Electronic Supplementary Material (ESI) for Green Chemistry.

On-demand, in situ, generation of ammonium caroate (peroxomonosulfate) for the dihydroxylation of alkenes to vicinal diols

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KEYWORDS: dihydroxylation, oxidation, electrochemical, biphasic, process analysis

Datasets associated with this publication are available from the Imperial College London Research Data Repository under Collection DOI 10.14469/hpc/10417

General

Unless otherwise stated, NMR spectra were recorded on a Bruker 400 Avance NMR Spectrometer (^{1}H: 400 MHz, ^{13}C: 101 MHz), using $CDCl_3$ as the solvent. Chemical shifts are reported in δ (ppm) referenced to the residual signals of $CHCl_3$ (δ_H 7.27) and $CDCl_3$ (δ_H 77), respectively. Infra-red spectra were recorded on a PerkinElmer Spectrum RX-I FTIR Spectrophotometer. MS analysis was performed using the Micromass AutoSpec Premier Mass Spectrometer and GC/MS analysis when coupled with the Agilent HP6890 GC system. Melting points were determined using a Stanford Research Systems OptiMelt MPA100 and were uncorrected. HPLC data was obtained using a HP 1100 LC system with a DAD detector, fitted with a C18 column. GC-FID analysis was performed using an Agilent HP6890 gas chromatography system fitted with an Agilent 7683 injector and FID detectors. The chemicals were obtained from commercial sources and were used without prior purification.

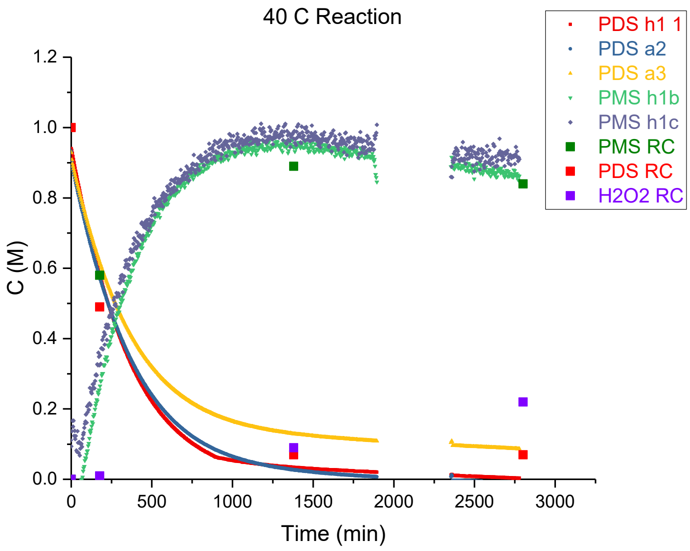
Description of the electrochemical reactor

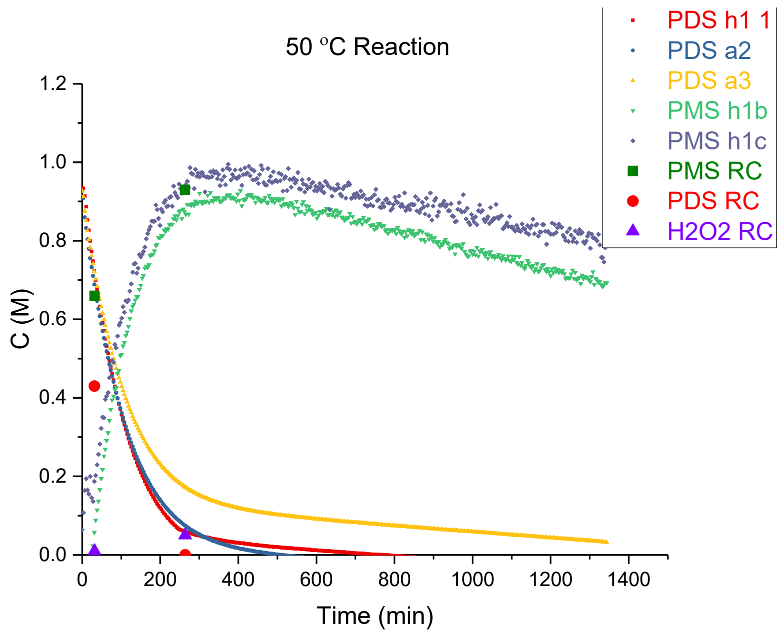
Full details and a schematic depiction of the electrochemical reactor have been reported previously.[1] The electrochemical reactor was constructed from a commercially available electrolytic flow cell (MFC, EC ElectroCell®) powered by a DC power supply (IPS 3303, ISO-TECH). The MFC flow cell contained a boron doped diamond anode (BDD coated on a Niobium substrate) and a stainless steel (SS316) cathode, separated by a Nafion® 424 ion-selective membrane. The electrolytic layers of the flow cell were sandwiched between two cooling plates through which ice-cold water was continuously circulated by a peristaltic pump.

The electrolyte solutions were held in two glass conical flasks (250 mL, separate flasks for anolyte and catholyte) and circulated through the flow cell by a dual-channel peristaltic pump (MasterFlex L/S, Cole-Parmer).

Kinetic experiments

See experimental section for procedure.





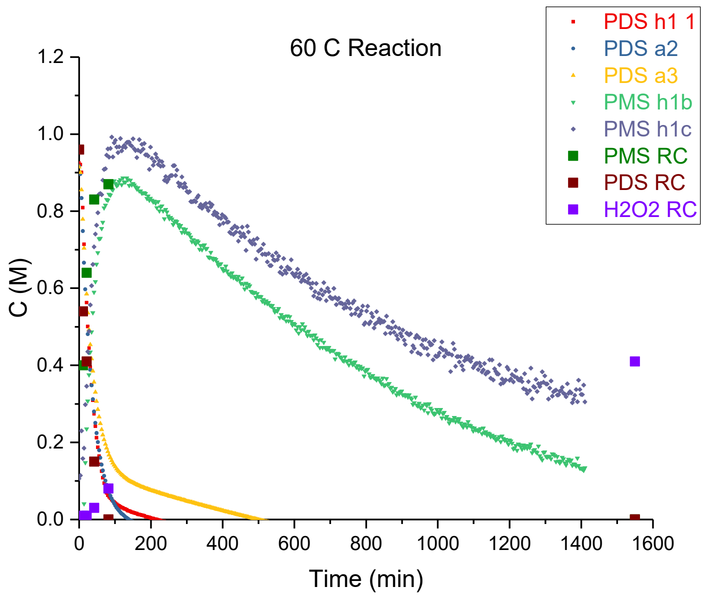


Figure S1. Decomposition of PDS in acidic solutions at 40, 50 and 60 °C. Concentrations of PDS and PMS were monitored by IR spectroscopy and the concentrations of H_2O_2 was determined by redox colorimetric method.[2]

Fitting of kinetic model

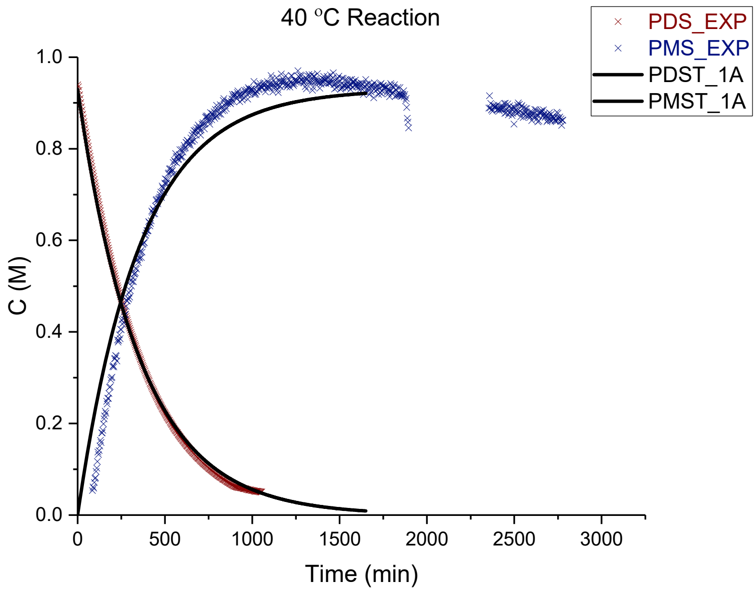
Simplified model which considers only the decomposition of PDS to PMS (Pseudo first order).

PDS -> PMS

Kinetic constants were extracted by fitting the model to experimental data using Berkeley Madonna differential equation solver.[3]

```
METHOD RK4

STARTTIME = 0
STOPTIME=3000
DT = 1

; Reactions

; RXN1: PDS  --> PMS

; Reaction Constants

k1 = 0.01

;Reaction

INIT PDS = 0.93 ; M
INIT PMS = 0  ; M

RXN1= K1*PDS

d/dt(PDS) = -RXN1
d/dt(PMS) =  RXN1
```

Entry	T (C)	k1 fitting	RMS
1	40	0.00281	0.01102
2	50	0.00961	0.01949
3	60	0.02822	0.04840



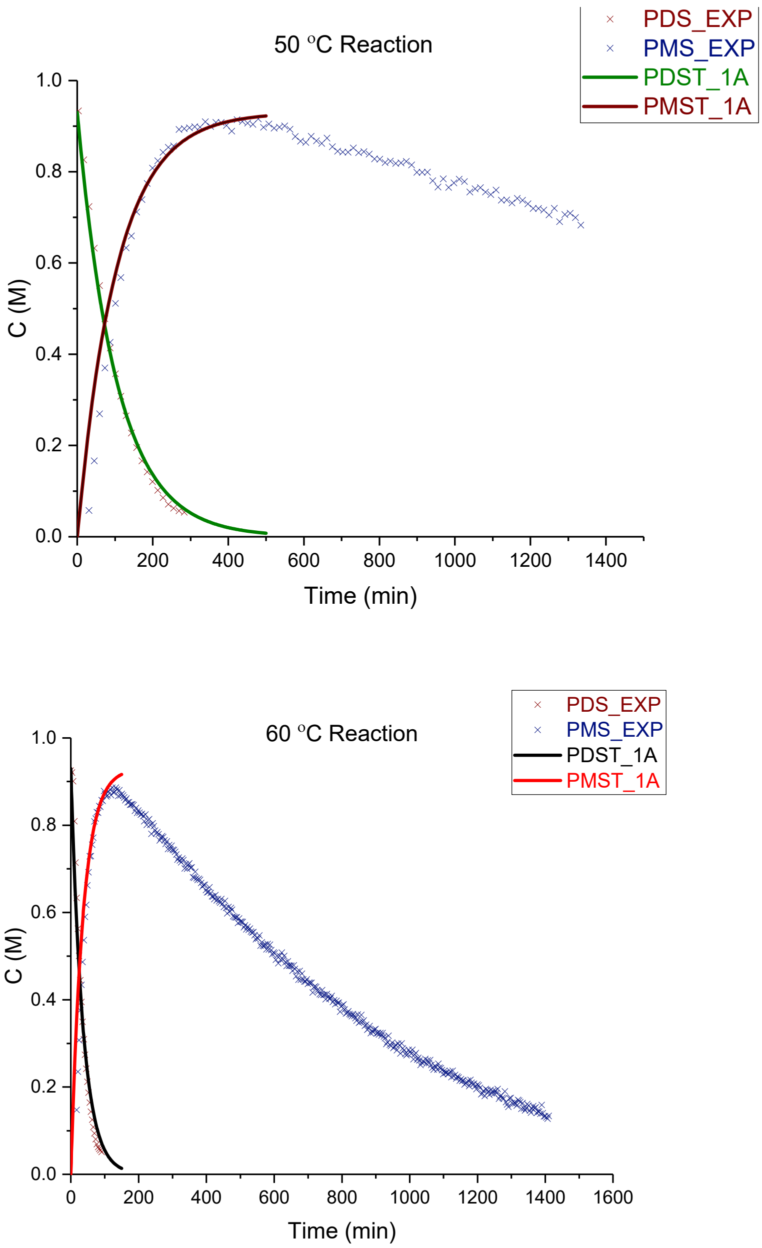
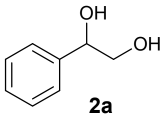


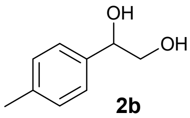
Figure S2. Fitting of pseudo first order kinetics to the decomposition of ammonium PDS in H_2SO_4 (Table 2).

Characterisation data for diols



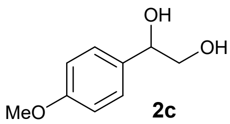
2a

*1-Phenyl-1,2-ethanediol (**2a**)*. General procedure followed using styrene (**1a**) (269 µL, 2.35 mmol) and 0.94 M PMS (5 mL, 4.70 mmol, 2 equiv.). Pale yellow crystals (261.1 mg, 1.89 mmol, 80%). mp 61.3 – 64.2 °C. δ_H 7.41 – 7.19 (5 H, m), 4.76 (1 H, dd, *J* = 8.3, 2.9 Hz, 1H), 4.46 (2 H, br s), 3.66 (1 H, dd, *J* = 11.6, 2.9), 3.60 (1 H, dd, *J* = 11.6, 8.3). δ_C 140.6, 128.5, 127.9, 126.2, 74.8, 68.0. ν_{max}/cm^{-1} 3202. Data in concordance with literature.[4]



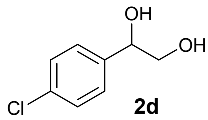
2b

*1-(4-Methylphenyl)ethane-1,2-diol (**2b**)*. General procedure followed using 4-methylstyrene (**1b**) (310 µL, 2.35 mmol) and 0.94 *M* PMS (5 mL, 4.70 mmol, 2 equiv.). Off-white crystalline flakes (376.9 mg, 2.48 mmol, 91%). mp 75.0 – 76.5 °C. δ_H 7.26 (2 H, d, *J* = 8.1), 7.18 (2 H, d, *J* = 8.1), 4.80 (1 H, dd, *J* = 8.1, 3.7), 3.75 (1 H, dd, *J* = 11.2, 3.7), 3.66 (1 H, dd, *J* = 11.2, 8.1), 2.35 (3 H, s), 1.59 (2 H, s). δ_C 137.6, 129.2, 126.1, 74.6, 68.0, 21.1. ν_{max}/cm^{-1} 3247 and 1073. Data in concordance with literature.[5]



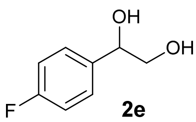
2c

*1-(4-Methoxyphenyl)ethane-1,2-diol (**2c**)*. General procedure followed using 4-methoxystyrene (**1c**) (313 µL, 2.35 mmol) and 0.94 *M* PMS (5 mL, 4.70 mmol, 2 equiv.). Brown crystalline solid (350.3 mg, 2.08 mmol, 87%). mp 72.6 – 78.4 °C. δ_H 7.29 (2 H, d, *J* = 8.6), 6.89 (2 H, d, *J* = 8.6), 4.77 (1 H, dd, *J* = 8.1, 3.5), 3.80 (3 H, s), 3.72 (1 H, dd, *J* = 11.3, 3.5), 3.65 (1 H, dd, *J* = 11.3, 8.1), 2.63 (1 H, s), 2.25 (1 H, s). δ_C 159.2, 132.8, 127.4, 113.9, 74.3, 68.0, 55.3. ν_{max}/cm^{-1} 3295 and 1080. Data in concordance with literature. [5]



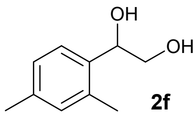
2d

*1-(4-Chlorophenyl)ethane-1,2-diol (**2d**)*. General procedure followed using 4-chlorostyrene (**1d**) (282 µL, 2.35 mmol) and 0.94 *M* PMS (5 mL, 4.70 mmol, 2 equiv.). Yellow crystals (292.8 mg, 1.70 mmol, 72%). mp 71.3 – 74.0 °C. δ_H 7.34 – 7.24 (4 H, m), 4.77 (1 H, dd, *J* = 8.3, 3.3), 3.71 (1 H, dd, *J* = 11.4, 3.3), 3.58 (1 H, dd, *J* = 11.4, 8.3), 3.04 (2 H, s). δ_C 139.0, 133.9, 128.8, 127.6, 74.1, 68.0. ν_{max}/cm^{-1} 3246 and 1069. Data in concordance with literature.[6]



