Supporting Information

For

An eco-compatible pathway to new hydrotropes from tetraols

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1. General information

All reagents were obtained from commercial sources and used as received. Erythritol (>99% purity) was purchased from Sigma-Aldrich. Pentaerythritol (>98% purity) was purchased from ACROS. Valeraldehyde, octanal, decanal and dodecanal were supplied by Sigma-Aldrich or Alfa-Aesar. Camphorsulfonic acid was purchased from Chimex, Amberlyst 35 were bought from Rohm and Haas. Pd/C (5 or 10%, Pd on activated carbon, reduced and dry, Escat 1431) was purchased from Strem Chemicals. All other reagents and solvents were used as received without further purification. Reactions were monitored by TLC or GC and chromatographic separations were performed over silica gel 60H (40-63µ). Acetalisation were carried in a 20mL sealed tube and methylation in 50mL sealed tube. Direct reductive alkylations were undertaken in a 300mL steel Parr autoclave equipped with a mechanic stirrer.

All compounds were characterized by spectroscopic data. The nuclear magnetic resonance (NMR) spectra were recorded either on a Bruker ALS 300 (^{1}H: 300 MHz, ^{13}C: 75 MHz), a DRX 300 (^{1}H: 300 MHz, ^{13}C: 75 MHz) or a Bruker DRX 400 (^{1}H: 400 MHz, ^{13}C: 100 MHz) spectrometer, in $CDCl_3$ or DMSO-d_6 at 293K. Chemical shifts are reported in parts per million (ppm) and are calibrated on residual solvent peaks: $CDCl_3$ 7.16 ppm in ^{1}H and 77.16 ppm in ^{13}C, or DMSO-d_6 2.50 ppm in ^{1}H and 39.52 ppm in ^{13}C. Spin-spin coupling constants (*J*) are

given in Hz. The peak patterns are indicated as follows: (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet, and br. for broad).

IR spectra were recorded by using a Spectro Nicolet IS10 Smart ITR instrument with an Attenuated Total Reflectance (ATR) diamond.

ESI HRMS mass spectra were recorded in positive-ion mode by using a hybrid quadrupole time-of-flight mass spectrometer (Micro-TOFQ-II, Bruker Daltonics, Bremen) with an ESI ion source. The flow of spray gas was 0.4 bar, and the capillary voltage was 3.5 kV. The solutions were infused at 10 $mL.min^{-1}$ in a mixture of solvents (methanol/ dichloromethane/water=45:40:15). The mass range of the analysis was m/z=50–1000, and the calibration was performed with sodium formate.

GC analyses were performed on a Shimadzu Gas Chromatograph GC-2025 equipped with a ZB-5-MS column (30.0 m × 0.25mm × 0.25µm). The carrier gas was N_2 at a flow rate of 1.27 mL/min. Column temperature was initially 70 °C for 2 min, then gradually increased to 310 °C at 15 °C/min and finally kept at 310 °C for 10 min. The injector temperature was 280 °C and for detection a FID was used at 315 °C.

General procedure for the preparation of acetals.

In a round bottom flask equipped with a condenser fitted with a $CaCl_2$ guard, under an argon atmosphere, the corresponding polyol (10.00 g, 85.0mmol, 3 equiv.) was dissolved in dry EtOH. The aldehyde (27.3 mmol, 1 equiv.) was added dropwise, followed by camphorsulfonic acid (10 wt%/aldehyde). Then, the mixture was magnetically stirred at 80 °C for 15 hours. The mixture was cooled down to room temperature and the solvent was evaporated under reduced pressure. The residue was triturated in EtOAc and the excess of polyol was filtered off and washed with cold EtOAc. This operation was repeated two times in order to remove any traces of polyol. The residue was concentrated, in the case of erythritol, a mixture of regioisomers acetals were obtained. The ratio of compounds has been determined by H^1 NMR. Acetal is crystalized with the minimum of diethylether in a 250mL round bottom flask. The solution is cold-filtered by a sintered glass. Purified acetal contained in the solid part is dried under reduced pressure.

General procedure for the recycling of acid catalyst and excess of polyol.

Same protocol as described above was made for the acetalisation, but after cooling, the acid resin and the excess of polyol recrystallized and filtrated were dried under vacuum. The mixture of polyol and acid resin obtained was completed by fresh polyol to restart acetalisation.

General procedure for the preparation of corresponding ethers.

The corresponding acetal (10 mmol) was diluted in dry CPME (100 mL) and 5%-Pd/C (1.06 g, 5 mol% in Pd) was added in a 300 mL stainless steel autoclave. The reactor was tightly closed, purged three times with hydrogen and hydrogen pressure was introduced (30 bar). The system was heated at 130 °C and mechanically stirred for 15 hours. After cooling to room temperature, hydrogen pressure was released and the reaction mixture was then dissolved in EtOAc (100 mL), filtered (Millipore Durapore filter 0.01 μm), and washed two times with 20 mL of EtOAc. The filtrate was evaporated under reduced pressure and the residue was purified by flash chromatography to give corresponding ethers, in erythritol case, a mixture of 2 regioisomers were obtained. The ratio of compounds has been determined by GC.

Thermogravimetric analysis

ThermoGravimetric Analysis (TGA) experiments were performed with a TA Instruments TGA Q500 apparatus. Approximately 20 mg of each compound was introduced into a platinum crucible and a temperature ramp of 5 °C/min was applied from 20 °C to 300 °C under nitrogen atmosphere. The decrease of the mass was followed as a function of time and temperature.

Table 1: Boiling point/decomposition temperature, half-evaporation-temperature $T_{50\%}$ of *O*-alkylerythritols (mixture of 1-*O* and 2-*O* isomers), *O*-alkylpentaerythritols, 1-*O*-alkylglycerols

	R	Ratio **2** : **9**	T_{boil} T_{dec}[c] (°C)	$T_{50\%}$ (°C)
2.a: 9.a	C_4H_9	60 :40	245	191
2.a: 9.a	C_4H_9	100 :0	316	n.d.
2.b : 9.b	C_5H_{11}	58 :42	329	201
2.c : 9.c	C_7H_{17}	59 :41	307[c]	221
2.d : 9.d	C_9H_{19}	70:30	350	230
2.e : 9.e	$C_{11}H_{23}$	65:35	342	237
13.a	C_4H_9	-	302	196
13.b	C_5H_{11}	-	333[c]	210
13.c	C_7H_{17}	-	315[c]	223
13.d	C_9H_{19}	-	338[c]	239
13.e	$C_{11}H_{23}$	-	329[c]	246
15.a	C_4H_9	-	258	n.d.
15.b	C_5H_{11}	-	n.d.	n.d.
15.c	C_7H_{17}	-	301[c]	149
15.d	C_9H_{19}	-	328[c]	184
15.e	$C_{11}H_{23}$	-	n.d.[c]	204

The table 1 described the values of the second endothermic peak observed by DSC, which are assigned to the boiling point or the decomposition temperature of the compounds. ThermoGravimetric Analysis (TGA) is carried out to distinguish whether the endothermic peaks observed with *O*-alkylerythritol and *O*-alkylpentaerythritol correspond to the boiling or degradation of the compound tested. Since the ATG analysis is carried out in an open capsule and under a stream of nitrogen, compounds evaporate at a temperature substantially below its boiling point (□T ≈ 60-100°C), thus avoiding its decomposition. Figure 1 shows the TGA profile of the *O*-alkylpentaerythritol derivatives

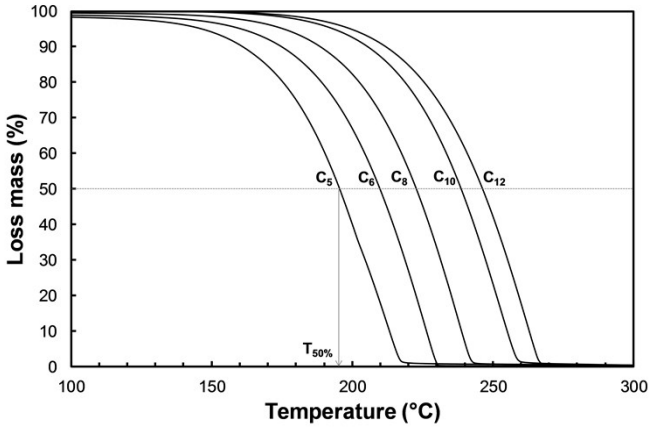


Figure 1 : Mass loss (%) vs temperature of *O*-alkylpentaerythritol derivatives determined by TGA (5°C/min)

while Figure 2 show the evolution of the boiling points of the most volatile amphiphiles and the half-evaporation-temperature $T_{50\%}$ of heavier compounds.

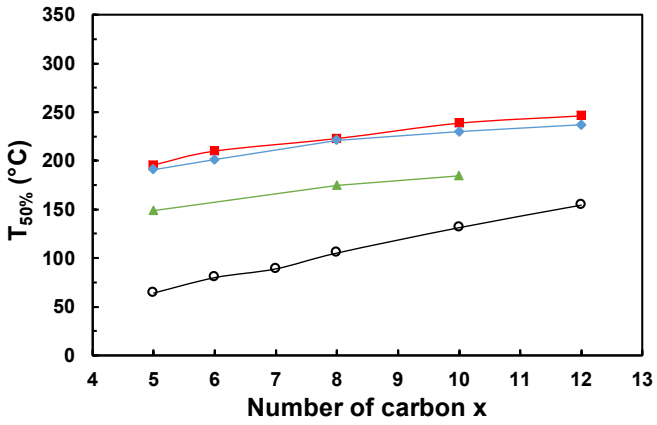
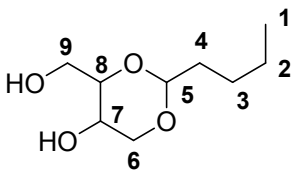


Figure 2: Half-evaporation-temperature, $T_{50\%}$ (°C) vs number of carbon x of the alkyl chain of (◆)***O***-alkylerythritol (mixture of 1-*O* and 2-*O* isomers), (⭘) *O*-alkylpentaerythritol and (●) 1-*O*-alkylglycerol and (○) n-alcohols C_x-OH

2. Characterization data

All erythritol compounds are a mixture of diastereoisomers

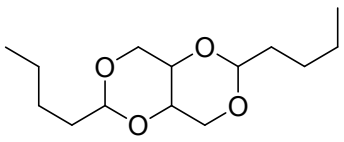
2-butyl-4-(hydroxymethyl)-1,3-dioxan-5-ol **4a:**



Chemical Formula: $C_9H_{18}O_4$
Molecular Weight: 190,24

White solid (Mp =102-104°C). Eluent for isolation: 6:4~4:6 cyclohexane/EtOAc, or crystallization in Et_2O. **^{1}H NMR:** δ = 0.89 (t, *J* = 7.1 Hz, 3H, CH_3 [1]), 1.25-1.42 (m, 4H, 2xCH_2 [2,3]), 1.58-1.64 (m, 2H, CH_2 [4]), 2.6 (br, 2H, 2xOH), 3.38 (t, *J* = 10.4 Hz, 1H, CH_2 [6]), 3.40-3.46 (dt, *J* = 8.3, 3.8 Hz, 1H, CH [8]), 3.72-3.81 (m, 1H, CH [7]), 3.83-3.85 (dd, *J* = 4.1, 1.9 Hz, 2H, CH_2 [9]), 4.15 (dd, *J* = 10.7, 5.4 Hz, 1H, CH_2 [6]), 4.52 (t, *J* = 5.2 Hz, 1H, CH [5]) **^{13}C NMR:** δ = 14.2 (CH_3 [1]), 24.6 (CH_2 [2]), 26.4 (CH_2 [3]), 34.2 (CH_2 [4]), 62.5 (CH_2 [9]), 62.7 (CH [7]), 70.7 (CH_2 [6]), 80.8 (CH [8]), 102.2 (CH [5]). **GC:** retention time: 9.9 min.; MS (ESI+) calcd for $C_9H_{18}O_4$: $[M^+Na]^+$ = 213.1103; found: 213.1097.

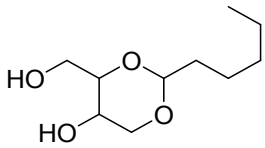
2,6-dibutyltetrahydro-[1,3]dioxino[5,4-d][1,3]dioxine **6:**



Chemical Formula: $C_{14}H_{26}O_4$
Exact Mass: 258,1831

White solid (Mp = 74-76°C). Eluent for isolation: 8:2 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.88 (t, *J* = 7.1 Hz, 6H, 2xCH_3), 1.27-1.38 (m, 8H, 4xCH_2), 1.56-1.63 (m, 4H, 2xCH_2), 3.48-3.61 (m, 4H, 2xCH and CH_2), 4.11 (dd, *J* = 9.4, 3.1 Hz, 2H, CH_2), 4.60 (t, *J* = 5.2 Hz, 2H, 2xCH); **^{13}C NMR:** δ = 14.1 (2xCH_3), 22.6 (2xCH_2), 26.3 (2xCH_2), 34.2 (2xCH_2), 68.6 (2xCH_2), 73.7 (2xCH), 103.1 (2xCH). **GC:** retention time: 11.75 min.; MS (ESI+) calcd for $C_{14}H_{26}O_4$: $[M^+Na]^+$ = 281.1729; found: 281.1721.

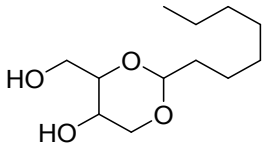
2-pentyl-4-(hydroxymethyl)-1,3-dioxan-5-ol **4b:**



Chemical Formula: $C_{10}H_{20}O_4$
Exact Mass: 204,1362

White solid (Mp = 104-106°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.89 (t, *J* = 6.8 Hz, 3H, CH_3), 1.25-1.42 (m, 6H, 3xCH_2), 1.58-1.64 (m, 2H, CH_2), 2.04-2.22 (br, 2H, 2xOH), 3.39 (t, *J* = 10.4 Hz, 1H, CH_2), 3.43-3.49 (dt, *J* = 9.1, 4.5 Hz, 1H, CH), 3.73-3.82 (m, 1H, CH), 3.86 (t, *J* = 3.9 Hz, 2H, CH_2), 4.15 (dd, *J* = 10.7, 5.4 Hz, 1H, CH_2), 4.52 (t, *J* = 5.2 Hz, 1H, CH); **^{13}C NMR:** δ = 14.1 (CH_3) 22.7 (CH_2), 24.0 (CH_2), 31.8 (CH_2), 34.5 (CH_2), 63.0 (CH and CH_2), 70.7 (CH_2), 80.7 (CH), 102.2 (CH). **GC:** retention time: 10.2 min.; MS (ESI+) calcd for $C_{10}H_{20}O_4$: $[M^+Na]^+$ = 227.1259; found: 227.1254.

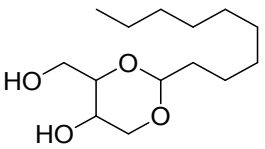
2-heptyl-4-(hydroxymethyl)-1,3-dioxan-5-ol **4c:**



Chemical Formula: $C_{12}H_{24}O_4$
Exact Mass: 232,17

White solid (Mp =108°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.87 (t, *J* = 6.6 Hz, 3H, CH_3), 1.27-1.38 (m, 10H, 5xCH_2), 1.57-1.64 (m, 2H, CH_2), 2.27 (br, 2H, 2xOH), 3.39 (t, *J* = 10.5 Hz, 1H, CH_2), 3.42-3.47 (dt, *J* = 12.6, 3.3 Hz, 1H, CH), 3.73-3.81 (m, 1H, CH), 3.85 (dd, *J* = 4.2, 2.6 Hz, 2H, CH_2), 4.15 (dd, *J* = 10.7, 5.3 Hz, 1H, CH_2), 4.53 (t, *J* = 5.2 Hz, 1H, CH); **^{13}C NMR:** δ = 14.2 (CH_3), 22.8 (CH_2), 24.3 (CH_2), 29.3 (CH_2), 29.5 (CH_2), 31.9 (CH_2), 34.5 (CH_2), 62.5 (CH_2), 62.9 (CH), 70.7 (CH_2), 80.8 (CH), 102.2 (CH). **GC:** retention time: 12.4 min.; MS (ESI+) calcd for $C_{12}H_{24}O_4$: $[M^+Na]^+$ = 255.1572; found: 255.1562.

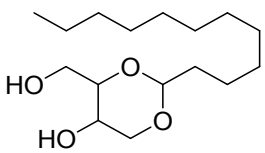
2-nonyl-4-(hydroxymethyl)-1,3-dioxan-5-ol **4d:**



Chemical Formula: $C_{14}H_{28}O_4$
Exact Mass: 260,1988

White solid (Mp =114°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.88 (t, *J* = 6.7 Hz, 3H, CH_3), 1.26-1.40 (m, 14H, 7xCH_2), 1.58-1.62 (m, 2H, CH_2), 1.96-2.00 (br, 1H, OH), 2.10 (d, *J* = 5 Hz, 1H, OH), 3.39 (t, *J* = 10.4 Hz, 1H, CH_2), 3.43-3.49 (dt, *J* = 9.2, 4.6 Hz, 1H, CH), 3.73-3.81 (m, 1H, CH), 3.83-3.88 (m, 2H, CH_2), 4.16 (dd, *J* = 10.7, 5.4 Hz, 1H, CH_2), 4.54 (t, *J* = 5.2 Hz, 1H, CH); **^{13}C NMR:** δ = 14.3 (CH_3), 22.8 (CH_2), 24.3 (CH_2), 29.4 (CH_2), 29.6 (CH_2), 29.7 (3xCH_2), 32.0 (CH_2), 34.5 (CH_2), 62.9 (CH and CH_2), 70.7 (CH_2), 80.7 (CH), 102.2 (CH). **GC:** retention time: 13.9 min.; MS (ESI+) calcd for $C_{14}H_{28}O_4$: $[M^+Na]^+$ = 283.1885; found: 283.1881.

2-undecyl-4-(hydroxymethyl)-1,3-dioxan-5-ol **4e:**

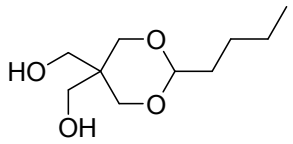


Chemical Formula: $C_{16}H_{32}O_4$
Exact Mass: 288,2301

White solid (Mp =114-116°C). Crystallization in EtOH. **^{1}H NMR:** δ = 0.88 (t, *J* = 6.7 Hz, 3H, CH_3), 1,25-1.36 (m, 18H, 9xCH_2), 1.57-1.64 (m, 2H, CH_2), 2.00 (br, 2H, 2xOH), 3.39 (t, *J* = 10.4 Hz, 1H, CH_2), 3.43-3.49 (dt, *J* = 9.2, 4.6 Hz, 1H, CH), 3.74-3.82 (m, 1H, CH), 3.86 (t, *J* = 3.8 Hz, 2H, CH_2), 4.15 (dd, *J* = 10.7, 5.3 Hz, 1H, CH_2), 4.53 (t, *J* = 5.2 Hz, 1H, CH); **^{13}C NMR:** δ = 13.9 (CH_3), 22.1 (CH_2), 23.8 (CH_2), 28.7 (CH_2), 28.9 (CH_2), 29.0

(5xCH_2), 31.3 (CH_2), 34.1 (CH_2), 60,8 (CH_2), 60.9 (CH), 70.4 (CH_2), 82.6 (CH), 101.9 (CH). **GC:** retention time: 15.2 min.; MS (ESI+) calcd for $C_{16}H_{32}O_4$: $[M^+Na]^+$ = 311.2198; found: 311.2193.

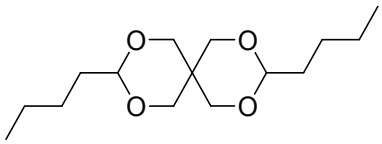
(2-butyl-1,3-dioxane-5,5-diyl)dimethanol **11a** [775-74-6] [1] :



Chemical Formula: $C_{10}H_{20}O_4$
Exact Mass: 204,1362

White solid (Mp = 80-81°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.79 (t, *J* = 6.9 Hz, 3H, CH_3), 1.15-1.32 (m, 4H, 2xCH_2), 1.48-1.57 (m, 2H, CH_2), 2.13 (br, 2H, 2xOH), 3.36 (s, 2H, CH_2), 3.44 (d, *J* = 11.8 Hz 2H, CH_2), 3.90 (d, J = 14.4 Hz, 2H, CH_2), 3.93 (s, 2H, CH_2), 4.35 (t, *J* = 5.0 Hz, 1H, CH); **^{13}C NMR:** δ = 14.1 (CH_3), 22.7 (CH_2), 26.1 (CH_2), 34.6 (CH_2), 38.9 (C), 64.3 (CH_2), 65.8 (CH_2), 69.7 (2xCH_2), 103.0 (CH). **GC:** retention time: 10.8 min.; MS (ESI+) calcd for $C_{10}H_{20}O_4$: $[M^+Na]^+$ = 227.1259; found: 227.1248.

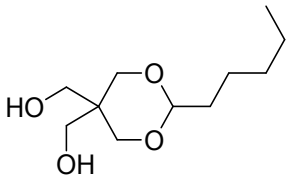
3,9-dibutyl-2,4,8,10-tetraoxaspiro[5.5]undecane **12** [5703-81-1][1]**:**



Chemical Formula: $C_{15}H_{28}O_4$
Exact Mass: 272,1988

White solid (Mp = 57-59°C). Extraction with cyclohexane. **^{1}H NMR:** δ = 0.86 (t, *J* = 7.0 Hz, 6H, 2xCH_3), 1.22-1.35 (m, 8H, 4xCH_2), 1.53-1.60 (m, 4H, 2xCH_2), 3.23-3.33 (m, 2H, CH_2), 3.46-3.55 (m, 4H, 2xCH_2), 4.39 (t, *J* = 5.0 Hz, 2H, 2xCH), 4.51 (m, 2H, CH_2); **^{13}C NMR:** δ = 14.0 (2xCH_3), 22.6 (2xCH_2), 25.9 (2xCH_2), 32.4 (C), 34.5 (2xCH_2), 70.2 (2xCH_2), 70.6 (2xCH_2), 103.0 (2xCH). **GC:** retention time: 12.3 min.; MS (ESI+) calcd for $C_{15}H_{28}O_4$: $[M^+Na]^+$ = 295.1885; found: 295.1877.

2-pentyl-1,3-dioxane-5,5-diyl)dimethanol **11b** [5684-87-7] [2] :

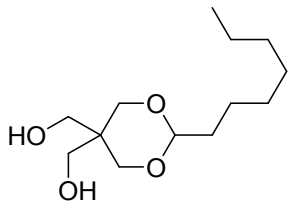


Chemical Formula: $C_{11}H_{22}O_4$
Exact Mass: 218,1518

White solid (Mp = 82-84°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.88 (t, *J* = 6.8 Hz, 3H, CH_3), 1.27-1.30 (m, 6H, 3xCH_2), 1.57-1.64 (m, 2H, CH_2), 3.47 (s, 2H, CH_2), 3.53 (d, *J* = 11.9 Hz, 2H, CH_2), 4.01 (d, *J* = 11.9 Hz, 2H, CH_2), 4.04 (s, 2H, CH_2), 4.45 (t, *J* = 5.0 Hz, 1H, CH); **^{13}C NMR:** δ = 14.2 (CH_3), 22.7 (CH_2), 23.7 (CH_2), 31.8 (CH_2), 34.9 (CH_2), 38.9 (C), 64.5 (CH_2), 65.9 (CH_2), 69.7 (2xCH_2), 103.0 (CH). **GC:** retention time: 11.5 min.; MS (ESI+) calcd for $C_{11}H_{22}O_4$: $[M^+Na]^+$ = 241.1416; found: 241.1413.

[1] W. Conrad, B. Gesner, L. Levasseur, R. Murphy and H. Conrad, *J. Org. Chem.*, 1961, **26**, 3571–3574.
[2] G.-W. Wang, X.-Y. Yuan, Y.-C. Liu and X.-G. Lei, *J. Am. Oil Chem. Soc.*, 1994, **71**, 727–730.

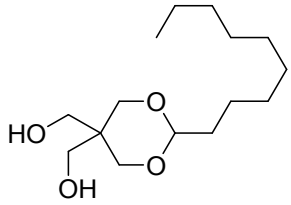
2-heptyl-1,3-dioxane-5,5-diyl)dimethanol **11c** [2] :



Chemical Formula: $C_{13}H_{26}O_4$
Exact Mass: 246,1831

White solid (Mp = 85-86°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.86 (t, *J* = 6.6 Hz, 3H, CH_3), 1.25-1.38 (m, 10H, 5xCH_2), 1.55-1.62 (m, 2H, CH_2), 3.07 (br, 2H, 2xOH), 3.42 (s, 2H, CH_2), 3.50 (d, *J* = 11.9 Hz, 2H, CH_2), 3.96 (d, *J* = 11.6 Hz, 2H, CH_2), 4.98 (s, 2H, CH_2), 4.43 (t, *J* = 5.0 Hz, 1H, CH); **^{13}C NMR:** δ = 14.2 (CH_3), 22.8 (CH_2), 24.0 (CH_2), 29.3 (CH_2), 29.6 (CH_2), 31.9 (CH_2), 34.9 (CH_2), 39.0 (C), 63.6 (CH_2), 65.2 (CH_2), 69.7 (2xCH_2), 103.0 (CH). **GC:** retention time: 13.1 min.; MS (ESI+) calcd for $C_{13}H_{26}O_4$: $[M^+Na]^+$ = 269.1729; found: 269.1717.

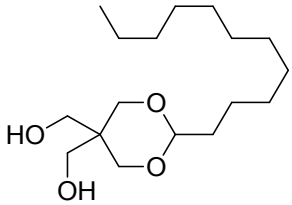
2-nonyl-1,3-dioxane-5,5-diyl)dimethanol **11d** [2] :



Chemical Formula: $C_{15}H_{30}O_4$
Exact Mass: 274,2144

White solid (Mp = 88-90°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.87 (t, *J* = 6.7 Hz, 3H, CH_3), 1.25-1.36 (m, 14H, 7xCH_2), 1.57-1.64 (m, 2H, CH_2), 2.18 (br, 2H, 2xOH), 3.47 (s, 2H, CH_2), 3.53 (d, *J* = 11.9 Hz, 2H, CH_2), 4.00 (d, *J* = 11.9 Hz, 2H, CH_2), 4.04 (s, 1H, CH_2), 4.45 (t, *J* = 5.1 Hz, 1H, CH); **^{13}C NMR:** δ = 14.3 (CH_3), 22,8 (CH_2), 24.0 (CH_2), 29.4 (CH_2), 29.7 (3xCH_2), 32.0 (CH_2), 35.0 (CH_2), 38.9 (C), 64.5 (CH_2), 65.9 (CH_2), 69.7 (2xCH_2), 103.0 (CH). **GC:** retention time: 14.4 min.; MS (ESI+) calcd for $C_{15}H_{30}O_4$: $[M^+Na]^+$ = 297.2042; found: 297.2036.

