2H, d, J = 8.7 Hz, ArH), 5.80 (1H, ddt, J = 16.8, 10.2 and 6.4 Hz, =CH), 5.07-4.91 (2H, m, $=CH_2$), 4.54 (1H, t, J = 6.6 Hz, CHPh), 3.76 (3H, s, CH_3O), 2.76 (1H, br s,

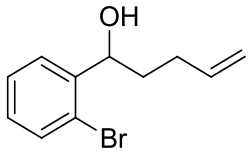
OH), 2.18-1.58 (4H, m, 4 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 158.7, 138.1, 136.6, 127.0, 114.6, 113.5, 73.2, 55.0, 37.7, 29.9.

1-(2-Chlorophenyl)pent-4-en-1-ol (1i)



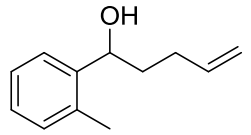
Colorless oil; 69% yield; ^{1}H NMR ($CDCl_3$) δ: 7.51 (1H, d, J = 6.8 Hz, ArH), 7.34-7.11 (3H, m, ArH), 5.86 (1H, ddt, J = 15.9, 9.9 and 6.5 Hz, =CH), 5.20-4.93 (3H, m, CHPh and =CH_2), 2.34-2.15 (2H, m, CH_2), 2.03 (1H, br s, OH), 1.95-1.65 (2H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 142.0, 138.0, 131.6, 129.3, 128.3, 127.0, 126.9, 115.0, 70.0, 36.5, 29.9; HRMS exact mass calculated for $[M+Na]^+$ ($C_{11}H_{13}ClNaO$) requires *m/z* 219.0547, found *m/z* 219.0550.

1-(2-Bromophenyl)pent-4-en-1-ol (1j)



Colorless oil; 66% yield; ^{1}H NMR ($CDCl_3$) δ: ^{1}H NMR ($CDCl_3$) δ 7.51 (2H, m, ArH), 7.31 (1H, t, J = 7.5 Hz, ArH), 7.10 (1H, t, J = 7.5 Hz, ArH), 5.86 (1H, ddt, J = 16.0, 10.2 and 6.5 Hz, =CH), 5.13-4.95 (3H, m, CHPh and =CH_2), 2.51 (1H, s, OH), 2.37-2.05 (2H, m, CH_2), 1.96-1.60 (2H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 144.5, 138.0, 132.5, 128.7, 127.6, 127.2, 121.8, 115.0, 72.3, 36.5, 30.0; HRMS exact mass calculated for $[M+Na]^+$ ($C_{11}H_{13}BrNaO$) requires *m/z* 263.0042, found *m/z* 263.0041.

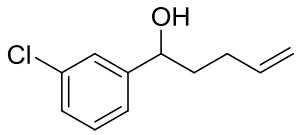
1-(o-Tolyl)pent-4-en-1-ol (1k)[6]



Colorless oil; 79% yield; ^{1}H NMR ($CDCl_3$) δ: 7.47 (1H, dd, J = 8.0 and 1.5 Hz, ArH), 7.31-7.11 (3H, m, ArH), 5.95-5.81 (1H, m, =CH), 5.17-5.00 (2H, m, =CH_2), 4.89 (1H,

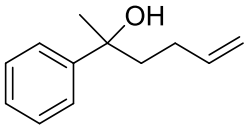
t, *J* = 6.3 Hz, CHAr), 2.96 (1H, br s, OH), 2.33 (3H, s, CH_3), 2.29-2.08 (2H, m, 1 x CH_2), 1.88-1.72 (2H, m, 1 x CH_2); ^{13}C NMR ($CDCl_3$) *δ*: 142.6, 138.0, 134.1, 130.0, 126.8, 126.0, 125.1, 114.7, 69.6, 36.8, 30.0, 18.8.

1-(3-Chlorophenyl)pent-4-en-1-ol (1l)[7]



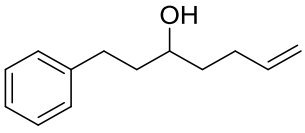
Colorless oil; 89% yield; ^{1}H NMR ($CDCl_3$) *δ*: 7.40-7.07 (4H, m, ArH), 5.75 (1H, dt, J = 16.7, 10.1 and 6.5 Hz, =CH), 5.06-4.89 (2H, m, =CH_2), 4.52 (1H, t, *J* = 6.2 Hz, CHAr), 3.46 (1H, br s, OH), 2.15-1.94 (2H, m, 1 x CH_2), 1.85-1.67 (2H, m, 1 x CH_2); ^{13}C NMR ($CDCl_3$) *δ*: 146.6, 137.7, 134.0, 129.5, 127.3, 125.9, 123.9, 114.9, 72.9, 37.8, 29.6.

2-Phenylhex-5-en-2-ol (1m)[8]



Colorless oil; 63% yield; ^{1}H NMR ($CDCl_3$) *δ*: 7.48-7.18 (5H, m, ArH), 5.79 (1H, ddt, *J* = 16.8, 10.1 and 6.5 Hz, =CH), 5.0.5-4.87 (2H, m, =CH_2), 2.11-1.85 (5H, m, OH and 2 x CH_2), 1.56 (3H, s, CH_3); ^{13}C NMR ($CDCl_3$) *δ*: 147.5, 138.6, 128.1, 126.5, 124.7, 114.5, 74.6, 42.9, 30.2, 28.4.

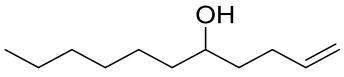
1-Phenylhept-6-en-3-ol (1n)[9]



Colorless oil; 45% yield; ^{1}H NMR ($CDCl_3$) *δ*: 7.36-7.15 (5H, m, ArH), 5.85 (1H, ddt, *J* = 16.9, 10.1 and 6.6 Hz, =CH), 5.12-4.92 (2H, m, =CH_2), 3.74-3.59 (1H, m, OCH), 2.93-1.59 (2H, m, CH_2Ph), 2.28-2.03 (2H, m, CH_2), 1.86-1.51 (5H, m, OH and 2 x

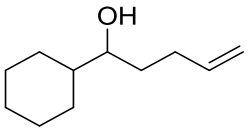
CH_2); ^{13}C NMR ($CDCl_3$) δ: 142.0, 138.4, 128.3, 125.7, 114.8, 70.8, 39.0, 36.5, 32.0, 30.0.

Undec-1-en-5-ol (1o)[10]



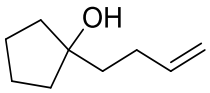
Colorless oil; 52% yield; ^1H NMR ($CDCl_3$) δ: 5.82 (1H, ddt, J = 16.8, 10.1 and 6.6 Hz, =CH), 5.09-4.89 (2H, m, $=CH_2$), 3.68-3.53 (1H, m, OCH), 2.25-2.00 (2H, m, CH_2), 1.65-1.12 (13H, m, OH and 6 x CH_2), 0.86 (3H, t, J = 6.2 Hz, CH_3); ^{13}C NMR ($CDCl_3$) δ: 138.6, 114.6, 71.4, 37.4, 36.4, 31.8, 30.0, 29.3, 25.6, 22.6, 14.0.

1-Cyclohexylpent-4-en-1-ol (1p)[11]



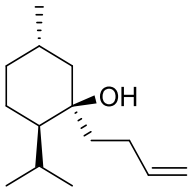
Colorless oil; 80% yield; 1:1 mixture of diastereomers; ^1H NMR ($CDCl_3$) δ: 5.77 (1H, ddt, J = 16.7, 10.1 and 6.6 Hz, =CH), 5.05-4.84 (2H, m, $=CH_2$), 3.36-3.17 (1H, m, OCH), 2.29-1.91 (2H, m, OH and CH_2), 1.65-1.12 (13H, m, CH and 6 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 138.7, 114.3, 75.3, 43.5, 33.0, 30.2, 29.0, 27.7, 26.4, 26.2, 26.1.

1-(But-3-en-1-yl)cyclopentan-1-ol (1q)



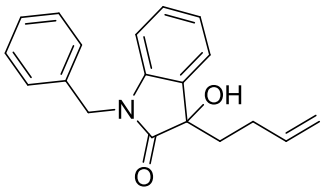
Colorless oil; 48% yield; ^1H NMR ($CDCl_3$) δ: 5.83 (1H, ddt, J = 16.8, 10.1 and 6.6 Hz, =CH), 5.07-4.83 (2H, m, $=CH_2$), 2.24-2.05 (2H, =CHCH_2), 1.90-1.42 (11H, m, OH and 5 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 139.2, 114.2, 82.3, 40.4, 39.5, 29.2, 23.6; HRMS exact mass calculated for $[M+Na]^+$ ($C_9H_{16}NaO$) requires *m/z* 163.1093, found *m/z* 163.1093.

(1*R*,2*R*,5*S*)-1-(But-3-en-1-yl)-2-isopropyl-5-methylcyclohexan-1-ol (1r)



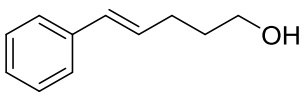
Colorless oil; 52% yield; $[\alpha]_D$ = +3.1 (c=1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ: 5.79 (1H, ddt, *J* = 16.6, 10.0 and 6.4 Hz, =CH), 5.07-4.83 (2H, m, $=CH_2$), 2.14-1.94 (3H, OH and =CHC*H*$_2$), 1.79-1.20 (8H, m, 3 x CH and 5 x C*H*H), 1.12-0.73 (12H, m, 3 x C*H*H and 3 x CH_3); ^{13}C NMR ($CDCl_3$) δ: 138.9, 114.2, 74.9, 47.8, 46.6, 40.1, 35.0, 28.3, 27.9, 25.4, 23.5, 22.4, 20.4, 18.1; HRMS exact mass calculated for $[M+Na]^+$ ($C_{14}H_{26}NaO$) requires *m/z* 233.1876, found *m/z* 233.1881.

1-Benzyl-3-(but-3-en-1-yl)-3-hydroxyindolin-2-one (1s)



Yellow solid; mp 92-95 ºC; 34% yield; ^{1}H NMR ($CDCl_3$) δ: 7.45-7.00 (8H, m, ArH), 6.72 (1H, d, *J* = 7.6 Hz, ArH), 5.71 (1H, ddt, *J* = 12.6, 10.2 and 6.3 Hz, =CH), 5.03-4.62 (4H, m, $PhCH_2$ and $=CH_2$), 3.90 (1H, s, OH), 2.21-1.84 (4H, m, 2 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 180.1, 142.3, 138.6, 138.5, 134.3, 129.1, 128.2, 127.6, 127.6, 125.6, 123.9, 114.3, 111.0, 73.9, 44.2, 38.1, 30.3; HRMS exact mass calculated for $[M+Na]^+$ ($C_{19}H_{19}NNaO_2$) requires *m/z* 316.1318, found *m/z* 316.1310.

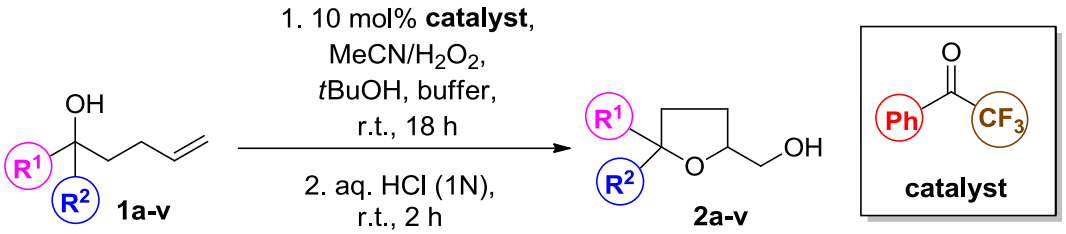
(*E*)-5-phenylpent-4-en-1-ol (1v)[12]



^{1}H NMR ($CDCl_3$) δ: 7.43-7.13 (5H, m, ArH), 6.43 (1H, d, *J* = 15.9 Hz, PhCH), 6.23 (1H, dt, *J* = 15.9 and 6.8 HZ, =CH), 3.70 (2H, t, *J* = 6.3 Hz, OCH_2), 2.30 (2H, q, *J* =

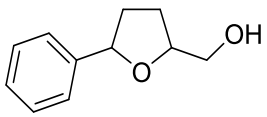
6.8 Hz, =CHC*H*$_2$), 1.84-1.67 (3H, m, OH and 1 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 137.6, 130.3, 130.0, 128.5, 126.9, 125.9, 62.4, 32.2, 29.3.

