

## Supporting Information

# Simple and Expeditious Pinacol Coupling of Non Usual $\alpha,\beta$ Unsaturated Carbonyl Compounds in Water

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### List of Contents

1. General information	1
2. General procedure for the synthesis of pinacol products <b>2</b>	1
3. Spectral data of all compounds	2 – 6
4. Spectra of all compounds	7 - 24

### General information:

All commercially available products and solvents were used without further purification. Reactions were monitored by TLC (Kieselgel 60F254 aluminum sheet) with detection by UV light or potassium permanganate acidic solution. Column chromatography was performed on silica gel 40–60  $\mu\text{m}$ . Flash column chromatography was performed on an automatic apparatus, using silica gel cartridges.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a 400 MHz/54 mm ultralong hold. Chemical shifts ( $\delta$ ) are quoted in parts per million (ppm) and are referenced to TMS as an internal standard. Coupling constants (J) are quoted in hertz. The *dl/meso* ratios were determined by  $^1\text{H}$  NMR of the crude product.

### General procedure for the synthesis of pinacol products **2**

A 10 mL flask was charged with the desired carbonyl compound (2,3 mmol) and zinc (2 eq., 300 mg). The solution of AcOH (2 eq., 250  $\mu\text{L}$  in 4,75 mL of water) was then added. The mixture was vigorously stirred at room temperature for 20 minutes. At the end of the reaction, zinc is filtered and the final product is extracted from the filtrate with EtOAc ( $3 \times 5$  mL). The organic phase was dried over anhydrous sodium sulphate and concentrated in vacuo. The crude product was purified over a column of silica gel and eluted with a gradient of cyclohexane / ethyl acetate to give the pinacol products **2**.

## Spectral Data

### (2E,6E)-octa-2,6-diene-4,5-diol (*dl* and *meso*) (2a)<sup>i</sup>

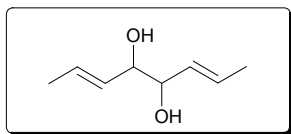


Table 4, entry 1, colorless oil:

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ ppm: 1.66 (t, 6H, *J* = 6,5 Hz, CH<sub>3</sub>), 2.55-2.47 (bs, 2H, OH), 3.84 (d, 2H, *J* = 4.7 Hz, CH-OH, *dl form*), 3.99 (d, 2H, *J* = 6.2 Hz, CH-OH, *meso form*), 5.45-5.36 (m, 2H, CH=CH), 5.73-5.66 (m, 2H, CH=CH),

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ ppm: 17.8 (CH<sub>3</sub>), 17.9 (CH<sub>3</sub>), 75.6 (CH), 75.8 (CH), 128.9 (CH=CH), 129.1 (CH=CH), 129.4 (CH=CH), 129.8 (CH=CH),

HRMS (ESI): found 165.0888; calculated 165.0891 for C<sub>8</sub>H<sub>14</sub>O<sub>2</sub>Na

### (4E,8E)-dodeca-4,8-diene-6,7-diol (*dl* and *meso*) (2b)<sup>ii</sup>

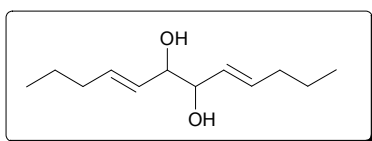


Table 4, entry 2, colorless oil:

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ ppm: 0.86-0.79 (m, 6H, 2 CH<sub>3</sub>), 1.35-1.29 (m, 4H, 2 CH<sub>2</sub>), 1.97-1.92 (m, 4H, 2 CH<sub>2</sub>), 2.82 (brs, 2H, OH), 3.81 (d, *J* = 8.4 Hz, 2H, CH-OH *dl form*), 4.00 (d, *J* = 8.4 Hz, 2H, CH-OH *meso form*), 5.42-5.31 (m, 2H, CH=CH), 5.68-5.60 (m, 2H, CH=CH),

*dl-2b*: <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ ppm: 13.6 (CH<sub>3</sub>), 22.2 (CH<sub>2</sub>), 34.4 (CH<sub>2</sub>), 76.1 (CH), 128.8 (CH=CH), 134.2 (CH=CH),

*meso-2b*: <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ ppm: 13.6 (CH<sub>3</sub>), 22.2 (CH<sub>2</sub>), 34.4 (CH<sub>2</sub>), 75.7 (CH), 128.0 (CH=CH), 134.5 (CH=CH),

HRMS (ESI): found 221.1519; calculated 221.1517 for C<sub>12</sub>H<sub>22</sub>O<sub>2</sub>Na

### (8E,12E)-icosa-8,12-diene-10,11-diol (*dl* and *meso*) (2c)

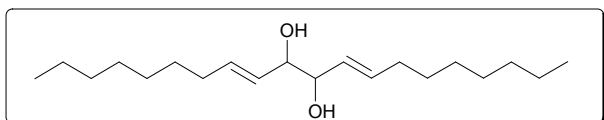


Table 4, entry 3, white solid:

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ ppm: 0.82-0.79 (m, 6H, 2 CH<sub>3</sub>), 1.32-1.19 (m, 20H, 8 CH<sub>2</sub>, 2 OH, 1 H from non equivalent CH<sub>2</sub>), 2.01-1.94 (m, 6H, 2 CH<sub>2</sub>, 1 H from non equivalent CH<sub>2</sub>), 3.84 (dd, *J* = 8.4, 1.6 Hz, 2H, CH-OH *dl form*), 3.99 (dd, *J* = 8.4, 1.6 Hz, 2H, CH-OH *meso form*), 5.42-5.33 (m, 2H, CH=CH), 5.70-5.64 (m, 2H, CH=CH),

*dl-2c*: <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ ppm: 13.1 (CH<sub>3</sub>), 21.7 (CH<sub>2</sub>), 28.1 (CH<sub>2</sub>), 28.1 (CH<sub>2</sub>), 28.2 (CH<sub>2</sub>), 31.4 (CH<sub>2</sub>), 75.0 (CH), 127.4 (CH=CH), 133.8 (CH=CH),

*meso-2c*: <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ ppm: 13.1 (CH<sub>3</sub>), 21.7 (CH<sub>2</sub>), 28.1 (CH<sub>2</sub>), 28.1 (CH<sub>2</sub>), 28.2 (CH<sub>2</sub>), 30.8 (CH<sub>2</sub>), 74.6 (CH), 126.7 (CH=CH), 134.3 (CH=CH),

mp= 30-32°C

HRMS (ESI): found 333.2756; calculated 333.2770 for C<sub>20</sub>H<sub>38</sub>O<sub>2</sub>Na

### (6E,8E,12E,14E)-icosa-6,8,12,14-tetraene-10,11-diol (*meso*) (2d)

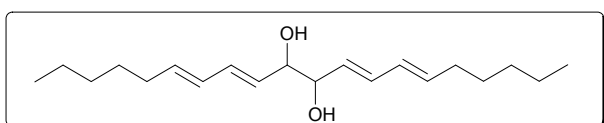


Table 4, entry 4, colorless oil:

**meso-2d:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 0.83-0.79 (m, 10H, 2  $\text{CH}_3$ , 1  $\text{CH}_2$ , 2 OH), 1.23-1.18 (m, 12H, 6  $\text{CH}_2$ ), 2.00 (dd, 2H, 1  $\text{CH}_2$ ), 4.10 (d,  $J = 8.4$  Hz, 2H,  $\text{CH-OH}$ ), 5.49 (dd,  $J = 15.6, 6.0$  Hz, 2H,  $\text{CH=CH}$ ), 5.65 (td,  $J = 14.4, 6.82$  Hz, 2H,  $\text{CH=CH}$ ), 5.97 (dd,  $J = 15.2, 10.4$  Hz, 2H,  $\text{CH=CH}$ ), 6.20 (dd,  $J = 15.2, 10.4$  Hz, 2H,  $\text{CH=CH}$ ),  
 $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 14.1 ( $\text{CH}_3$ ), 22.5 ( $\text{CH}_2$ ), 28.8 ( $\text{CH}_2$ ), 31.4 ( $\text{CH}_2$ ), 32.6 ( $\text{CH}_2$ ), 75.5 (CH), 127.8 ( $\text{CH=CH}$ ), 129.3 ( $\text{CH=CH}$ ), 133.8 ( $\text{CH=CH}$ ), 136.5 ( $\text{CH=CH}$ ),  
 HRMS (ESI): found 329.2468; calculated 329.2457 for  $\text{C}_{20}\text{H}_{34}\text{O}_2\text{Na}$

**(2E,6E)-3,6-dimethylocta-2,6-diene-4,5-diol (meso) (2e)**

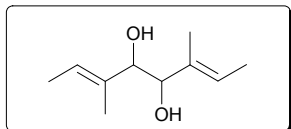


Table 4, entry 5, colorless oil:

**meso-2e:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 1.54-1.53 (m, 12H, 4  $\text{CH}_3$ ), 2.25-2.14 (bs, 2H, 2 OH), 3.93 (s, 2H, 2  $\text{CH-OH}$ ), 5.50-5.44 (m, 2H, 2  $\text{CH=CH}$ )  
 $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 11.9 ( $\text{CH}_3$ ), 13.1 ( $\text{CH}_3$ ), 78.4 (CH), 122.8 ( $\text{CH=C}$ ), 134.5 ( $\text{CH=C}$ ),  
 HRMS (ESI): found 193.1201; calculated 193.1204 for  $\text{C}_{10}\text{H}_{18}\text{O}_2\text{Na}$

**(3E,7E)-4,7-dimethyldeca-3,7-diene-5,6-diol (meso) (2f)**

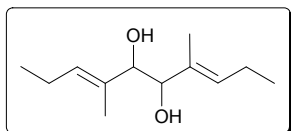


Table 4, entry 6, colorless oil:

**meso-2f:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 0.92 (t, 6H, 2  $\text{CH}_3$ ), 1.59 (s, 6H, 2  $\text{CH}_3$ ), 2.00 (quint,  $J = 7.6$  Hz, 4H, 2  $\text{CH}_2$ ), 2.36 (bs, 2H, 2 OH), 3.97 (s, 2H, 2  $\text{CH-OH}$ ), 5.41 (td, 2H, 2  $\text{CH=C}$ )  
 $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 11.8 ( $\text{CH}_3$ ), 13.9 ( $\text{CH}_3$ ), 20.8 ( $\text{CH}_2$ ), 78.8 (CH), 130.7 ( $\text{CH=C}$ ), 133.0 ( $\text{CH=C}$ ),  
 HRMS (ESI): found 221.1521; calculated 221.1517 for  $\text{C}_{12}\text{H}_{22}\text{O}_2\text{Na}$

**(6E,10E)-2,6,11,15-tetramethylhexadeca-2,6,10,14-tetraene-8,9-diol (dl and meso) (2g)<sup>iii</sup>**

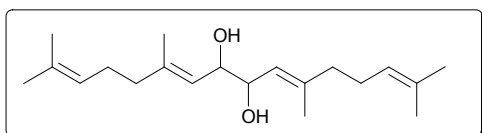


Table 4, entry 7, yellow oil:

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 1.76-1.60 (m, 18H, 6  $\text{CH}_3$ ), 2.13-2.07 (m, 8H, 4  $\text{CH}_2$ ), 4.18 (m, 1H,  $\text{CH-OH dl form}$ ), 4.30 (m, 1H,  $\text{CH-OH meso form}$ ), 5.26-5.07 (m, 4H, 4  $\text{CH=C}$ )  
 $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 16.9 ( $\text{CH}_3$ ), 17.1 ( $\text{CH}_3$ ), 17.7 ( $\text{CH}_3$ ), 17.7 ( $\text{CH}_3$ ), 23.5 (2  $\text{CH}_3$ ), 23.6 (2  $\text{CH}_3$ ), 25.7 (2  $\text{CH}_3$ ), 25.7 (2  $\text{CH}_3$ ), 26.3 ( $\text{CH}_2$ ), 26.4 ( $\text{CH}_2$ ), 26.6 ( $\text{CH}_2$ ), 26.7 ( $\text{CH}_2$ ), 32.6 ( $\text{CH}_2$ ), 32.7 ( $\text{CH}_2$ ), 39.7 ( $\text{CH}_2$ ), 39.8 ( $\text{CH}_2$ ), 70.9 (CH), 71.3 (CH), 71.8 (CH), 72.1 (CH), 122.8 ( $\text{CH=C}$ ), 123.1 ( $\text{CH=C}$ ), 123.6 ( $\text{CH=C}$ ), 123.8 ( $\text{CH=C}$ ), 123.9 ( $\text{CH=C}$ ), 123.9 ( $\text{CH=C}$ ), 123.9 ( $\text{CH=C}$ ), 124.0 ( $\text{CH=C}$ ), 131.7 ( $\text{CH=C}$ ), 131.9 ( $\text{CH=C}$ ), 132.4 ( $\text{CH=C}$ ), 132.5 ( $\text{CH=C}$ ), 141.5 ( $\text{CH=C}$ ), 141.6 ( $\text{CH=C}$ ), 141.7 ( $\text{CH=C}$ ), 142.1 ( $\text{CH=C}$ ),  
 HRMS (ESI): found 329.2461; calculated 329.2457 for  $\text{C}_{20}\text{H}_{34}\text{O}_2\text{Na}$