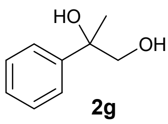
2e

*1-(4-Fluorophenyl)ethane-1,2-diol (**2e**)*. General procedure followed using 4-fluorostyrene (**1e**) (280 µL, 2.35 mmol) and 0.94 *M* PMS (5 mL, 4.70 mmol, 2 equiv.). Off-white crystals (293.9 mg, 1.88 mmol, 71%). mp 48.9 – 51.7 °C. δ_H 7.38 – 7.28 (2 H, m), 7.04 (2 H, t, J = 8.7), 4.80 (1 H, dd, J = 8.2, 3.4), 3.72 (1 H, dd, J = 11.3, 3.4), 3.61 (1 H, dd, J = 11.3, 8.2), 2.45 (2 H, s). δ_C 162.4 (d, J_{C-F} = 246.1), 136.2 (d, J_{C-F} = 2.1), 127.8 (d, J_{C-F} = 8.0), 115.3 (d, J_{C-F} = 21.3), 74.1, 67.8. ν_{max}/cm^{-1} 3206 and 1078. Data in concordance with literature.[5]



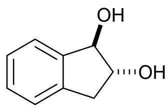
2f

*1-(2,4-Dimethylphenyl)ethane-1,2-diol (**2f**)*. General procedure followed using 2,4-dimethylstyrene (**1f**) (343 µL, 2.35 mmol) and 0.94 *M* PMS (5 mL, 4.70 mmol, 2 equiv.). Orange solid (326.5 mg, 1.96 mmol, 84%). mp 76.5 – 80.3 °C. δ_H 7.36 (1 H, t, J = 7.3), 7.05 (1 H, t, J = 7.3), 6.97 (1 H, d, J = 5.0), 5.03 (1 H, m), 3.70 (1 H, m), 3.60 (1 H, m), 2.47 (2 H, br s), 2.31 (6 H, m). δ_C 137.1, 135.6, 134.7, 131.2, 127.0, 125.8, 71.5, 67.0, 21.1, 18.93. ν_{max}/cm^{-1} 3214 and 1086. Data in concordance with literature. [5]



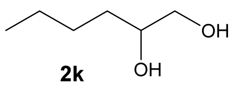
2g

*2-Phenyl-1,2-propanediol (**2g**)*. General procedure followed using α-methylstyrene (**1g**) (306 µL, 2.35 mmol) and 0.94 *M* PMS (5 mL, 4.70 mmol, 2 equiv.). Colourless oil (300.4 mg, 1.97 mmol, 84%). δ_H 7.27 (2 H, d, J = 7.2), 7.19 (2 H, t, J = 7.2), 7.11 (1 H, t, J = 7.2), 3.71 (1 H, s), 3.51 (1 H, d, J = 11.3), 3.38 (1 H, d, J = 11.3), 1.32 (2 H, s). δ_C 145.1, 128.2, 126.9, 125.1, 74.9, 70.6, 25.8. ν_{max}/cm^{-1} 3366. Data in concordance with literature.[7]



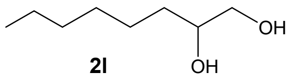
2h

*Trans-1,2-indanediol (**2h**)*. General procedure followed using indene (**1h**) (238 µL, 2.35 mmol) and 0.81 *M* PMS (5 mL, 4.05 mmol, 2 equiv.). Orange solid (198.7 mg, 1.32 mmol, 65%). mp 145.1 – 149.5 °C. δ_H (DMSO-d_6) 7.28-7.14 (4 H, m), 5.36 (1 H, d, J = 6.1), 5.14 (1 H, d, J = 4.8), 4.69 (1 H, t, J = 5.4), 4.08 (1 H, m), 3.07 (1 H, dd, J = 15.5, 7.0), 2.59 (1 H, dd, J = 15.5, 7.0). δ_C (DMSO-d_6) 144.1, 139.4, 127.4, 126.4, 124.5, 124.2, 80.6, 79.9, 38.0. ν_{max}/cm^{-1} 3216 and 1072. Data in in concordance with literature.[8]



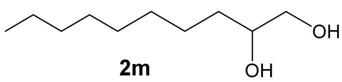
2k

*1,2-Hexanediol (**2k**)*. General procedure followed using 1-hexene (**1k**) (292 µL, 2.35 mmol) and 0.94 *M* PMS (5 mL, 4.70 mmol, 2 equiv.). Pale yellow oil (157.0 mg, 1.33 mmol, 67%). δ_H 3.81 – 3.55 (2 H, m), 3.43 (1 H, dd, J = 11.0, 7.8), 2.61 (2 H, s), 1.44-1.32 (6 H, m), 0.91 (3 H, t, J = 6.8). δ_C 72.4, 66.7, 32.8, 27.7, 22.7, 13.9. ν_{max}/cm^{-1} 3339 and 1061. Data in concordance with literature.[9]



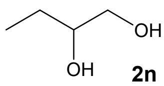
2l

*1,2-Octanediol (**2l**)*. General procedure followed using 1-octene (**1l**) (369 µL, 2.35 mmol) and 0.94 *M* PMS (5 mL, 4.70 mmol, 2 equiv.). Pale yellow oil (243.2 mg, 1.66 mmol, 85%). δ_H 3.89 – 3.53 (2 H, m), 3.43 (1 H, dd, *J* = 11.0, 7.8), 2.35 (2 H, s), 1.68 – 1.12 (10 H, m), 0.88 (3 H, t, *J* = 6.6). δ_C 72.4, 66.7, 33.1, 31.8, 29.3, 25.56, 22.6, 14.0. v_{max}/cm^{-1} 3347 and 1067. Data in concordance with literature.[7]



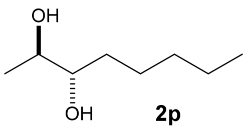
2m

*1,2-Decanediol (**2m**)*. General procedure followed using 1-decene (**1m**) (454 µL, 2.40 mmol) and 0.94 *M* PMS (5 mL, 4.7 mmol, 2 equiv.). Pale yellow oil (132.8 mg, 0.76 mmol, 32%). δ_H 3.82 – 3.35 (2 H, m), 2.16 (2 H, s), 1.26 (12 H, s), 0.87 (3 H, t, *J* = 6.6). δ_C 72.5, 66.7, 33.2, 32.0, 29.8, 29.7, 29.4, 25.7, 22.7, 14.1. v_{max}/cm^{-1} 3371. Data in concordance with literature.[10]



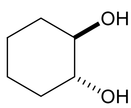
2n

*1,2-Dodecanediol (**2n**)*. General procedure followed using 1-dodecene (**1n**) (292.9 mg, 1.32 mmol) and 0.66 *M* PMS (5mL, 3.30 mmol, 2 equiv.). White solid (30.5 mg, 0.15 mmol, 11%). m.p. 49.2-51.8 °C. δ_H 3.75 – 3.59 (2 H, m), 3.48 – 3.37 (1 H, m), 2.42 (2 H, s), 1.34 (18 H, m), 0.87 (3 H, t, *J* = 6.7). δ_C 72.5, 67.0, 33.4, 32.1, 29.8, 29.8, 29.7, 29.5, 25.7, 22.8, 14.3. v_{max}/cm^{-1} 3360. Data in concordance with literature.[11]



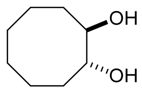
2p

*2,3-Octanediol (**2p**)*. General procedure followed using trans-2-octene (**1p**) (328 µL, 2.10 mmol) and 0.84 *M* H_2SO_5 (5 mL, 4.20 mmol, 2 equiv.). Colourless oil (222.0 mg, 1,52 mmol, 63%). δ_H 4.07 (2 H, s), 3.68 (1 H, qd, *J* = 6.4, 2.9), 3.52 (1 H, ddd, *J* = 7.8, 4.9, 2.9), 1.26 (8 H, m), 1.04 (3 H, d, *J* = 6.4), 0.82 (3 H, t, *J* = 6.8). δ_C 74.9, 70.4, 31.9, 31.7, 25.8, 22.5, 16.1, 13.9. v_{max}/cm^{-1} 3350, 2931, 2859 and 1057. Data in concordance with literature.[12]



2q

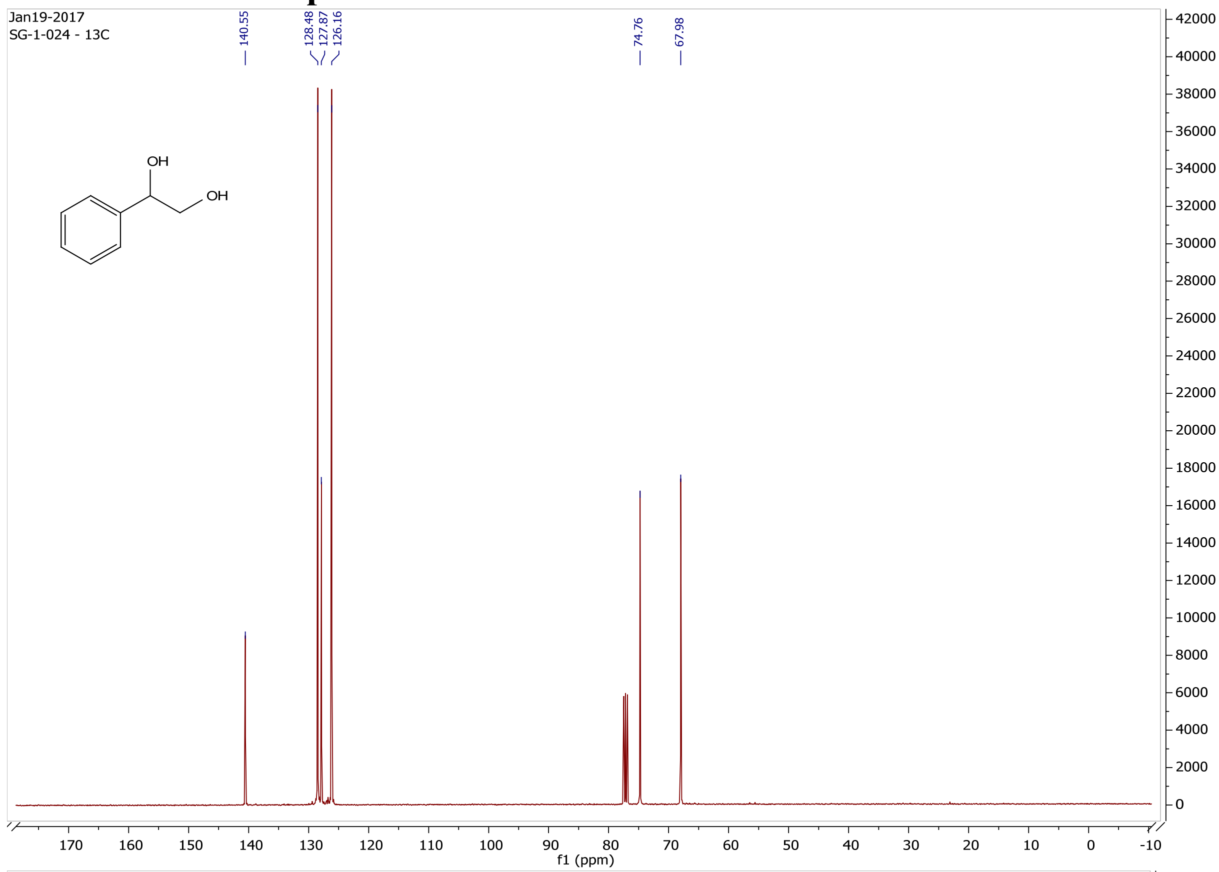
*Trans-1,2-cyclohexanediol (**2q**)*. General procedure followed using cyclohexene (**1q**) (213 µL, 2.10 mmol) and 0.84 *M* H_2SO_5 (5 mL, 4.20 mmol, 2 equiv.). White crystalline solid (146.8 mg, 1.26 mmol, 62%). mp 95.2 – 101.0 °C. δ_H 3.36 (2 H, s), 2.23 (2 H, br), 1.71 (4 H, s), 1.27 (4 H, m). δ_C (101 MHz, $CDCl_3$) 75.5, 32.9, 24.3. v_{max}/cm^{-1} 3284, 2931, 2858 and 1064. Data in concordance with literature.[13-15]