One-pot Organocatalytic Oxidative Synthesis of Tetrahydrofuranyl derivatives from Alkenes



Alkene (0.50 mmol) was placed in a round bottom flask and dissolved in *tert*-butanol (0.4 mL). 2,2,2-Trifluoro-1-phenylethanone (8.7 mg, 0.05 mmol), aqueous buffer solution (0.4 mL, 0.6M K_2CO_3 - 4x10^{-4}M EDTA disodium salt), acetonitrile (0.40 mL, 8.00 mmol) and 30% aqueous H_2O_2 (0.84 mL, 8.00 mmol) were added consecutively. The reaction mixture was left stirring for 18 hours at room temperature and then aqueous HCl 1N (1.0 mL, 1.00 mmol) was added and the reaction mixture was stirred for 2 hours. Then, EtOAc (20 mL) was added into the mixture and the organic layer was washed with HCl 1N (10 mL) and brine (10 mL), dried over Na_2SO_4, filtered and concentrated *in vacuo*. If the product required further purification, filtration through a short silica plug was performed (30-50% EtOAc in Pet. Ether).

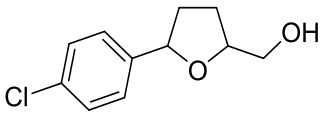
(5-Phenyltetrahydrofuran-2-yl)methanol (2a)[13]



Colorless oil; 94% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.43-7.17 (5H, m, ArH), 5.04-4.86 (1H, m, CHPh), 4.44-4.29 (0.5H, m, OCH), 4.27-4.13 (0.5H, m, OCH), 3.85-3.53 (2H, m, OCH_2), 2.45-2.23 (1H, m, C*H*H), 2.17-1.96 (1H, m, C*H*H), 1.95-1.74 (2H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 142.4, 141.9, 128.3, 128.3,

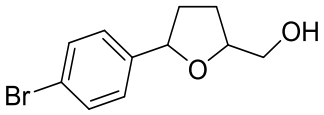
127.5, 127.4, 125.9, 125.6, 81.6, 81.0, 80.3, 80.1, 65.3, 65.0, 35.2, 34.0, 27.7, 27.4; MS (ESI) 201 [$(M+Na)^+$, 100%].

(5-(4-Chlorophenyl)tetrahydrofuran-2-yl)methanol (2b)



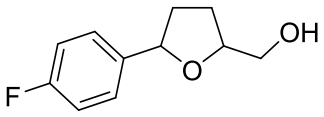
Colorless oil; 81% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.33-7.17 (4H, m, ArH), 4.96-4.79 (1H, m, CHPh), 4.40-4.23 (0.5H, m, OCH), 4.22-3.88 (1.5H, m, OH and OCH), 3.78-3.47 (2H, m, OCH_2), 2.40-2.18 (1H, m, C*H*H), 2.13-1.93 (1H, m, C*H*H), 1.89-1.65 (2H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 141.2, 140.7, 133.0, 132.8, 128.4, 128.4, 127.2, 126.9, 80.7, 80.3, 80.2, 80.1, 65.1, 64.8, 35.4, 34.2, 27.7, 27.3; HRMS exact mass calculated for $[M+Na]^+$ ($C_{11}H_{13}ClNaO_2$) requires *m/z* 235.0496, found *m/z* 235.0491.

(5-(4-Bromophenyl)tetrahydrofuran-2-yl)methanol (2c)



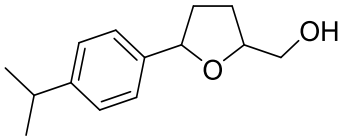
Colorless oil; 90% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.57-7.38 (2H, m, ArH), 7.23-7.12 (2H, m, ArH), 4.96-4.77 (1H, m, CHPh), 4.38-4.22 (0.5H, m, OCH), 4.21-4.05 (0.5H, m, OCH), 3.80-3.45 (2H, m, OCH_2), 2.47 (1H, br s, OH), 2.36-2.18 (1H, m, C*H*H), 2.11-1.91 (1H, m, C*H*H), 1.90-1.65 (2H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 141.9, 141.4, 131.3, 131.3, 127.5, 127.2, 121.1, 120.9, 80.7, 80.2, 80.4, 80.1, 65.2, 64.9, 35.4, 34.2, 27.7, 27.3; HRMS exact mass calculated for $[M+Na]^+$ ($C_{11}H_{13}BrNaO_2$) requires *m/z* 278.9991, found *m/z* 278.9990.

(5-(4-Fluorophenyl)tetrahydrofuran-2-yl)methanol (2d)[13]



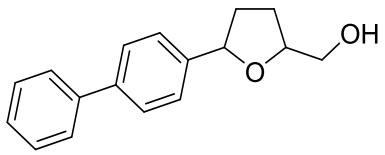
Colorless oil; 85% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.46-7.18 (2H, m, ArH), 7.08-6.91 (2H, m, ArH), 4.99-4.78 (1H, m, CHPh), 4.38-4.22 (0.5H, m, OCH), 4.21-4.06 (0.5H, m, OCH), 3.81-3.45 (2H, m, OCH_2), 2.70 (1H, br s, OH), 2.40-2.17 (1H, m, C*H*H), 2.12-1.89 (1H, m, C*H*H), 1.85-1.67 (2H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 162.1 (d, J = 245.0 Hz), 162.0 (d, J = 245.0 Hz), 138.4 (d, J = 3.0 Hz), 137.9 (d, J = 3.1 Hz), 127.5 (d, J = 8.1 Hz), 127.2 (d, J = 8.0 Hz), 115.1 (d, J = 21.4 Hz), 115.1 (d, J = 21.4 Hz), 80.9, 80.3, 80.2, 80.0, 65.2, 64.9, 35.4, 34.2, 27.8, 27.4; ^{19}F NMR ($CDCl_3$) δ: -35.67 – -25.93 (m), -35.96 – -35.22 (m); MS (ESI) 219 [$(M+Na)^+$, 100%].

(5-(4-Isopropylphenyl)tetrahydrofuran-2-yl)methanol (2e)



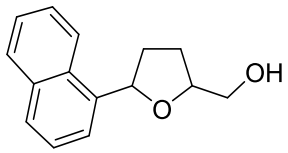
Colorless oil; 91% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.32-7.14 (4H, m, ArH), 5.00-4.83 (1H, m, CHPh), 4.42-4.27 (0.5H, m, OCH), 4.23-4.15 (0.5H, m, OCH), 3.85-3.49 (2H, m, OCH_2), 2.90 [1H, sept, J = 6.9 Hz, C*H*$(CH_3)_2$], 2.71 (1H, br s, OH), 2.39-2.19 (1H, m, C*H*H), 2.13-1.68 (3H, m, CH_2 and C*H*H), 1.23 (6H, d, J = 6.9 Hz, 2 x CH_3); ^{13}C NMR ($CDCl_3$) δ: 148.1, 148.0, 139.9, 139.3, 126.3, 126.3, 126.0, 125.7, 81.4, 80.8, 80.0, 79.8, 65.2, 65.0, 35.1, 33.9, 33.7, 27.8, 27.5, 23.9; HRMS exact mass calculated for $[M+Na]^+$ ($C_{14}H_{20}NaO_2$) requires *m/z* 243.1356, found *m/z* 243.1351.

(5-([1,1'-Biphenyl]-4-yl)tetrahydrofuran-2-yl)methanol (2f)[13]



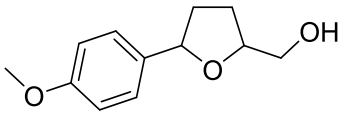
Colorless oil; 88% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.70-7.53 (4H, m, ArH), 7.51-7.30 (5H, m, ArH), 5.10-4.92 (1H, m, CHPh), 4.47-4.33 (0.5H, m, OCH), 4.29-4.15 (0.5H, m, OCH), 3.89-3.55 (2H, m, OCH_2), 2.72 (1H, br s, OH), 2.47-2.26 (1H, m, C*H*H), 2.16-1.75 (3H, m, CH_2 and C*H*H); ^{13}C NMR ($CDCl_3$) δ: 141.8, 141.2, 140.7, 140.7, 140.3, 140.1, 128.6, 128.6, 127.1, 127.1, 127.0, 127.0, 126.9, 126.9, 126.3, 126.0, 81.2, 80.6, 80.2, 80.0, 65.2, 64.9, 35.3, 34.1, 27.8, 27.4; MS (ESI) 277 [$(M+Na)^+$, 100%].

(5-(Naphthalen-1-yl)tetrahydrofuran-2-yl)methanol (2g)[13]



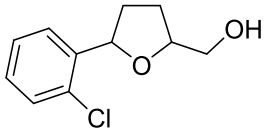
Orange oil; 55% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 8.03-7.57 (4H, m, ArH), 7.56-7.26 (3H, m, ArH), 5.89-5.57 (1H, m, CHPh), 4.56-4.40 (0.5H, m, OCH), 4.35-4.20 (0.5H, m, OCH), 3.92-3.57 (2H, m, OCH_2), 2.68-2.47 (1H, m, C*H*H), 2.31 (1H, br s, OH), 2.18-1.75 (3H, m, CH_2); ^{13}C NMR ($CDCl_3$) δ: 138.7, 138.1, 133.6, 133.6, 130.4, 128.8, 128.7, 127.7, 127.5, 125.8, 125.8, 125.4, 125.4, 123.3, 123.3, 121.9, 121.6, 80.0, 78.3, 78.2, 65.3, 65.1, 34.3, 33.3, 27.6, 27.4; MS (ESI) 251 [$(M+Na)^+$, 100%].

(5-(4-Methoxyphenyl)tetrahydrofuran-2-yl)methanol (2h)[13]



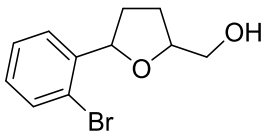
Colorless oil; 57% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.32-7.15 (2H, m, ArH), 6.90-6.72 (2H, m, ArH), 4.97-4.75 (1H, m, CHPh), 4.39-4.23 (0.5H, m, OCH), 4.22-4.03 (0.5H, m, OCH), 3.87-3.46 (5H, m, OCH_2 and CH_3), 2.36-2.13 (1H, m, C*H*H), 2.36-1.65 (4H, m, 3 x C*H*H, and OH); ^{13}C NMR ($CDCl_3$) δ: 158.9, 158.8, 134.4, 133.9, 127.3, 127.0, 113.6, 113.6, 81.3, 80.6, 79.9, 79.7, 65.1, 64.9, 55.2, 35.2, 33.9, 27.8, 27.4; MS (ESI) 231 [$(M+Na)^+$, 100%].

(5-(2-Chlorophenyl)tetrahydrofuran-2-yl)methanol (2i)



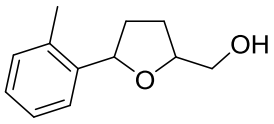
Colorless oil; 94% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.58-7.46 (1H, m, ArH), 7.33-7.09 (3H, m, ArH), 5.33-5.17 (1H, m, CHPh), 4.47-4.32 (0.5H, m, OCH), 4.24-4.09 (0.5H, m, OCH), 3.87-3.44 (2H, m, OCH_2), 2.76 (1H, br s, OH), 2.62-2.41 (1H, m, CH*H*), 2.12-1.55 (3H, m, C*H*H and CH_2); ^{13}C NMR ($CDCl_3$) δ: 140.8, 140.4, 138.8, 138.5, 129.2, 129.2, 128.2, 128.1, 126.8, 126.8, 126.3, 126.1, 80.5, 80.2, 78.1, 78.0, 65.2, 64.9, 33.6, 32.8, 27.5, 27.2; HRMS exact mass calculated for $[M+Na]^+$ ($C_{11}H_{13}ClNaO_2$) requires *m/z* 235.0496, found *m/z* 235.0489.

(5-(2-Bromophenyl)tetrahydrofuran-2-yl)methanol (2j)



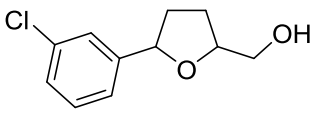
Colorless oil; 75% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.59-7.45 (2H, m, ArH), 7.36-7.24 (1H, m, ArH), 7.15-7.04 (1H, m, ArH), 5.30-5.13 (1H, m, CHPh), 4.49-4.25 (0.5H, m, OCH), 4.26-4.12 (0.5H, m, OCH), 3.88-3.53 (2H, m, OCH_2), 2.68-2.43 (1H, m, C*H*H), 2.31 (1H, br s, OH), 2.13-1.55 (3H, m, C*H*H and CH_2); ^{13}C NMR ($CDCl_3$) δ: 142.5, 142.0, 132.5, 132.5, 128.6, 128.5, 127.5, 127.4, 126.6, 126.4, 121.5, 121.2, 80.6, 80.2, 80.1, 80.1, 65.3, 65.0, 33.8, 33.1, 27.5, 27.2; HRMS exact mass calculated for $[M+Na]^+$ ($C_{11}H_{13}BrNaO_2$) requires *m/z* 278.9991, found *m/z* 278.9987.

