Supporting Information

Synthesis of 2-hydroxytetrahydrofurans by Wacker-type oxidation of 1,1-disubstituted alkenes

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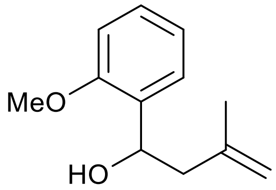
General Information

$PdCl_2(MeCN)_2$[1] was prepared as described in the literature. Deionized water was used as a solvent. Dehydrated DMF was purchased from FUJIFILM Wako Pure Chemical Corporation. BQ was purified by sublimation. Isoprenol (**1a**) was obtained from Kuraray Co., Ltd and was used as received. Other chemicals were also commercially available and were used without further purification. Flash column chromatography was performed using silica gel SILICYCLE SiliaFlash F60 (40–63 μm, 230–400 mesh). NMR spectra were recorded on either a Bruker AV-300N (300 MHz (^{1}H), 75 MHz (^{13}C)) spectrometer or a JEOL JNM-AL400 (400 MHz (^{1}H), 100 MHz (^{13}C)) spectrometer. Chemical shift values (σ) were expressed relative to $SiMe_4$. High-resolution mass spectra were recorded on a JEOL JMS-T100LC spectrometer (ESI-TOF MS) with positive ionization mode.

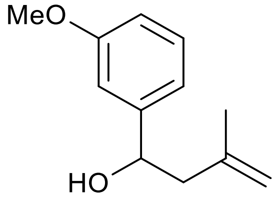
Preparation of Substrates 1

Substrate **1b** was prepared as described in the literature.[2] Substrates **1c**–**1p** were synthesized similarly to **1b**.[2] Substrate **1q** was prepared similarly as described in the literature.[3]



Synthesis of 1-(2-methoxyphenyl)-3-methyl-3-buten-1-ol (1c)

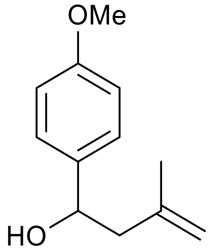
The reaction was performed under an argon atmosphere. DMF (54.5 mL) and *β*-methallyl chloride (3.21 mL, 33.0 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, *o*-anisaldehyde (4.09 g, 30.0 mmol, 1.0 eq.), $SnCl_2 \cdot 2H_2O$ (10.2 g, 45.0 mmol, 1.5 eq.), and NaI (6.75 g, 45.0 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 30 mL) and *t*-butyl methyl ether (60 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (30 mL × 3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1c** as a white solid (2.64 g, 13.7 mmol, 46% yield). The NMR spectral data for **1c** were in accordance with those reported in the literature.[4]



Synthesis of 1-(3-methoxyphenyl)-3-methyl-3-buten-1-ol (1d)

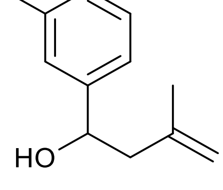
The reaction was performed under an argon atmosphere. DMF (54.5 mL), *m*-methoxybenzadehyde (3.66 mL, 30.0 mmol, 1.0 eq.), and *β*-methallyl chloride (3.21 mL, 33.0 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (10.2 g, 45.0 mmol, 1.5 eq.) and NaI (6.75 g, 45.0 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 30 mL) and *t*-butyl methyl ether (60 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (30 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1d** as a yellow oil (3.71 g, 19.3 mmol, 64% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38–6.76 (m, 4H), 4.96 (t, J = 1.6 Hz, 1H), 4.89 (d, J = 0.9 Hz, 1H), 4.82 (dt, J = 2.3, 6.7 Hz, 1H), 3.84 (s, 3H), 2.44 (d, J = 6.5 Hz, 2H), 2.21 (s, 1H), 1.83 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ159.9, 146.0, 142.5, 129.6, 118.2, 114.2, 113.1, 111.4, 71.5, 55.4, 48.5, 22.5. HRMS (ESI): *m*/*z* calcd for $C_{12}H_{16}NaO_2$ [M+Na]$^+$ 215.1048, found 215.1046.



Synthesis of 1-(4-methoxyphenyl)-3-methyl-3-buten-1-ol (1e)

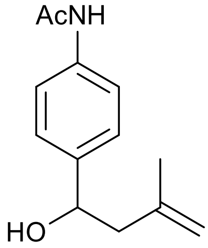
The reaction was performed under an argon atmosphere. DMF (54.5 mL), *p*-methoxybenzadehyde (3.65 mL, 30.0 mmol, 1.0 eq.), and *β*-methallyl chloride (3.21 mL, 33.0 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (10.2 g, 45.0 mmol, 1.5 eq.) and NaI (6.75 g, 45.0 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 30 mL) and *t*-butyl methyl ether (60 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (30 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1e** as a pale yellow oil (3.73 g, 19.4 mmol, 65% yield). The NMR spectral data for **1e** were in accordance with those reported in the literature.[5]



Synthesis of 3-methyl-1-(3-phenoxyphenyl)-3-buten-1-ol (1f)

The reaction was performed under an argon atmosphere. DMF (27.3 mL), 3-phenoxybenzaldehyde (2.58 mL, 15.0 mmol, 1.0 eq.), and *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1f** as a pale yellow oil (3.30 g, 13.0 mmol, 86% yield).

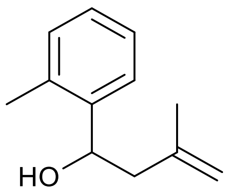
^1H NMR (300 MHz, $CDCl_3$) δ 7.42–6.83 (m, 9H), 4.91 (s, 1H), 4.83 (s, 1H), 4.81–4.73 (m, 1H), 2.42–2.35 (m, 2H), 2.09 (s, 1H), 1.77 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 157.5, 157.4, 146.4, 142.3, 129.9, 129.9, 123.3, 120.8, 119.0, 118.0, 116.5, 114.4, 71.2, 48.5, 22.5. HRMS (ESI): *m/z* calcd for $C_{17}H_{18}NaO_2$ $[M+Na]^+$ 277.1205, found 277.1200.



Synthesis of 1-(4-acetamidophenyl)-3-methyl-3-buten-1-ol (1g)

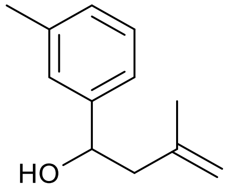
The reaction was performed under an argon atmosphere. DMF (27.3 mL), 4-acetamidobenzaldehyde (2.45 g, 15.0 mmol, 1.0 eq.), and *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 2/3) to afford compound **1g** as a white solid (1.20 g, 5.49 mmol, 37% yield).

^1H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, J = 8.5 Hz, 2H), 7.31 (d, J = 8.5 Hz, 2H), 7.13 (br s, 1H), 4.90 (s, 1H), 4.83 (s, 1H), 4.77 (dt, J = 2.1, 6.9 Hz, 1H), 2.38 (d, J = 6.9 Hz, 2H), 2.16 (s, 3H), 2.08 (d, J = 2.3 Hz, 1H), 1.77 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 168.4, 142.4, 140.2, 137.3, 126.6 (2C), 120.0 (2C), 114.3, 71.2, 48.4, 24.7, 22.5. HRMS (ESI): *m/z* calcd for $C_{13}H_{17}NNaO_2$ $[M+Na]^+$ 242.1157, found 242.1163.



Synthesis of 3-methyl-1-(2-methylphenyl)-3-buten-1-ol (1h)

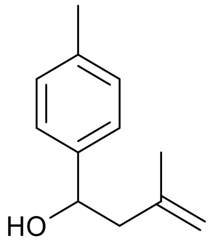
The reaction was performed under an argon atmosphere. DMF (27.3 mL), *o*-tolualdehyde (1.76 mL, 15.0 mmol, 1.0 eq.), and *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1h** as a pale yellow oil (1.56 g, 8.86 mmol, 59% yield). The NMR spectral data for **1h** were in accordance with those reported in the literature.[5]



Synthesis of 3-methyl-1-(3-methylphenyl)-3-buten-1-ol (1i)

The reaction was performed under an argon atmosphere. DMF (27.3 mL), *m*-tolualdehyde (1.76 mL, 15.0 mmol, 1.0 eq.), and *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1i** as a pale yellow oil (1.42 g, 8.06 mmol, 54% yield).

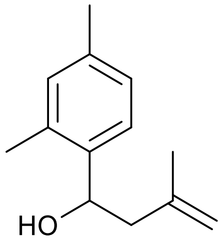
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.04 (m, 4H), 4.92 (s, 1H), 4.85 (d, J = 0.7 Hz, 1H), 4.82–4.73 (m, 1H), 2.47– 2.31 (m, 2H), 2.34 (s, 3H), 2.07 (s, 1H), 1.79 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 144.1, 142.6, 138.2, 128.5, 128.4, 126.6, 123.0, 114.2, 71.5, 48.5, 22.5, 21.6. HRMS (ESI): *m*/*z* calcd for $C_{12}H_{16}NaO$ $[M+Na]^+$ 199.1099, found 199.1109.



Synthesis of 3-methyl-1-(4-methylphenyl)-3-buten-1-ol (1j)

The reaction was performed under an argon atmosphere. DMF (40.0 mL), *p*-tolualdehyde (2.36 mL, 20.0 mmol, 1.0 eq.), and *β*-methallyl chloride (2.14 mL, 22.0 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2·2H_2O$ (6.77 g, 30.0 mmol, 1.5 eq.) and NaI (4.50 g, 30.0 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 25 mL) and *t*-butyl methyl ether (50 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (30 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1j** as a pale yellow oil (1.31 g, 7.44 mmol, 37% yield).

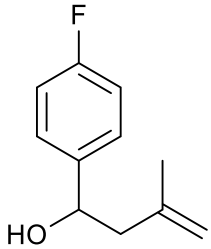
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 (d, J = 8.0 Hz, 2H), 7.14 (d, J = 8.0 Hz, 2H), 4.90 (d, J = 1.1 Hz, 1H), 4.84 (d, J = 1.1 Hz, 1H), 4.79–4.74 (m, 1H), 2.41–2.39 (m, 2H), 2.33 (s, 3H), 2.06 (d, J = 2.4 Hz, 1H), 1.78 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 142.7, 141.2, 137.3, 129.2 (2C), 125.9 (2C), 114.1, 71.4, 48.5, 22.5, 21.3. HRMS (ESI): *m*/*z* calcd for $C_{12}H_{16}NaO$ $[M+Na]^+$ 199.1099, found 199.1109.



Synthesis of 3-methyl-1-(2,4-dimethylphenyl)-3-buten-1-ol (1k)

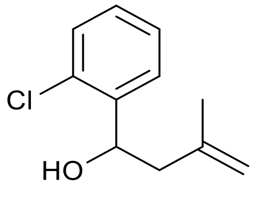
The reaction was performed under an argon atmosphere. DMF (27.3 mL), 2,4-dimethylbenzaldehyde (2.16 mL, 15.0 mmol, 1.0 eq.), and *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2·2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1k** as a yellow oil (2.11 g, 11.1 mmol, 74% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (d, J = 7.9 Hz, 1H), 7.08–6.91 (m, 2H), 5.04–4.96 (m, 1H), 4.92 (t, J = 1.6 Hz, 1H), 4.88 (d, J = 8.4 Hz, 1H), 2.46–2.25 (m, 2H), 2.30 (s, 3H), 2.29 (s, 3H), 2.00 (d, J = 2.3 Hz, 1H), 1.82 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 142.9, 139.3, 136.8, 134.2, 131.2, 127.1, 125.2, 114.0, 67.9, 47.3, 22.4, 21.1, 19.0.



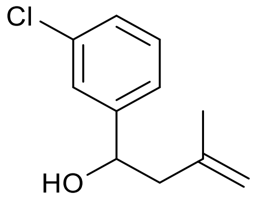
Synthesis of 1-(4-fluorophenyl)-3-methyl-3-buten-1-ol (1l)

The reaction was performed under an argon atmosphere. DMF (54.5 mL), *p*-fluorobenzadehyde (3.16 mL, 30.0 mmol, 1.0 eq.), and β-methallyl chloride (3.21 mL, 33.0 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2{\cdot}2H_2O$ (10.2 g, 45.0 mmol, 1.5 eq.) and NaI (6.75 g, 45.0 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 30 mL) and *t*-butyl methyl ether (60 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (30 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1l** as a pale yellow oil (3.66 g, 20.3 mmol, 68% yield). The NMR spectral data for **1l** were in accordance with those reported in the literature.[5]



Synthesis of 1-(2-chlorophenyl)-3-methyl-3-buten-1-ol (1m)

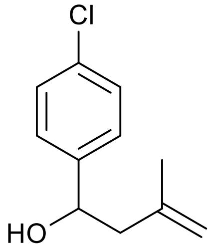
The reaction was performed under an argon atmosphere. DMF (27.3 mL), *o*-chlorobenzaldehyde (1.69 mL, 15.0 mmol, 1.0 eq.), and β-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2{\cdot}2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1m** as a pale yellow oil (2.20 g, 11.2 mmol, 75% yield). The NMR spectral data for **1m** were in accordance with those reported in the literature.[6]



Synthesis of 1-(3-chlorophenyl)-3-methyl-3-buten-1-ol (1n)

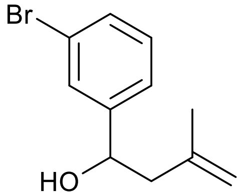
The reaction was performed under an argon atmosphere. DMF (27.3 mL), *m*-chlorobenzaldehyde (1.69 mL, 15.0 mmol, 1.0 eq.), and *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1n** as a pale yellow oil (2.37 g, 12.0 mmol, 80% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (d, J = 1.8 Hz, 1H), 7.29–7.20 (m, 3H), 4.94 (t, J = 1.5 Hz, 1H), 4.85 (s, 1H), 4.81–4.73 (m, 1H), 2.45–2.29 (m, 2H), 2.16 (d, J = 2.4 Hz, 1H), 1.79 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 146.3, 142.1, 134.5, 129.8, 127.7, 126.1, 124.0, 114.7, 70.8, 48.6, 22.4.



Synthesis of 1-(4-chlorophenyl)-3-methyl-3-buten-1-ol (1o)

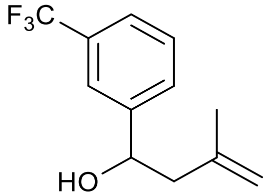
The reaction was performed under an argon atmosphere. DMF (54.5 mL) and *β*-methallyl chloride (3.21 mL, 33.0 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, *p*-chlorobenzadehyde (4.22 g, 30.0 mmol, 1.0 eq.), $SnCl_2 \cdot 2H_2O$ (10.2 g, 45.0 mmol, 1.5 eq.), and NaI (6.75 g, 45.0 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 30 mL) and *t*-butyl methyl ether (60 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (30 mL × 3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1o** as a pale yellow oil (3.70 g, 18.8 mmol, 63% yield). The NMR spectral data for **1o** were in accordance with those reported in the literature.[7]



Synthesis of 1-(3-bromophenyl)-3-methyl-3-buten-1-ol (1p)

The reaction was performed under an argon atmosphere. DMF (27.3 mL) and 3-bromobenzaldehyde (2.13 mL, 15.0 mmol, 1.0 eq.), *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.), and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL × 3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1p** as a pale yellow oil (2.26 g, 9.36 mmol, 62% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (t, J = 1.7 Hz, 1H), 7.43–7.16 (m, 3H), 4.94 (t, J = 1.5 Hz, 1H), 4.85 (d, J = 0.8 Hz, 1H), 4.80–4.72 (m, 1H), 2.46–2.29 (m, 2H), 2.16 (d, J = 2.4 Hz, 1H), 1.79 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 146.5, 142.1, 130.6, 130.1, 129.0, 124.5, 122.7, 114.7, 70.7, 48.6, 22.4.

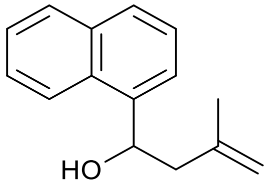


Synthesis of 3-methyl-1-(3-trifluoromethylphenyl)-3-buten-1-ol (1q)

The reaction was performed under an argon atmosphere. DMF (27.3 mL), 3-(trifluoromethyl)benzaldehyde (2.01 mL, 15.0 mmol, 1.0 eq.), and *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1q** as a pale yellow oil (2.58 g, 11.2 mmol, 75% yield).

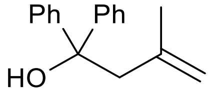
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.56–7.42 (m, 3H), 4.96 (t, J = 1.5 Hz, 1H), 4.87 (s, 1H), 4.89–4.81 (m, 1H), 2.47–2.30 (m, 2H), 2.22 (d, J = 2.2 Hz, 1H), 1.80 (s, 3H). ^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$) δ 145.1, 142.0, 130.9 (q, $^2J_{CF}$ = 32 Hz), 129.3 (q, $^4J_{CF}$ = 1.0 Hz), 129.0, 124.4 (q, $^3J_{CF}$ = 3.8

Hz), 124.3 (q, ${}^{1}J_{CF} = 270.6$ Hz), 122.7 (q, ${}^{3}J_{CF} = 3.8$ Hz), 114.9, 70.8, 48.7, 22.4.



Synthesis of 3-methyl-1-(1-naphthyl)-3-buten-1-ol (1r)

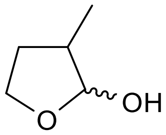
The reaction was performed under an argon atmosphere. DMF (27.3 mL), 1-naphthylaldehyde (2.20 mL, 15.0 mmol, 1.0 eq.), and *β*-methallyl chloride (1.61 mL, 16.5 mmol, 1.1 eq.) were added to a dried reaction vessel. To the solution, $SnCl_2 \cdot 2H_2O$ (5.08 g, 22.5 mmol, 1.5 eq.) and NaI (3.37 g, 22.5 mmol, 1.5 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. A mixture of NH_4F aq. (25% solution, 15 mL) and *t*-butyl methyl ether (30 mL) was added to the reaction mixture. After separation, the aqueous layer was extracted with *t*-butyl methyl ether (20 mL×3). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford compound **1r** as a white solid (1.64 g, 7.71 mmol, 51% yield). The NMR spectral data for **1r** were in accordance with those reported in the literature.[8]



Synthesis of 1,1-diphenyl-3-methyl-3-buten-1-ol (1s)

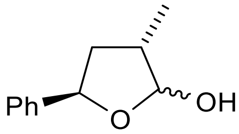
The reaction was performed under an argon atmosphere. A reaction vessel equipped with a reflux condenser and a three-way cock was dried. Mg (1.64 g, 67.5 mmol, 2.25 eq.) and THF (22.0 mL) were added to the reaction vessel. The reaction mixture was cooled to 5 °C. *β*-Methallyl chloride (4.38 mL, 45.0 mmol, 1.5 eq.) was added dropwise to the mixture, which was kept at 5 °C during the addition. After stirring at room temperature for 1 h, the mixture was refluxed for 2 h. A THF (12.0 mL) solution of benzophenone (5.47 g, 30.0 mmol, 1.0 eq.) was added dropwise to the reaction mixture over 15 min at 0 °C. After stirring at room temperature for 16 h, saturated NH_4Cl aq. (23 mL) was added. After separation, the aqueous layer was extracted with ethyl acetate (20 mL × 4). The collected organic layer was dried over Na_2SO_4. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 12/1) to afford compound **1s** as a white solid (4.98 g, 20.9 mmol, 70% yield). The NMR spectral data for **1s** were in accordance with those reported in the literature.[9]

Synthesis of 2-Hydroxytetrahydrofurans 2



Synthesis of 2-hydroxy-3-methyltetrahydrofuran (2a)

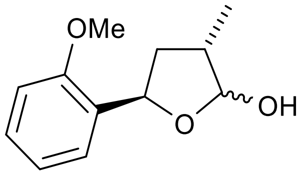
To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. H_2O (2.0 mL) was added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1a** (50.5 µL, 0.50 mmol) was added to the mixture over 10 h by a syringe pump, and the reaction mixture was stirred for an additional 8 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 5), and the combined organic layer was dried over Na_2SO_4. After filtration, the filtrate was concentrated under vacuum carefully. Compound **2a** was obtained as a yellow oil (45.8 mg, 0.45 mmol, 90% yield). The ^{1}H and ^{13}C NMR data were in accordance with those reported previously.[10]



Synthesis of 2-hydroxy-3-methyl-5-phenyltetrahydrofuran (2b)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.40 mL) and H_2O (1.60 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1b** (82.8 µL, 0.50 mmol) was added to the mixture over 10 h by a syringe pump, and the reaction mixture was stirred for an additional 8 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2b** as a pale yellow oil (53.7 mg, 0.30 mmol, 60% yield, *trans*:*cis* = 92:8).

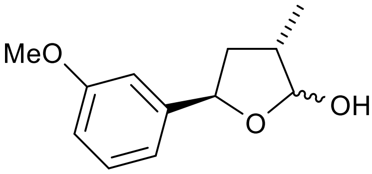
^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.46–7.18 (m, 5H), 5.23 (d, J = 2.5 Hz, 1H), 5.17 (dd, J = 6.8, 9.0 Hz, 1H), 2.81 (d, J = 3.3 Hz, 1H), 2.45–1.83 (m, 3H), 1.10 (d, J = 7.1 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.46–7.18 (m, 5H), 5.52 (t, J = 3.6 Hz, 1H), 5.31 (dd, J = 3.3, 8.5 Hz, 1H), 2.51 (d, J = 3.2 Hz, 1H), 2.45–1.83 (m, 3H), 1.08 (d, J = 5.5 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 143.3, 128.5 (2C), 127.5, 126.4 (2C), 104.6, 81.6, 41.3, 40.0, 16.5. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 144.0, 128.5 (2C), 127.4, 125.5 (2C), 100.2, 79.4, 39.3, 37.4, 12.9. HRMS (ESI): *m/z* calcd for $C_{11}H_{14}NaO_2$ $[M+Na]^+$ 201.0892, found 201.0893.



Synthesis of 2-hydroxy-5-(2-methoxyphenyl)-3-methyltetrahydrofuran (2c)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol), BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. A DMF solution of **1c** (2.5 M, 200 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2c** as a yellow oil (46.1 mg, 0.22 mmol, 44% yield, *trans*:*cis* = 93:7).

^1H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.66–7.12 (m, 2H), 7.03–6.73 (m, 2H), 5.46 (t, J = 7.7 Hz, 1H), 5.21 (d, J = 4.2 Hz, 1H), 3.82 (s, 3H), 3.16 (d, J = 4.5 Hz, 1H), 2.48–1.74 (m, 3H), 1.09 (d, J = 7.2 Hz, 3H). ^1H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.66–7.12 (m, 2H), 7.03–6.73 (m, 2H), 5.58–5.51 (m, 2H), 3.82 (s, 3H), 2.75 (d, J = 3.2 Hz, 1H), 2.48–1.74 (m, 3H), 1.05 (d, J = 5.9 Hz, 3H). $^{13}C\{^1H\}$ NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 156.2, 131.7, 128.3, 127.1, 120.8, 110.3, 104.8, 75.3, 55.4, 41.0, 38.1, 16.5. $^{13}C\{^1H\}$ NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 156.0, 132.3, 128.0, 125.6, 120.3, 110.1, 100.1, 77.4, 55.4, 37.9, 36.9, 12.8. HRMS (ESI): *m*/*z* calcd for $C_{12}H_{16}NaO_3$ $[M+Na]^+$ 231.0997, found 231.0987.

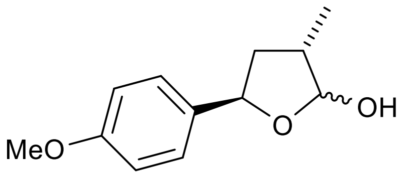


Synthesis of 2-hydroxy-5-(3-methoxyphenyl)-3-methyltetrahydrofuran (2d)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1d** (94.5 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.7 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2d** as a yellow oil (48.2 mg, 0.23 mmol, 46% yield, *trans*:*cis* = 94:6).

^1H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.32–6.68 (m, 4H), 5.21 (d, J = 2.0 Hz, 1H), 5.14 (dd, J = 8.9, 6.8 Hz, 1H), 3.79 (s, 3H), 3.26 (d, J = 3.1 Hz, 1H), 2.41–1.87 (m, 3H), 1.08 (d, J = 7.1 Hz, 3H). ^1H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.32–6.68 (m, 4H), 5.50

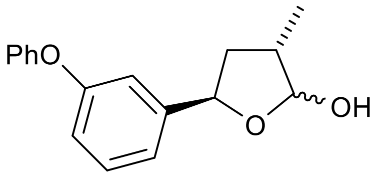
(t, J = 3.4 Hz, 1H), 5.28 (dd, J = 3.2, 8.4 Hz, 1H), 3.79 (s, 3H), 2.94 (d, J = 2.7 Hz, 1H), 2.41−1.87 (m, 3H), 1.06 (d, J = 6.5 Hz, 3H). $^{13}C\{^1H\}$ NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 159.8, 145.1, 129.5, 118.7, 113.0, 111.9, 104.6, 81.4, 55.3, 41.1, 40.0, 16.5. $^{13}C\{^1H\}$ NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 159.8, 145.7, 129.5, 117.8, 113.0, 112.5, 100.2, 79.2, 55.3, 39.3, 37.2, 12.8. HRMS (ESI): *m/z* calcd for $C_{12}H_{16}NaO_3$ $[M+Na]^+$ 231.0997, found 231.0997.



Synthesis of 2-hydroxy-5-(4-methoxyphenyl)-3-methyltetrahydrofuran (2e)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.40 mL) and H_2O (1.60 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1e** (94.5 μL, 0.50 mmol) was added to the mixture over 10 h by a syringe pump, and the reaction mixture was stirred for an additional 5 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2e** as a brown solid (54.1 mg, 0.26 mmol, 52% yield, *trans*:*cis* = 57:43).

^1H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.46−7.05 (m, 2H), 7.00−6.68 (m, 2H), 5.18 (d, J = 2.3 Hz, 1H), 5.11 (dd, J = 6.6, 9.2 Hz, 1H), 3.78 (s, 3H), 3.20 (d, J = 3.1 Hz, 1H), 2.41−1.87 (m, 3H), 1.07 (d, J = 8.7 Hz, 3H). ^1H NMR for other three diastereomers (300 MHz, $CDCl_3$) δ 7.46−7.05 (m, 2H), 7.00−6.68 (m, 2H), 5.57−4.84 (m, 2H), 3.78 (s, 3H), 3.14−2.76 (m, 1H), 2.54−1.37 (m, 3H), 1.18−1.01 (m, 3H). $^{13}C\{^1H\}$ NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 159.1, 135.2, 127.9 (2C), 113.8 (2C), 104.3, 81.3, 55.4, 41.1, 40.0, 16.5. $^{13}C\{^1H\}$ NMR for other three diastereomers (75 MHz, $CDCl_3$) δ 159.1, 158.8, 135.9, 135.0, 134.0, 127.3 (2C), 126.8 (2C), 113.9 (2C), 113.8 (2C), 104.9, 100.0, 99.4, 82.1, 79.8, 79.0, 55.4, 42.4, 42.0, 40.2, 40.1, 39.3, 37.4, 17.8, 12.9, 12.7. HRMS (ESI): *m/z* calcd for $C_{12}H_{16}NaO_3$ $[M+Na]^+$ 231.0997, found 231.0988.

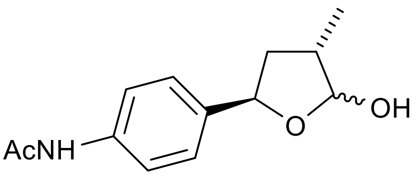


Synthesis of 2-hydroxy-3-methyl-5-(3-phenoxyphenyl)tetrahydrofuran (2f)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (162.1 mg, 1.50 mmol) were added, and O_2 was purged. DMF (1.0 mL) and H_2O (1.0 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1f** (117.8 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total).

The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1.0 M, 2.0 mL × 3), and was dried over Na_2SO_4. After filtration, the filtrate was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2f** as a yellow solid (52.9 mg, 0.20 mmol, 39% yield, *trans*:*cis* = 89:11).

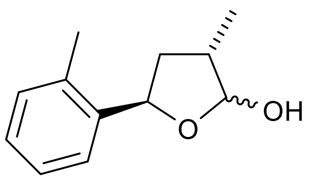
^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.37−7.21 (m, 3H), 7.18−6.80 (m, 6H), 5.20 (d, J = 1.7 Hz, 1H), 5.13 (dd, J = 6.7, 9.0 Hz, 1H), 2.67 (d, J = 3.3 Hz, 1H), 2.40−2.10 (m, 2H), 2.05−1.89 (m, 1H), 1.07 (d, J = 7.2 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.37−7.21 (m, 3H), 7.18−6.80 (m, 6H), 5.47 (d, J = 1.4 Hz, 1H), 5.27 (dd, J = 3.0, 8.3 Hz, 1H), 2.43 (d, J = 2.9 Hz, 1H), 2.40−2.10 (m, 2H), 2.05−1.89 (m, 1H), 1.06 (d, J = 6.5 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 157.4, 157.3, 145.7, 129.8, 129.8, 123.3, 121.2, 119.0, 117.8, 116.9, 104.6, 81.1, 41.1, 40.0, 16.5. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 157.4, 157.3, 146.3, 129.9, 129.8, 123.3, 120.4, 118.9, 117.5, 116.1, 100.2, 79.1, 39.3, 37.2, 12.8. HRMS (ESI): *m*/*z* calcd for $C_{17}H_{18}NaO_3$ $[M+Na]^+$ 293.1154, found 293.1158.



Synthesis of 5-(4-acetamidophenyl)-2-hydroxy-3-methyltetrahydrofuran (2g)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.33 mL) and H_2O (1.67 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1g** (3.3 M, 150.6 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 1/1.5) afforded **2g** as a brown oil (58.4 mg, 0.25 mmol, 50% yield, *trans*:*cis* = 92:8).

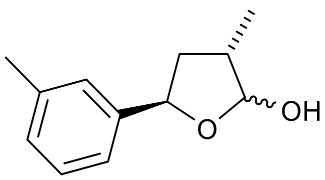
^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.49−7.18 (m, 4H), 5.22 (d, J = 2.7 Hz, 1H), 5.12 (dd, J = 6.8, 9.0 Hz, 1H), 3.21 (d, J = 3.1 Hz, 1H), 2.43−1.82 (m, 3H), 2.15 (s, 3H), 1.08 (d, J = 7.2 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.49−7.18 (m, 4H), 5.50 (t, J = 3.9 Hz, 1H), 5.26 (dd, J = 3.2, 8.6 Hz, 1H), 2.64 (d, J = 3.2 Hz, 1H), 2.43−1.82 (m, 3H), 2.15 (s, 3H), 1.07 (d, J = 6.6 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 169.3, 139.1, 137.2, 127.0 (2C), 120.3 (2C), 104.4, 81.0, 41.1, 40.0, 24.4, 16.4. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 169.2, 139.8, 137.0, 126.1 (2C), 120.3 (2C), 100.0, 78.9, 39.3, 37.2, 24.4, 12.9. HRMS (ESI): *m*/*z* calcd for $C_{13}H_{17}NNaO_3$ $[M+Na]^+$ 258.1106, found 258.1096.



Synthesis of 2-hydroxy-3-methyl-5-(2-methylphenyl)tetrahydrofuran (2h)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1h** (90.7 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2h** as an orange oil (44.3 mg, 0.23 mmol, 46% yield, *trans*:*cis* = 94:6).

^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.73–7.01 (m, 4H), 5.40 (dd, *J* = 7.6, 11.7 Hz, 1H), 5.24 (s, 1H), 3.29 (s, 1H), 2.40–2.01 (m, 3H), 2.29 (s, 3H), 1.11 (d, *J* = 7.2 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.73–7.01 (m, 4H), 5.57 (d, *J* = 3.8 Hz, 1H), 5.48 (dd, *J* = 3.1, 8.2 Hz, 1H), 2.97 (s, 1H), 2.40–2.01 (m, 3H), 2.27 (s, 3H), 1.07 (d, *J* = 6.4 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 142.1, 134.1, 130.1, 127.0, 126.4, 125.6, 104.6, 78.1, 40.9, 38.7, 19.3, 16.4. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 141.6, 133.9, 130.3, 126.9, 126.0, 124.3, 100.2, 76.9, 38.0, 37.0, 19.3, 12.8. HRMS (ESI): *m*/*z* calcd for $C_{12}H_{16}NaO_2$ $[M+Na]^+$ 215.1048, found 215.1055.

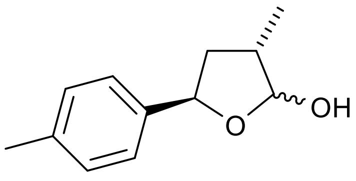


Synthesis of 2-hydroxy-3-methyl-5-(3-methylphenyl)tetrahydrofuran (2i)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.40 mL) and H_2O (1.60 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1i** (90.9 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2i** as an orange oil (32.9 mg, 0.17 mmol, 34% yield, *trans*:*cis* = 88:12).

^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.28–7.01 (m, 4H), 5.22 (d, *J* = 2.4 Hz, 1H), 5.14 (dd, *J* = 6.6, 9.2 Hz, 1H), 2.91 (d, *J* = 3.4 Hz, 1H), 2.43–1.89 (m, 3H), 2.34 (s, 3H), 1.09 (d, *J* = 7.2 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.28–7.01 (m, 4H), 5.52

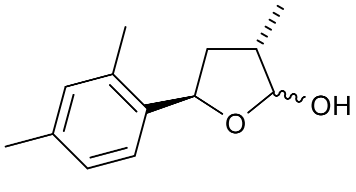
(t, *J* = 3.8, 3.9 Hz, 1H), 5.28 (dd, *J* = 3.3, 8.5 Hz, 1H), 2.62 (d, *J* = 3.2 Hz, 1H), 2.43−1.89 (m, 3H), 2.33 (s, 3H), 1.07 (d, *J* = 6.7 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) *δ* 143.2, 138.1, 128.4, 128.3, 127.1, 123.5, 104.6, 81.6, 41.3, 39.9, 21.6, 16.5. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) *δ* 143.9, 138.1, 128.4, 128.0, 126.2, 122.6, 100.2, 79.4, 39.3, 37.4, 21.6, 12.9. HRMS (ESI): *m*/*z* calcd for $C_{12}H_{16}NaO_2$ [M+Na]$^+$ 215.1048, found 215.1048.



Synthesis of 2-hydroxy-3-methyl-5-(4-methylphenyl)tetrahydrofuran (2j)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.40 mL) and H_2O (1.60 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1j** (90.7 µL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2j** as a yellow solid (31.3 mg, 0.16 mmol, 33% yield, *trans*:*cis* = 86:14).

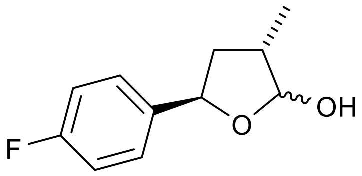
^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) *δ* 7.34−7.08 (m, 4H), 5.19 (d, *J* = 1.6 Hz, 1H), 5.13 (dd, *J* = 6.6, 9.1 Hz, 1H), 3.30 (d, *J* = 3.1 Hz, 1H), 2.45−2.07 (m, 2H), 2.33 (s, 3H), 2.02−1.87 (m, 1H), 1.08 (d, *J* = 7.1 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) *δ* 7.34−7.08 (m, 4H), 5.49 (dd, *J* = 2.3, 4.1 Hz, 1H), 5.28 (dd, *J* = 3.3, 8.5 Hz, 1H), 2.99 (d, *J* = 2.5 Hz, 1H), 2.45−2.07 (m, 2H), 2.32 (s, 3H), 2.02−1.87 (m, 1H), 1.06 (d, *J* = 6.7 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) *δ* 140.2, 137.2, 129.2 (2C), 126.5 (2C), 104.4, 81.5, 41.2, 40.0, 21.2, 16.5. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) *δ* 140.9, 136.9, 129.1 (2C), 125.5 (2C), 100.1, 79.3, 39.3, 37.3, 21.2, 12.9. HRMS (ESI): *m*/*z* calcd for $C_{12}H_{16}NaO_2$ [M+Na]$^+$ 215.1048, found 215.1039.



Synthesis of 2-hydroxy-3-methyl-5-(2,4-dimethylphenyl)tetrahydrofuran (2k)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (81.1 mg, 0.75 mmol) were added, and O_2 was purged. DMF (0.67 mL) and H_2O (1.33 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1k** (98.8 µL, 0.50 mmol) was added to the mixture over 10 h by a syringe pump, and the reaction mixture was stirred for an additional 8 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3), and was dried over Na_2SO_4. The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 6/1) afforded **2k** as a brown oil (49.1 mg, 0.24 mmol, 48% yield, *trans*:*cis* = 90:10).