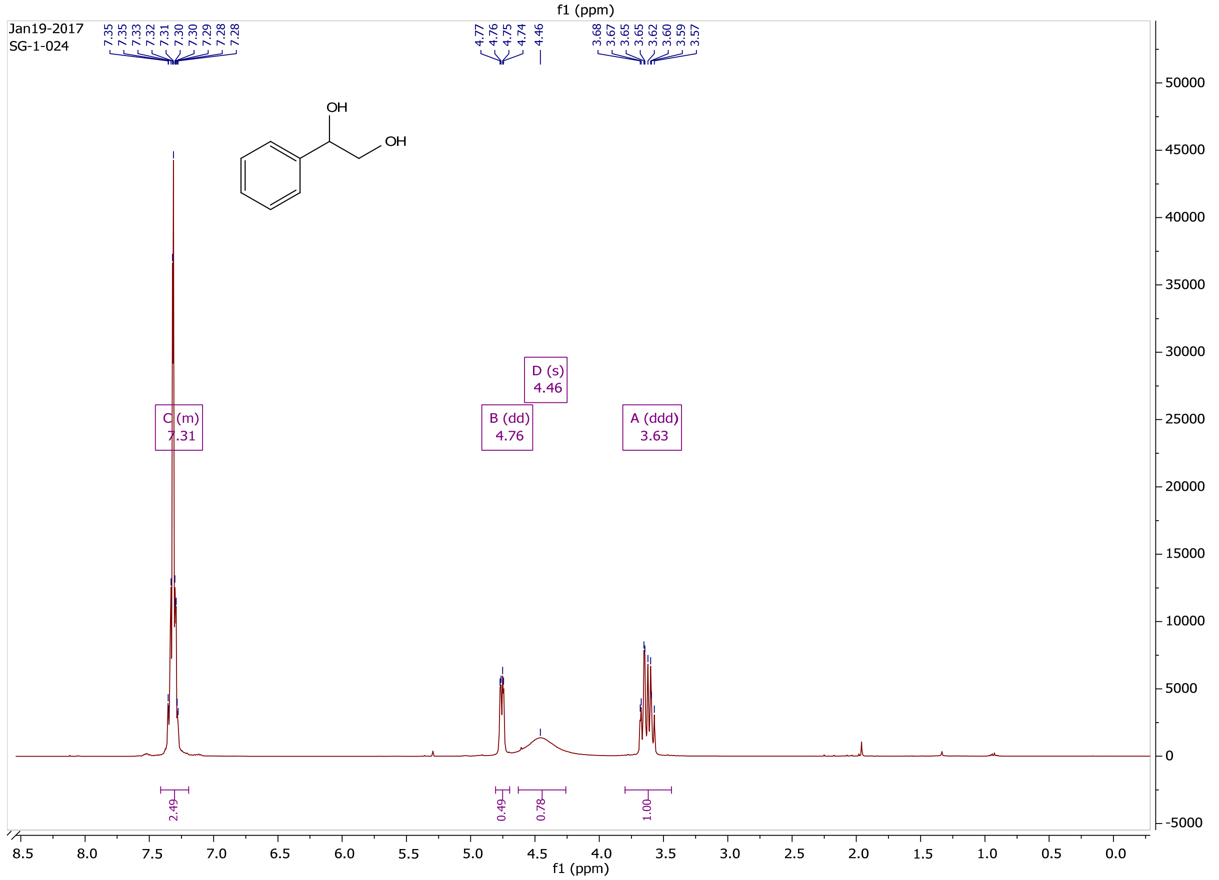


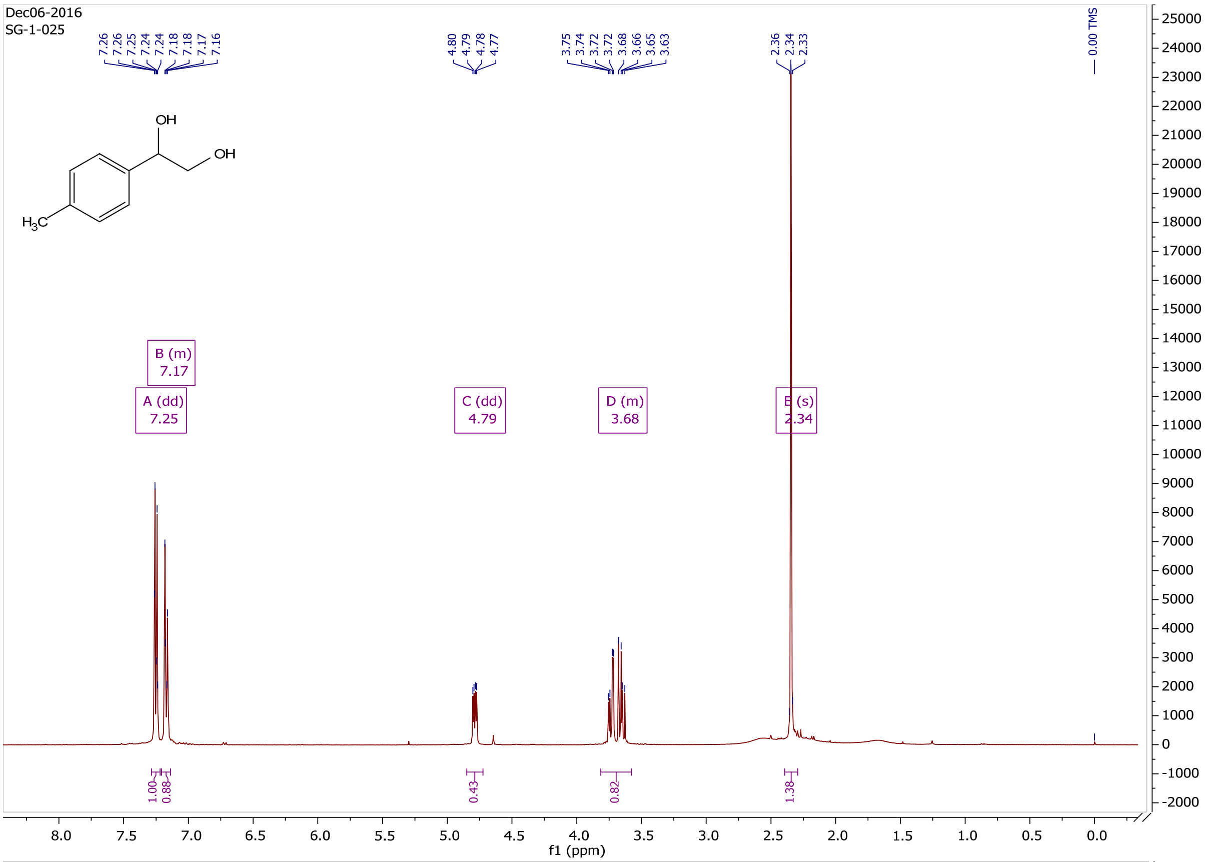
2r

*Trans-1,2-cyclooctanediol (**2r**)*. General procedure followed using cyclooctene (**1r**) (273 µL, 2.10 mmol) and 0.84 *M* H_2SO_5 (5 mL, 4.20 mmol, 2 equiv.). Purified by flash column chromatography on a silica gel column using a gradient of 25-50% (v/v) EtOAc in petroleum ether. Pale yellow oil (41.8 mg, 0.29 mmol, 14%). R_f 0.08 (1:1 EtOAc/pet. ether). δ_H 3.56 (2 H, d, *J* = 5.7), 3.22 (2 H, s), 1.89 – 1.76 (2 H, m), 1.72 – 1.60 (4 H, m), 1.60 – 1.48 (4 H, m), 1.49 – 1.41 (2 H, m). δ_C 76.2, 31.9, 26.2, 23.7. ν_{max}/cm^{-1} 3368, 2922, 2855 and 1033. Data in concordance with literature.[14,16]

[1]H and [13]C NMR Spectra



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SG-1-025
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C (dd) 4.79
D (m) 3.68
E (s) 2.34
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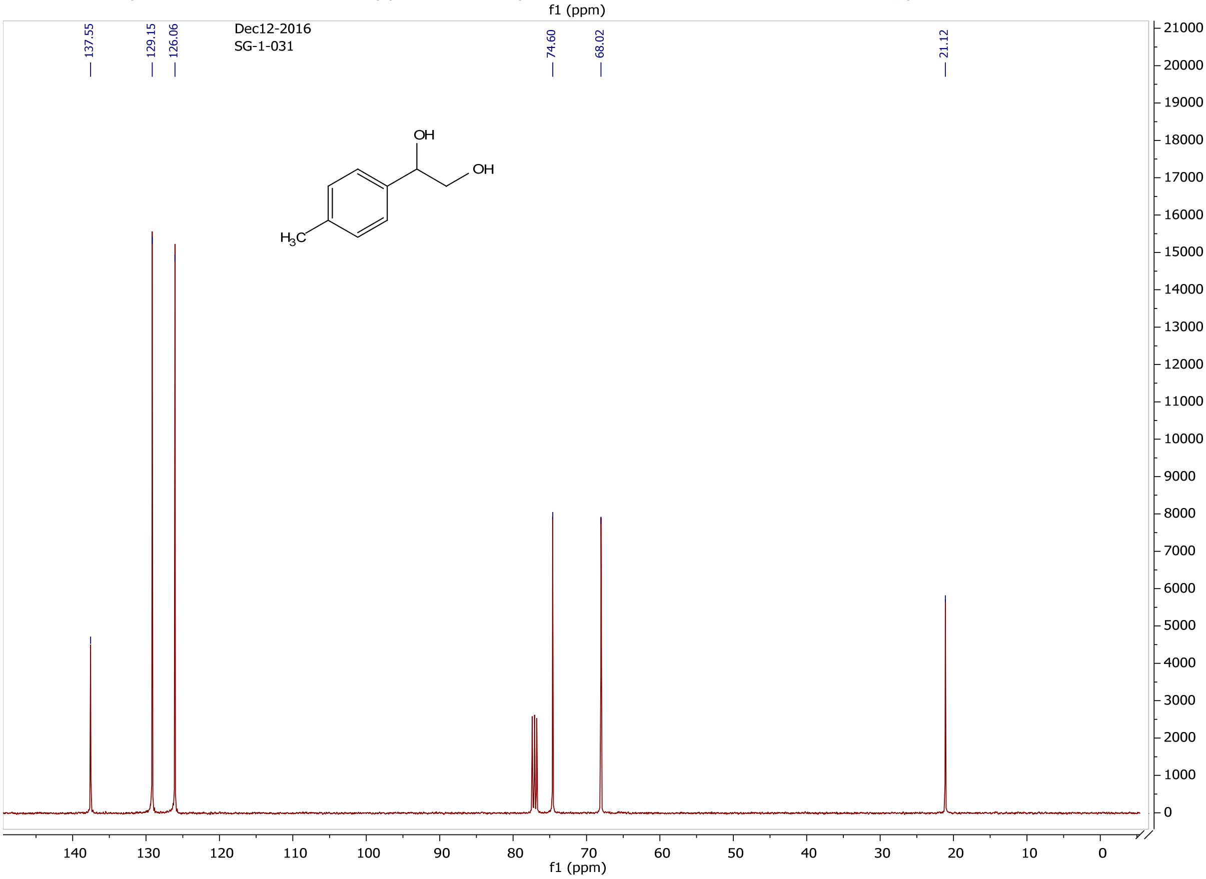
f1 (ppm)


Dec12-2016
SG-1-031
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126.06
74.60
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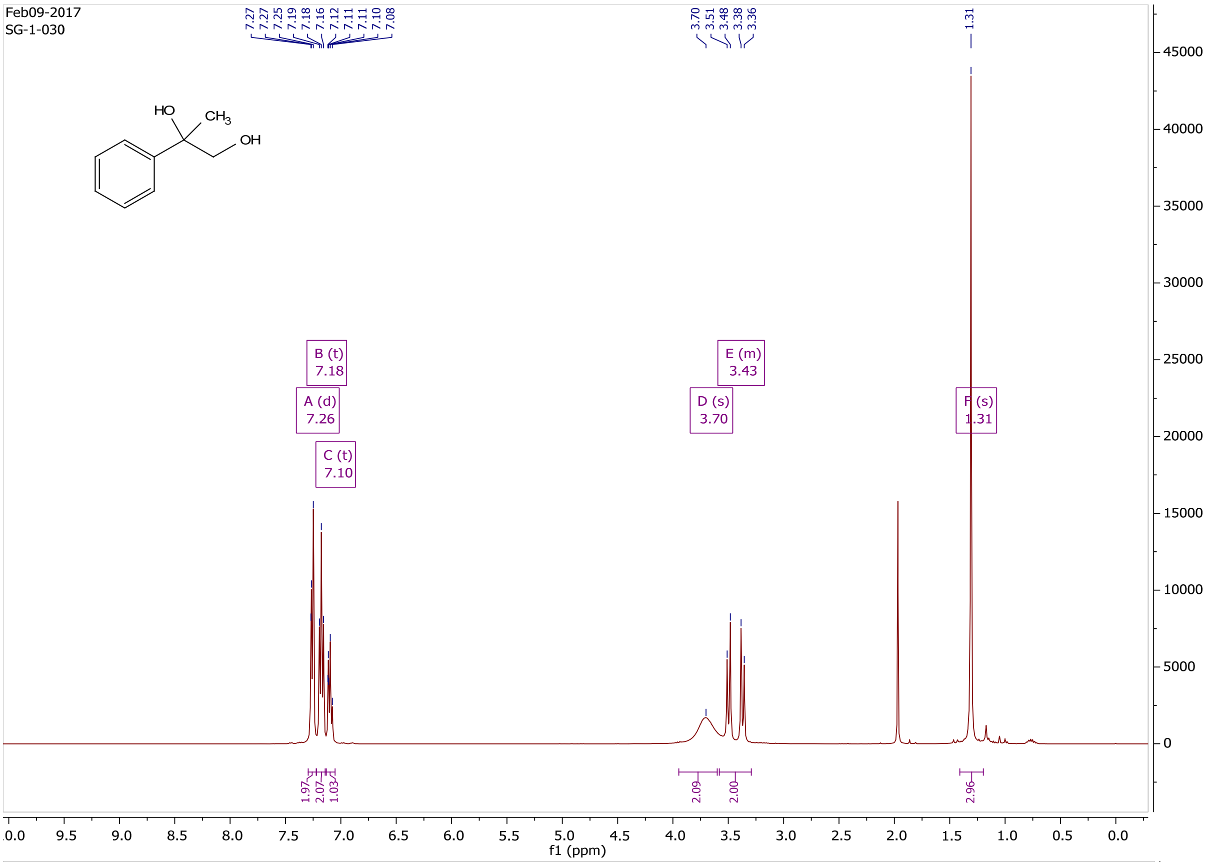
21.12
f1 (ppm)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OH
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7.27
7.25
7.19
7.18
7.16
7.12
7.11
7.11
7.10
7.08
3.70
3.51
3.48
3.38
3.36
1.31
B (t)
7.18
A (d)
7.26
C (t)
7.10
E (m)
3.43
D (s)
3.70
F (s)
1.31
1.97
2.07
1.03
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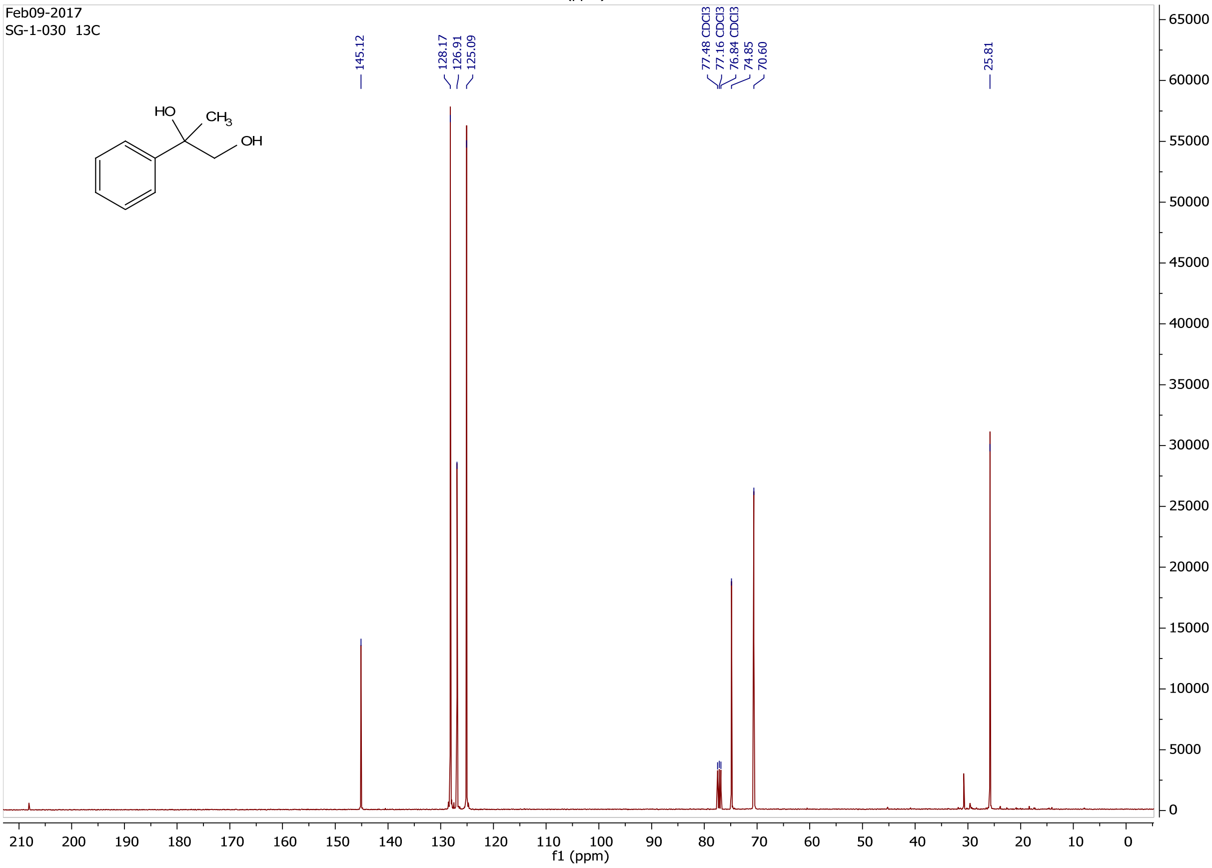
2.00
2.96
f1 (ppm)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OH
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125.09
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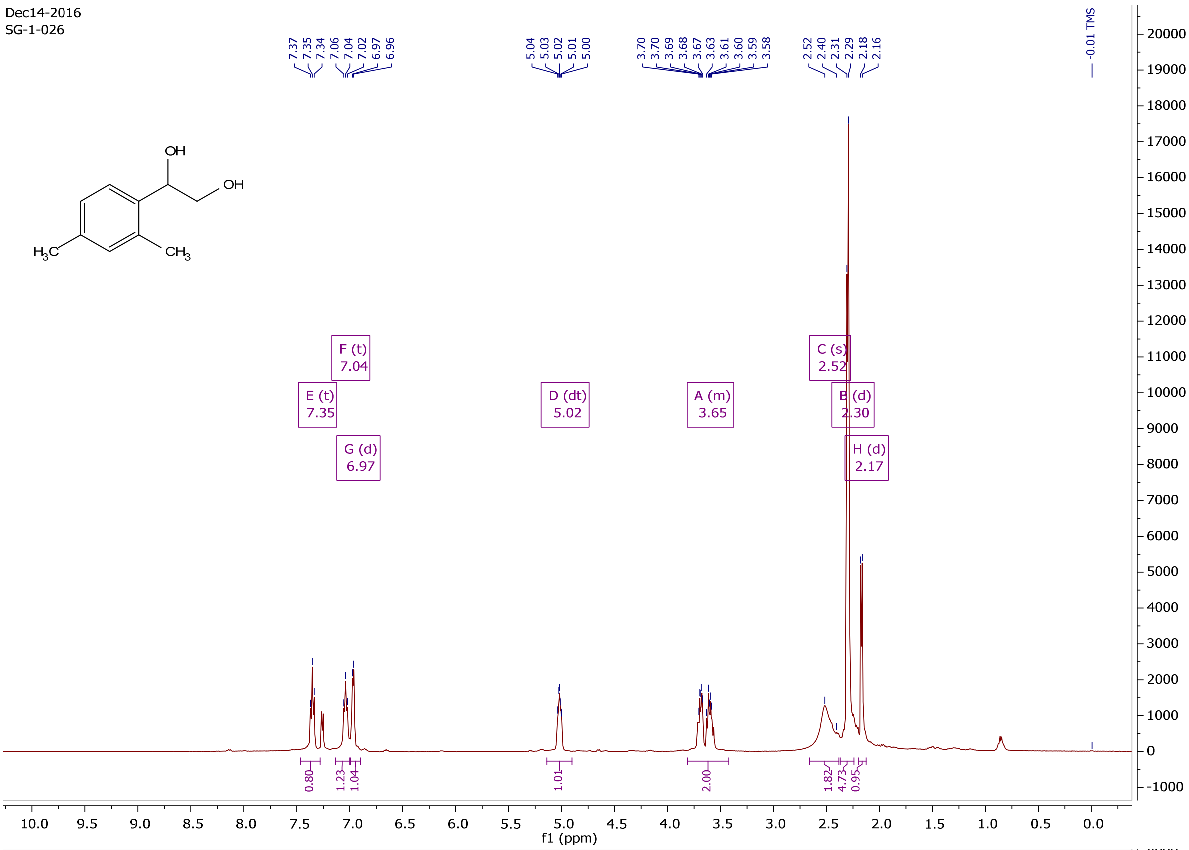
f1 (ppm)