(5-(o-Tolyl)tetrahydrofuran-2-yl)methanol (2k)



Colorless oil; 79% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.49-7.38 (1H, m, ArH), 7.26-7.00 (3H, m, ArH), 5.24-5.04 (1H, m, CHPh), 4.48-4.34 (0.5H, m, OCH), 4.25-4.11 (0.5H, m, OCH), 3.87-3.54 (2H, m, OCH_2), 2.93 (1H, br s, OH), 2.50-2.19 (4H, m, C*H*H and CH_3), 2.14-1.58 (3H, m, C*H*H and CH_2); ^{13}C NMR ($CDCl_3$) δ: 140.6, 139.9, 130.2, 127.2, 127.0, 126.1, 124.6, 124.4, 80.1, 79.9, 78.5, 78.3, 65.3, 64.9, 33.6, 32.6, 27.6, 27.3, 19.1; HRMS exact mass calculated for $[M+Na]^+$ ($C_{12}H_{16}NaO_2$) requires *m/z* 215.1043, found *m/z* 215.1042.

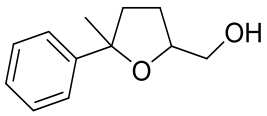
(5-(3-Chlorophenyl)tetrahydrofuran-2-yl)methanol (2l)



Colorless oil; 89% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.35-7.12 (4H, m, ArH), 4.98-4.79 (1H, m, CHPh), 4.39-4.26 (0.5H, m, OCH), 4.22-4.08 (0.5H, m, OCH), 3.80-3.49 (2H, m, OCH_2), 2.56-2.20 (1H, m, OH and C*H*H), 2.12-1.57 (3H, m, C*H*H and CH_2); ^{13}C NMR ($CDCl_3$) δ: 144.9, 144.3, 134.1, 134.0, 129.6, 127.5, 127.3, 125.8, 125.5, 124.0, 123.6, 80.7, 80.40, 80.2, 80.1, 65.1, 64.8, 35.2, 34.0, 27.6,

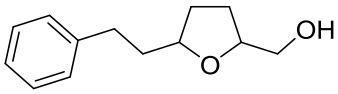
27.2; HRMS exact mass calculated for $[M+Na]^+$ ($C_{11}H_{13}ClNaO_2$) requires *m/z* 235.0496, found *m/z* 235.0490.

(5-Methyl-5-phenyltetrahydrofuran-2-yl)methanol (2m)[14]



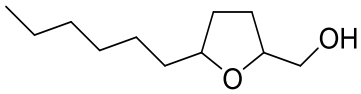
Colorless oil; 98% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.46-7.17 (5H, m, ArH), 4.38-4.25 (0.5H, m, OCH), 4.24-4.05 (0.5H, m, OCH), 3.87-3.47 (2H, m, OCH_2), 2.35-1.96 (3H, m, 3 x CH*H*), 1.79-1.62 (1H, m, C*H*H), 1.54 (1.5H, s, CH_3), 1.52 (1.5H, s, CH_3); ^{13}C NMR ($CDCl_3$) δ: 148.0, 147.4, 128.3, 127.9, 126.3, 126.3, 124.4, 124.4, 85.2, 84.9, 79.5, 79.1, 65.4, 65.1, 39.1, 39.1, 30.2, 29.4, 27.4, 27.2; MS (ESI) 215 [(M+Na)+, 100%].

(5-Phenethyltetrahydrofuran-2-yl)methanol (2n)[15]



Yellow oil; 84% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.33-7.10 (5H, m, ArH), 4.21-3.82 (2H, m, 2 x OCH), 3.77-3.60 (1H, m, OC*H*H), 3.53-3.42 (1H, m, OC*H*H), 2.84-2.56 (2H, m, CH_2Ph), 2.17-1.42 (7H, m, OH and 3 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 141.8, 128.3, 125.7, 79.4, 79.0, 78.7, 65.2, 64.9, 37.4, 37.2, 32.4, 31.9, 31.2, 27.4, 26.9; MS (ESI) 229 [$(M+Na)^+$, 100%].

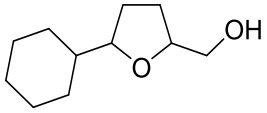
(5-Hexyltetrahydrofuran-2-yl)methanol (2o)[16]



Colorless oil; 92% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 4.15-3.76 (2H, m, 2 x OCH), 3.71-3.40 (2H, m, OCH_2), 2.44 (1H, br s, OH), 2.06-1.10 (14H, m, 7 x CH_2), 0.84 (3H, t, J = 6.5 Hz, CH_3); ^{13}C NMR ($CDCl_3$) δ: 80.2, 79.5, 79.2, 78.9,

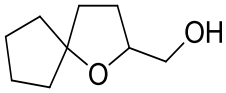
65.2, 65.0, 35.8, 35.7, 31.9, 31.7, 31.3, 29.3, 27.5, 27.0, 26.1, 26.1, 22.5, 14.0; MS (ESI) 209 [$(M+Na)^+$, 100%].

(5-Cyclohexyltetrahydrofuran-2-yl)methanol (2p)



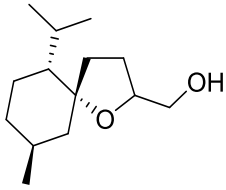
Colorless oil; 78% yield; 1:1 mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 4.13-3.91 (1H, m, OCH), 3.71-3.38 (3H, m, OCH and OCH_2), 2.22-0.81 (16H, m, OH, CH and 7 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 84.4, 83.8, 79.1, 78.9, 65.1, 64.7, 42.8, 42.8, 29.7, 29.4, 28.7, 28.6, 27.5, 27.0, 26.3, 25.8, 25.7; HRMS exact mass calculated for $[M+Na]^+$ ($C_{11}H_{20}NaO_2$) requires *m/z* 207.1356, found *m/z* 207.1352.

(1-Oxaspiro[4.4]nonan-2-yl)methanol (2q)[14]



Pale yellow oil; 70% yield; ^{1}H NMR ($CDCl_3$) δ: ^{13}C NMR ($CDCl_3$) δ: 4.13-3.97 (1H, m, OCH), 3.65 (IH, dd, J = 11.7, 3.2 Hz, OC*H*H), 3.48 (1H, dd, J = 11.7, 6.1 Hz, OC*H*H), 2.03-1.38 (13H, m, OH and 6 x CH_2); ^{13}C NMR ($CDCl_3$) δ: 92.4, 78.8, 65.4, 38.6, 37.9, 36.3, 27.5, 23.8; MS (ESI) 179 [$(M+Na)^+$, 100%].

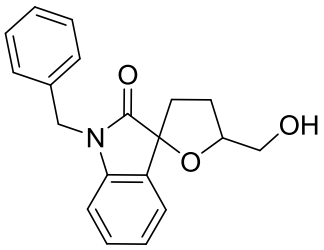
((5*R*,6*R*,9*S*)-6-Isopropyl-9-methyl-1-oxaspiro[4.5]decan-2-yl)methanol (2r)



Colorless oil; 1:1 mixture of diastereomers; 85% yield; ^{1}H NMR ($CDCl_3$) δ: 4.11-3.88 (1H, m, OCH), 3.75-3.33 (2H, m, OCH_2), 2.23-1.35 (12H, m, OH and 3 x CH and 8 x

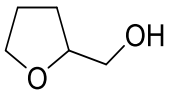
C*H*H), 1.15-0.97 (2H, m, C*H*H), 0.93-0.75 (9H, m, 3 x CH_3); ^{13}C NMR ($CDCl_3$) δ: 86.2, 85.9, 79.7, 77.8, 65.3, 50.5, 49.2, 48.8, 47.2, 35.9, 35.2, 35.2, 34.9, 28.9, 28.7, 28.1, 27.3, 26.0, 25.9, 24.0, 23.7, 22.4, 22.3, 22.3, 21.9, 18.7, 17.7; HRMS exact mass calculated for $[M+Na]^+$ ($C_{14}H_{26}NaO_2$) requires *m/z* 249.1825, found *m/z* 249.1818.

1'-Benzyl-5-(hydroxymethyl)-4,5-dihydro-3H-spiro[furan-2,3'-indolin]-2'-one (2s)



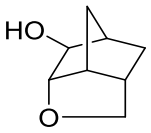
Yellow solid; 62% yield; (insoluble to reaction mixture; 0.2 mL EtOAc was added); mixture of diastereomers; ^{1}H NMR ($CDCl_3$) δ: 7.45-6.97 (8H, m, ArH), 6.76-6.64 (1H, m, ArH), 5.03-4.62 (3H, m, CH_2Ph and OCH), 4.05-3.36 (2H, m, OCH_2), 2.72-1.48 (5H, m, 2 x CH_2 and OH); ^{13}C NMR ($CDCl_3$) δ: 180.3, 180.3, 142.3, 142.3, 135.7, 135.5, 129.9, 129.9, 129.1, 129.1, 128.0, 127.9, 127.4, 123.8, 123.8, 123.6, 123.5, 109.9, 109.7, 83.9, 83.8, 83.7, 82.1, 65.1, 64.4, 44.1, 43.9, 38.1, 36.3, 27.8, 27.3; HRMS exact mass calculated for $[M+Na]^+$ ($C_{19}H_{19}NNaO_3$) requires *m/z* 332.1257, found *m/z* 332.1247.

(Tetrahydrofuran-2-yl)methanol (2t)[17]



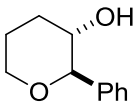
Colorless oil; 98% yield; ^{1}H NMR ($CDCl_3$) δ: 4.47 (1H, br s, OH), 3.95 (1H, ddd, *J* = 13.1, 6.2, 3.3 Hz, OCH), 3.87-3.65 (2H, m, OCH_2), 3.60 (1H, dd, *J* = 11.7, 3.3 Hz, OC*H*H), 3.44 (1H, dd, *J* = 11.7, 6.3 Hz, OC*H*H), 1.93-1.75 (3H, m, CH_2 and 1 x C*H*H), 1.66-1.47 (1H, m, C*H*H); ^{13}C NMR ($CDCl_3$) δ: 79.5, 68.1, 64.6, 27.0, 25.8; MS (ESI) 125 [$(M+Na)^+$, 100%].

Hexahydro-2*H*-3,5-methanocyclopenta[*b*]furan-6-ol (2u)[18]



Colorless oil; 49% yield (only *endo* stereoisomer cyclized); ^{1}H NMR ($CDCl_3$) δ: 3.93 (1H, d, J = 4.4 Hz, OCH), 3.77 (1H, dd, J = 3.8, 1.7 Hz, OCH), 3.75-3.70 (1H, m, OCH*H*), 3.60 (1H, d, J = 8.0 Hz, OCH*H*), 3.42 (1H, s, OH) 2.61-2.51 (1H, m, CH), 2.38-2.19 (1H, m, CH), 2.09-2.01 (1H, m, CH), 1.91 (1H, d, J = 10.2 Hz, C*H*H), 1.84-1.73 (1H, m, C*H*H), 1.45 (1H, d, J = 10.6 Hz, CHH), 0.93 (1H, dd, J = 12.8 and 1.7 Hz, C*H*H); ^{13}C NMR ($CDCl_3$) δ: 87.5, 80.8, 75.1, 44.5, 40.1, 37.2, 33.7, 33.5; MS (ESI) 163 [$(M+Na)^+$, 100%].

2-Phenyltetrahydro-2H-pyran-3-o (2v)[19]



Colorless oil; 66% yield; ^{1}H NMR ($CDCl_3$) δ: 7.46-7.27 (5H, m, ArH), 4.08-4.03 (1H, m, OCH), 3.90 (1H, d, J = 9.0 Hz, PhCH), 3.56-3.39 (2H, m, OCH_2), 2.23-2.07 (1H, m, 1 x C*H*H), 1.94-1.72 (3H, m, 1 x CH_2 and OH), 1.68-1.41 (1H, m, 1 x C*H*H); ^{13}C NMR ($CDCl_3$) δ: 139.4, 128.4, 128.2, 127.4, 85.3, 71.1, 68.0, 31.7, 25.5; MS (ESI) 175 [$(M-H)^-$, 100%].