**(6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)-2-(6,6-dimethylbicyclo[3.1.1]hept-2-en-3-yl)ethane-1,2-diol (meso) (2h)**

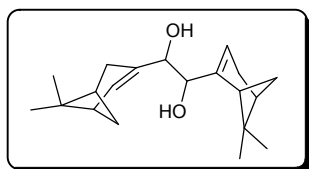


Table 4, entry 8, yellow oil:

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 0.80 (s, 6H, 2  $\text{CH}_3$ ), 1.07 (d,  $J = 8.7$  Hz 2H, 2 CH), 1.23 (s, 6H, 2  $\text{CH}_3$ ), 1.35 (s, 2H, 2 OH), 2.05 (m, 4H, 2  $\text{CH}_2$ ), 2.24 (m, 4H, 2  $\text{CH}_2$ ), 2.36 (td,  $J = 8.7$  Hz,  $J = 5.5$  Hz, 2H, 2 CH), 3.84 (s, 2H, 2  $\text{CH-OH}$  *meso form*), 5.59 (m, 2H, 2  $\text{CH=C}$ )

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 26.3 (2  $\text{CH}_3$ ), 26.9 (2  $\text{CH}_3$ ), 31.2 (2 CH/ $\text{CH}_2$ ), 31.9 (2 CH/ $\text{CH}_2$ ), 38.1 (2  $\text{C}_{\text{IV}}$ ), 41.1 (2 CH/ $\text{CH}_2$ ), 43.1 (2 CH/ $\text{CH}_2$ ), 73.2 (2 CH-OH), 119.1 (2 CH), 147.1 (2  $\text{C}_{\text{IV}}$ ),

HRMS (ESI): found 325.2128; calculated 325.2144 for  $\text{C}_{20}\text{H}_{30}\text{O}_2\text{Na}$

**1-((6S)-6-isopropylbicyclo[3.1.1]hept-2-en-2-yl)-2-((6R)-6-isopropylbicyclo[3.1.1]hept-2-en-3-yl)ethane-1,2-diol (*meso*) (2i)**

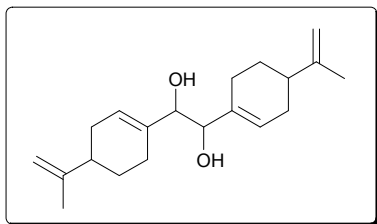


Table 4, entry 9, yellow oil:

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 1.48-1.38 (m, 2H, 2 CH), 1.67 (s, 6H, 2  $\text{CH}_3$ ), 2.27-1.79 (m, 18H, 8  $\text{CH}_2$ , 2 OH), 3.89 (dd,  $J = 12$  Hz,  $J = 4.5$  Hz, 2  $\text{CH-OH}$ ), 4.66 (m, 4H, 2  $\text{C=CH}_2$ ), 5.76 (m, 2H, 2  $\text{CH=C}$ )

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 20.8 ( $\text{CH}_3$ ), 20.9 ( $\text{CH}_3$ ), 23.6 ( $\text{CH}_2$ ), 23.6 ( $\text{CH}_2$ ), 27.2 ( $\text{CH}_2$ ), 27.4 ( $\text{CH}_2$ ), 30.4 ( $\text{CH}_2$ ), 30.7 ( $\text{CH}_2$ ), 40.7 (CH), 41.3 (CH), 76.6 (CH-OH), 76.8 (CH-OH), 108.8 ( $\text{CH}_2=\text{C}$ ), 108.8 ( $\text{CH}_2=\text{C}$ ), 126.5 ( $\text{CH}=\text{C}$ ), 127.0 ( $\text{CH}=\text{C}$ ), 136.3 ( $\text{C}=\text{C}$ ), 136.3 ( $\text{C}=\text{C}$ ), 149.4 ( $\text{C}=\text{CH}_2$ ), 149.6 ( $\text{C}=\text{CH}_2$ ),

HRMS (ESI): found 325.2148; calculated 325.2144 for  $\text{C}_{20}\text{H}_{34}\text{O}_2\text{Na}$

**(1E,5E)-1,6-diphenylhexa-1,5-diene-3,4-diol (*dl* and *meso*) (2j)**

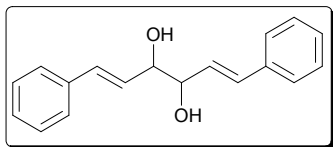


Table 4 entry 10, white solid:

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 4.21 (dd,  $J = 11.4$ , 5.4 Hz, 2H,  $\text{CH-OH}$  *dl form*), 4.36 (dd,  $J = 9.4$ , 3.8 Hz, 2H,  $\text{CH-OH}$  *meso form*), 6.25-6.17 (m, 2H, CH=CH), 6.68-6.61 (m, 2H, CH=CH), 7.33-7.22 (m, 10H,  $\text{CH}_{\text{Ar}}$ ),

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 75.8 (CH *meso form*), 75.9 (CH *dl form*), 126.6 (CH), 126.7 (CH), 127.1 (CH), 127.8 (CH), 127.9 (CH), 128.0 (CH), 128.6 (2 CH), 132.7 (CH), 133.0 (CH), 136.4 ( $\text{C}_{\text{IV}}$ ), 136.5 ( $\text{C}_{\text{IV}}$ ),

MS (ESI): 267.13 [ $\text{M} + \text{H}$ ] $^+$ , 289.12 [ $\text{M} + \text{Na}$ ] $^+$

mp= 110-112°C [*lit.* 106-155°C]

**1,2-Diphenyl-1,2-ethanediol (*dl* and *meso*) (2m)**

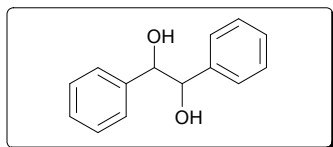


Table 5 entry 1, white solid:

***dl*-2m:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 2.25-2.10 (bs, 2H, OH), 4.61 (s, 2H,  $\text{CH-OH}$ ), 7.23-7.02 (m, 10H,  $\text{CH}_{\text{Ar}}$ ),

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 79.1 ( $\text{CH-OH}$ ), 127.0 (2  $\text{CH}_{\text{Ar}}$ ), 128.0 ( $\text{CH}_{\text{Ar}}$ ), 128.1 (2  $\text{CH}_{\text{Ar}}$ ), 139.8 ( $\text{C}_{\text{IV}}$ ),

***meso*-2m:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 2.25-2.10 (bs, 2H, OH), 4.74 (s, 2H,  $\text{CH-OH}$ ), 7.23-7.02 (m, 10H,  $\text{CH}_{\text{Ar}}$ ),

$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 78.1 ( $\text{CH-OH}$ ), 127.1 (2  $\text{CH}_{\text{Ar}}$ ), 128.1 ( $\text{CH}_{\text{Ar}}$ ), 128.2 (2  $\text{CH}_{\text{Ar}}$ ), 139.7 ( $\text{C}_{\text{IV}}$ ),

MS (ESI): 215.1 [M + H]<sup>+</sup>, 237.1 [M + Na]<sup>+</sup>,  
mp= 120-124°C [lit. 119-159°C]

### 1,2-Bis(4-methylphenyl)-1,2-ethanediol (*dl* and *meso*) (2n)

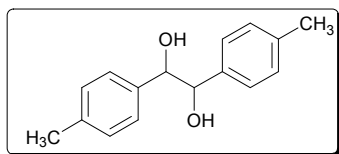


Table 5 entry 2, beige solid:

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ □□ppm: 2.22 (s, 6H, 2 CH<sub>3</sub>), 2.26 (s, 6H, 2 CH<sub>3</sub>), 4.57 (s, 2H, CH-OH *dl form*), 4.65 (s, 2H, CH-OH *meso form*), 7.10-6.93 (m, 8H, CH<sub>Ar</sub>),

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ□□ □□ppm: 21.2 (4 CH<sub>3</sub>), 78.1 (CH-OH *meso form*), 78.8 (CH-OH *dl form*), 126.9 (2 CH<sub>Ar</sub>), 127.1 (2 CH<sub>Ar</sub>), 128.8 (2 CH<sub>Ar</sub>), 129.0 (2 CH<sub>Ar</sub>), 137.0 (2 C<sub>IV</sub>), 137.5 (C<sub>IV</sub>), 137.8 (C<sub>IV</sub>),

MS (ESI): 243.13 [M + H]<sup>+</sup>, 267.12 [M + Na]<sup>+</sup>,  
mp= 161-163°C [lit 161-180°C]

### 1,2-Bis(4-chlorophenyl)-1,2-ethanediol (*dl* and *meso*) (2o)

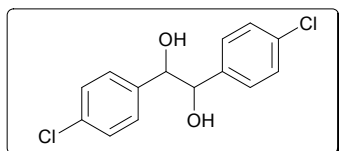


Table 5 entry 3, white solid:

*dl*-2o: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ□□ □□ppm: 4.55 (s, 2H, CH-OH), 6.95 (d, *J* = 8.4 Hz, 4H, 2 CH<sub>Ar</sub>), 7.14 (d, *J* = 8.4 Hz, 4H, 2 CH<sub>Ar</sub>),

*meso*-2o: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ□□ □□ppm: 4.77 (s, 2H, CH-OH), 7.03 (dd, *J* = 8.4, 1.6 Hz, 4H, 2 CH<sub>Ar</sub>), 7.18 (m, 4H, CH<sub>Ar</sub>),

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ□□ □□ppm: 77.2 (CH *meso form*), 78.6 (CH *dl form*), 128.4 (CH<sub>Ar</sub>), 128.4 (CH<sub>Ar</sub>), 128.4 (CH<sub>Ar</sub>), 128.9 (CH<sub>Ar</sub>), 131.6 (C<sub>IV</sub>), 133.9 (C<sub>IV</sub>), 137.8 (C<sub>IV</sub>), 137.9 (C<sub>IV</sub>),

MS (ESI): 283.02 (100%), 285.02 (64%), 284.02 (15%) [M + H]<sup>+</sup>  
mp= 147-149°C

### 1,2-Bis(4-bromophenyl)-1,2-ethanediol (*dl* and *meso*) (2p)

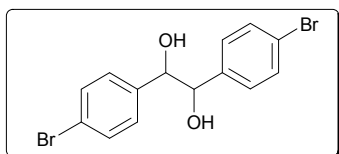


Table 5 entry 4, white solid:

*dl*-2p: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ□□ □□ppm: 4.53 (s, 2H, CH-OH), 6.89 (d, *J* = 8.4 Hz, 4H, CH<sub>Ar</sub>), 7.30 (d, *J* = 8.4 Hz, 4H, CH<sub>Ar</sub>),

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ□□ □□ppm: 78.5 (CH), 122.2 (C<sub>IV</sub>), 128.7 (2 CH<sub>Ar</sub>), 131.4 (2 CH<sub>Ar</sub>), 138.4 (C<sub>IV</sub>),

*meso*-2p: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ□□ □□ppm: 4.74 (s, 2H, CH-OH), 6.98 (dd, *J* = 8.4, 1.6 Hz, 4H, CH<sub>Ar</sub>), 7.34 (d, *J* = 8.4, 1.6 Hz, 4H, CH<sub>Ar</sub>),

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ□□ □□ppm: 77.2 (CH), 122.1 (C<sub>IV</sub>), 128.7 (2 CH<sub>Ar</sub>), 131.3 (2 CH<sub>Ar</sub>), 138.3 (C<sub>IV</sub>),

MS (ESI): 370.92 (51%) [M + H]<sup>+</sup>, 372.92 (100%), 374.92 (49%)  
mp= 155-157°C