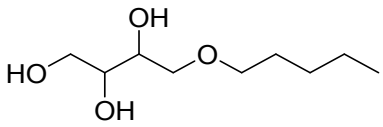
2-undecyl-1,3-dioxane-5,5-diyl)dimethanol **11e** [2] :



Chemical Formula: $C_{17}H_{34}O_4$
Exact Mass: 302,2457

White solid (Mp = 93-94°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.87 (t, *J* = 6.7 Hz, 3H, CH_3), 1.25-1.36 (m, 18H, 9xCH_2), 1.57-1.63 (m, 2H, CH_2), 2.48 (br, 2H, 2xOH), 3.46 (s, 2H, CH_2), 3.52 (d, *J* = 11.8 Hz, 2H, CH_2), 4.00 (d, *J* = 14.4 Hz, 2H, CH_2), 4.02 (s, 2H, CH_2), 4.44 (t, *J* = 5.1 Hz, 1H, CH); **^{13}C NMR:** δ = 14.3 (CH_3), 22.9 (CH_2), 24.0 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.7 (2xCH_2), 29.8 (2xCH_2), 32.1 (CH_2), 35.0 (CH_2), 38.9 (C), 64.3 (CH_2), 65.7 (CH_2), 69.7 (2xCH_2), 103.0 (CH). **GC:** retention time: 15.2 min.; MS (ESI+) calcd for $C_{17}H_{34}O_4$: $[M^+Na]^+$ = 325.2355; found: 325.2348.

1-O-pentylerythritol **2a:**

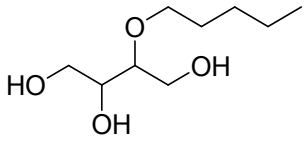


Chemical Formula: $C_9H_{20}O_4$
Exact Mass: 192,14

Colorless liquid. Eluent for isolation: 4:6~2:8 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.87 (t, *J* = 6.9 Hz, 3H, CH_3), 1.26-1.31 (m, 4H, 2xCH_2), 1.52-1.59 (m, 2H, CH_2), 3.45 (t, *J* = 6.8 Hz, 2H, CH_2), 3.48-3.58 (m, 2H, CH_2), 3.64-3.68 (m, 1H, CH), 3.70-3.71 (m, 2H, CH_2), 3.77-3.81 (m, 1H, CH), 3.95 (br, 3H, 3xOH); **^{13}C NMR:** δ = 14.1 (CH_3), 22.6 (CH_2), 28.3 (CH_2), 29.2 (CH_2), 63.5 (CH_2), 71.1 (CH), 72.0 (CH_2), 72.1 (CH_2), 72.9 (CH). **GC:** retention time: 10.2 min.; MS (ESI+) calcd for $C_9H_{20}O_4$: $[M^+Na]^+$ = 215.1259;

found: 215.1247.

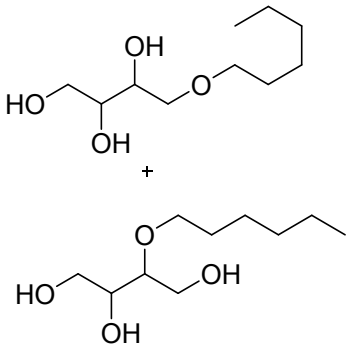
2-O-pentylerythritol **9a:**



Chemical Formula: $C_9H_{20}O_4$
Exact Mass: 192,14

Colorless liquid. Eluent for isolation: 4:6~2:8 cyclohexane/EtOAc. **^{1}H NMR:** δ =0.87 (t, *J* = 6.9 Hz, 3H, CH_3), 1.26-1.31 (m, 4H, 2xCH_2), 1.51-1.60 (m, 2H, CH_2), 3.33-3.38 (m, 1H, CH), 3.41-3.48 (m, 1H, CH_2), 3.57-3.65 (m, 5H, 2xH of CH_2 and 3xOH), 3.66-3.69 (m, 2H, CH_2), 3.72-3.83 (m, 2H, 1xCH and 1xH of CH_2); **^{13}C NMR:** δ = 14.1 (CH_3), 22.6 (CH_2), 28.3 (CH_2), 29.8 (CH_2), 60.6 (CH_2), 63.4 (CH_2), 71.0 (CH_2), 71.8 (CH), 80.0 (CH). **GC:** retention time: 10.5 min.; MS (ESI+) calcd for $C_9H_{20}O_4$: $[M^+Na]^+$ = 215.1259; found: 215.1254.

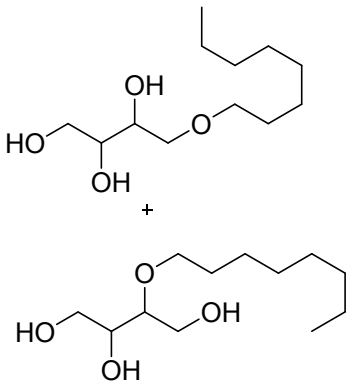
1-O-hexylerythritol + 2-O-hexylerythritol **2b:**



Chemical Formula: $C_{10}H_{22}O_4$
Exact Mass: 206,1518

Colorless liquid. Eluent for isolation: 4:6~2:8 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.87 (t, *J* = 6.8 Hz, 3H, CH_3), 1.28-1.33 (m, 6H, 3xCH_2), 1.52-1.59 (m, 2H, CH_2), 2.52 (br, 3H, 3xOH), 3.25-3.30 (m, 0.4x1H, CH secondary ether), 3.44-3.81 (m, 10.6H, 0.58x(3xCH_2 and 2xCH for primary ether), 0.42x(CH and 3xCH_2 for secondary ether), 3H for -OH); **^{13}C NMR:** δ = 14.1 (CH_3), 22.7 (CH_2), 25.8 (CH_2), 29.6 (CH_2), 30.0 (CH_2 secondary ether), 31.8 (CH_2), 60,8 (CH_2 secondary ether), 64.0 and 63.6 (CH_2), 70.7 (CH_2 secondary ether), 71.1 (CH primary ether), 71.6 (CH_2 primary ether), 72.0 (CH_2 primary ether), 72.1 (CH secondary ether), 72.9 (CH for primary ether), 80.1 (CH secondary ether). **GC:** retention time: 10.4 and 10.7 min.; MS (ESI+) calcd for $C_{10}H_{22}O_4$: $[M^+Na]^+$ = 229.1416; found: 229.1416.

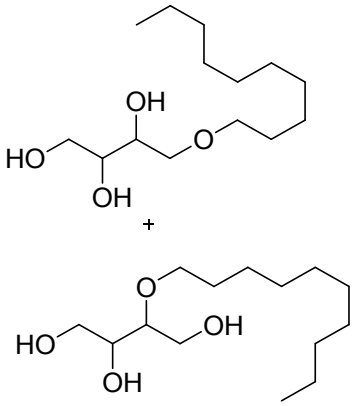
1-O-octylerythritol + 2-O-octylerythritol **2c:**



Chemical Formula: $C_{12}H_{26}O_4$
Exact Mass: 234,1831

Sticky white solid. Eluent for isolation: 4:6~1:9 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.88 (t, *J* = 6.7 Hz, 3H, CH_3), 1.27-1.36 (m, 10H, 5xCH_2), 1.55-1.60 (m, 2H, CH_2), 2.52 (s, 3H, 3xOH), 3.37-3.41 (m, 0.4x1H, CH secondary ether), 3.46-3.85 (m, 7.6H, 0.59x(3xCH_2 and 2xCH for primary ether), 0.41x(CH and 3xCH_2 for secondary ether); **^{13}C NMR:** δ = 14.1 (CH_3), 22.7 (CH_2), 26.1 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.6 (CH_2 primary ether), 30.0 (CH_2 secondary ether), 31.9 (CH_2), 60.5 (CH_2 secondary ether), 64.0 and 63.4 (CH_2), 70.5 (CH_2 secondary ether), 71.0 (CH primary ether), 71.4 (CH_2 primary ether), 72.0 (CH_2 primary ether), 72.1 (CH secondary ether), 72.8 (CH for primary ether), 80.1 (CH secondary ether). **GC:** retention time: 12.7 and 12.9 min.; MS (ESI+) calcd for $C_{12}H_{26}O_4$: $[M^+Na]^+$ = 257.1729; found: 257.1734.

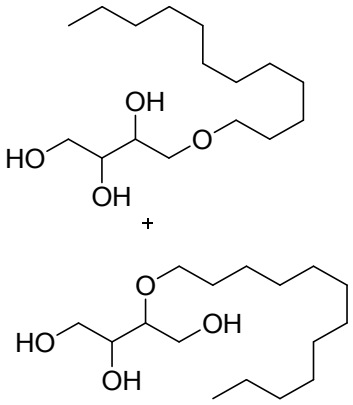
1-O-decylerythritol + 2-O-decylerythritol **2d:**



Chemical Formula: $C_{14}H_{30}O_4$
Exact Mass: 262,2144

White solid (Mp = 39-41°C). Eluent for isolation: 4:6~1:9 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.86 (t, *J* = 6.7 Hz, 3H, CH_3), 1.22-1.29 (m, 14H, 7xCH_2), 1.51-1.58 (m, 2H, CH_2), 3.25-3.27 (m, 0.30x1H, CH secondary ether), 3.45 (m, 10.7H, 0.70x(3xCH_2 and 2xCH for primary ether), 0.30x(CH and 3xCH_2 for secondary ether), 3H for -OH); **^{13}C NMR:** δ = 14.2 (CH_3), 22.8 (CH_2), 26.2 (CH_2), 29.4 (CH_2), 29.6 (CH_2), 29.7 (CH_2 primary ether), 29.7 (2xCH_2), 30.1 (CH_2 secondary ether), 32.0 (CH_2), 60,7 (CH_2 secondary ether), 64.0 and 63.5 (CH_2), 70.7 (CH_2 secondary ether), 71.1 (CH primary ether), 71.5 (CH_2 primary ether), 72.0 (CH_2 primary ether), 72.1 (CH secondary ether), 72.9 (CH for primary ether), 80.1 (CH secondary ether). **GC:** retention time: 14.1 and 14.3 min.; MS (ESI+) calcd for $C_{14}H_{30}O_4$: $[M^+Na]^+$ = 285.2042; found: 285.2029

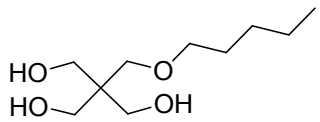
1-O-dodecylerythritol + 2-O-dodecylerythritol **2e:**



Chemical Formula: $C_{16}H_{34}O_4$
Exact Mass: 290,2457

White solid (Mp = 49-51°C). Eluent for isolation: 4:6~1:9 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.88 (t, *J* = 6.7 Hz, 3H, CH_3), 1.24-1.36 (m, 18H, 9xCH_2), 1.53-1.60 (m, 2H, CH_2), 2.34 (s, 3H, 3xOH), 3.36-3.41 (m, 0.35x1H, CH secondary ether), 3.46-3.84 (m, 7.7H, 0.65x(3xCH_2 and 2xCH for primary ether), 0.35x(CH and 3xCH_2 for secondary ether); **^{13}C NMR:** δ = 14.2 (CH_3), 22.8 (CH_2), 26.2 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.7 (CH_2 primary ether), 29.8 (4xCH_2), 30.1 (CH_2 secondary ether), 32.0 (CH_2), 60,7 (CH_2 secondary ether), 64.0 and 63.6 (CH_2), 70.7 (CH_2 secondary ether), 71.1 (CH primary ether), 71.5 (CH_2 primary ether), 72.0 (CH secondary ether and CH_2 primary ether), 72.9 (CH for primary ether), 80.1 (CH secondary ether). **GC:** retention time: 15.4 and 15.6 min.; MS (ESI+) calcd for $C_{16}H_{34}O_4$: $[M^+Na]^+$ = 313.2355; found: 313.2335

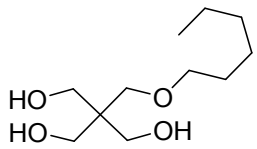
O-pentylpentaerythritol **13a:**



Chemical Formula: $C_{10}H_{22}O_4$
Exact Mass: 206,1518

Colorless liquid. Eluent for isolation: 4:6~1:9 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.89 (t, *J* = 6.9 Hz, 3H, CH_3), 1.27-1.32 (m, 4H, 2xCH_2), 1.51-1.58 (m, 2H, CH_2), 3.40 (t, *J* = 6.6 Hz, 2H, CH_2), 3.43 (s, 2H, CH_2), 3.53 (br, 3H, 3xOH), 3.68 (s, 6H, 3xCH_2); **^{13}C NMR:** δ = 14.1 (CH_3), 22.6 (CH_2), 28.4 (CH_2), 29.3 (CH_2), 45.0 (C), 64.3 (3xCH_2), 72.3 (CH_2), 73.6 (CH_2). **GC:** retention time: 11.0 min.; MS (ESI+) calcd for $C_{10}H_{22}O_4$: $[M^+Na]^+$ = 229.1416; found: 229.1407.

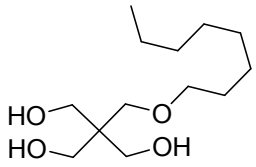
O-hexylpentaerythritol **13b:**



Chemical Formula: $C_{11}H_{24}O_4$
Exact Mass: 220,1675

Colorless liquid. Eluent for isolation: 4:6~1:9 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.87 (t, *J* = 6.9 Hz, 3H, CH_3), 1.27-1.30 (m, 6H, 3xCH_2), 1.49-1.58 (m, 2H, CH_2), 3.24 (br, 3H, 3xOH), 3.40 (t, *J* = 6.6 Hz, 2H, CH_2), 3.43 (s, 2H, CH_2), 3.67 (s, 6H, 3xCH_2); **^{13}C NMR:** δ = 14.1 (CH_3), 22.7 (CH_2), 25.9 (CH_2), 29.5 (CH_2), 31.7 (CH_2), 45.0 (C), 64.4 (3xCH_2), 72.3 (CH_2), 73.7 (CH_2). **GC:** retention time: 11.8 min.; MS (ESI+) calcd for $C_9H_{20}O_4$: $[M^+Na]^+$ = 243.1572; found: 243.1560.

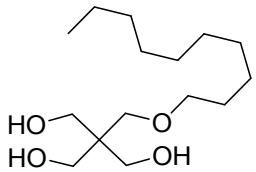
O-octylpentaerythritol **13c:**



Chemical Formula: $C_{13}H_{28}O_4$
Exact Mass: 248,1988

Colorless liquid. Eluent for isolation: 2:8 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.85 (t, *J* = 6.7 Hz, 3H, CH_3), 1.21-1.30 (m, 10H, 5xCH_2), 1.47-1.54 (m, 2H, CH_2), 3.37 (t, *J* = 6.6 Hz, 2H, CH_2), 3.39 (s, 2H, CH_2), 3.63 (s, 6H, 3xCH_2), 3.69 (br, 3H, 3xOH); **^{13}C NMR:** δ = 14.2 (CH_3), 22.7 (CH_2), 26.2 (CH_2), 29.3 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 31.9 (CH_2), 45.0 (C), 63.9 (3xCH_2), 72.2 (CH_2), 73.4 (CH_2). **GC:** retention time: 13.2 min.; MS (ESI+) calcd for $C_{13}H_{28}O_4$: $[M^+Na]^+$ = 271.1885; found: 271.1870.

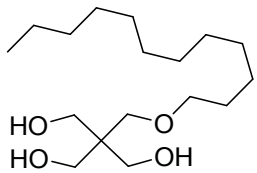
O-decylpentaerythritol **13d**[3]**:**



Chemical Formula: $C_{15}H_{32}O_4$
Exact Mass: 276,2301

White solid (Mp = 93-94°C). Crystallization in Et_2O. **^{1}H NMR:** δ = 0.87 (t, *J* = 6.7 Hz, 3H, CH_3), 1.25-1.32 (m, 18H, 9xCH_2), 1.50-1.56 (m, 2H, CH_2), 3.01 (br, 3H, 3xOH), 3.40 (t, *J* = 6.6 Hz, 2H, CH_2), 3.44 (s, 2H, CH_2), 3.69 (s, 6H, 3xCH_2); **^{13}C NMR:** δ = 14.3 (CH_3), 22.8 (CH_2), 26.2 (CH_2), 29.4 (CH_2), 29.6 (2xCH_2), 29.7 (2xCH_2), 32.0 (CH_2), 45.0 (C), 64.7 (3xCH_2), 72.3 (CH_2), 73.7 (CH_2). **GC:** retention time: 14.3 min.; MS (ESI+) calcd for $C_{15}H_{32}O_4$: $[M^+Na]^+$ = 299.2198; found: 299.2196.

O-dodecylpentaerythritol **13e:**

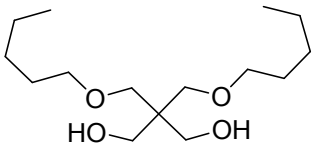


Chemical Formula: $C_{17}H_{36}O_4$
Exact Mass: 304,2614

White solid (Mp = 61°C). Eluent for isolation: 3:7~1:9 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.86 (t, *J* = 6.7 Hz, 3H, CH_3), 1.22-1.29 (m, 18H, 9xCH_2), 1.49-1.58 (m, 2H, CH_2), 3.39 (t, *J* = 6.7 Hz, 2H, CH_2), 3.42 (s, 2H, CH_2), 3.53 (br, 3H, 3xOH), 3.67 (s, 6H, 3xCH_2); **^{13}C NMR:** δ = 14.2 (CH_3), 22.8 (CH_2), 26.2 (CH_2), 29.5 (CH_2), 29.6 (2xCH_2), 29.7 (2xCH_2), 29.8 (2xCH_2), 32.0 (CH_2), 45.0 (C), 64.3 (3xCH_2), 72.3 (CH_2), 73.6 (CH_2). **GC:** retention time: 15.4 min.; MS (ESI+) calcd for $C_{17}H_{36}O_4$: $[M^+Na]^+$ = 327.2511; found: 327.2508.

[3] J. Berry, *J. Org. Chem.*, 1960, **25**, 1272–1274.

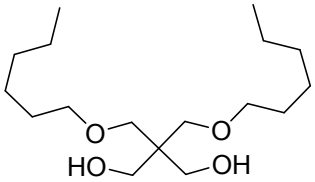
2,2-bis((pentyloxy)methyl)propane-1,3-diol **14a:**



Chemical Formula: $C_{15}H_{32}O_4$
Exact Mass: 276,2301

Colorless liquid. Eluent for isolation: 6:4 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.88 (t, *J* = 6.8 Hz, 6H, 2xCH_3), 1.22-1.32 (m, 8H, 4xCH_2), 1.45-1.55 (m, 4H, 2xCH_2), 2.99 (s, 2H, 2xOH), 3.39 (t, *J* = 6.5 Hz, 4H, 2xCH_2), 3.47 (s, 4H, 2xCH_2), 3.62 (s, 4H, 2xCH_2); **^{13}C NMR:** δ = 14.1 (2xCH_3), 22.6 (2xCH_2), 28.4 (2xCH_2), 29.3 (2xCH_2), 44.6 (C), 65.4 (2xCH_2), 72.1 (2xCH_2), 73.1 (2xCH_2). **GC:** retention time: 12.7 min.; MS (ESI+) calcd for $C_{21}H_{44}O_4$: $[M^+Na]^+$ = 299.2198; found: 299.2182.

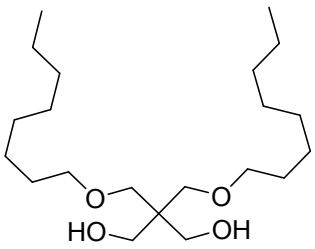
2,2-bis((hexyloxy)methyl)propane-1,3-diol **14b:**



Chemical Formula: $C_{17}H_{36}O_4$
Exact Mass: 304,2614

Colorless liquid. Eluent for isolation: 6:4 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.84 (t, *J* = 7.0 Hz, 6H, 2xCH_3), 1.26-1.33 (m, 12H, 6xCH_2), 1.48-1.57 (m, 4H, 2xCH_2), 3.01 (br, 2H, 2xOH), 3.37 (t, *J* = 6.6 Hz, 4H, 2xCH_2), 3.46 (s, 4H, 2xCH_2), 3.62 (s, 4H, 2xCH_2); **^{13}C NMR:** δ = 14.1 (2xCH_3), 22.7 (2xCH_2), 25.8 (2xCH_2), 29.5 (2xCH_2), 31.7 (2xCH_2), 44.6 (C), 65.2 (2xCH_2), 72.1 (2xCH_2), 73.0 (2xCH_2). **GC:** retention time: 13.9 min.; MS (ESI+) calcd for $C_{17}H_{36}O_4$: $[M^+Na]^+$ = 327.2511; found: 327.2508.

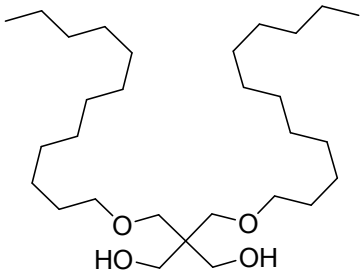
2,2-bis((octyloxy)methyl)propane-1,3-diol **14c:**



Chemical Formula: $C_{21}H_{44}O_4$
Exact Mass: 360,3240

Colorless liquid. Eluent for isolation: 8:2 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.85 (t, *J* = 6.7 Hz, 6H, 2xCH_3), 1.22-1.32 (m, 20H, 10xCH_2), 1.45-1.55 (m, 4H, 2xCH_2), 2.99 (s, 2H, 2xOH), 3.39 (t, *J* = 6.5 Hz, 4H, 2xCH_2), 3.47 (s, 4H, 2xCH_2), 3.62 (s, 4H, 2xCH_2); **^{13}C NMR:** δ = 14.2 (2xCH_3), 22.7 (2xCH_2), 26.2 (2xCH_2), 29.3 (2xCH_2), 29.5 (2xCH_2), 29.6 (2xCH_2), 29.8 (2xCH_2), 31.9 (2xCH_2), 44.6 (C), 65.3 (2xCH_2), 72.1 (2xCH_2), 73.0 (2xCH_2). **GC:** retention time: 15.5 min.; MS (ESI+) calcd for $C_{21}H_{44}O_4$: $[M^+Na]^+$ = 383.3137; found: 383.3119.

2,2-bis((dodecyloxy)methyl)propane-1,3-diol **14e:**

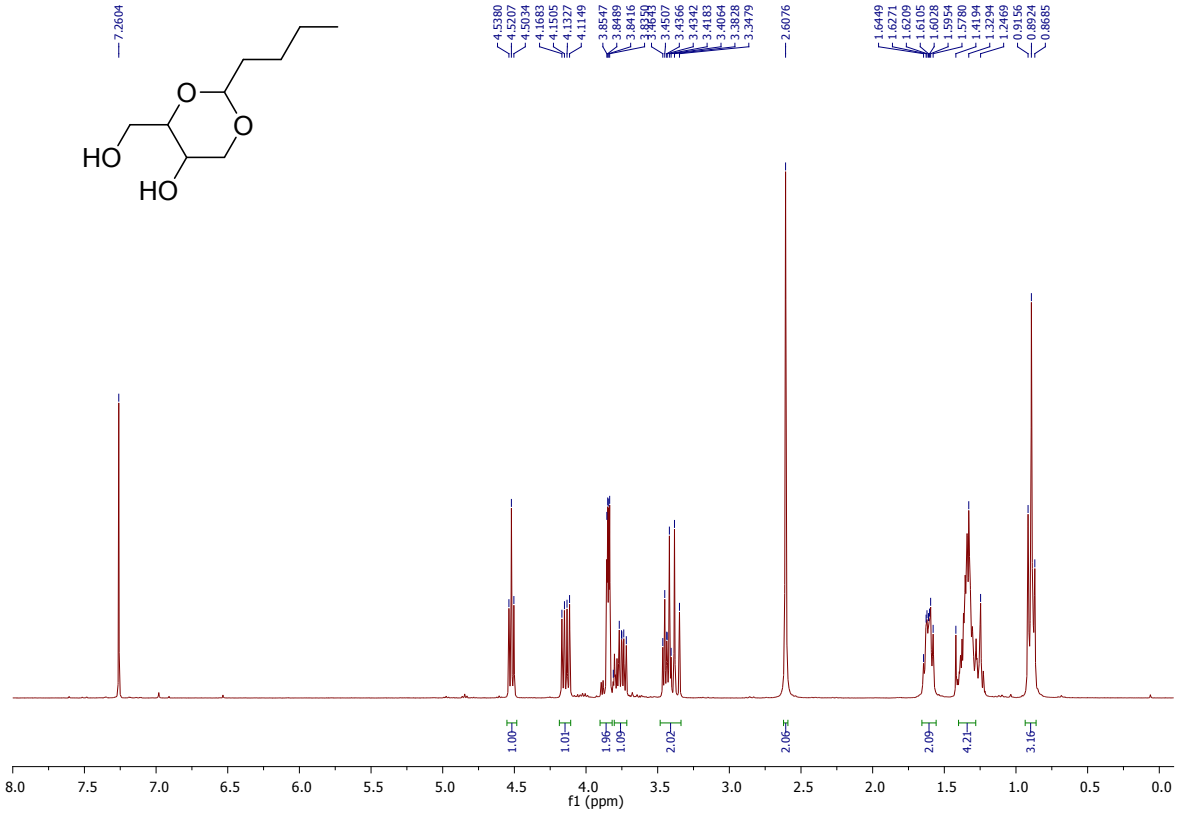


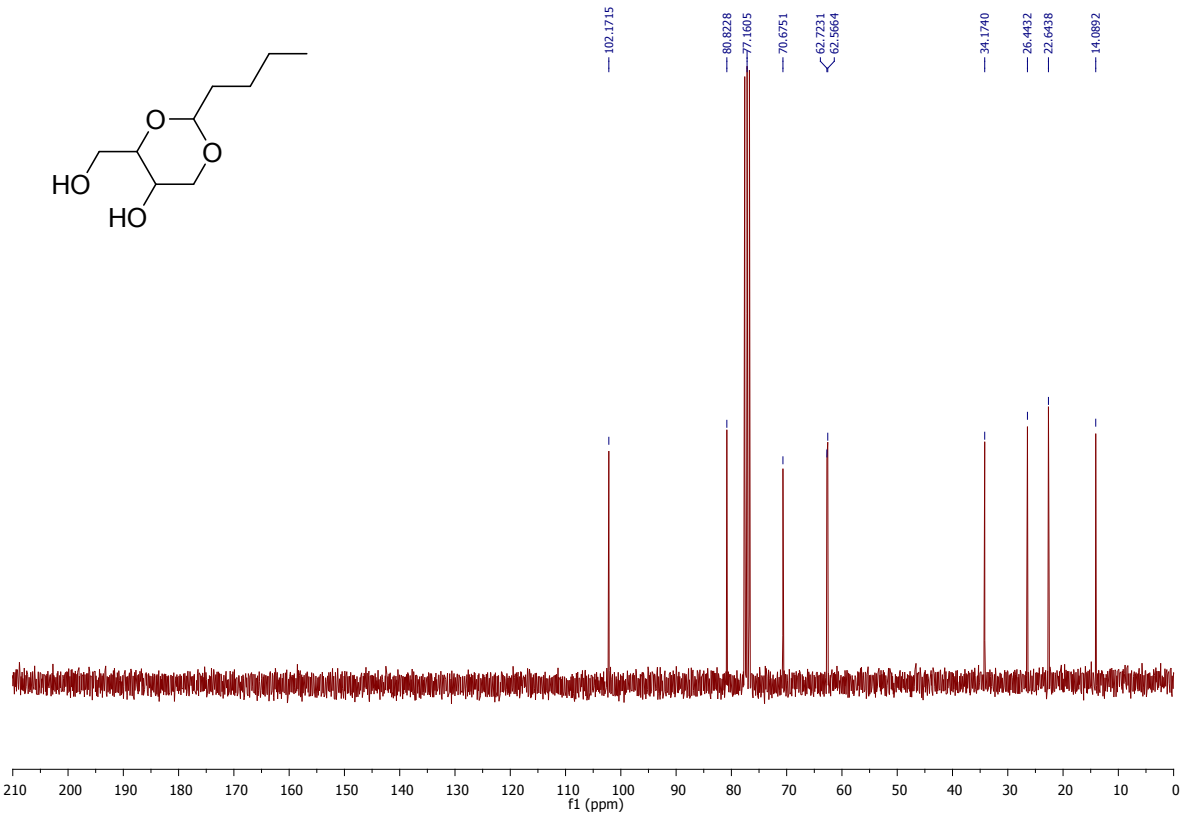
Chemical Formula: $C_{29}H_{60}O_4$
Exact Mass: 472,4492

White solid (Mp = 54-56°C). Eluent for isolation: 8:2 cyclohexane/EtOAc. **^{1}H NMR:** δ = 0.87 (t, *J* = 6.7 Hz, 6H, 2xCH_3), 1.23-1.31 (m, 36H, 18xCH_2), 1.50-1.57 (m, 4H, 2xCH_2), 2.79 (s, 2H, 2xOH), 3.41 (t, *J* = 6.5 Hz, 4H, 2xCH_2), 3.49 (s, 4H, 2xCH_2), 3.64 (s, 4H, 2xCH_2); **^{13}C NMR:** δ = 14.6 (2xCH_3), 22.8 (2xCH_2), 26.2 (2xCH_2), 29.5 (2xCH_2), 29.6 (2xCH_2), 29.7 (4xCH_2), 29.8 (6xCH_2), 32.1 (2xCH_2), 44.6 (C), 65.5 (2xCH_2), 72.2 (2xCH_2), 73.3 (2xCH_2). **GC:** retention time: 20.4 min.; MS (ESI+) calcd for $C_{29}H_{60}O_4$: $[M^+Na]^+$ = 495.4389; found: 495.4378.