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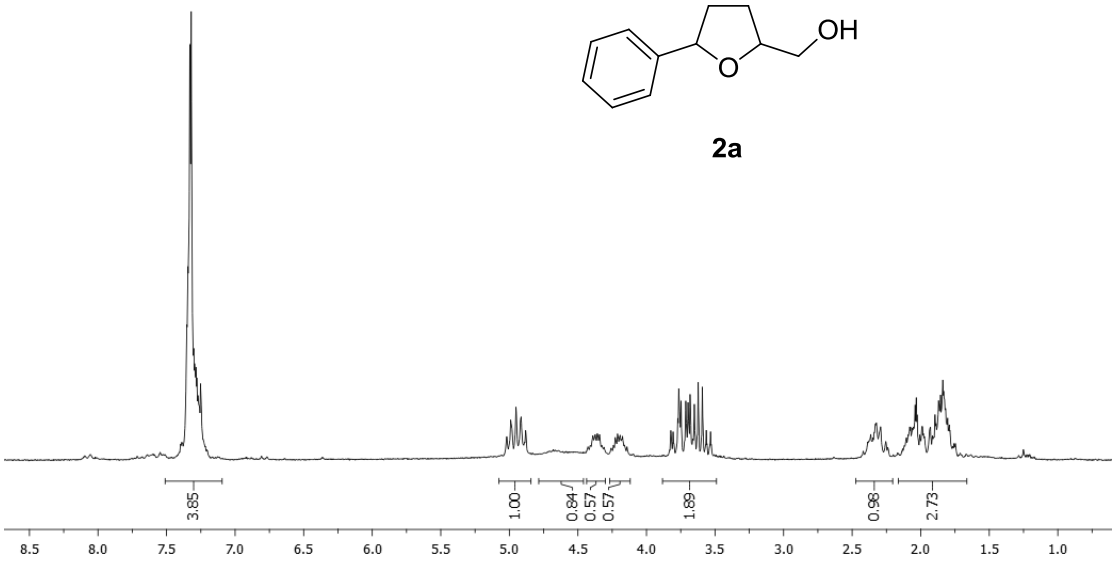
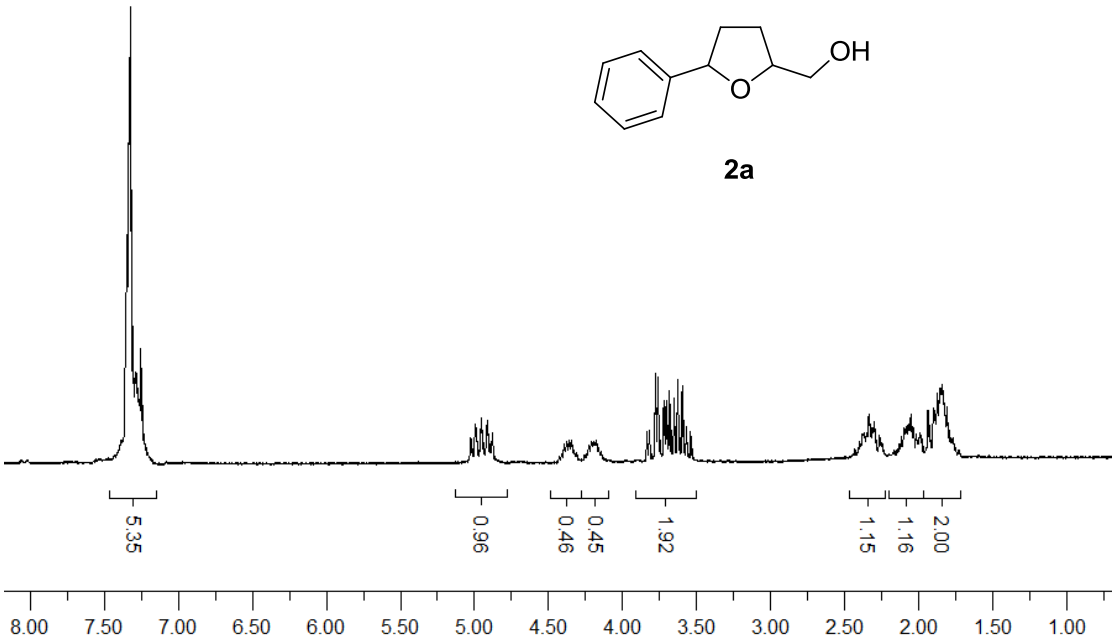
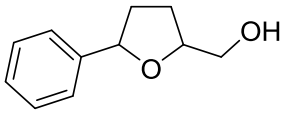
NMR Spectra of the Crude Reaction after Extractions and After Column Chromatography

NMR spectrum of the Crude Reaction Mixture after Extractions



NMR spectrum after Column Chromatography

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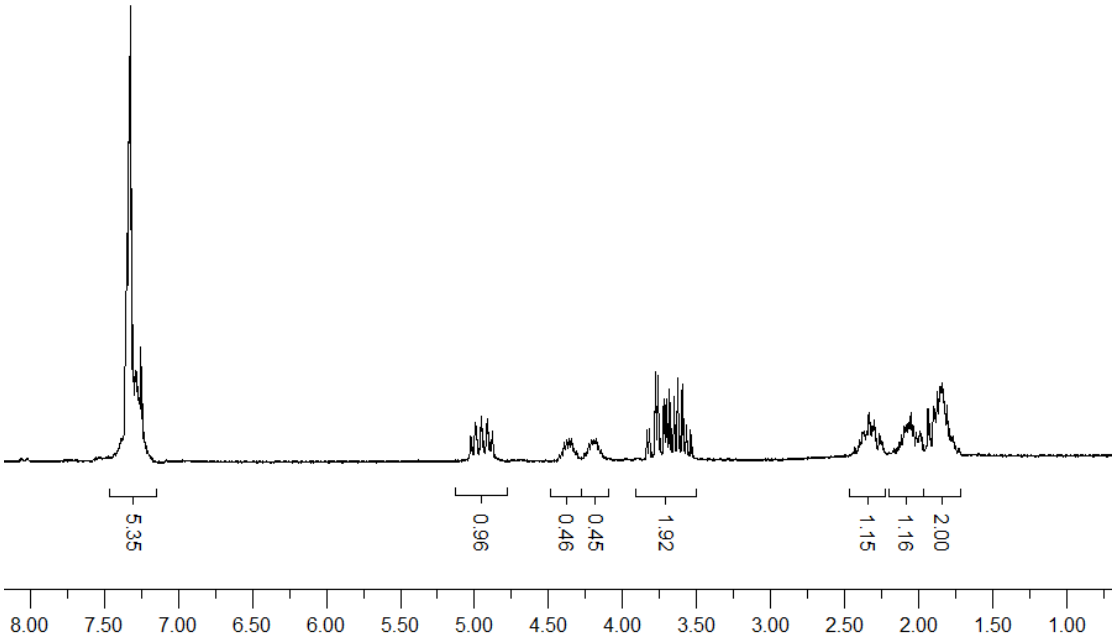
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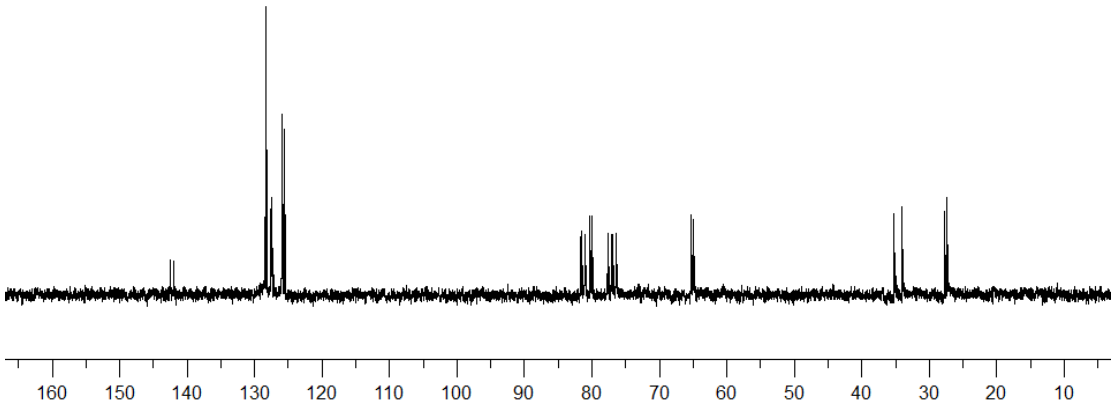
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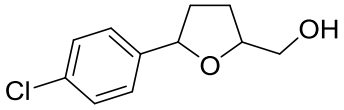
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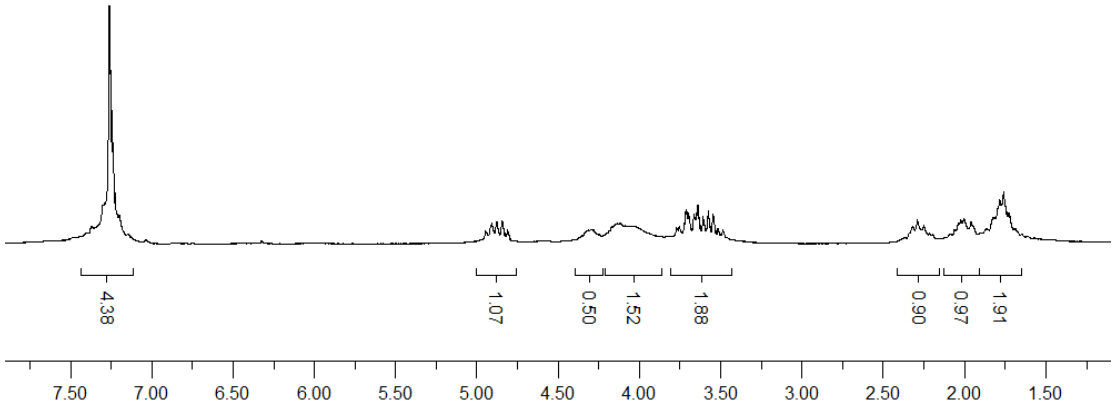
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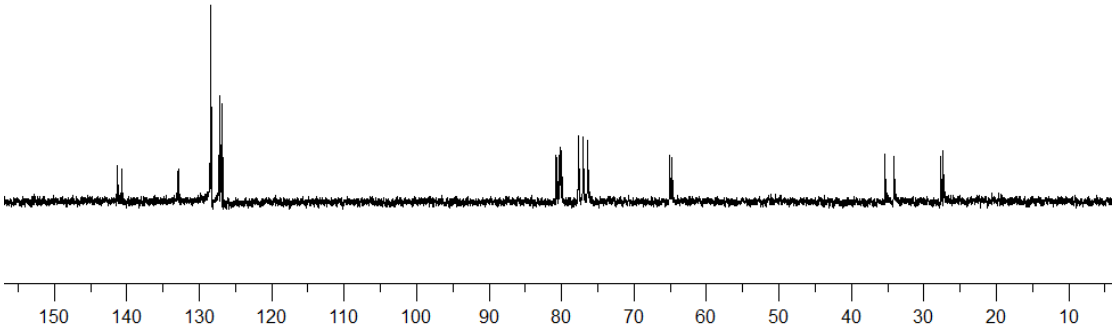
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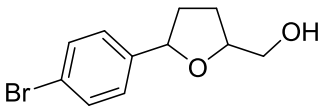
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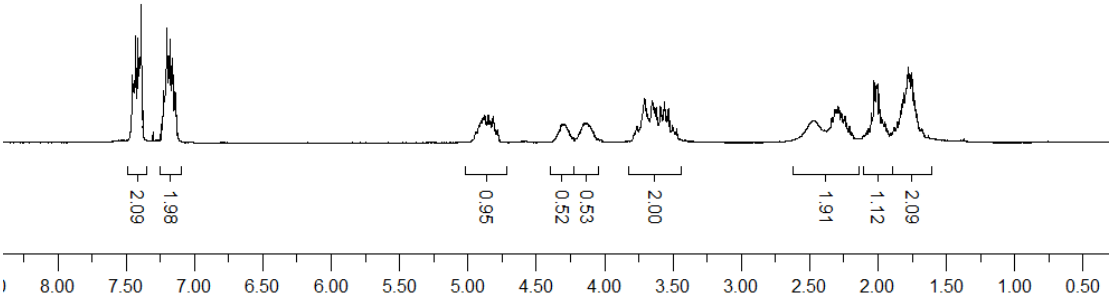
O
Br