### 1,2-Bis(2,3-dichlorophenyl)-1,2-ethanediol (*dl* and *meso*) (2r)

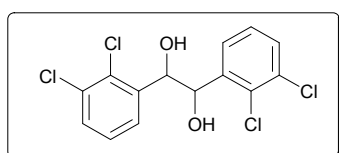


Table 6 entry 17, white solid:

**dl-2r:**  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 2.12 (bs, 2H, OH), 5.31 (s, 2H,  $\text{CH-OH}$ ), 7.20 (t,  $J = 8.0$  Hz, 2H,  $\text{CH}_{\text{Ar}}$ ), 7.36 (dd,  $J = 8.0, 1.6$  Hz, 2H,  $\text{CH}_{\text{Ar}}$ ), 7.55 (dd,  $J = 8.0, 1.6$  Hz, 2H,  $\text{CH}_{\text{Ar}}$ ),

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 73.0 (CH), 129.8 ( $\text{C}_{\text{IV}}$ ), 127.2 ( $\text{CH}_{\text{Ar}}$ ), 127.3 ( $\text{CH}_{\text{Ar}}$ ), 130.1 ( $\text{CH}_{\text{Ar}}$ ), 131.5 ( $\text{C}_{\text{IV}}$ ), 139.7 ( $\text{C}_{\text{IV}}$ ),

**meso-2r:**  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 2.12 (bs, 2H, OH), 5.57 (s, 2H,  $\text{CH-OH}$ ), 7.03 (t,  $J = 8.0$  Hz, 2H,  $\text{CH}_{\text{Ar}}$ ), 7.11 (dd,  $J = 7.9, 1.7$  Hz, 2H,  $\text{CH}_{\text{Ar}}$ ), 7.55 (dd,  $J = 8.0, 1.6$  Hz, 2H,  $\text{CH}_{\text{Ar}}$ ),

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 77.1 (CH), 126.9 (2  $\text{CH}_{\text{Ar}}$ ), 129.8 ( $\text{CH}_{\text{Ar}}$ ), 129.8 ( $\text{C}_{\text{IV}}$ ), 132.5 ( $\text{C}_{\text{IV}}$ ), 138.5 ( $\text{C}_{\text{IV}}$ ),

MS (ESI): 352.94 (100%), 350.94 (78%), 354.94 (48%) [ $\text{M} + \text{H}$ ] $^+$

mp= 177-179°C

### 1,2-di(furan-2-yl)ethane-1,2-diol (*dl* and *meso*) (2s)

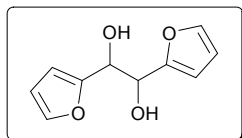


Table 6 entry 21, yellow oil:

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 2.75 (bs, 2H, OH *dl* and *meso* forms), 4.98 (s, 2H,  $\text{CH-OH}$  *dl* form), 5.01 (s, 2H,  $\text{CH-OH}$  *meso* form), 6.33-6.23 (m, 4H,  $\text{CH}_{\text{Ar}}$  *dl* and *meso* forms),

7.38-7.34 (m, 2H,  $\text{CH}_{\text{Ar}}$  *dl* and *meso* forms),

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 69.9 ( $\text{CH-OH}$  *dl* form), 70.1 ( $\text{CH-OH}$  *meso* form), 108.0 ( $\text{CH}_{\text{Ar}}$ ), 108.2 ( $\text{CH}_{\text{Ar}}$ ), 110.3 ( $\text{CH}_{\text{Ar}}$ ), 110.4 ( $\text{CH}_{\text{Ar}}$ ), 142.4 ( $\text{CH}_{\text{Ar}}$ ), 142.4 ( $\text{CH}_{\text{Ar}}$ ), 152.6 ( $\text{C}_{\text{IV}}$ ), 152.8 ( $\text{C}_{\text{IV}}$ ),

MS (ESI): 195.06 [ $\text{M} + \text{H}$ ] $^+$ , 218.06 [ $\text{M} + \text{Na}$ ] $^+$ ,

### 2,3-diphenylbutane-2,3-diol (*dl* and *meso*) (2t)

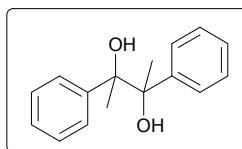


Table 6 entry 22, white solid:

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 1.41 (s, 6H,  $\text{CH}_3$  *dl* form), 1.49 (s, 6H,  $\text{CH}_3$  *meso* form), 2.39 (bs, 2H, OH *dl* and *meso* forms), 7.16-7.10 (m, 10H,  $\text{CH}_{\text{Ar}}$  *dl* and *meso* forms),

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 25.0 ( $\text{CH}_3$  *dl* form), 25.1 ( $\text{CH}_3$  *meso* form), 78.7 ( $\text{C}_{\text{IV}}$  *meso* form), 78.9 ( $\text{C}_{\text{IV}}$  *dl* form), 126.9 ( $\text{CH}_{\text{Ar}}$ ), 127.0 (2  $\text{CH}_{\text{Ar}}$ ), 127.1 ( $\text{CH}_{\text{Ar}}$ ), 127.2 (2  $\text{CH}_{\text{Ar}}$ ), 127.3 (2  $\text{CH}_{\text{Ar}}$ ), 127.4 (2  $\text{CH}_{\text{Ar}}$ ), 143.4 ( $\text{C}_{\text{IV}}$ ), 143.8 ( $\text{C}_{\text{IV}}$ ),

MS (ESI): 243.13 [ $\text{M} + \text{H}$ ] $^+$ , 267.12 [ $\text{M} + \text{Na}$ ] $^+$ ,

mp= 127-130°C

### 2,3-bis(4-chlorophenyl)butane-2,3-diol (*dl* and *meso*) (2u)

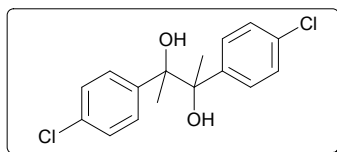


Table 6 entry 23, white solid:

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 1.39 (s, 6H,  $\text{CH}_3$  *dl* form), 1.46 (s, 6H,  $\text{CH}_3$  *meso* form), 2.22 (bs, 2H, OH *dl* and *meso* forms), 7.23-7.00 (m, 10H,  $\text{CH}_{\text{Ar}}$  *dl* and *meso* forms),

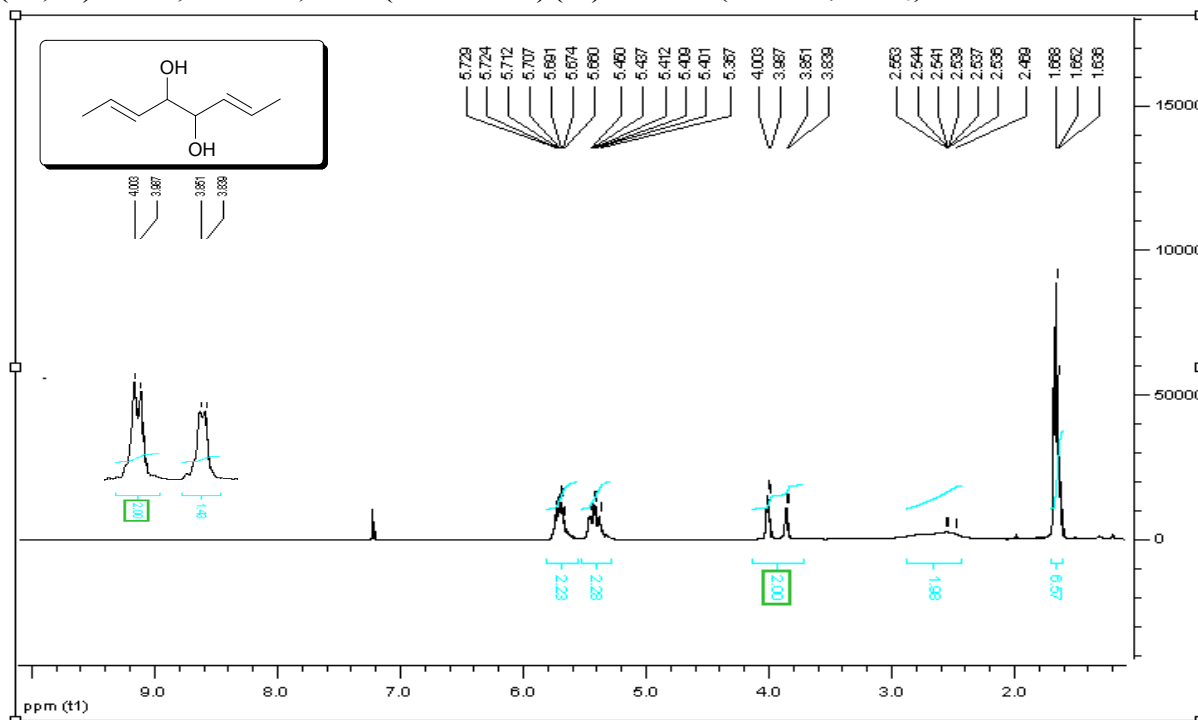
$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  ppm: 24.8 ( $\text{CH}_3$  *dl* form), 25.1 ( $\text{CH}_3$  *meso* form), 78.3 ( $\text{C}_{\text{IV}}$  *meso* form), 78.5 ( $\text{C}_{\text{IV}}$  *dl* form), 126.8 ( $\text{CH}_{\text{Ar}}$ ), 127.3 (2  $\text{CH}_{\text{Ar}}$ ), 127.4 ( $\text{CH}_{\text{Ar}}$ ), 128.5 (2  $\text{CH}_{\text{Ar}}$ ), 128.6 (2  $\text{CH}_{\text{Ar}}$ ), 128.8 (2  $\text{CH}_{\text{Ar}}$ ), 133.0 ( $\text{C}_{\text{IV}}$ ), 133.2 ( $\text{C}_{\text{IV}}$ ), 141.7 ( $\text{C}_{\text{IV}}$ ), 142.2 ( $\text{C}_{\text{IV}}$ ),

MS (ESI): 243.13 [ $\text{M} + \text{H}$ ] $^+$ , 267.12 [ $\text{M} + \text{Na}$ ] $^+$ ,