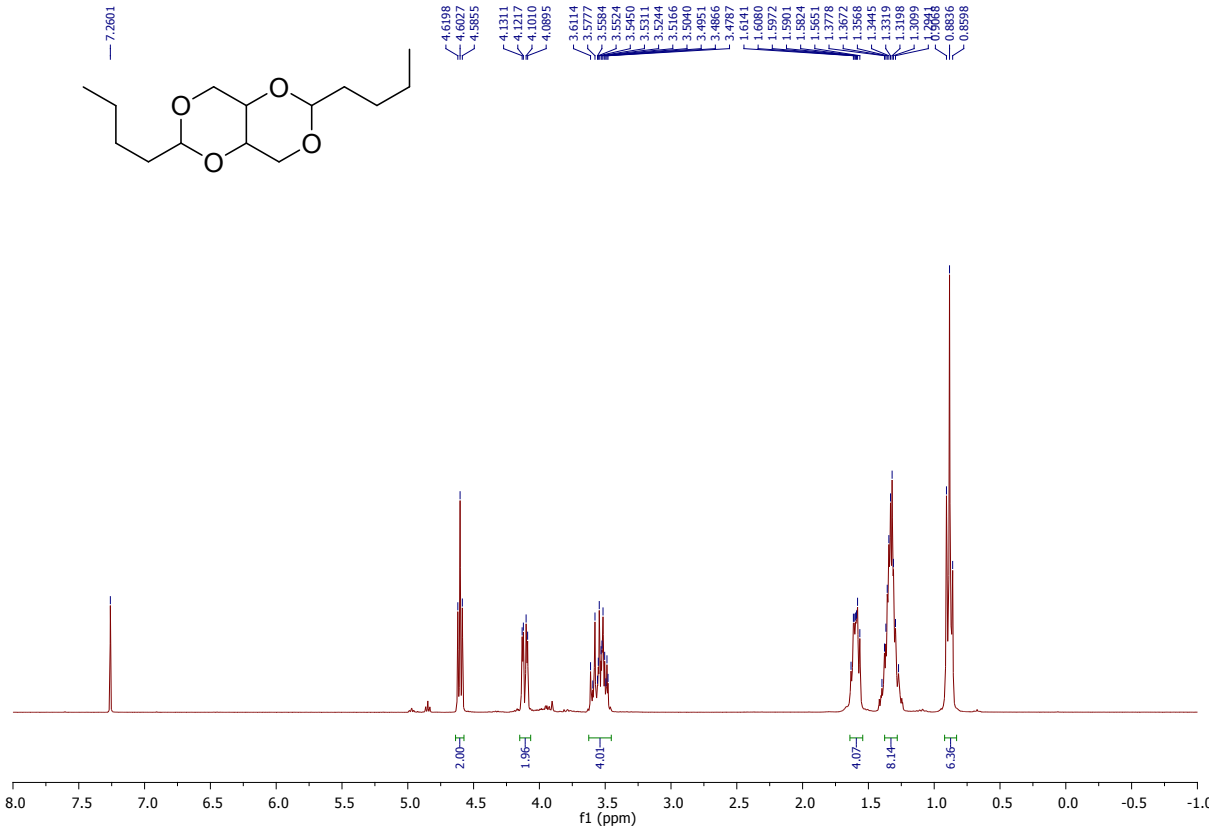
4. Spectra

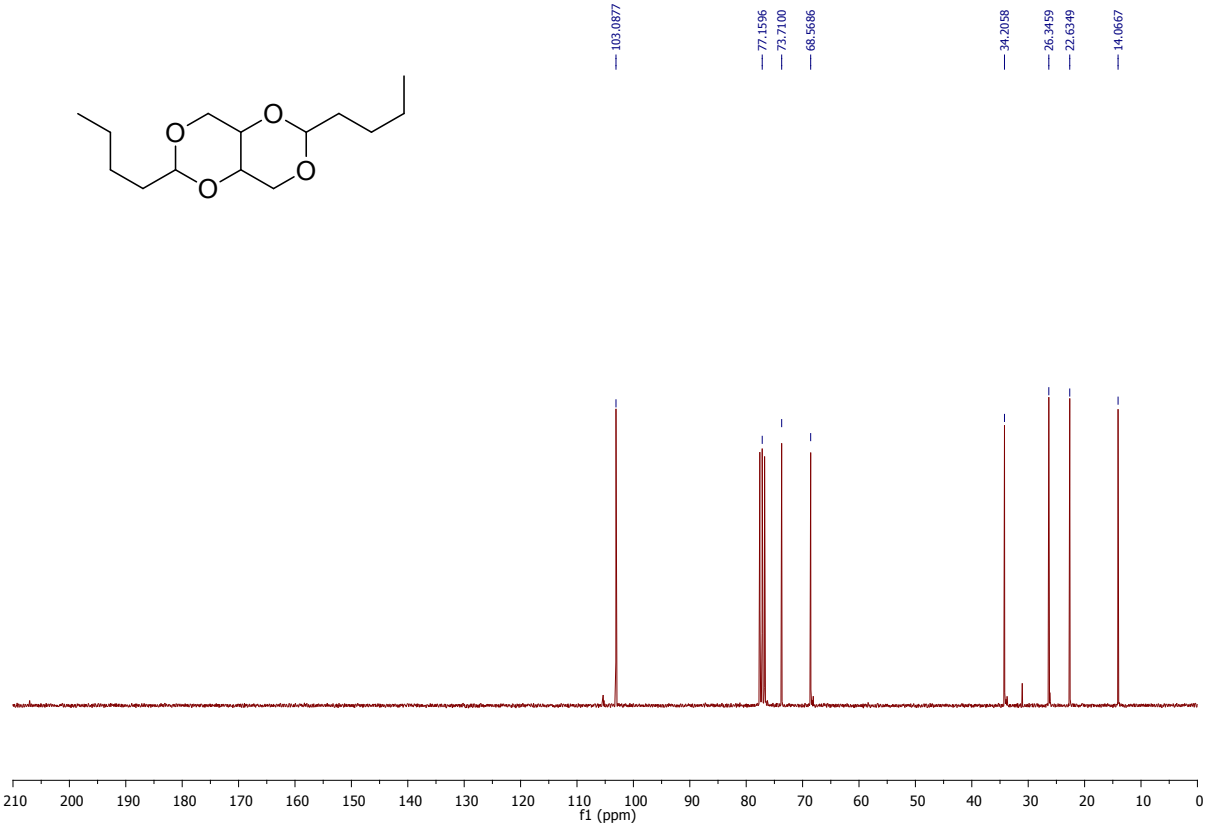
2-butyl-4-(hydroxymethyl)-1,3-dioxan-5-ol



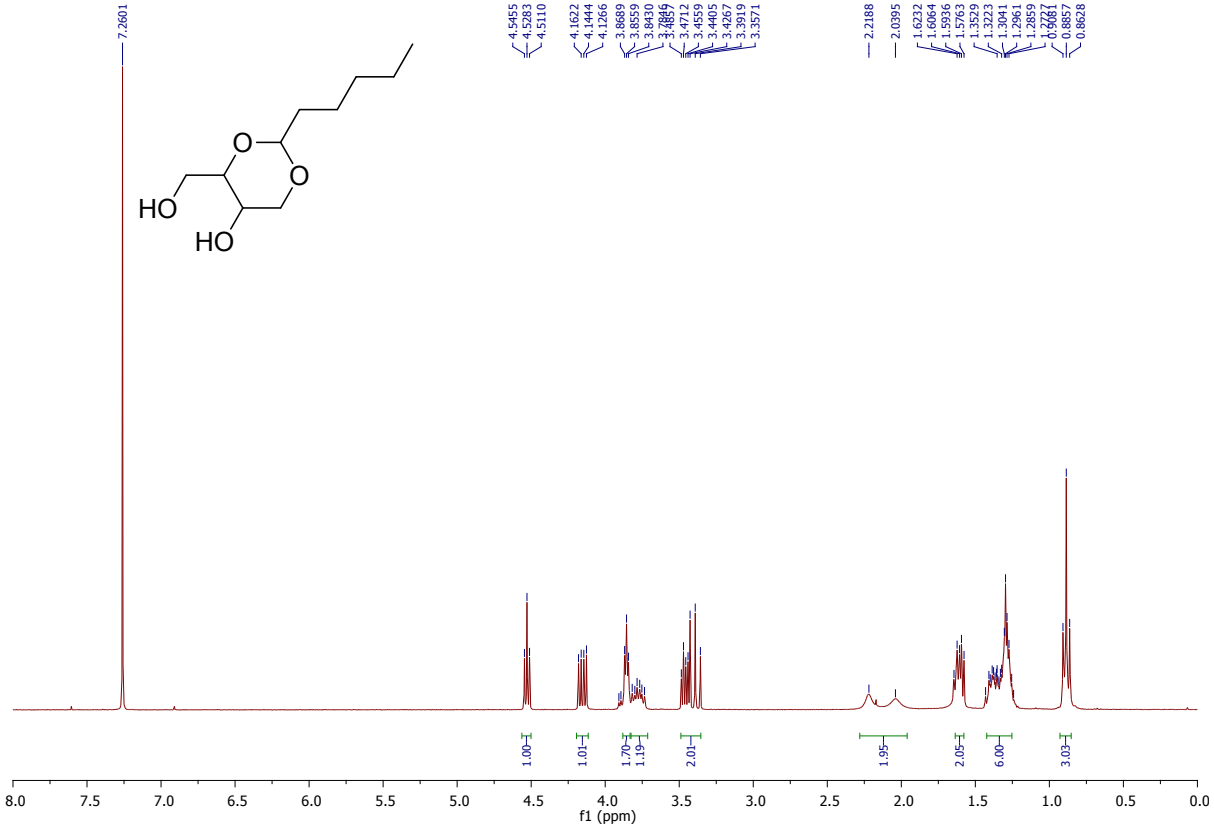


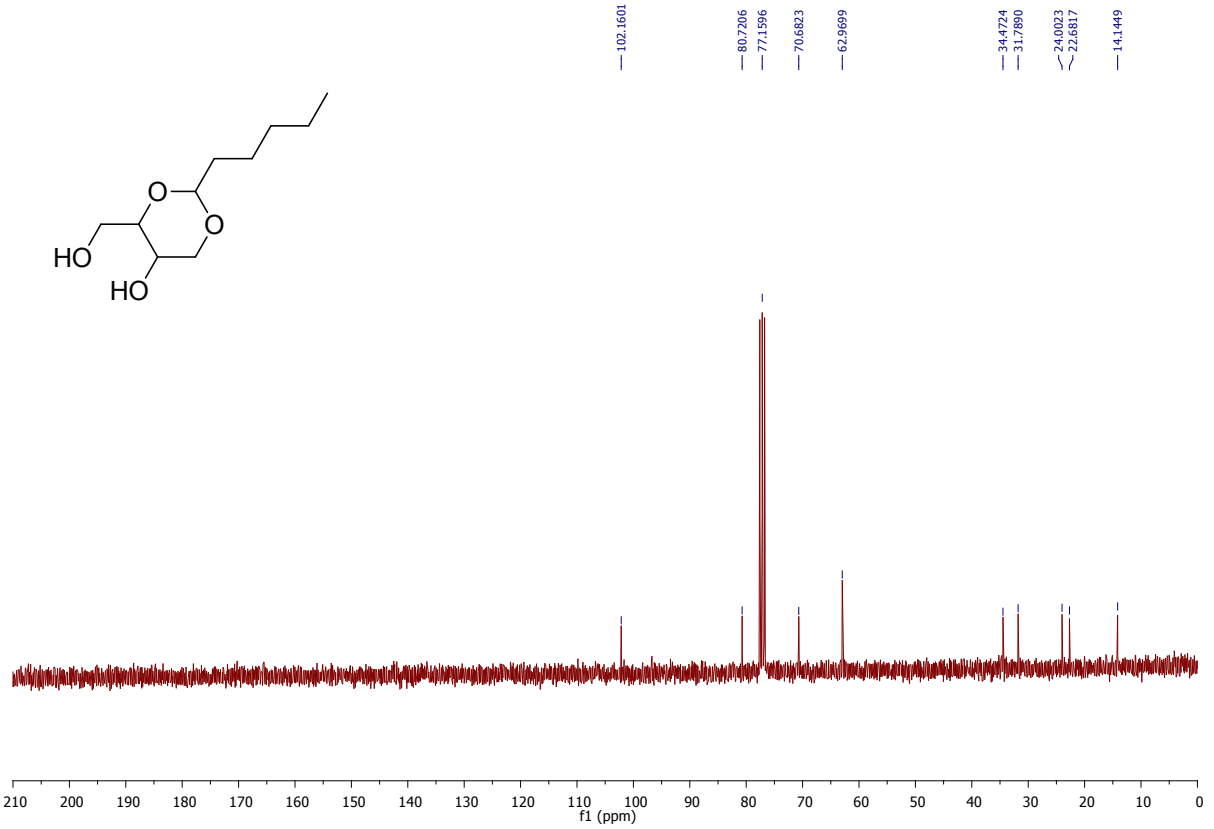
2,6-dibutyltetrahydro-[1,3]dioxino[5,4-d][1,3]dioxine



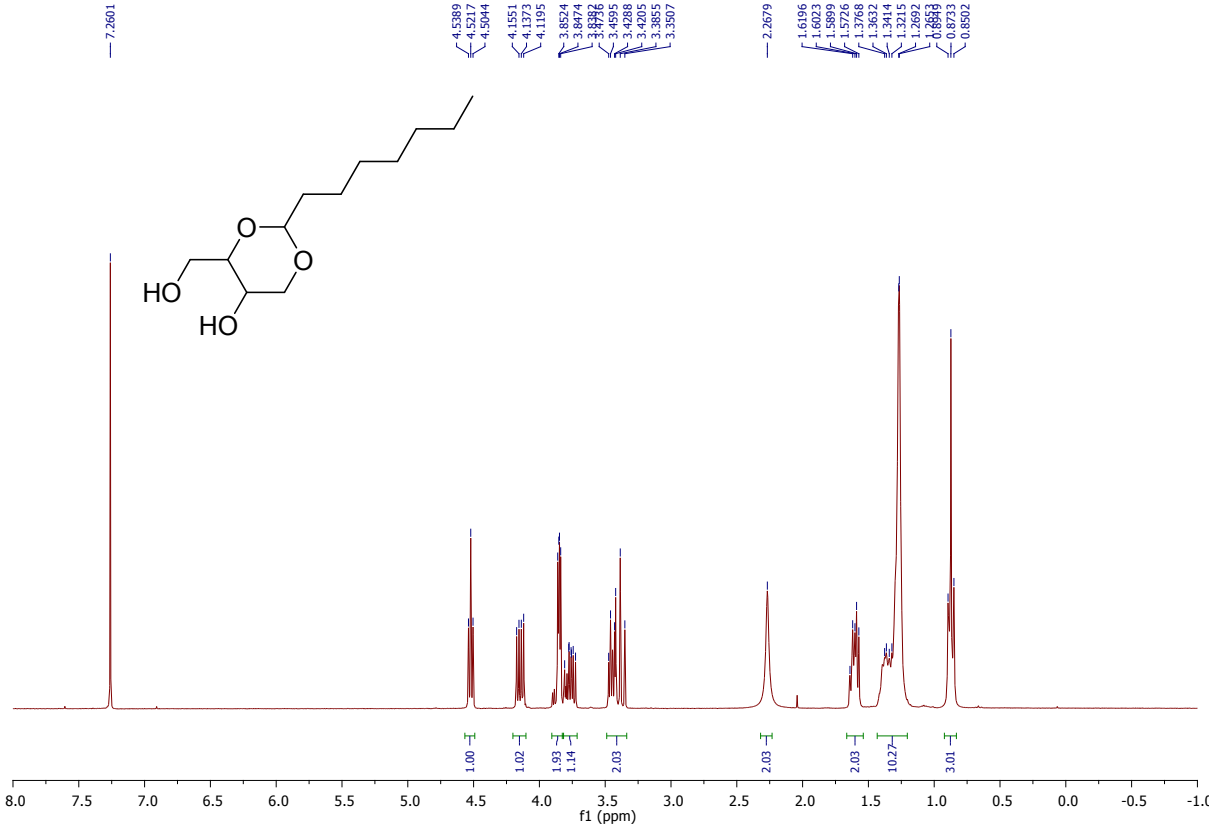


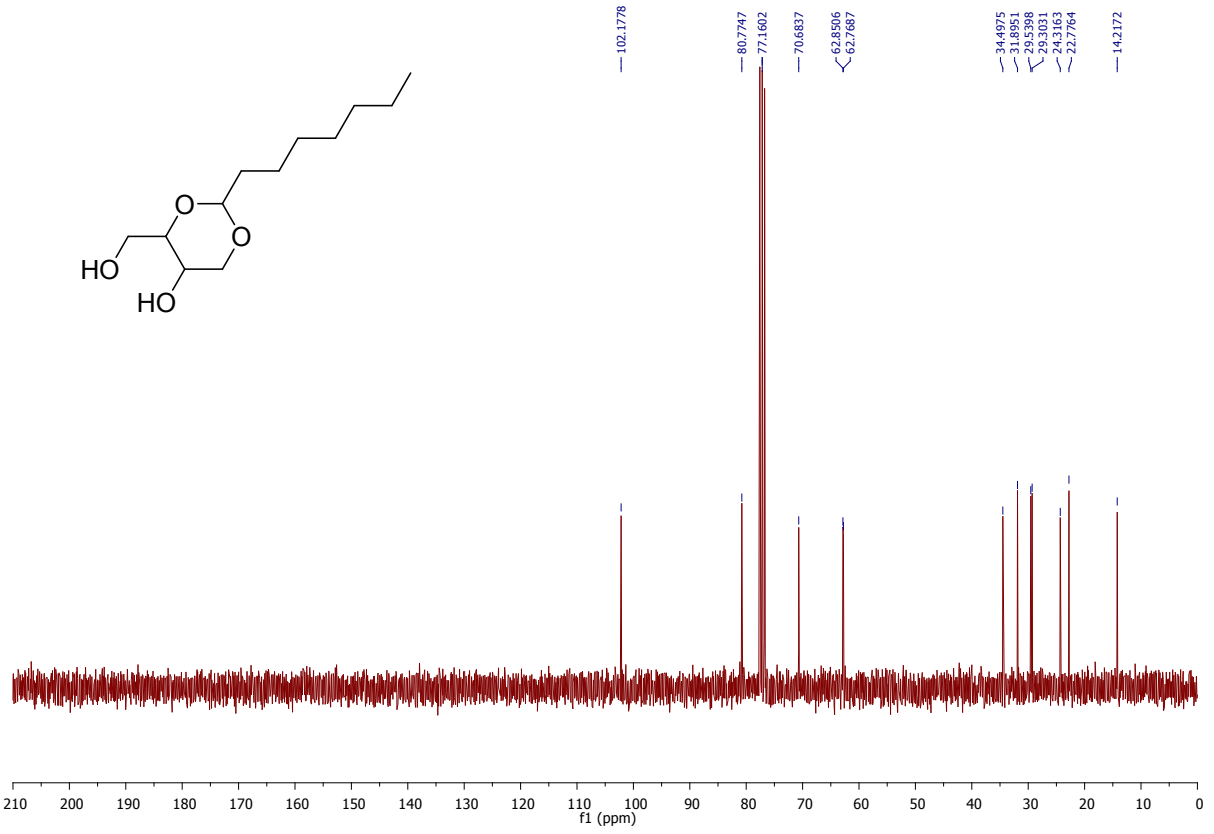
2-pentyl-4-(hydroxymethyl)-1,3-dioxan-5-ol



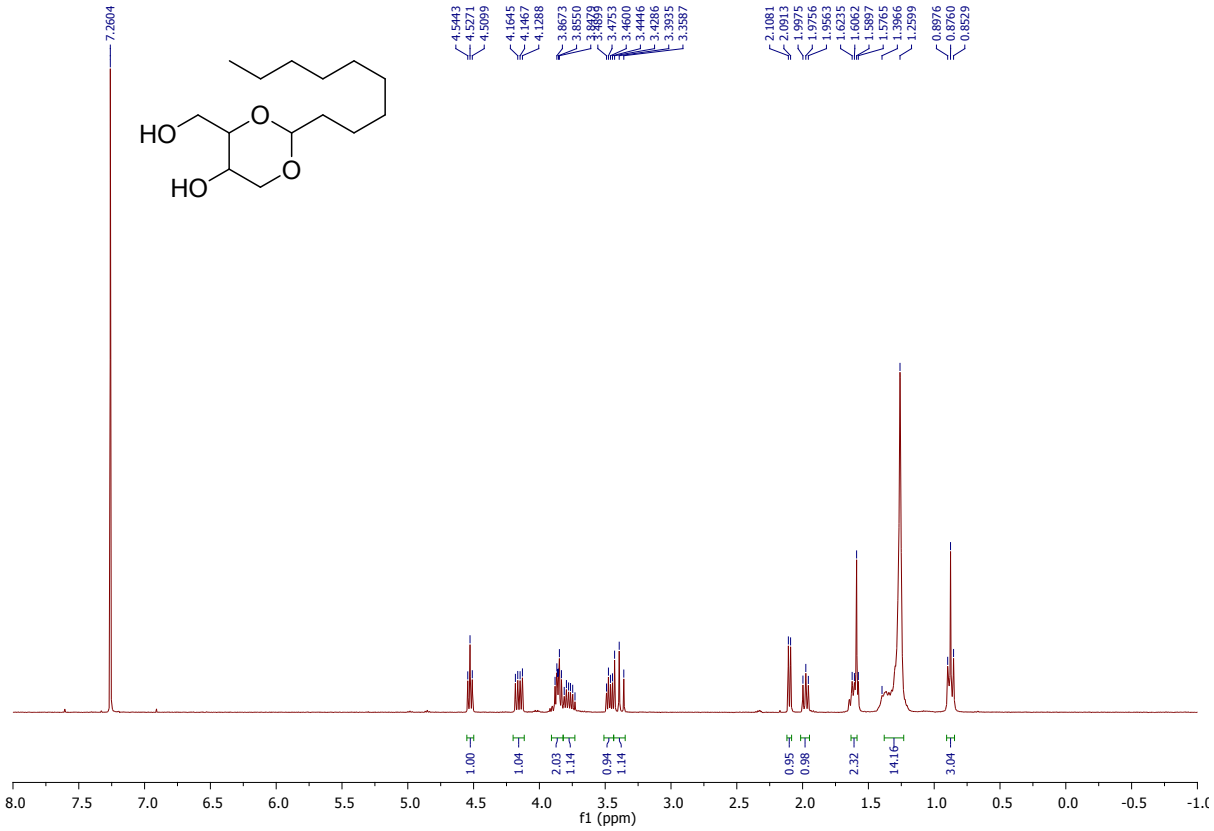


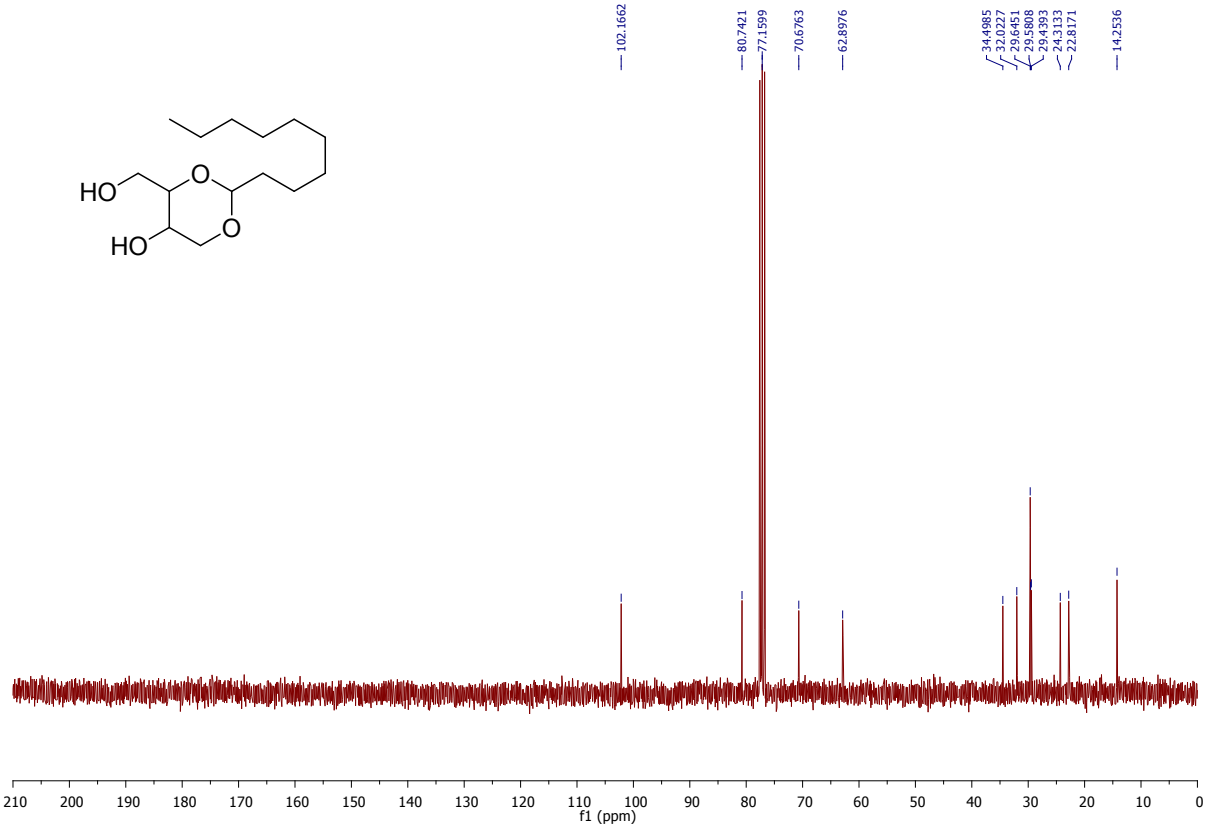
2-heptyl-4-(hydroxymethyl)-1,3-dioxan-5-ol