2c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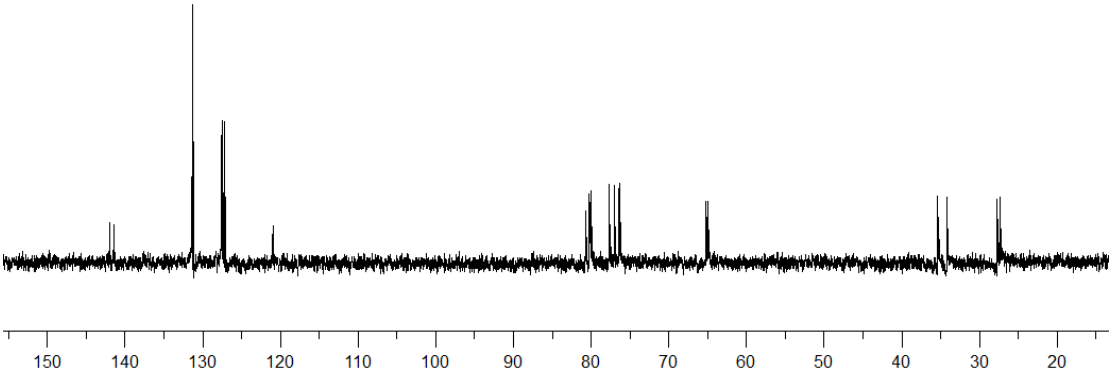
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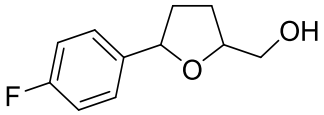
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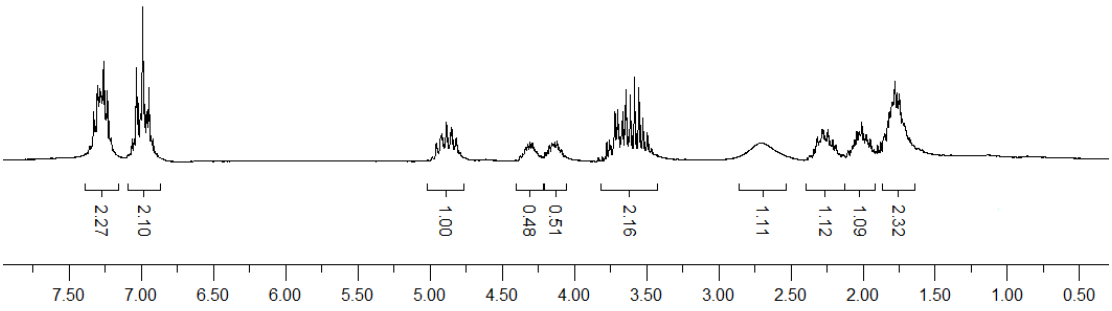
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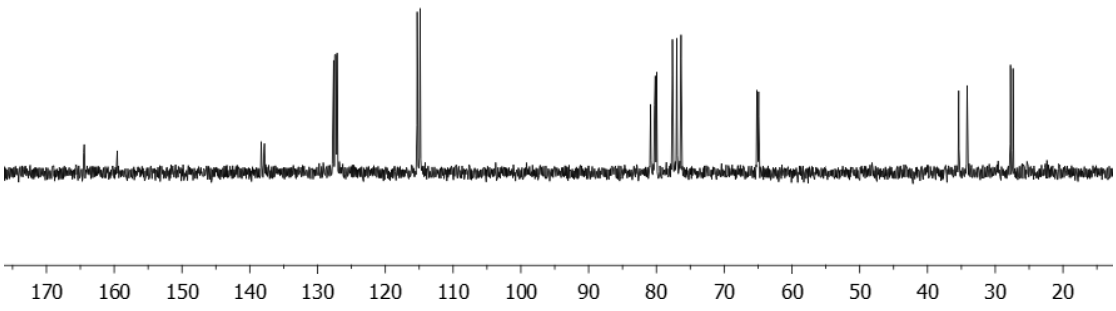
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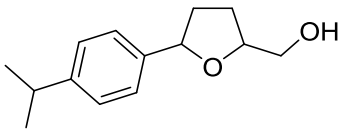
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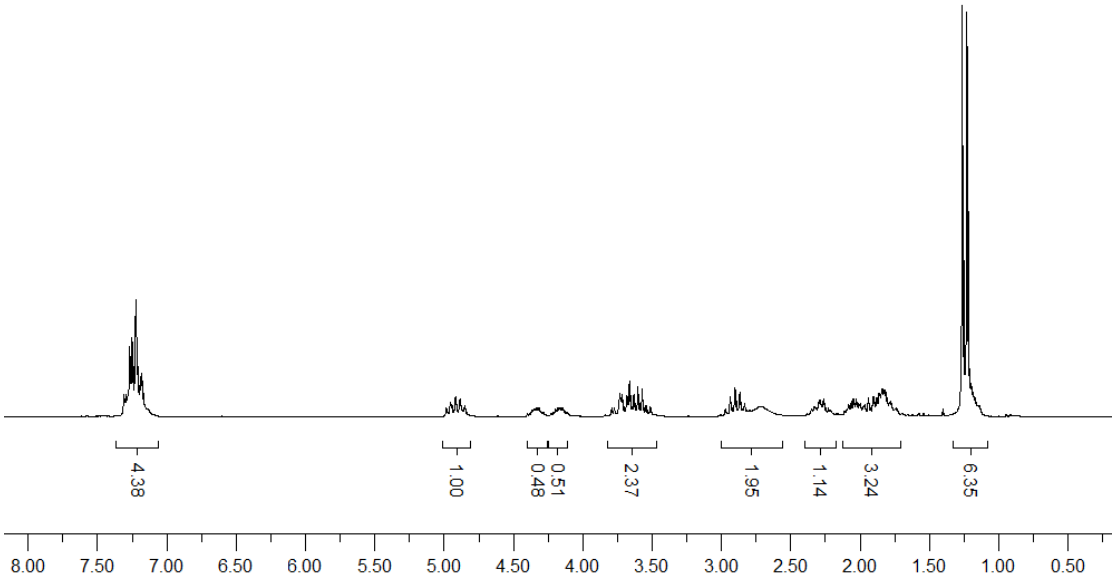
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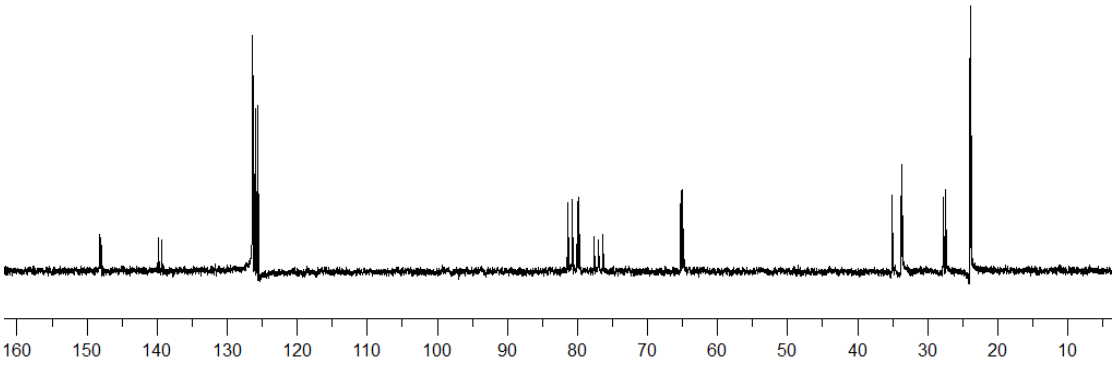
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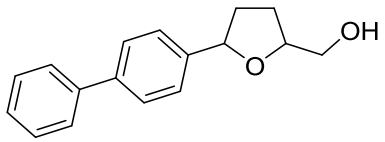
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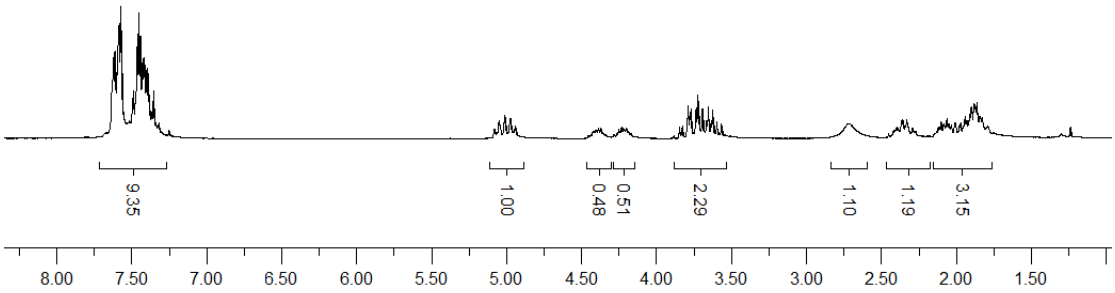
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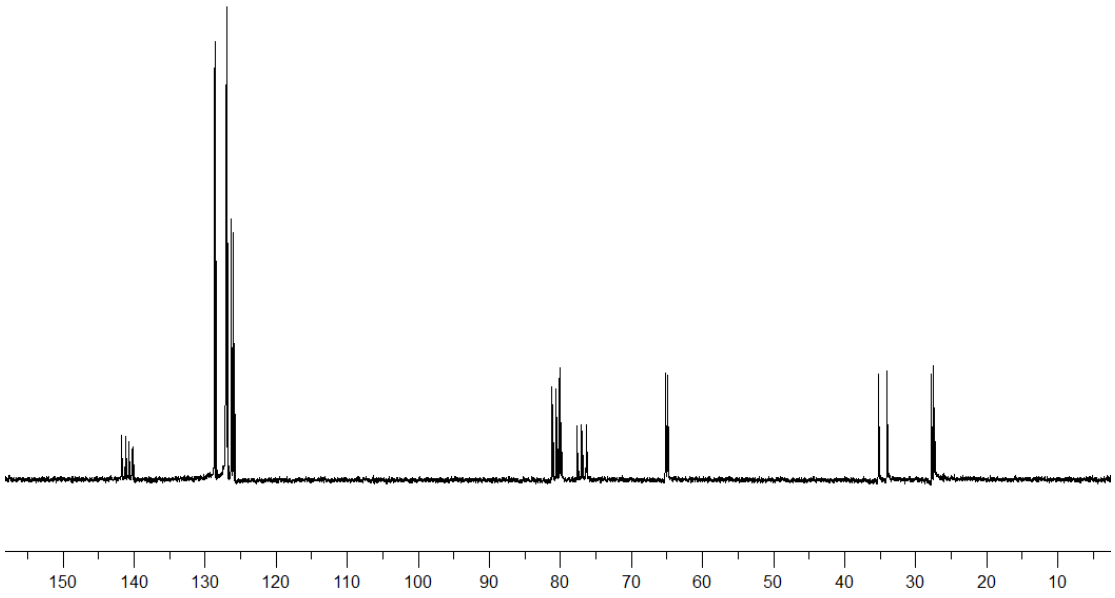
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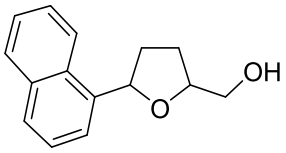
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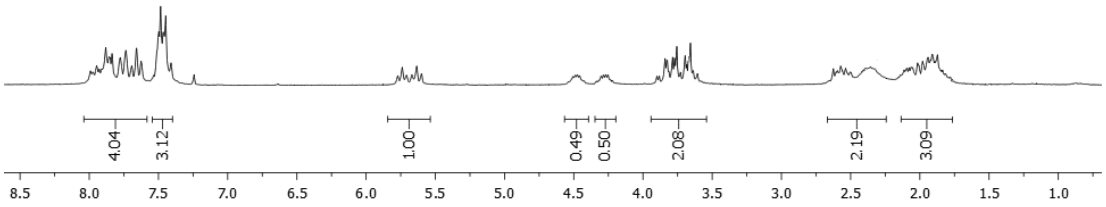
2g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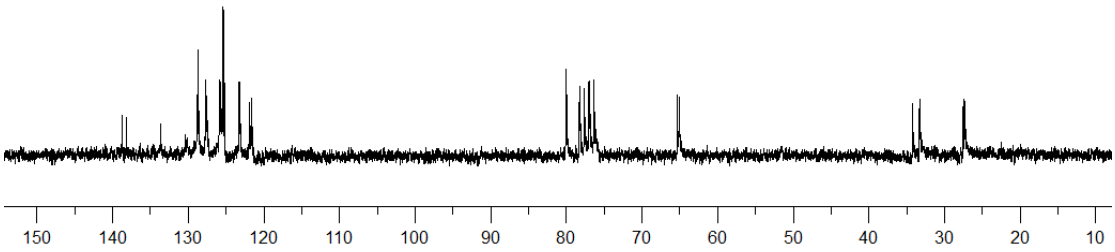
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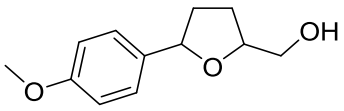
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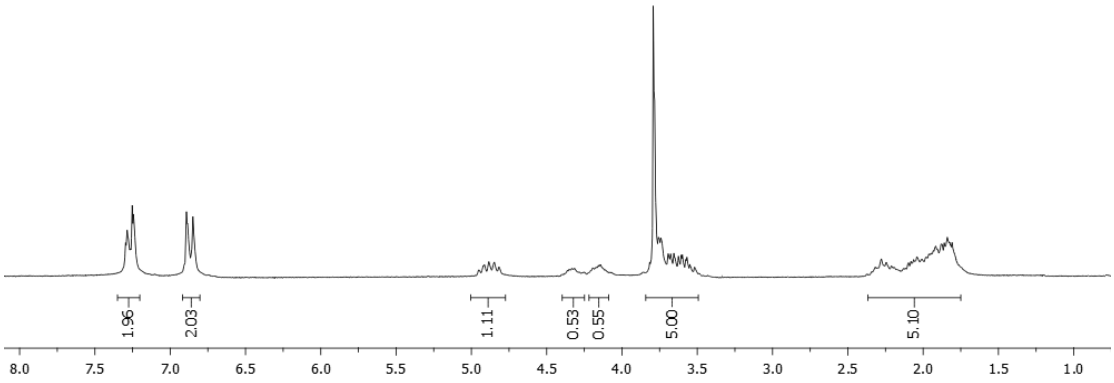
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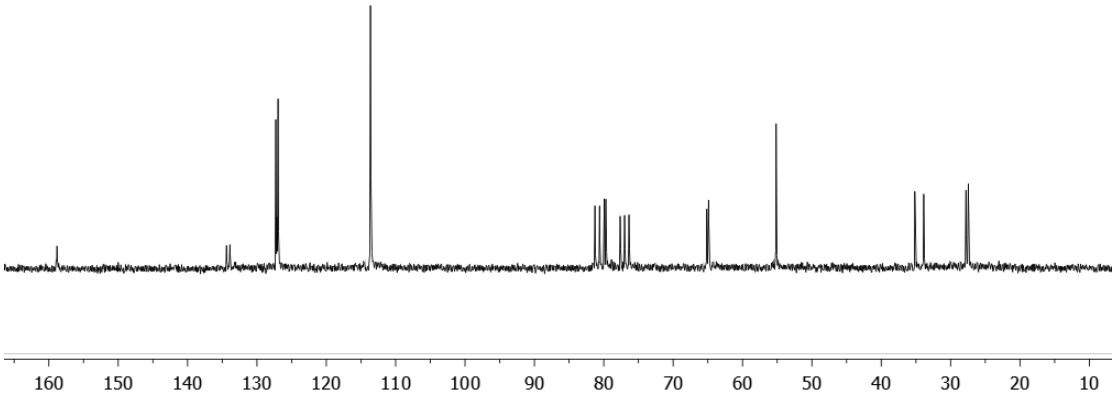
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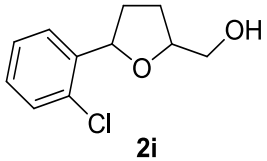
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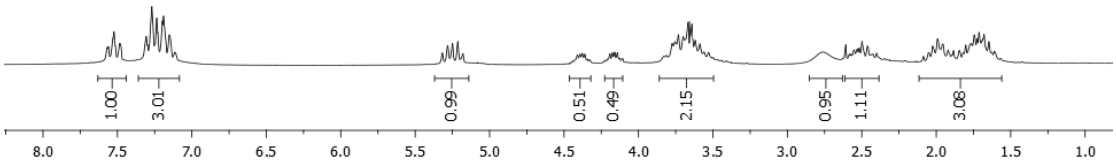
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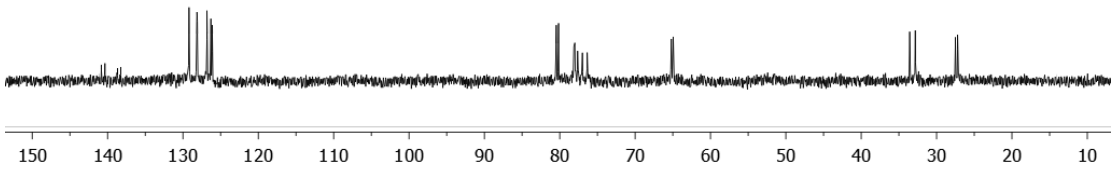