Dec14-2016
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OH
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CH3
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F (t) 7.04
G (d) 6.97
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B (d) 2.30
H (d) 2.17
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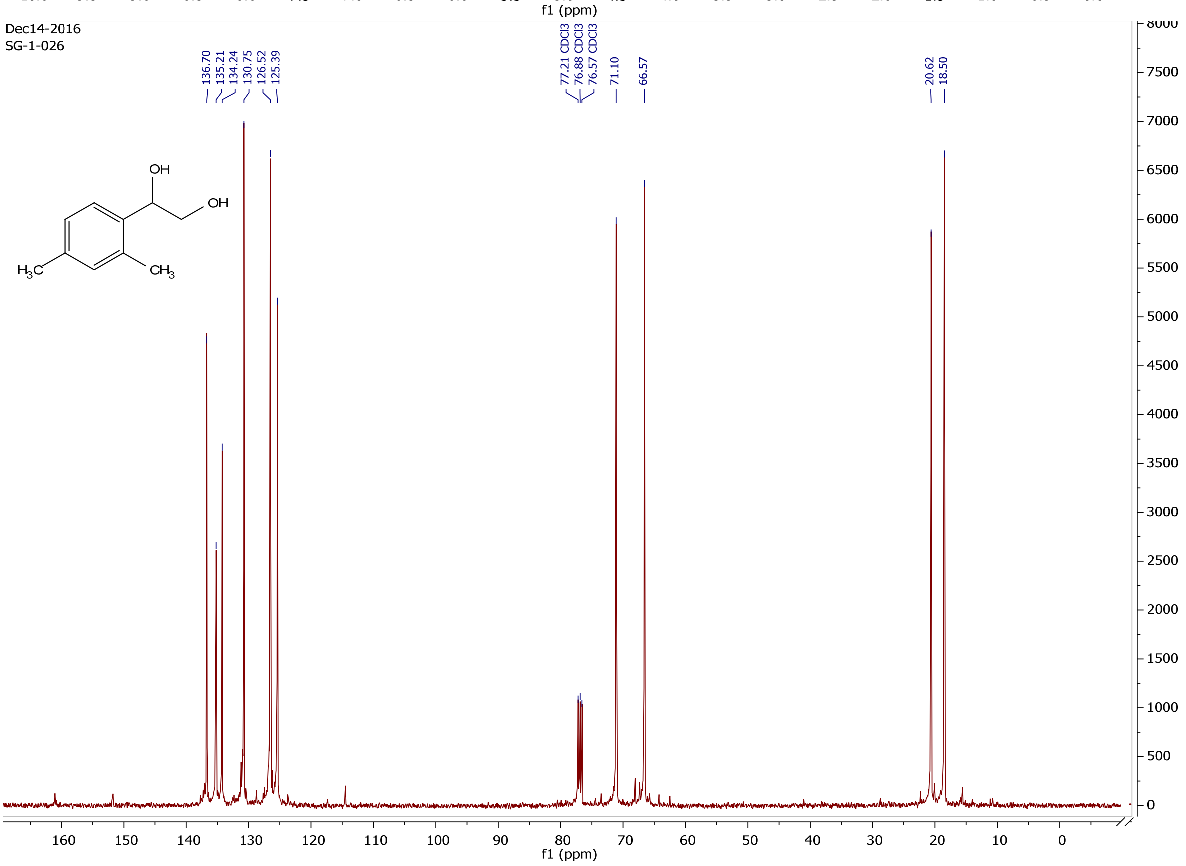
f1 (ppm)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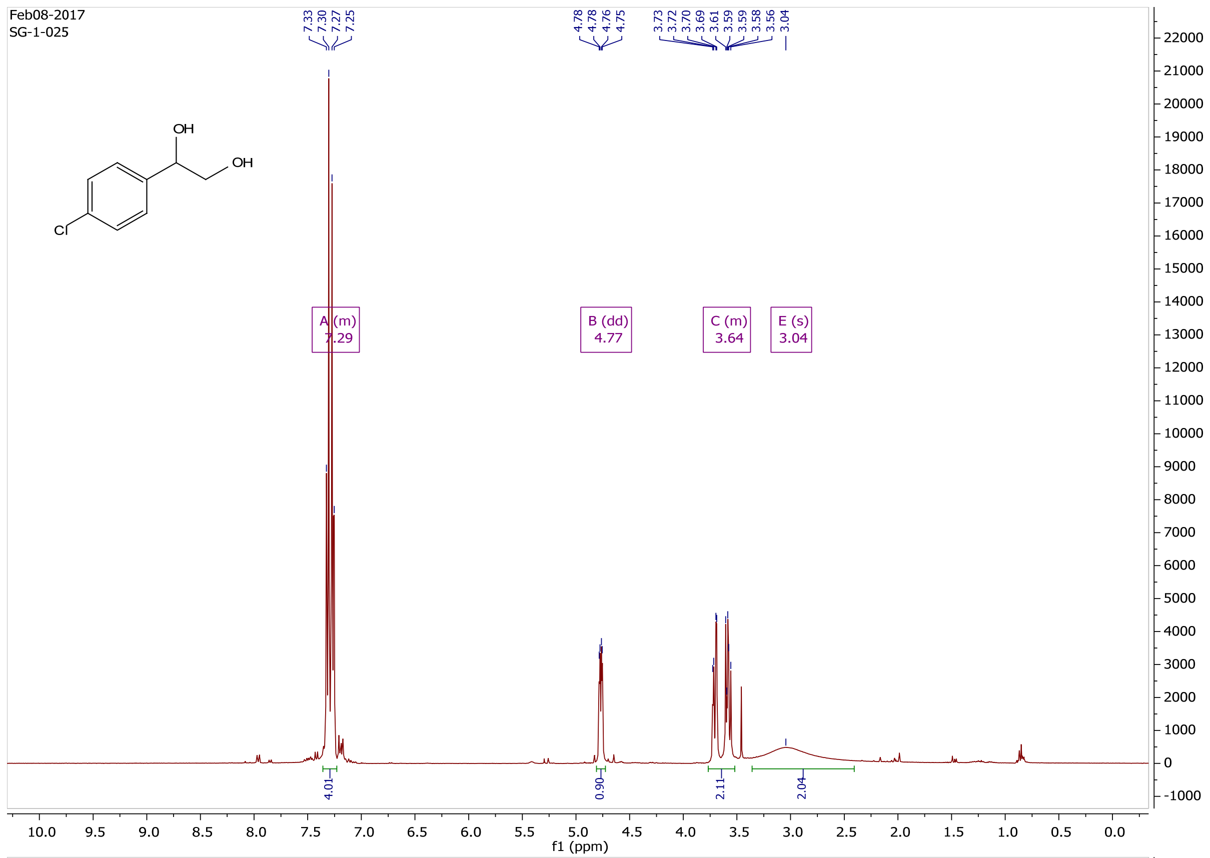
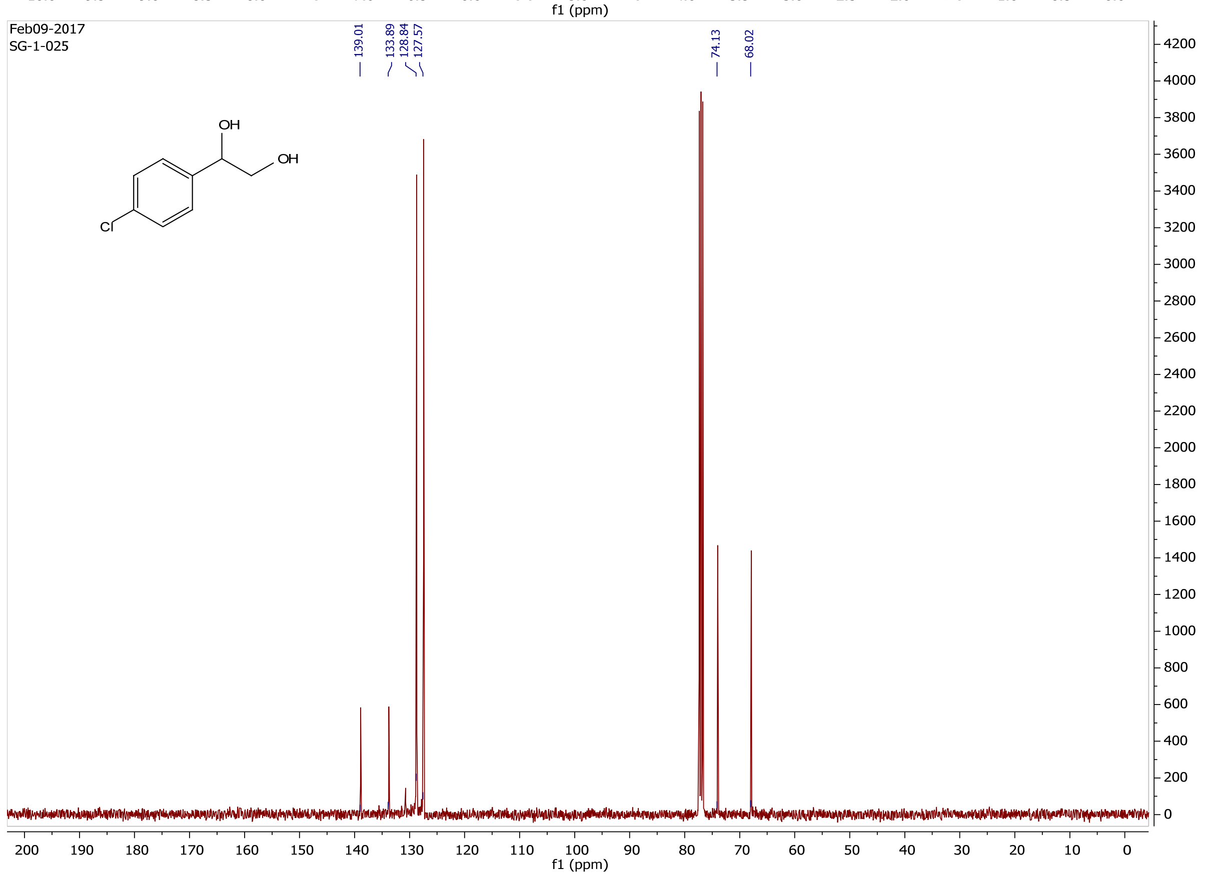
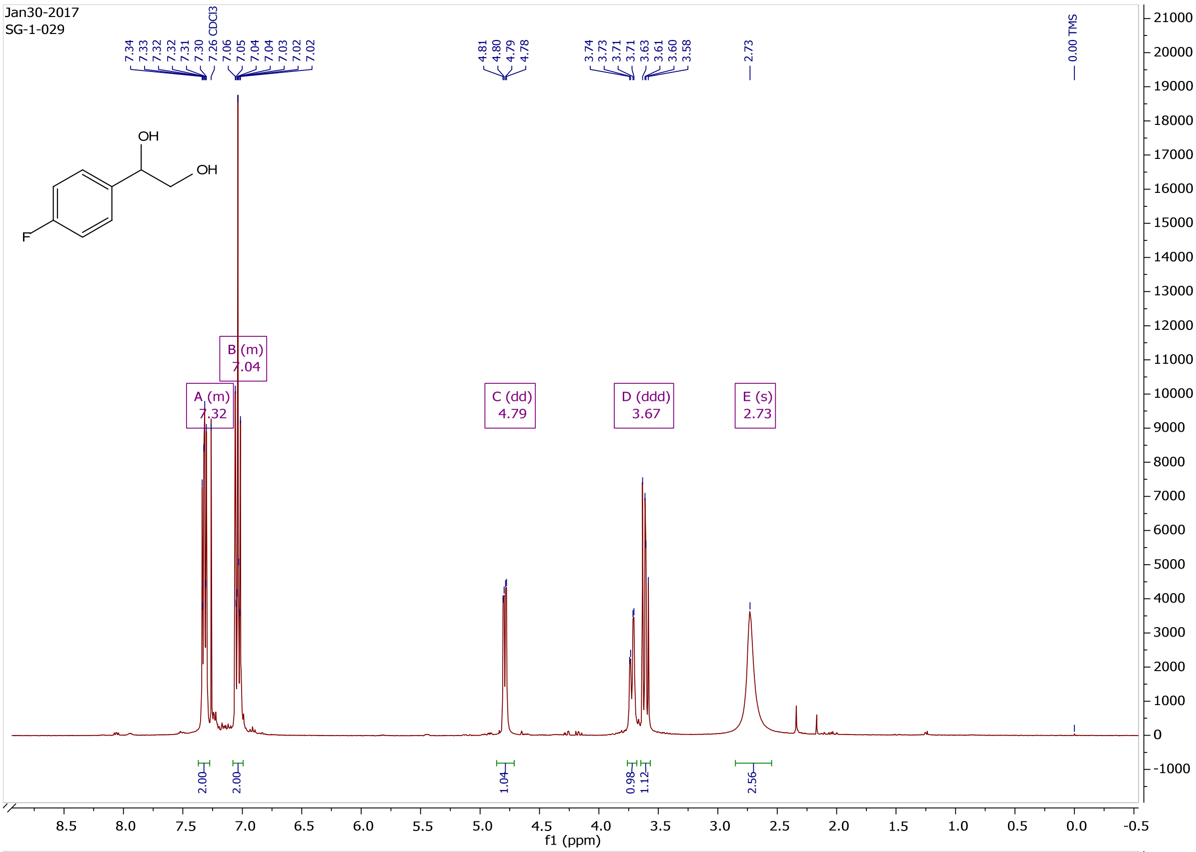
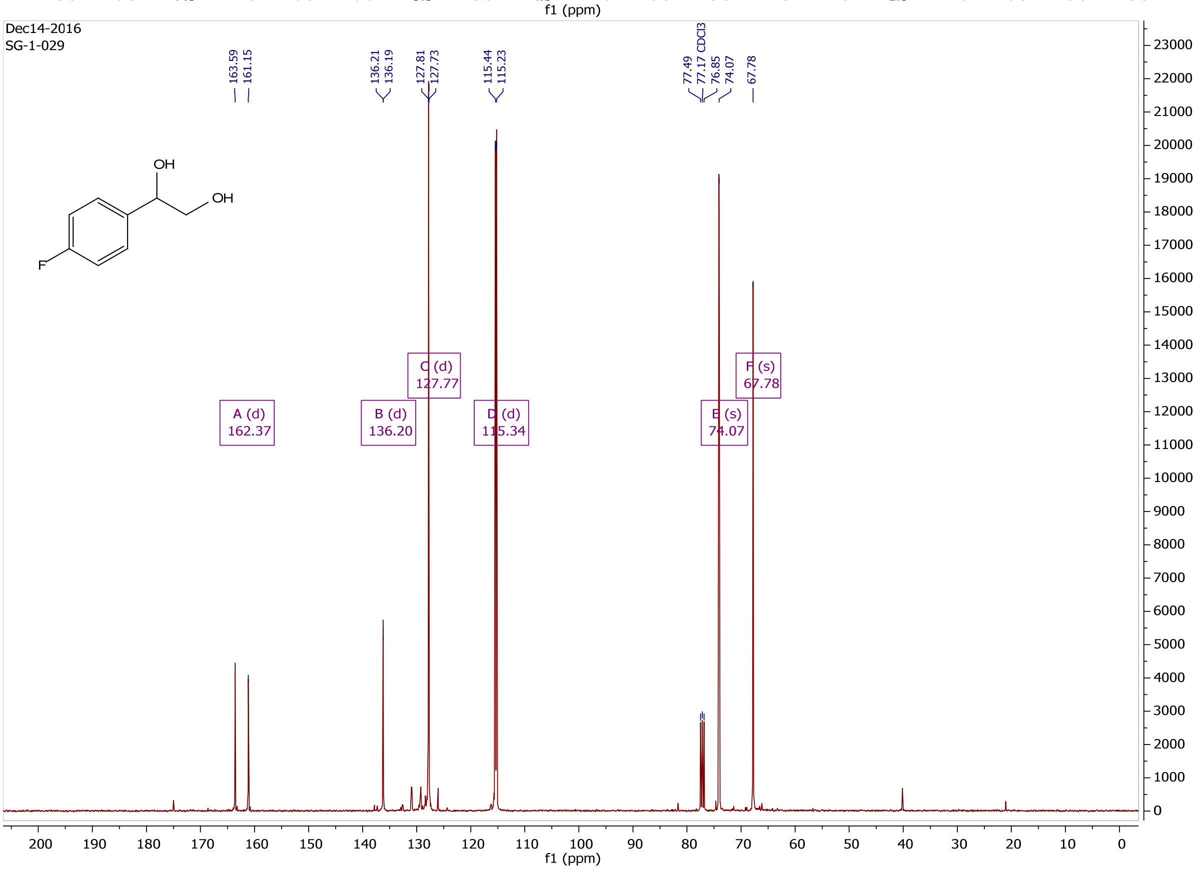
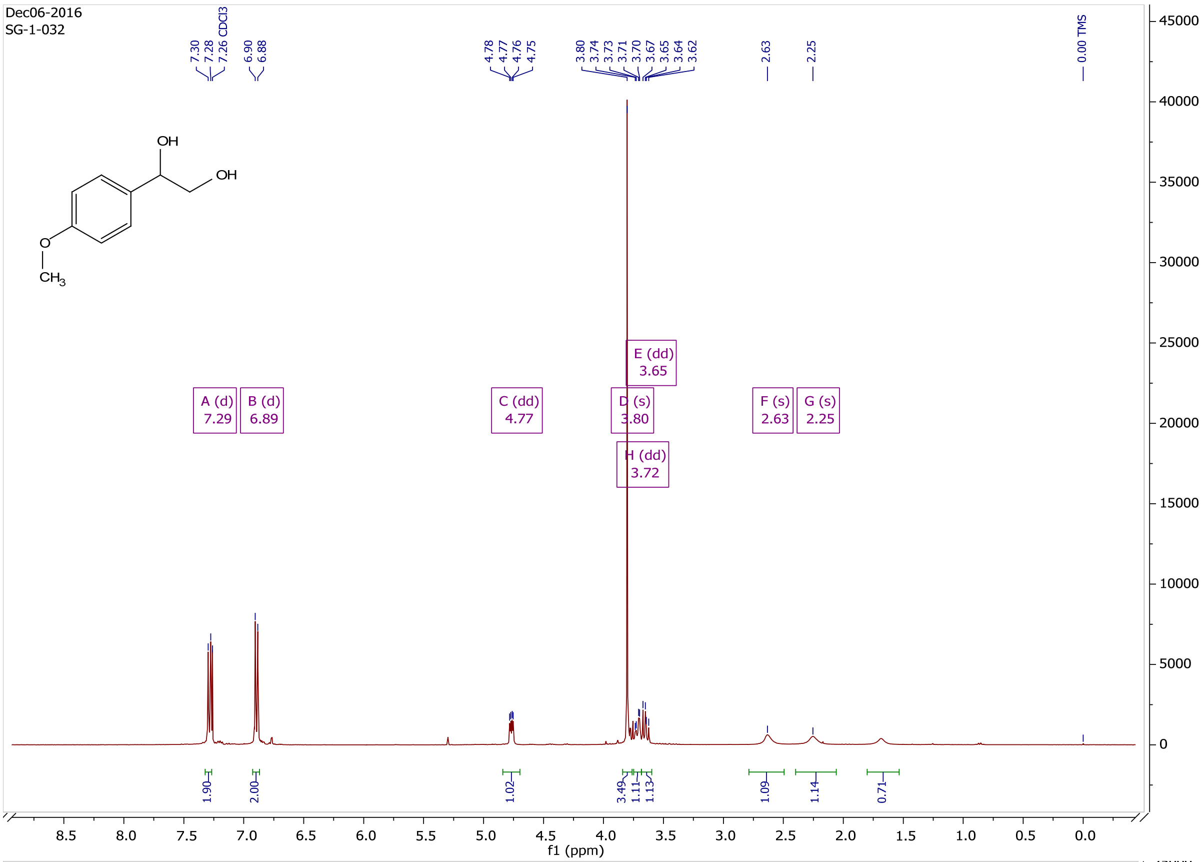
SG-1-026
136.70
135.21
134.24
130.75
126.52
125.39
77.21 CDCl3
76.88 CDCl3
76.57 CDCl3
71.10
66.57
20.62
18.50
OH
OH
H3C
CH3
f1 (ppm)