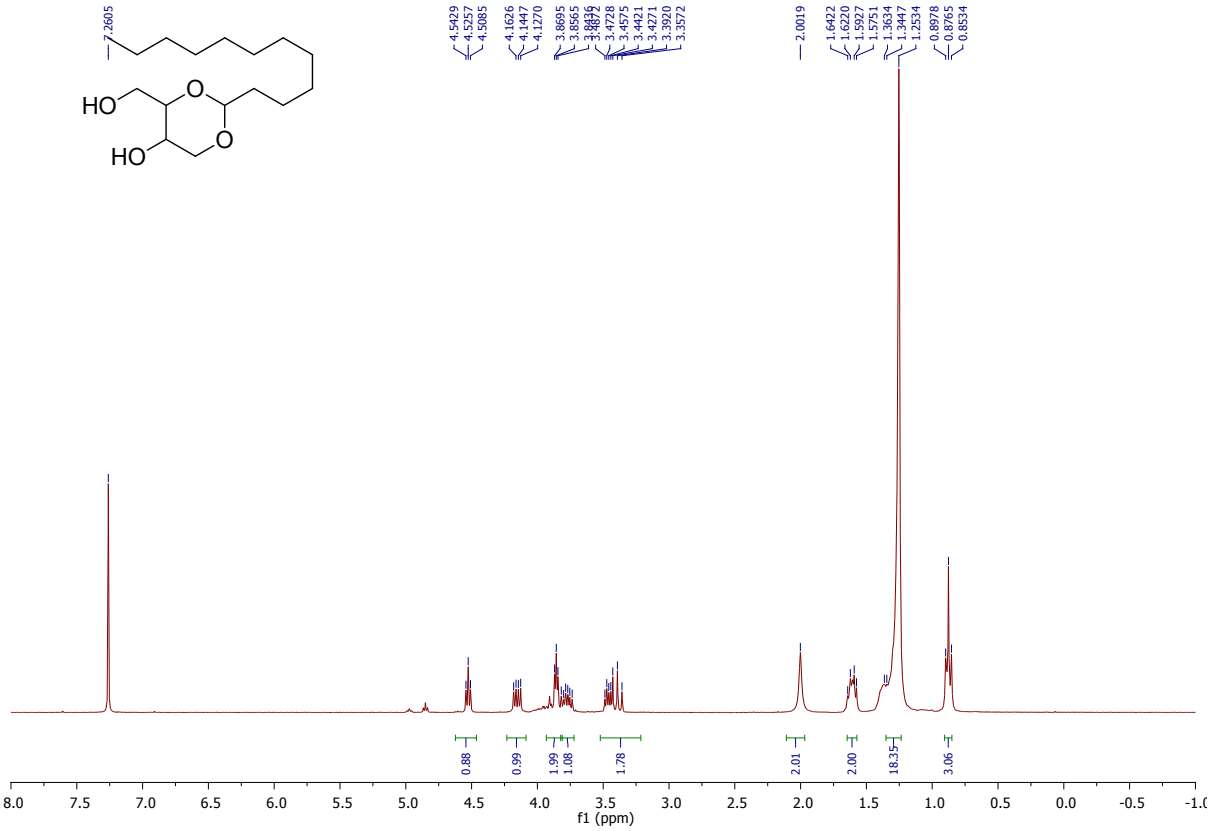


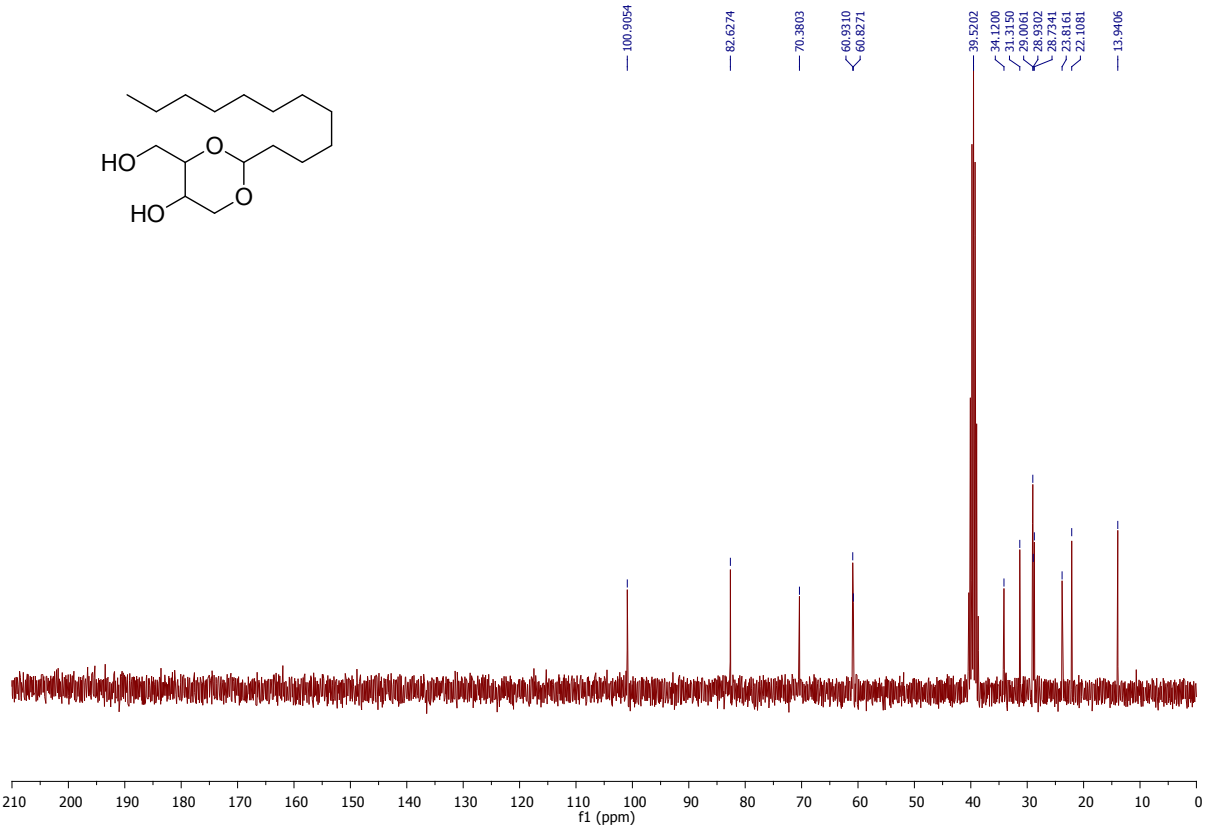
2-nonyl-4-(hydroxymethyl)-1,3-dioxan-5-ol



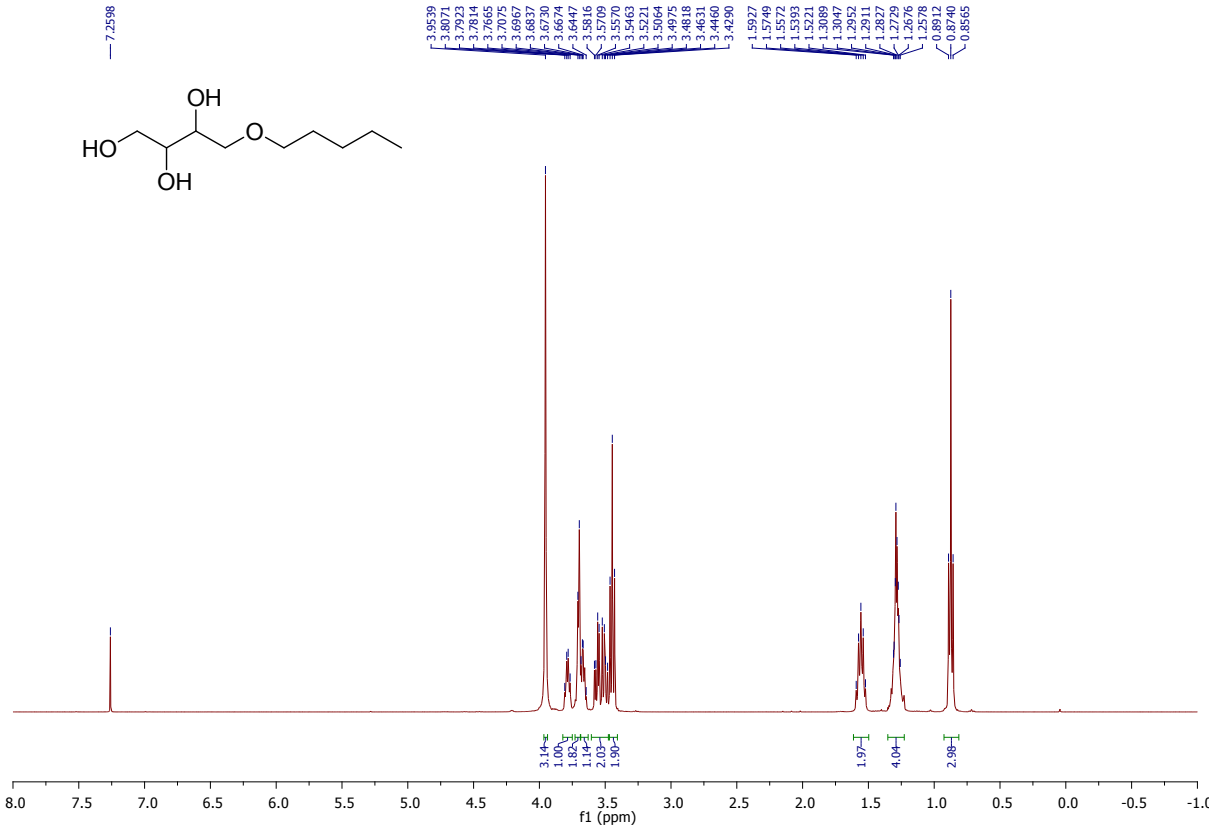


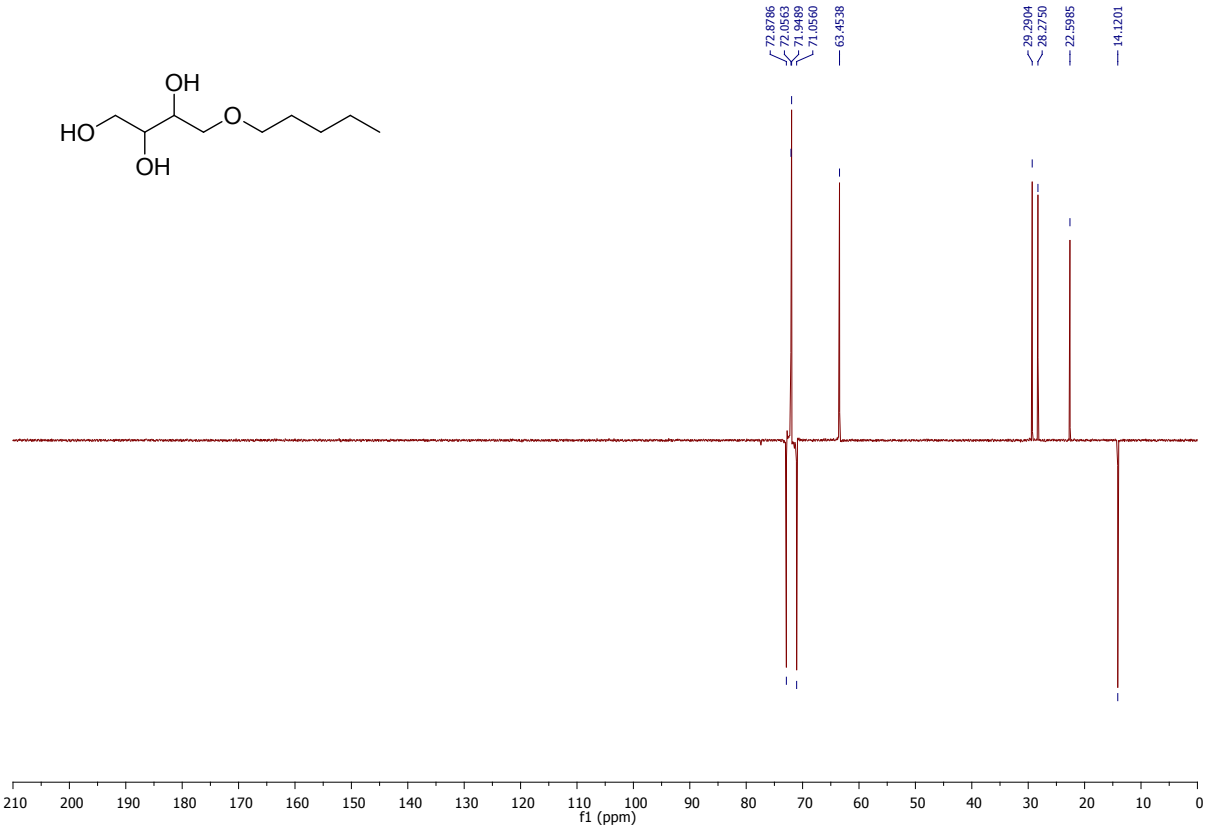
2-undecyl-4-(hydroxymethyl)-1,3-dioxan-5-ol