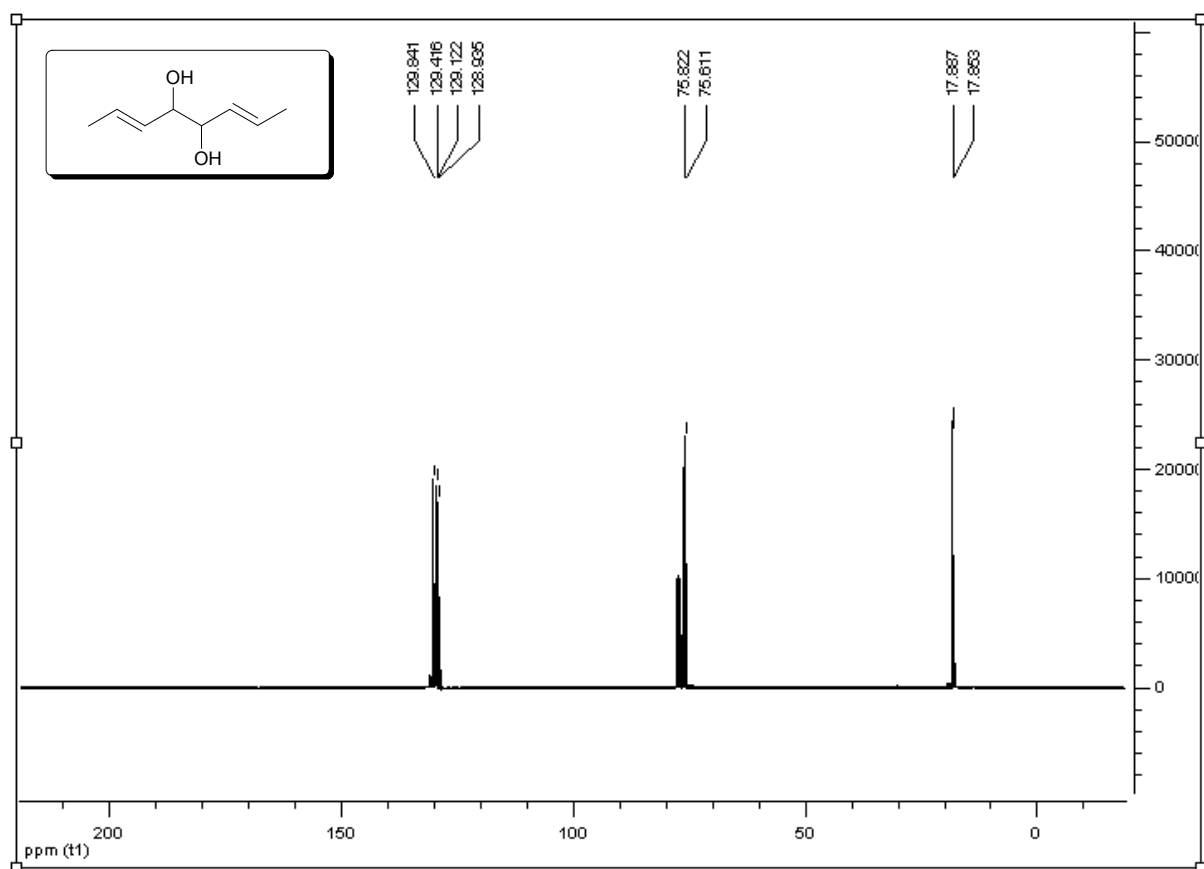
mp= 141-142°C

## SPECTRA

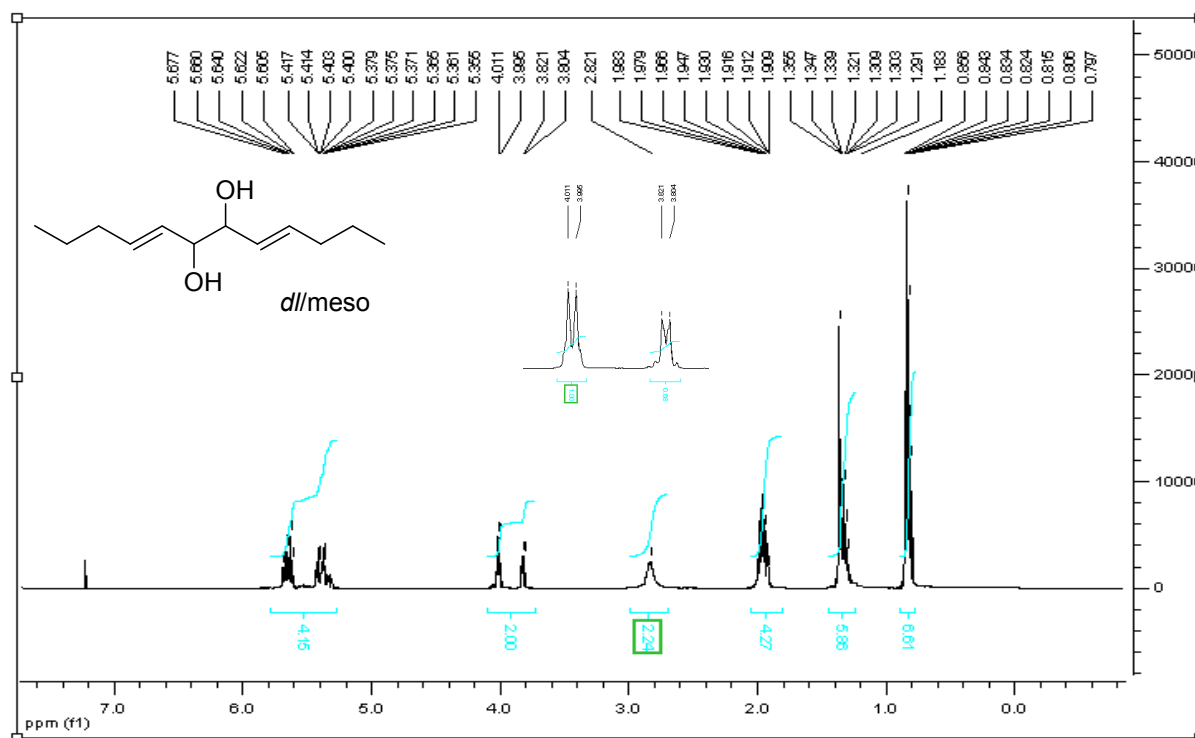
**(2E,6E)-octa-2,6-diene-4,5-diol (*dl* and *meso*) (2a): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



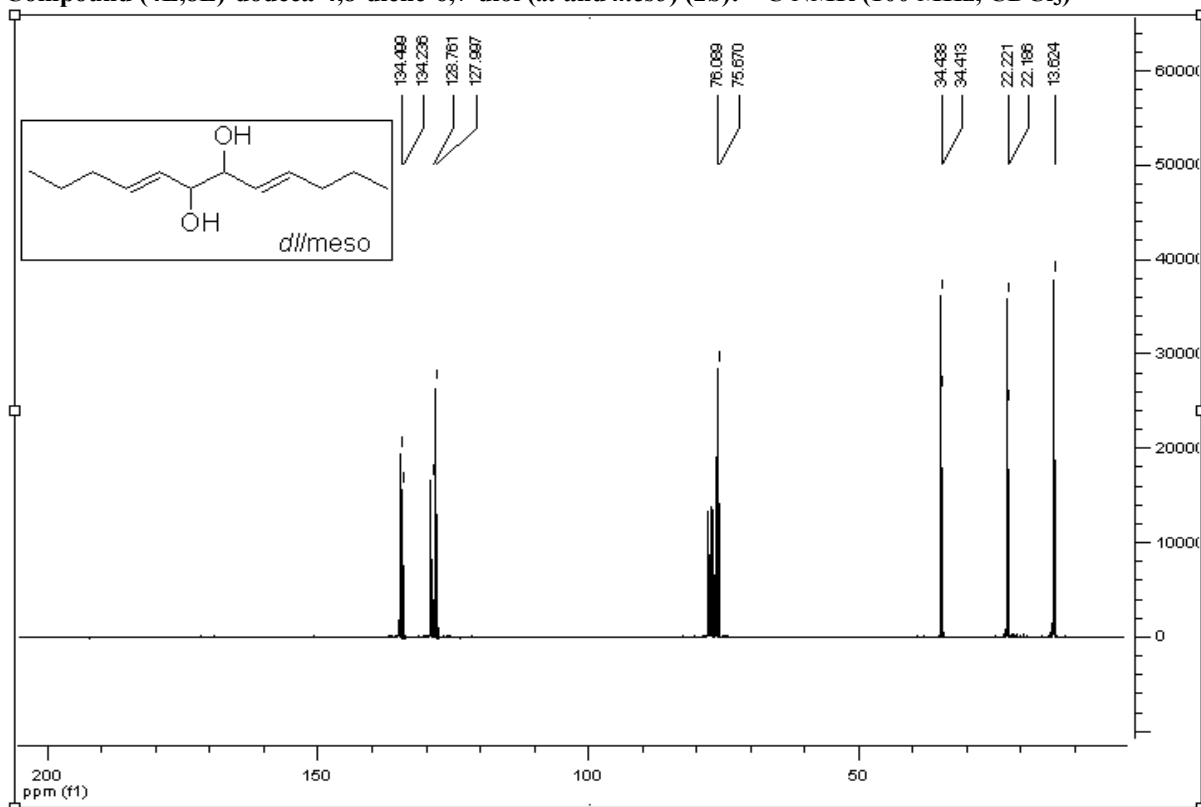
**(2E,6E)-octa-2,6-diene-4,5-diol (*dl* and *meso*) (2a): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



Compound (4E,8E)-dodeca-4,8-diene-6,7-diol (*dl* and *meso*) (2b):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )

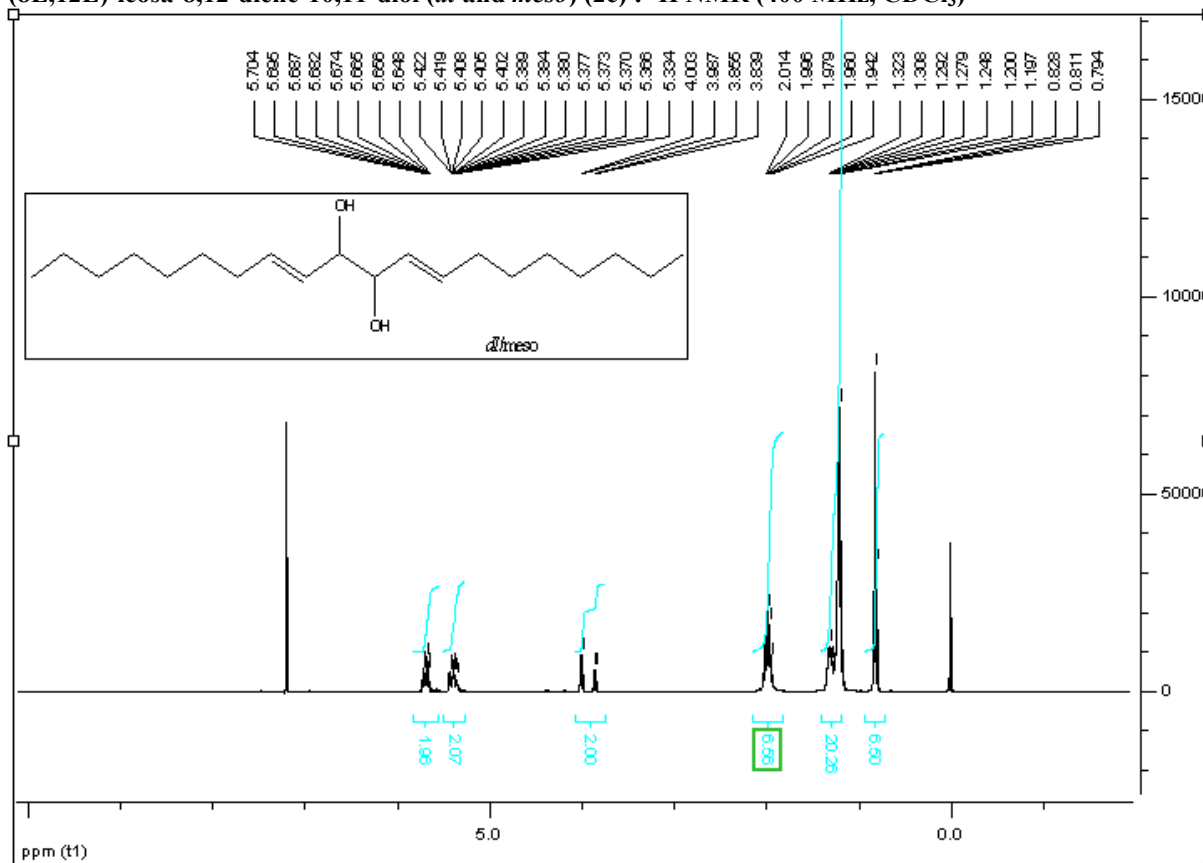


Compound (4E,8E)-dodeca-4,8-diene-6,7-diol (*dl* and *meso*) (2b):  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