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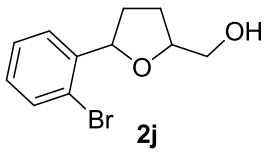
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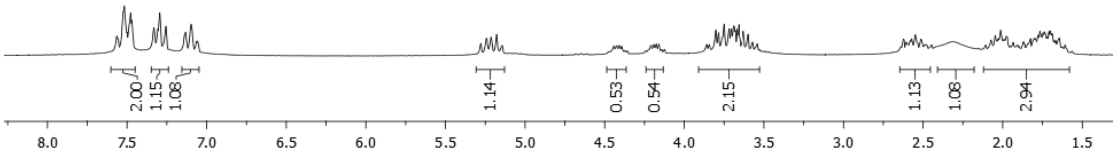
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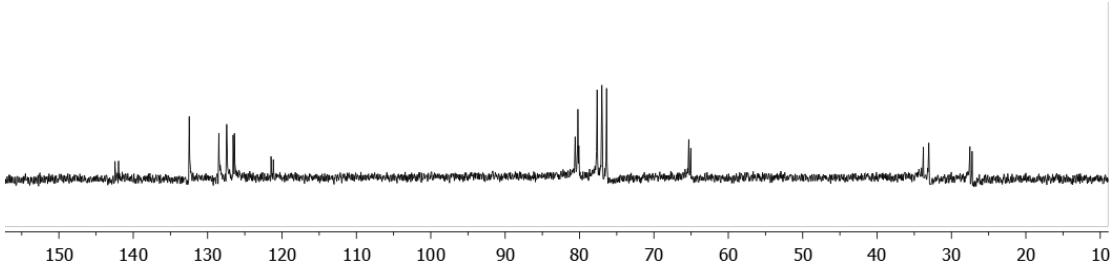
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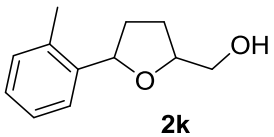
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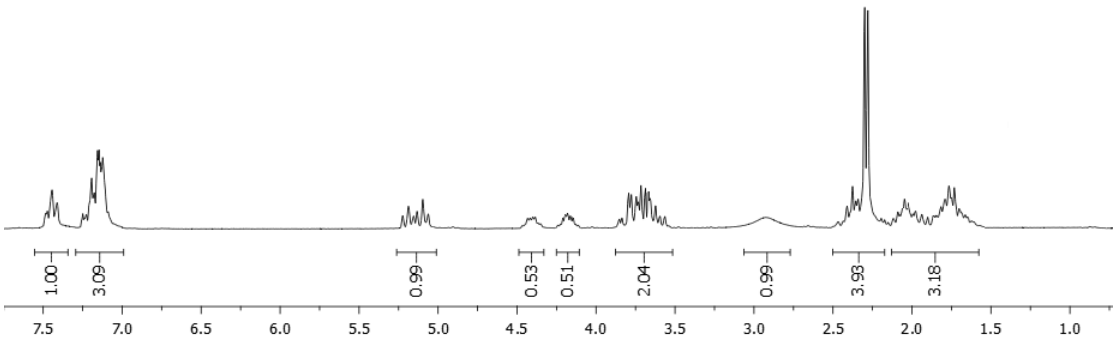
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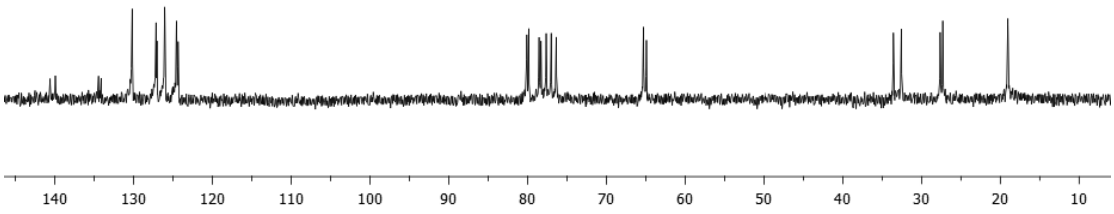
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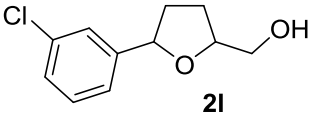
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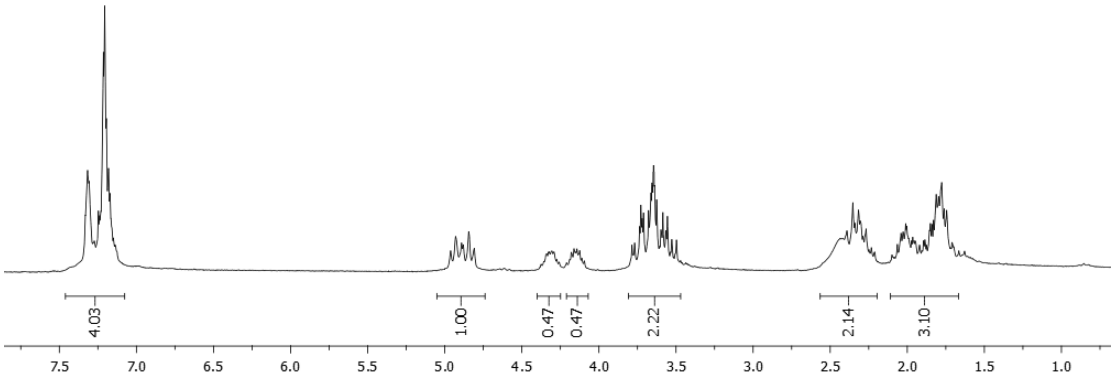
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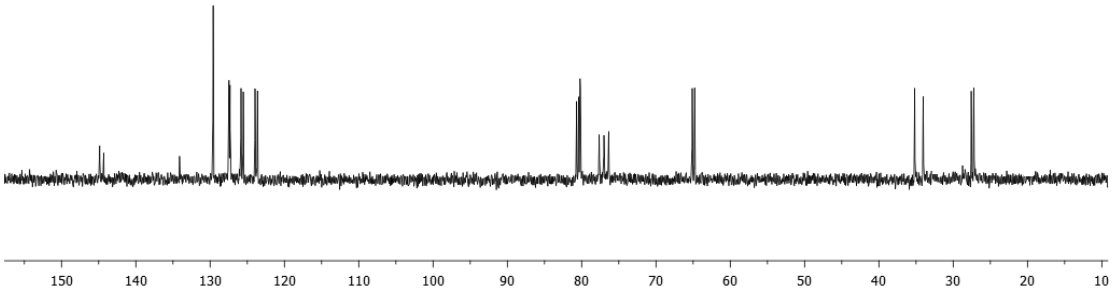
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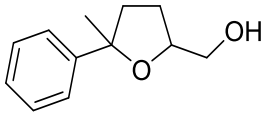
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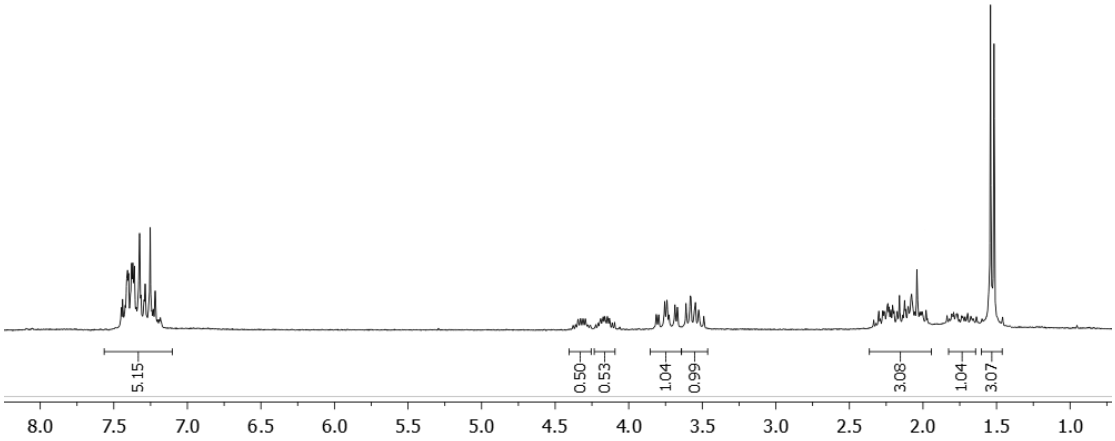
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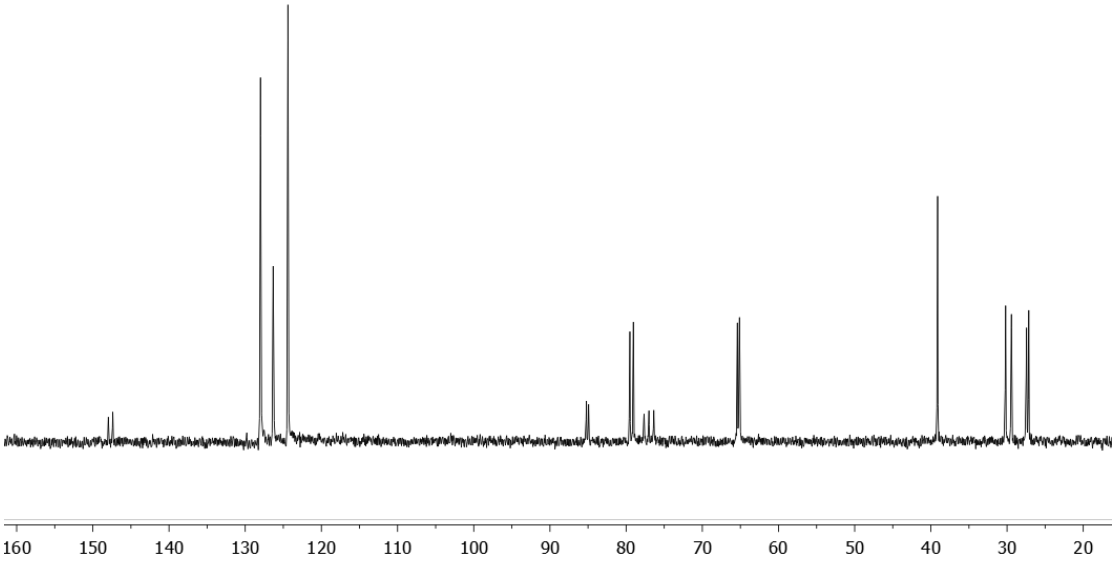
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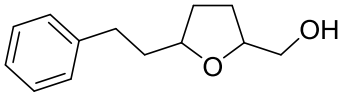
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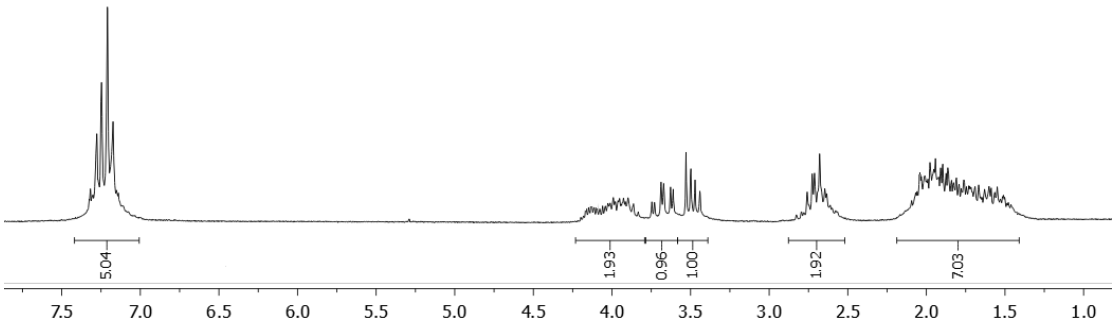
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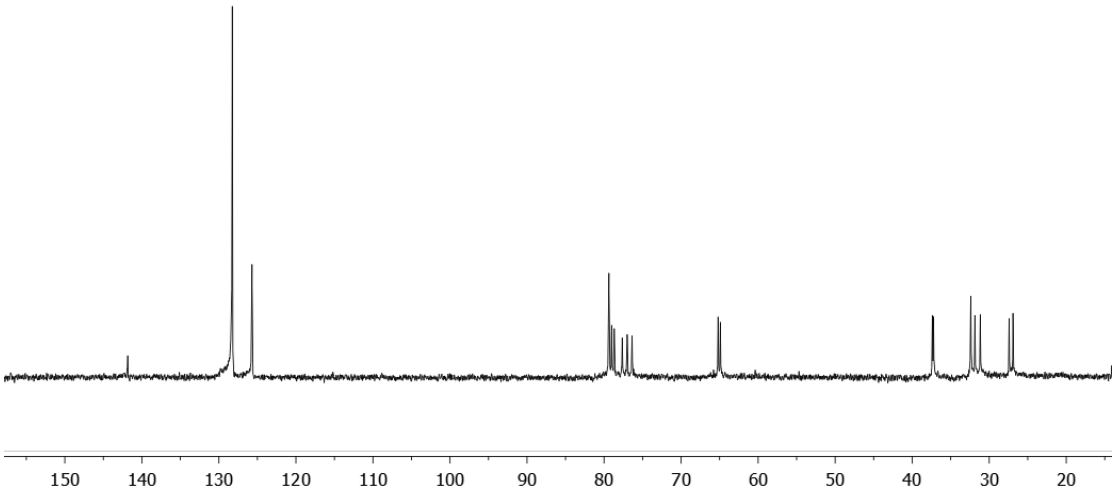
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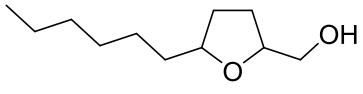
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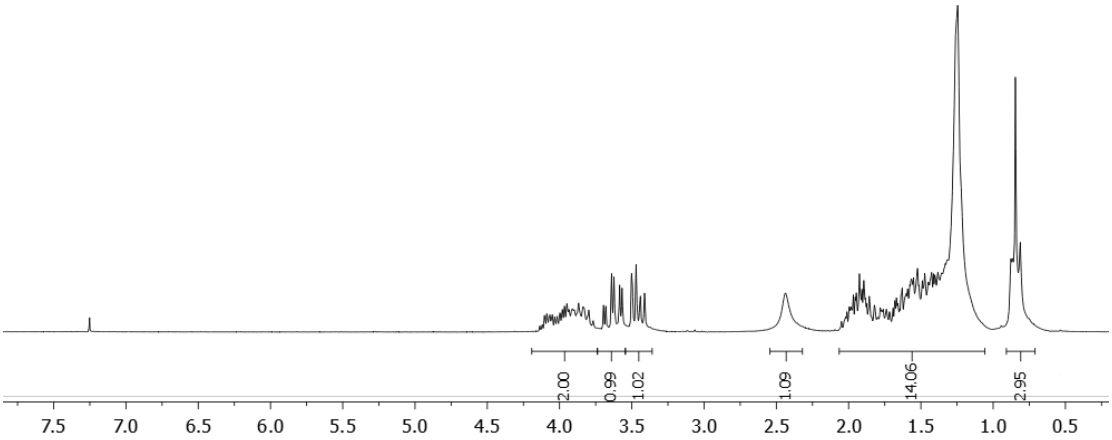
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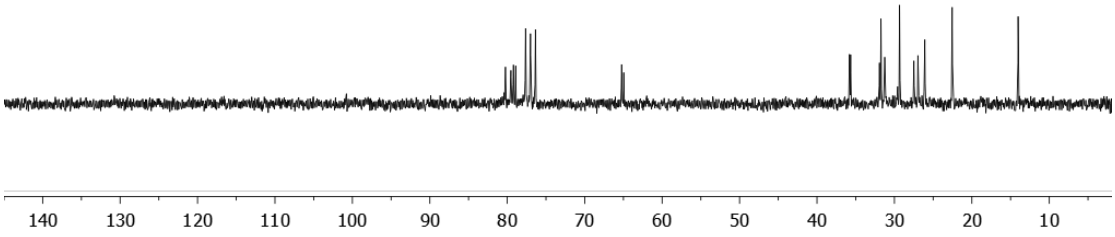
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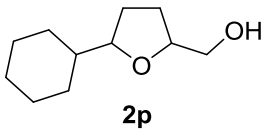