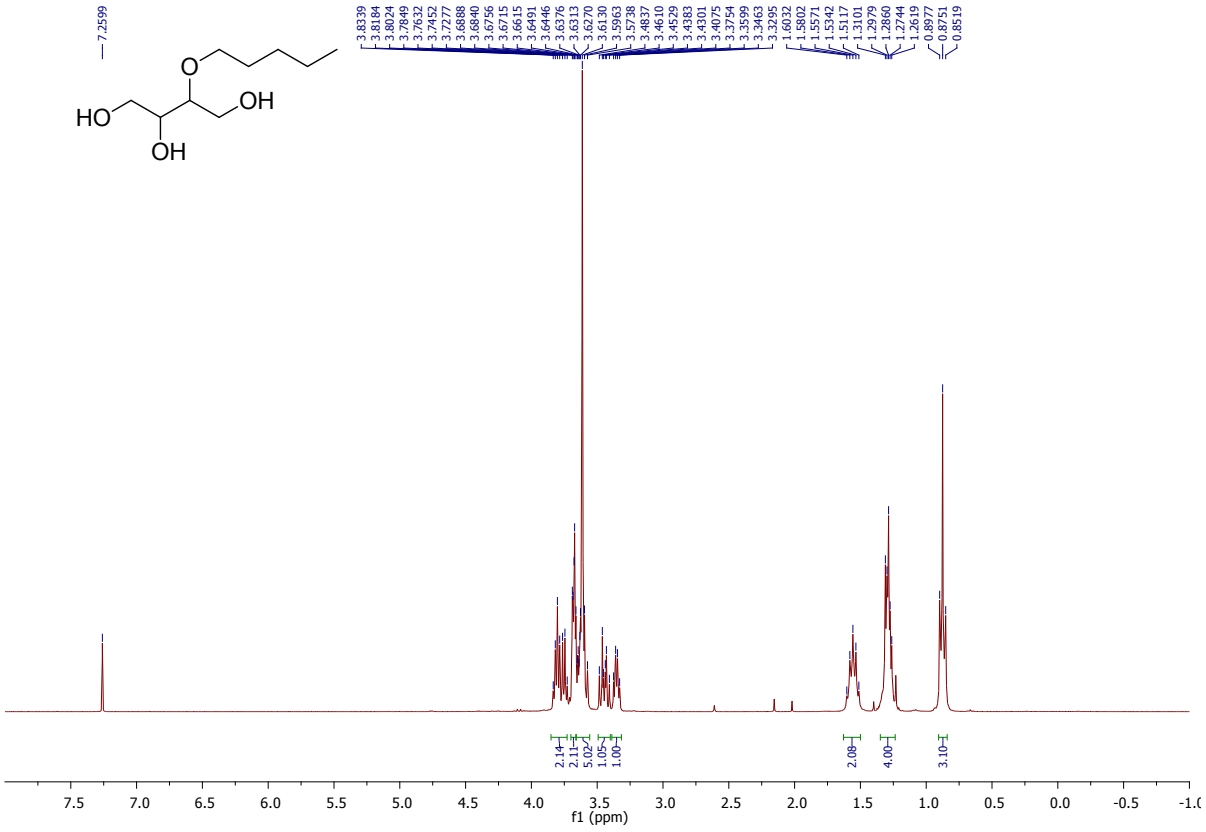


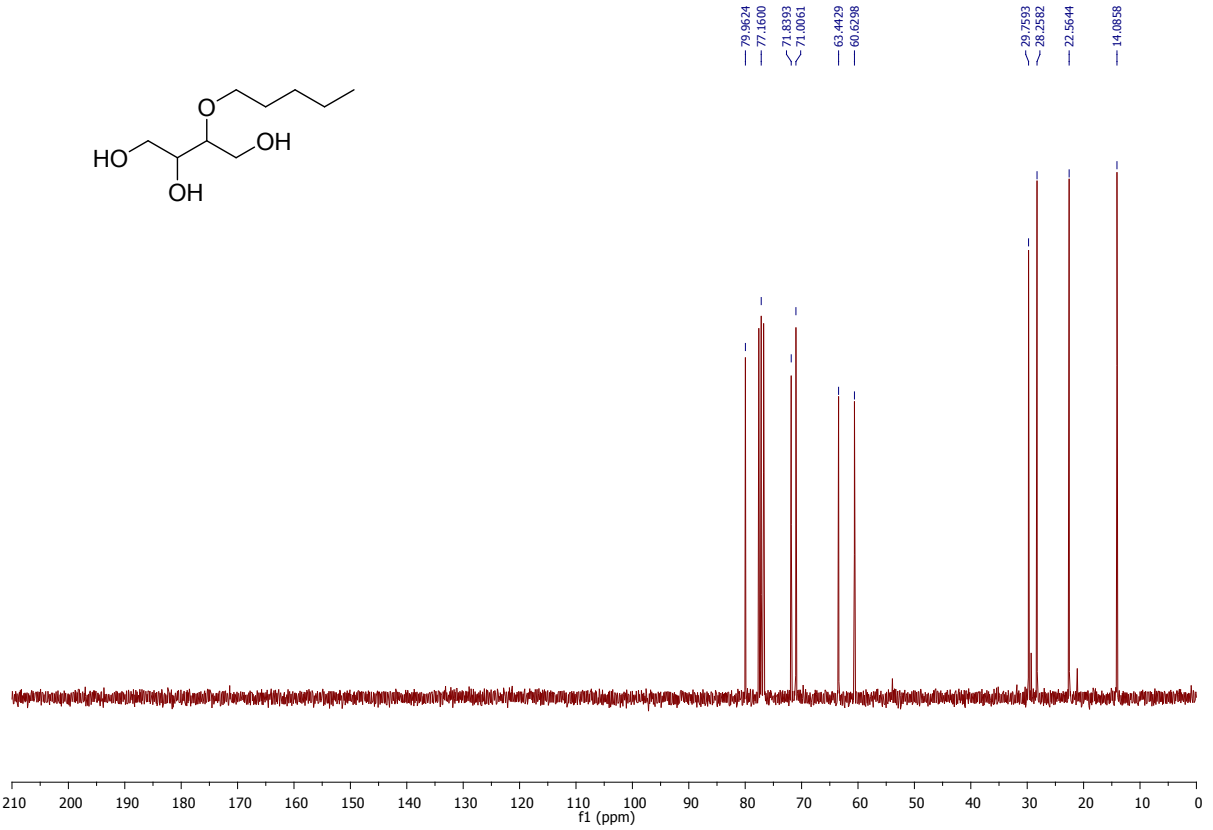
1-*O*-pentylerythritol



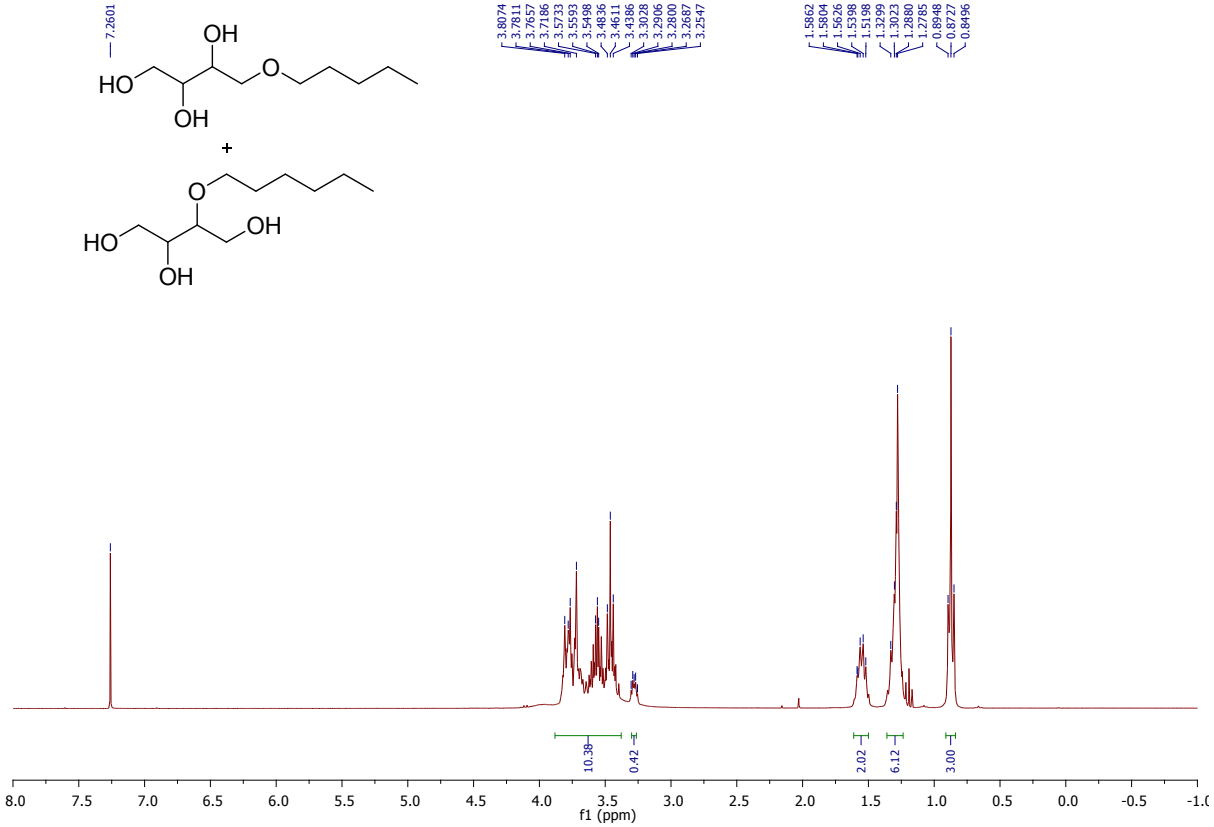


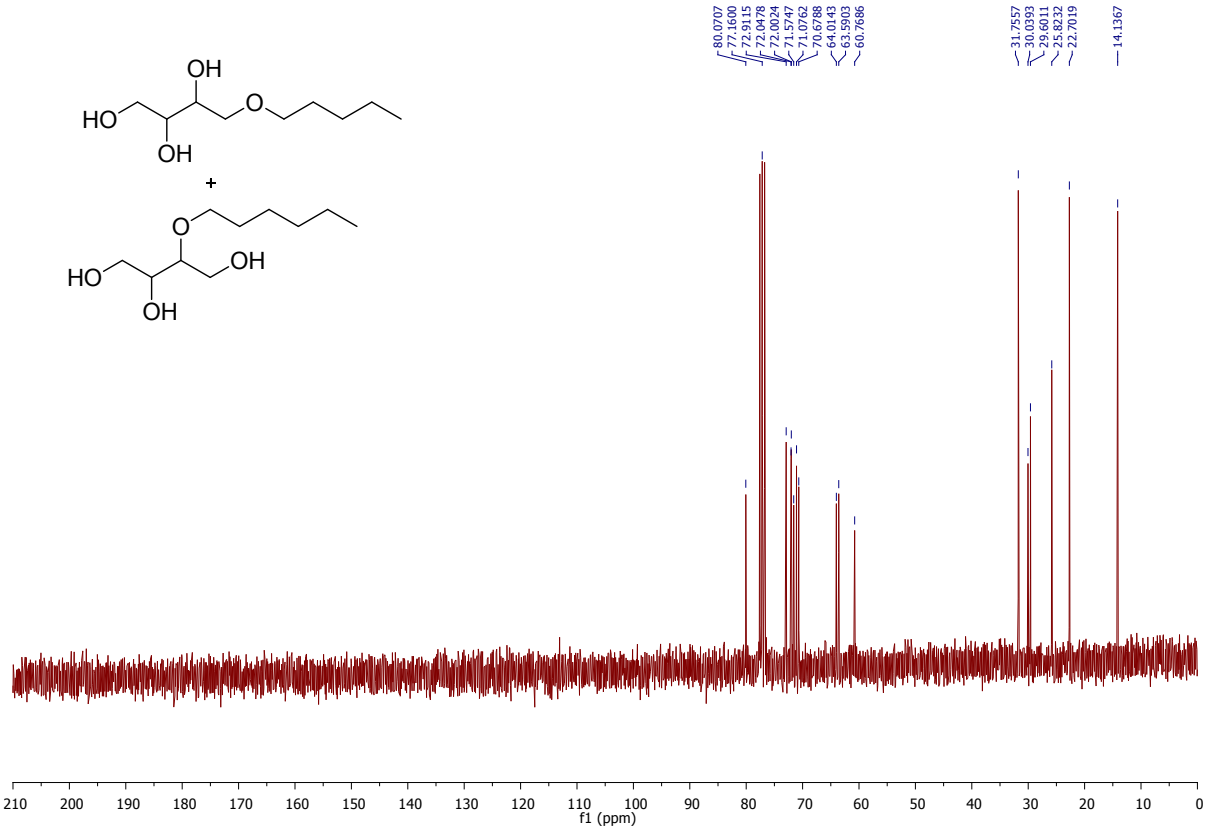
2-O-pentylerythritol



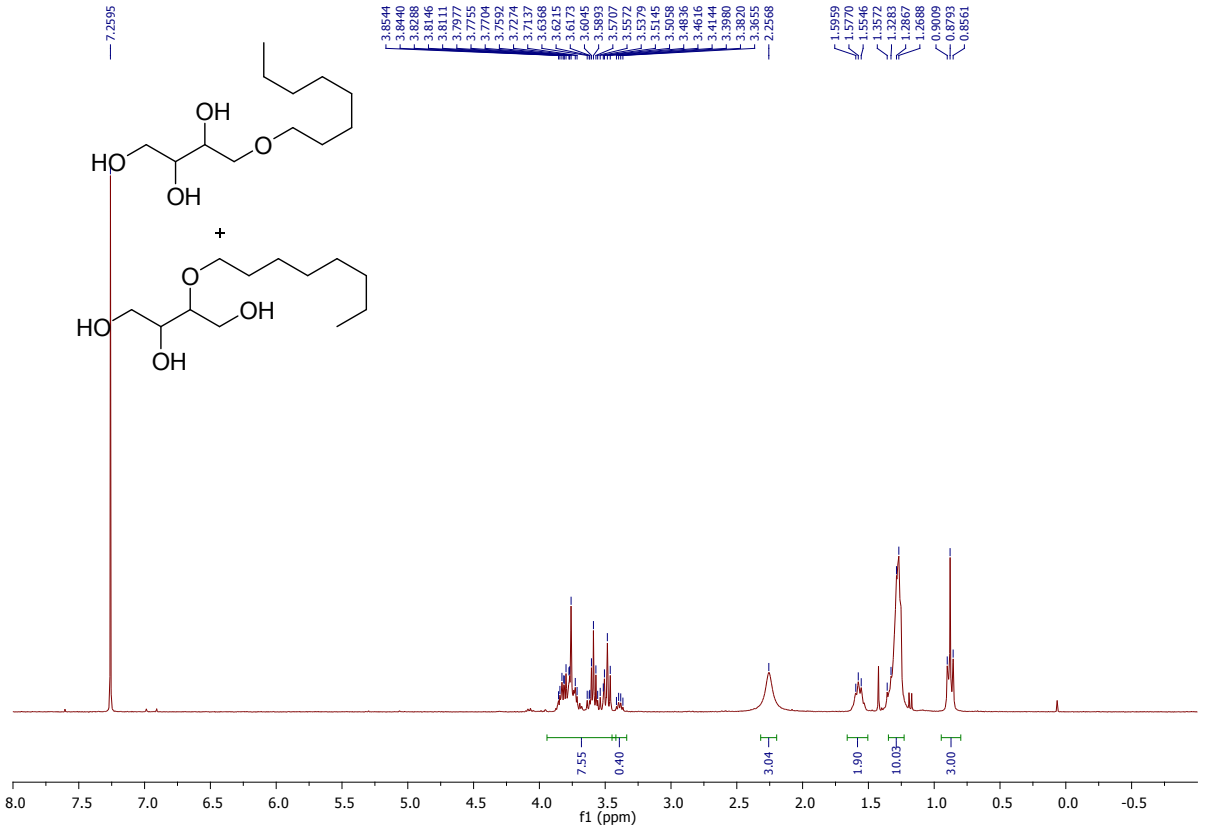


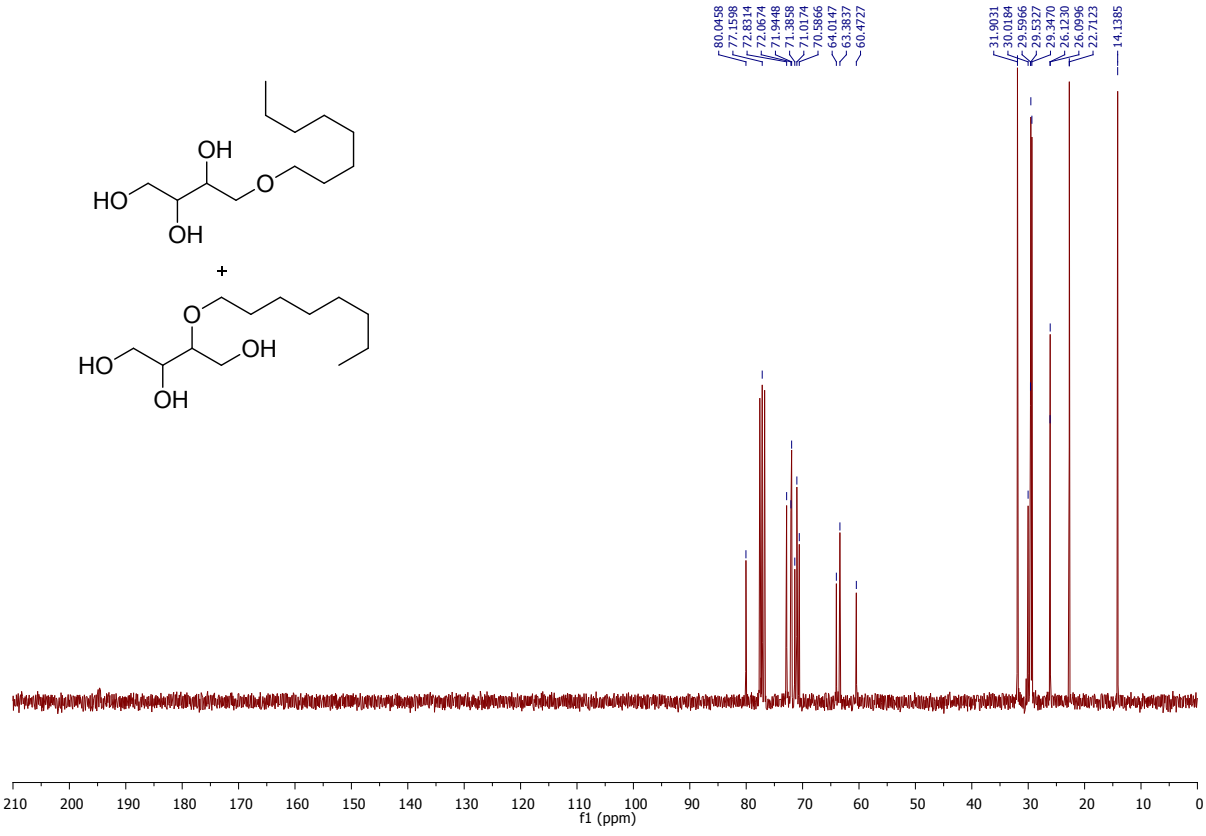
1-*O*-hexylerythritol + 2-*O*-hexylerythritol



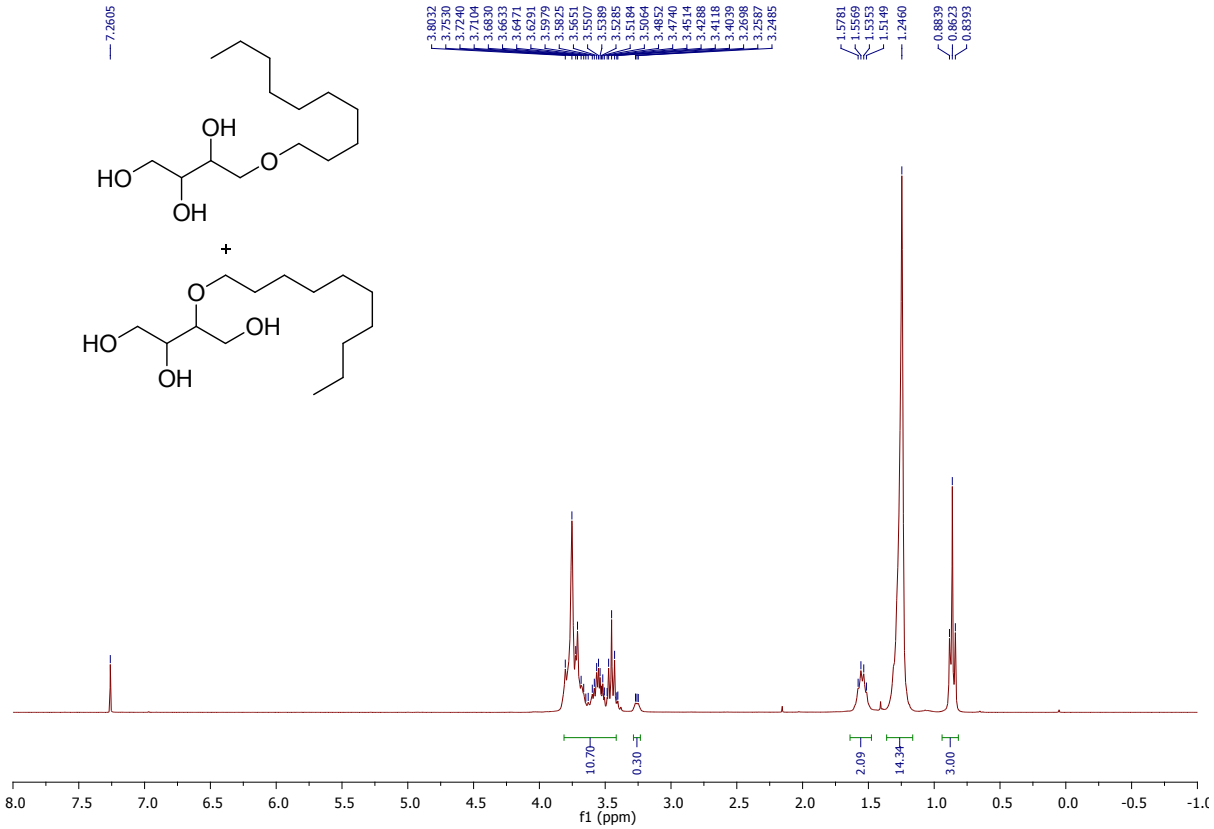


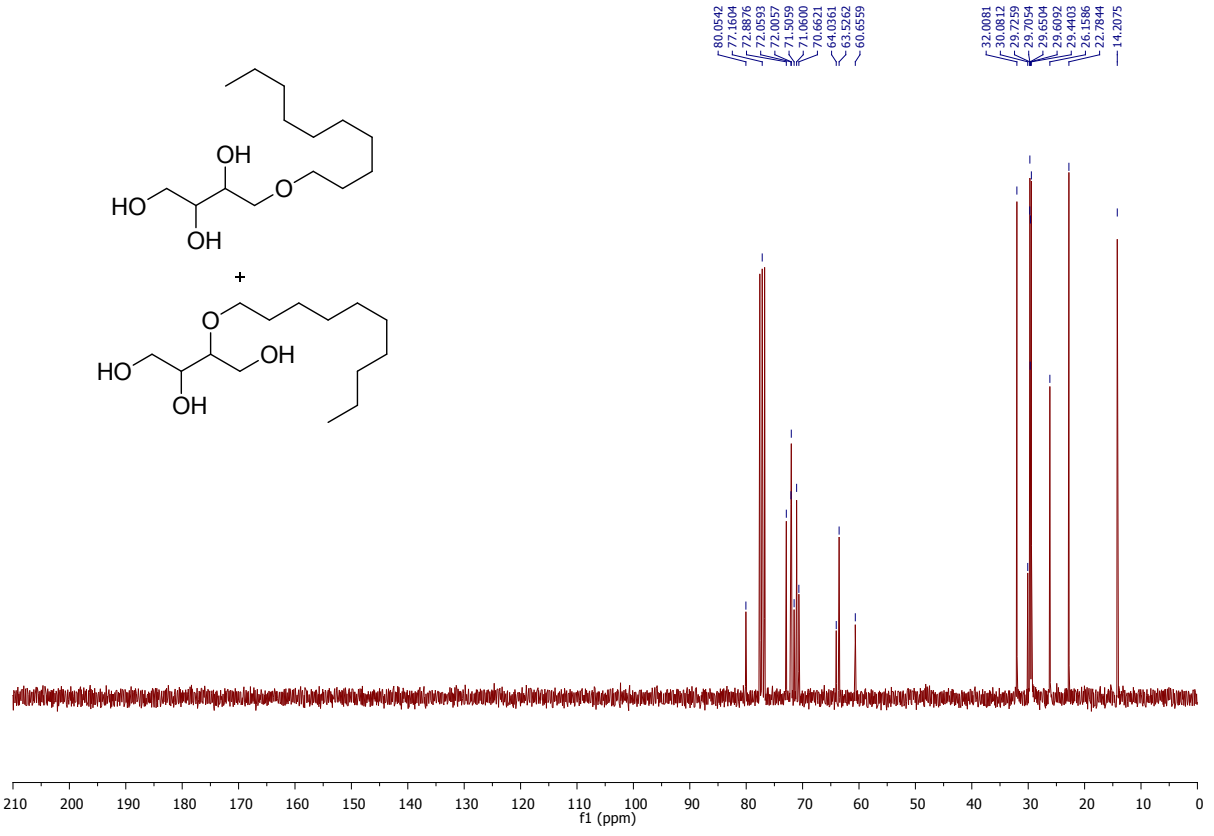
1-O-octylerythritol + 2-O-octylerythritol



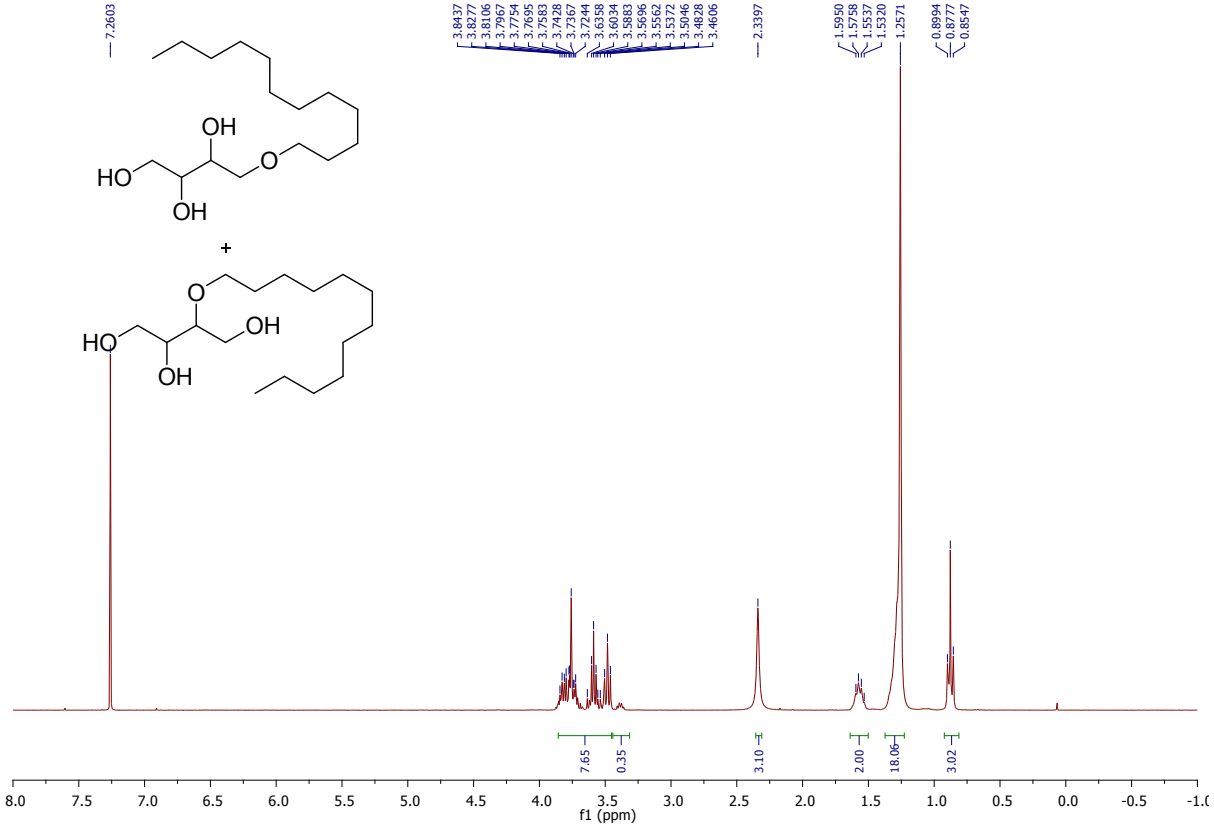


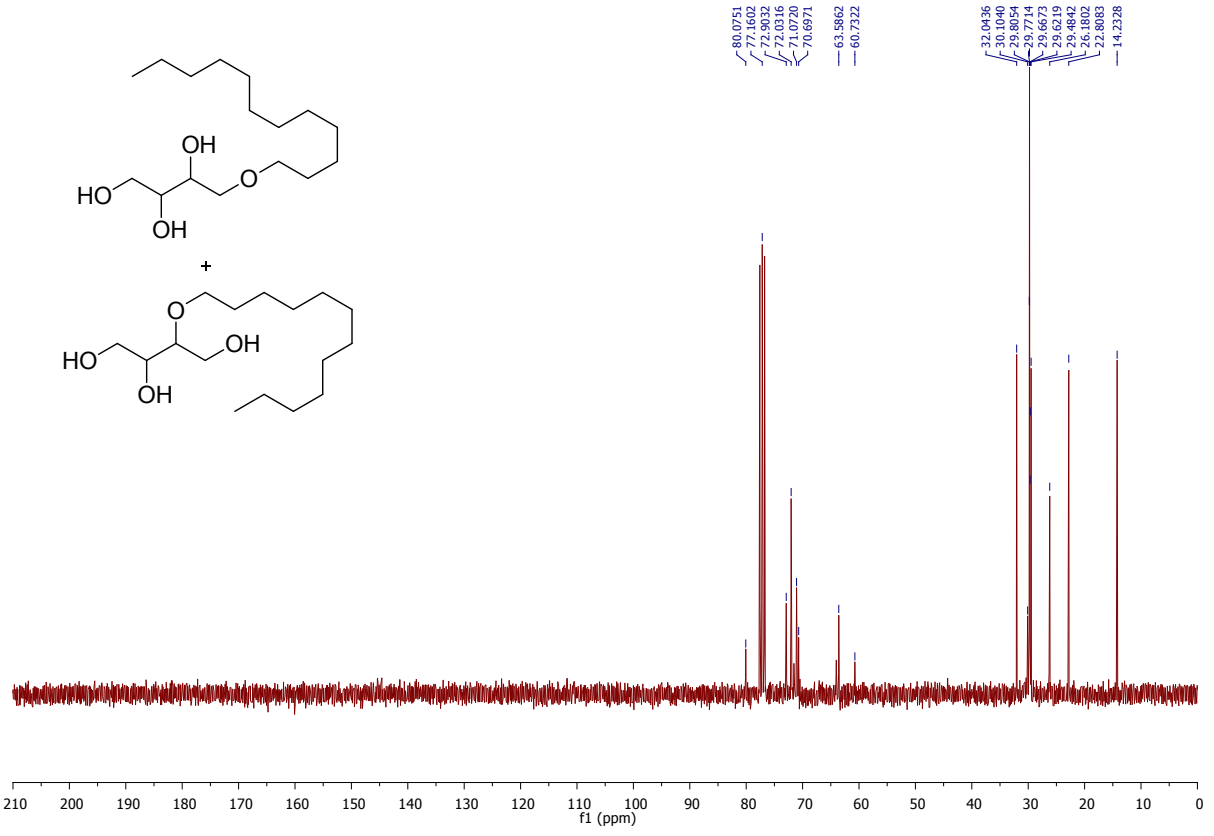
1-O-decylerythritol + 2-O-decylerythritol



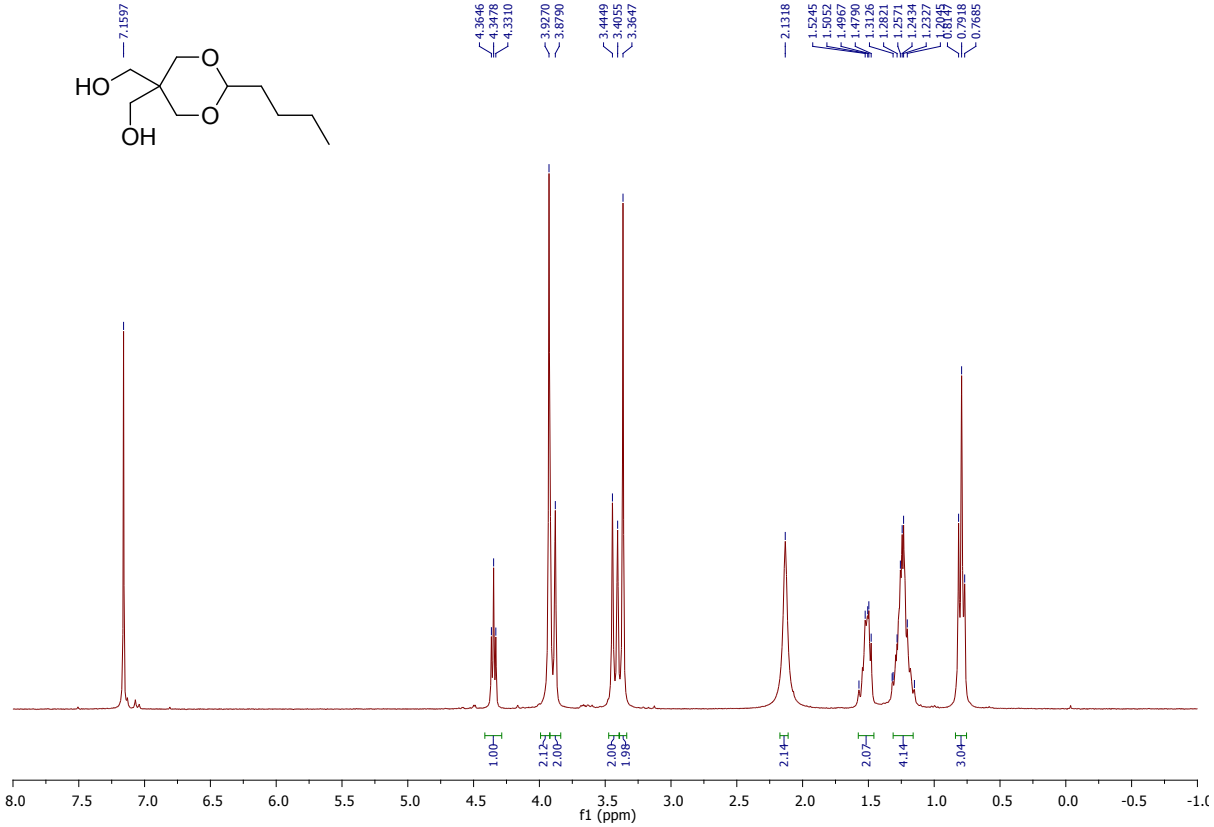


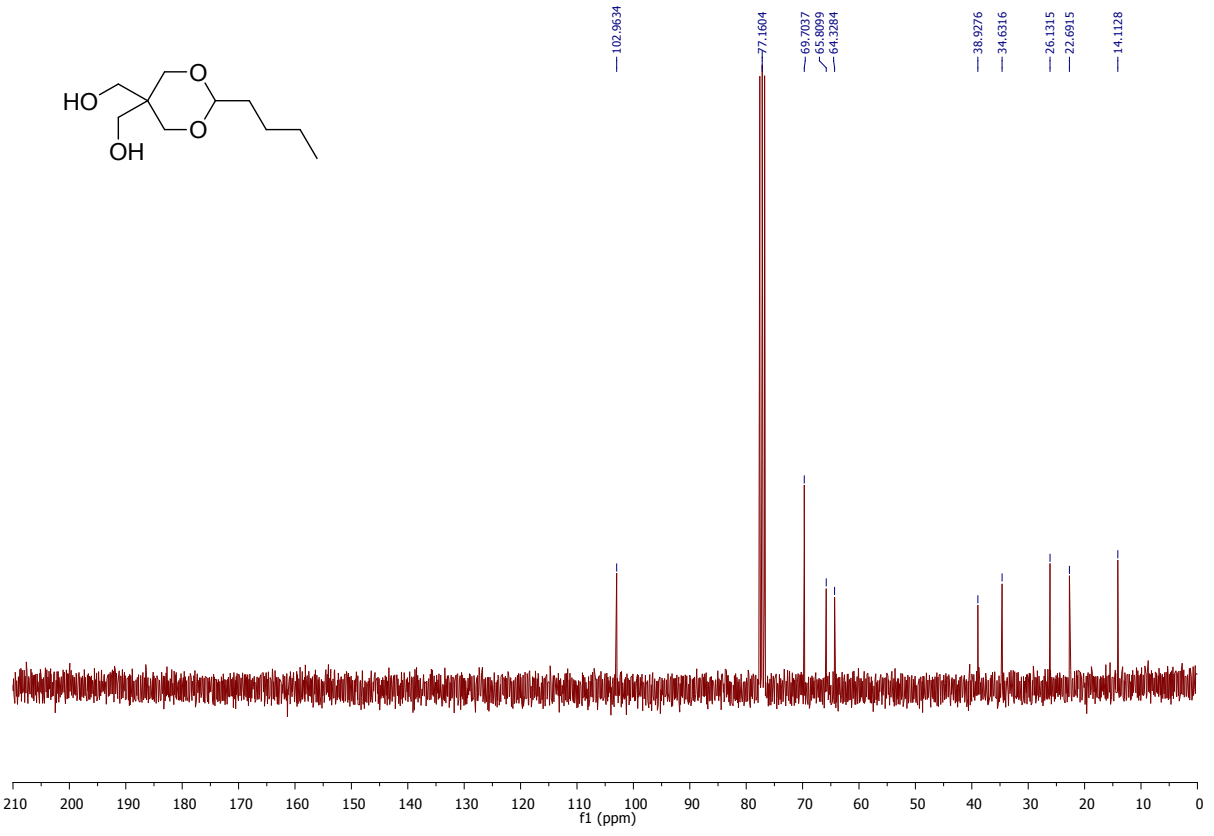
1-O-dodecylerythritol + 2-O-dodecylerythritol