^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.54 (d, J = 5.9 Hz, 1H), 7.01 (dd, J = 5.9, 11.6 Hz, 1H), 6.95 (d, J = 6.5 Hz, 1H), 5.38 (t, J = 5.7 Hz, 1H), 5.22 (d, J = 0.93 Hz, 1H), 3.53 (s, 1H), 2.48–1.92 (m, 2H), 2.29 (s, 3H), 2.27 (s, 3H), 2.14–1.91 (m, 1H), 1.10 (d, J = 5.4 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.25 (d, J = 5.5 Hz, 1H), 7.01 (dd, J = 5.9, 11.6 Hz, 1H), 6.95 (d, J = 6.5 Hz, 1H), 5.56 (t, J = 2.4 Hz, 1H), 5.49–5.43 (m, 1H), 3.22 (s, 1H), 2.48–1.92 (m, 2H), 2.29 (s, 3H), 2.24 (s, 3H), 2.14–1.91 (m, 1H), 1.07 (d, J = 4.8 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 138.5, 136.6, 134.1, 131.2, 126.6, 125.7, 104.6, 76.9, 40.9, 38.8, 21.0, 19.2, 16.4. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 139.1, 136.5, 133.8, 130.9, 127.1, 124.4, 100.1, 78.0, 38.1, 37.0, 21.1, 19.2, 12.8. HRMS (ESI): *m*/*z* calcd for $C_{13}H_{18}NaO_2$ $[M+Na]^+$ 229.1205, found 229.1212.

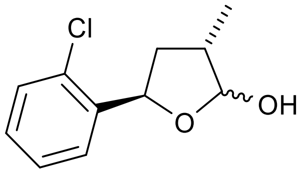


Synthesis of 5-(4-fluorophenyl)-2-hydroxy-3-methyltetrahydrofuran (2l)

Procedure A: To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.40 mL) and H_2O (1.60 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1l** (84.6 µL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2l** as a pale yellow oil (36.8 mg, 0.19 mmol, 37% yield, *trans*:*cis* = 97:3).

Procedure B: To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (81.1 mg, 0.75 mmol) were added, and O_2 was purged. DMF (0.40 mL) and H_2O (1.60 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1l** (84.6 µL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 8 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3), and was dried over Na_2SO_4. The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2l** as a pale yellow oil (50.8 mg, 0.26 mmol, 52% yield, *trans*:*cis* = 96:4).

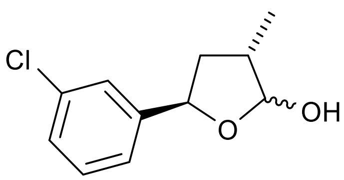
^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.47–7.16 (m, 2H), 7.13–6.96 (m, 2H), 5.20 (d, J = 2.7 Hz, 1H), 5.14 (dd, J = 6.9, 9.0 Hz, 1H), 3.10 (d, J = 3.1 Hz, 1H), 2.44–2.06 (m, 2H), 2.04−1.89 (m, 1H), 1.08 (d, $J = 7.2$ Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.47−7.16 (m, 2H), 7.13−6.96 (m, 2H), 5.49 (t, $J = 3.7$ Hz, 1H), 5.27 (dd, $J = 3.3$, 8.4 Hz, 1H), 2.75 (d, $J = 3.2$ Hz, 1H), 2.44−2.06 (m, 2H), 2.04−1.89 (m, 1H), 1.07 (d, $J = 6.4$ Hz, 3H). $^{13}C\{^1H\}$ NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 162.3 (d, $^1J_{CF} = 244$ Hz), 139.0 (d, $^4J_{CF} = 3.1$ Hz), 128.2 (d, $^3J_{CF} = 8.1$ Hz, 2C), 115.3 (d, $^2J_{CF} = 21.2$ Hz, 2C), 104.4, 81.0, 41.1, 40.1, 16.5. $^{13}C\{^1H\}$ NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 162.2 (d, $^1J_{CF} = 243$ Hz), 139.6 (d, $^4J_{CF} = 3.1$ Hz), 127.1 (d, $^3J_{CF} = 7.9$ Hz, 2C), 115.3 (d, $^2J_{CF} = 14.2$ Hz, 2C), 100.1, 78.8, 39.4, 37.3, 12.9. HRMS (ESI): *m*/*z* calcd for $C_{11}H_{13}FNaO_2$ $[M+Na]^+$ 219.0797, found 219.0801.



Synthesis of 5-(2-chlorophenyl)-2-hydroxy-3-methyltetrahydrofuran (2m)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol), BQ (81.1 mg, 0.75 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1m** (88.6 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3), and was dried over Na_2SO_4. The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2m** as an orange oil (38.8 mg, 0.18 mmol, 36% yield, *trans*:*cis* = 95:5).

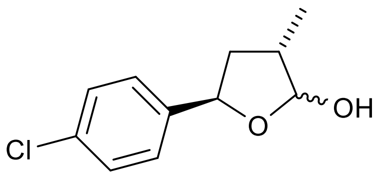
^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.84−7.07 (m, 4H), 5.63−5.46 (m, 1H), 5.26 (s, 1H), 3.56 (d, $J = 3.5$ Hz, 1H), 2.43−1.83 (m, 3H), 1.10 (d, $J = 7.0$ Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.84−7.07 (m, 4H), 5.63−5.46 (m, 2H), 3.19 (s, 1H), 2.43−1.83 (m, 3H), 1.06 (d, $J = 6.6$ Hz, 3H). $^{13}C\{^1H\}$ NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 141.3, 131.6, 129.2, 128.3, 127.5, 126.3, 105.0, 78.2, 40.5, 38.7, 16.3. $^{13}C\{^1H\}$ NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 141.5, 131.5, 129.5, 128.2, 127.1, 126.8, 100.4, 77.0, 38.0, 36.7, 12.6. HRMS (ESI): *m*/*z* calcd for $C_{11}H_{13}ClNaO_2$ $[M+Na]^+$ 235.0502, found 235.0501.



Synthesis of 5-(3-chlorophenyl)-2-hydroxy-3-methyltetrahydrofuran (2n)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol), BQ (162.1 mg, 1.50 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1n** (88.6 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3), and was dried over Na_2SO_4. The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2n** as an orange oil (55.3 mg, 0.26 mmol, 52% yield, *trans*:*cis* = 96:4).

^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.53−7.07 (m, 4H), 5.24 (d, J = 2.7 Hz, 1H), 5.13 (dd, J = 7.0, 8.8 Hz, 1H), 2.81 (d, J = 3.2 Hz, 1H), 2.42−1.85 (m, 3H), 1.09 (d, J = 7.2 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.53−7.07 (m, 4H), 5.52 (t, J = 3.5 Hz, 1H), 5.34−5.20 (m, 1H), 2.49 (d, J = 3.1 Hz, 1H), 2.42−1.85 (m, 3H), 1.08 (d, J = 6.3 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 145.6, 134.4, 129.8, 127.6, 126.6, 124.5, 104.6, 80.8, 41.0, 40.1, 16.4. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 146.1, 134.4, 129.7, 127.3, 125.6, 123.7, 100.2, 78.7, 39.3, 37.1, 12.7. HRMS (ESI): *m*/*z* calcd for $C_{11}H_{13}ClNaO_2$ $[M+Na]^+$ 235.0502, found 235.0511.

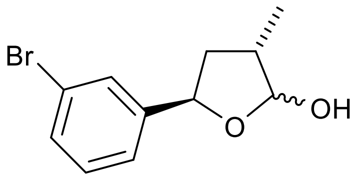


Synthesis of 5-(4-chlorophenyl)-2-hydroxy-3-methyltetrahydrofuran (2o)

Procedure A: To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1o** (88.6 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2o** as a yellow solid (34.4 mg, 0.18 mmol, 35% yield, *trans*:*cis* = 93:7).

Procedure B: To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (81.1 mg, 0.75 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1o** (88.6 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 8 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3), and was dried over Na_2SO_4. The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2o** as a yellow solid (42.3 mg, 0.20 mmol, 40% yield, *trans*:*cis* = 93:7).

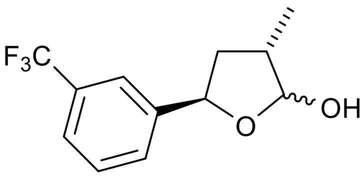
^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.43−7.12 (m, 4H), 5.20 (d, J = 2.4 Hz, 1H), 5.13 (dd, J = 6.8, 9.1 Hz, 1H), 3.24 (d, J = 3.2 Hz, 1H), 2.36−2.05 (m, 2H), 2.05−1.82 (m, 1H), 1.07 (d, J = 7.2 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.43−7.12 (m, 4H), 5.49 (t, *J* = 3.6 Hz, 1H), 5.26 (dd, *J* = 3.5, 8.2 Hz, 1H), 2.90 (d, *J* = 3.2 Hz, 1H), 2.36–2.05 (m, 2H), 2.05–1.82 (m, 1H), 1.06 (d, *J* = 6.4 Hz, 3H). $^{13}C\{^1H\}$ NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 141.9, 133.2, 128.6 (2C), 127.9 (2C), 104.5, 80.9, 41.1, 40.1, 16.4. $^{13}C\{^1H\}$ NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 142.5, 132.9, 128.6 (2C), 126.9 (2C), 100.2, 78.7, 39.3, 37.2, 12.8. HRMS (ESI): *m*/*z* calcd for $C_{11}H_{13}ClNaO_2$ $[M+Na]^+$ 235.0502, found 235.0499.



Synthesis of 5-(3-bromophenyl)-2-hydroxy-3-methyltetrahydrofuran (2p)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (81.1 mg, 0.75 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1p** (90.6 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3), and was dried over Na_2SO_4. The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 6/1) afforded **2p** as a yellow solid (48.5 mg, 0.19 mmol, 38% yield, *trans*:*cis* = 93:7).

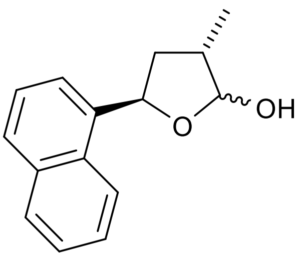
^1H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.62–7.11 (m, 4H), 5.49 (s, 1H), 5.11 (dd, *J* = 8.6, 10.4 Hz, 1H), 3.65 (d, *J* = 3.0 Hz, 1H), 2.40–2.07 (m, 2H), 2.05–1.81 (m, 1H), 1.06 (d, *J* = 7.2 Hz, 3H). ^1H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.62–7.11 (m, 4H), 5.26 (dd, *J* = 3.0, 8.1 Hz, 1H), 5.20 (d, *J* = 1.7 Hz, 1H), 3.29 (d, *J* = 2.8 Hz, 1H), 2.40–2.07 (m, 2H), 2.05–1.81 (m, 1H), 1.05 (d, *J* = 6.1 Hz, 3H). $^{13}C\{^1H\}$ NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 145.9, 130.1, 130.0, 129.5, 125.0, 122.6, 104.5, 80.8, 41.0, 40.1, 16.4. $^{13}C\{^1H\}$ NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 146.4, 130.5, 130.3, 128.5, 124.1, 122.7, 100.2, 78.6, 39.3, 37.1, 12.7. HRMS (ESI): *m*/*z* calcd for $C_{11}H_{13}ClNaO_2$ $[M+Na]^+$ 278.9997, found 278.9990.



Synthesis of 2-hydroxy-3-methyl-5-(3-trifluoromethylphenyl)tetrahydrofuran (2q)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol), BQ (162.1 mg, 1.50 mmol) were added, and O_2 was purged. DMF (0.50 mL) and H_2O (1.50 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1q** (99.2 μL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3) and was dried over Na_2SO_4. After filtration, the filtrate was concentrated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2q** as an orange oil (44.4 mg, 0.18 mmol, 36% yield, *trans*:*cis* = 93:7).

^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 7.73−7.36 (m, 4H), 5.25 (s, 1H), 5.21 (dd, J = 7.1, 9.2 Hz, 1H), 3.18 (d, J = 2.6 Hz, 1H), 2.47−1.90 (m, 3H), 1.09 (d, J = 7.2 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 7.73−7.36 (m, 4H), 5.53 (s, 1H), 5.34 (dd, J = 3.2, 8.1 Hz, 1H), 2.81 (s, 1H), 2.47−1.90 (m, 3H), 1.08 (d, J = 6.4 Hz, 3H). $^{13}C\{^1H\}$ NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 144.6, 130.8 (q, $^2J_{CF}$ = 32.1 Hz), 129.8, 129.0, 124.3 (q, $^1J_{CF}$ = 270.5 Hz), 124.3 (q, $^3J_{CF}$ = 3.7 Hz), 123.2 (q, $^3J_{CF}$ = 3.8 Hz), 104.7, 80.9, 41.3, 40.2, 16.4. $^{13}C\{^1H\}$ NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 145.1, 130.8 (q, $^2J_{CF}$ = 32.1 Hz), 129.8, 128.9,124.3 (q, $^1J_{CF}$ = 270.5 Hz), 124.1 (q, $^3J_{CF}$ = 3.9 Hz), 122.2 (q, $^3J_{CF}$ = 3.9 Hz), 100.2, 78.7, 39.3, 37.2, 12.8. HRMS (ESI): *m*/*z* calcd for $C_{24}H_{24}F_6NaO_3$ $[2M-H_2O+Na]^+$ 497.1527, found 497.1546.

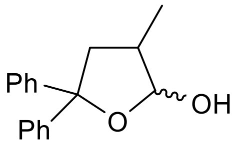


Synthesis of 2-hydroxy-3-methyl-5-(1-naphthyl)tetrahydrofuran (2r)

Procedure A: To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (5.4 mg, 0.050 mmol) were added, and O_2 was purged. DMF (0.67 mL) and H_2O (1.33 mL) were added to the mixture. After bubbling NO gas (2.24 mL, 0.10 mmol), the mixture was stirred and warmed up to 40 °C. Substrate **1r** (2.7 M, 183.2 μL, 0.50 mmol) was added to the mixture over 10 h by a syringe pump, and the reaction mixture was stirred for an additional 5 h (18 h in total). The reaction mixture was extracted with $CDCl_3$ (0.70 mL × 3). The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2r** as an orange solid (37.9 mg, 0.17 mmol, 33% yield, *trans*:*cis* = 93:7).

Procedure B: To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol) and BQ (81.1 mg, 0.75 mmol) were added, and O_2 was purged. DMF (0.67 mL) and H_2O (1.33 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. Substrate **1r** (2.7 M, 183.2 μL, 0.50 mmol) was added to the mixture over 10 h by a syringe pump, and the reaction mixture was stirred for an additional 8 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3), and was dried over Na_2SO_4. The combined organic layer was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2r** as an orange solid (47.1 mg, 0.21 mmol, 41% yield, *trans*:*cis* = 95:5).

^{1}H NMR for *trans*-major diastereomer (300 MHz, $CDCl_3$) δ 8.02−7.39 (m, 7H), 5.95 (t, J = 7.3 Hz, 1H), 5.65 (d, J = 4.3 Hz, 1H), 2.90 (s, 1H), 2.52−2.15 (m, 2H), 2.08−1.97 (m, 1H), 1.18 (d, J = 6.9 Hz, 3H). ^{1}H NMR for *trans*-minor diastereomer (300 MHz, $CDCl_3$) δ 8.02−7.39 (m, 7H), 6.03 (dd, J = 2.6, 8.9 Hz, 1H), 5.33 (s, 1H), 3.18 (d, J = 3.8 Hz, 1H), 2.52−2.15 (m, 2H), 2.08−1.97 (m, 1H), 1.09 (d, J = 6.7 Hz, 3H). ^{13}C{^{1}H} NMR for *trans*-major diastereomer (75 MHz, $CDCl_3$) δ 139.2, 133.8, 130.3, 129.0, 127.6, 126.0, 125.6, 125.5, 123.3, 121.6, 100.2, 77.0, 38.5, 37.2, 12.7. ^{13}C{^{1}H} NMR for *trans*-minor diastereomer (75 MHz, $CDCl_3$) δ 139.3, 133.7, 130.0, 129.0, 127.7, 126.0, 125.8, 125.6, 123.1, 122.6, 104.9, 78.0, 40.8, 39.4, 16.4. HRMS (ESI): *m*/*z* calcd for $C_{15}H_{16}NaO_2$ [M+Na]$^+$ 251.1048, found 251.1050.

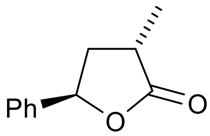


Synthesis of 5,5-diphenyl-2-hydroxy-3-methyltetrahydrofuran (2s)

To a reaction vessel, $PdCl_2(MeCN)_2$ (13.0 mg, 0.050 mmol), BQ (81.1 mg, 0.75 mmol) were added, and O_2 was purged. DMF (1.00 mL) and H_2O (1.00 mL) were added to the mixture. The mixture was stirred and warmed up to 40 °C. A DMF solution of **1s** (3.4 M, 146.2 µL, 0.50 mmol) was added to the mixture over 15 h by a syringe pump, and the reaction mixture was stirred for an additional 3 h (18 h in total). The reaction mixture was extracted with $CHCl_3$ (2.0 mL × 3). The combined organic layer was washed with NaOH aq. (1 M, 2.0 mL × 3) and was dried over Na_2SO_4. After filtration, the filtrate was concentrated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 9/1) afforded **2s** as a pale yellow oil (46.3 mg, 0.18 mmol, 36% yield).

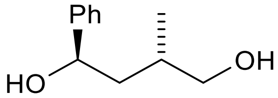
^{1}H NMR for major diastereomer (300 MHz, $CDCl_3$) δ 7.60−7.11 (m, 10H), 5.52 (t, J = 4.0 Hz, 1H), 2.75−2.61 (m, 2H), 2.43 (t, J = 12.4 Hz, 1H), 2.33−2.08 (m, 1H), 1.08 (d, J = 6.7 Hz, 3H). ^{1}H NMR for minor diastereomer (300 MHz, $CDCl_3$) δ 7.60−7.11 (m, 10H), 5.26 (t, J = 5.5 Hz, 1H), 3.03−2.87 (m, 2H), 2.29 (t, J = 10.9 Hz, 1H), 2.33−2.08 (m, 1H), 1.05 (d, J = 6.7 Hz, 3H). ^{13}C{^{1}H} NMR for major diastereomer (75 MHz, $CDCl_3$) δ 147.3, 146.6, 128.3 (2C), 128.2 (2C), 126.9 (2C), 126.8 (2C), 126.2 (2C), 125.7 (2C), 99.7, 88.9, 44.1, 38.2, 12.5. ^{13}C{^{1}H} NMR for minor diastereomer (75 MHz, $CDCl_3$) δ 147.5, 147.3, 128.3 (2C), 128.2 (2C), 126.8 (2C), 126.8 (2C), 125.9 (2C), 125.5 (2C), 105.7, 88.3 46.0, 41.7, 16.1. HRMS (ESI): *m*/*z* calcd for $C_{17}H_{18}NaO_2$ [M+Na]$^+$ 277.1205, found 277.1195.

Synthesis of 3–6



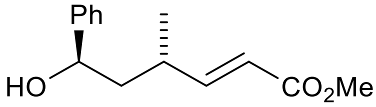
Synthesis of 3-methyl-5-phenyltetrahydrofuran-2-one (3)

To a reaction vessel, **2b** (66.2 mg, 0.37 mmol) and pyridinium dichromate (278.4 mg, 0.74 mmol) were added. To the mixture, CH_2Cl_2 (2.3 mL) was added and the reaction mixture was stirred for 24 h. Purification by silica gel column chromatography (hexane/ethyl acetate = 4/1) afforded **3** as a pale yellow oil (61.9 mg, 0.35 mmol, 95% yield, *trans*:*cis* = 90:10). The ^{1}H and ^{13}C NMR data were in accordance with those reported previously.[11]



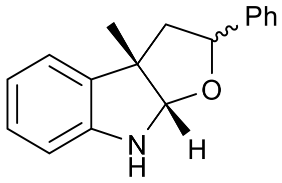
Synthesis of 3-methyl-1-phenyl-1,4-butanediol (4)

To a reaction vessel, **2b** (37.2 mg, 0.21 mmol) and $NaBH_4$ (16.1 mg, 0.43 mmol) were added. To the mixture, MeOH (0.27 mL) and THF (0.83 mL) were added and the reaction mixture was stirred for 26 h. The reaction was quenched with H_2O (4.8 mL). The resulting mixture was extracted with CH_2Cl_2. The combined organic layers were dried over $MgSO_4$. The solvent was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 6/1) afforded **4** as a pale yellow oil (30.9 mg, 0.17 mmol, 82% yield, *trans*:*cis* = 90:10). The ^{1}H and ^{13}C NMR data were in accordance with those reported previously.[12]



Synthesis of methyl 6-hydroxy-4-methyl-6-phenyl-2-hexenoate (5)

To a reaction vessel, **2b** (36.1 mg, 0.20 mmol) and (carbomethoxymethylene)triphenyl phosphorene (202.8 mg, 0.61 mmol) were added. To the mixture, CH_2Cl_2 (4.0 mL) was added and the reaction mixture was stirred for 47 h. The reaction mixture was evaporated under vacuum. Purification by silica gel column chromatography (hexane/ethyl acetate = 4/1) afforded **5** as a yellow oil (42.2 mg, 0.18 mmol, 89% yield, *trans*:*cis* = 91:9). The ^{1}H and ^{13}C NMR data were in accordance with those reported previously.[13]

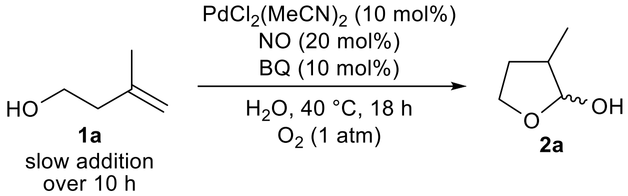


Synthesis of 6

To a reaction vessel, **2b** (47.4 mg, 0.27 mmol) and CH_3CO_2H/H_2O (1:1, 1.3 mL) were added. Phenylhydrazine (26.4 µL, 0.27 mmol) was added to the solution, and the reaction mixture was stirred at 60 °C for 5 h. After cooling to room temperature, saturated $NaHCO_3$ aq. (3.3 mL) was added to the reaction mixture. The mixture was extracted with ethyl acetate, and the organic layer was dried over $MgSO_4$. After filtration and concentration under vacuum, the residue was purified by silica gel column chromatography (hexane/ethyl acetate = 9/1) to afford **6** as a brown oil (35.9 mg, 0.14 mmol, 54% yield, diastereomer ratio = 63:37).

^{1}H NMR for major diastereomer (300 MHz, $CDCl_3$) δ 7.37–7.00 (m, 7H), 6.83–6.69 (m, 1H), 6.68–6.59 (m, 1H), 5.30 (s, 1H), 5.02 (dd, J = 6.4, 9.1 Hz, 1H), 4.80 (br s, 1H), 2.49 (dd, J = 6.4, 12.4 Hz, 1H), 2.19 (dd, J = 9.1, 12.5 Hz, 1H), 1.45 (s, 3H). ^{1}H NMR for minor diastereomer (300 MHz, $CDCl_3$) δ 7.37–7.00 (m, 7H), 6.83–6.69 (m, 1H), 6.68–6.59 (m, 1H), 5.48 (s, 1H), 4.76 (dd, J = 4.5, 11.2 Hz, 1H), 4.66 (br s, 1H), 2.53 (dd, J = 4.6, 11.9 Hz, 1H), 2.02 (dd, J = 11.3, 12.0 Hz, 1H), 1.51 (s, 3H). ^{13}C{^{1}H} NMR for major diastereomer (75 MHz, $CDCl_3$) δ 147.5, 141.6, 136.0, 128.3, 128.0, 127.6, 126.2 (2C), 123.1, 119.4, 109.6, 101.2, 79.0, 54.1, 49.0, 24.2. ^{13}C{^{1}H} NMR for minor diastereomer (75 MHz, $CDCl_3$) δ 149.3, 141.2, 134.1, 128.5, 128.3, 127.7, 126.0 (2C), 123.2, 119.1, 108.5, 99.7, 80.7, 54.8, 50.3, 25.0. HRMS (ESI): m/z calcd for $C_{17}H_{17}NNaO$ $[M+Na]^+$ 274.1208, found 274.1216.

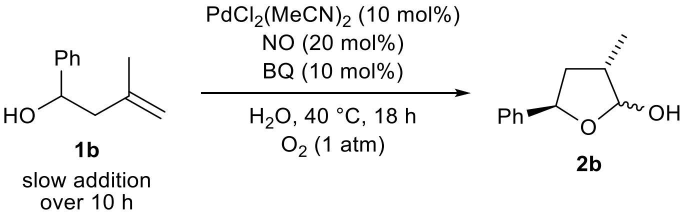
Table S1 Optimization of reaction conditions using **1a** as a substrate.[a]



entry	change from standard conditions	conv. of **1a** (%)[b]	yield of **2a** (%)[b]
1	None	100	90
2	No $PdCl_2(MeCN)_2$	0	0
3	No NO	73	13
4	No BQ	67	25
5	No NO, 300 mol% of BQ	100	79
6	No slow addition of **1a**	100	25
7	Air instead of O_2	93	56
8[c]	*t*-BuONO instead of NO	100	78
9	NO_2 instead of NO	100	86
10	CuCl instead of NO	50	9
11	$CuCl_2$ instead of NO	99	64
12	$PdCl_2$ instead of $PdCl_2(MeCN)_2$	93	66
13	$PdCl_2(PhCN)_2$ instead of $PdCl_2(MeCN)_2$	91	41
14	$PdCl_2$(cod) instead of $PdCl_2(MeCN)_2$	61	1
15	$Pd(OAc)_2$ instead of $PdCl_2(MeCN)_2$	86	36
16[d]	*t*-BuONO (10 mol%) instead of NO	85	49
17	$AgNO_2$ (10 mol%) instead of NO	63	11
18	$NaNO_2$ (10 mol%) instead of NO	83	13
19[c]	rt	97	28
20[c]	50 °C	100	55
21[d]	slow addition 5 h, total 6 h	71	29
22[d]	acetone instead of H_2O, H_2O 5.0 eq.	24	0
23[d]	1,4-dioxane instead of H_2O, H_2O 5.0 eq.	97	0
24[d]	DMF instead of H_2O, H_2O 5.0 eq.	100	0
25[d]	MeOH instead of H_2O, H_2O 5.0 eq.	25	0
26[d]	*t*-BuOH instead of H_2O, H_2O 5.0 eq.	58	13
27[d]	MeBQ instead of BQ	79	32
28[d]	2,6-Me_2BQ instead of BQ	62	16
29[d]	F_4BQ instead of BQ	70	23
30[d]	2-ClBQ instead of BQ	77	36
31[d]	2,5-Cl_2BQ instead of BQ	88	29
32[d]	maleimide instead of BQ	72	24

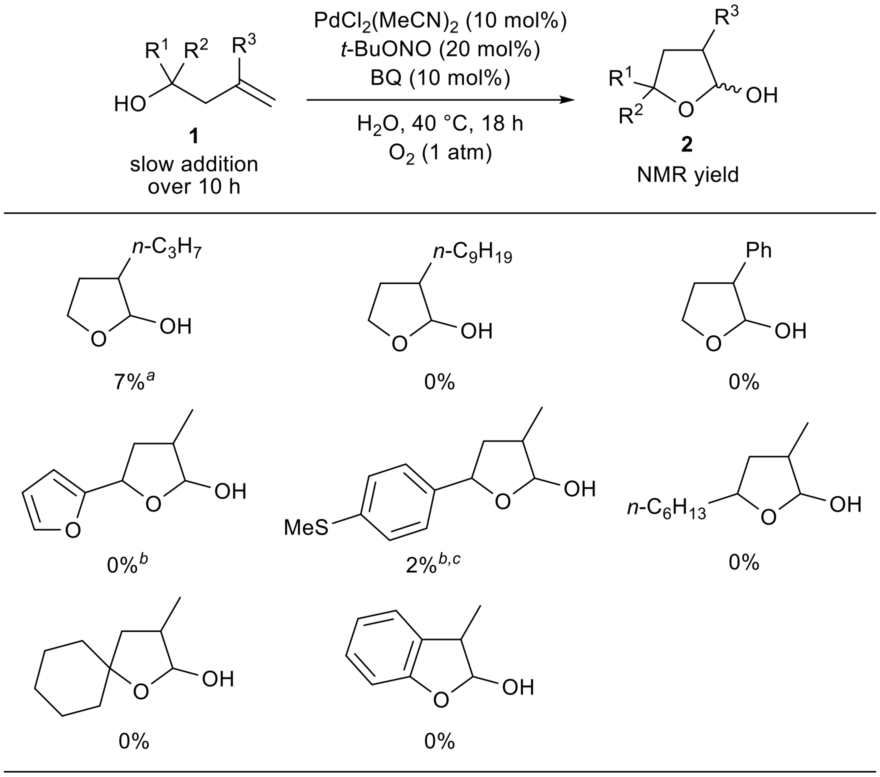
[a] Reaction conditions: **1a** (0.50 mmol), $PdCl_2(MeCN)_2$ (0.050 mmol), NO (0.10 mmol), BQ (0.050 mmol), H_2O (2.0 mL), 40 °C, O_2 (1 atm). **1a** was added over 10 h by a syringe pump, and the reaction mixture was stirred for an additional 8 h (18 h in total). [b] Determined by ^{1}H NMR. [c] *t*-BuONO (20 mol%) was used instead of NO. [d] *t*-BuONO (10 mol%) was used instead of NO.

Table S2 Optimization of reaction conditions using **1b** as a substrate.[a]



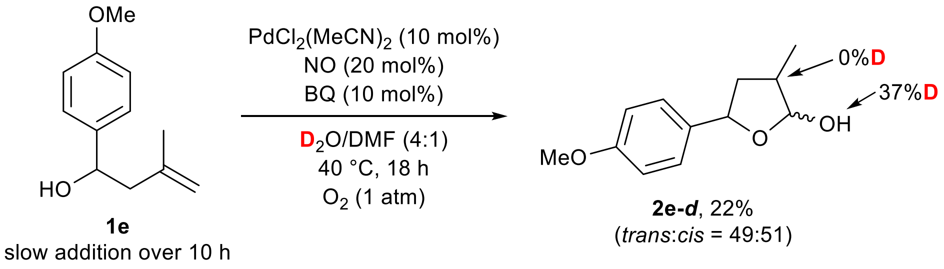
entry	change from standard conditions	conv. of **1b** (%)[b]	yield of **2b** (%)[b]
1	none	65	37
2	*n*-BuONO instead of NO	66	17
3[c]	*t*-BuONO instead of NO	60	24
4	NO_2 instead of NO	43	17
5[c]	no slow addition	53	1
6[c]	no slow addition, total 1 h	30	1
7[c]	slow addition over 15 h	49	42
8[c]	H_2O/hexane (3:1)	59	20
9[c]	H_2O/toluene (3:1)	43	5
10[c]	H_2O/dichloromethane (3:1)	38	8
11[c]	H_2O/1,2-dichloroethane (3:1)	40	1
12[c]	H_2O/acetone (10:1)	61	32
13	H_2O/acetone (5:1)	56	26
14[c]	H_2O/acetone (1:1)	43	17
15	H_2O/1,4-dioxane (5:1)	92	38
16	H_2O/acetonitrile (5:1)	81	40
17	H_2O/DMF (5:1)	98	47
18	H_2O/DMF (4:1), slow addition over 15 h	99	70 (60)[d]
19	H_2O/DMF (3:1), slow addition over 15 h	84	59
20	H_2O/DMF (2:1), slow addition over 15 h	68	46

[a] Reaction conditions: **1b** (0.50 mmol), $PdCl_2(MeCN)_2$ (0.050 mmol), NO (0.10 mmol), BQ (0.050 mmol), H_2O (2.0 mL), 40 °C, O_2 (1 atm). **1b** was added over 10 h by a syringe pump, and the reaction mixture was stirred for an additional 8 h (18 h in total). [b] Determined by ^1H NMR. [c] *t*-BuONO (20 mol%) was used instead of NO. [d] Isolated yield is shown in parentheses.



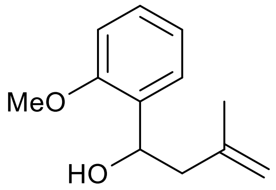
[a] NO_2 (20 mol%) was used instead of *t*-BuONO. [b] H_2O/DMF (4:1). NO (20 mol%) was used instead of *t*-BuONO. [c] **1** was added over 15 h by a syringe pump.

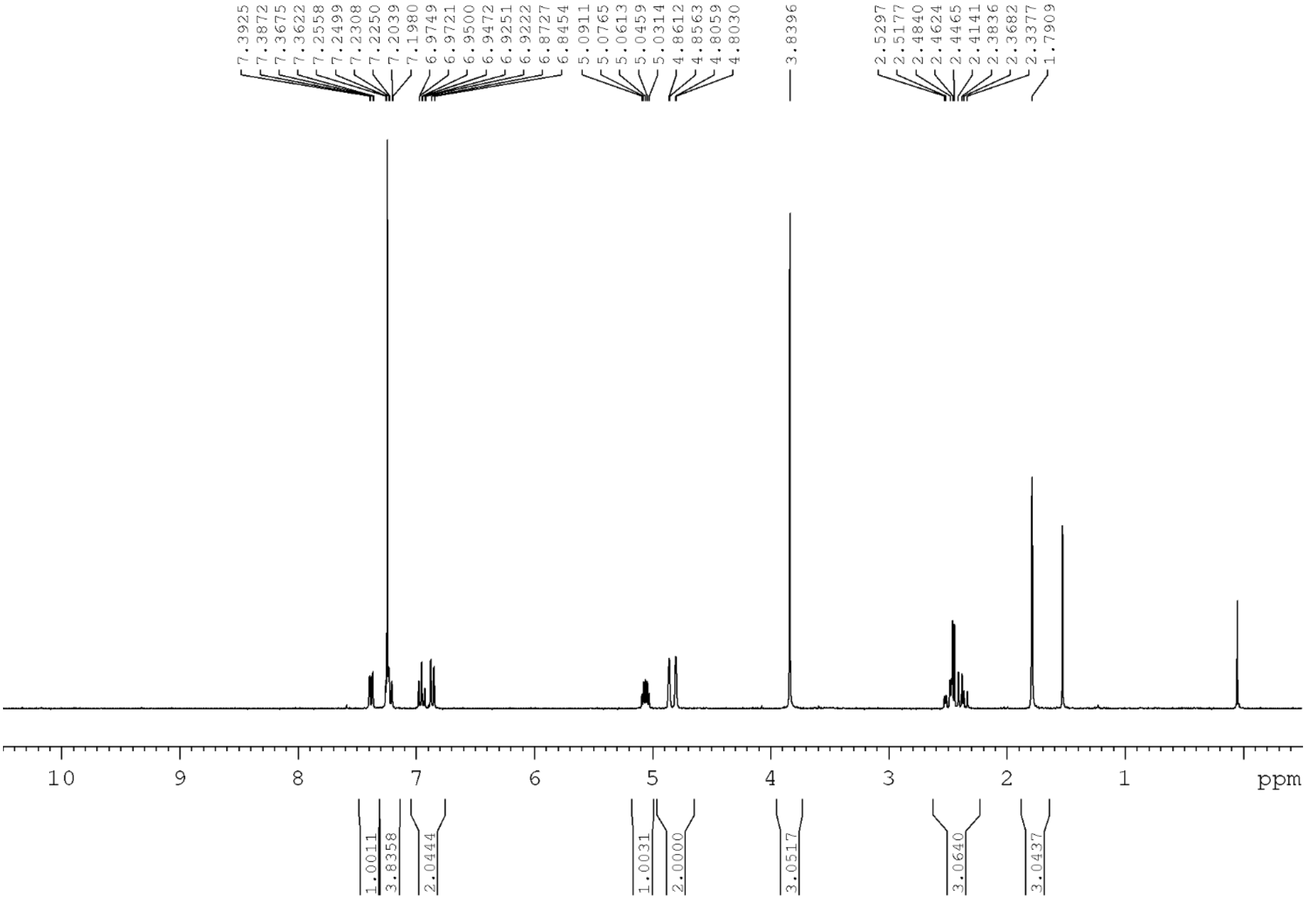
Scheme S1 Limitation of substrates.



Scheme S2 Control experiment using **1e** as a substrate in D_2O/DMF.