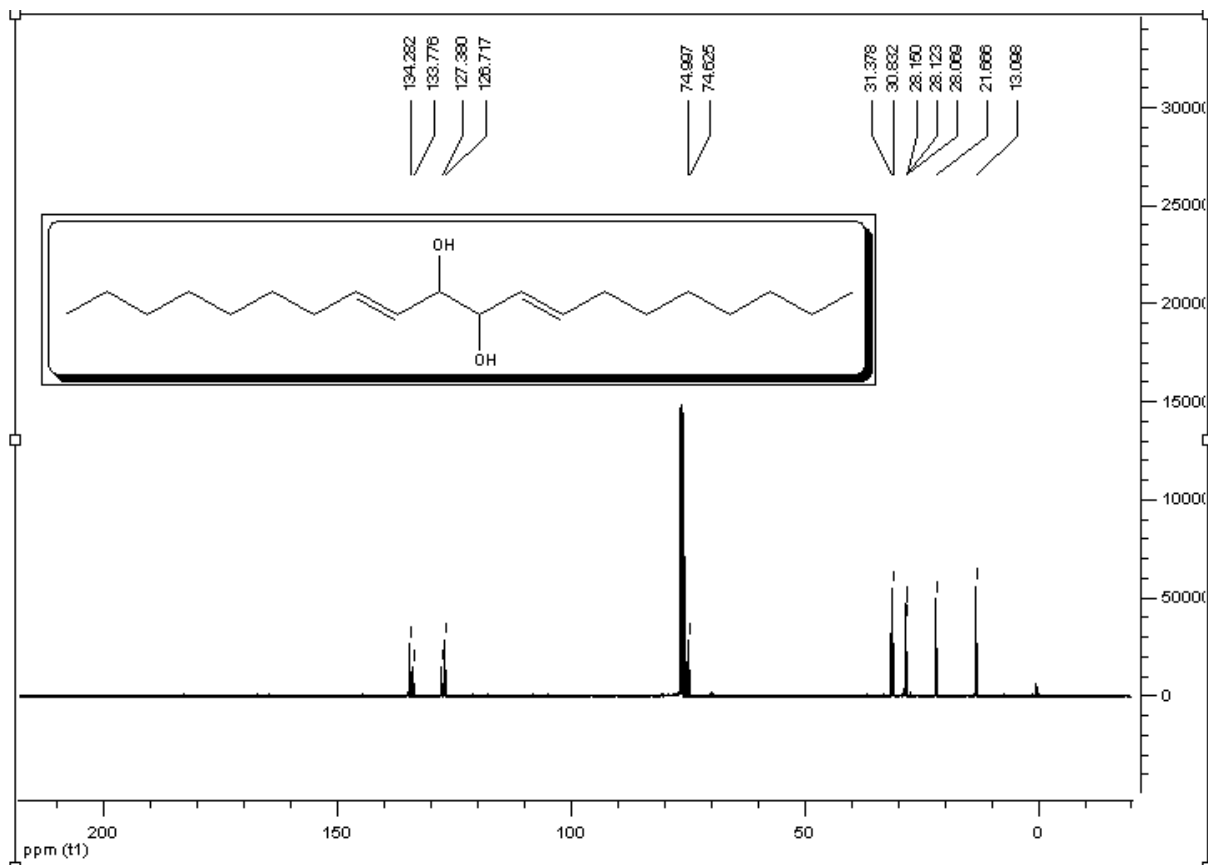




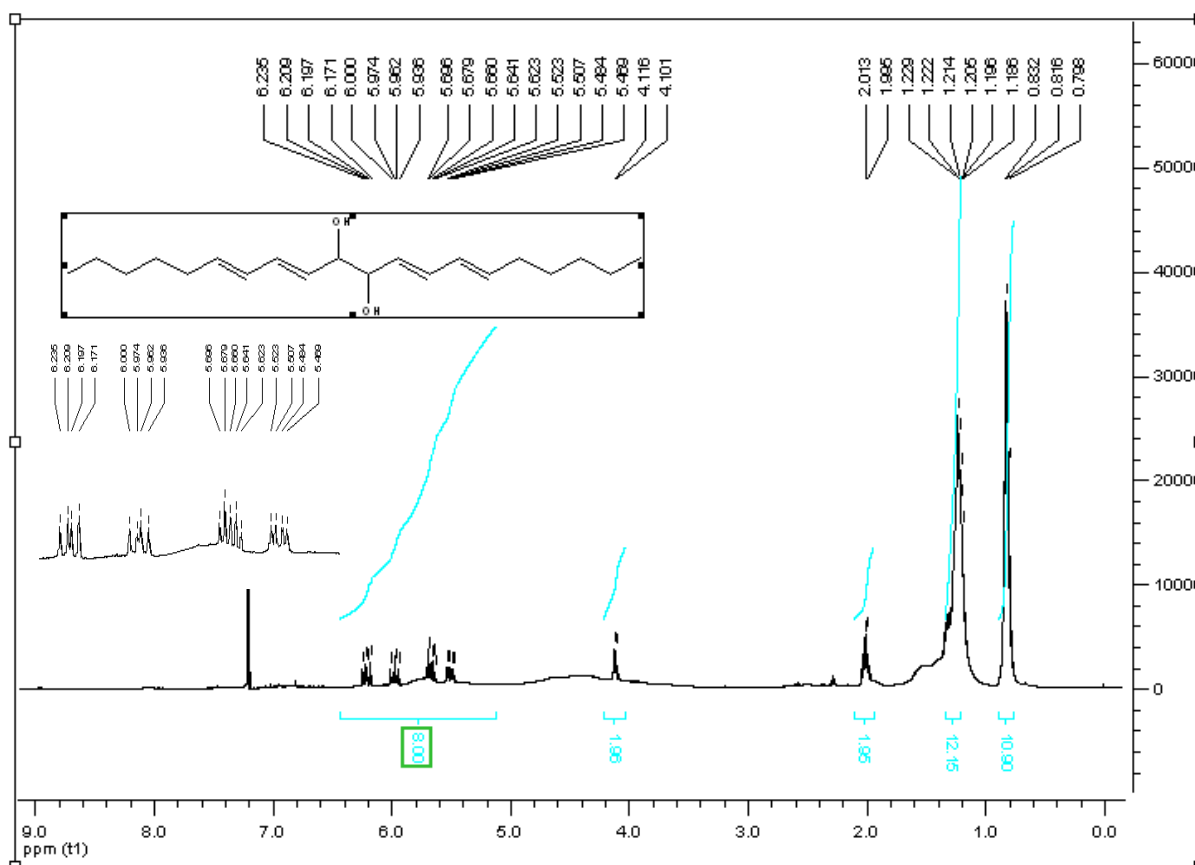
(8E,12E)-icosa-8,12-diene-10,11-diol (*dl* and *meso*) (2c) :  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



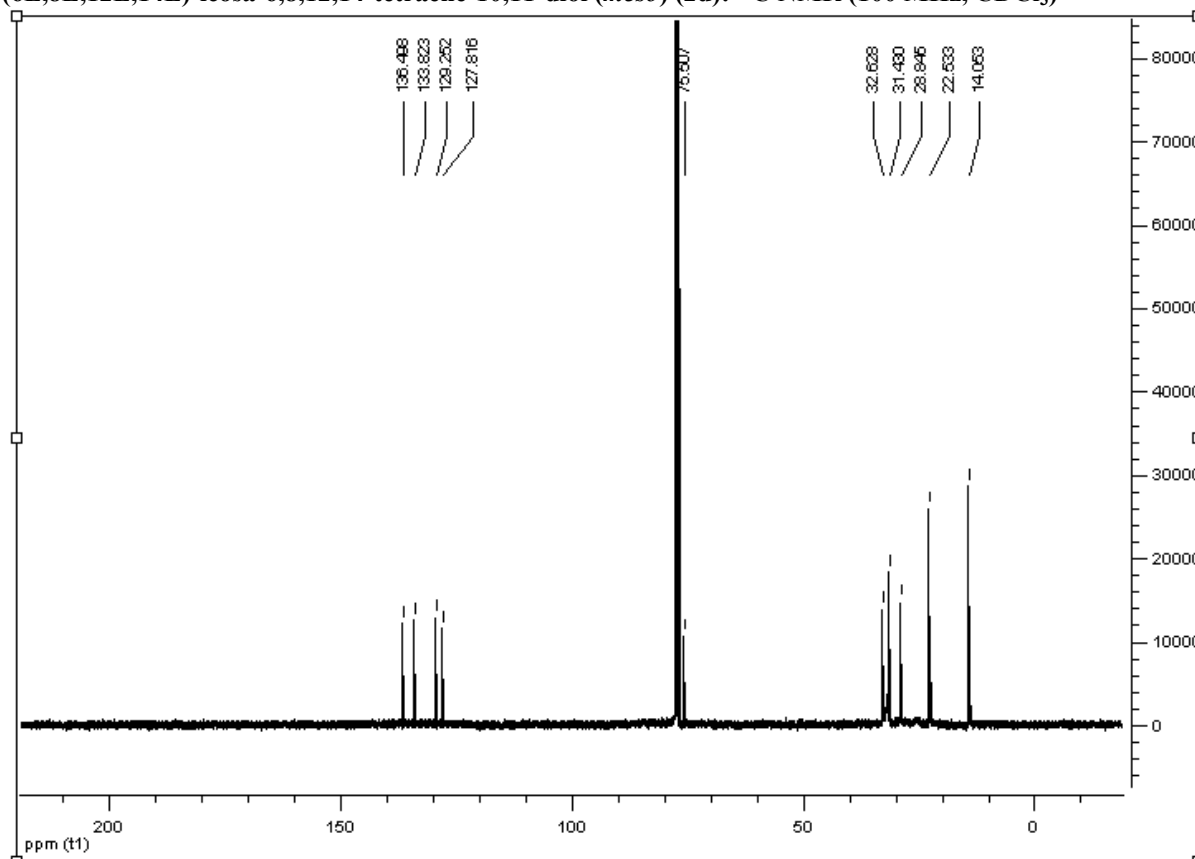
(8E,12E)-icosa-8,12-diene-10,11-diol (*dl* and *meso*) (2c) :  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



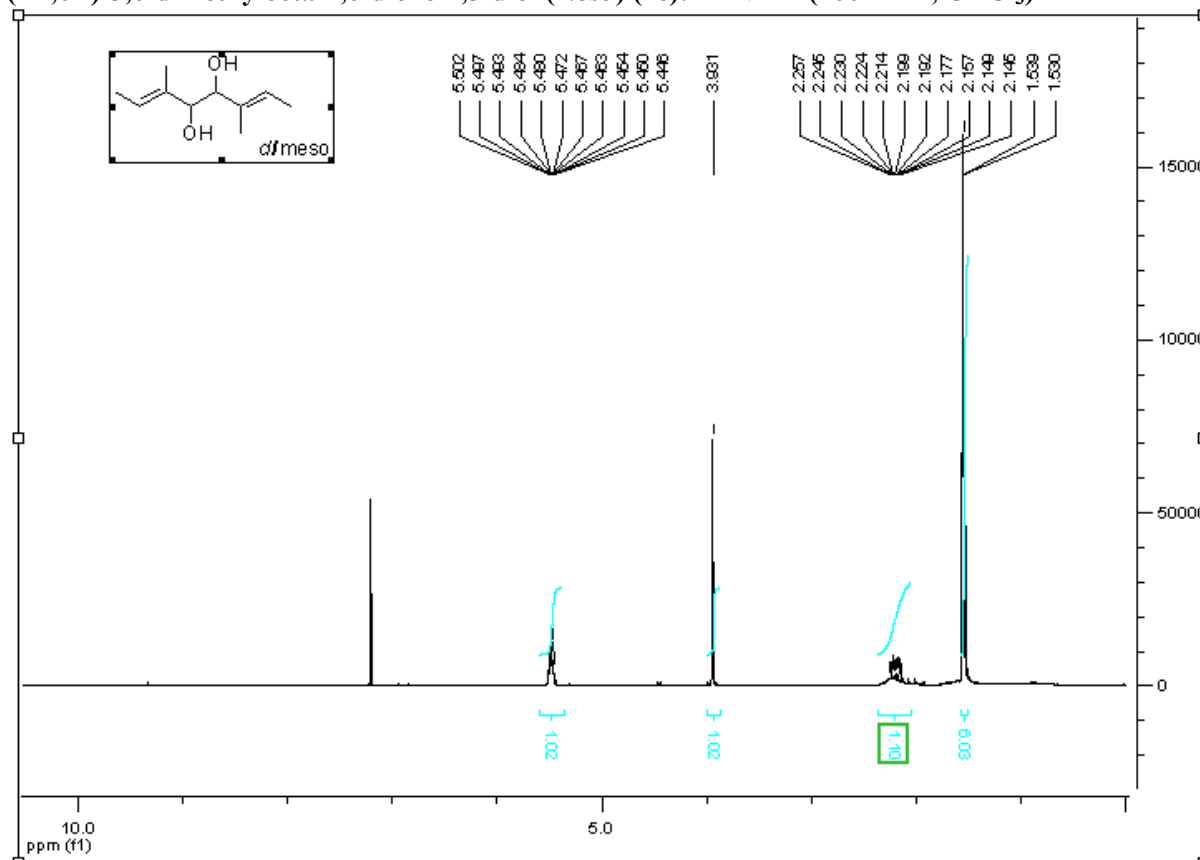
(6E,8E,12E,14E)-icosa-6,8,12,14-tetraene-10,11-diol (*meso*) (2d):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