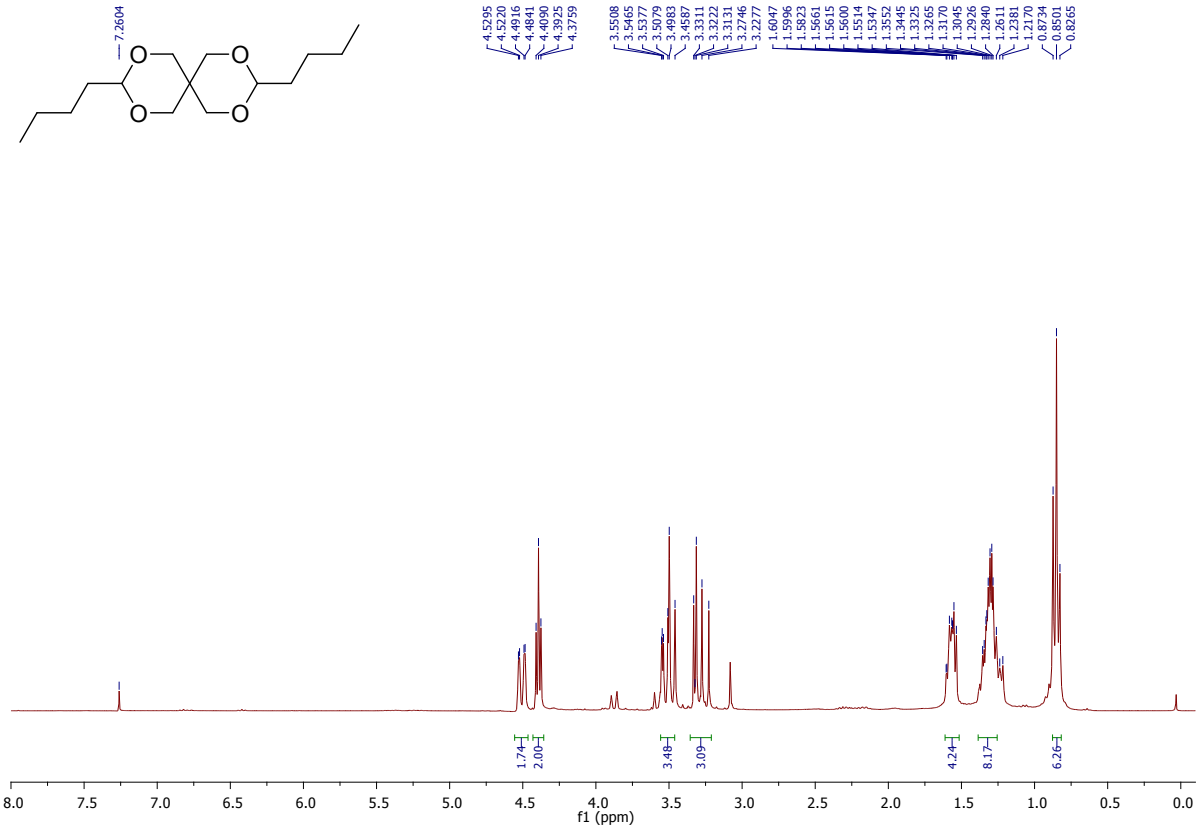


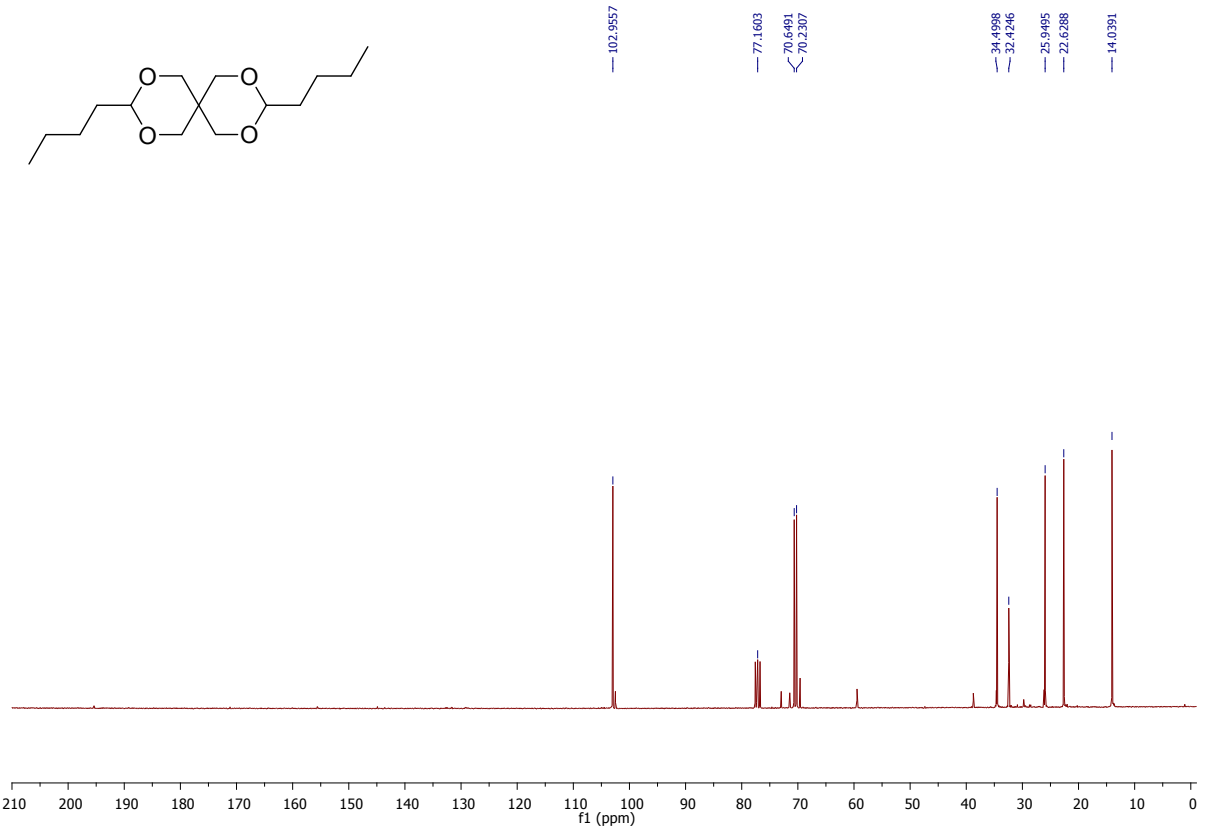
(2-butyl-1,3-dioxane-5,5-diyl)dimethanol



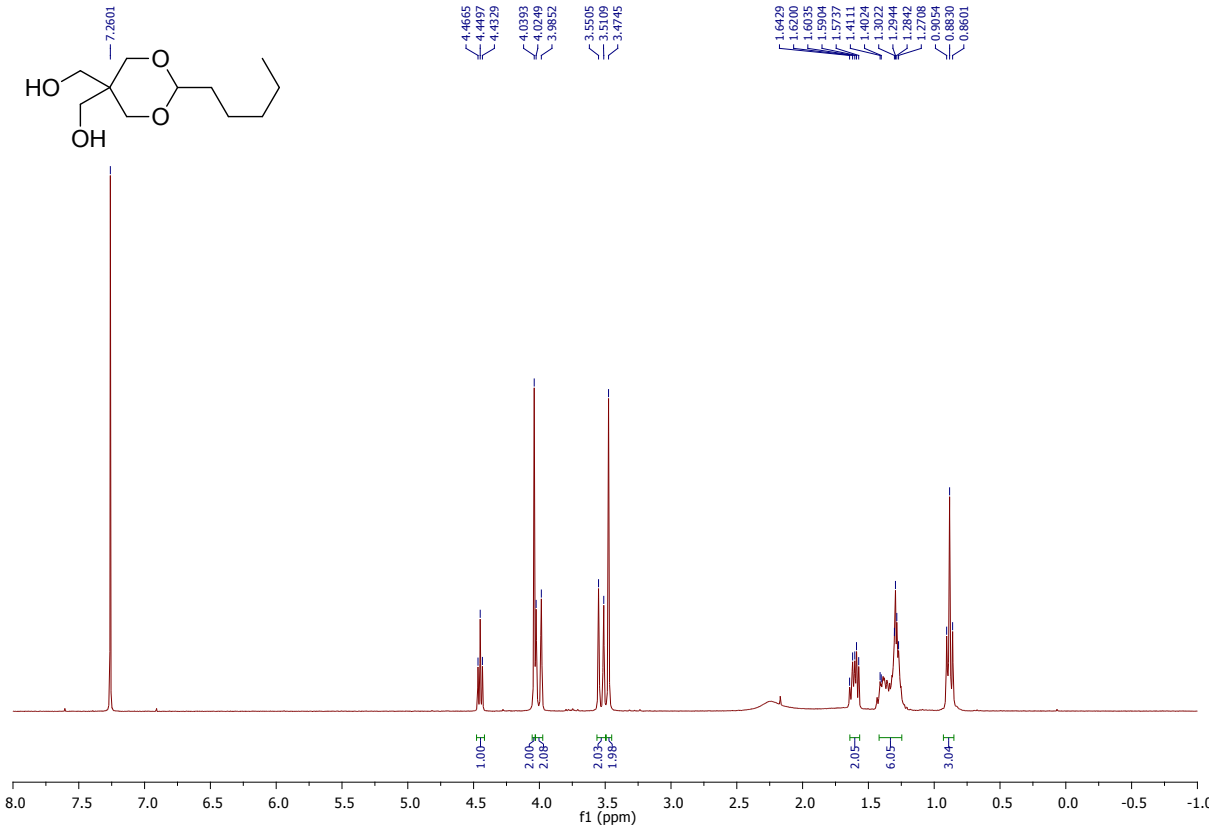


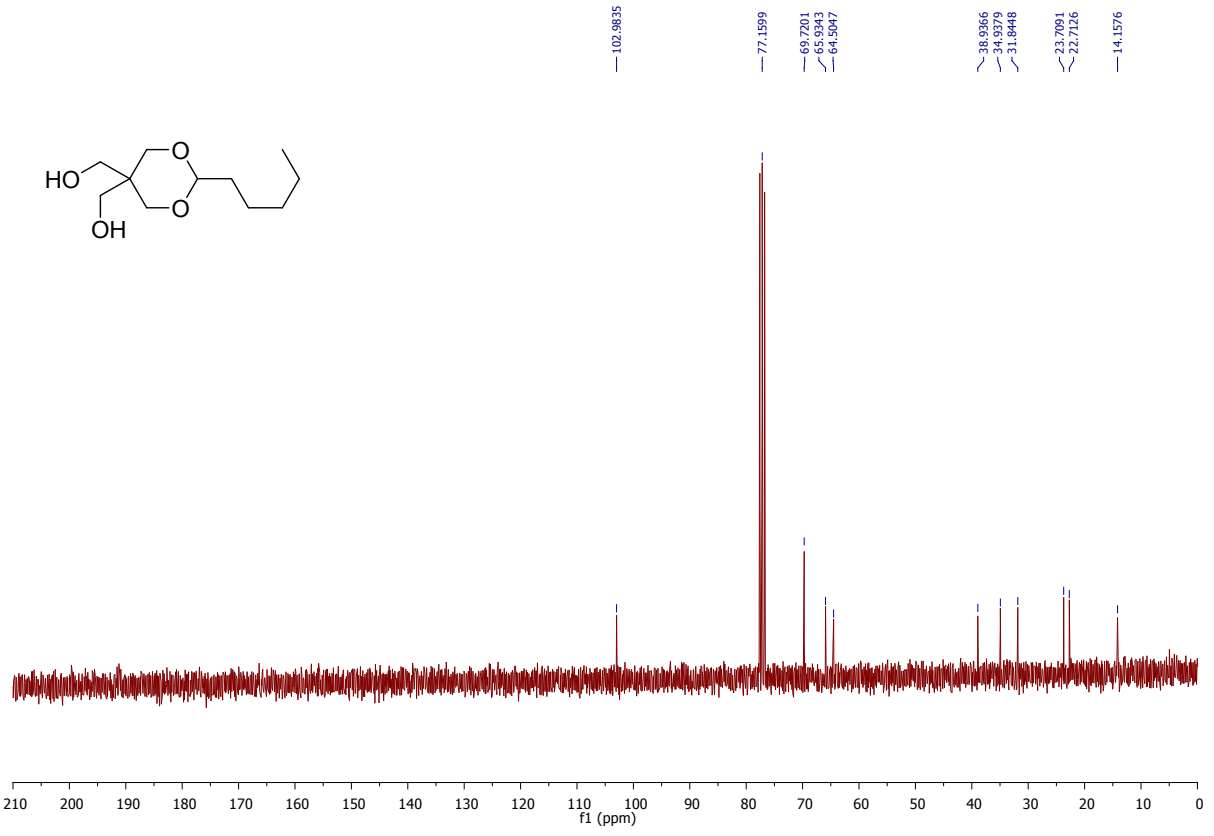
3,9-dibutyl-2,4,8,10-tetraoxaspiro[5.5]undecane



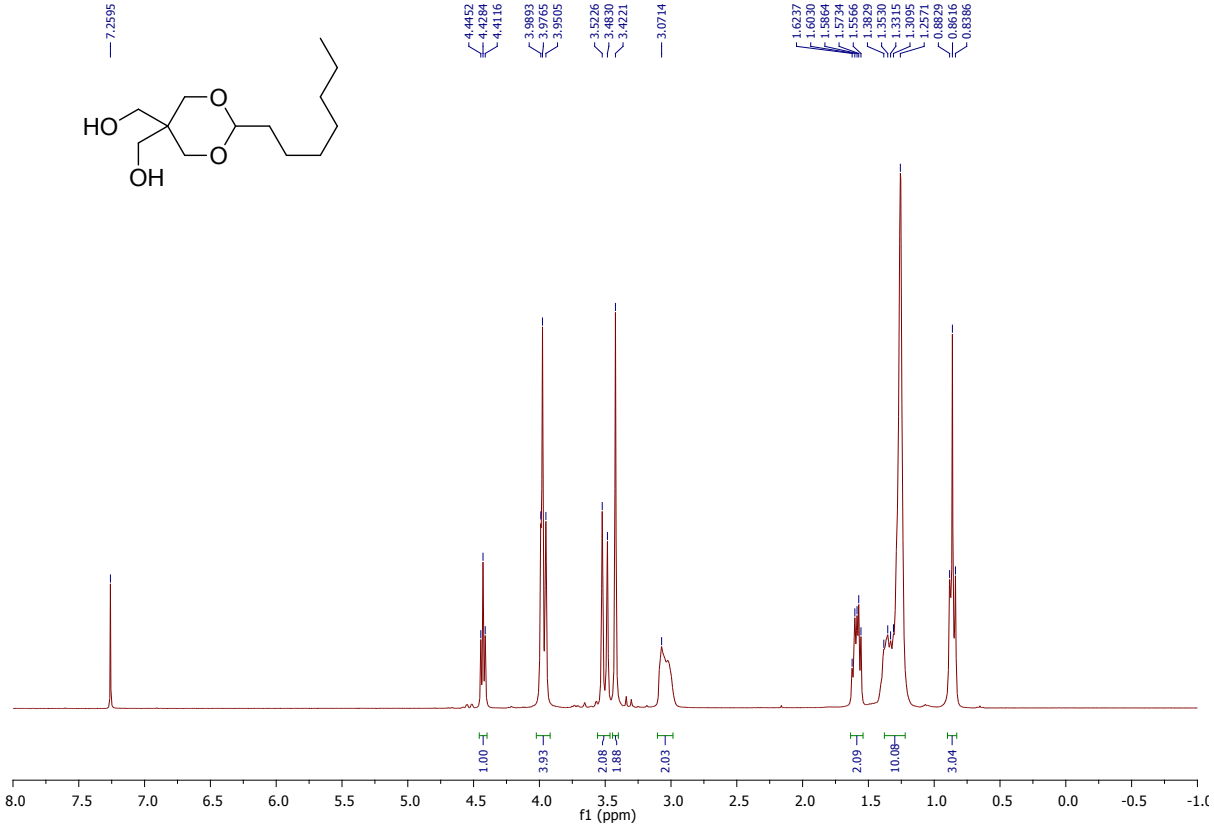


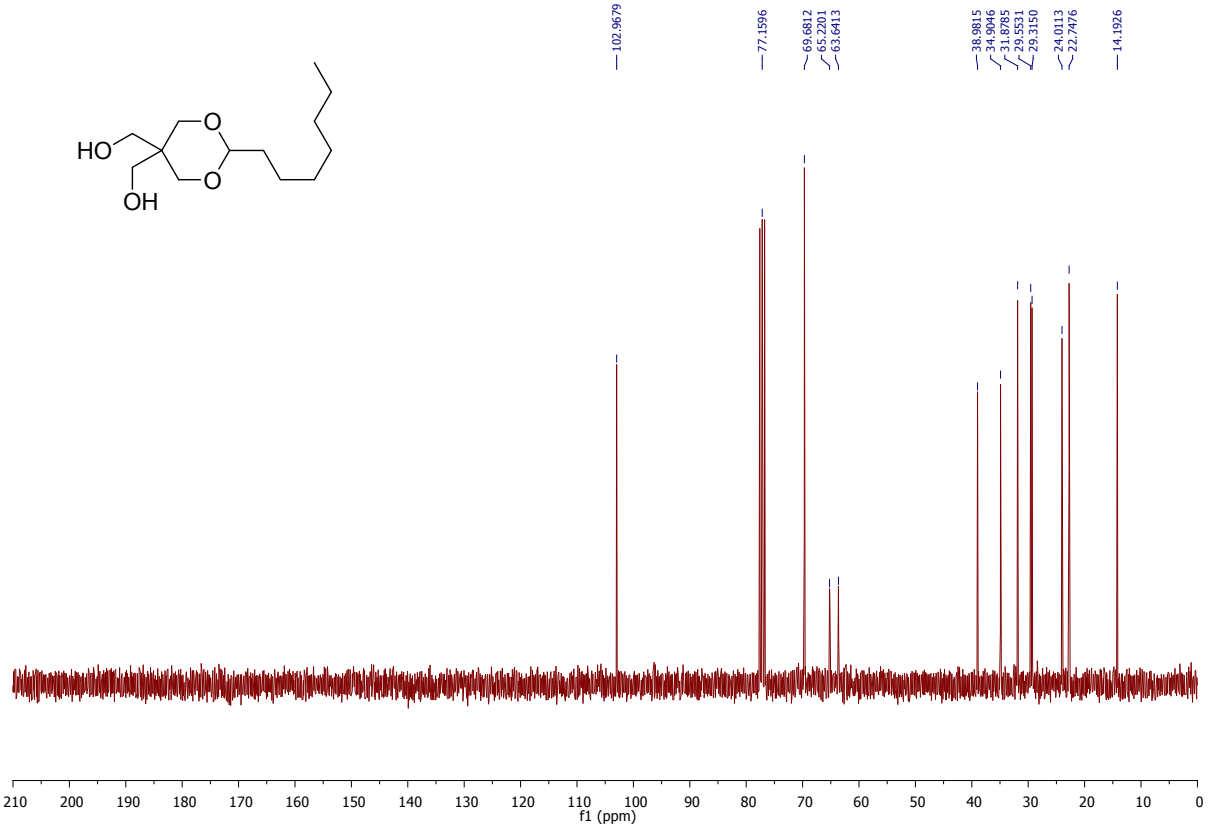
2-pentyl-1,3-dioxane-5,5-diyl)dimethanol



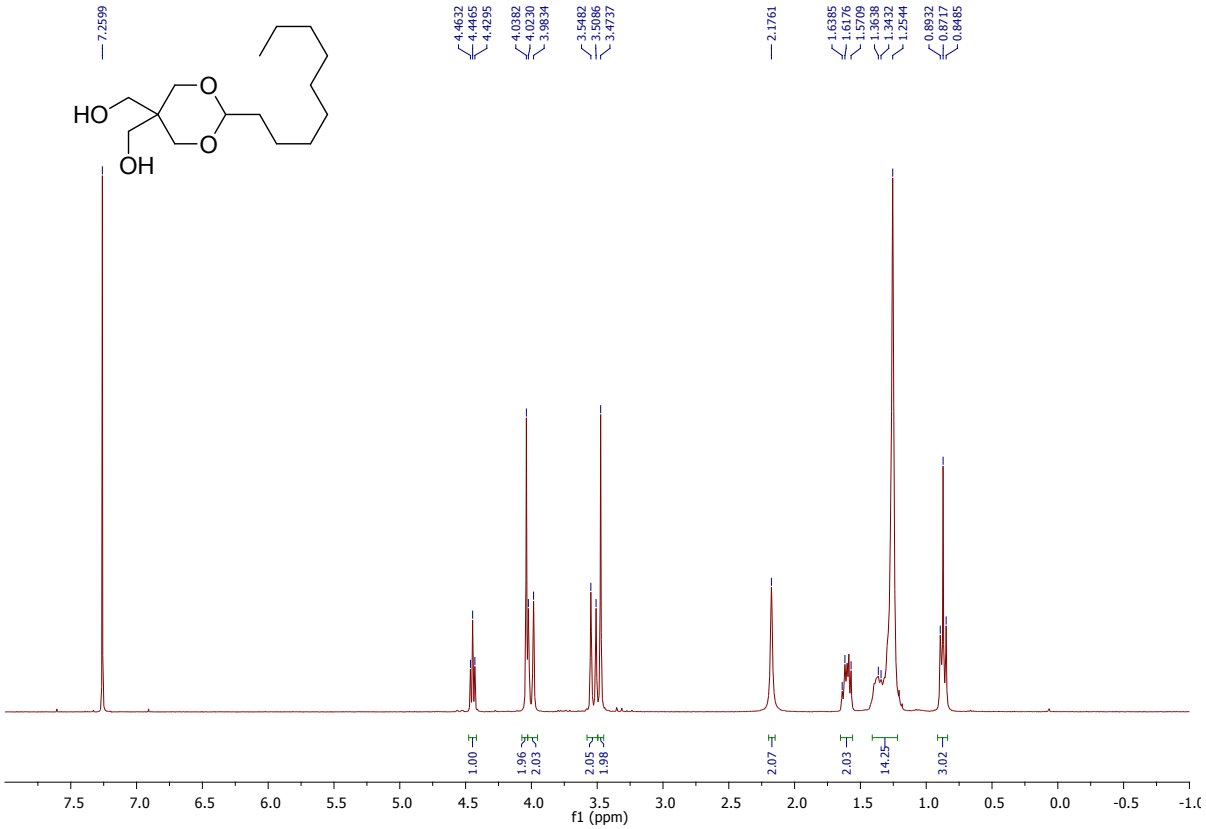


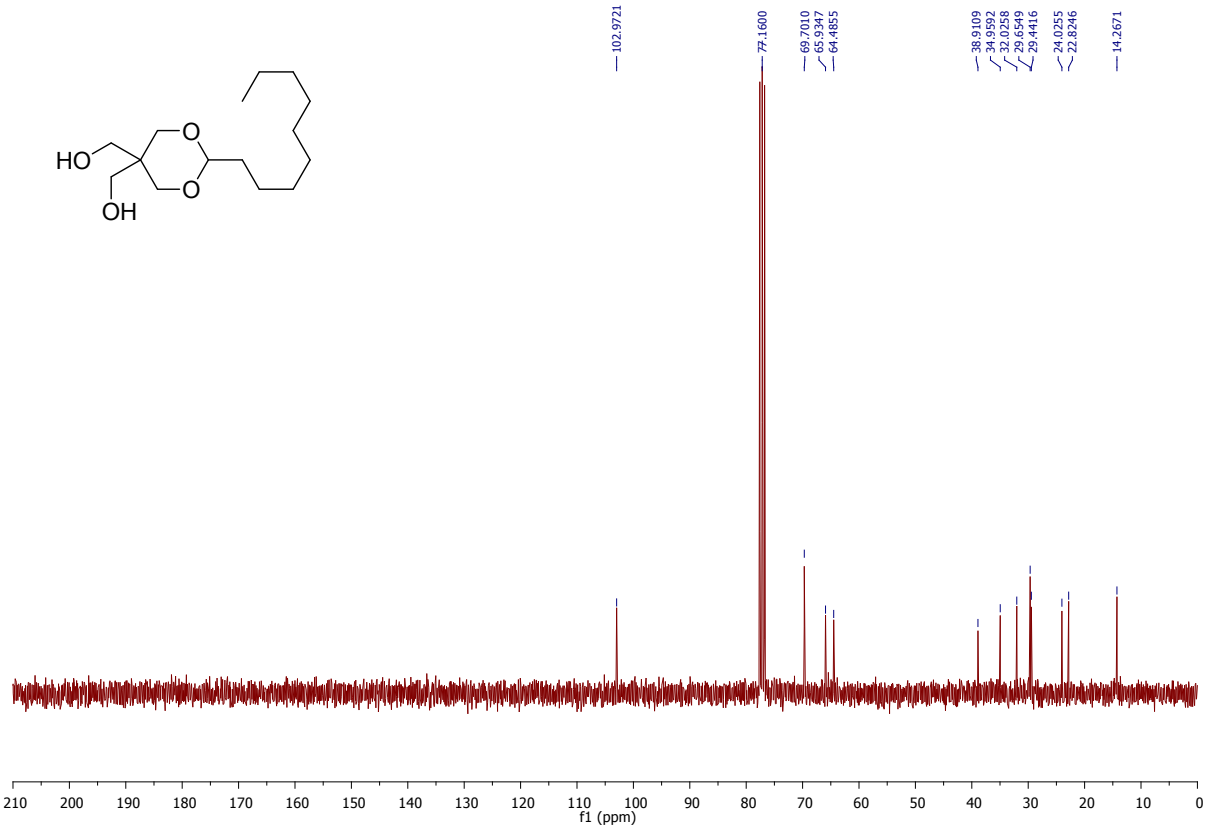
2-heptyl-1,3-dioxane-5,5-diyl)dimethanol