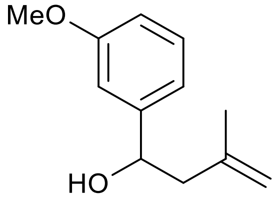
1-(2-methoxyphenyl)-3-methyl-3-buten-1-ol (1c)

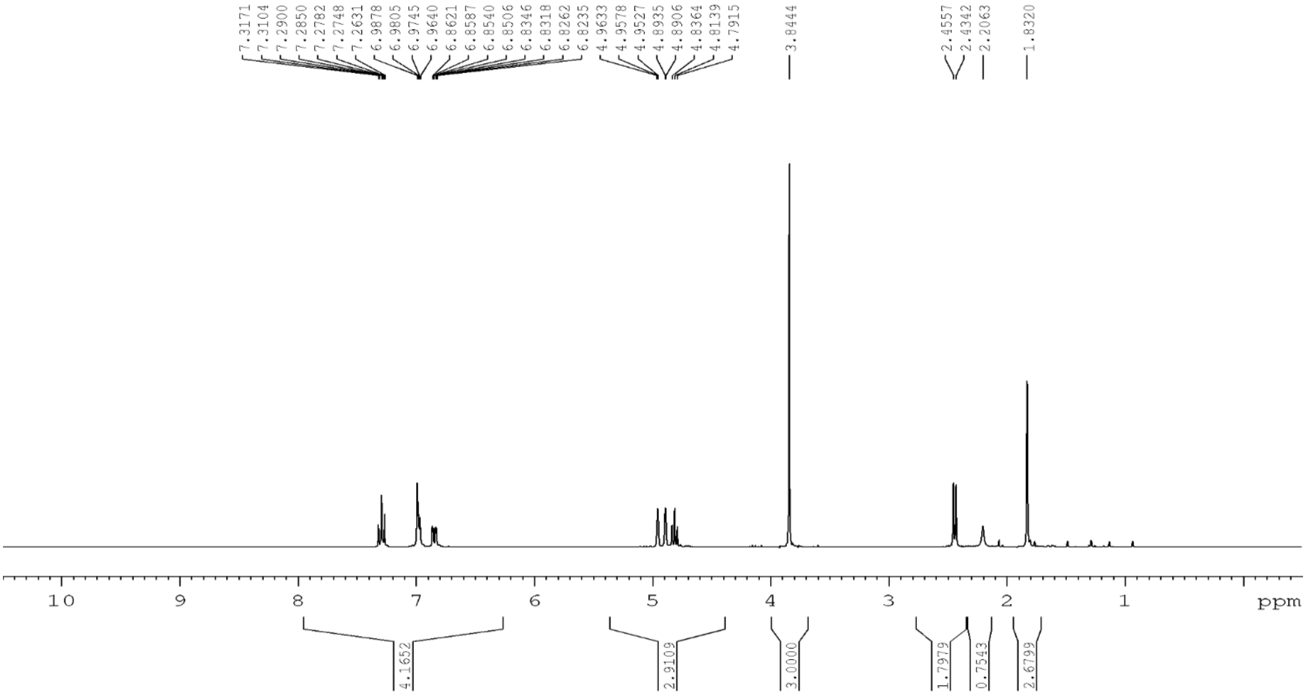




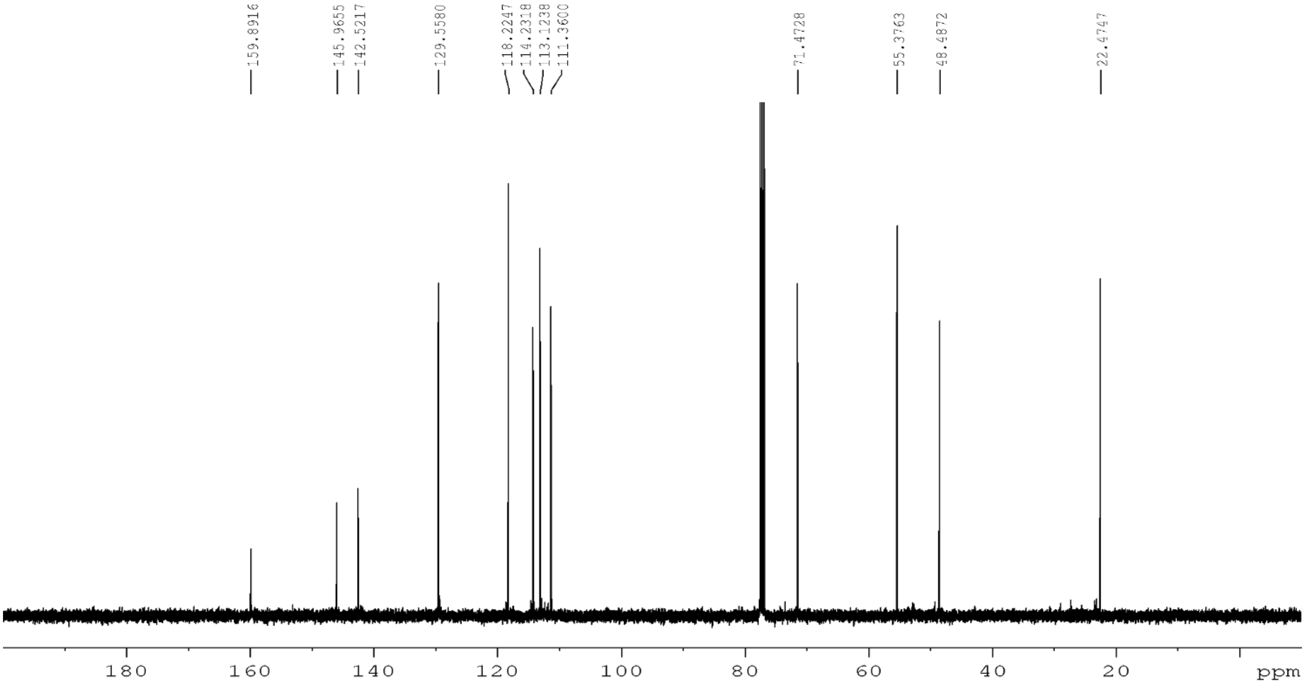
^{1}H NMR (300 MHz, CDCl$_3$)

1-(3-methoxyphenyl)-3-methyl-3-buten-1-ol (1d)



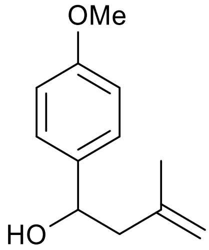


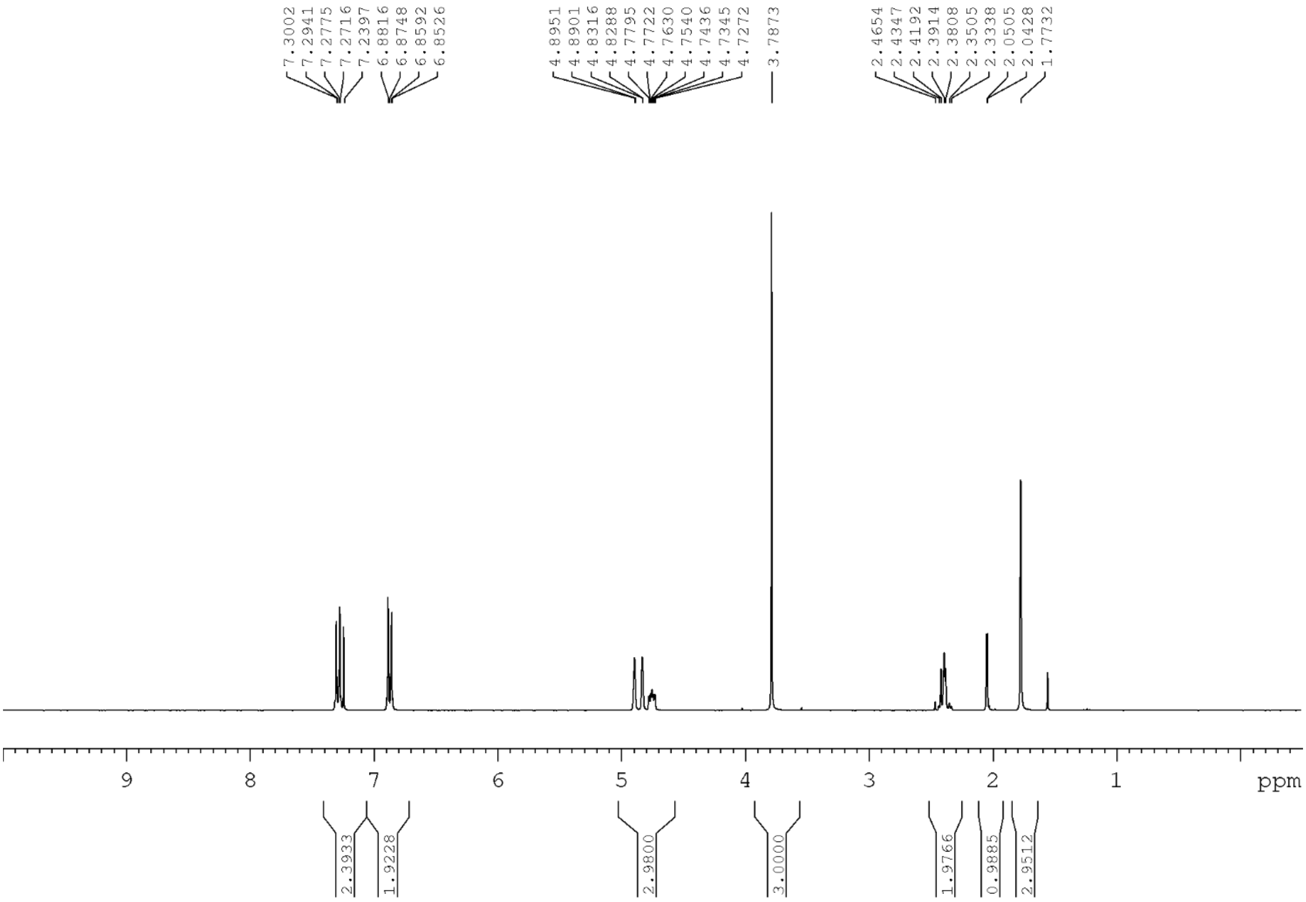
^{1}H NMR (300 MHz, CDCl$_3$)



^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$)

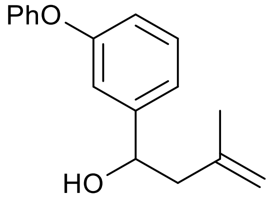
1-(4-methoxyphenyl)-3-methyl-3-buten-1-ol (1e)

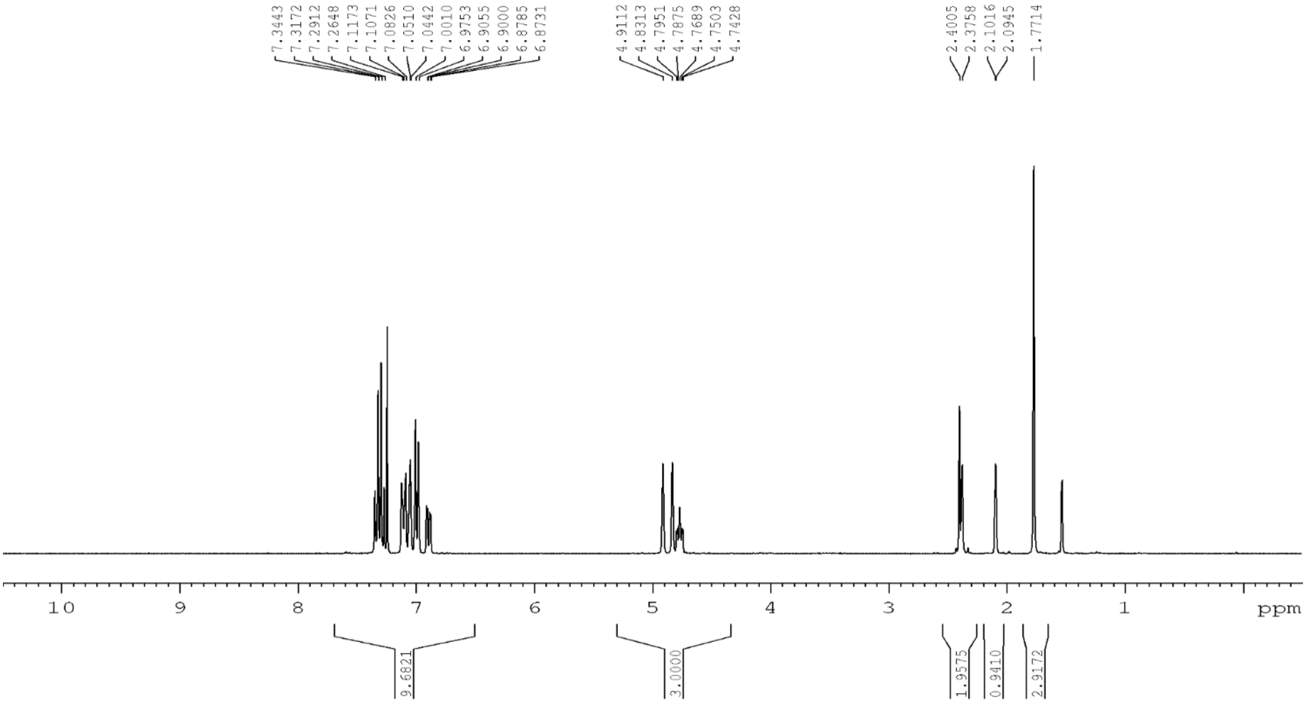




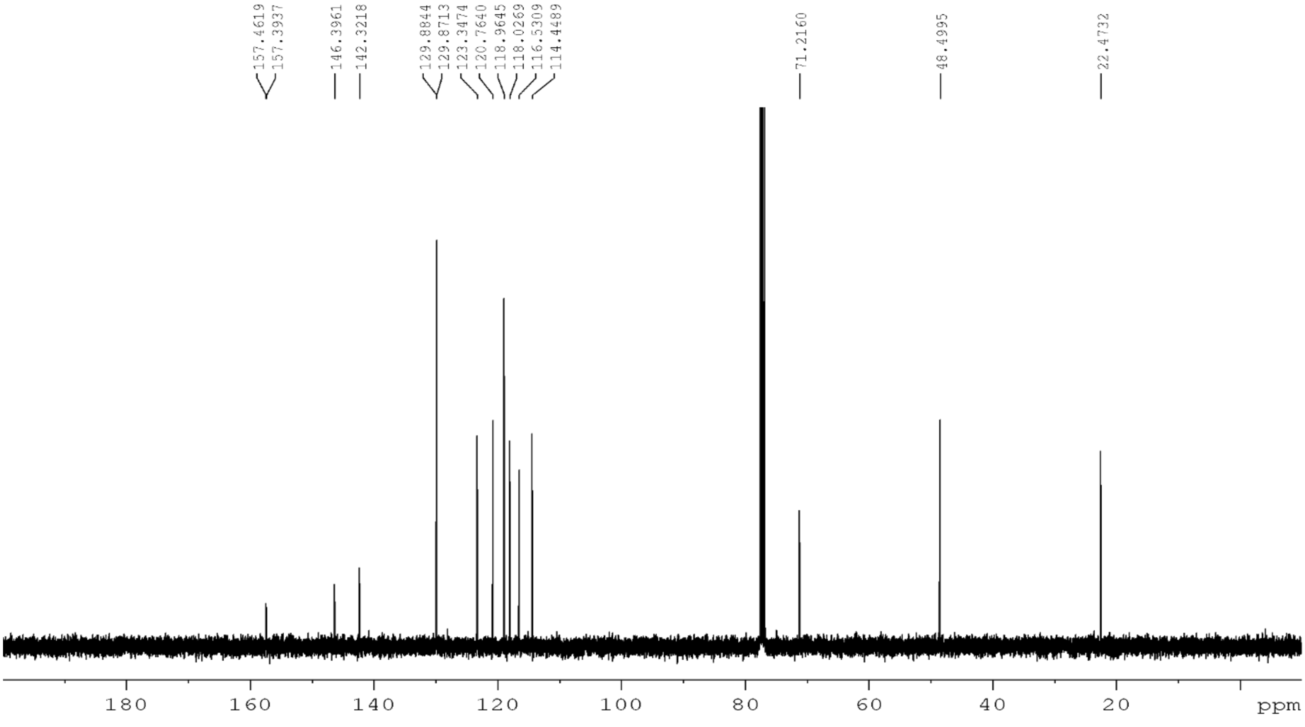
^{1}H NMR (300 MHz, $CDCl_3$)

3-methyl-1-(3-phenoxyphenyl)-3-buten-1-ol (1f)



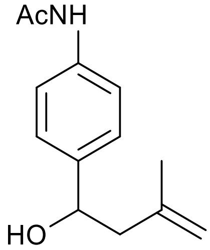


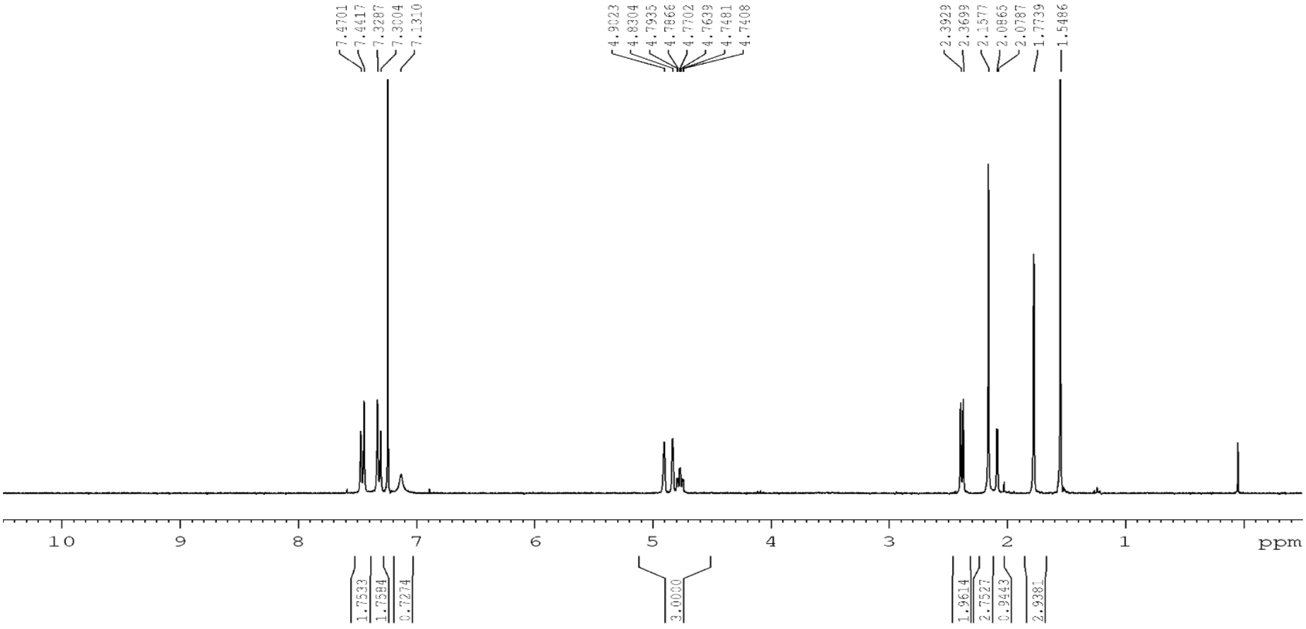
^{1}H NMR (300 MHz, CDCl$_3$)



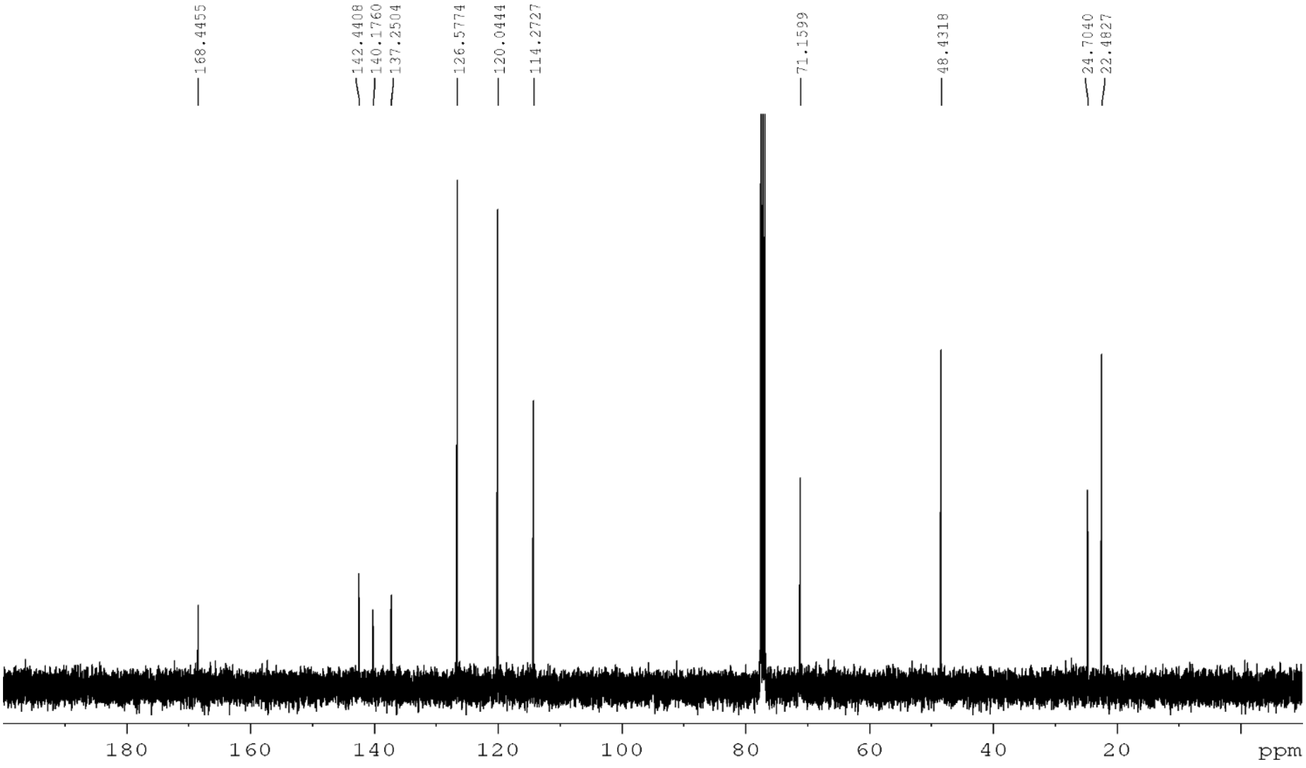
^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$)

1-(4-acetamidophenyl)-3-methyl-3-buten-1-ol (1g)



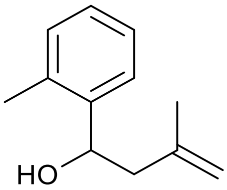


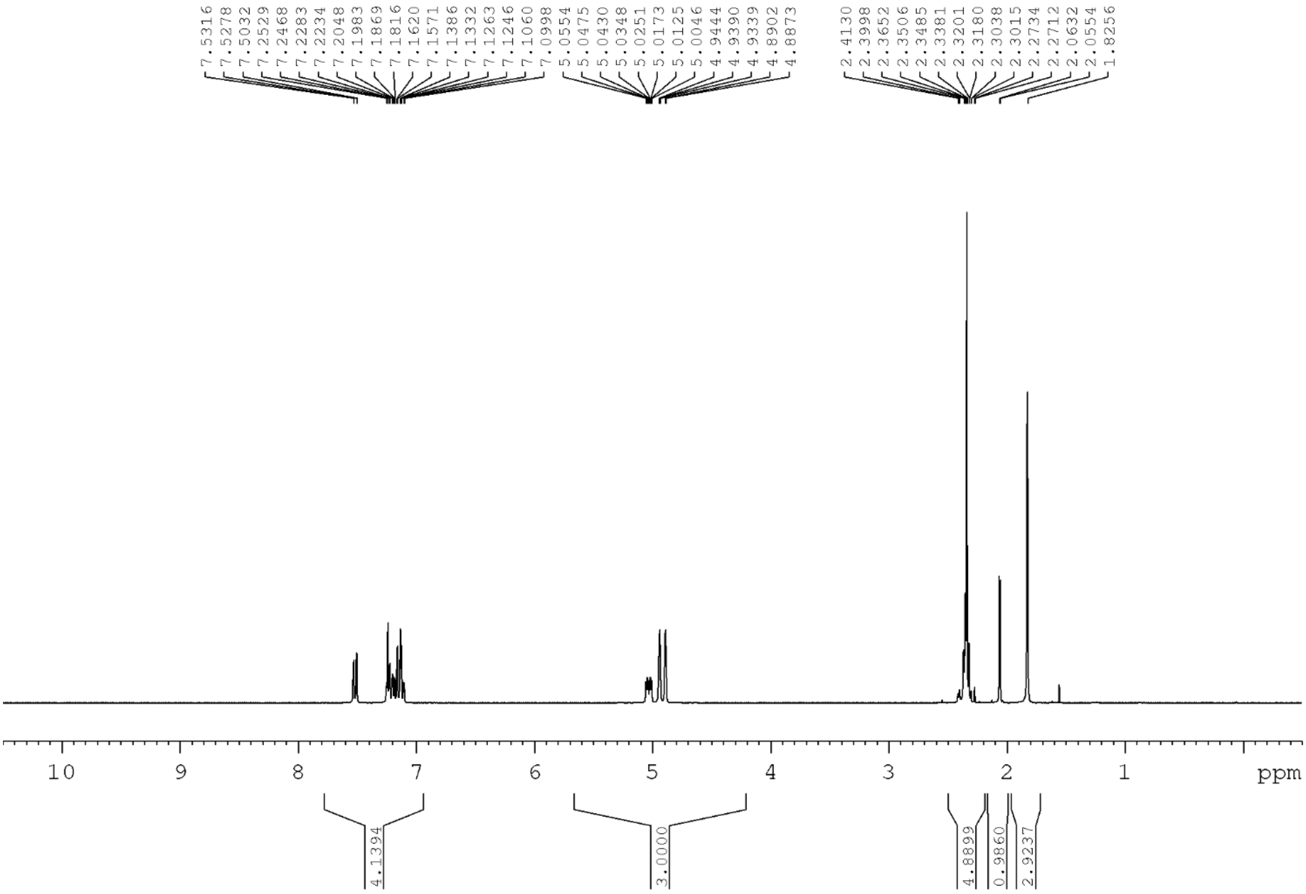
^{1}H NMR (300 MHz, CDCl$_3$)



^{13}C{^{1}H} NMR 100 MHz, CDCl$_3$)

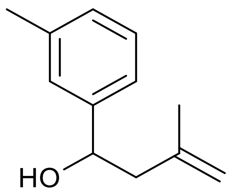
3-methyl-1-(2-methylphenyl)-3-buten-1-ol (1h)

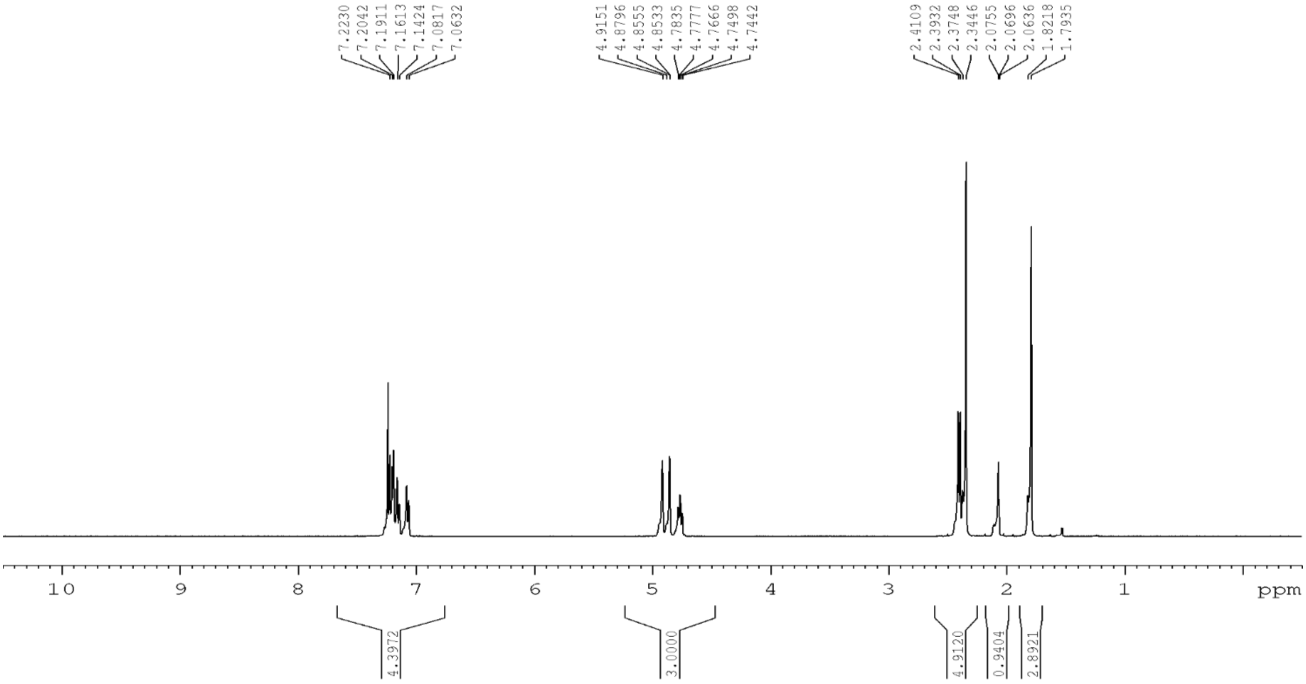




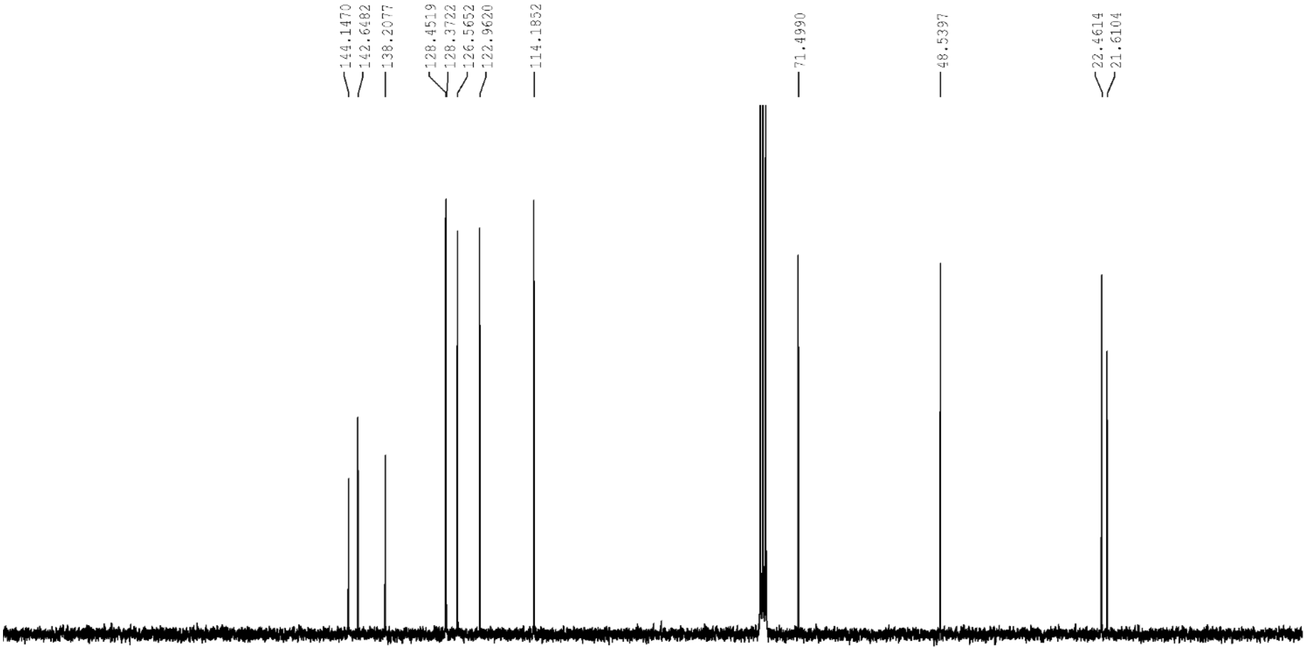
^{1}H NMR (300 MHz, $CDCl_3$)

3-methyl-1-(3-methylphenyl)-3-buten-1-ol (1i)



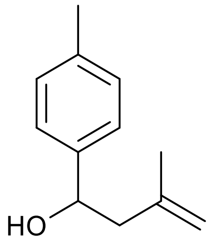


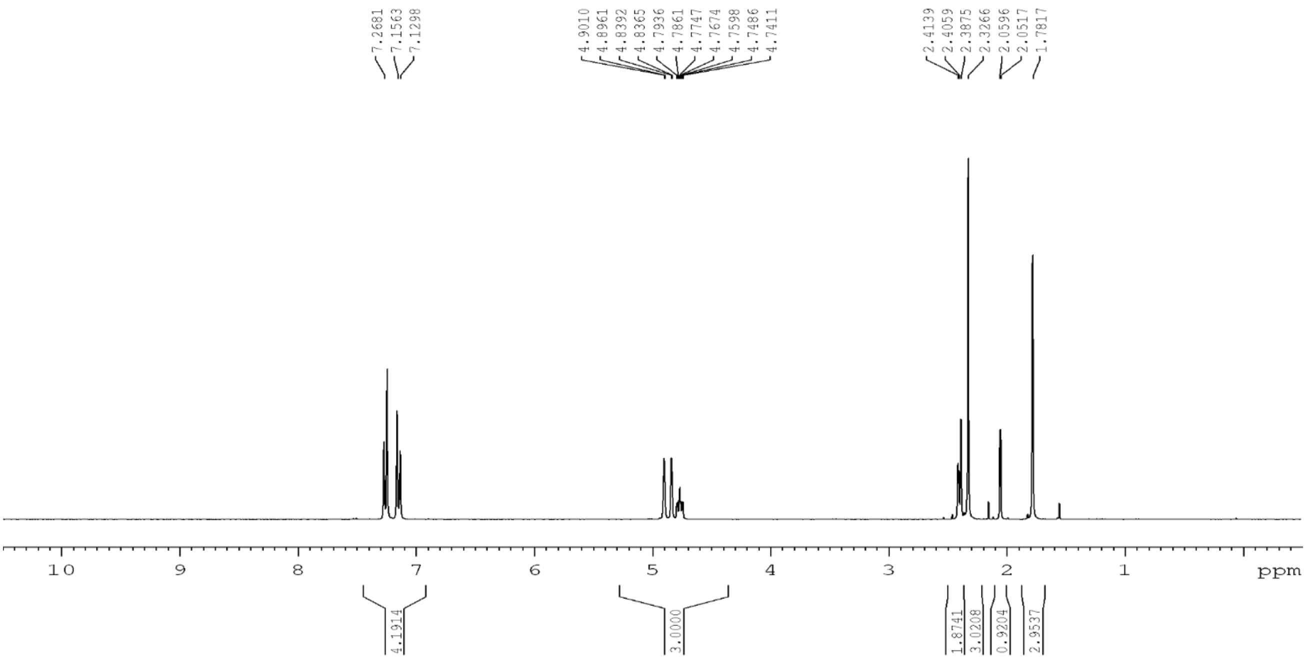
^{1}H NMR (300 MHz, CDCl$_3$)