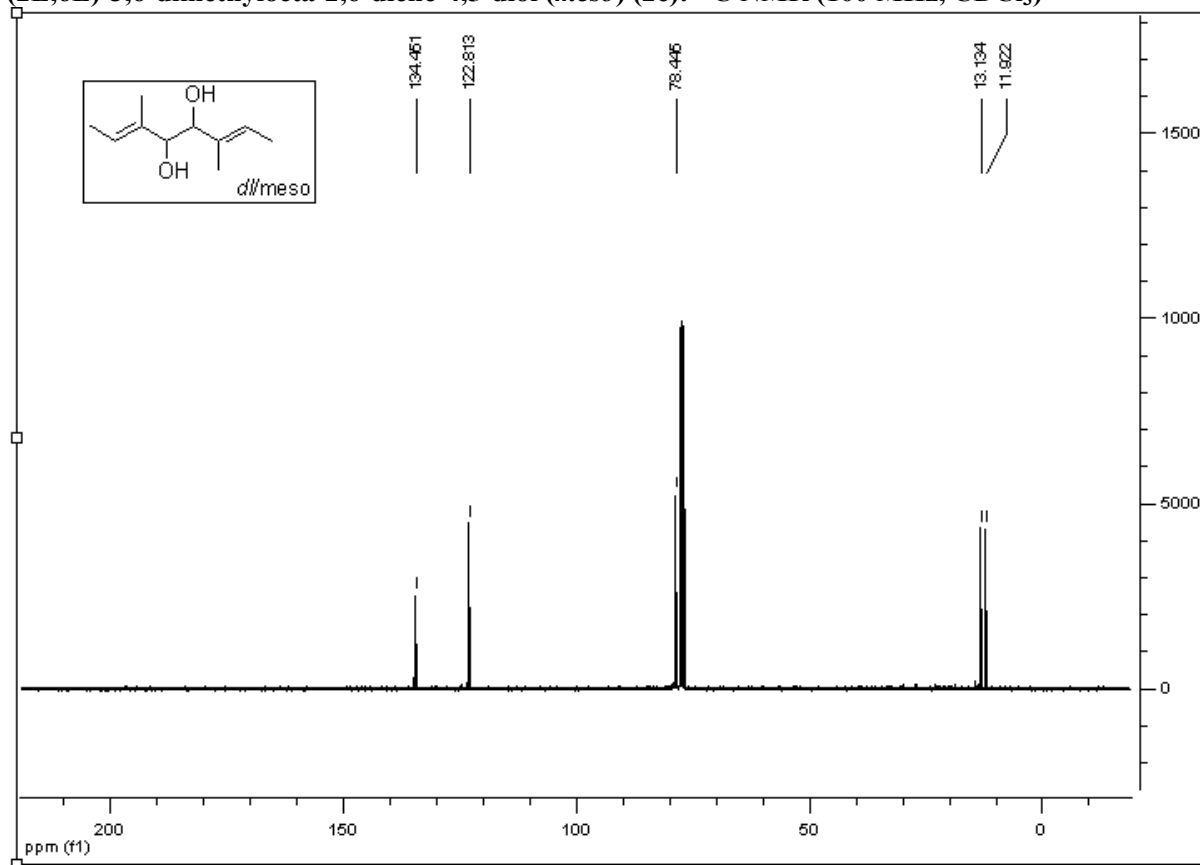
(6E,8E,12E,14E)-icosa-6,8,12,14-tetraene-10,11-diol (*meso*) (2d):  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



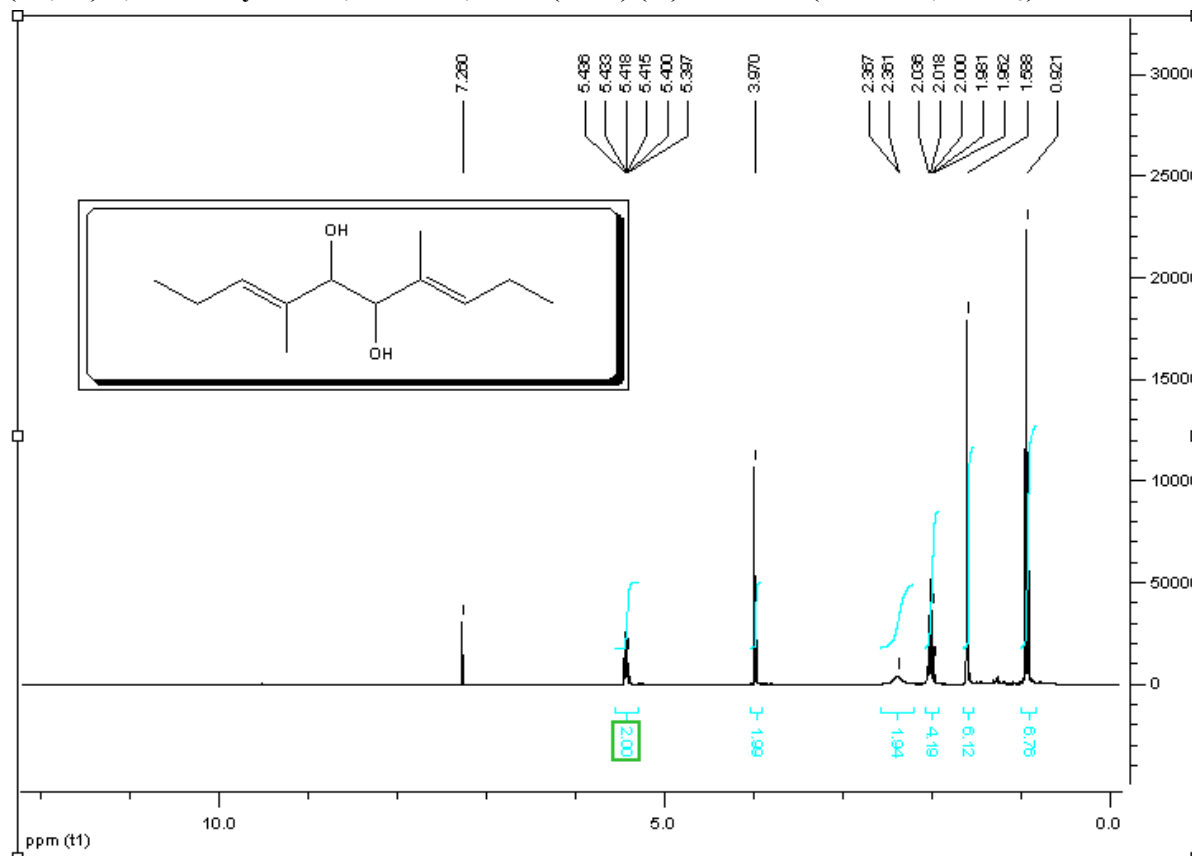
**(2E,6E)-3,6-dimethylocta-2,6-diene-4,5-diol (meso) (2e): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



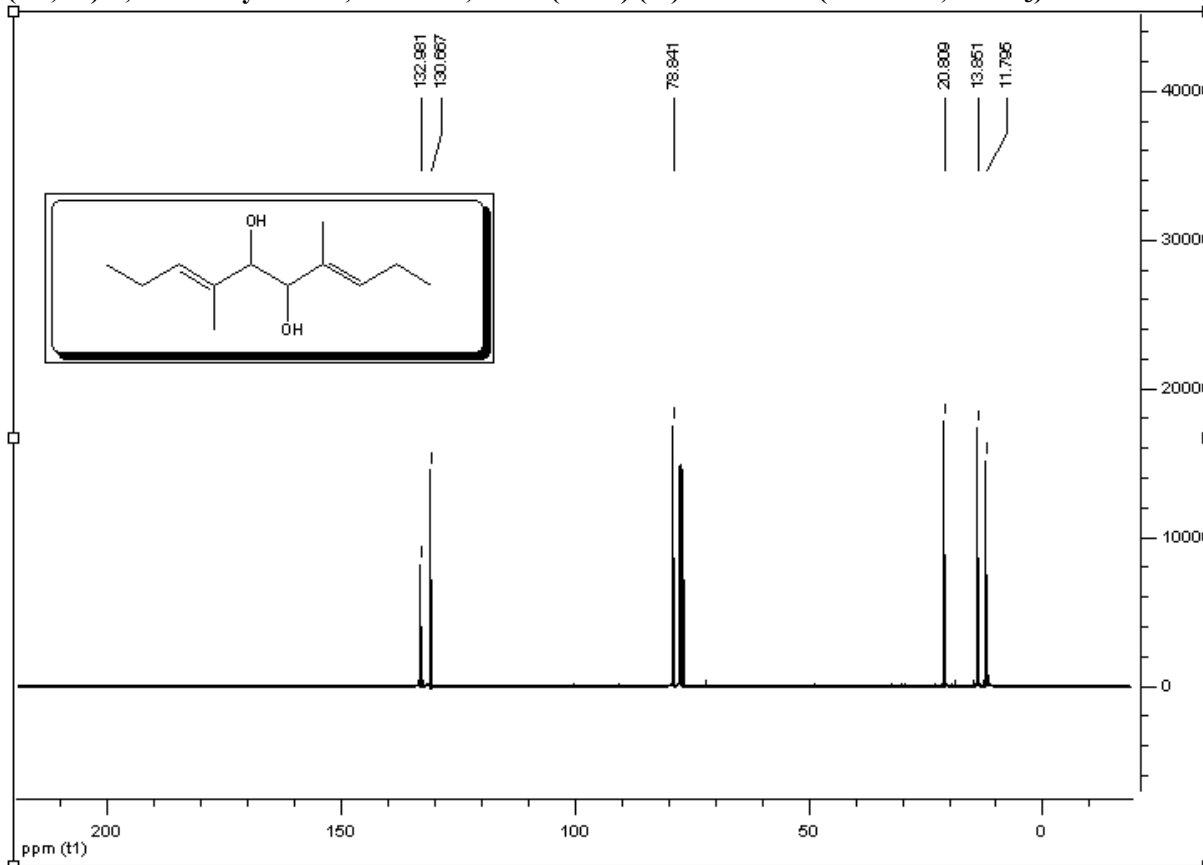
**(2E,6E)-3,6-dimethylocta-2,6-diene-4,5-diol (meso) (2e): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



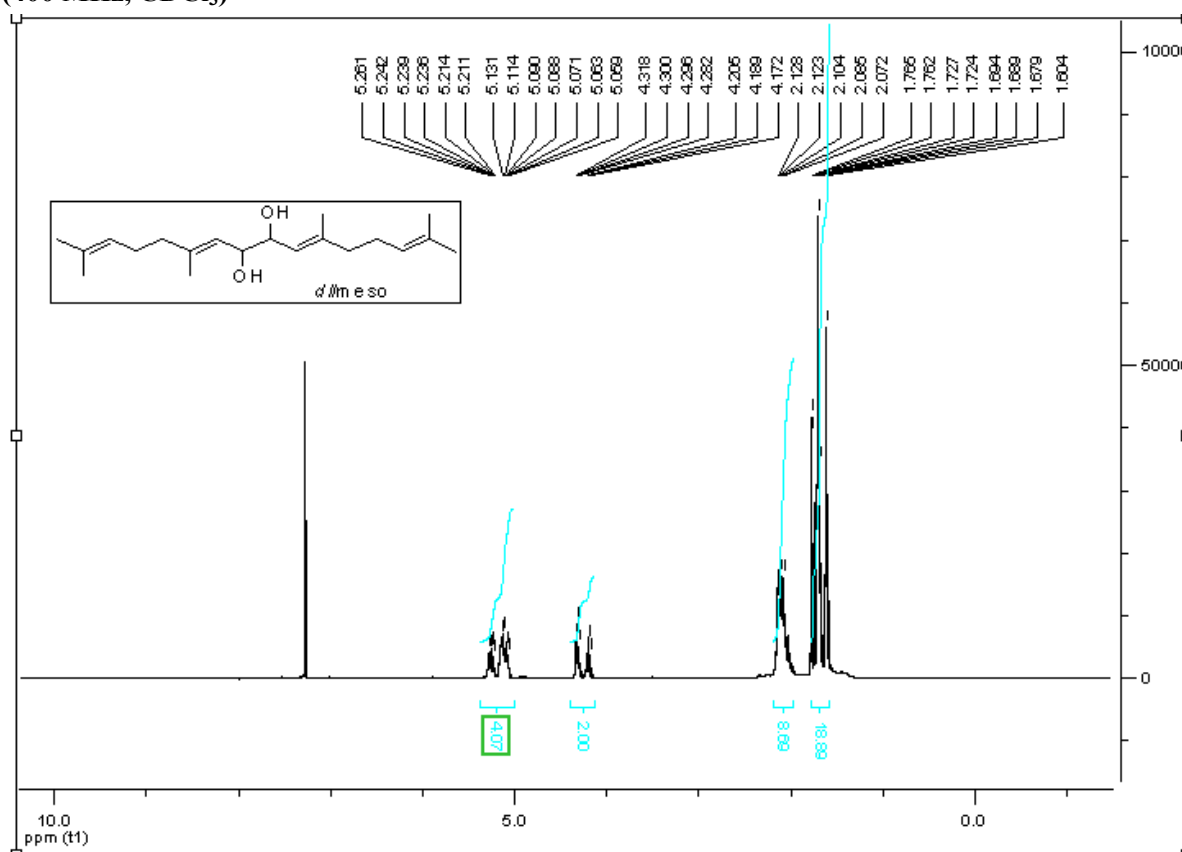
**(3E,7E)-4,7-dimethyldeca-3,7-diene-5,6-diol (meso) (2f):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )**