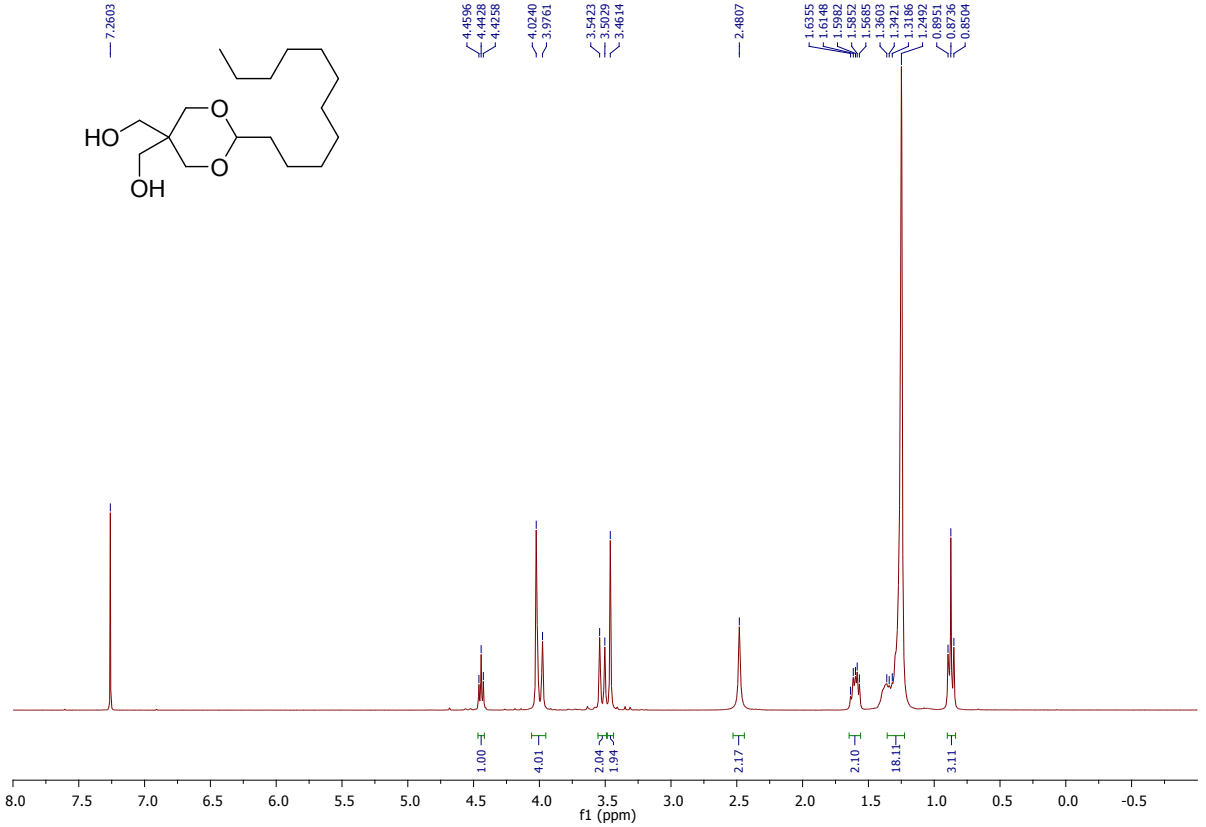


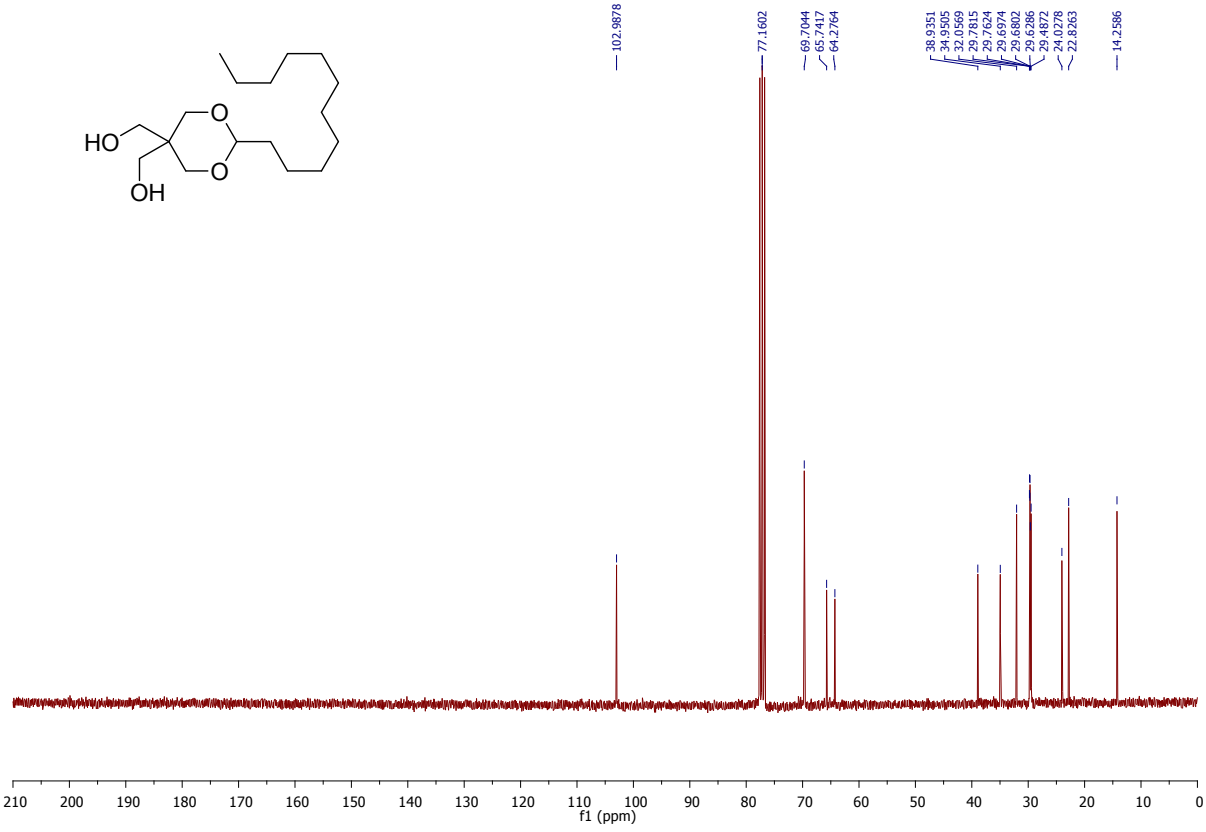
2-nonyl-1,3-dioxane-5,5-diyl)dimethanol



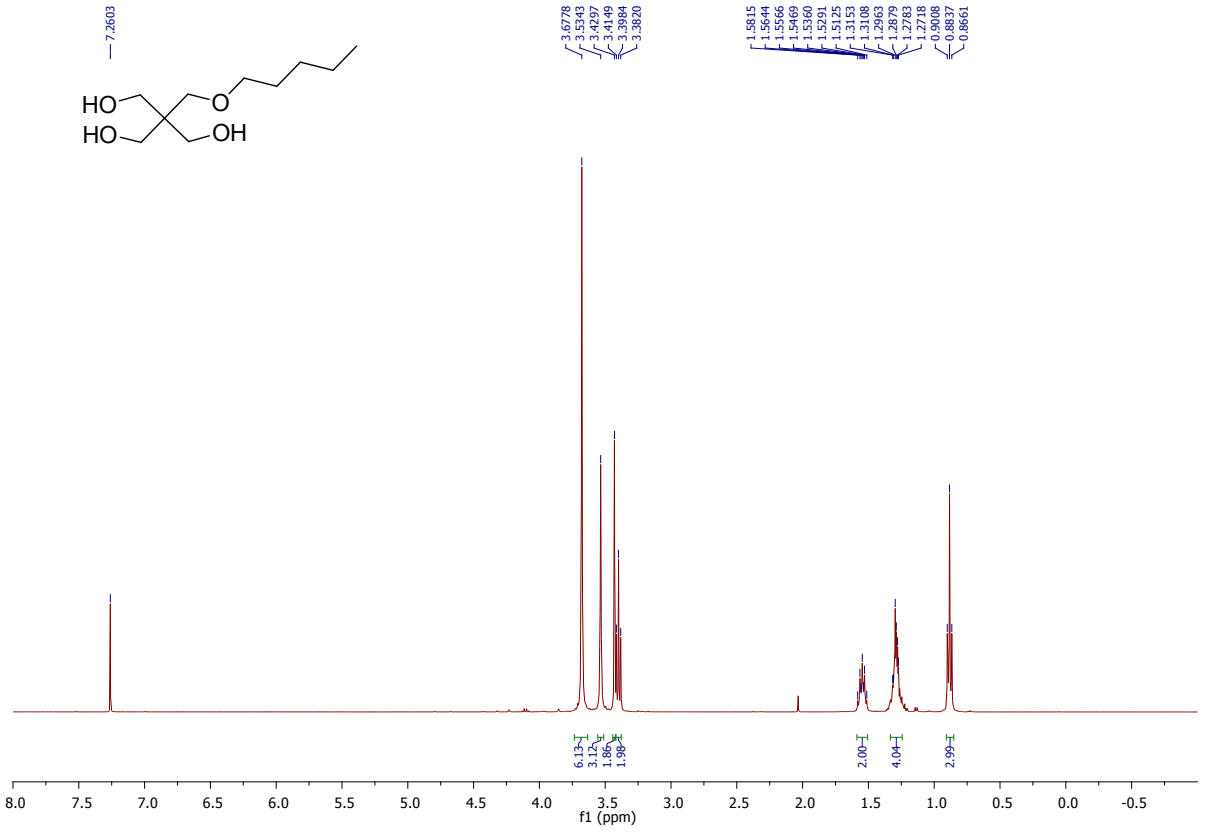


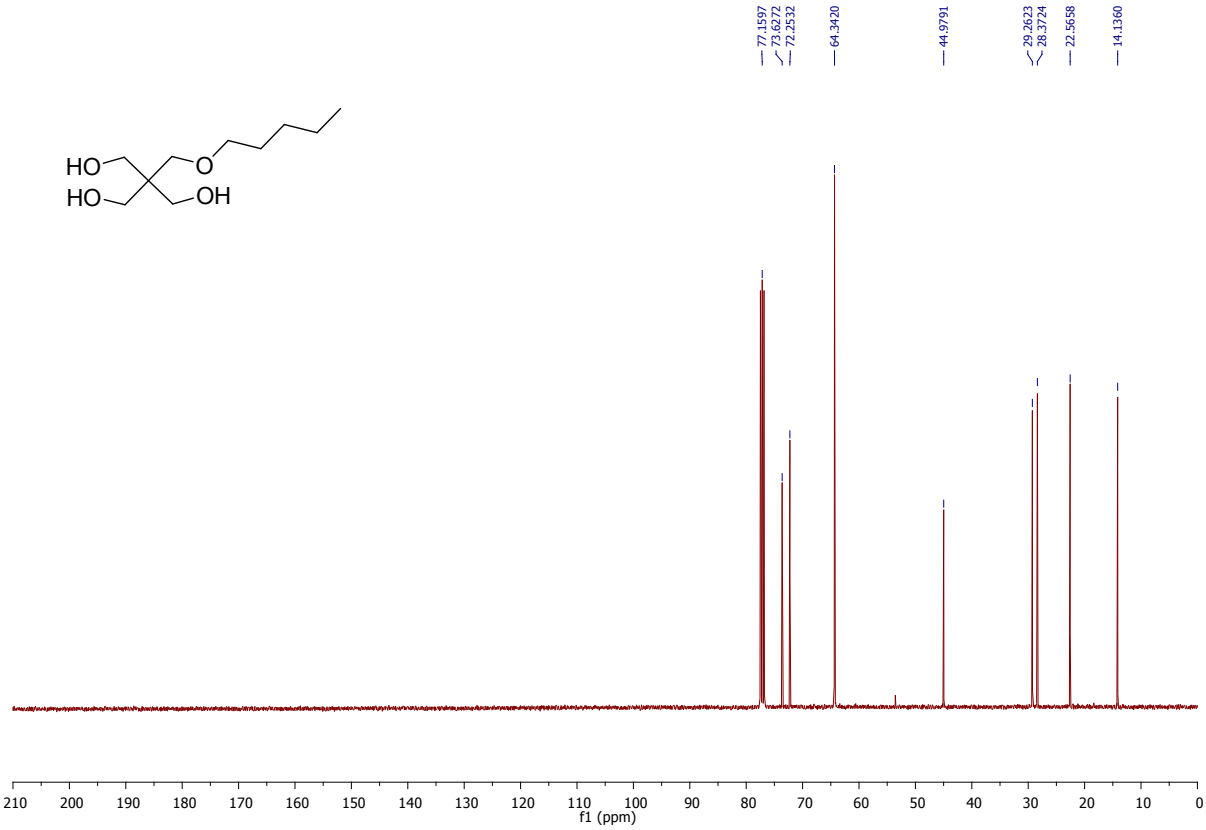
2-undecyl-1,3-dioxane-5,5-diyl)dimethanol