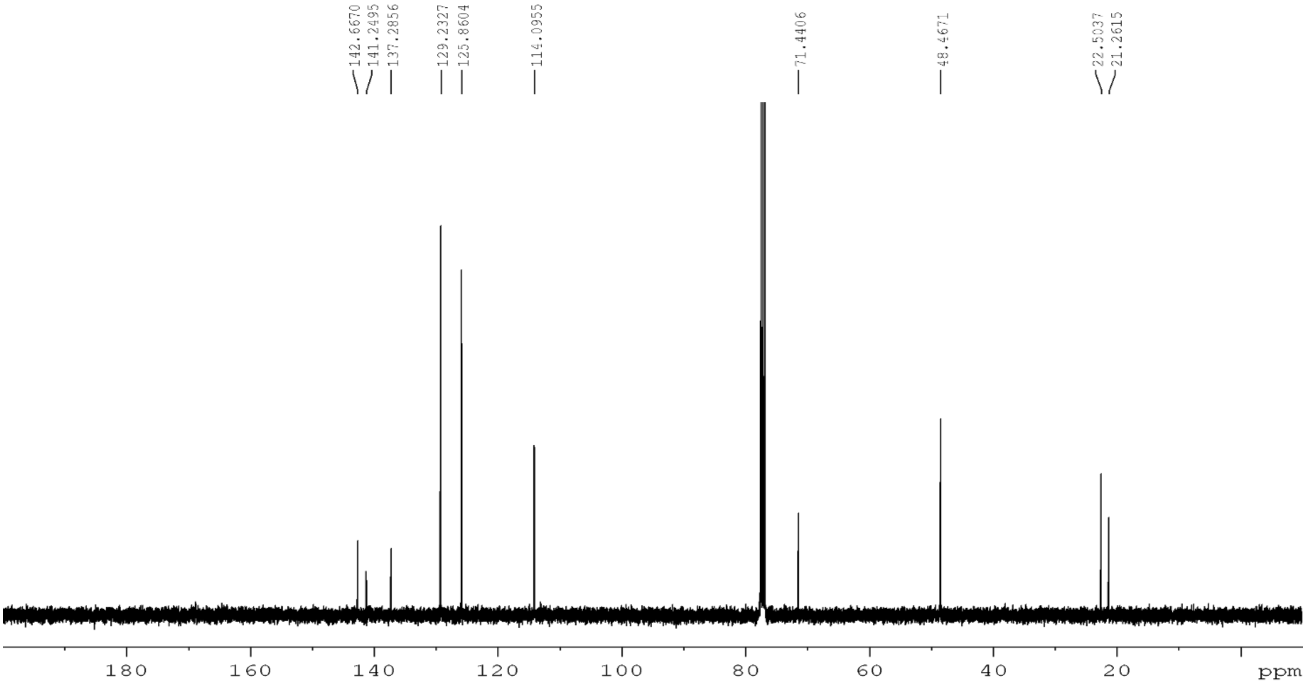
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

3-methyl-1-(4-methylphenyl)-3-buten-1-ol (1j)



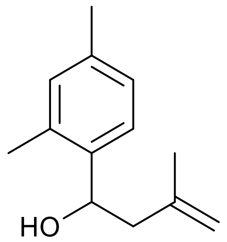


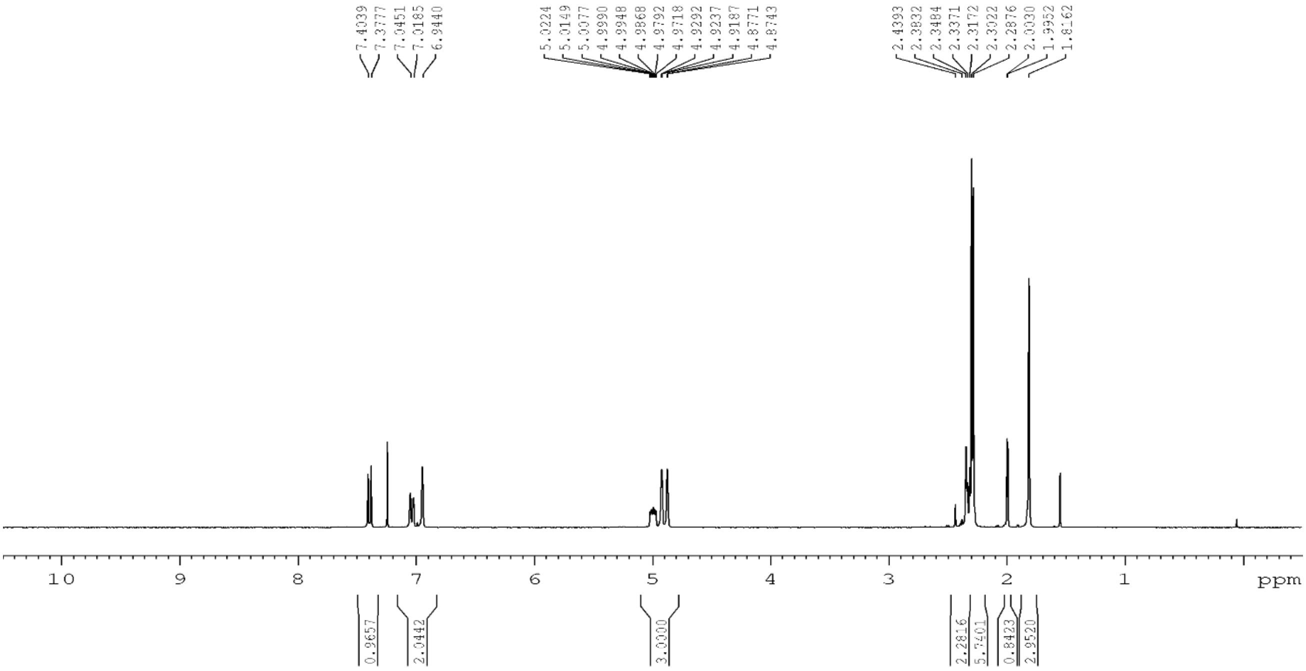
^{1}H NMR (300 MHz, CDCl$_3$)



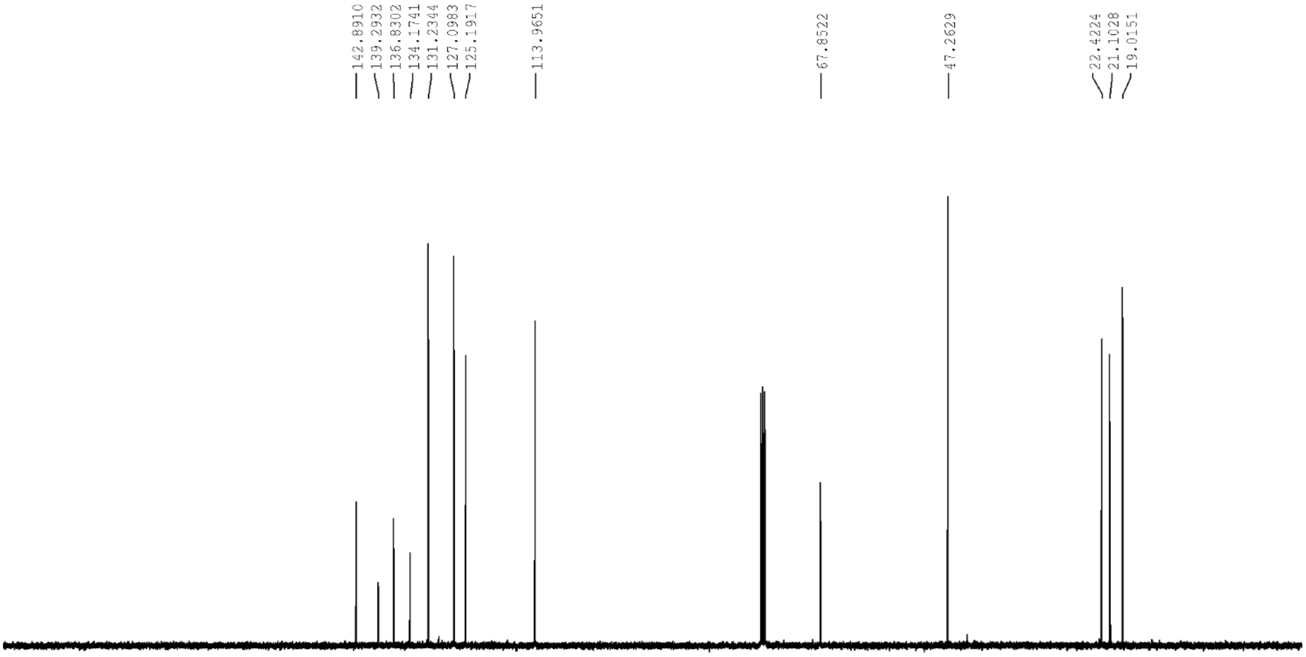
^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$)

3-methyl-1-(2,4-dimethylphenyl)-3-buten-1-ol (1k)



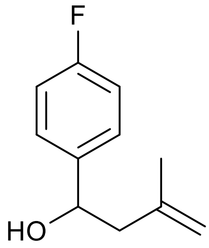


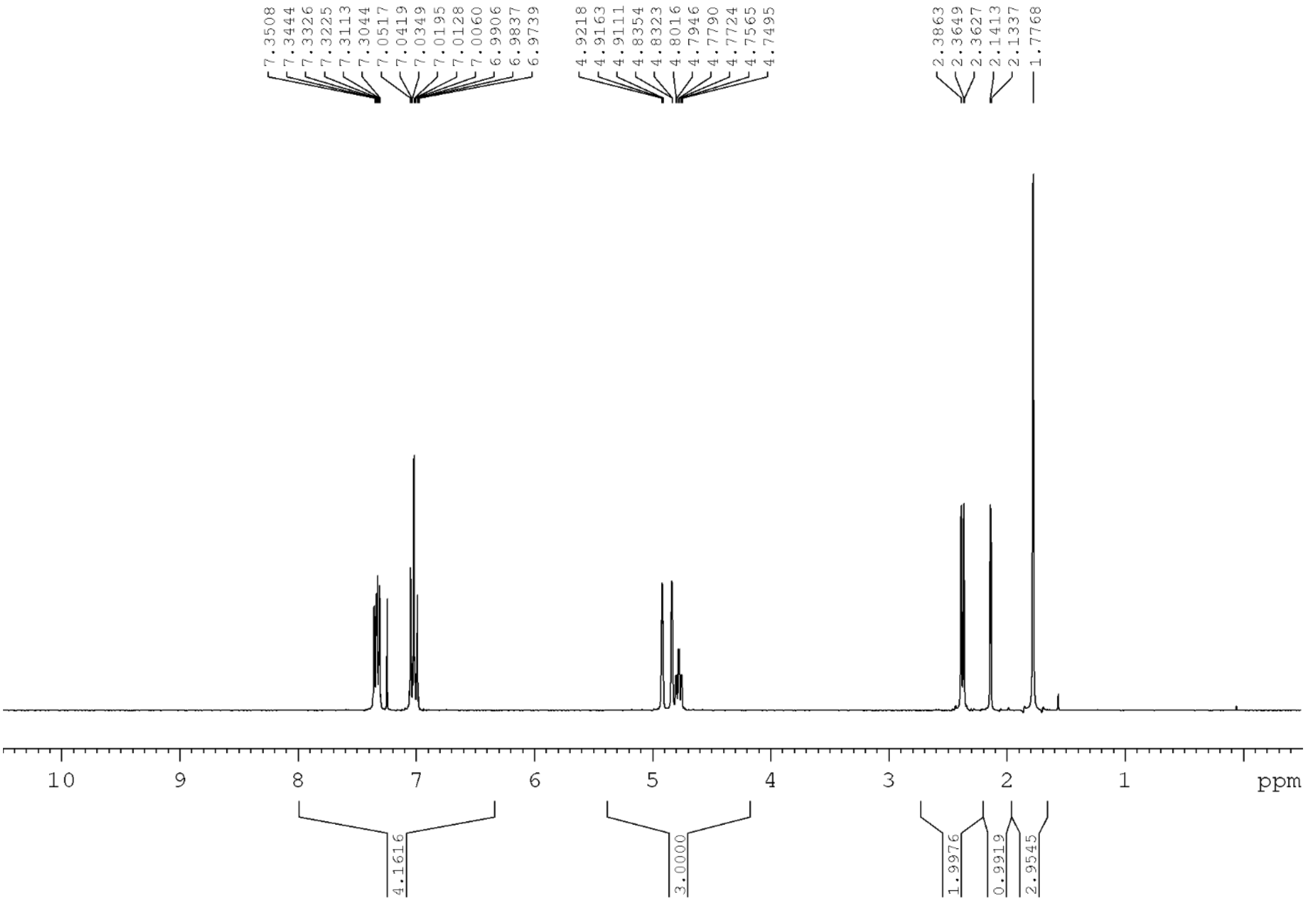
^{1}H NMR (300 MHz, CDCl$_3$)



^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$)

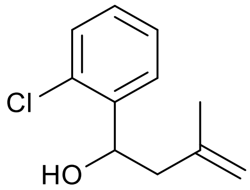
1-(4-fluorophenyl)-3-methyl-3-buten-1-ol (1l)

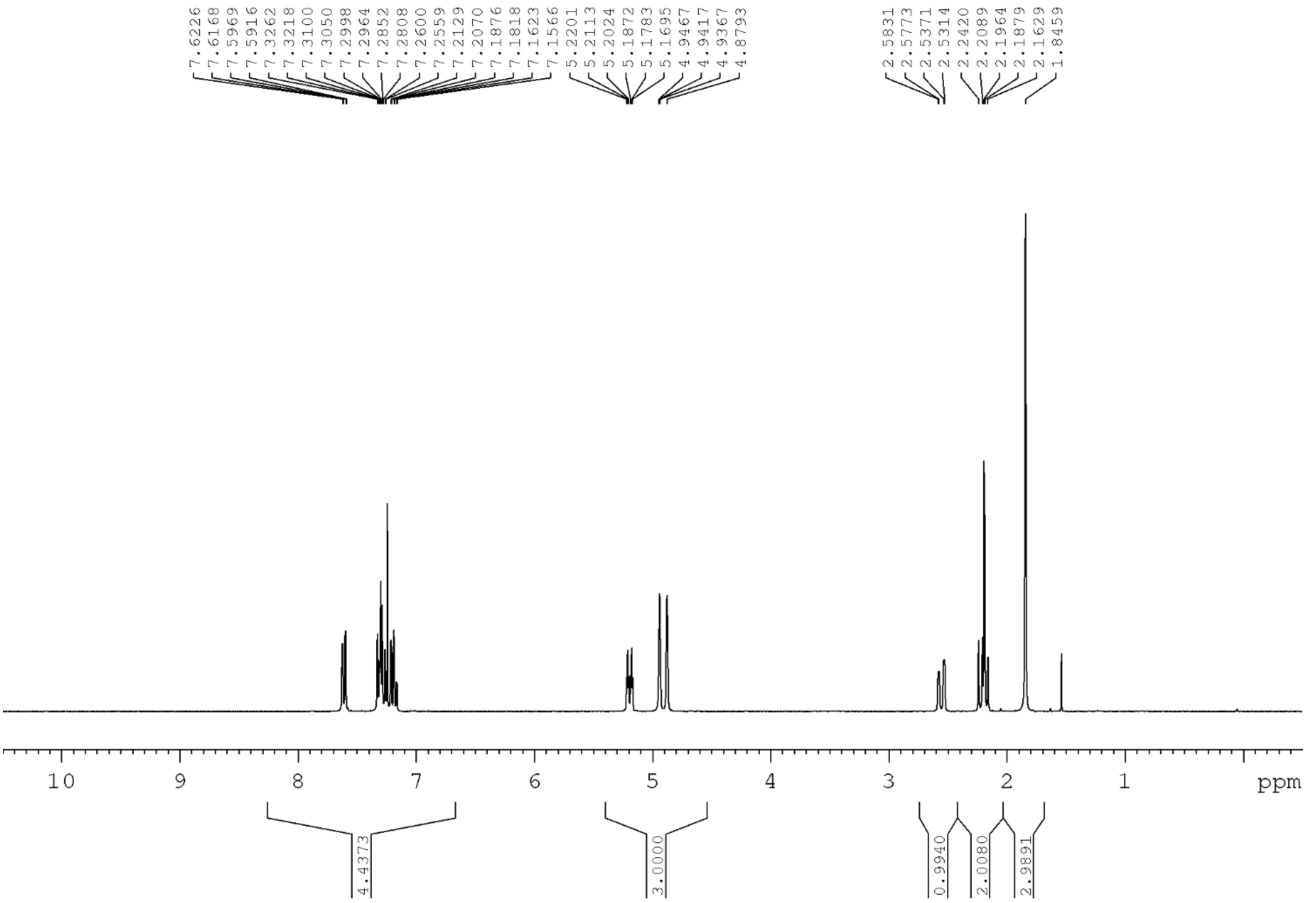




^{1}H NMR (300 MHz, CDCl$_3$)

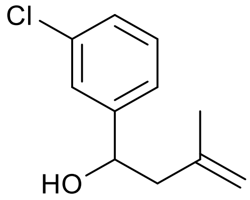
1-(2-chlorophenyl)-3-methyl-3-buten-1-ol (1m)

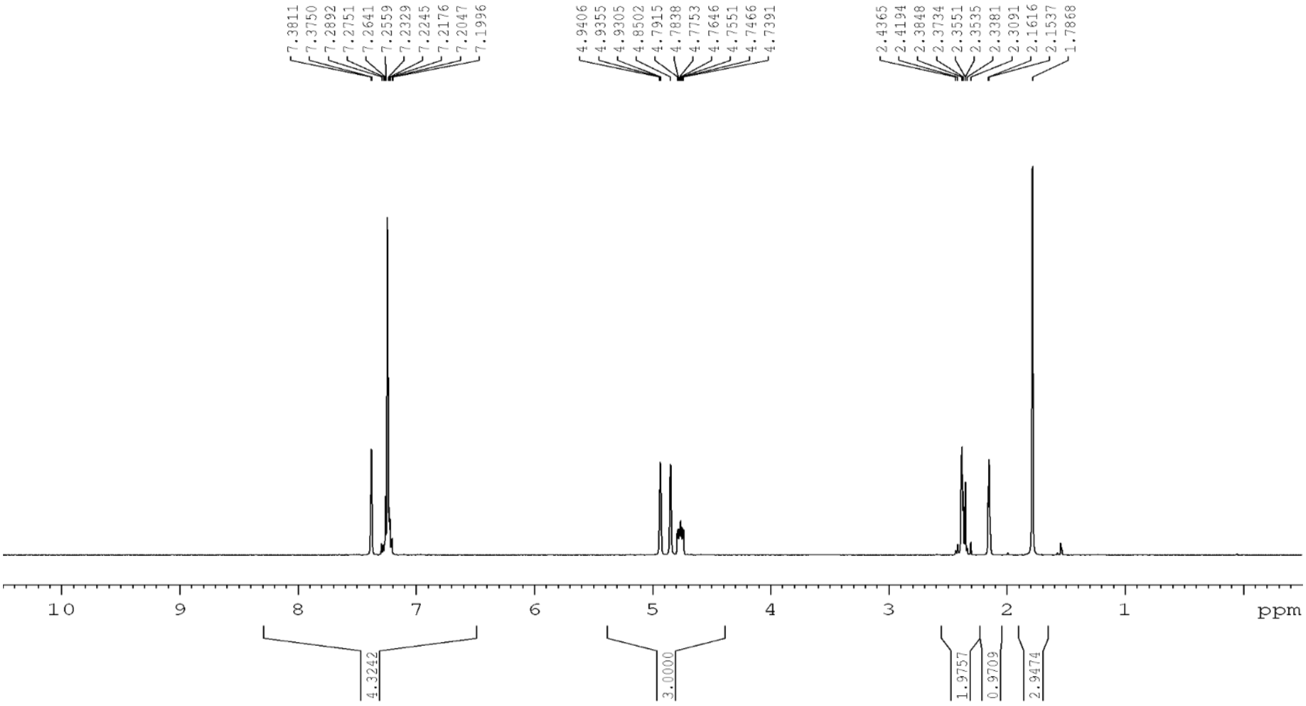




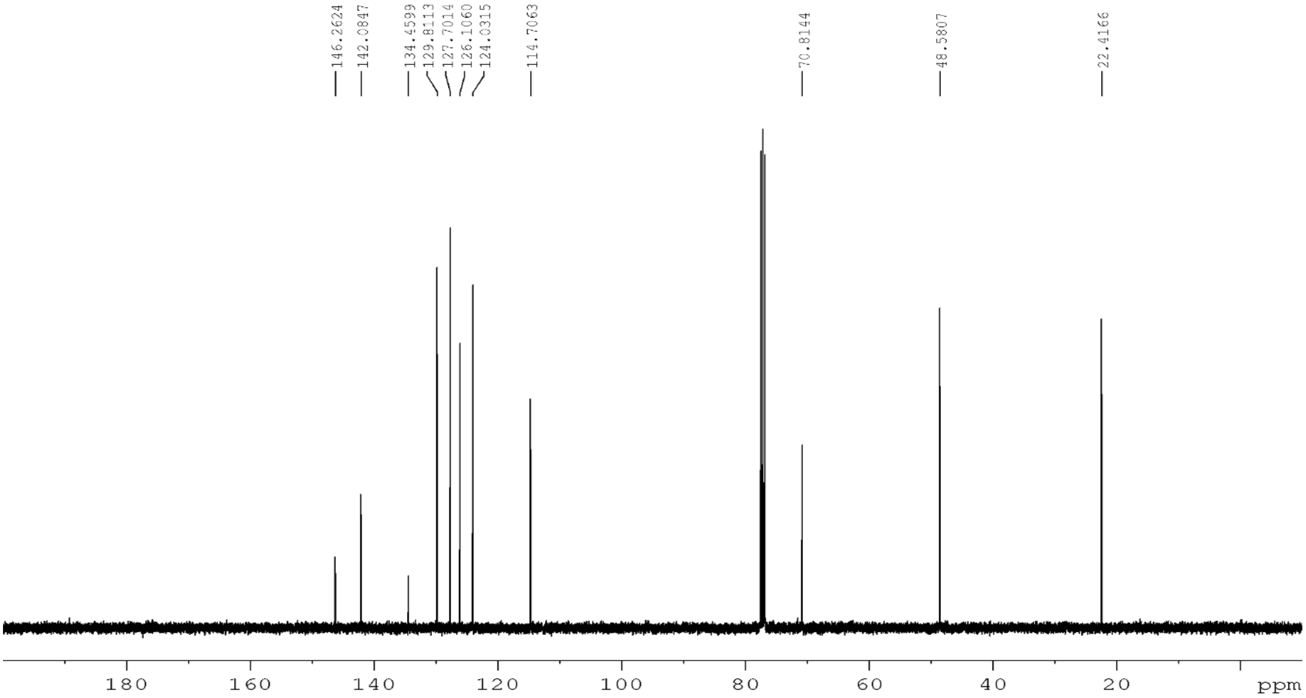
^{1}H NMR (300 MHz, CDCl$_3$)

1-(3-chlorophenyl)-3-methyl-3-buten-1-ol (1n)



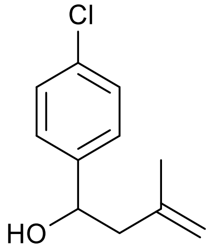


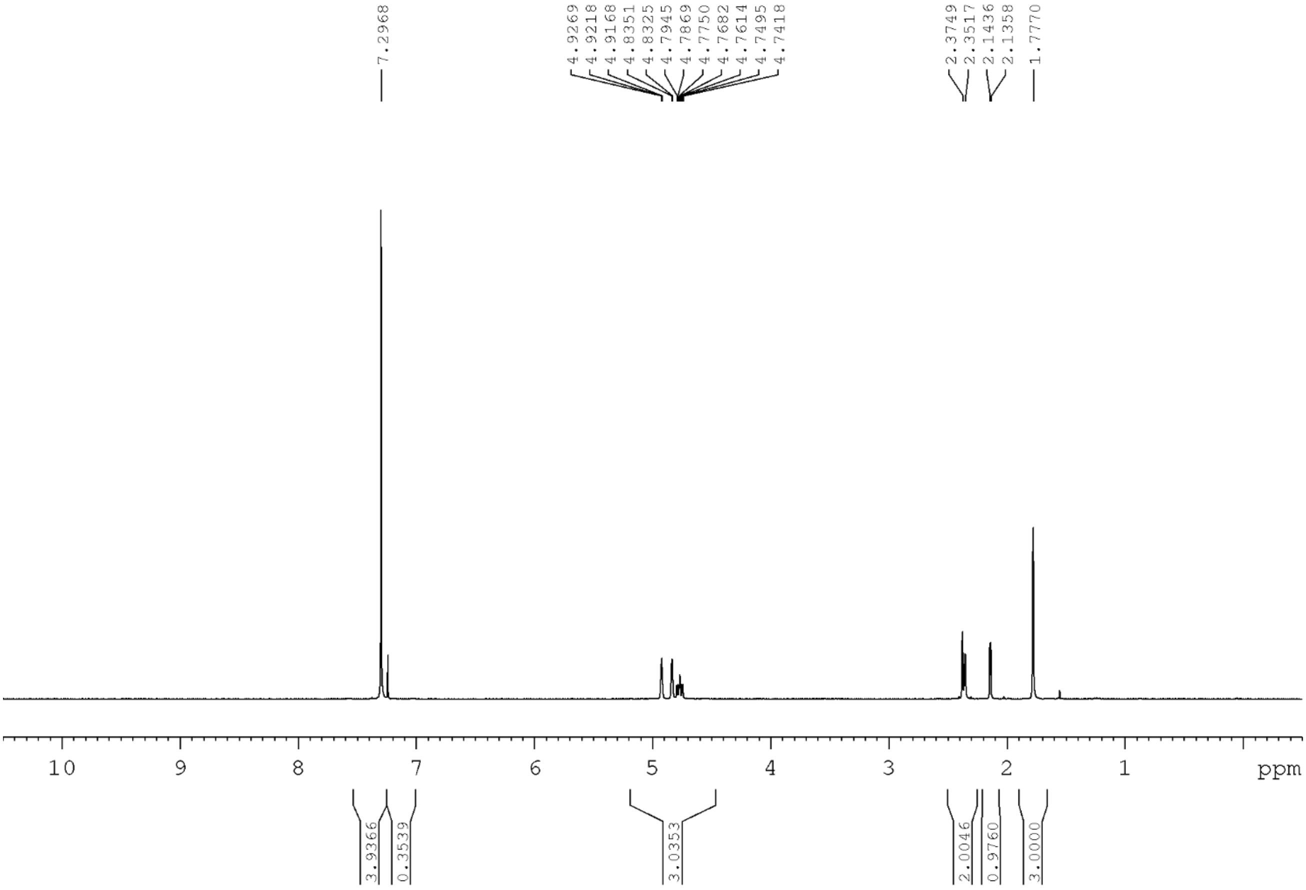
^{1}H NMR (300 MHz, CDCl$_3$)



^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$)

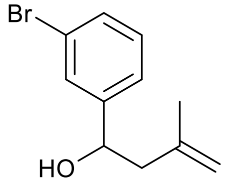
1-(4-chlorophenyl)-3-methyl-3-buten-1-ol (1o)

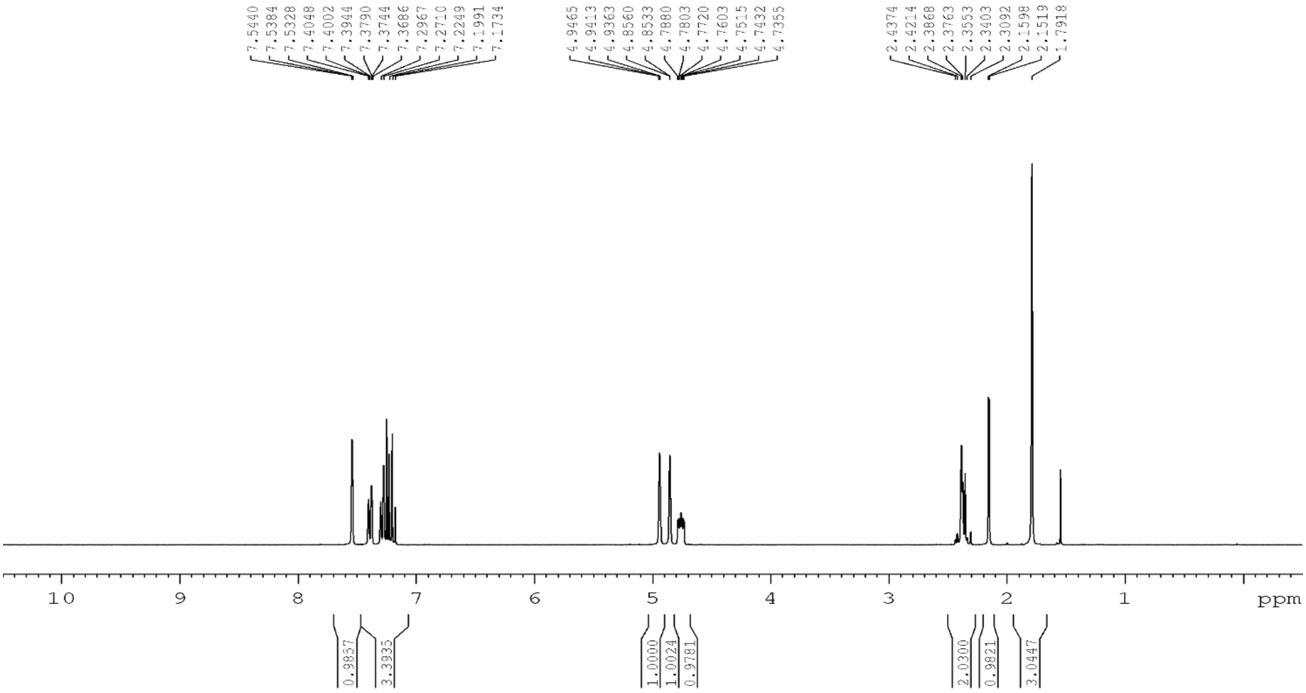




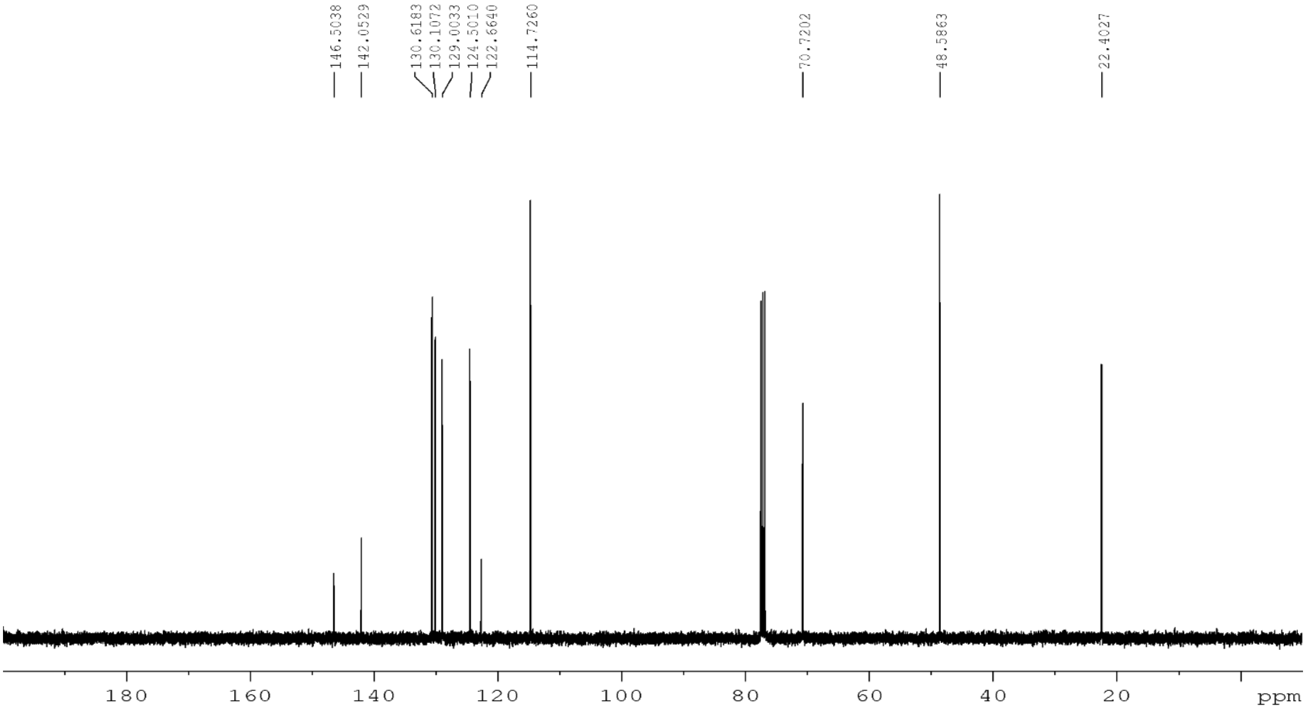
^{1}H NMR (300 MHz, CDCl$_3$)

1-(3-bromophenyl)-3-methyl-3-buten-1-ol (1p)



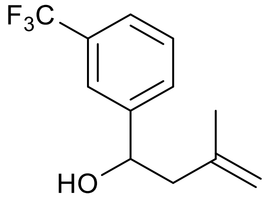


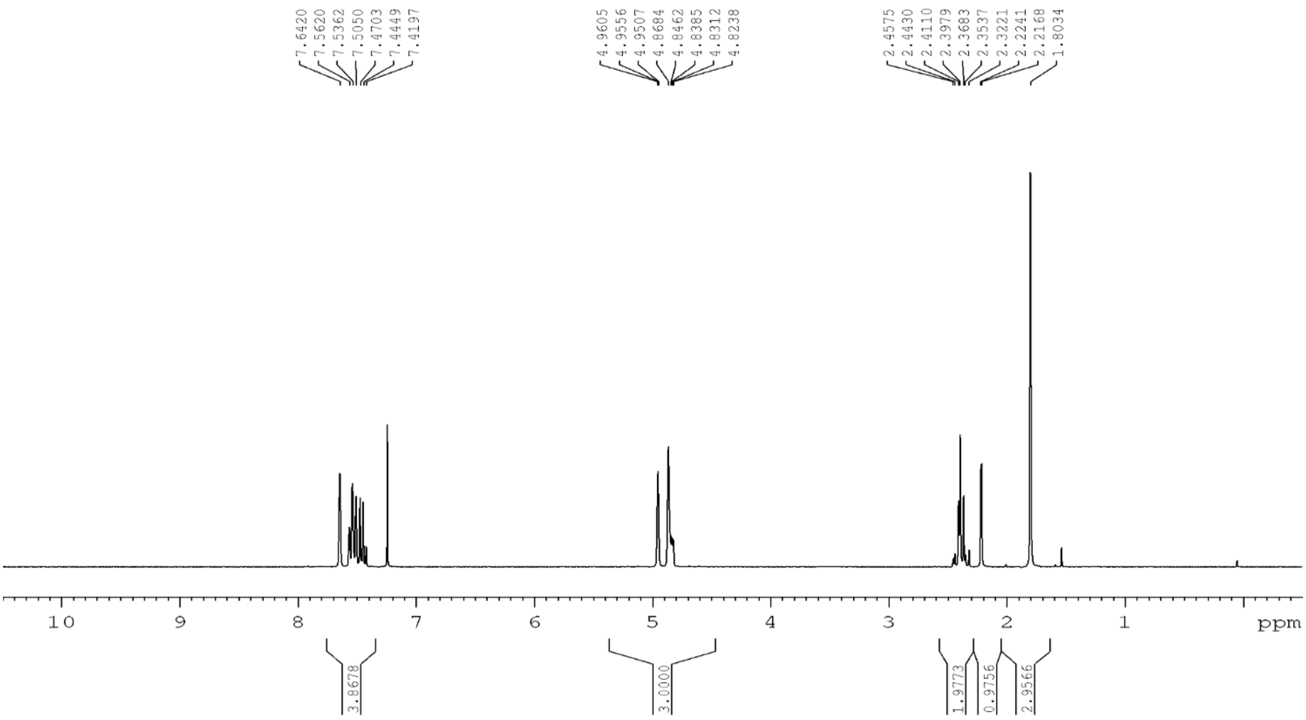
^{1}H NMR (300 MHz, CDCl$_3$)