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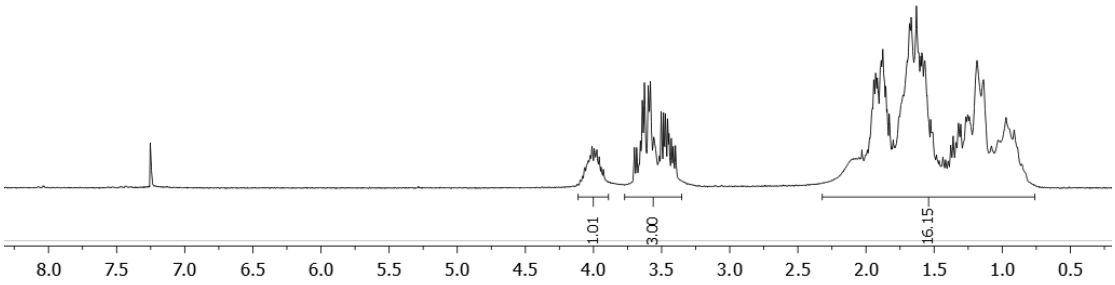
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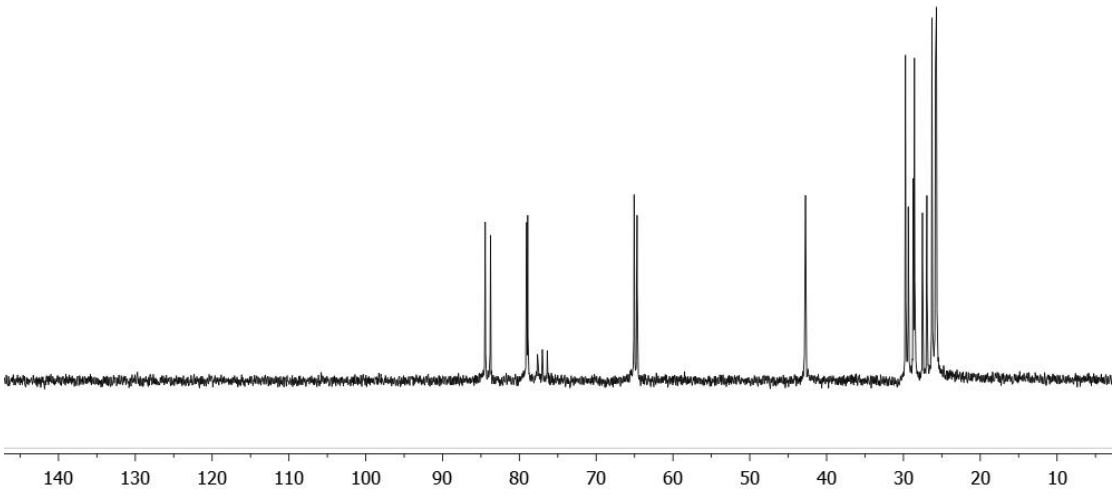
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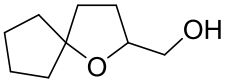
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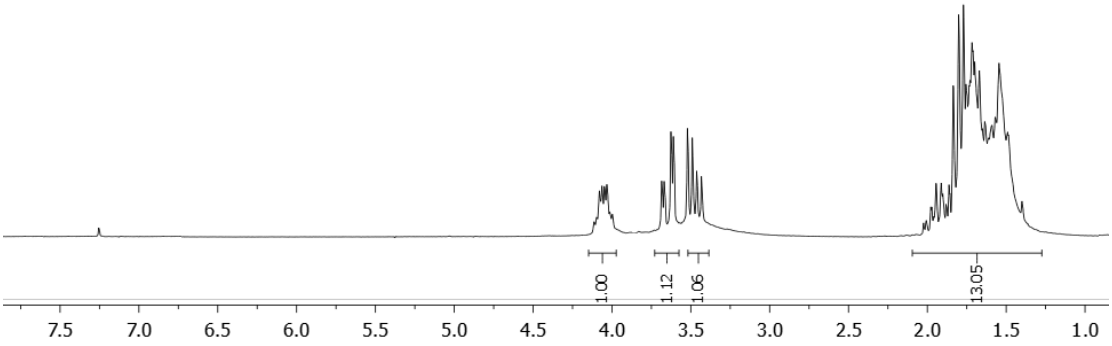
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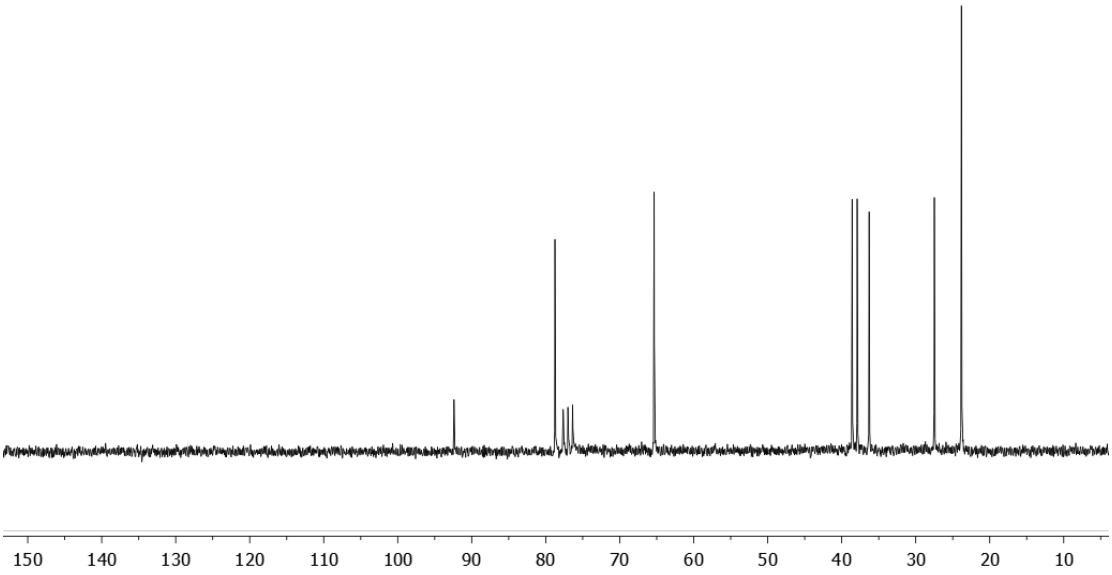
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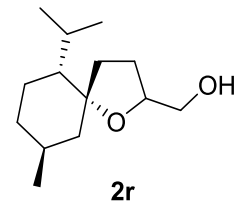
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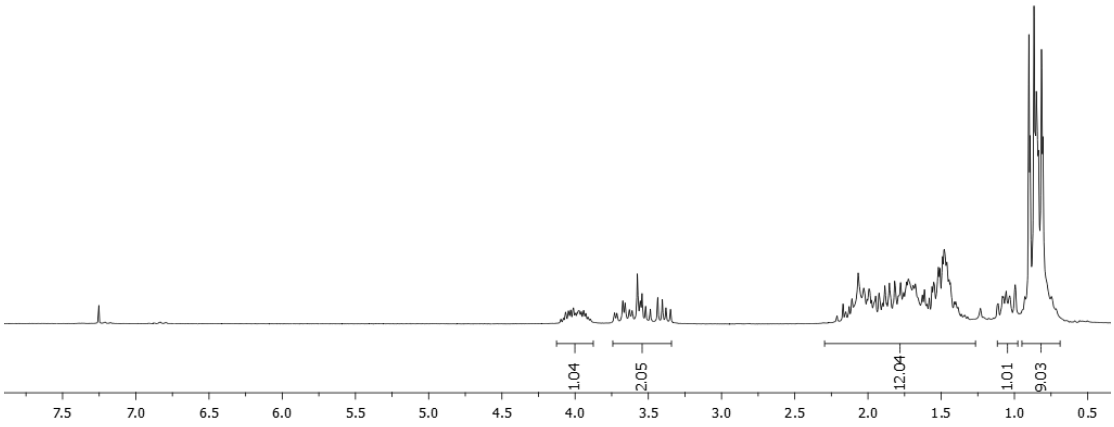
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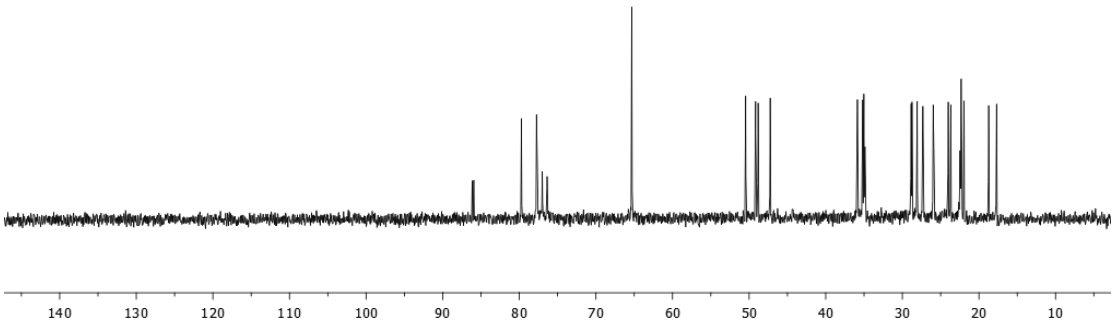
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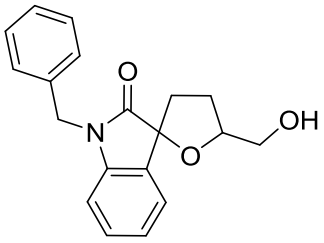
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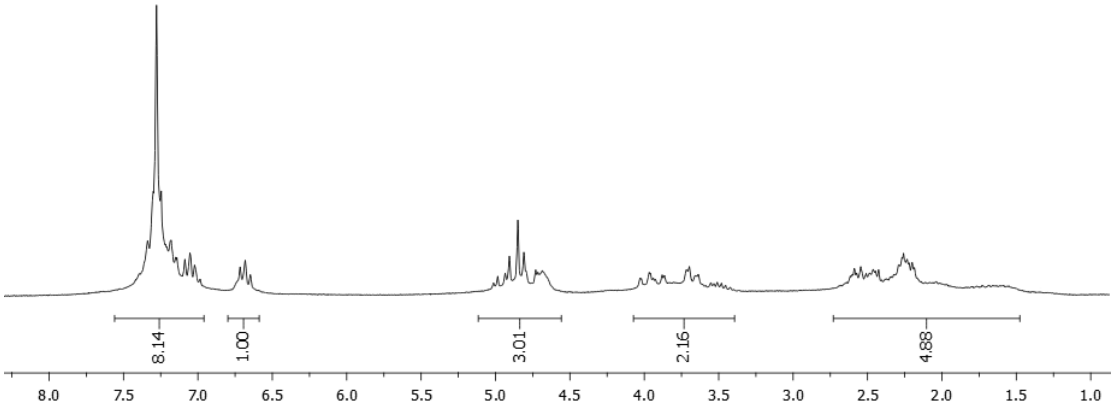
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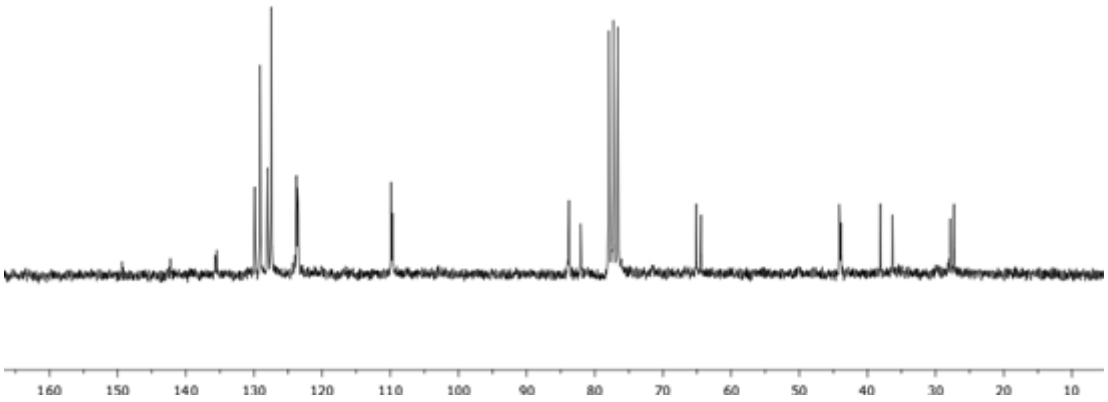
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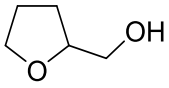
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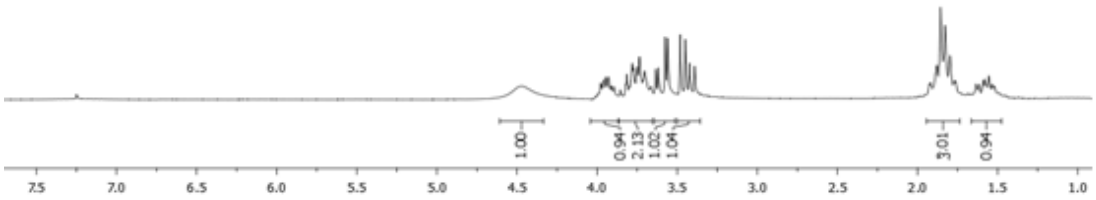
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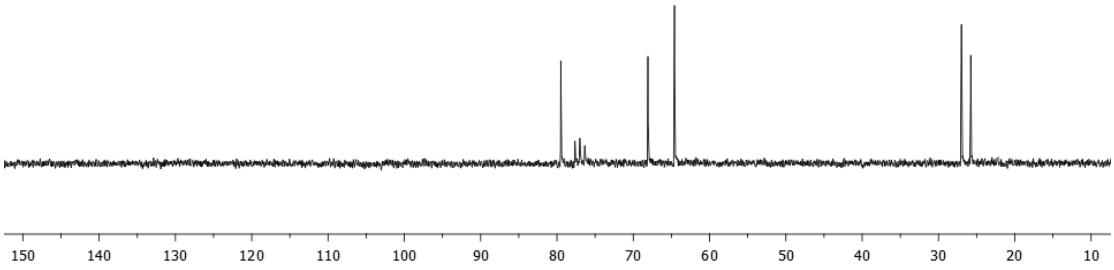
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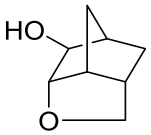
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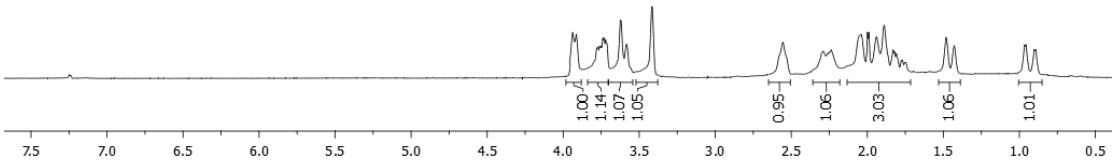
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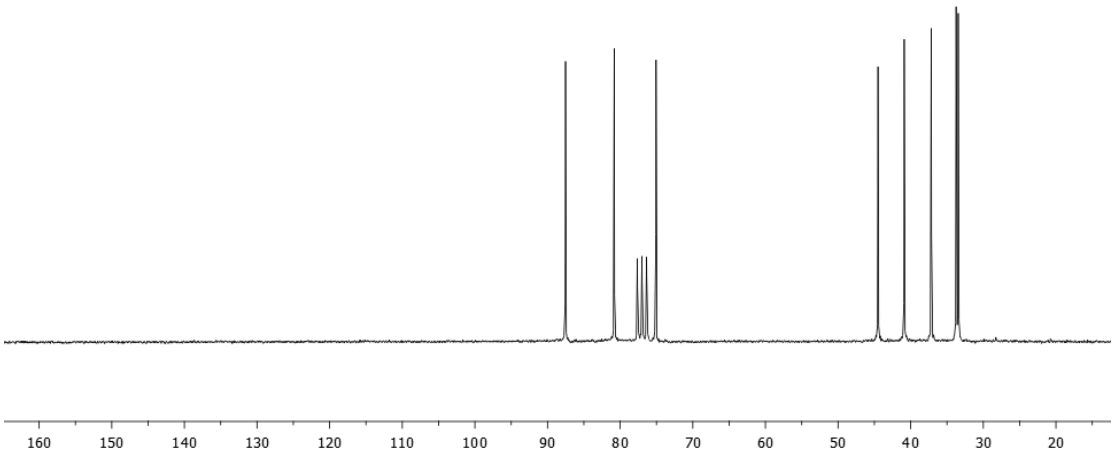
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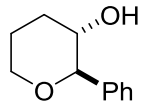
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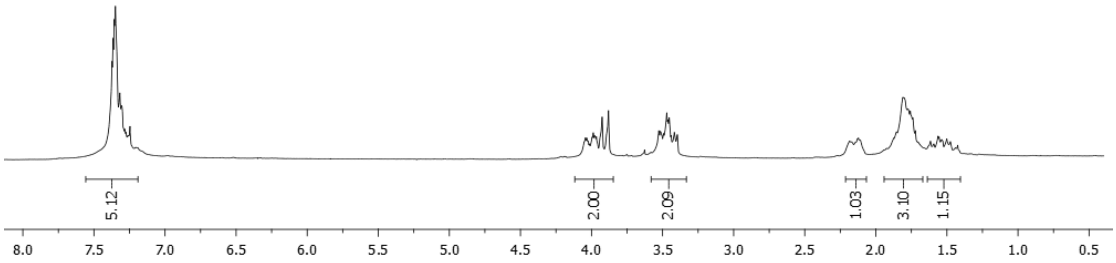
Ph