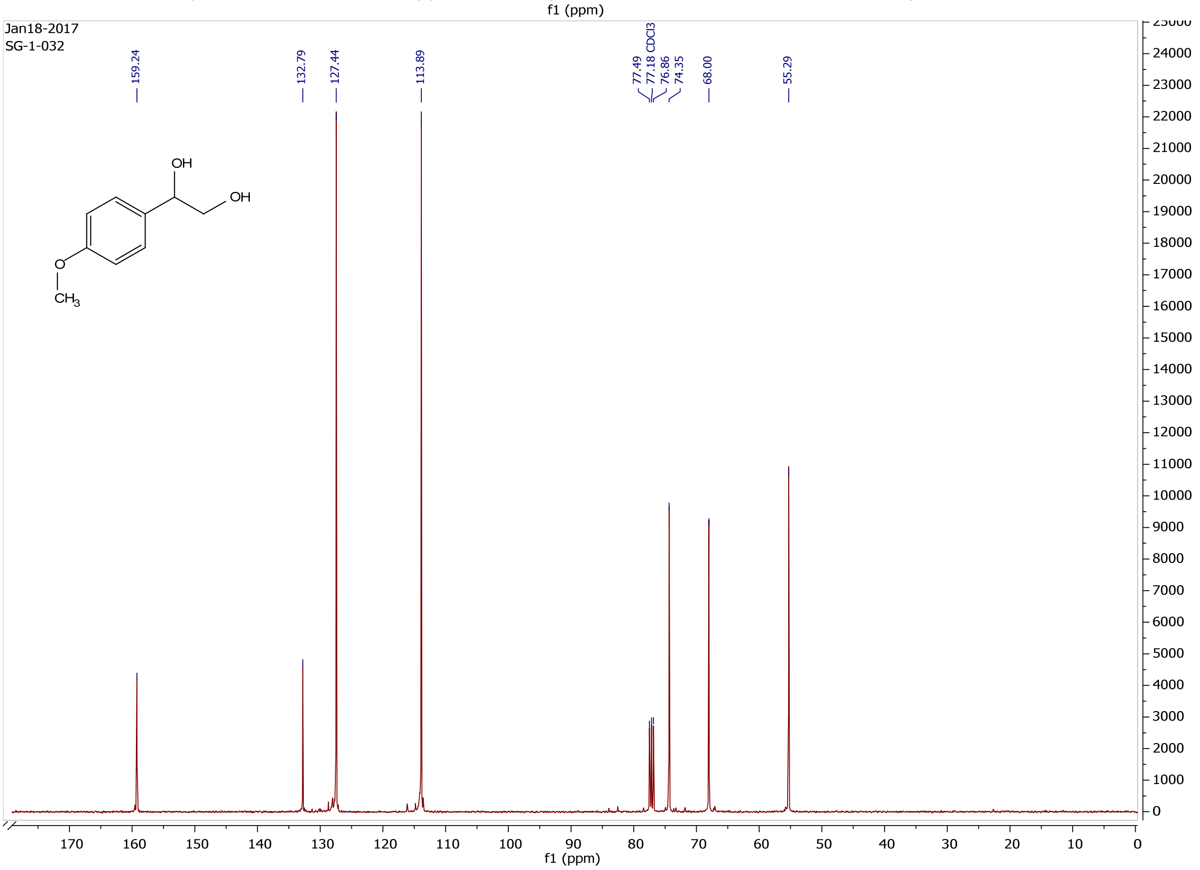

Feb08-2017
SG-1-025
OH
OH
Cl
7.33
7.30
7.27
7.25
4.78
4.78
4.76
4.75
3.73
3.72
3.70
3.69
3.61
3.59
3.59
3.58
3.56
3.04
A (m)
7.29
B (dd)
4.77
C (m)
3.64
E (s)
3.04
4.01
0.90
2.11
2.04
f1 (ppm)

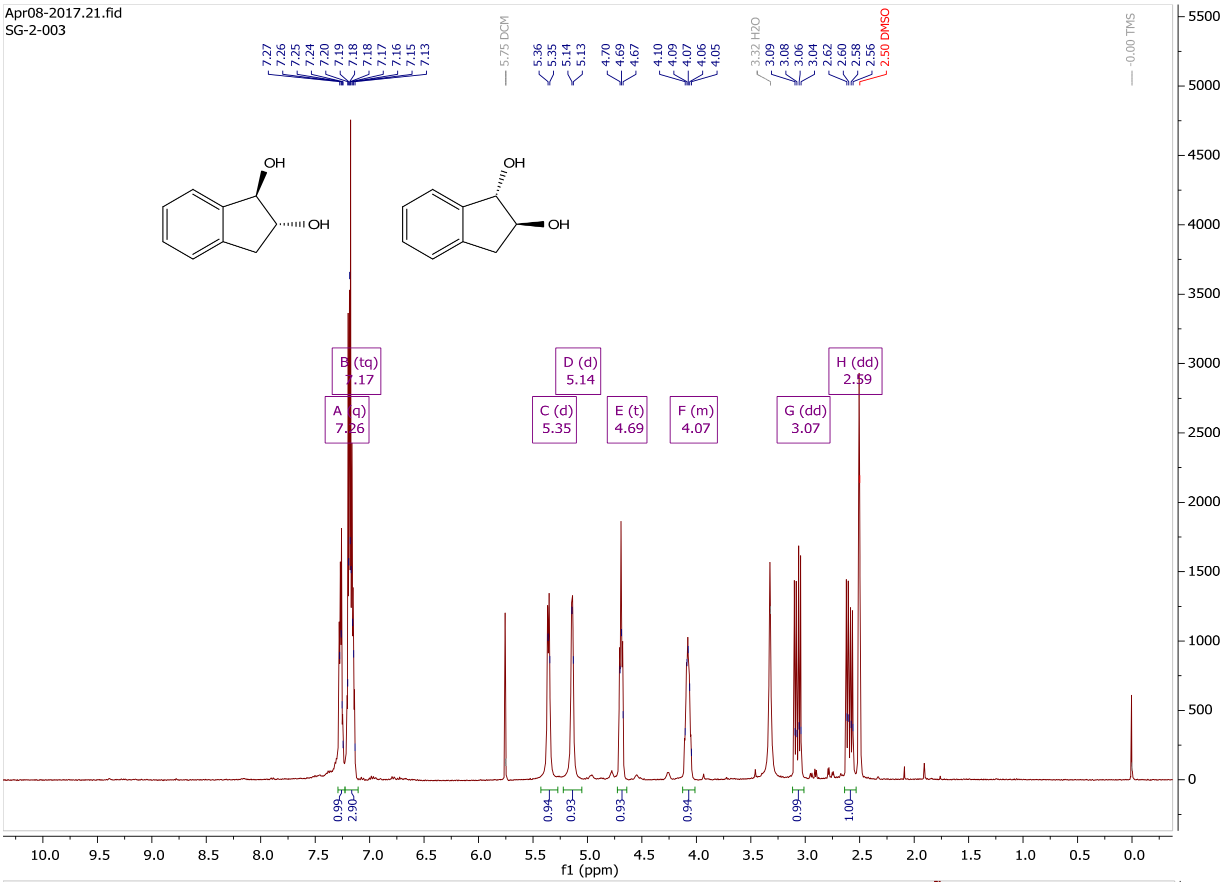

Feb09-2017
SG-1-025
OH
OH
Cl
139.01
133.89
128.84
127.57
74.13
68.02
f1 (ppm)


Jan30-2017
SG-1-029
A (m) 7.32
B (m) 7.04
C (dd) 4.79
D (ddd) 3.67
E (s) 2.73
f1 (ppm)

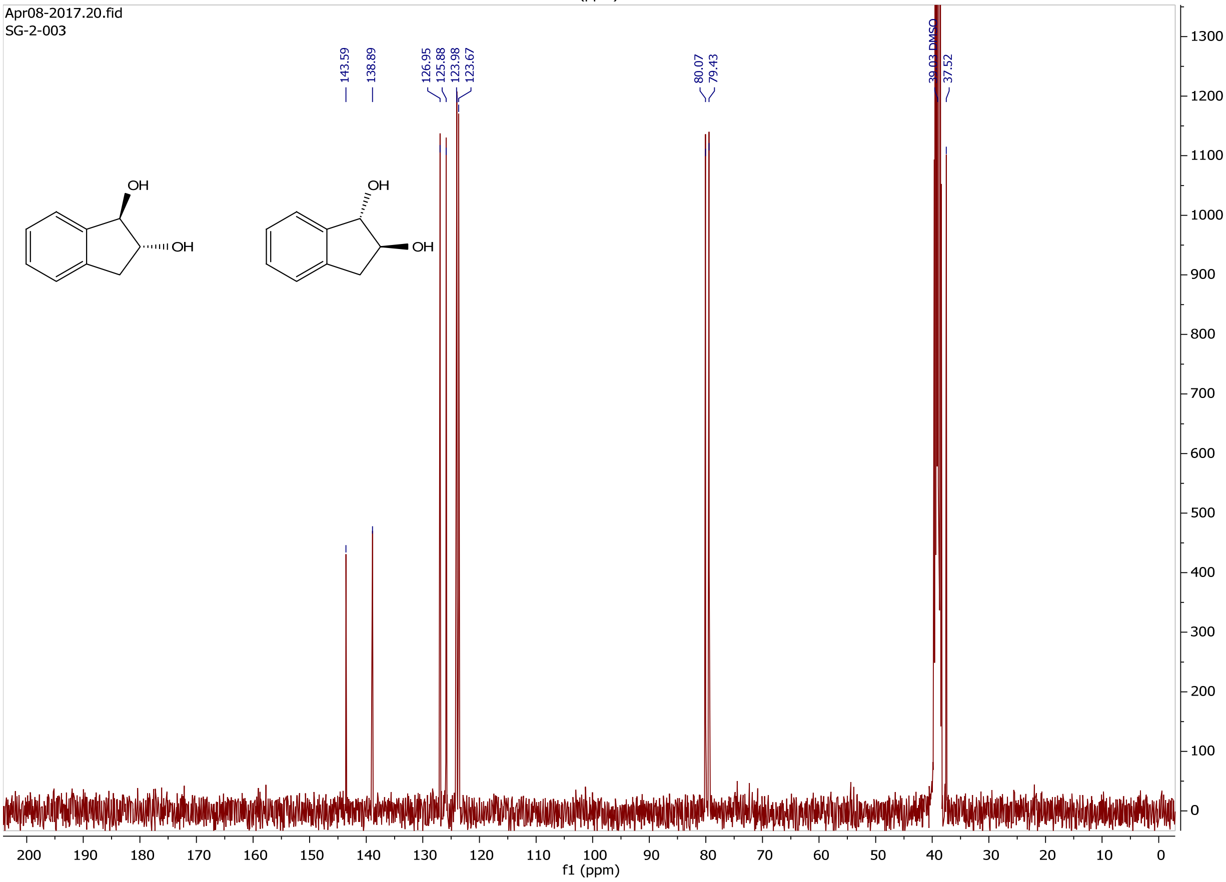

Dec14-2016
SG-1-029
A (d) 162.37
B (d) 136.20
C (d) 127.77
D (d) 115.34
E (s) 74.07
F (s) 67.78
f1 (ppm)