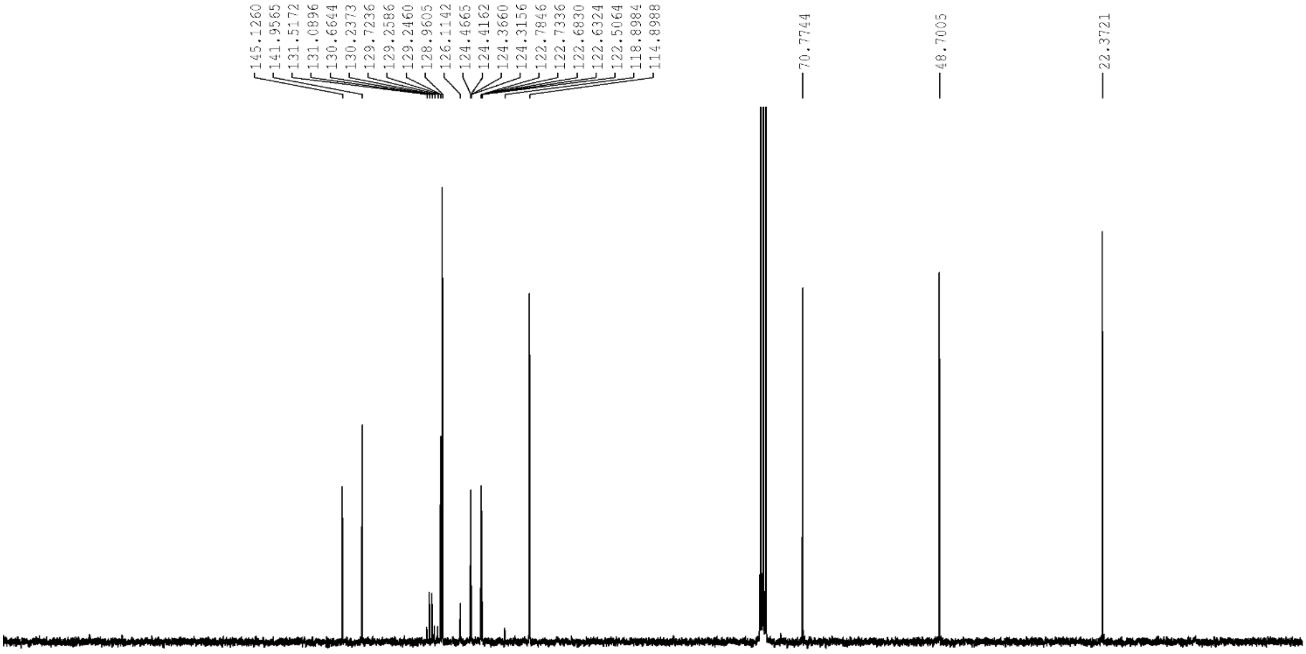
^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$)

3-methyl-1-(3-trifluoromethylphenyl)-3-buten-1-ol (1q)



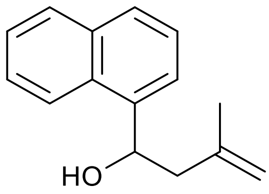


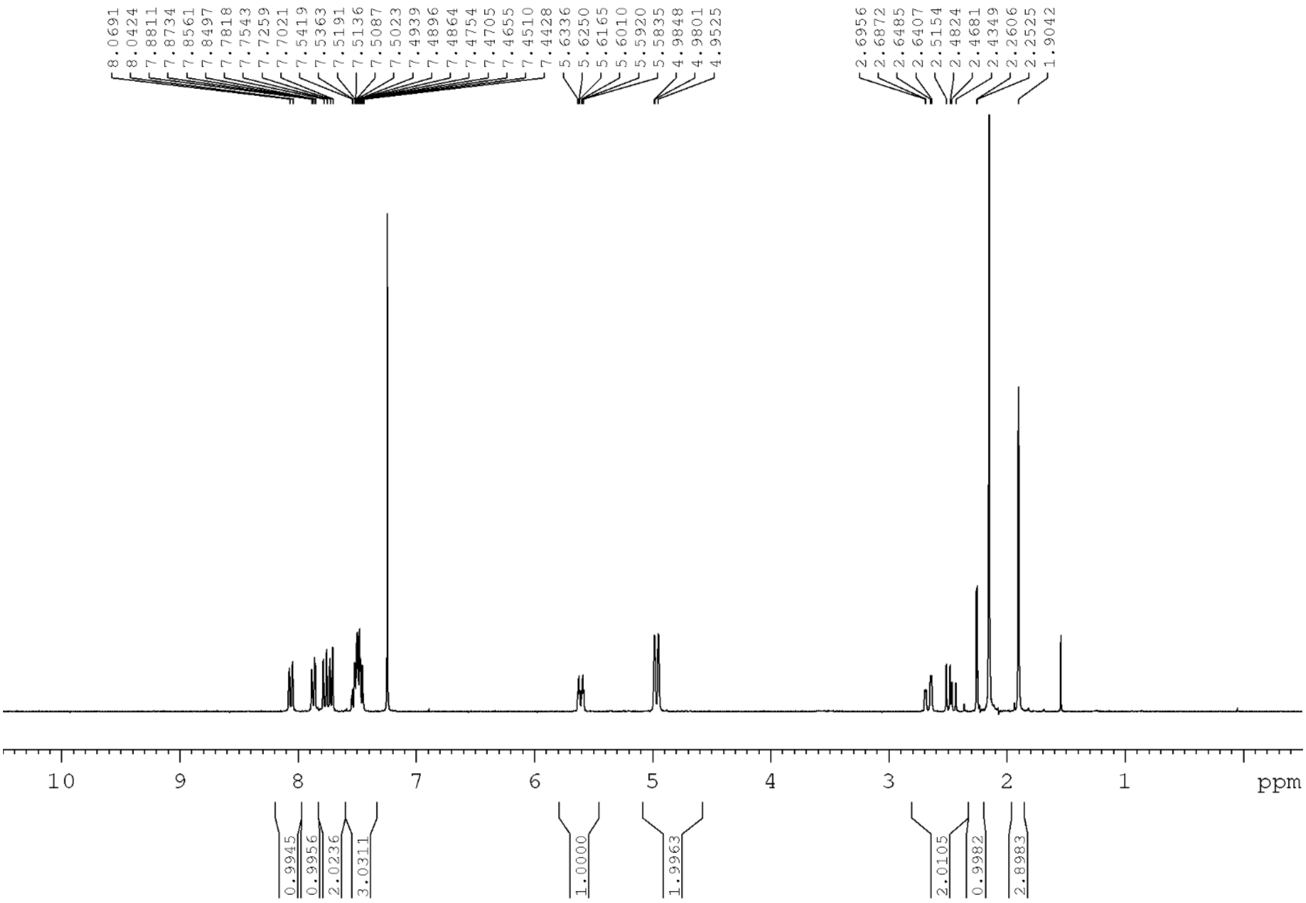
^{1}H NMR (300 MHz, CDCl$_3$)



^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

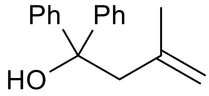
3-methyl-1-naphthyl-3-buten-1-ol (1r)

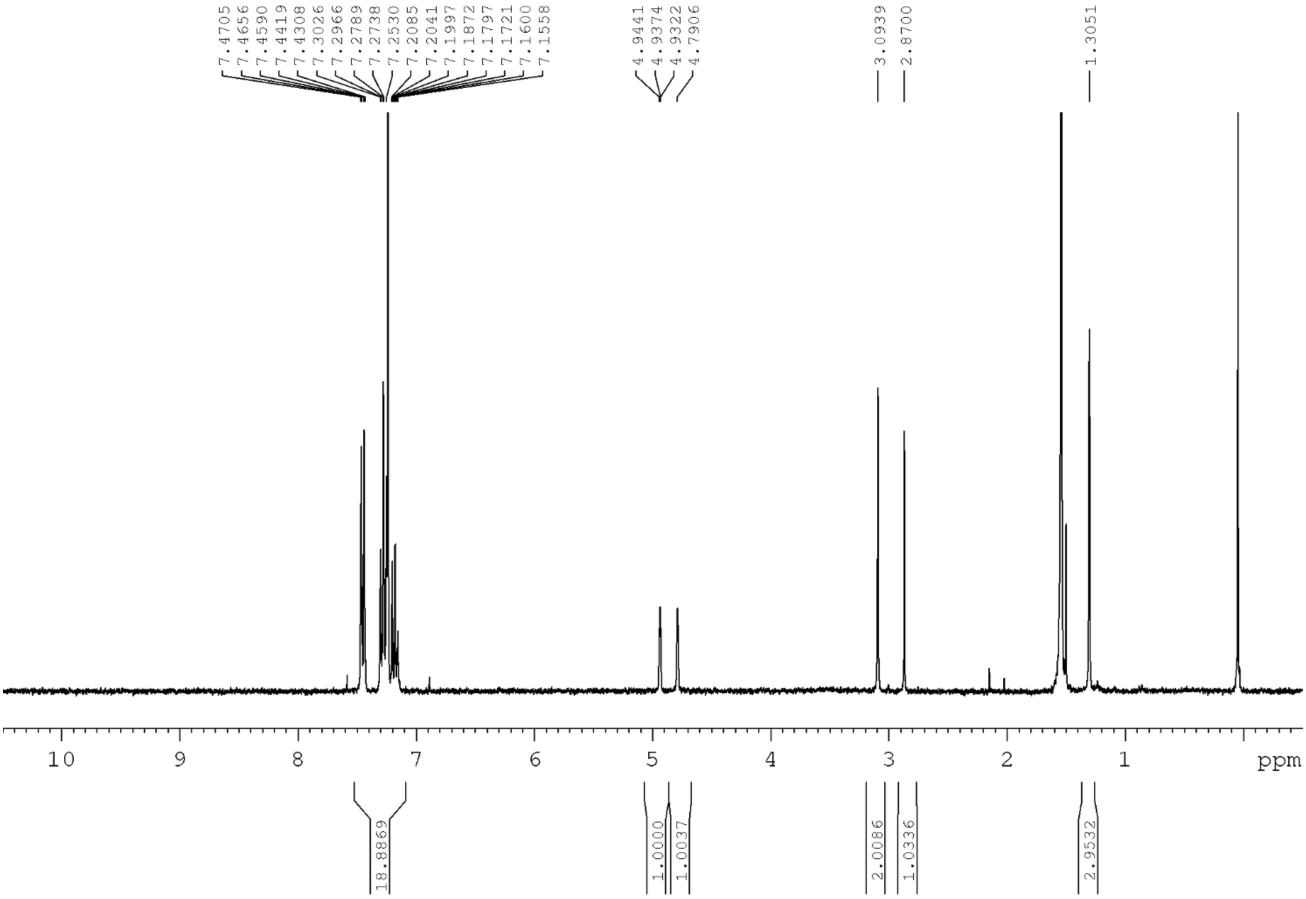




^{1}H NMR (300 MHz, $CDCl_3$)

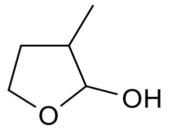
1,1-diphenyl-3-methyl-3-buten-1-ol (1s)

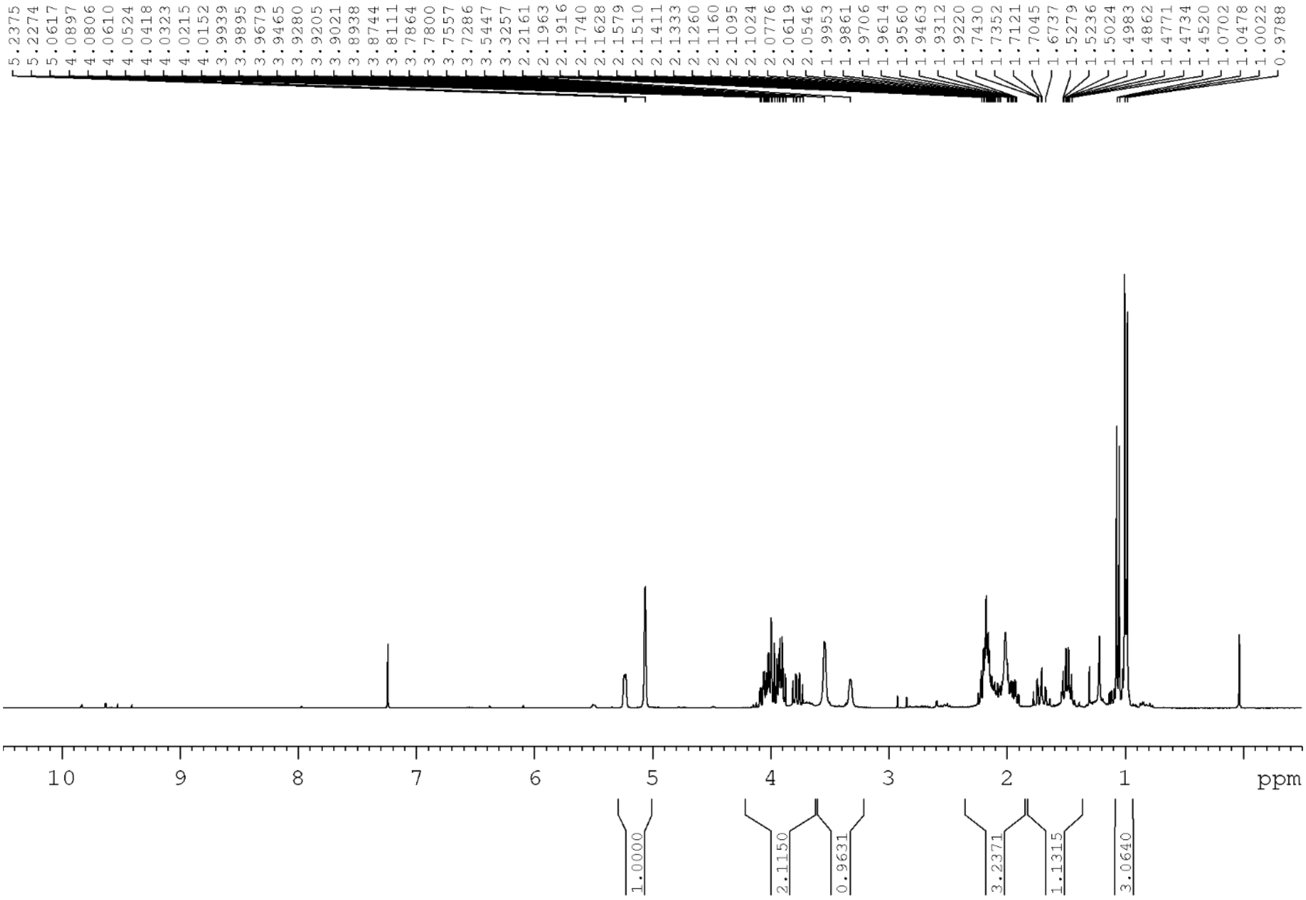




^{1}H NMR (300 MHz, $CDCl_3$)

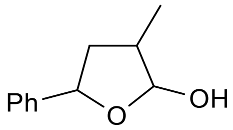
2-hydroxy-3-methyltetrahydrofuran (2a)

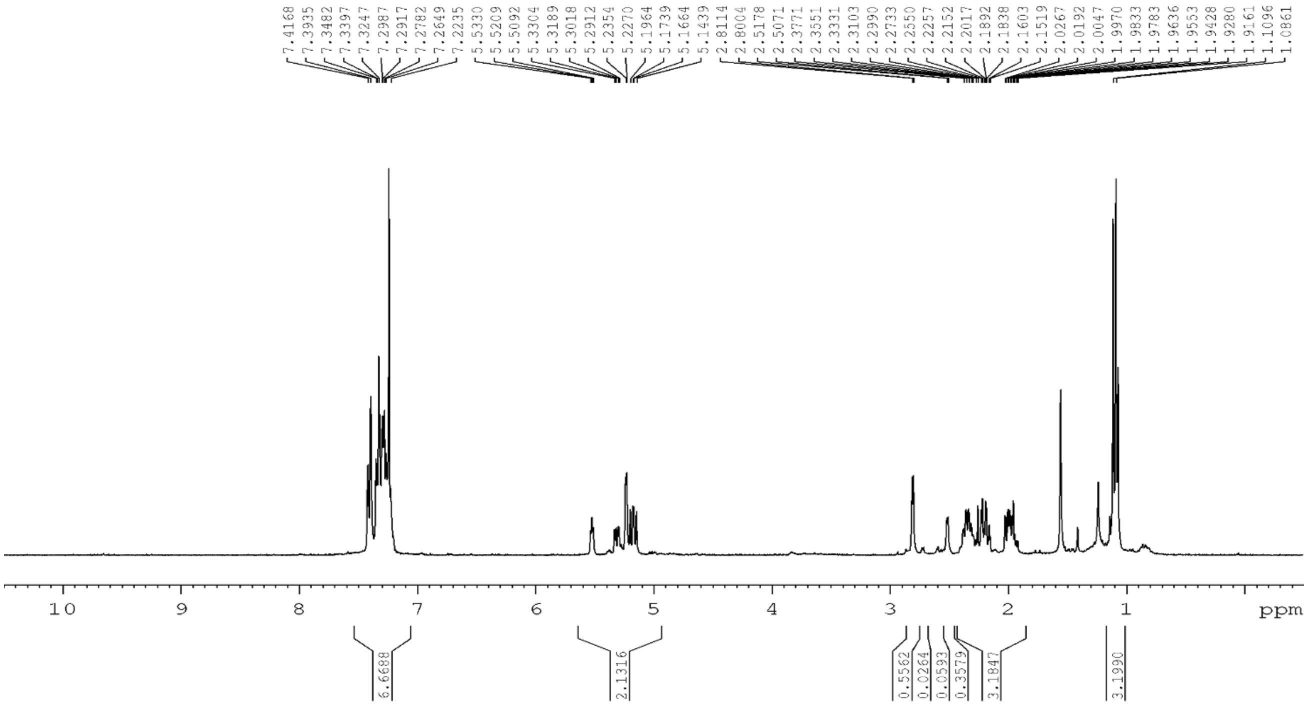




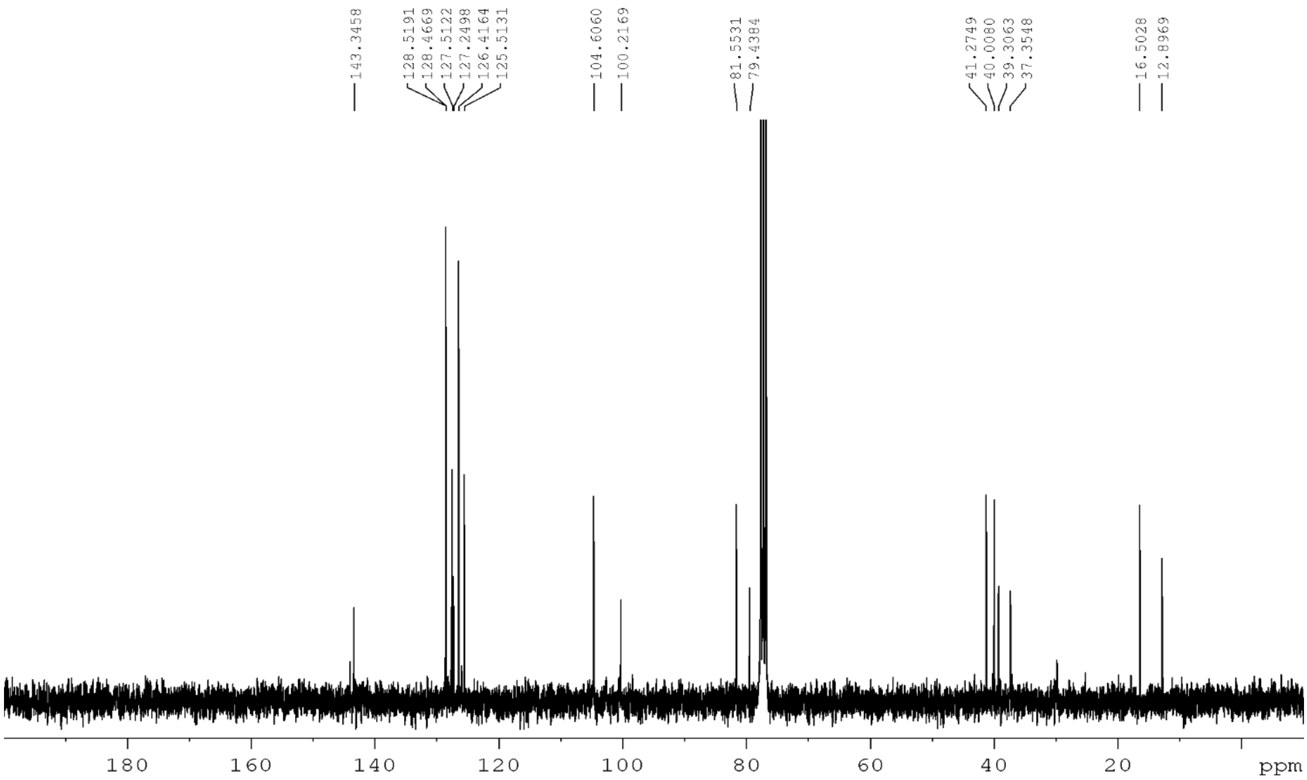
^{1}H NMR (300 MHz, $CDCl_3$)

2-hydroxy-3-methyl-5-phenyltetrahydrofuran (2b)



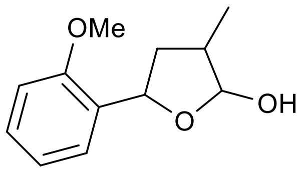


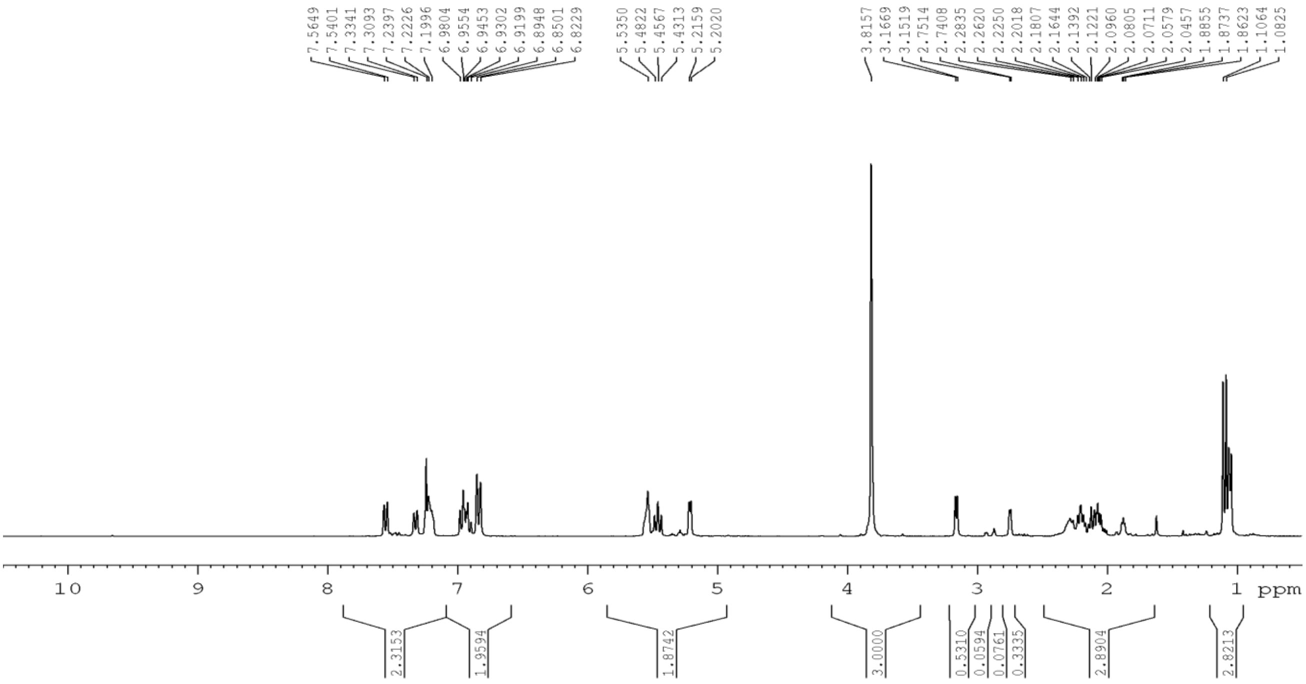
^{1}H NMR (300 MHz, CDCl$_3$)



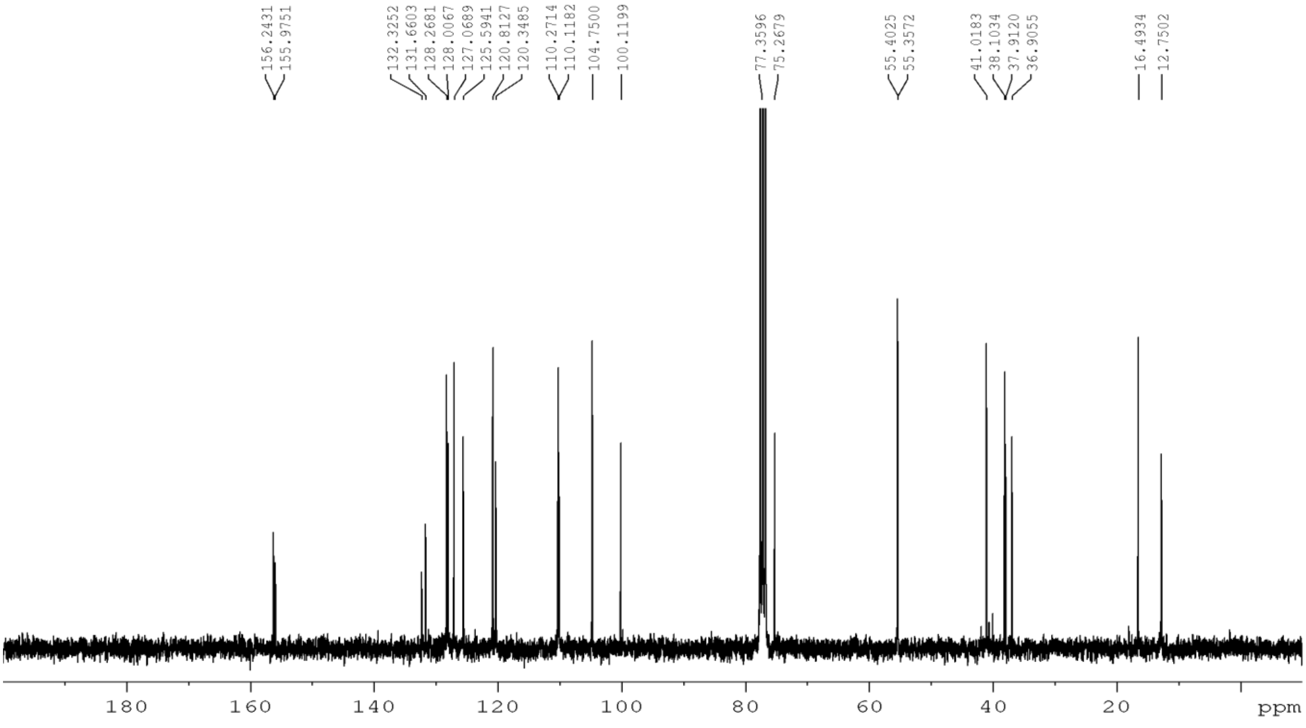
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-5-(2-methoxyphenyl)-3-methyltetrahydrofuran (2c)



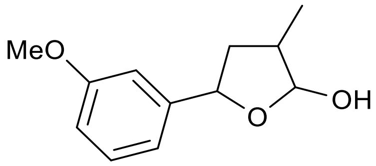


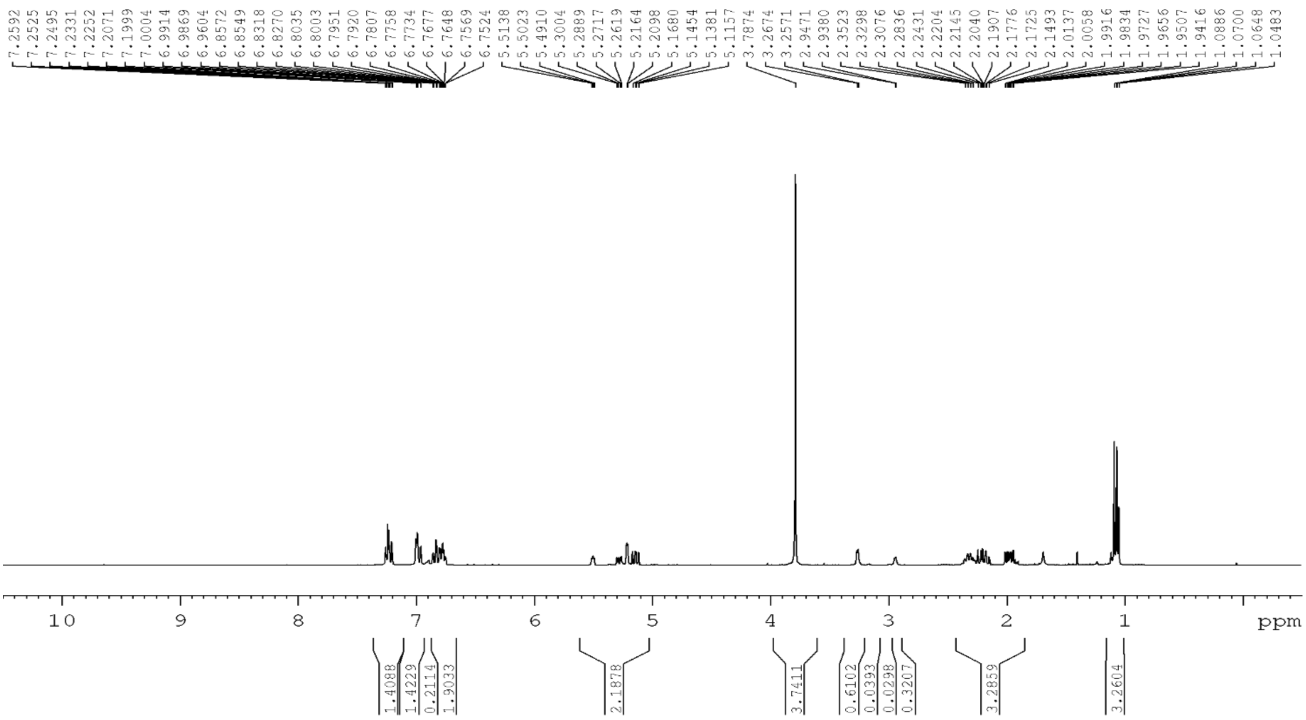
^{1}H NMR (300 MHz, CDCl$_3$)



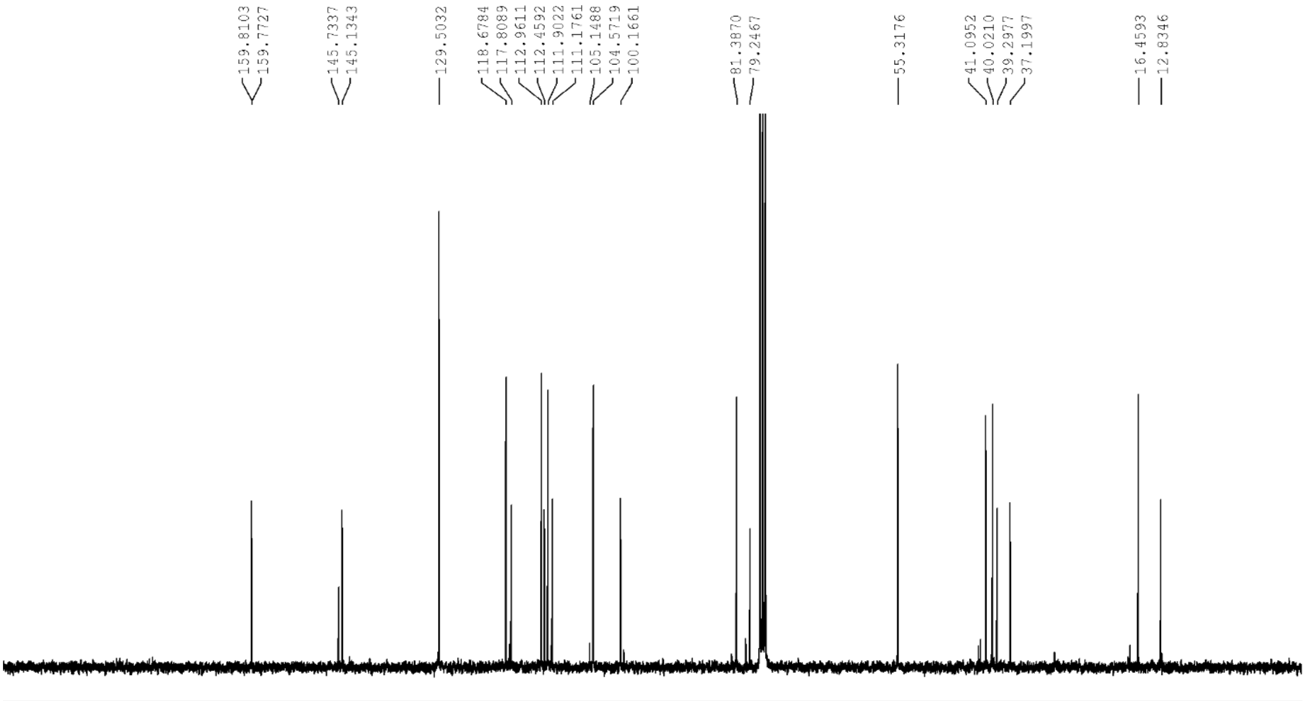
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-5-(3-methoxyphenyl)-3-methyltetrahydrofuran (2d)



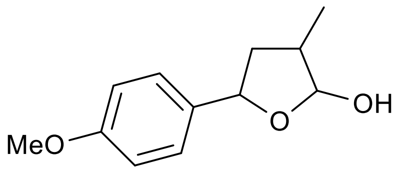


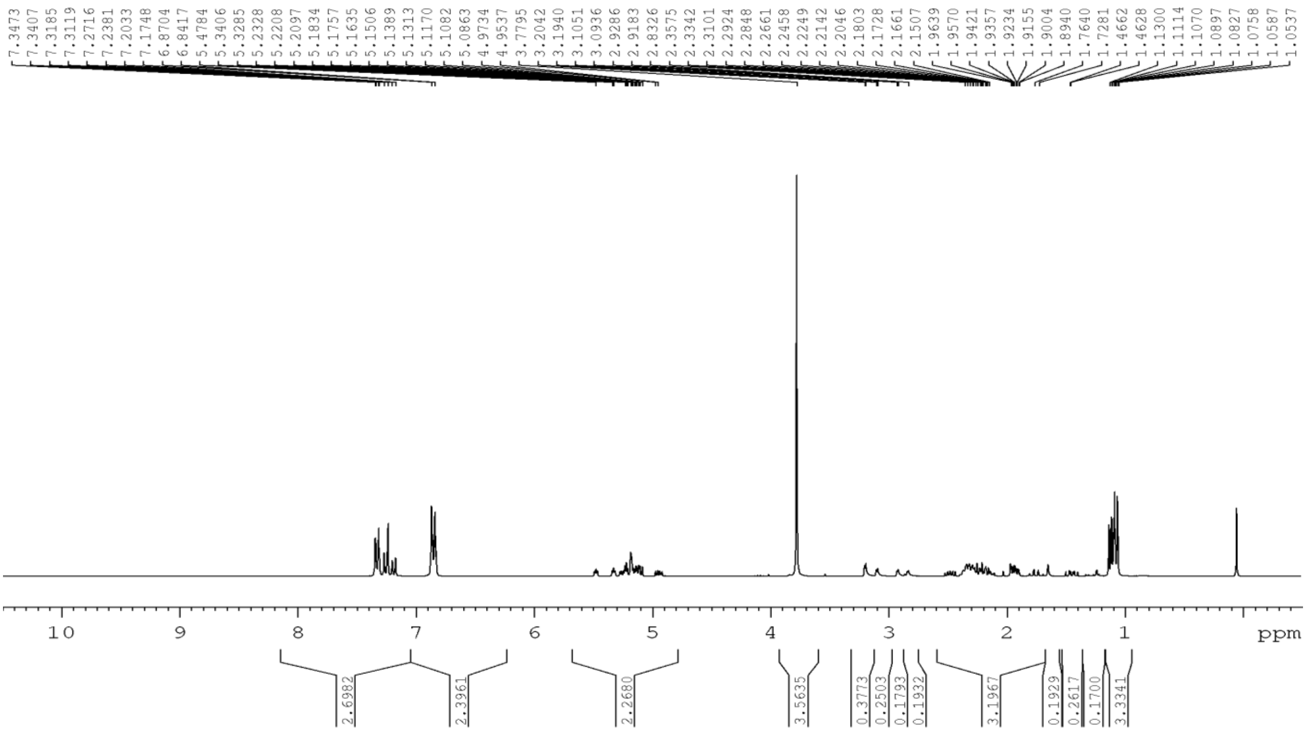
^{1}H NMR (300 MHz, CDCl$_3$)