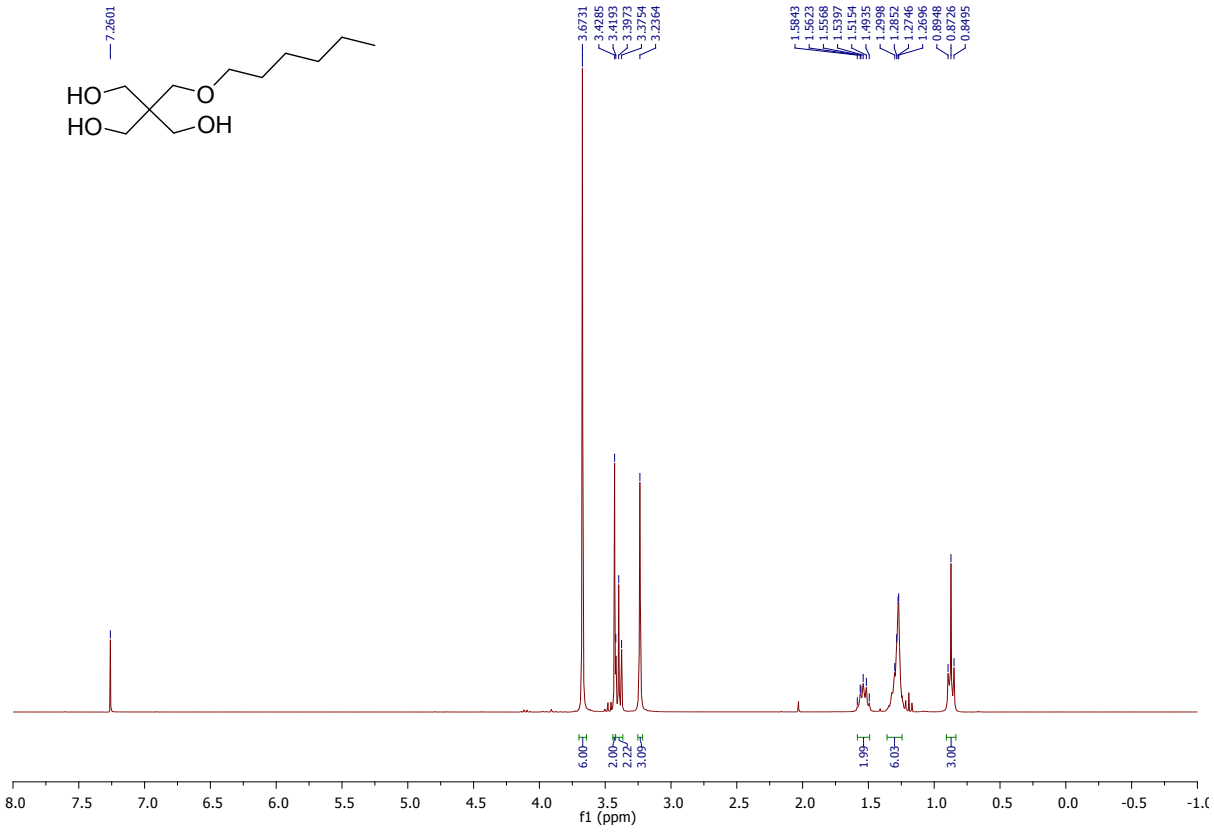


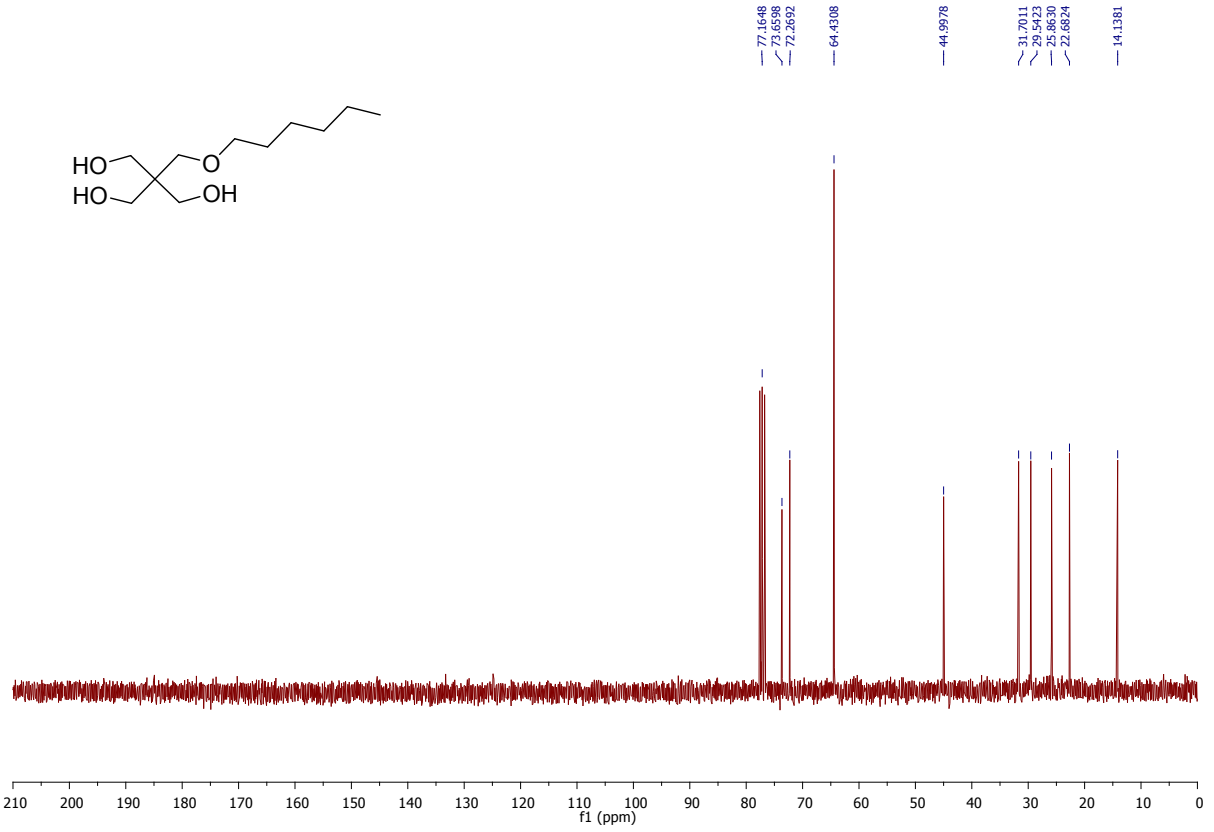
***O*-pentylpentaerythritol**



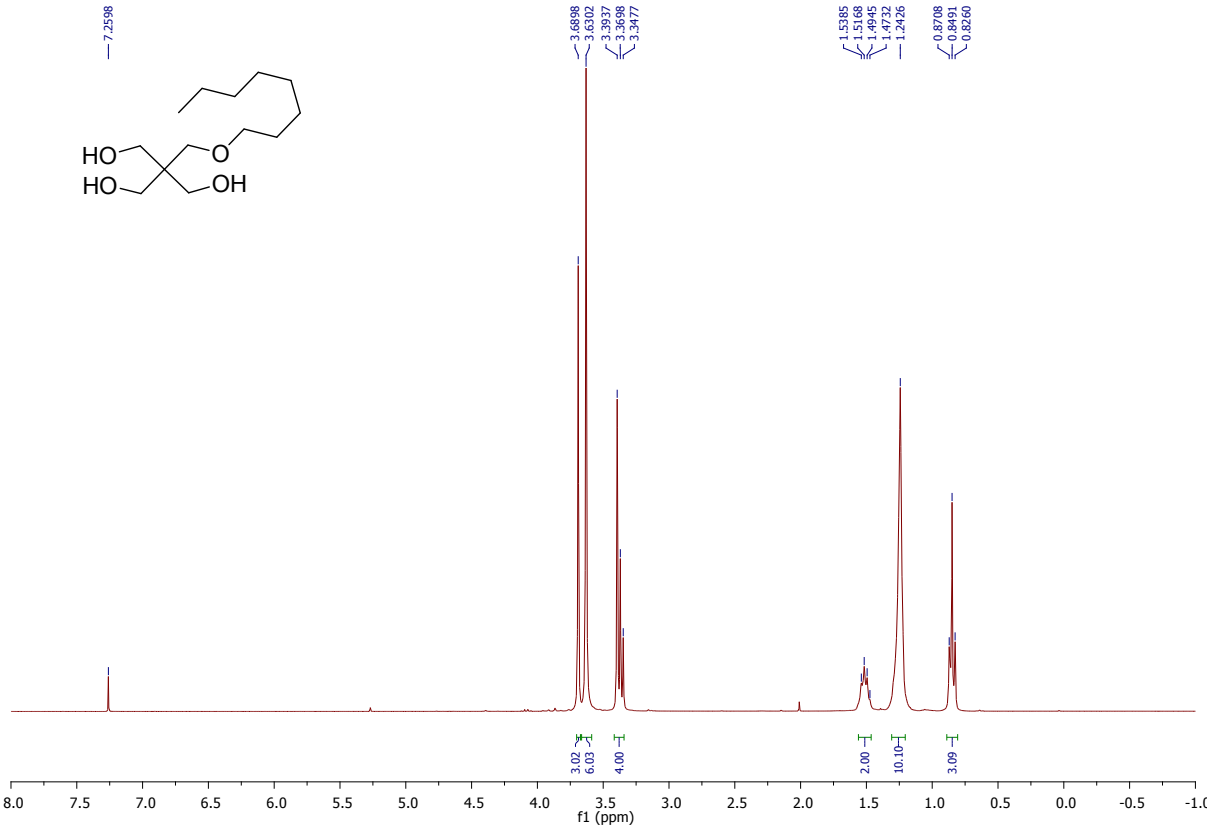


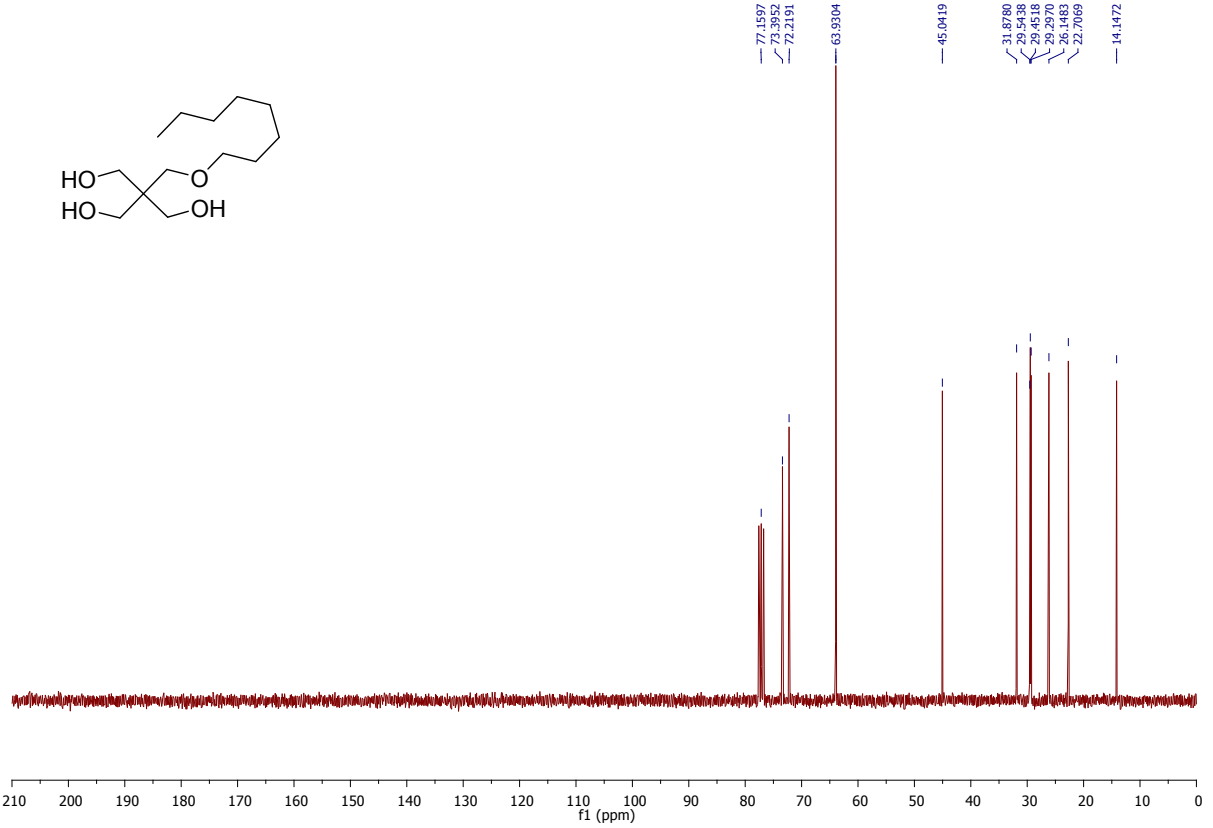
***O*-hexylpentaerythritol**



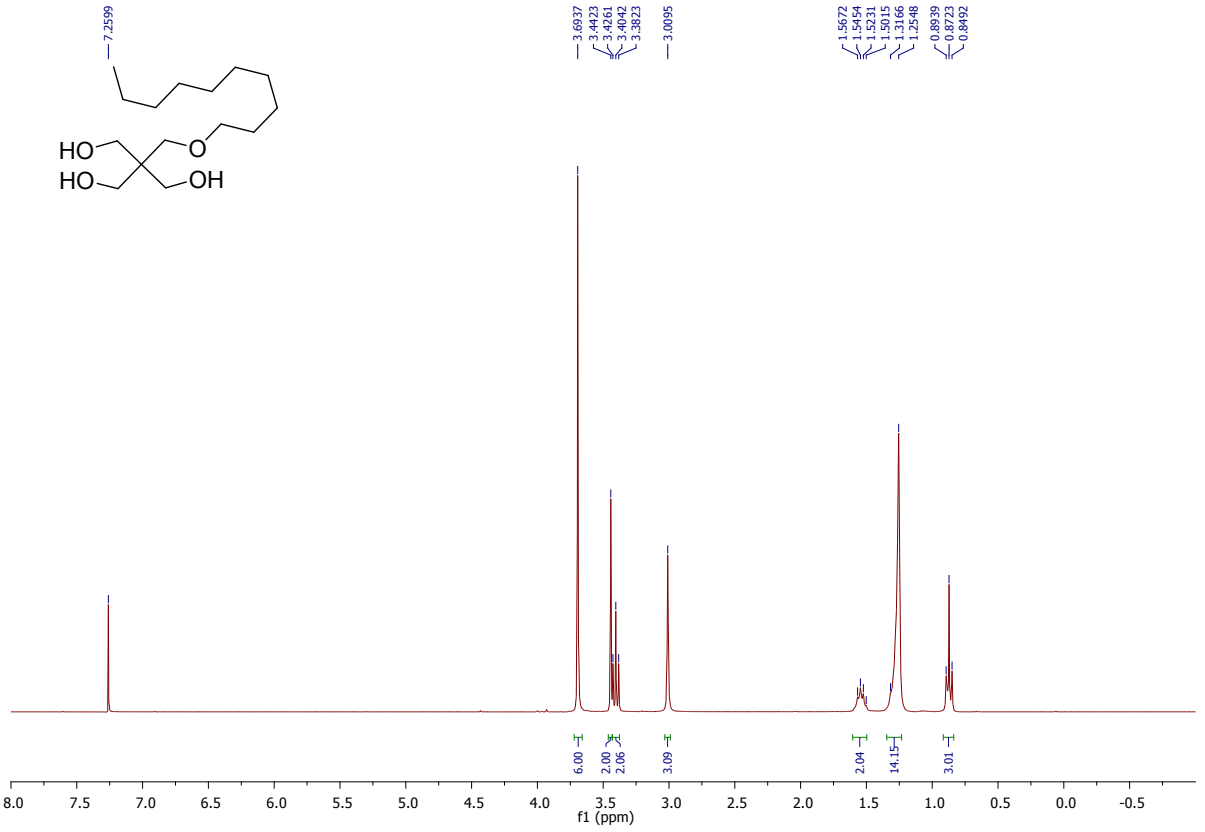


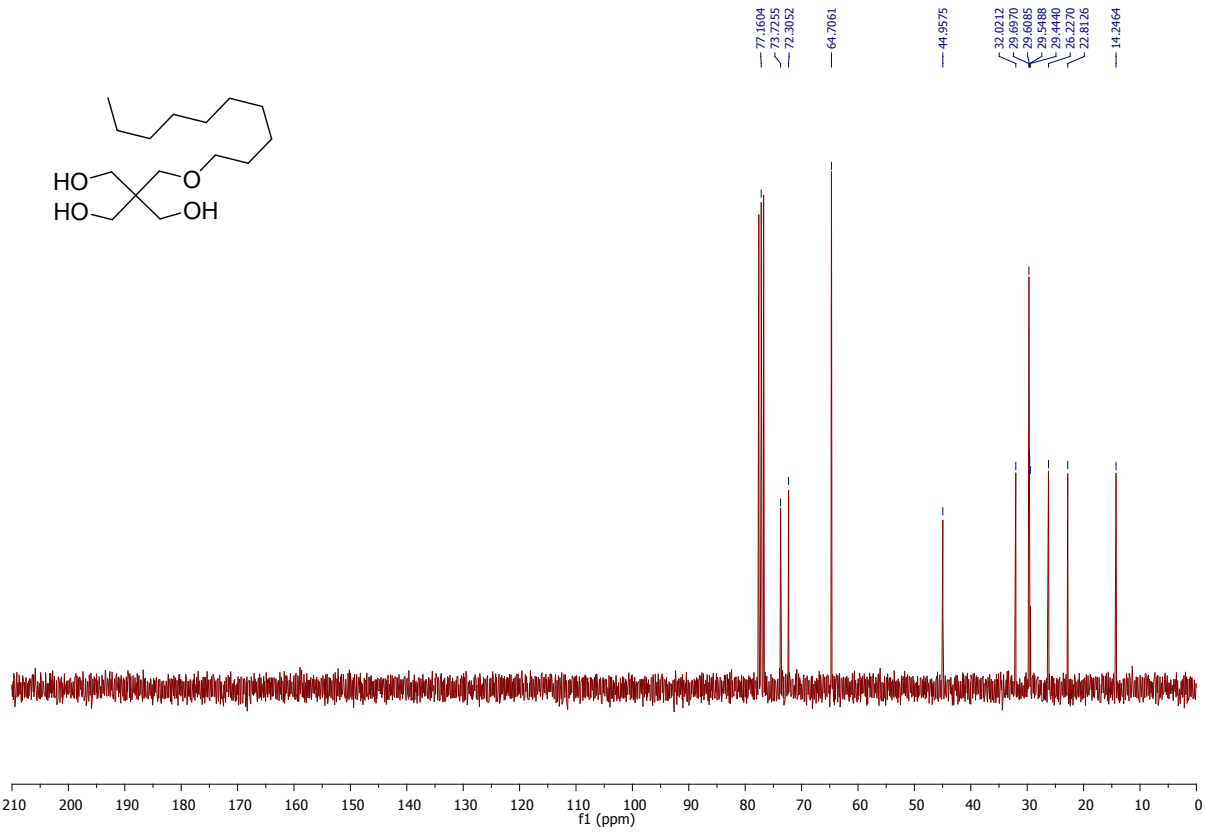
***O*-octylpentaerythritol**



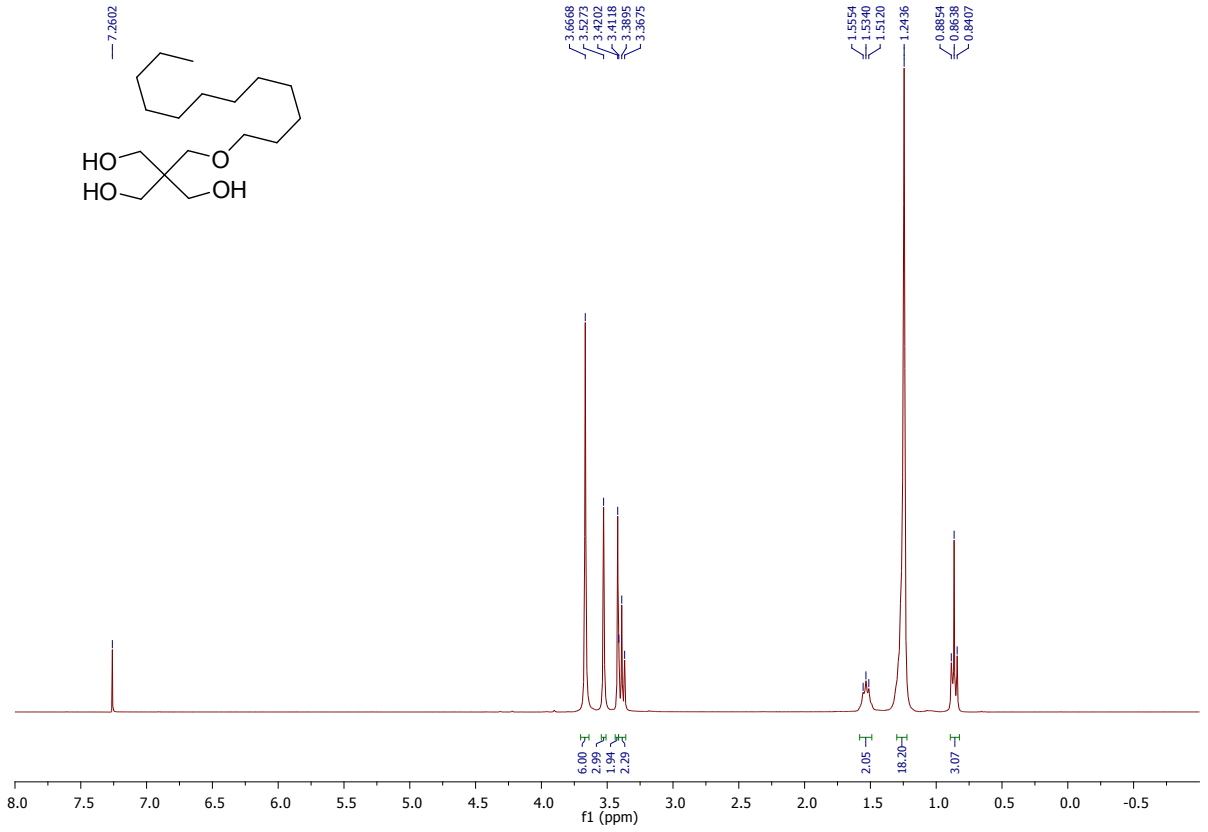


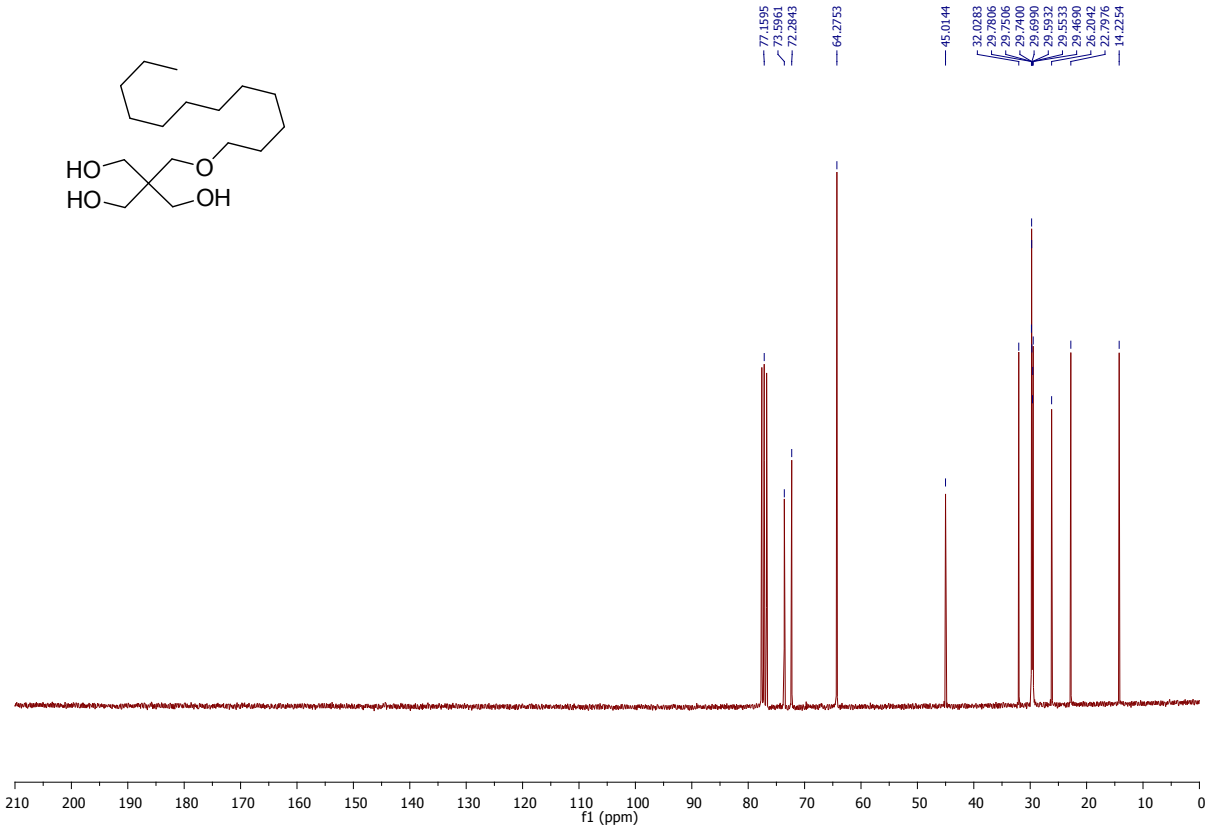
***O*-decylpentaerythritol**



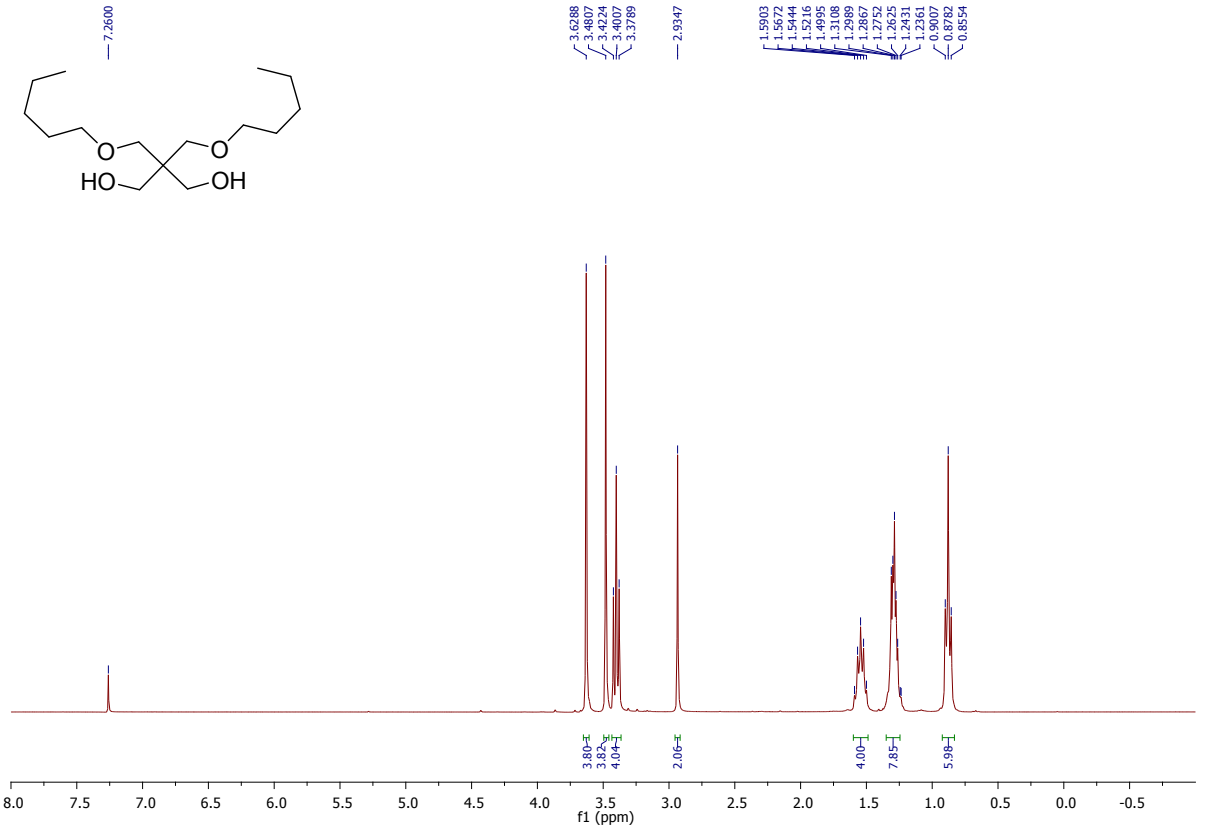


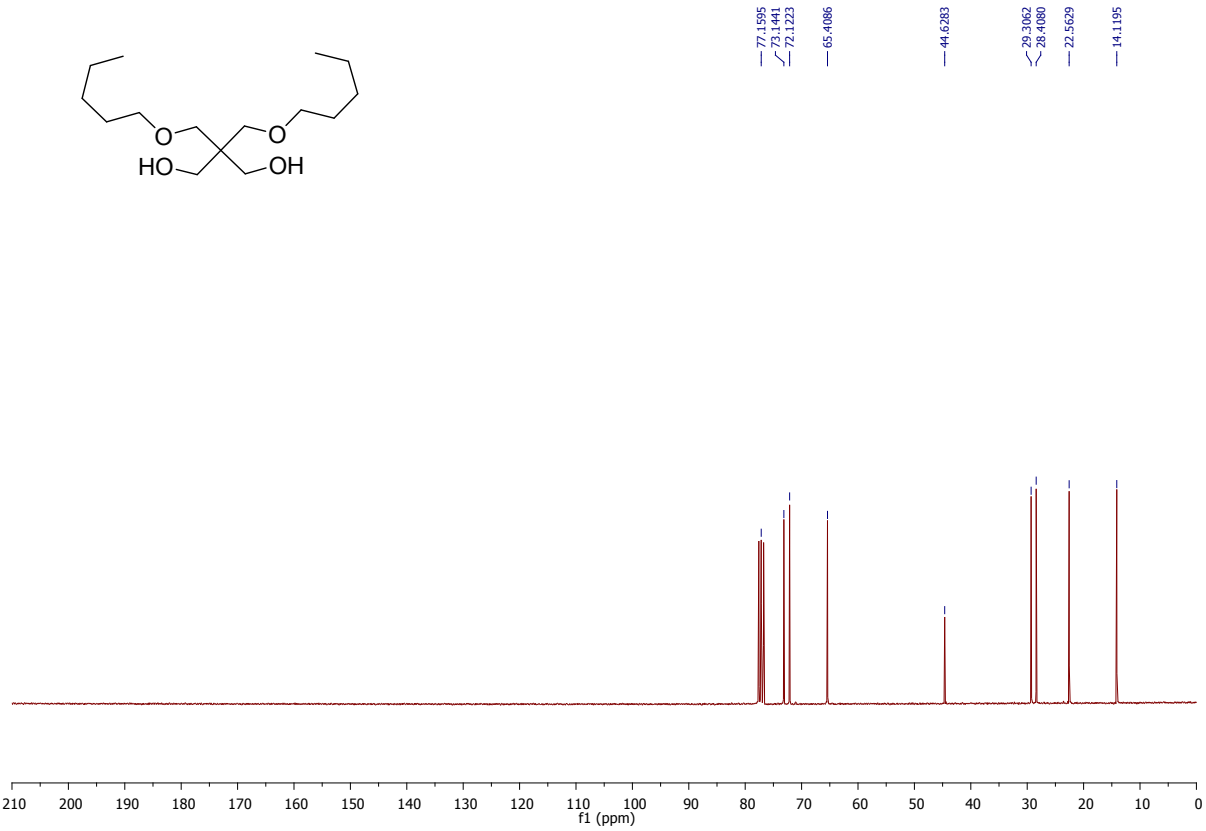
***O*-dodecylpentaerythritol**



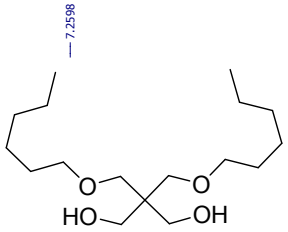


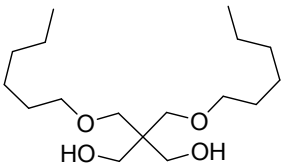
2,2-bis((pentyloxy)methyl)propane-1,3-diol



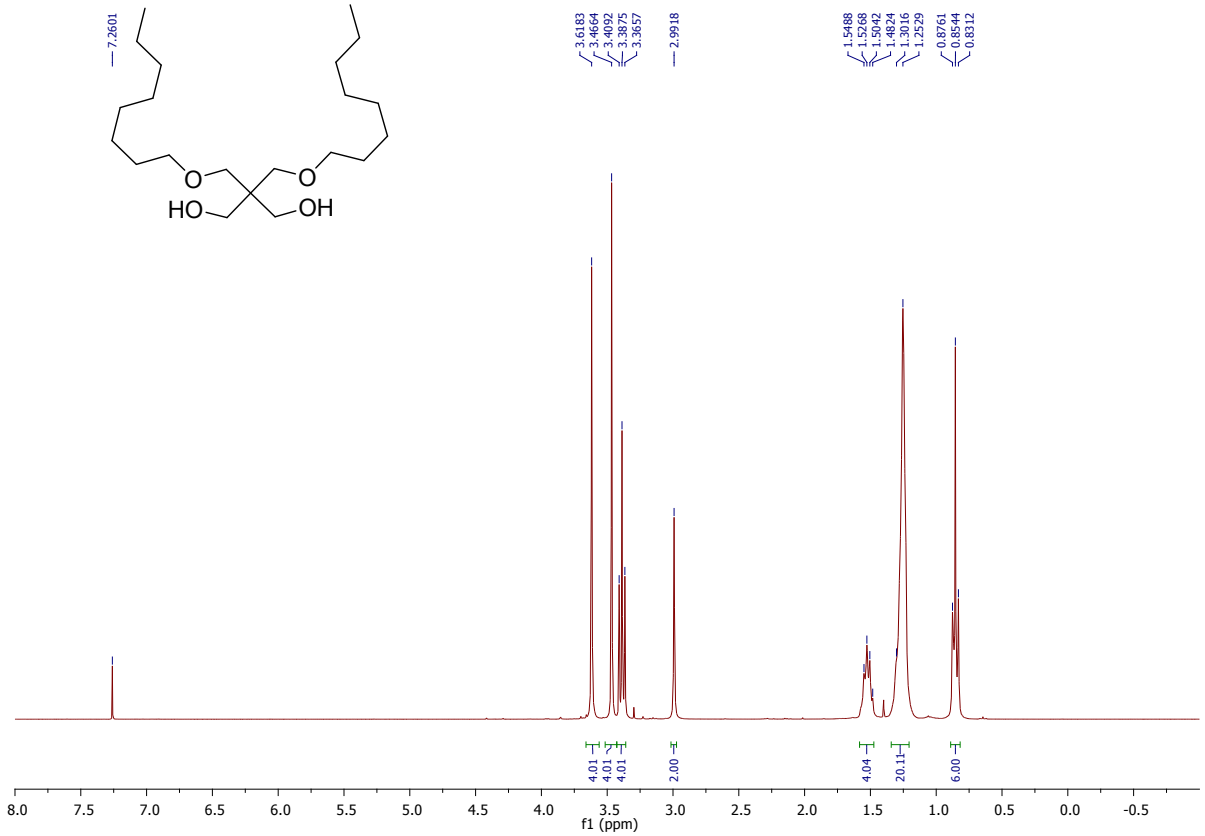


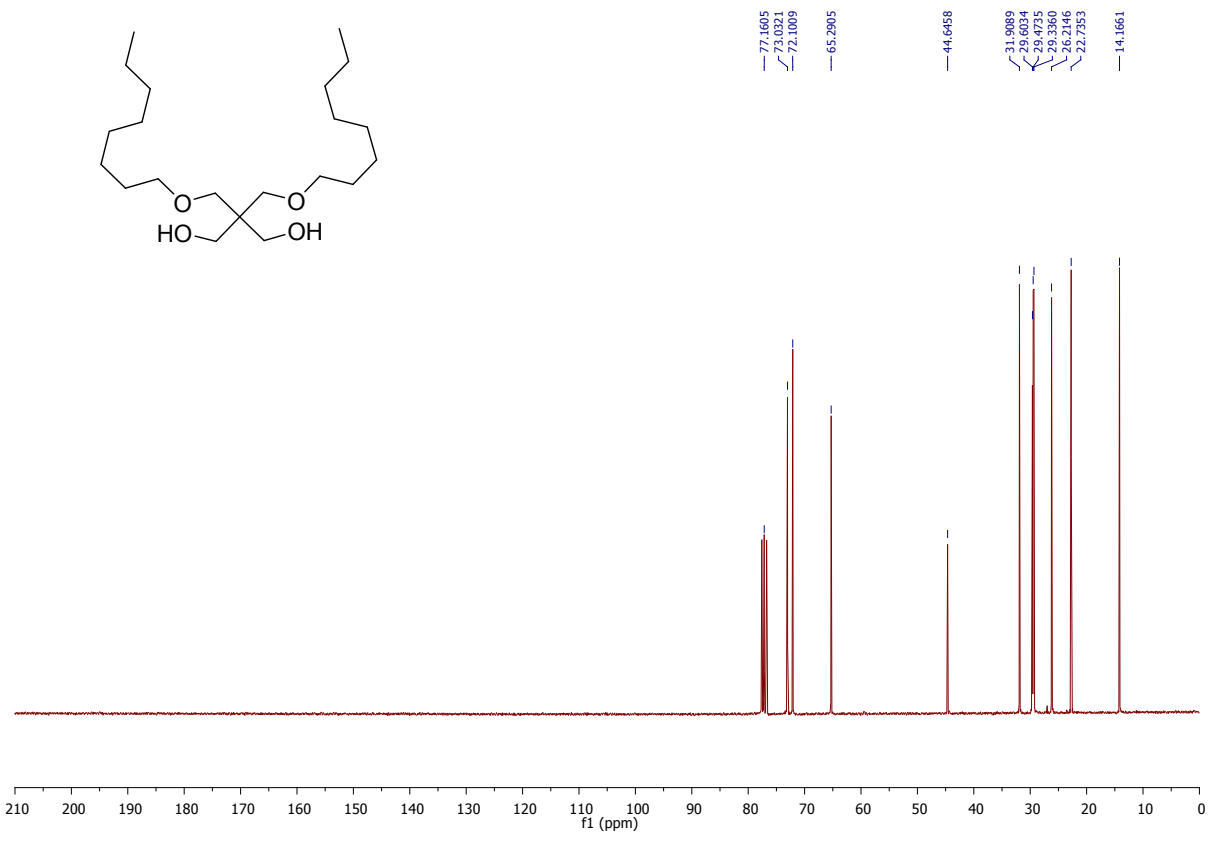
2,2-bis((hexyloxy)methyl)propane-1,3-diol



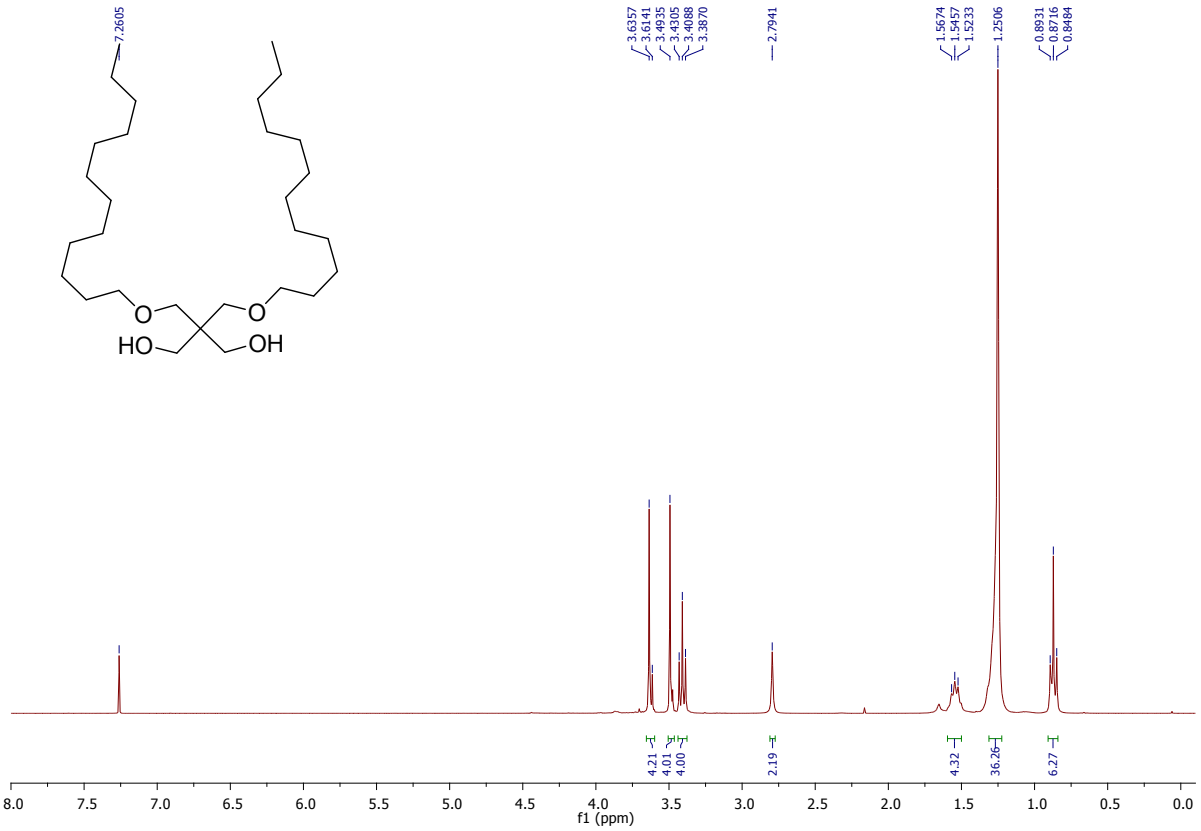


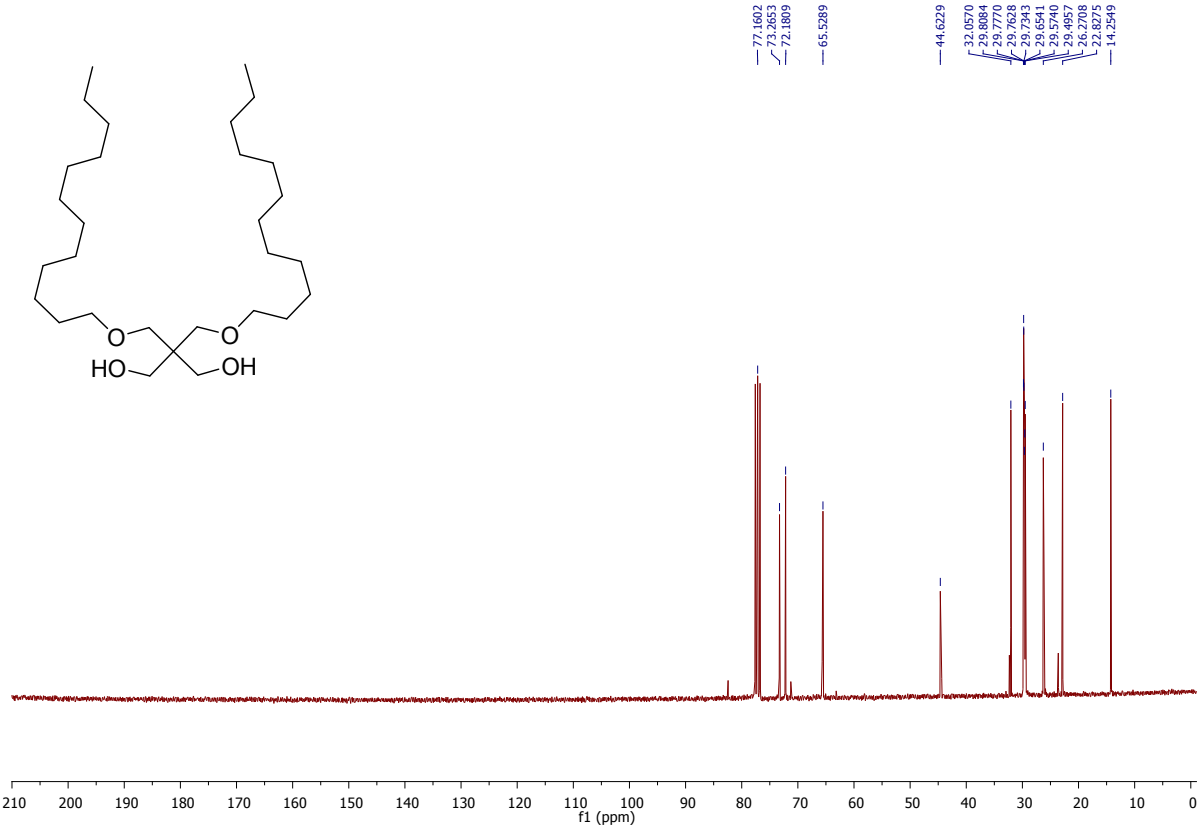
2,2-bis((octyloxy)methyl)propane-1,3-diol





2,2-bis((dodecyloxy)methyl)propane-1,3-diol


7.2605
3.6357
3.6141
3.4935
3.4305
3.4088
3.3870
2.7941
1.5674
1.5457
1.5233
1.2506
0.8931
0.8716
0.8484
HO
OH
4.21
4.01
4.00
2.19
4.32
36.26
6.27
f1 (ppm)


77.1602
73.2653
72.1809
65.5289
44.6229
32.0570
29.8084
29.7770
29.7628
29.7343
29.6541
29.5740
29.4957
26.2708
22.8275
14.2549
HO
OH
f1 (ppm)