Dec06-2016
SG-1-032
7.30
7.28
7.26 CDCl3
6.90
6.88
4.78
4.77
4.76
4.75
3.80
3.74
3.73
3.71
3.70
3.67
3.65
3.64
3.62
2.63
2.25
0.00 TMS
OH
OH
O
CH3
A (d) 7.29
B (d) 6.89
C (dd) 4.77
D (s) 3.80
E (dd) 3.65
H (dd) 3.72
F (s) 2.63
G (s) 2.25
1.90
2.00
1.02
3.49
1.11
1.13
1.09
1.14
0.71
f1 (ppm)

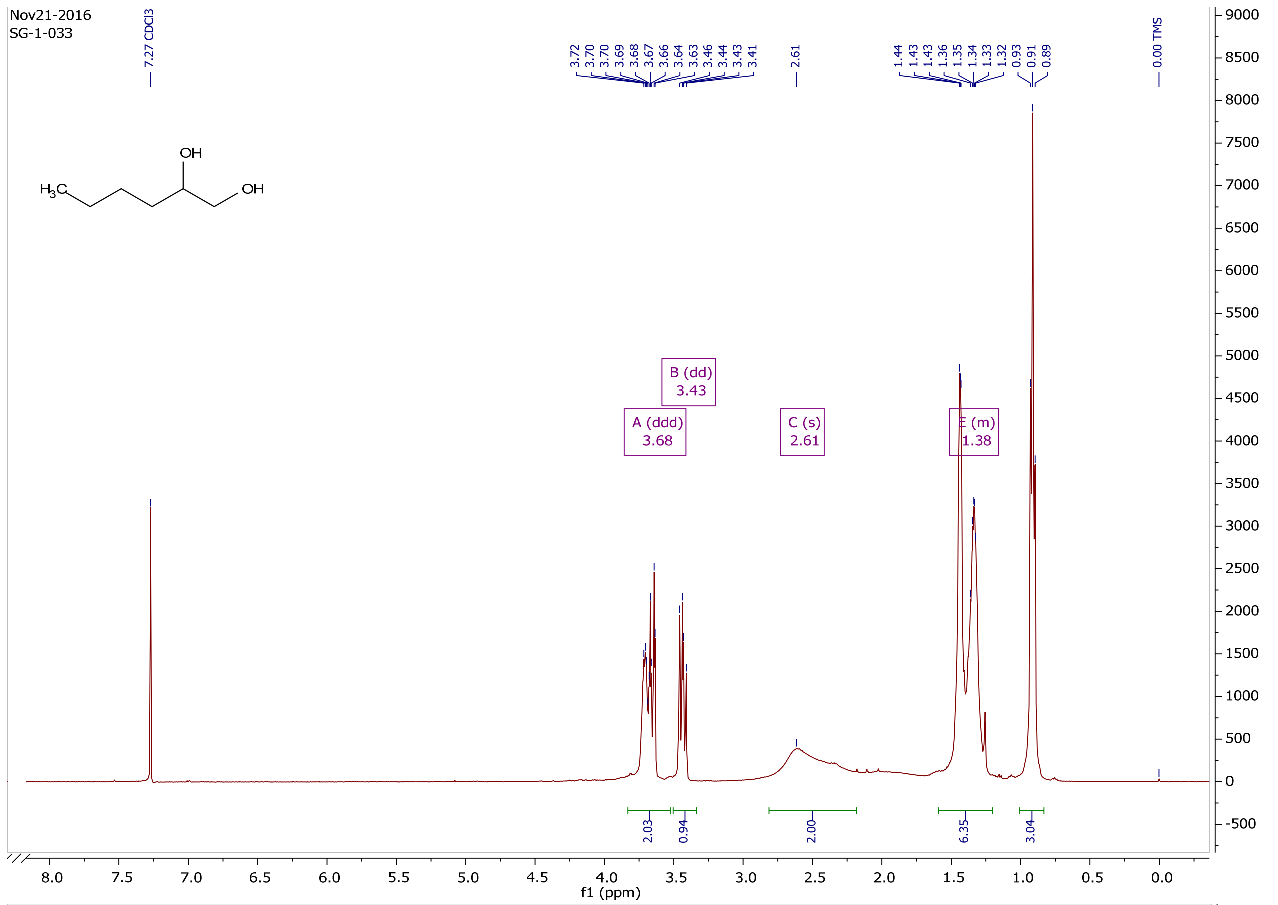

Jan18-2017
SG-1-032
159.24
132.79
127.44
113.89
77.49
77.18 CDCl3
76.86
74.35
68.00
55.29
OH
OH
O
CH3
f1 (ppm)

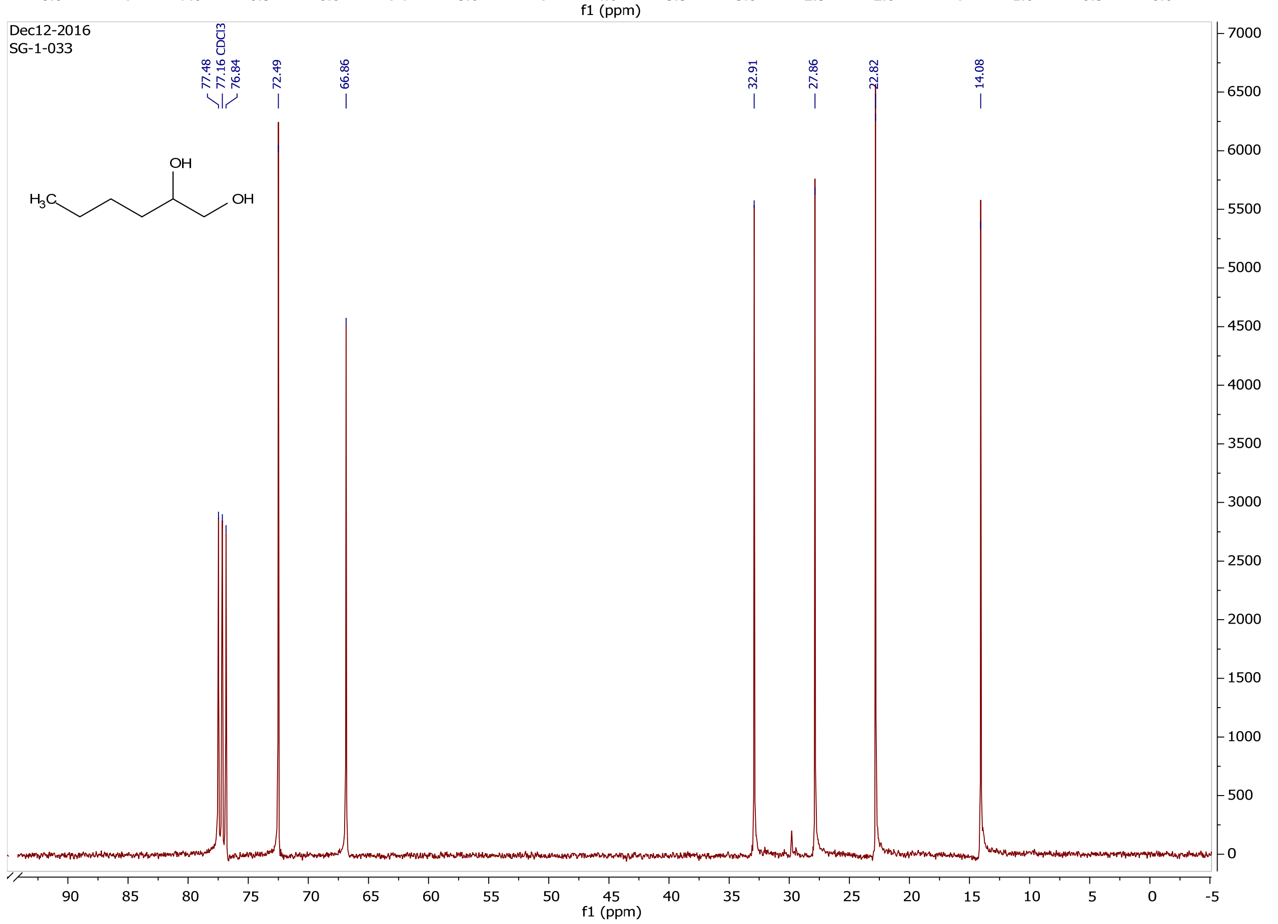


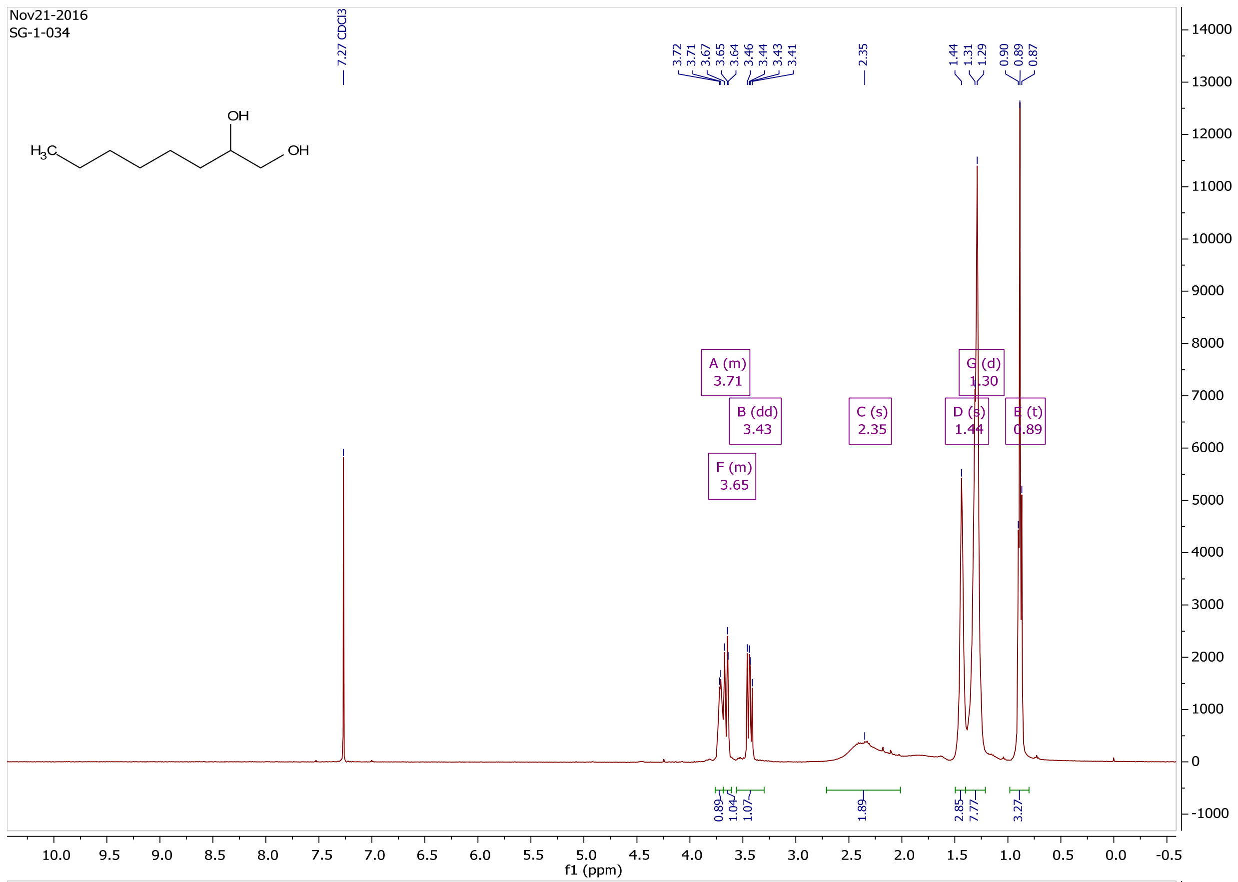
Apr08-2017.21.fid
SG-2-003
OH
OH
A (q) 7.26
B (tq) 7.17
C (d) 5.35
D (d) 5.14
E (t) 4.69
F (m) 4.07
G (dd) 3.07
H (dd) 2.59
5.75 DCM
3.32 H2O
2.50 DMSO
-0.00 TMS
f1 (ppm)