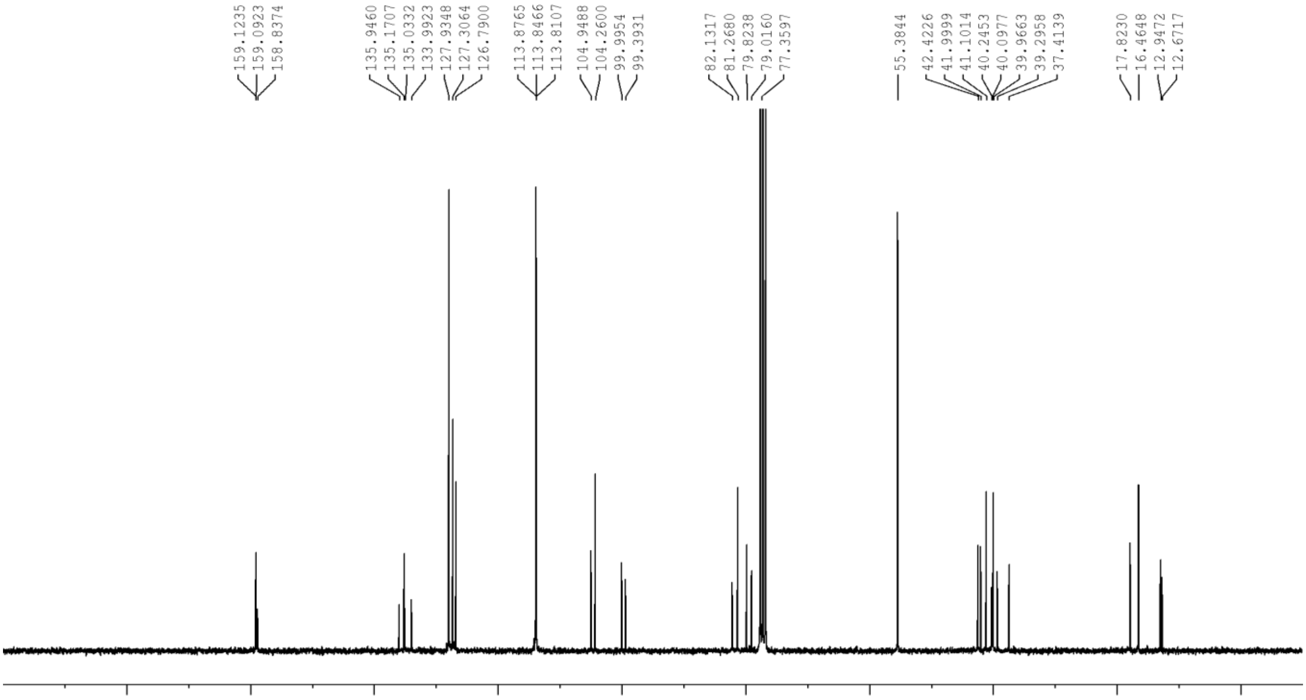
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-5-(4-methoxyphenyl)-3-methyltetrahydrofuran (2e)



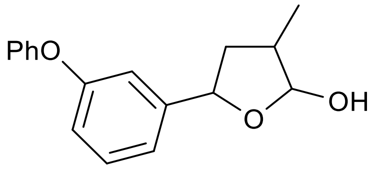


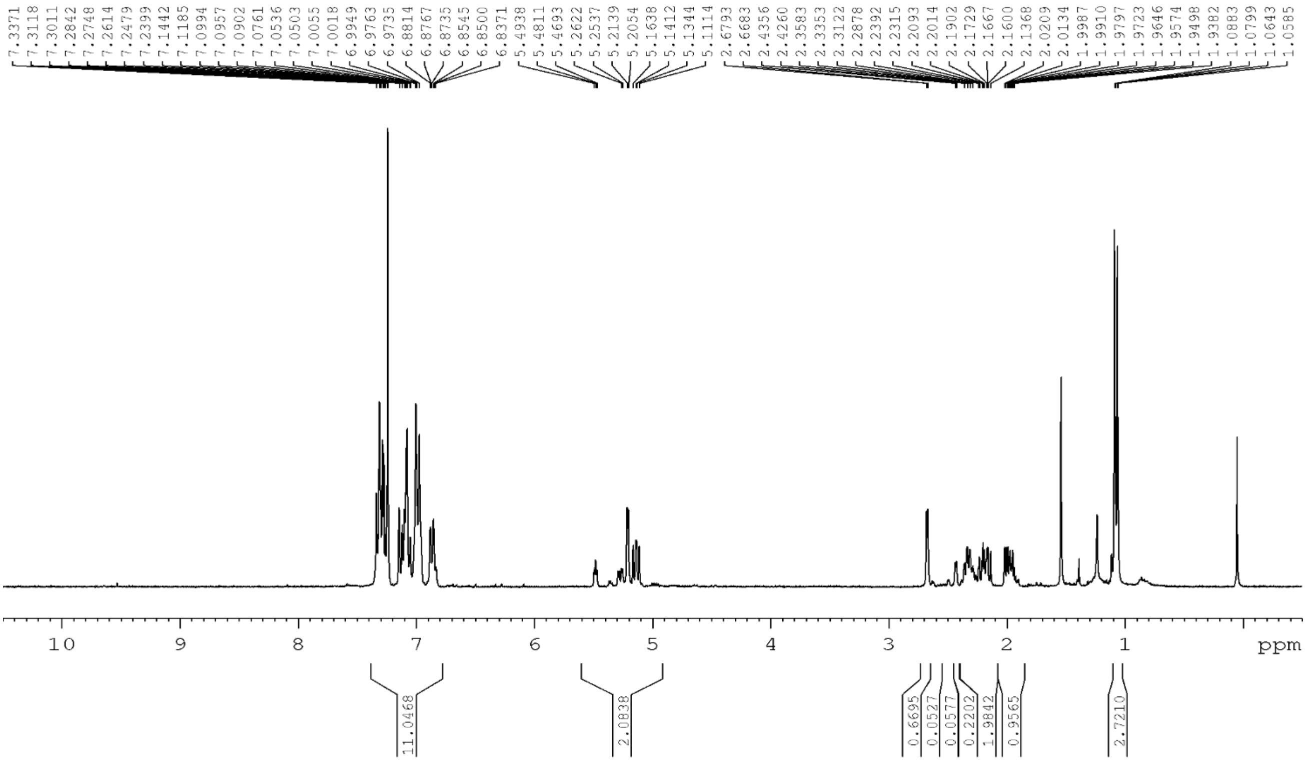
^{1}H NMR (300 MHz, CDCl$_3$)



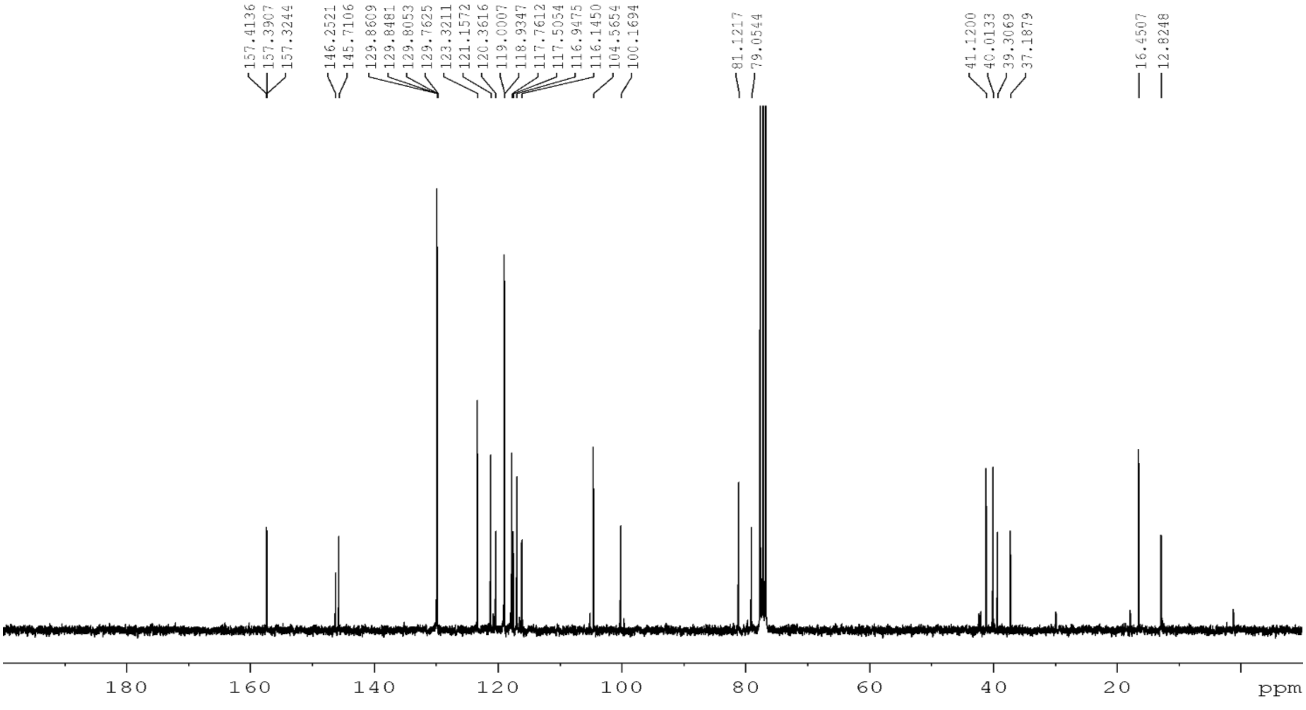
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-3-methyl-5-(3-phenoxyphenyl)tetrahydrofuran (2f)



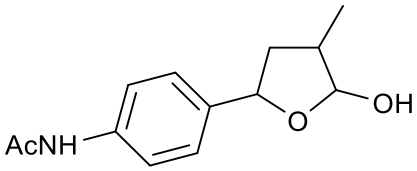


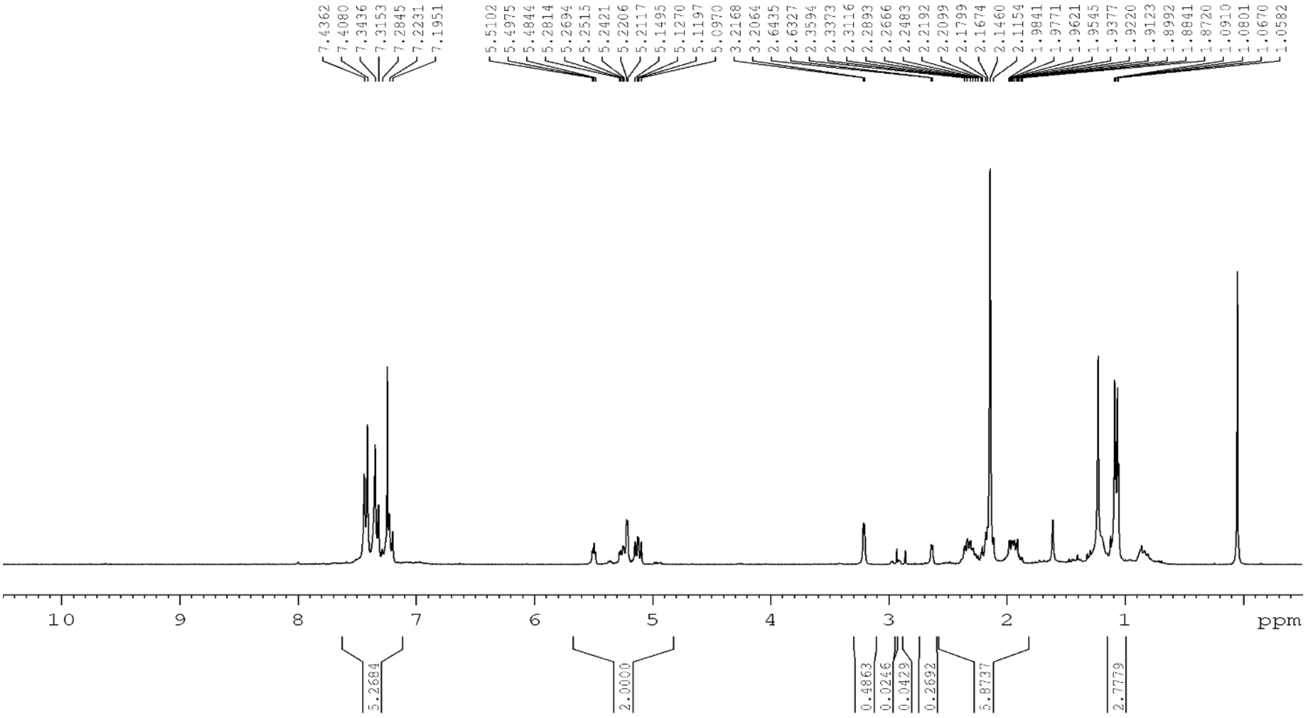
^{1}H NMR (300 MHz, CDCl$_3$)



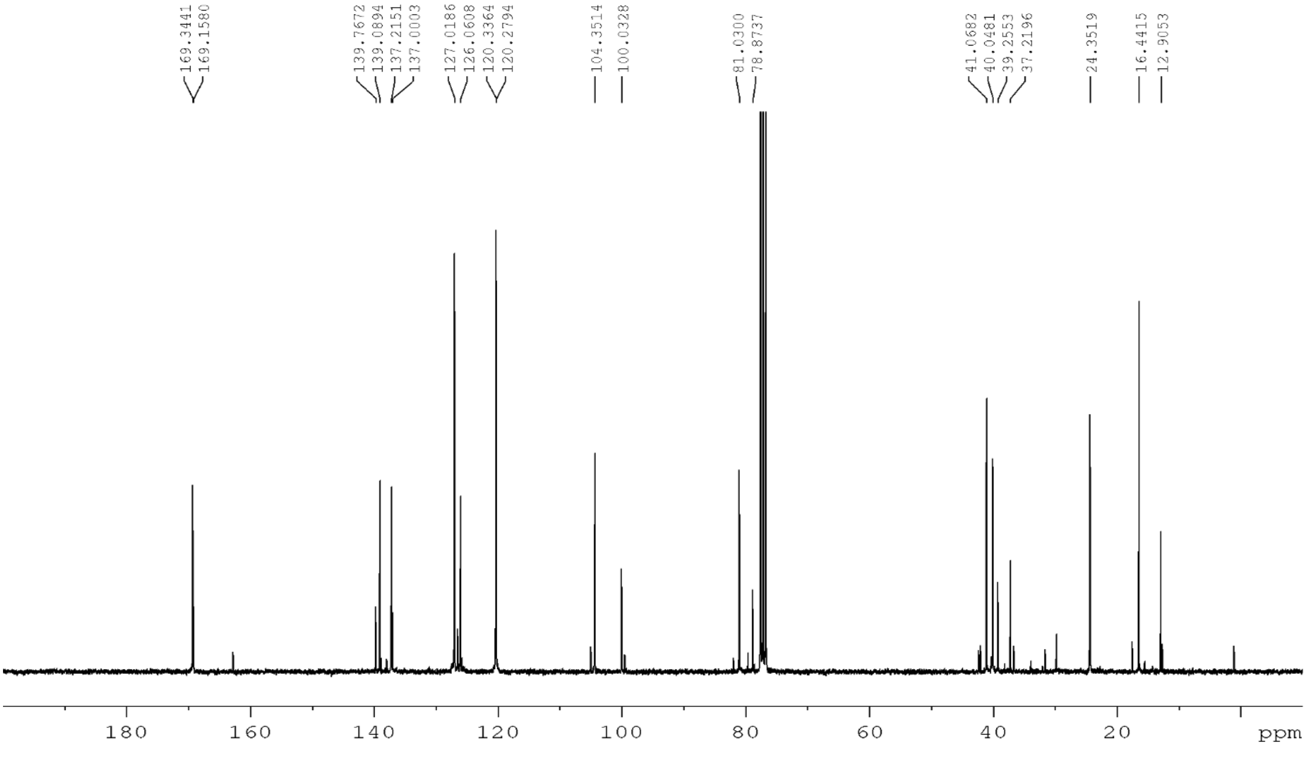
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

5-(4-acetamidophenyl)-2-hydroxy-3-methyltetrahydrofuran (2g)



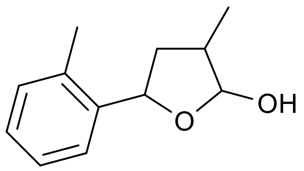


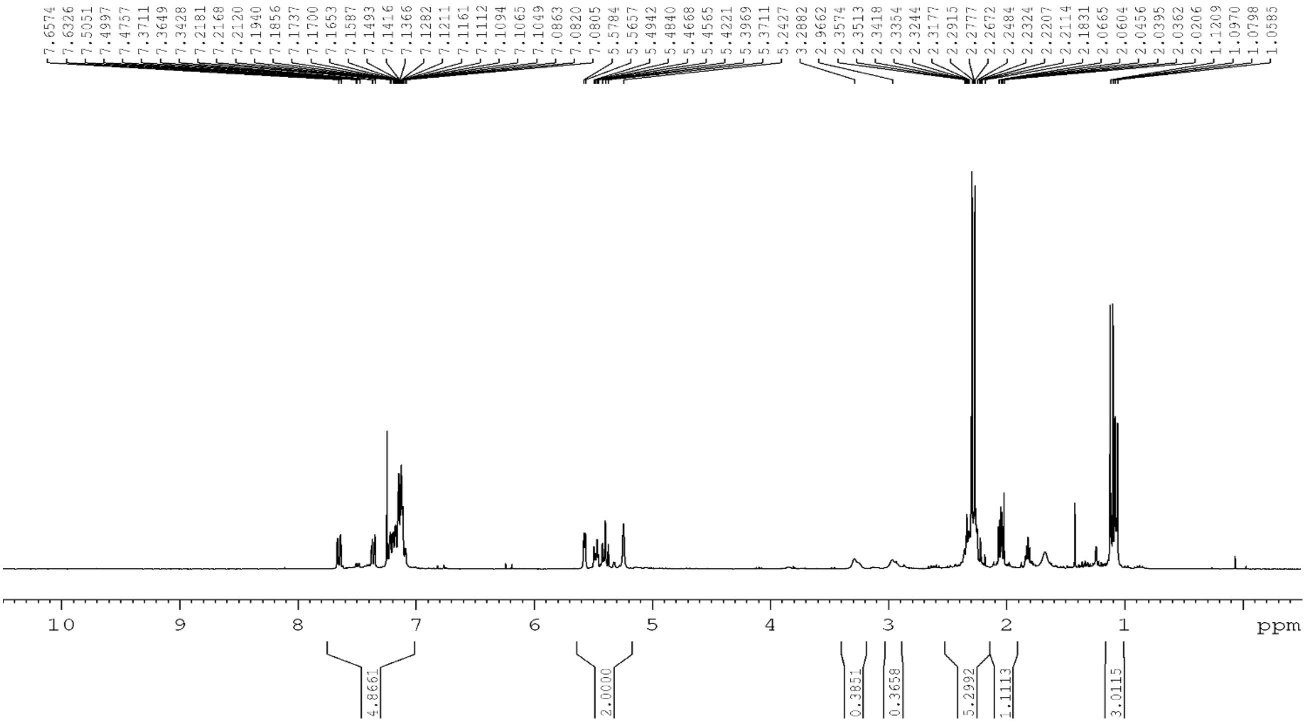
^{1}H NMR (300 MHz, CDCl$_3$)



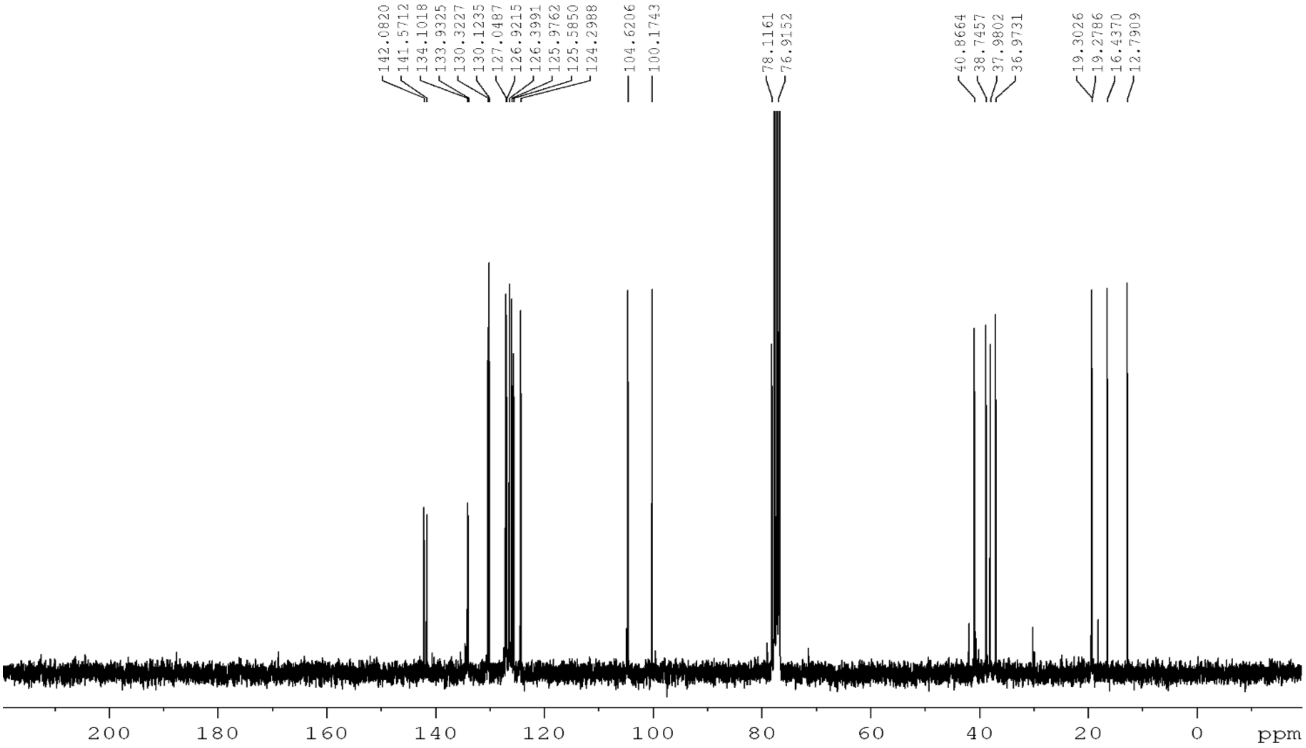
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-3-methyl-5-(2-methylphenyl)tetrahydrofuran (2h)



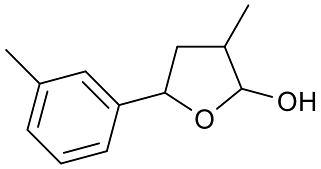


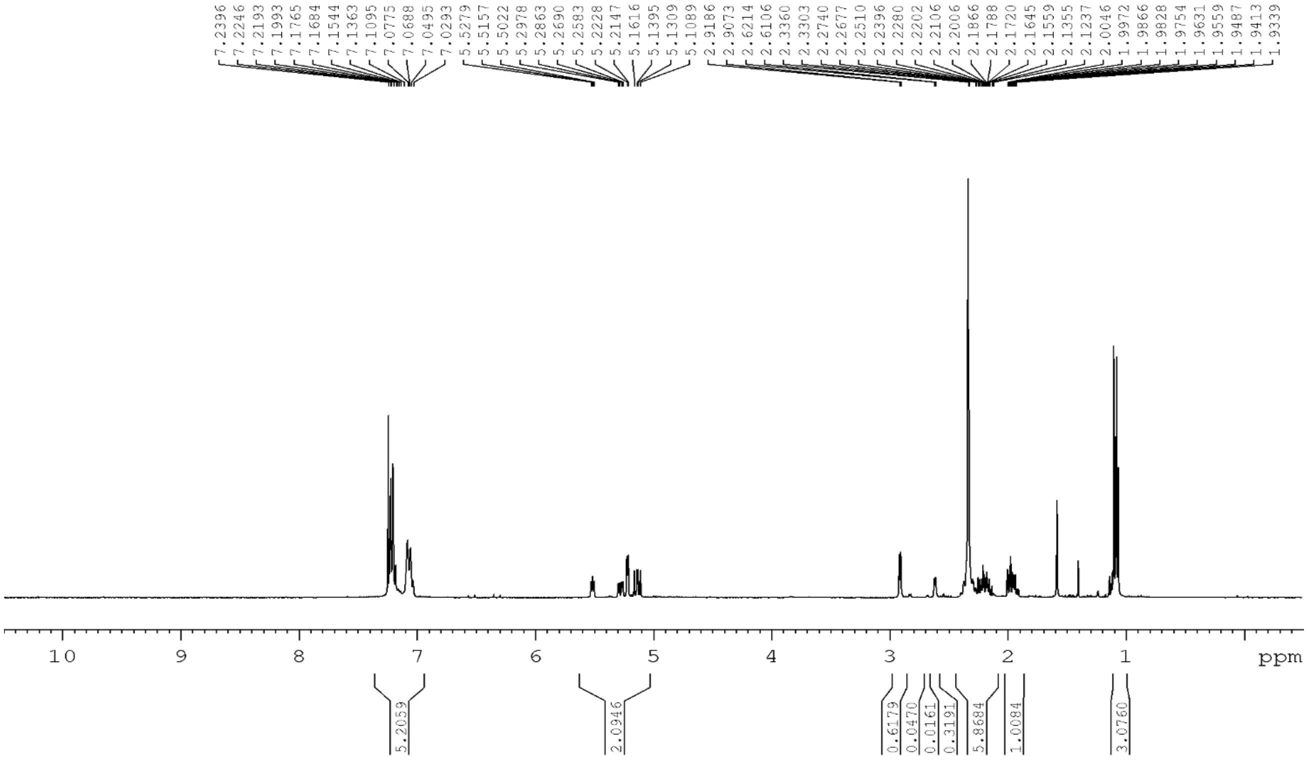
^{1}H NMR (300 MHz, CDCl$_3$)



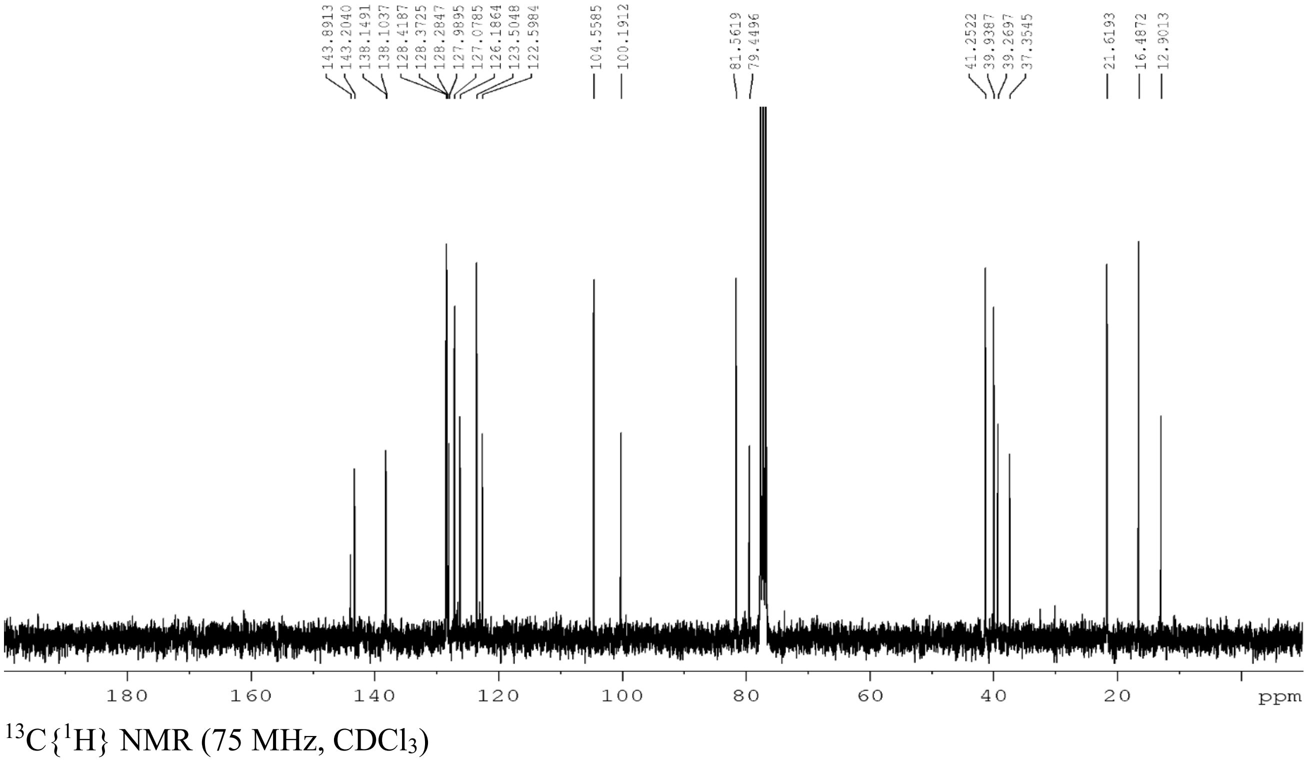
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-3-methyl-5-(3-methylphenyl)tetrahydrofuran (2i)



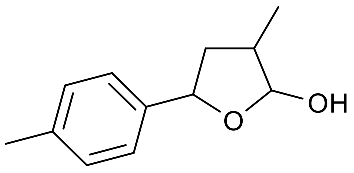


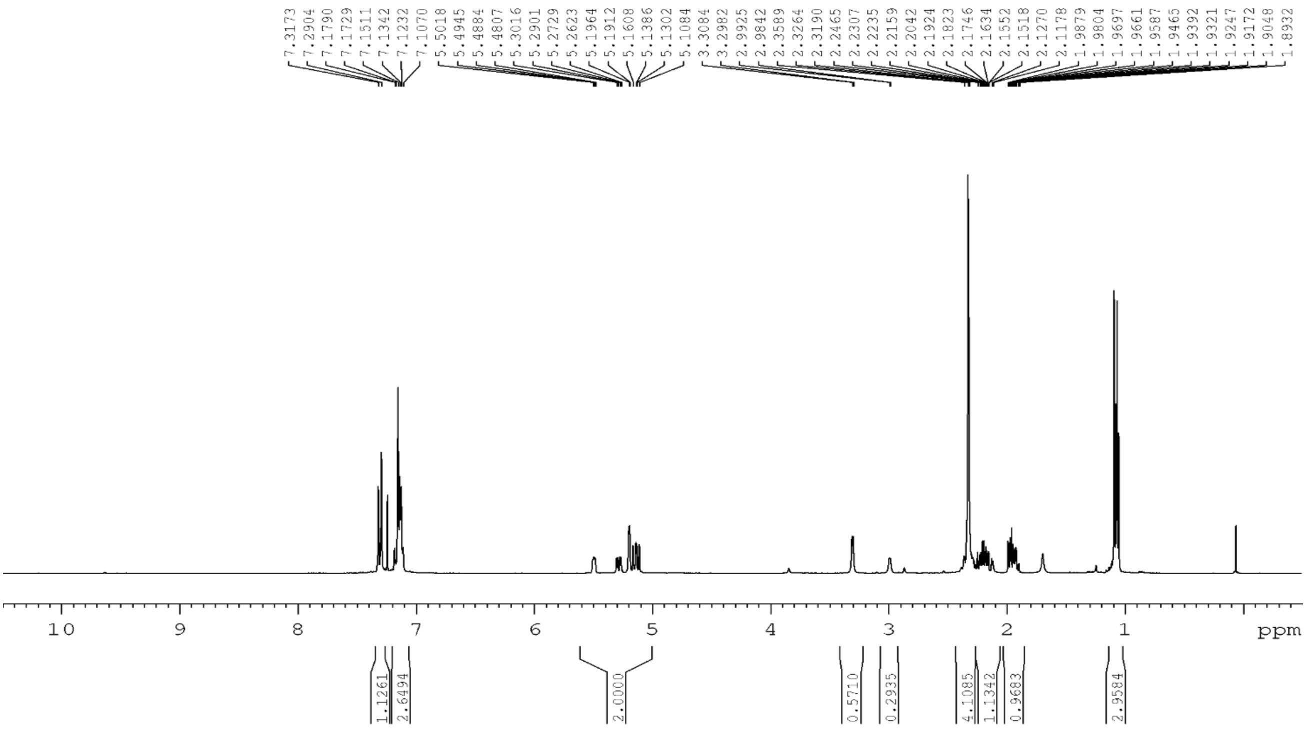
^{1}H NMR (300 MHz, CDCl$_3$)



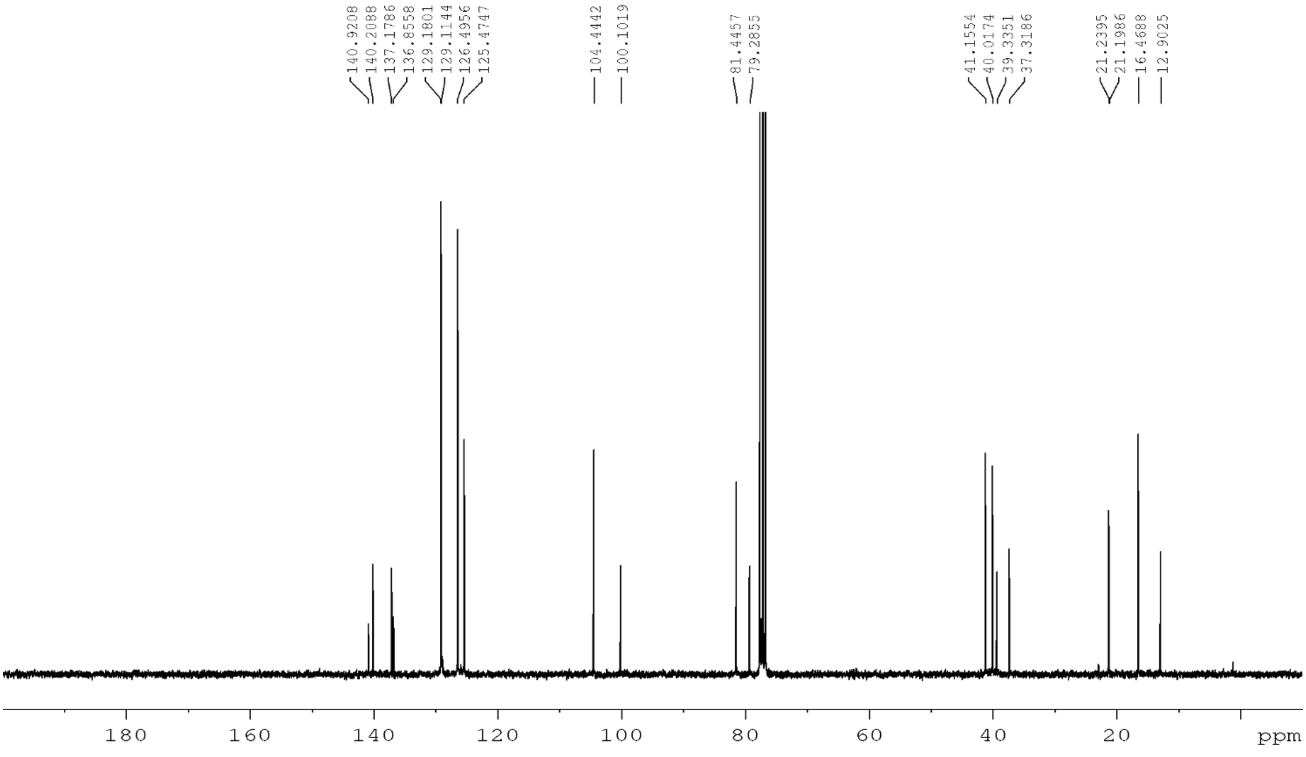
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-3-methyl-5-(4-methylphenyl)tetrahydrofuran (2j)