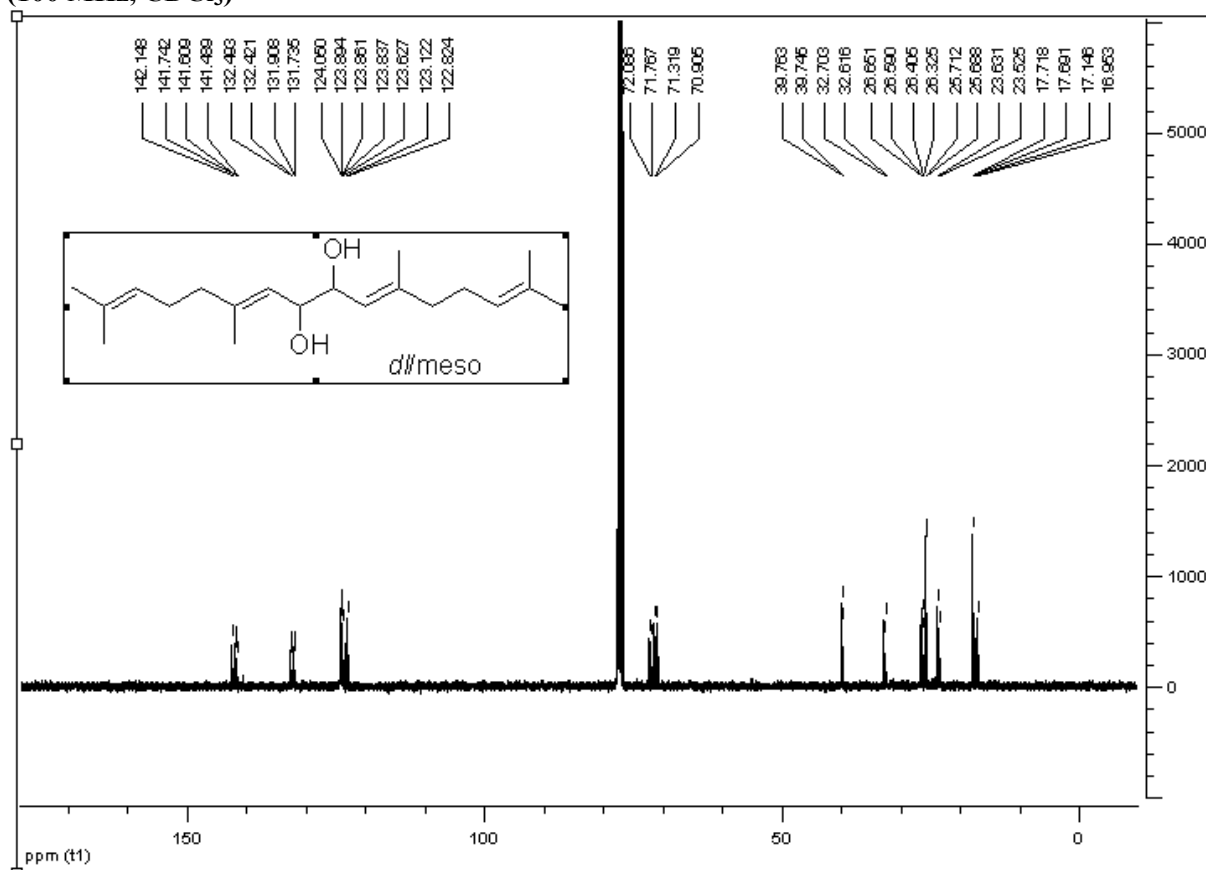
**(3E,7E)-4,7-dimethyldeca-3,7-diene-5,6-diol (meso) (2f):  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )**



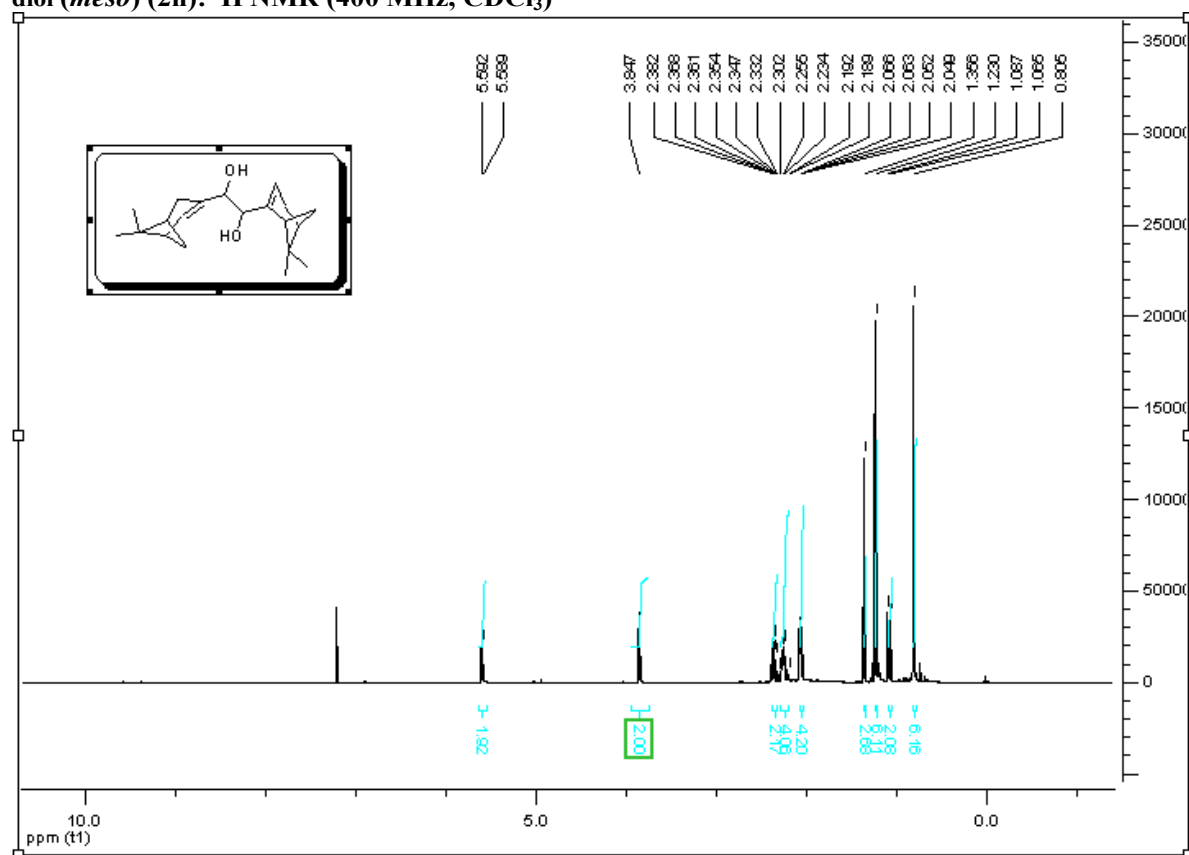
**(6E,10E)-2,6,11,15-tetramethylhexadeca-2,6,10,14-tetraene-8,9-diol (*dl* and *meso*) (2g): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



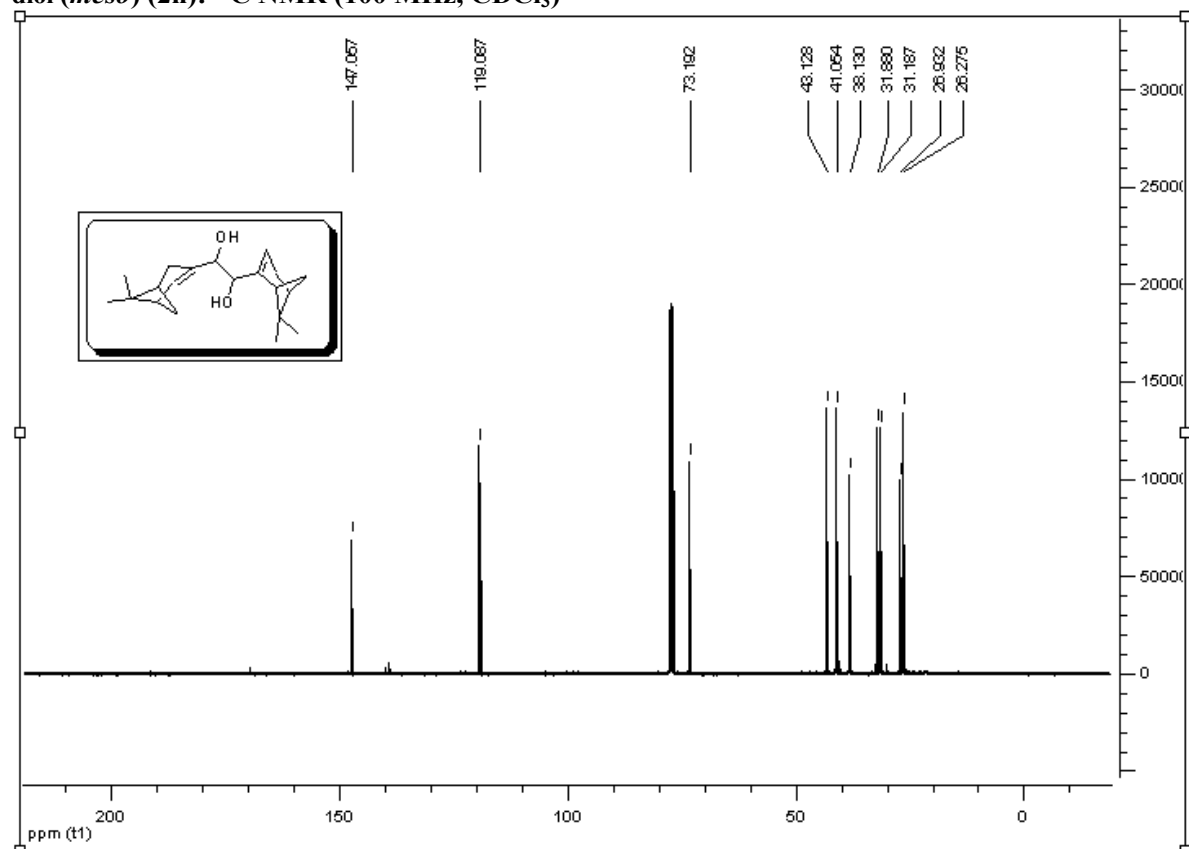
**(6E,10E)-2,6,11,15-tetramethylhexadeca-2,6,10,14-tetraene-8,9-diol (*dl* and *meso*) (2g): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