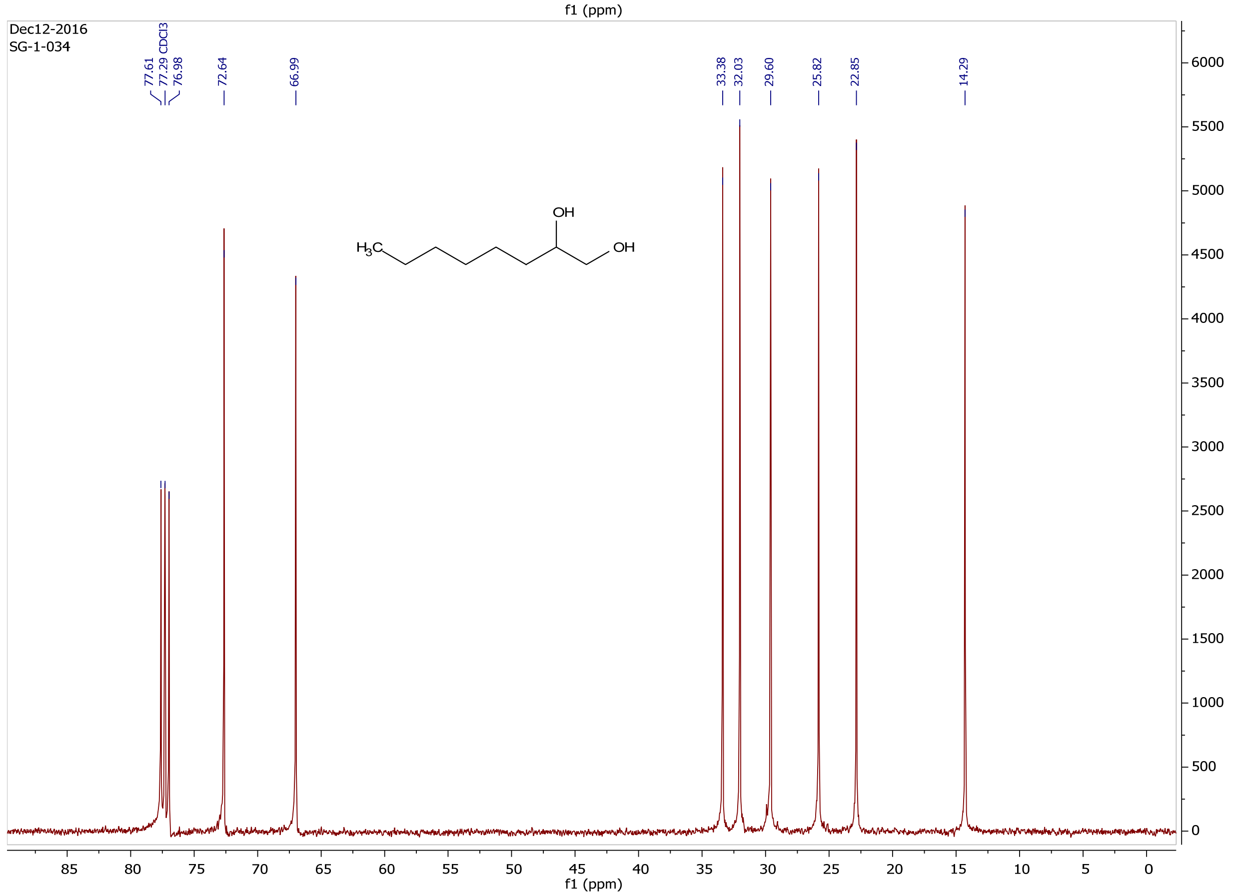


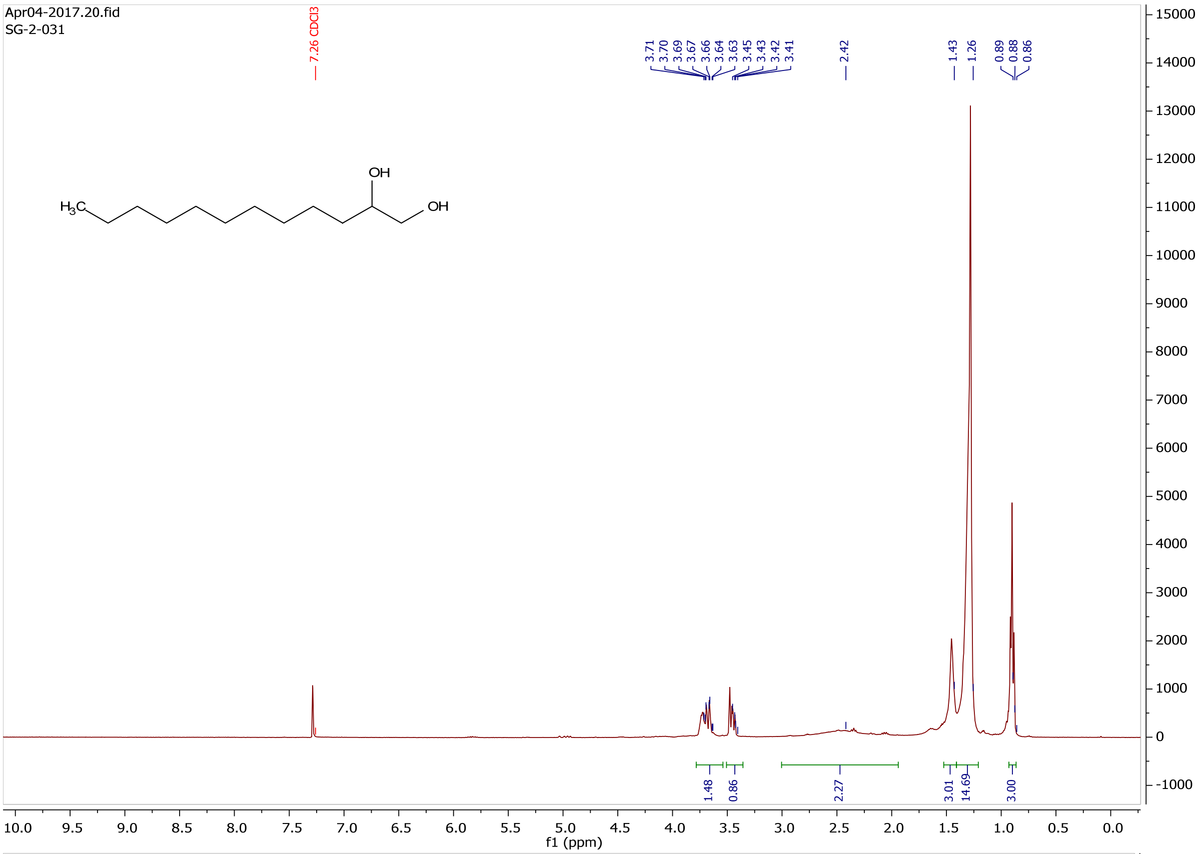
Apr08-2017.20.fid
SG-2-003
OH
OH
143.59
138.89
126.95
125.88
123.98
123.67
80.07
79.43
39.03 DMSO
37.52
f1 (ppm)

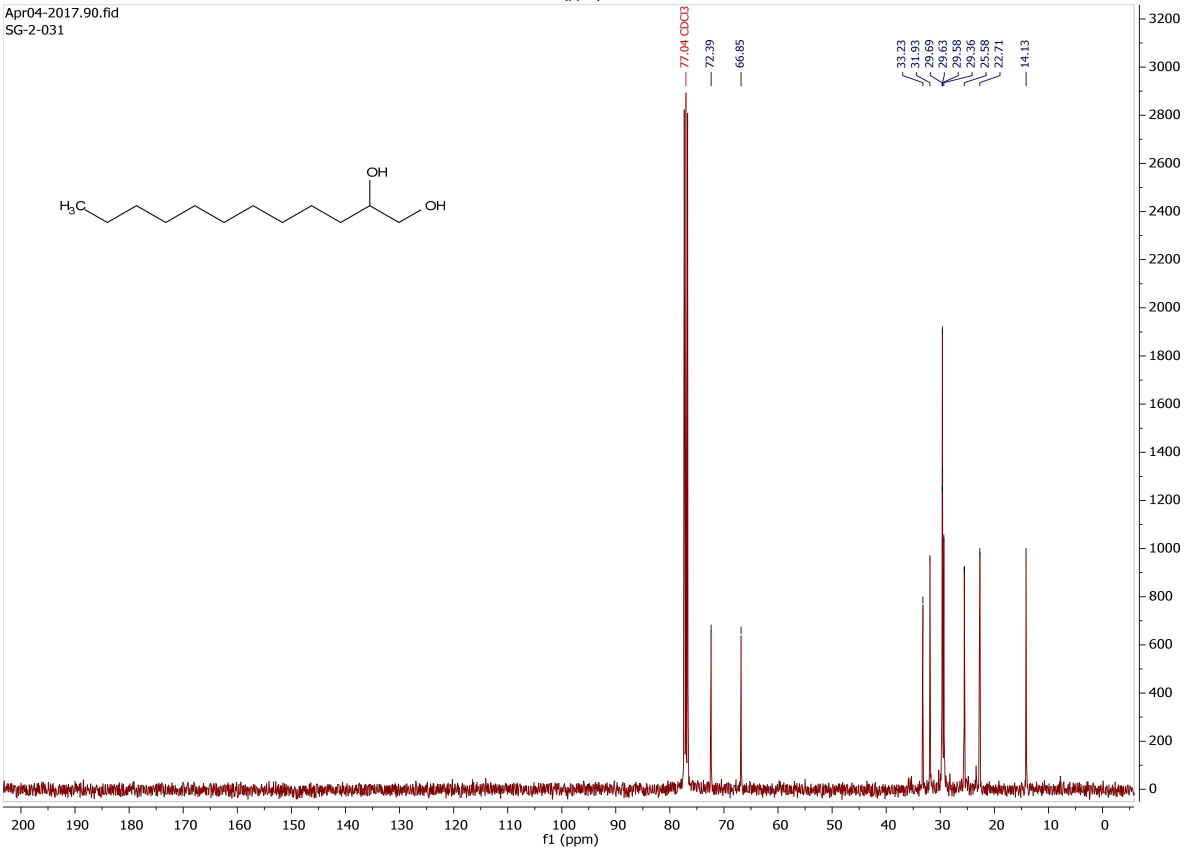

Nov21-2016
SG-1-033
7.27 CDCl3
3.72 3.70 3.70 3.69 3.68 3.67 3.66 3.64 3.63 3.46 3.44 3.43 3.43 3.41
2.61
1.44 1.43 1.43 1.36 1.35 1.34 1.33 1.32 0.93 0.91 0.89
0.00 TMS
B (dd) 3.43
A (ddd) 3.68
C (s) 2.61
E (m) 1.38
2.03 0.94 2.00 6.35 3.04
f1 (ppm)

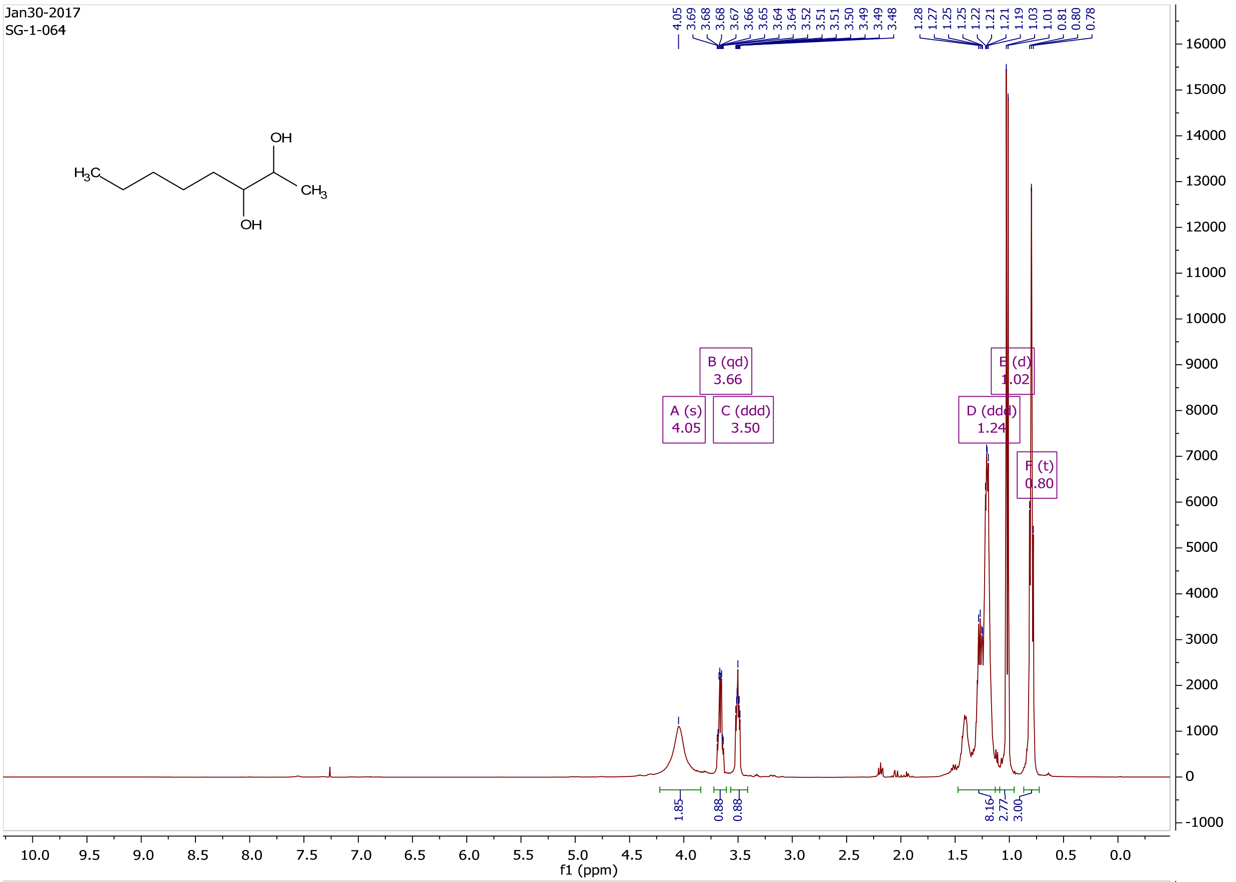

Dec12-2016
SG-1-033
77.48 77.16 CDCl3 76.84
72.49
66.86
32.91
27.86
22.82
14.08
f1 (ppm)

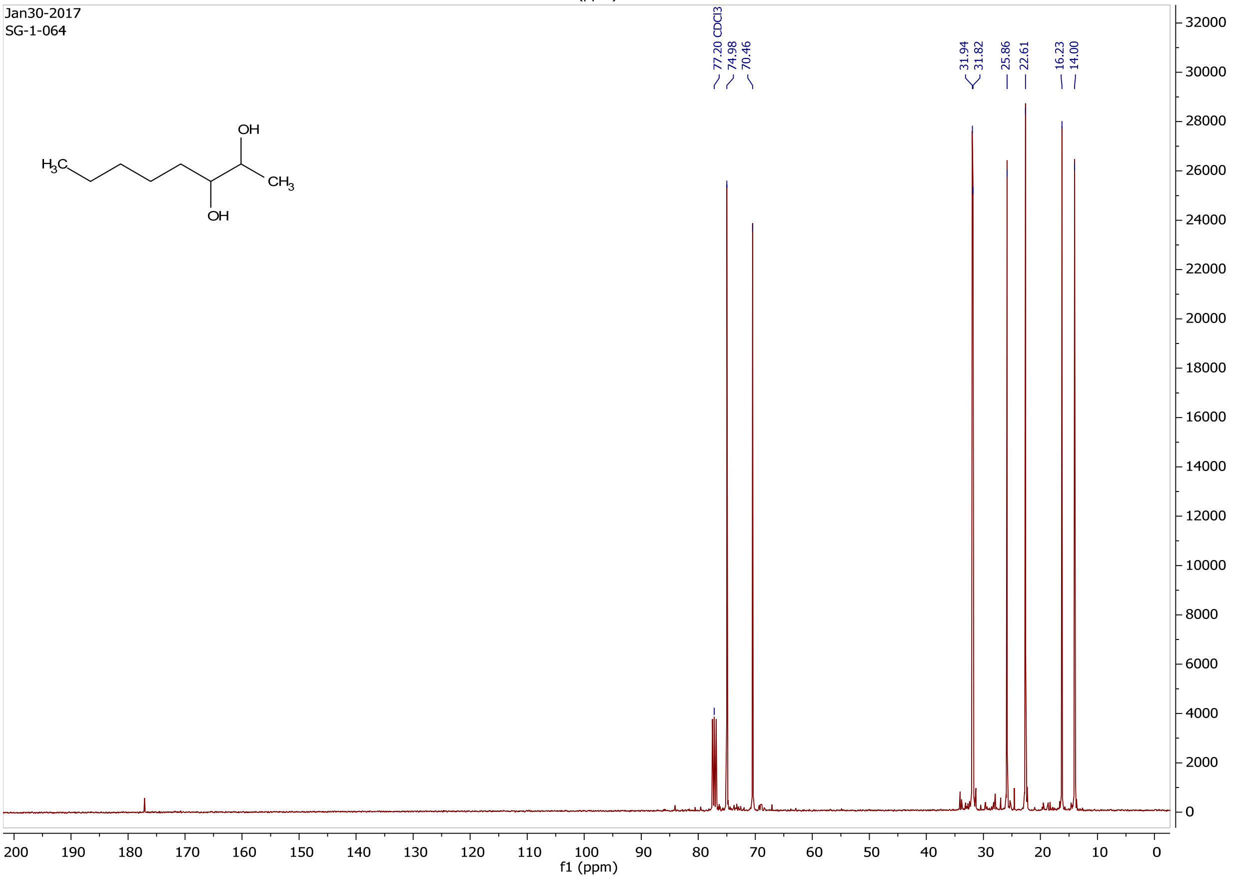

Nov21-2016
SG-1-034
7.27 CDCl3
3.72
3.71
3.67
3.65
3.64
3.46
3.44
3.43
3.41
2.35
1.44
1.31
1.29
0.90
0.89
0.87
OH
H3C
OH
A (m)
3.71
B (dd)
3.43
F (m)
3.65
C (s)
2.35
G (d)
1.30
D (s)
1.44
E (t)
0.89
0.89
1.04
1.07
1.89
2.85
7.77
3.27
f1 (ppm)