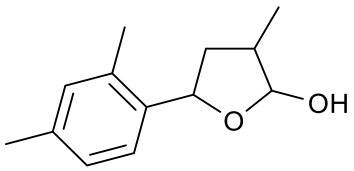


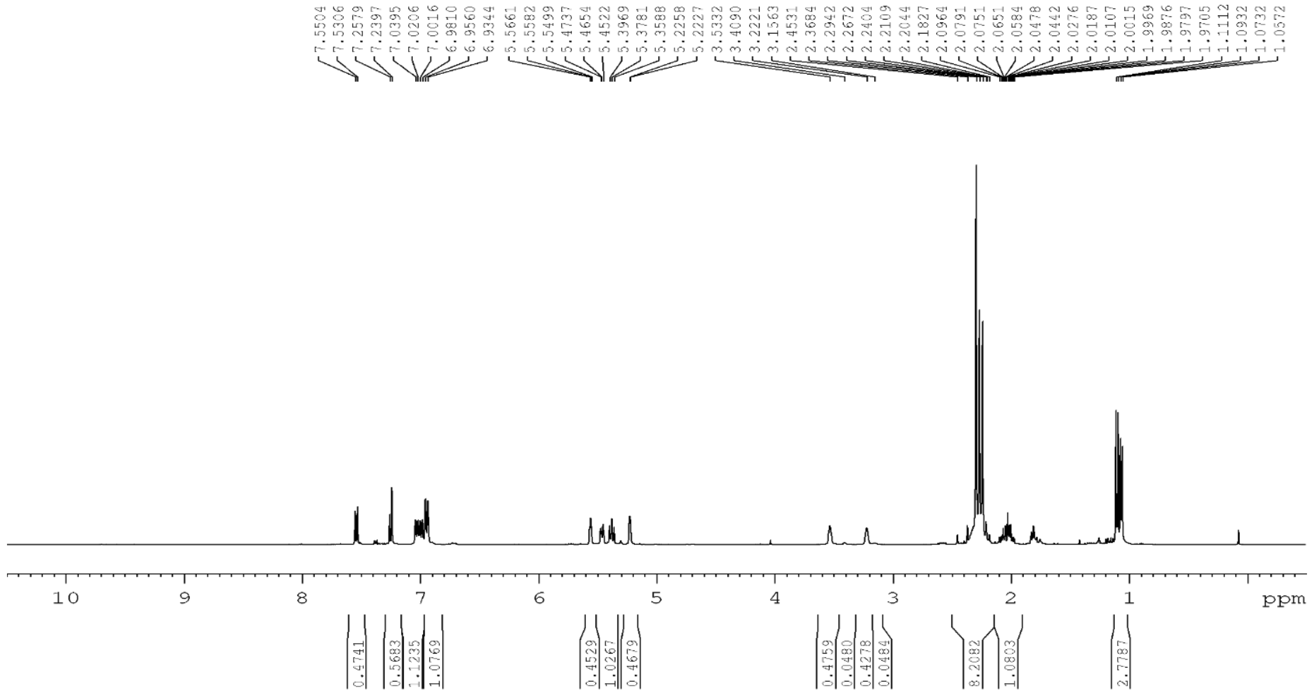
^{1}H NMR (300 MHz, CDCl$_3$)



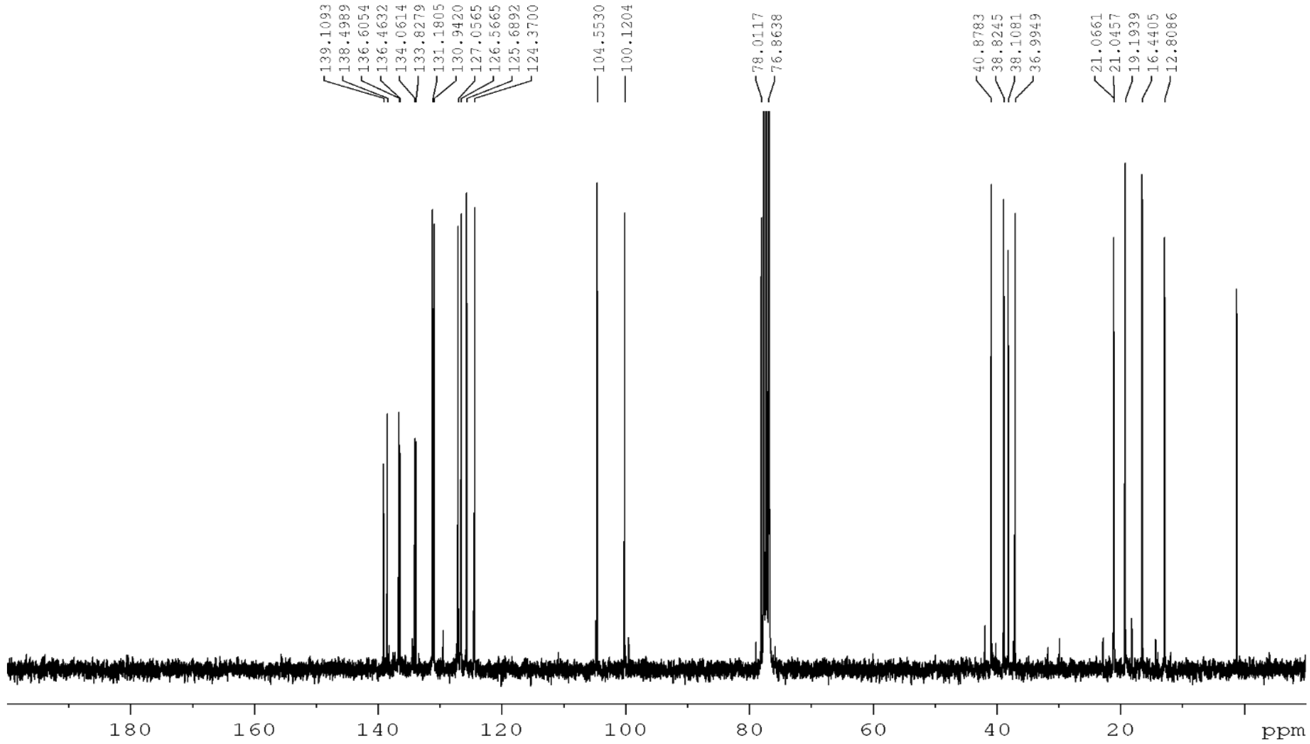
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-3-methyl-5-(2,4-dimethylphenyl)tetrahydrofuran (2k)



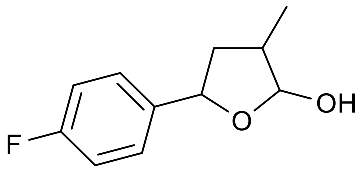


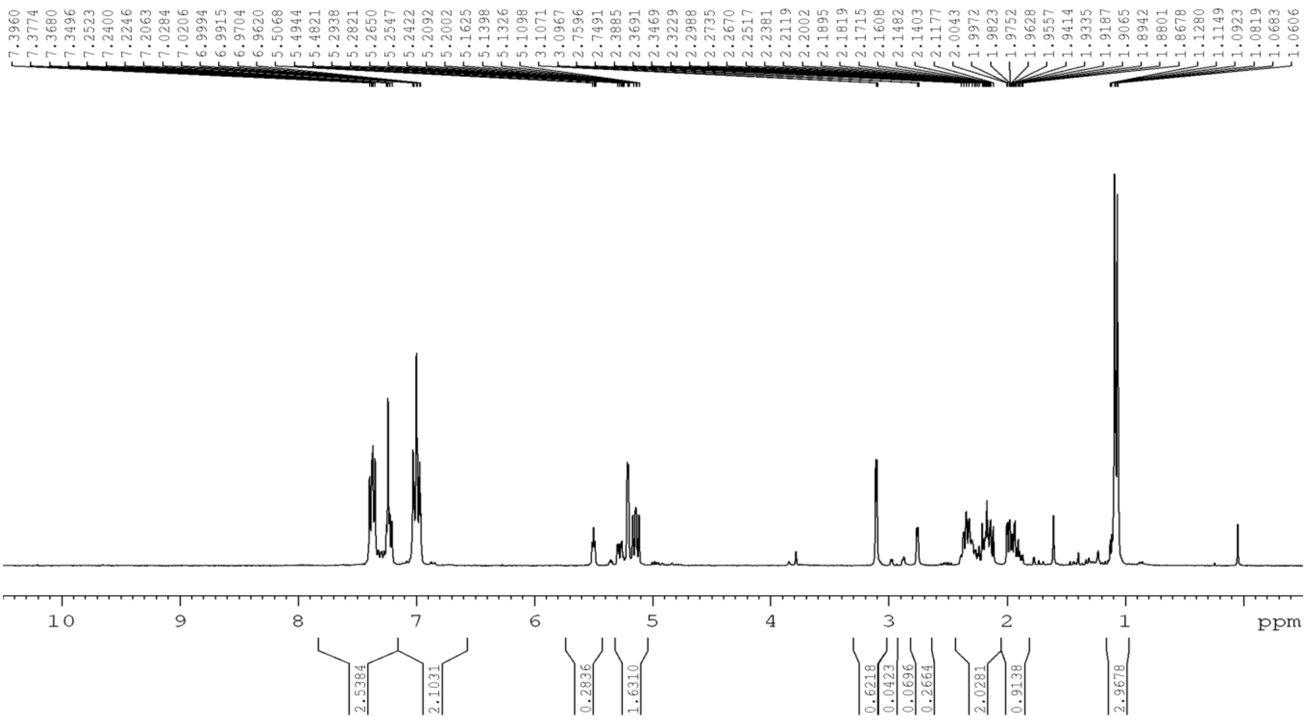
^{1}H NMR (300 MHz, CDCl$_3$)



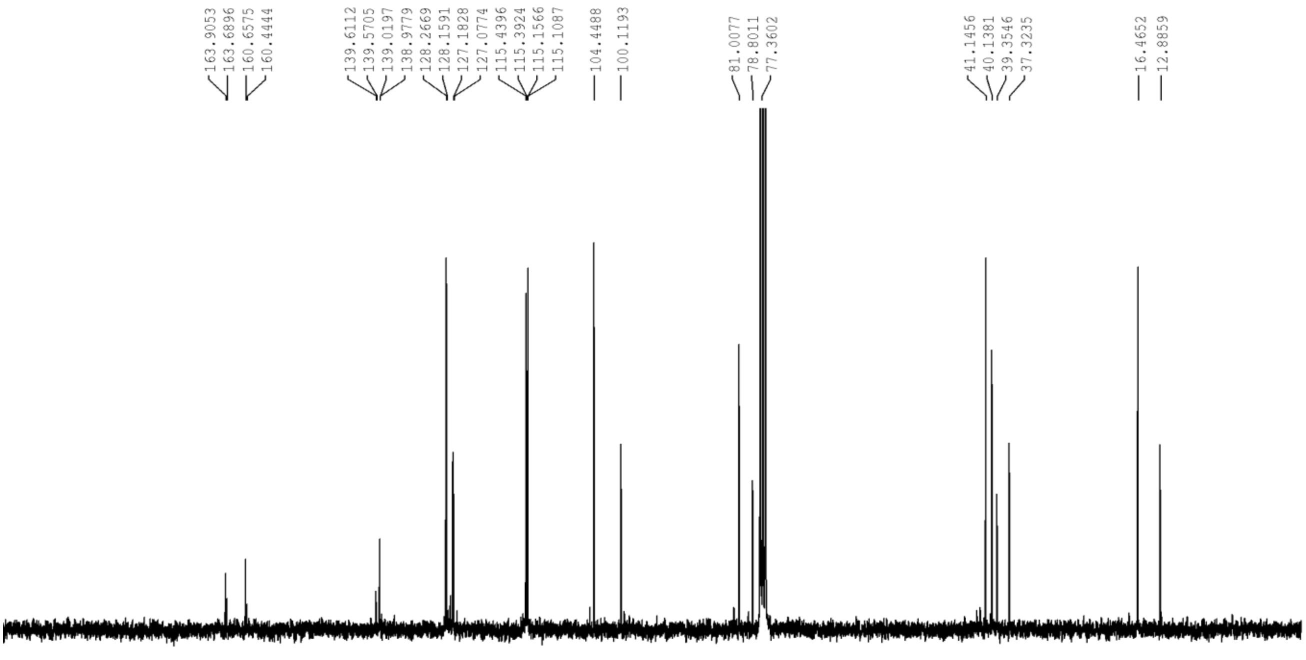
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

5-(4-fluorophenyl)-2-hydroxy-3-methyltetrahydrofuran (2l)



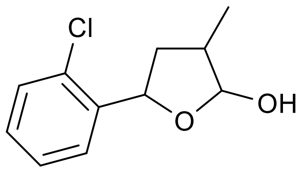


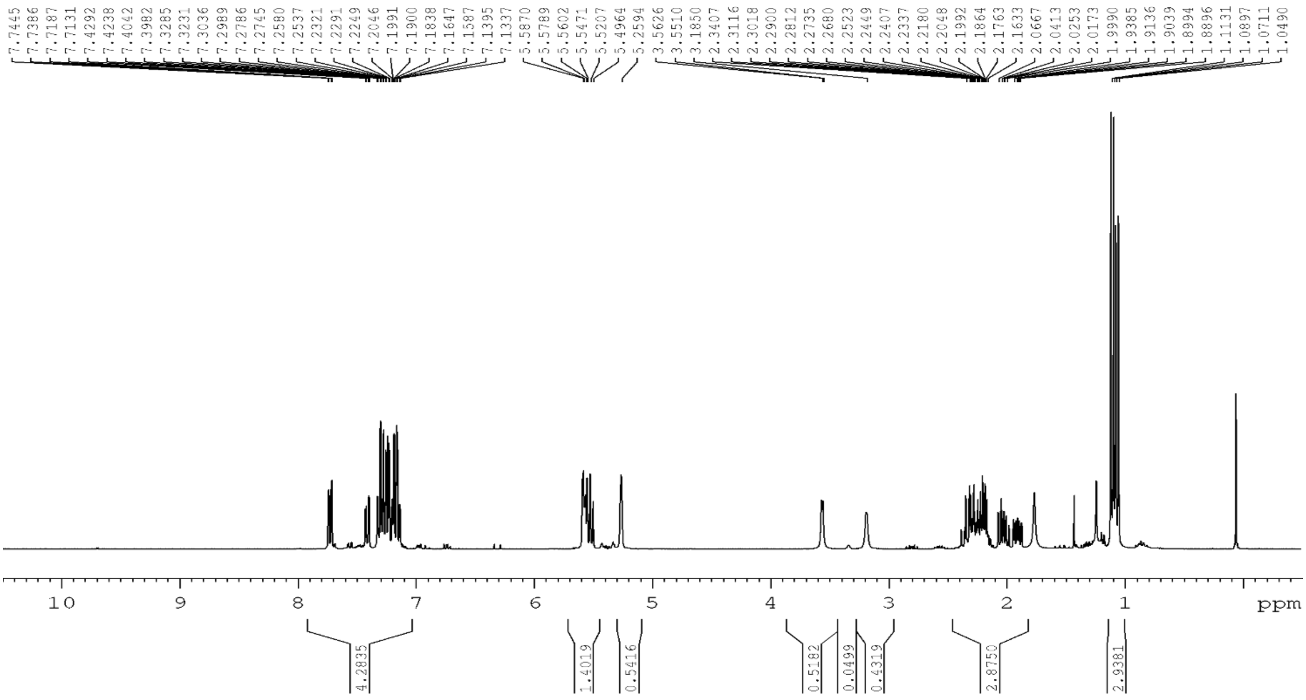
^{1}H NMR (300 MHz, CDCl$_3$)



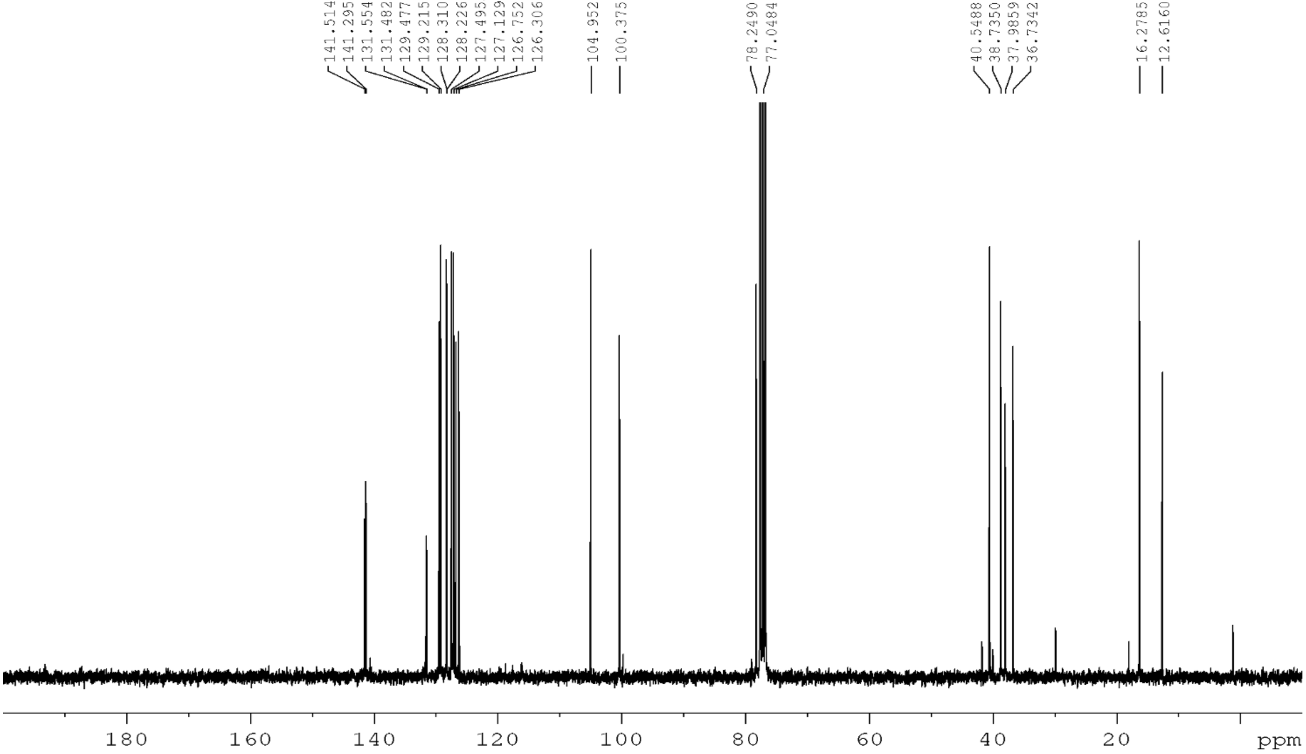
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

5-(2-chlorophenyl)-2-hydroxy-3-methyltetrahydrofuran (2m)



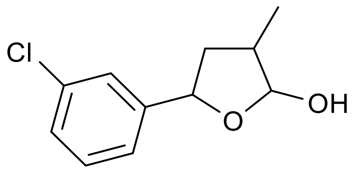


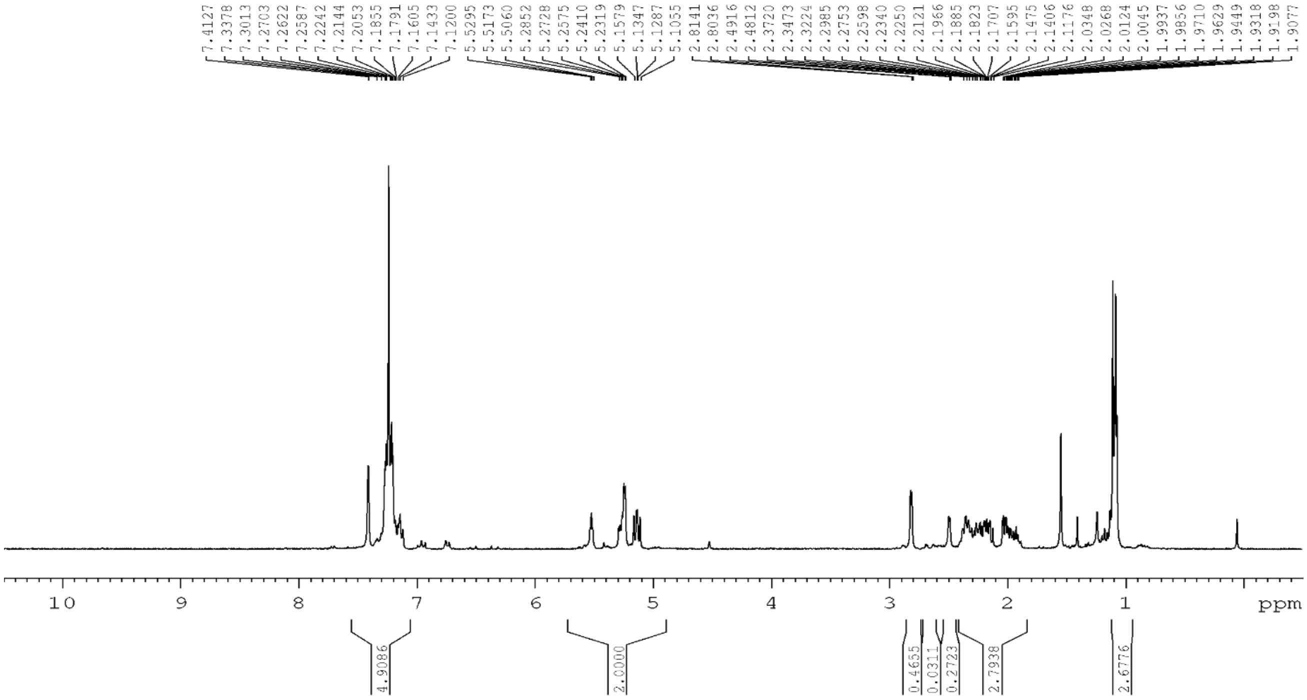
^{1}H NMR (300 MHz, CDCl$_3$)



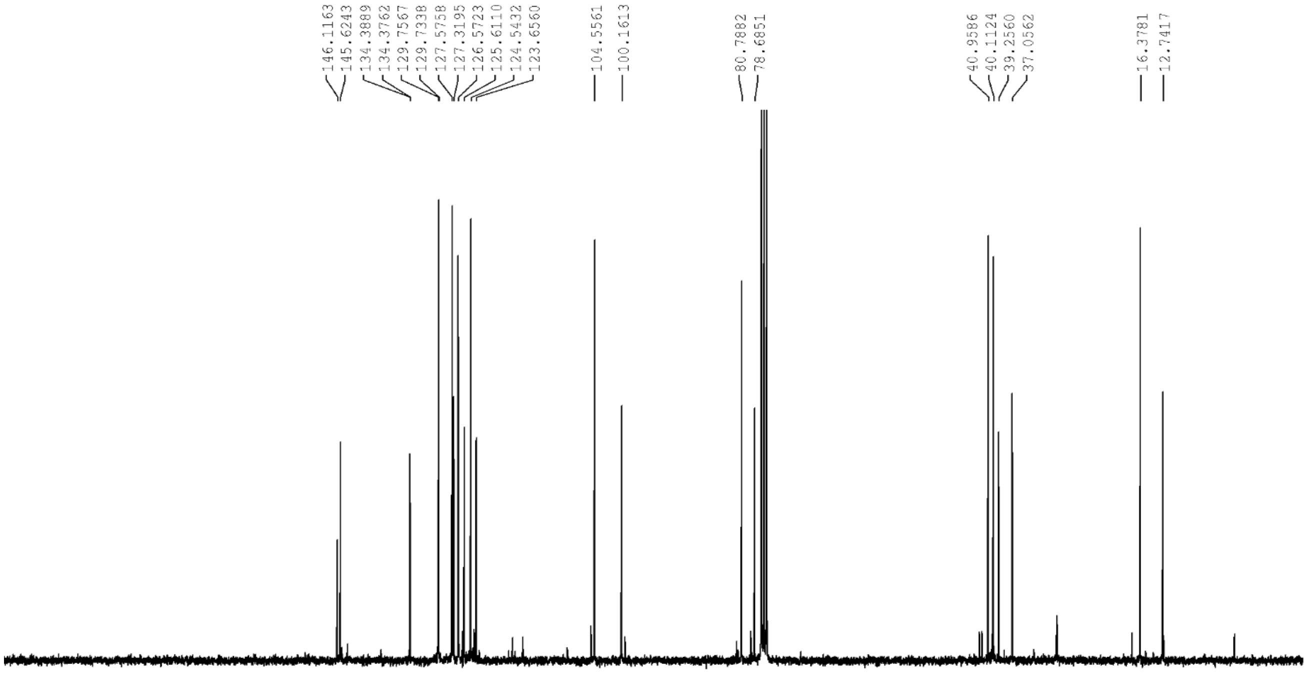
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

5-(3-chlorophenyl)-2-hydroxy-3-methyltetrahydrofuran (2n)



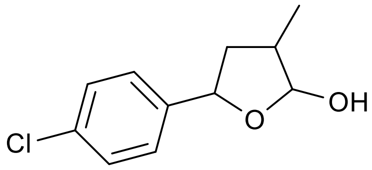


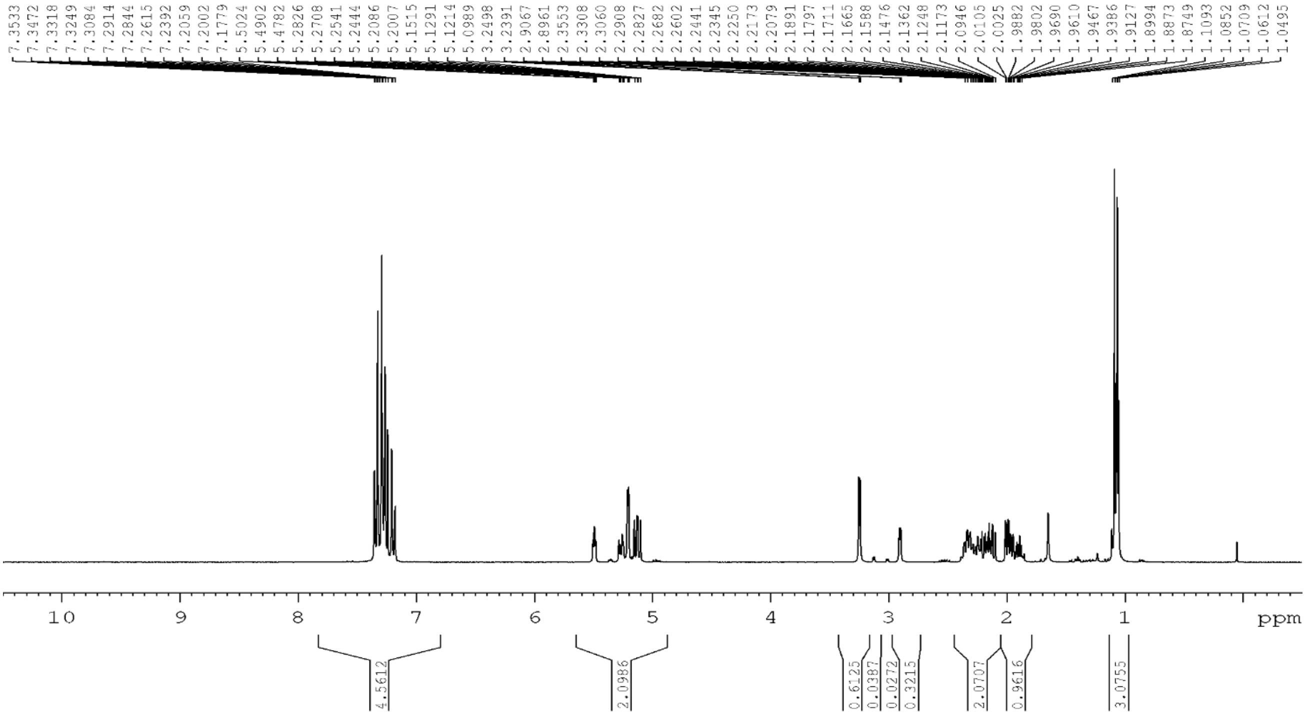
^{1}H NMR (300 MHz, CDCl$_3$)



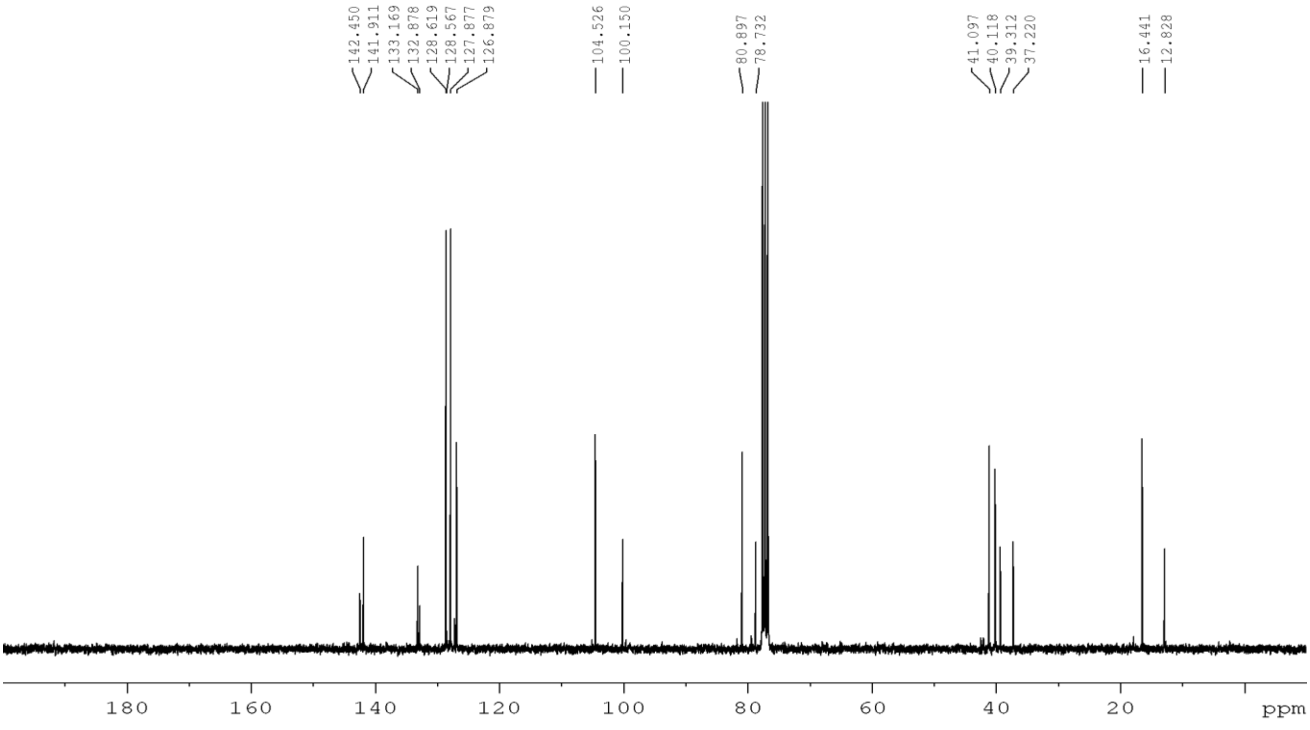
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

5-(4-chlorophenyl)-2-hydroxy-3-methyltetrahydrofuran (2o)



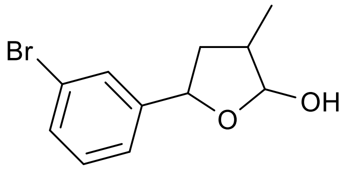


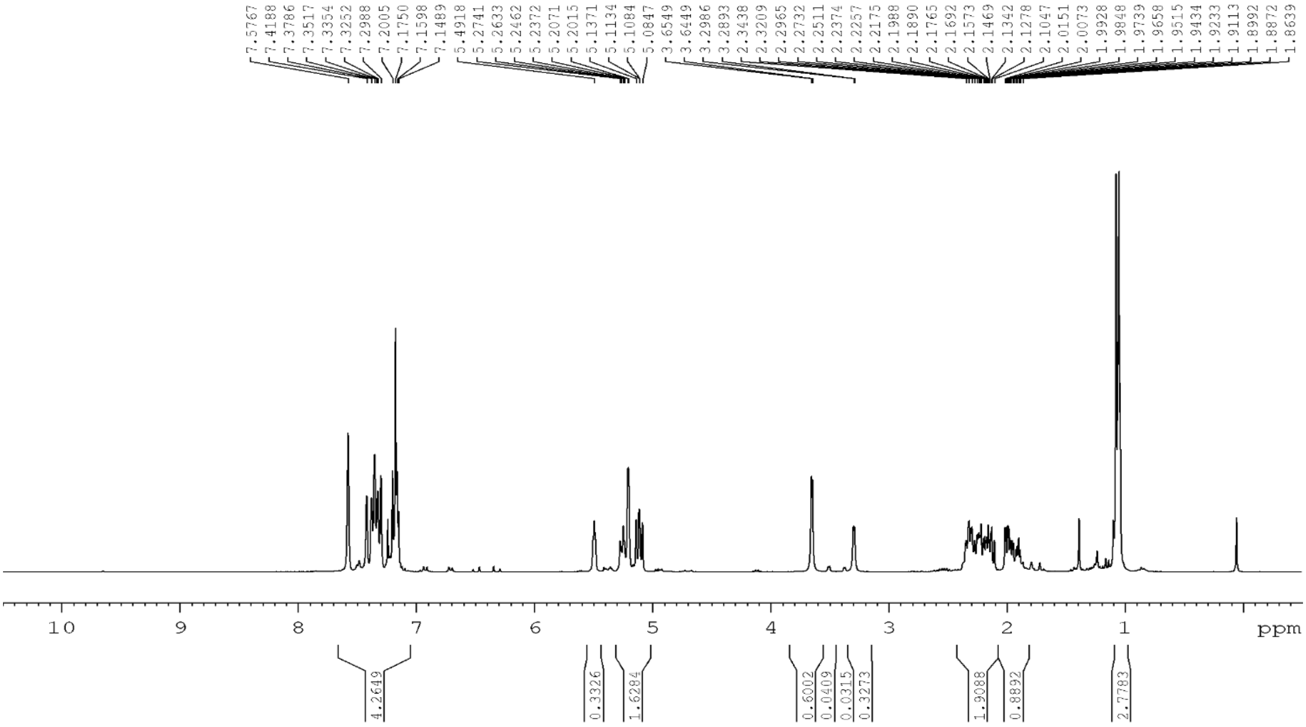
^{1}H NMR (300 MHz, CDCl$_3$)



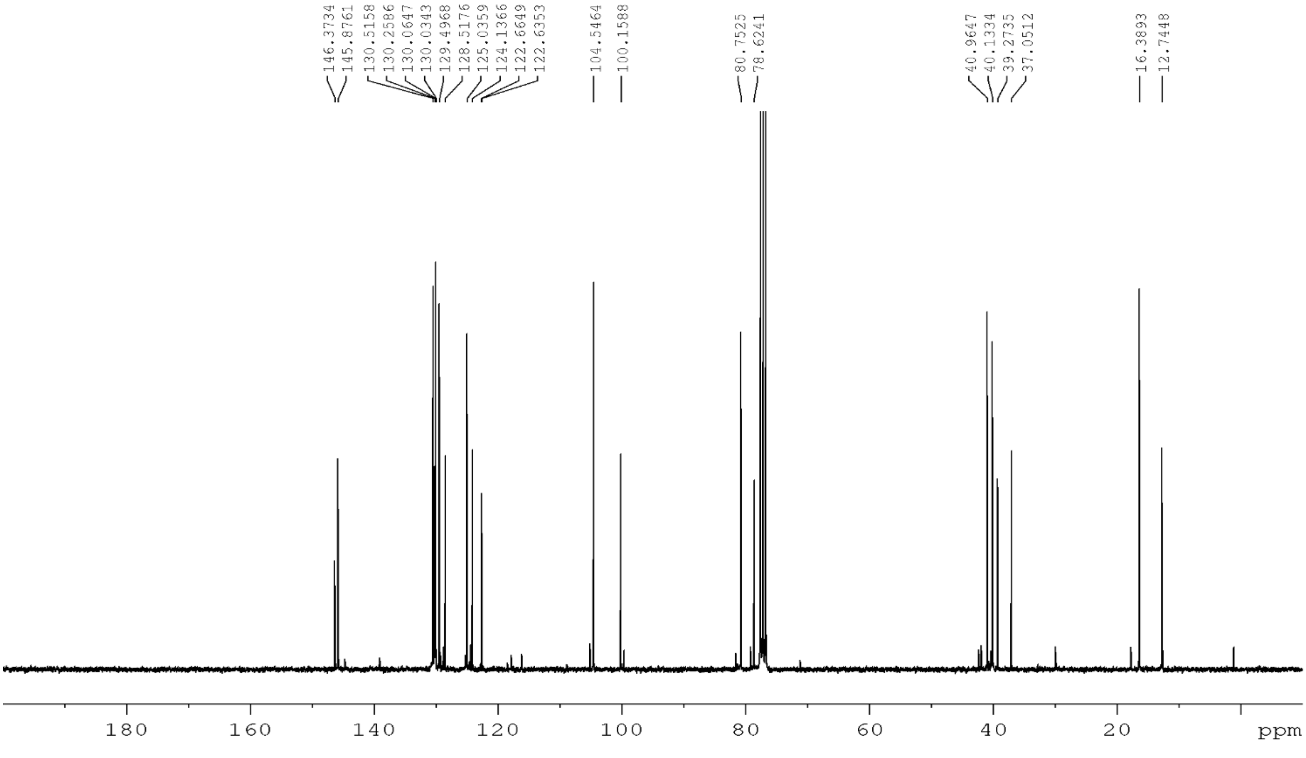
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

5-(3-bromophenyl)-2-hydroxy-3-methyltetrahydrofuran (2p)