2v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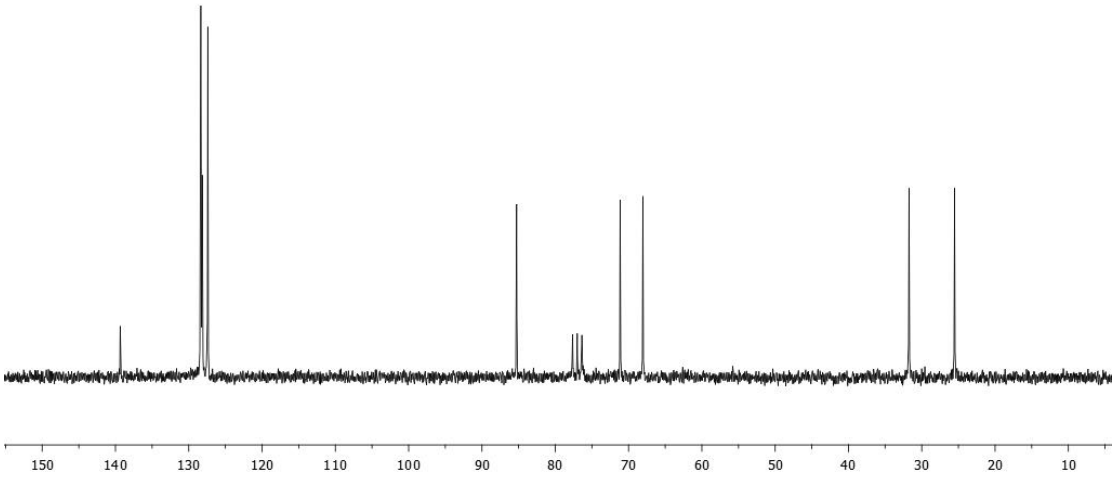
2.09
1.03
3.10
1.15
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5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140
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