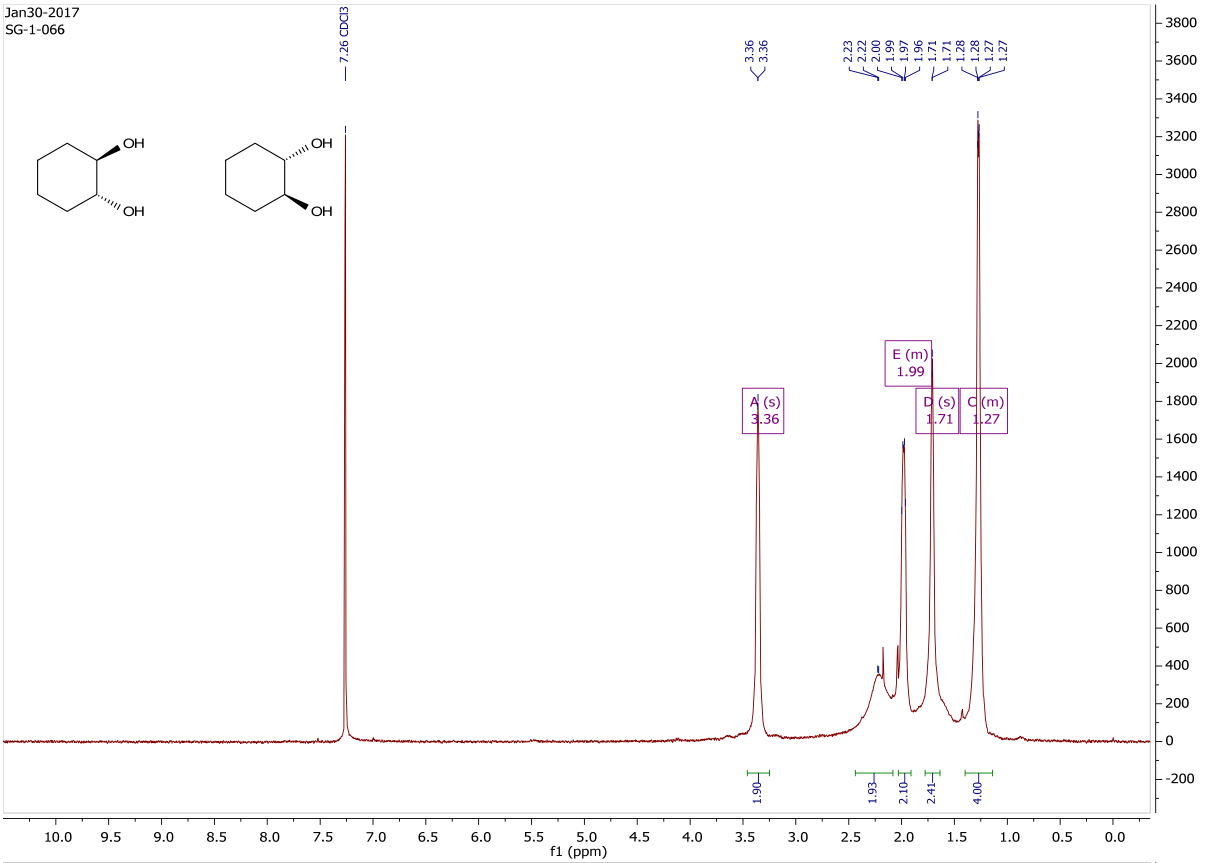

Dec12-2016
SG-1-034
77.61
77.29 CDCl3
76.98
72.64
66.99
33.38
32.03
29.60
25.82
22.85
14.29
OH
H3C
OH
f1 (ppm)

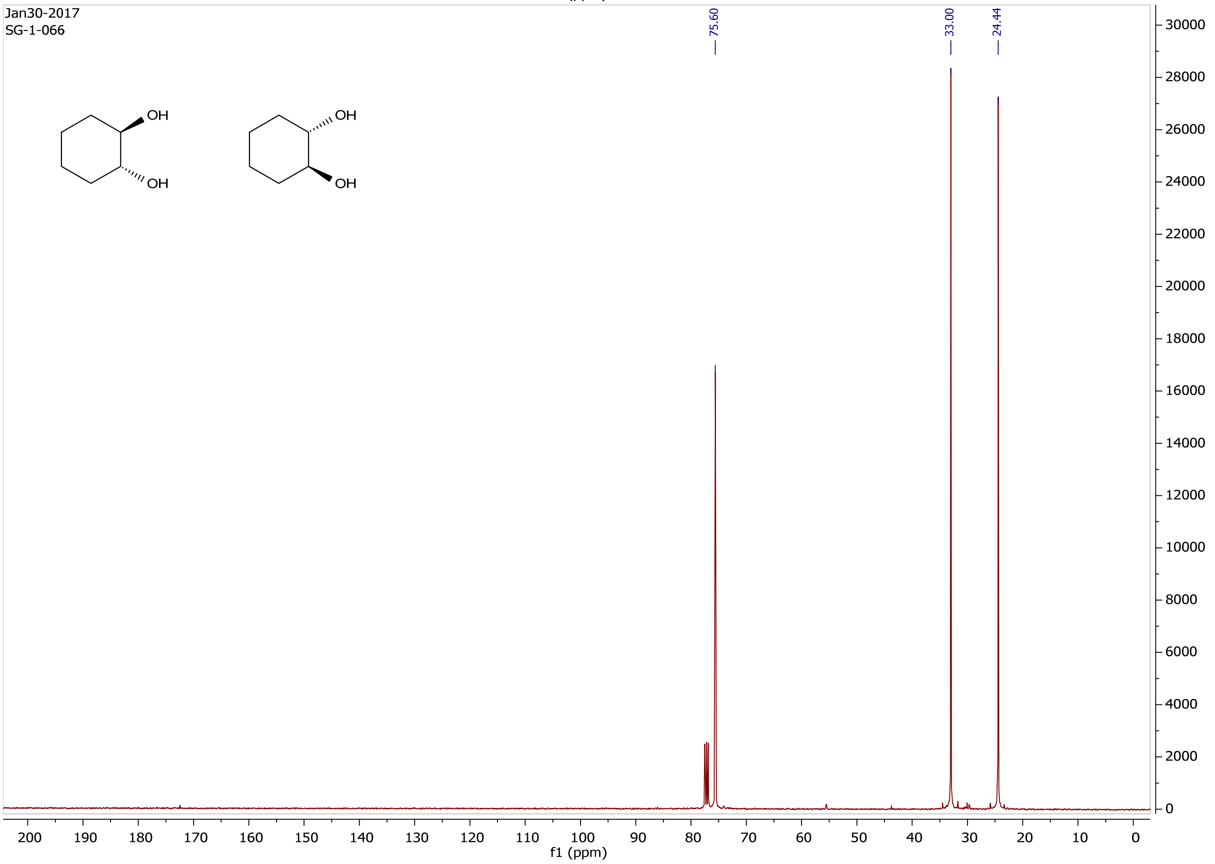

Apr04-2017.20.fid
SG-2-031
7.26 CDCl3
3.71
3.70
3.69
3.67
3.66
3.64
3.63
3.45
3.43
3.42
3.41
2.42
1.43
1.26
0.89
0.88
0.86
OH
H3C
OH
1.48
0.86
2.27
3.01
14.69
3.00
f1 (ppm)

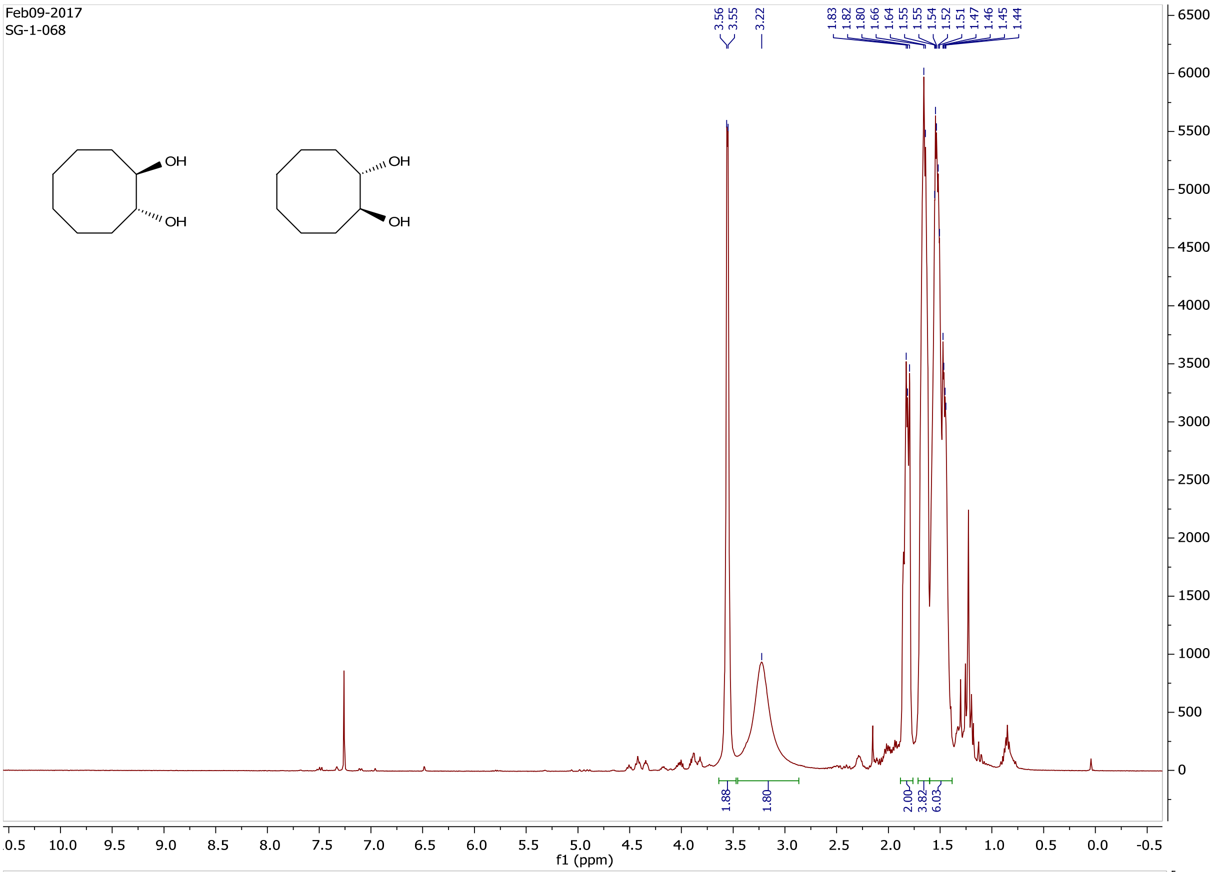

Apr04-2017.90.fid
SG-2-031
77.04 CDCl3
72.39
66.85
33.23
31.93
29.69
29.63
29.58
29.36
25.58
22.71
14.13
OH
H3C
OH
f1 (ppm)

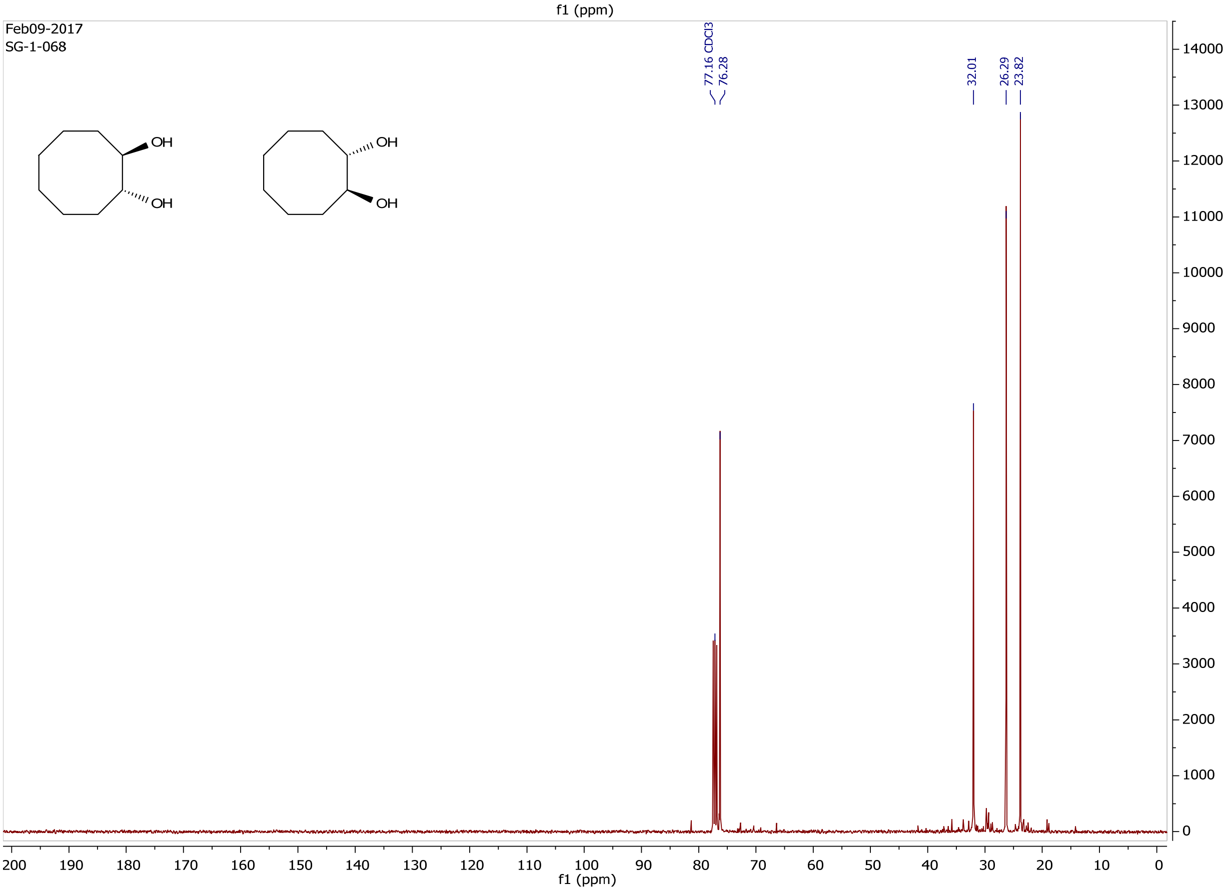

Jan30-2017
SG-1-064
4.05 3.69 3.68 3.68 3.67 3.66 3.65 3.64 3.64 3.52 3.51 3.51 3.50 3.49 3.49 3.48 1.28 1.27 1.25 1.25 1.22 1.21 1.21 1.19 1.03 1.01 0.81 0.80 0.78
OH
H_3C
CH_3
OH
B (qd) 3.66
A (s) 4.05
C (ddd) 3.50
E (d) 1.02
D (ddd) 1.24
F (t) 0.80
1.85
0.88
0.88
8.16
2.77
3.00
f1 (ppm)


Jan30-2017
SG-1-064
77.20 CDCl3
74.98
70.46
31.94
31.82
25.86
22.61
16.23
14.00
OH
H_3C
CH_3
OH
f1 (ppm)


Jan30-2017
SG-1-066
7.26 CDCl3
3.36
3.36
2.23
2.22
2.00
1.99
1.97
1.96
1.71
1.71
1.28
1.28
1.27
1.27
OH
OH
E (m)
1.99
A (s)
3.36
D (s)
1.71
C (m)
1.27
1.90
1.93
2.10
2.41
4.00
f1 (ppm)


Jan30-2017
SG-1-066
75.60
33.00
24.44
OH
OH
f1 (ppm)


Feb09-2017
SG-1-068
OH
OH
3.56
3.55
3.22
1.83
1.82
1.80
1.66
1.64
1.55
1.55
1.54
1.52
1.51
1.47
1.46
1.45
1.44
1.88
1.80
2.00
3.82
6.03
f1 (ppm)


Feb09-2017
SG-1-068
OH
OH
77.16 CDCl3
76.28
32.01
26.29
23.82
f1 (ppm)

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