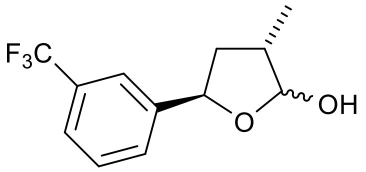


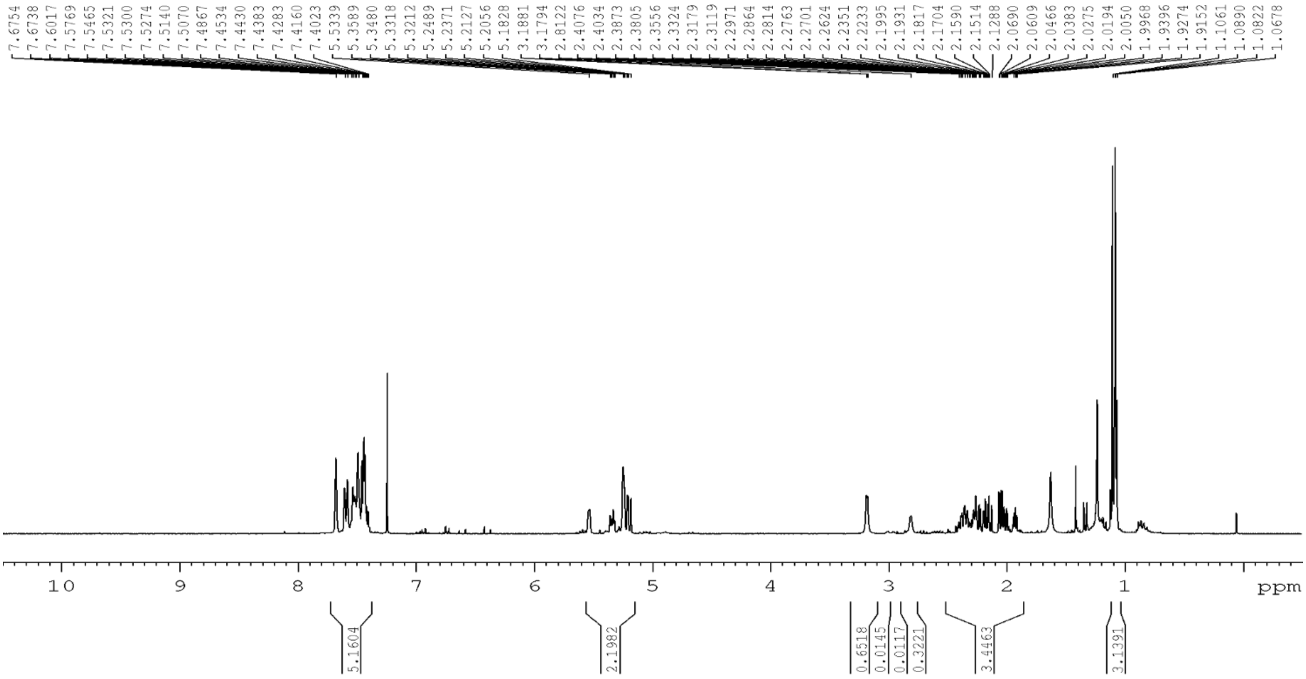
^{1}H NMR (300 MHz, CDCl$_3$)



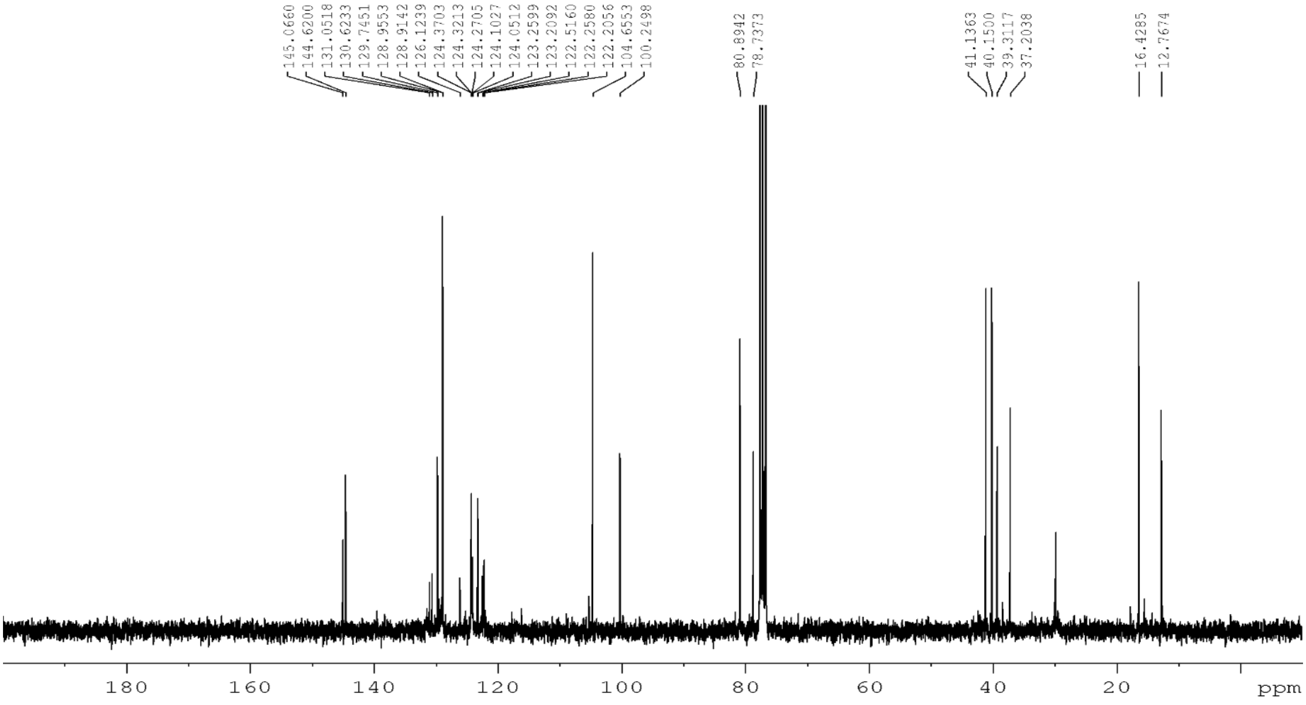
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-3-methyl-5-(3-trifluoromethylphenyl)tetrahydrofuran (2q)



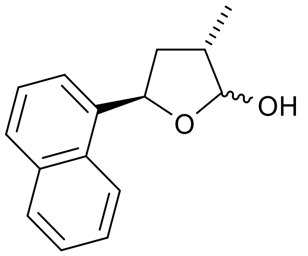


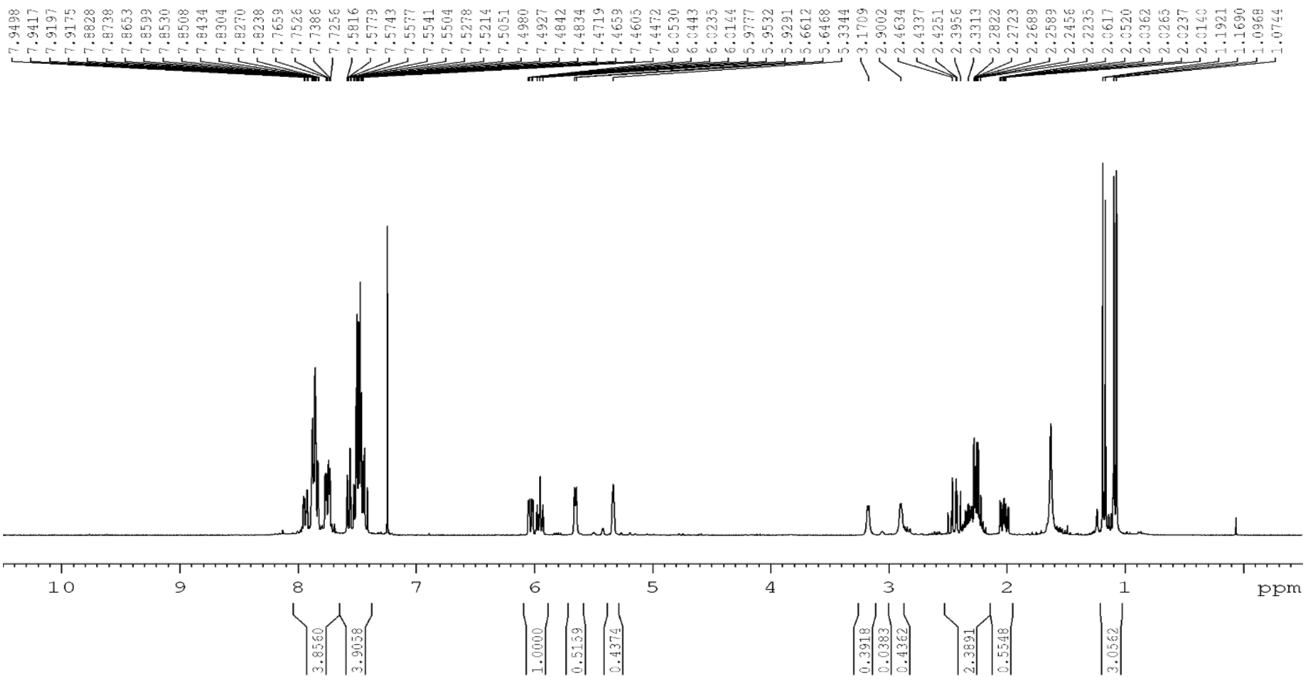
^{1}H NMR (300 MHz, CDCl$_3$)



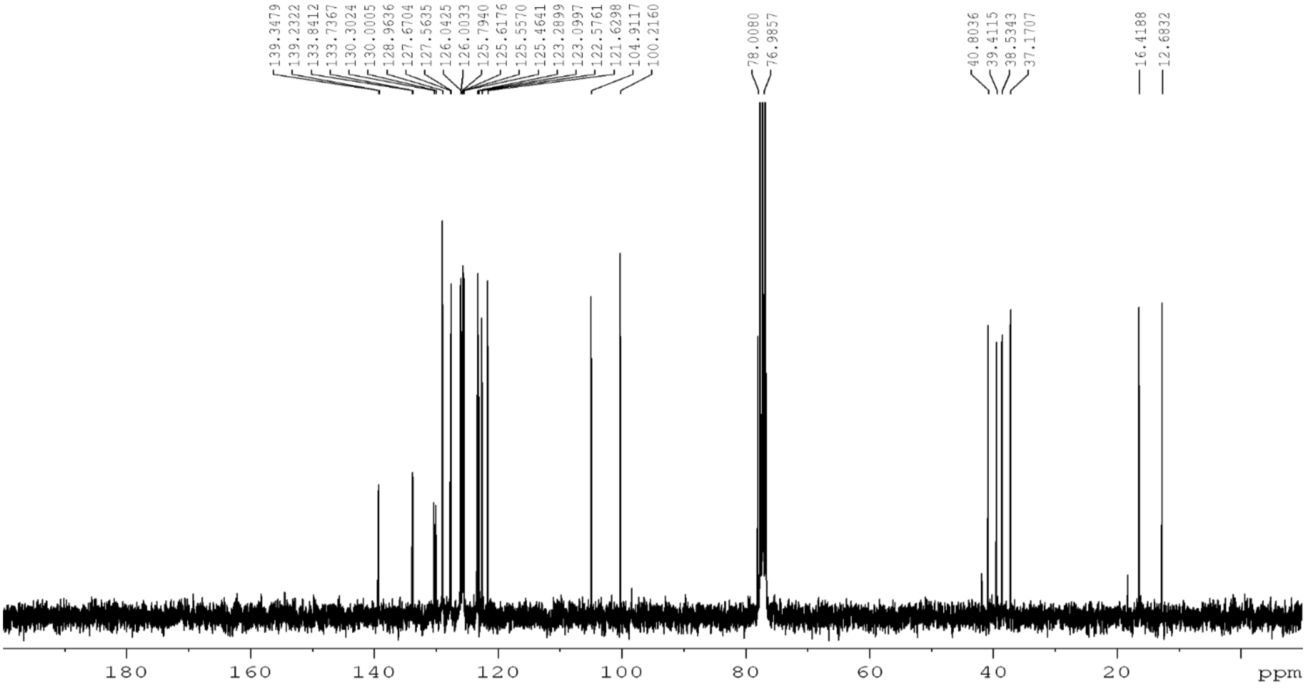
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

2-hydroxy-3-methyl-5-(1-naphthyl)tetrahydrofuran (2r)



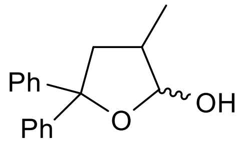


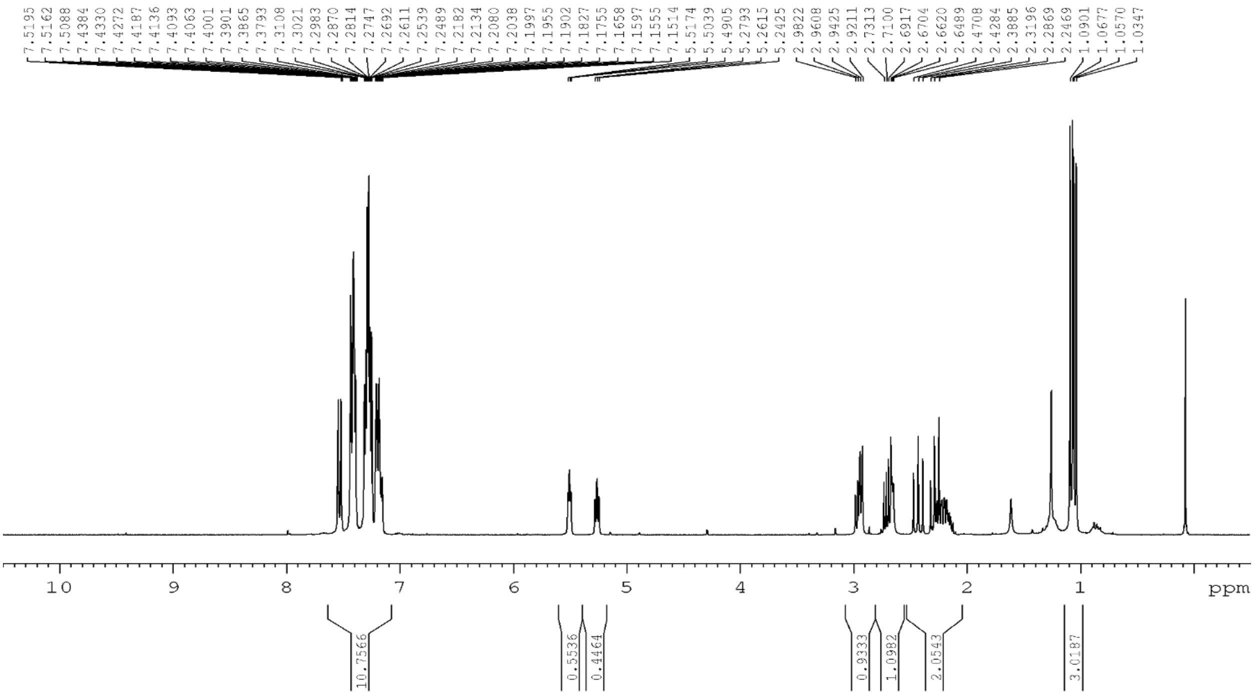
^{1}H NMR (300 MHz, CDCl$_3$)



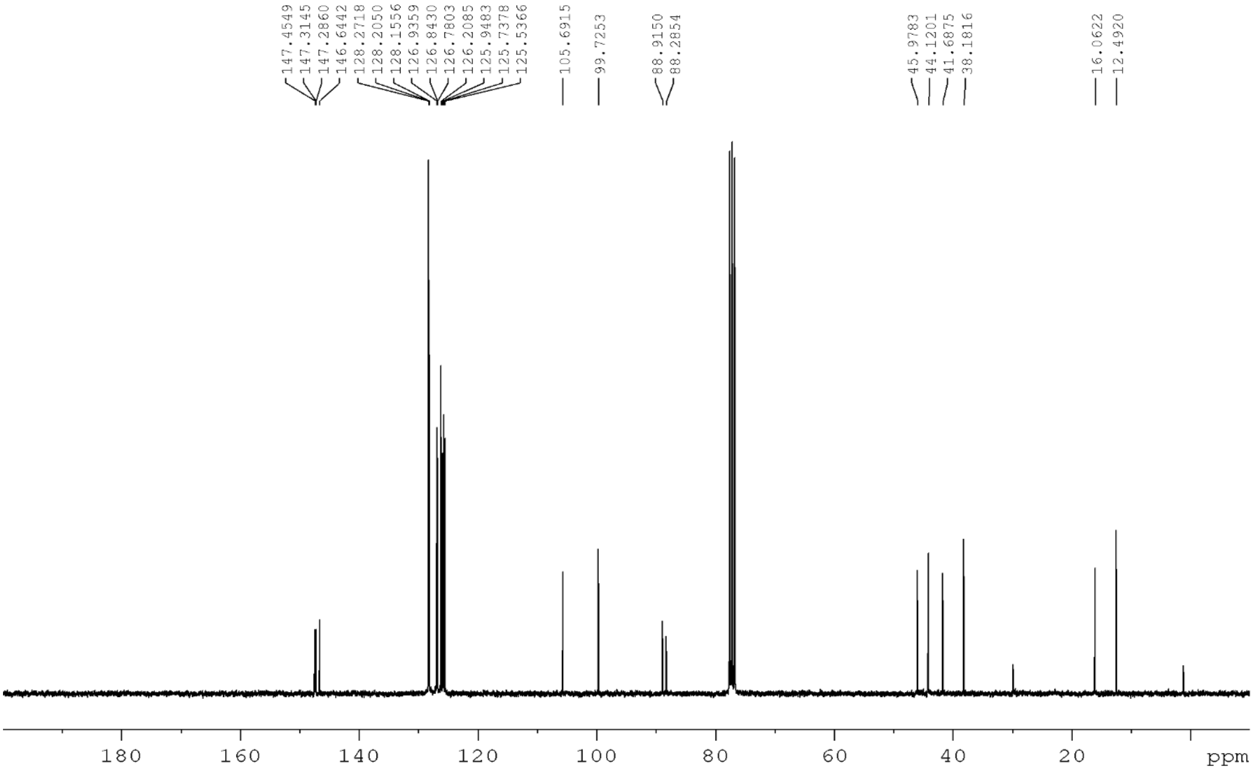
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

5,5-diphenyl-2-hydroxy-3-methyltetrahydrofuran (2s)



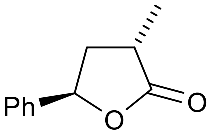


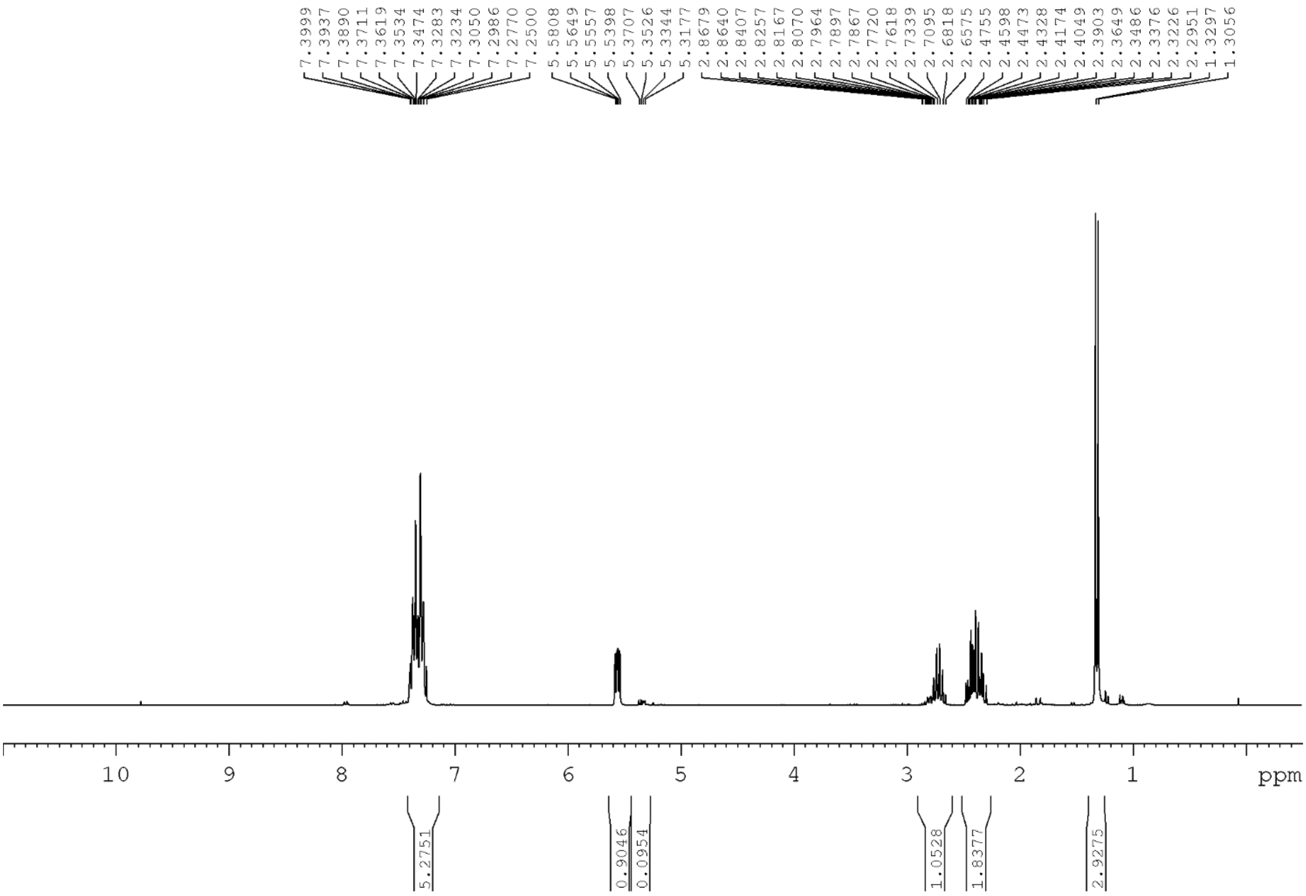
^{1}H NMR (300 MHz, CDCl$_3$)



^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)

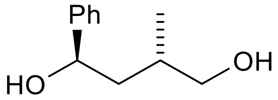
3-methyl-5-phenyltetrahydrofuran-2-one (3)

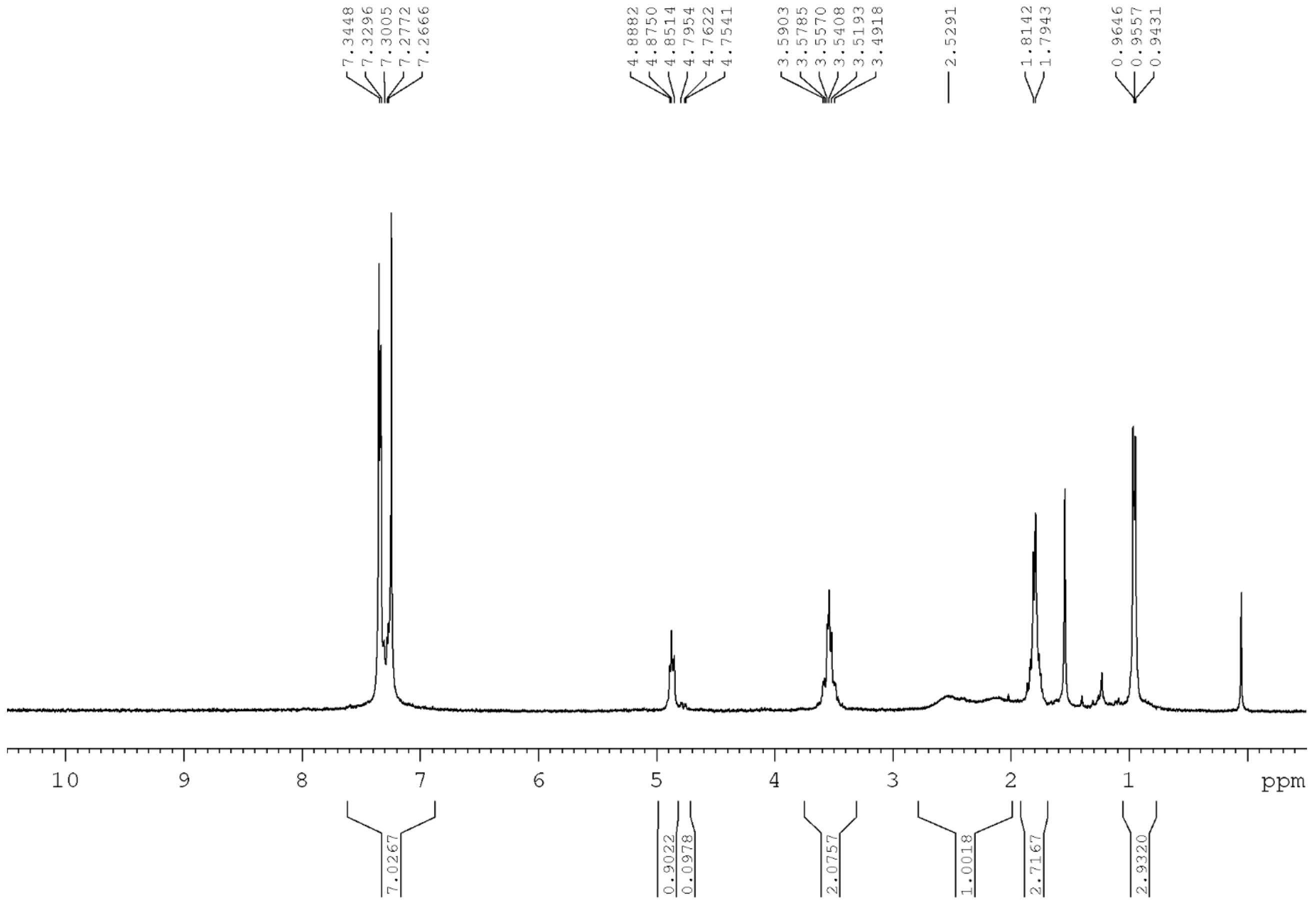




^{1}H NMR (300 MHz, CDCl$_3$)

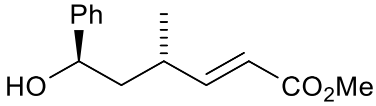
3-methyl-1-phenyl-1,4-butanediol (4)

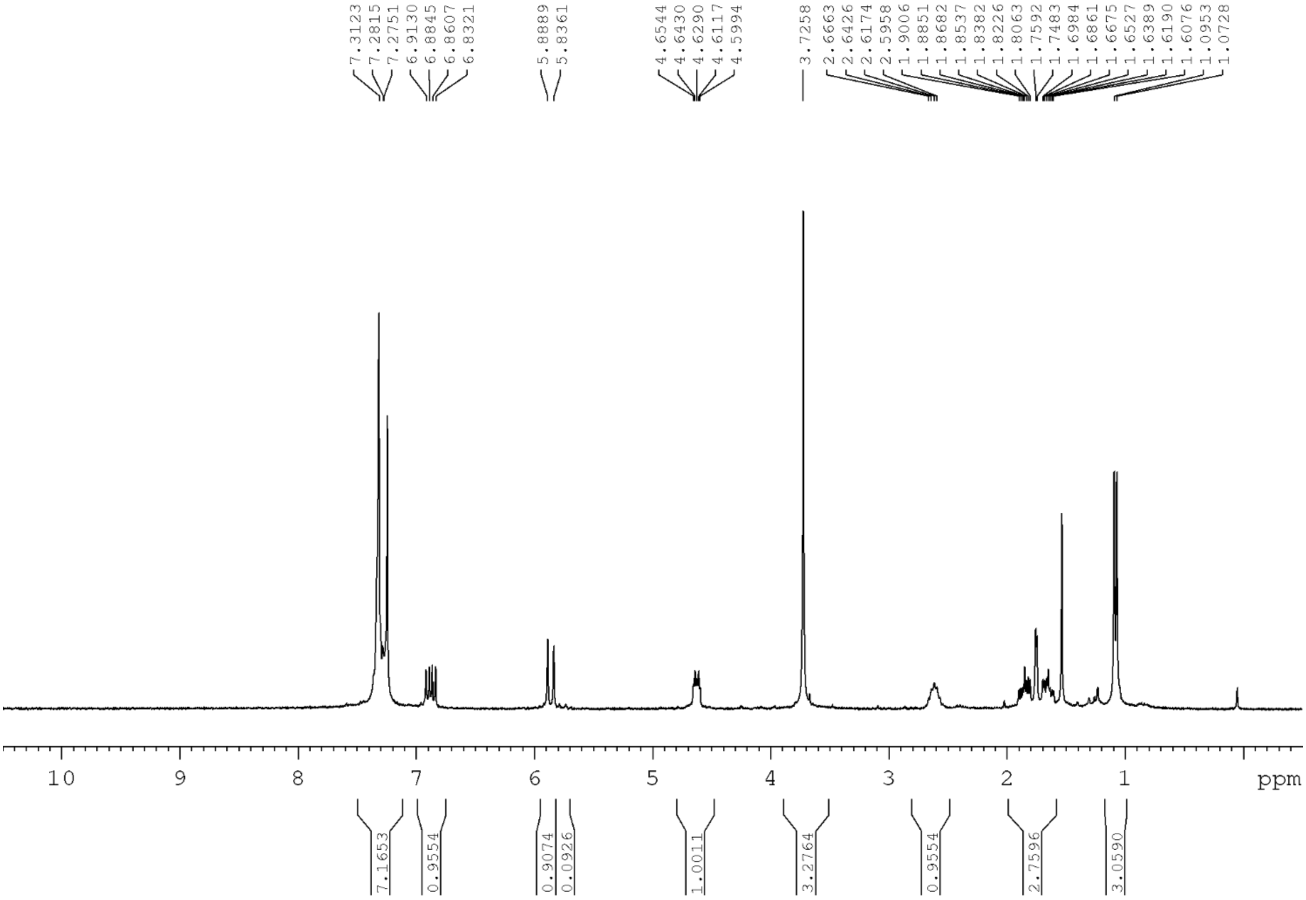




^{1}H NMR (300 MHz, CDCl$_3$)

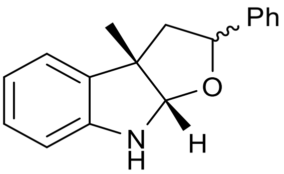
methyl 6-hydroxy-4-methyl-6-phenyl-2-hexenoate (5)

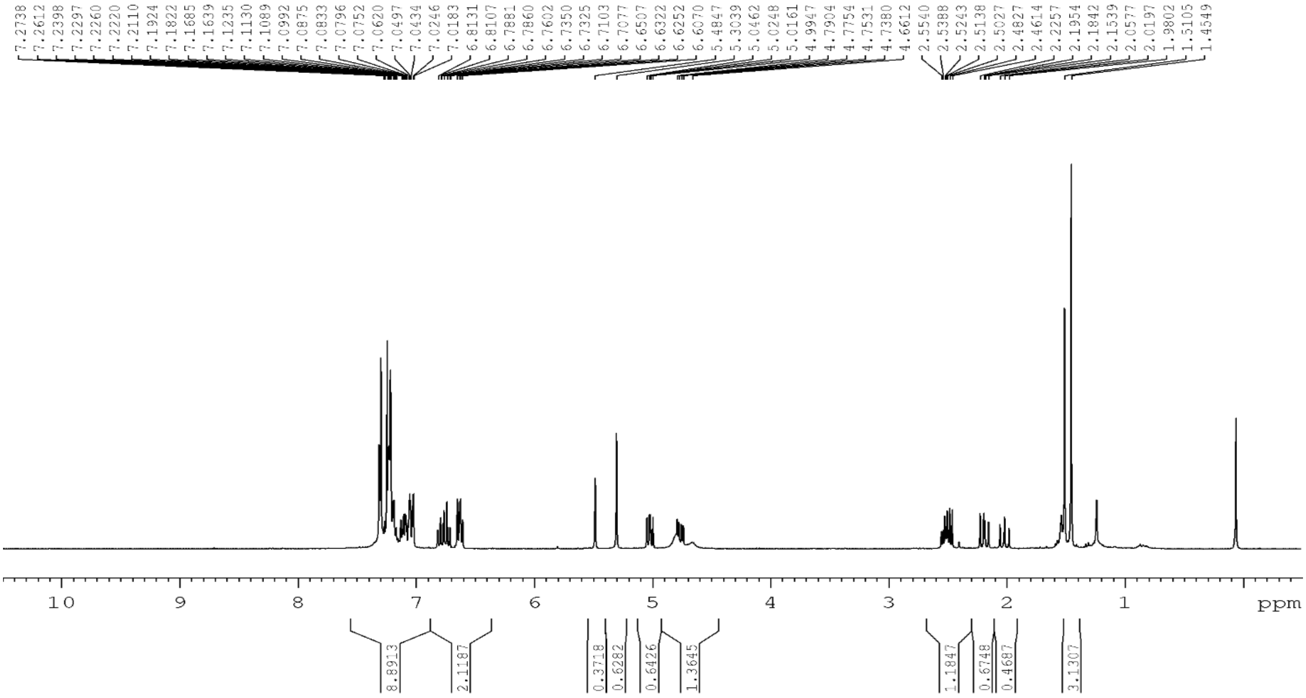




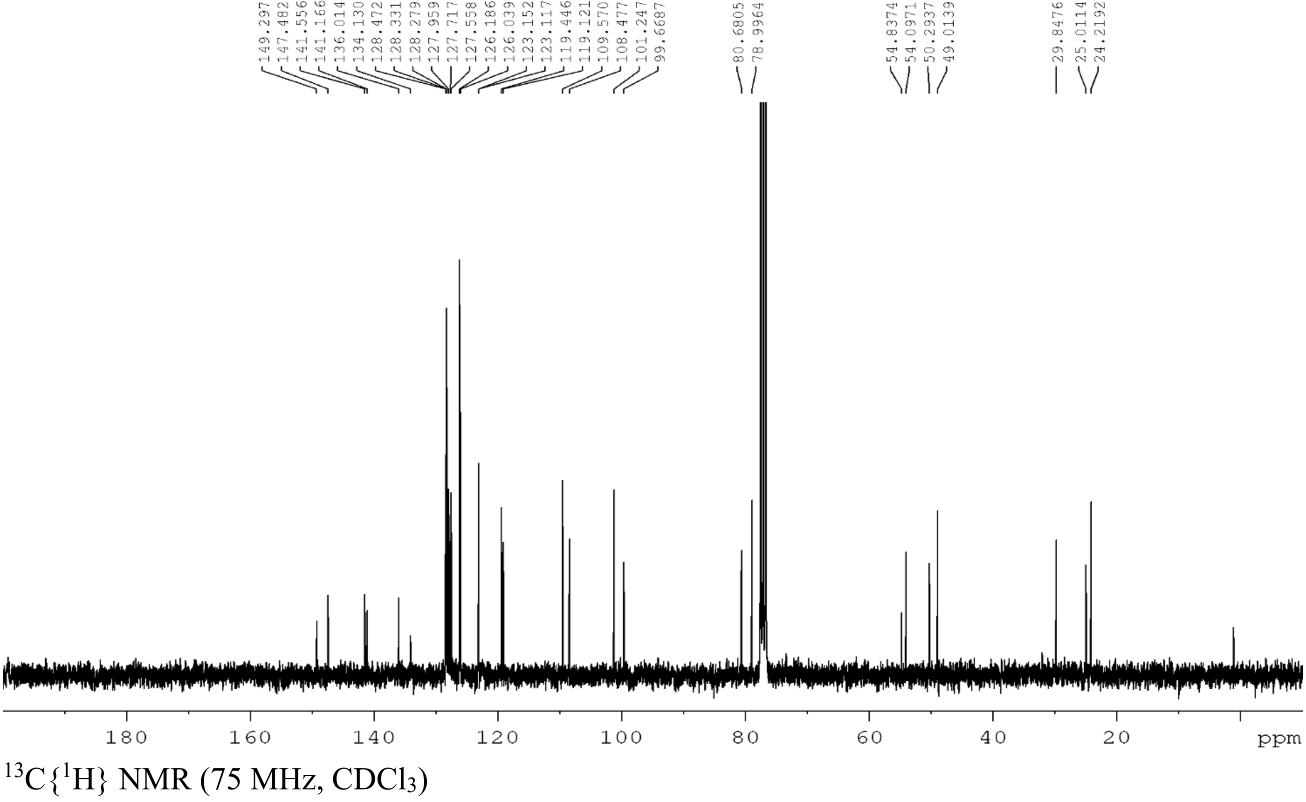
^{1}H NMR (300 MHz, $CDCl_3$)

Compound 6





^{1}H NMR (300 MHz, $CDCl_3$)



^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)

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