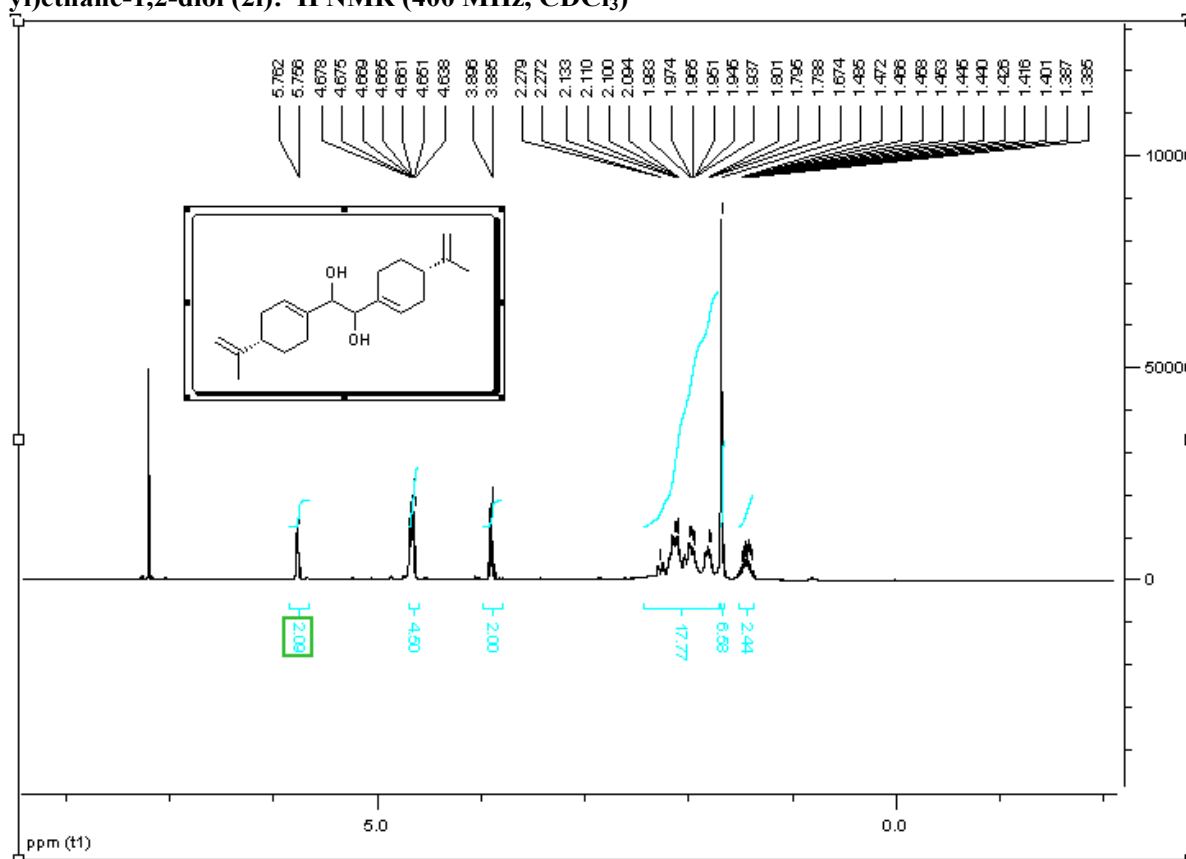
**(6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)-2-(6,6-dimethylbicyclo[3.1.1]hept-2-en-3-yl)ethane-1,2-diol (*meso*) (2h): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



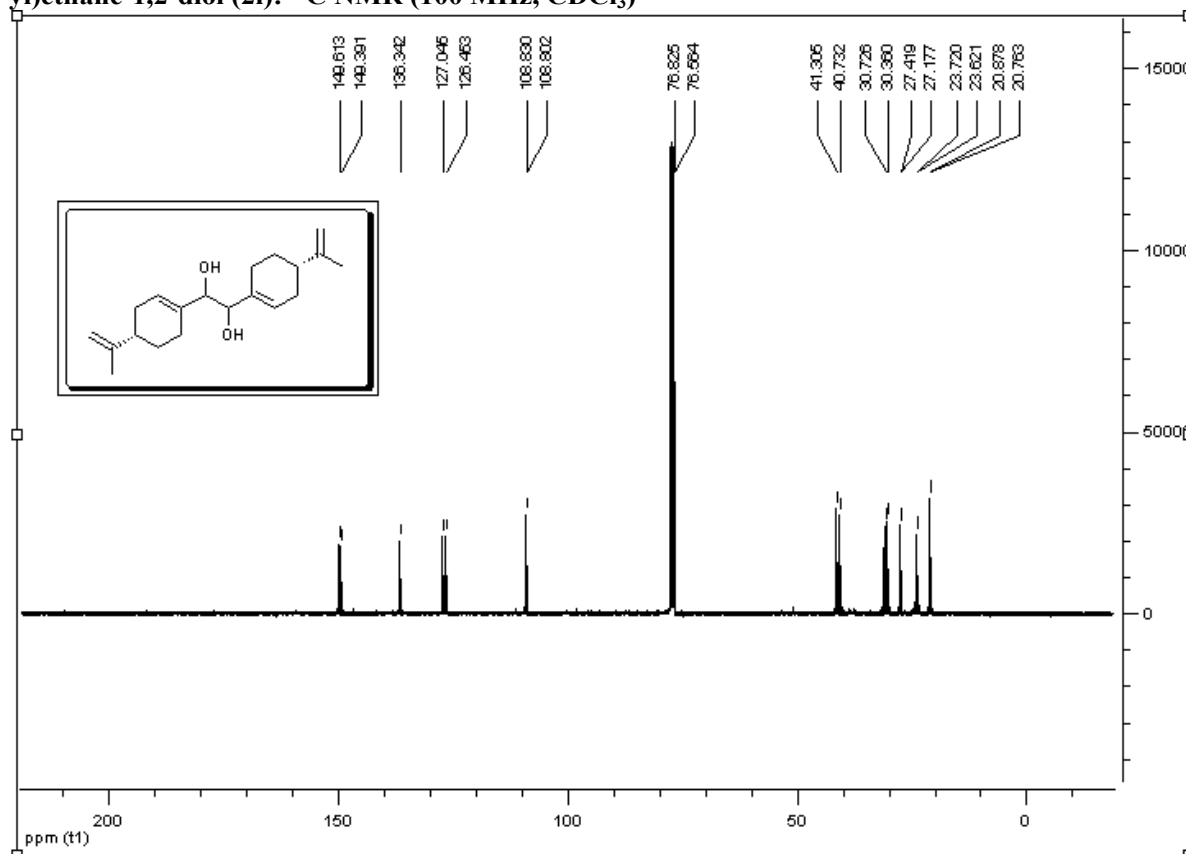
**(6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)-2-(6,6-dimethylbicyclo[3.1.1]hept-2-en-3-yl)ethane-1,2-diol (*meso*) (2h): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



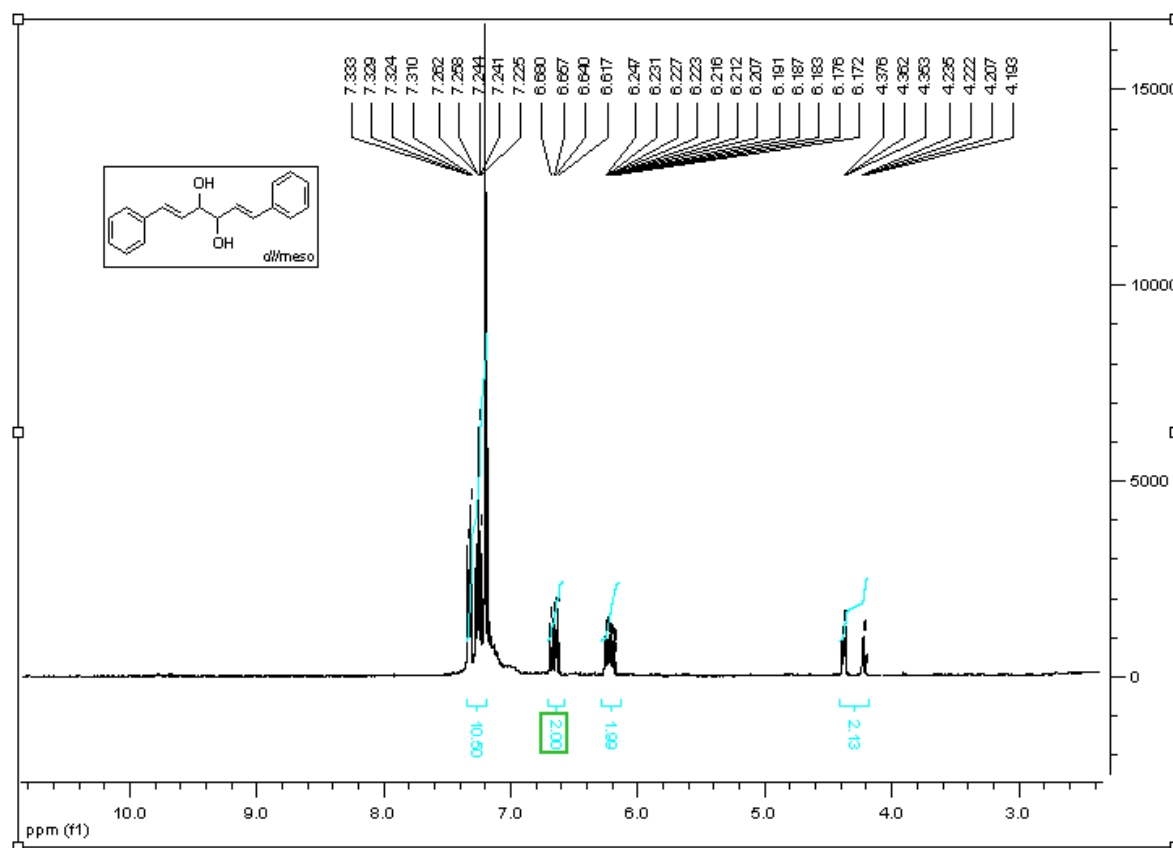
**1-((6S)-6-isopropylbicyclo[3.1.1]hept-2-en-2-yl)-2-((6R)-6-isopropylbicyclo[3.1.1]hept-2-en-3-yl)ethane-1,2-diol (2i): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



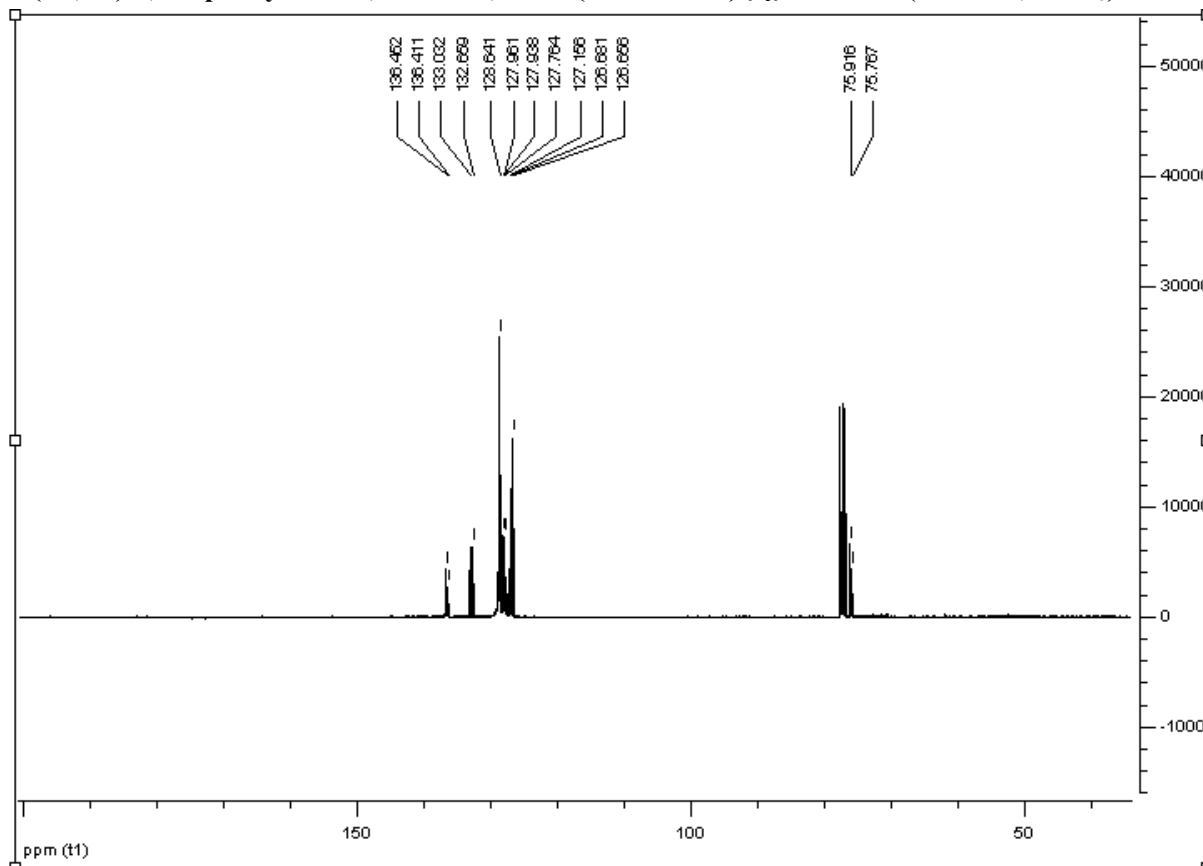
**1-((6S)-6-isopropylbicyclo[3.1.1]hept-2-en-2-yl)-2-((6R)-6-isopropylbicyclo[3.1.1]hept-2-en-3-yl)ethane-1,2-diol (2i): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



**(1E,5E)-1,6-diphenylhexa-1,5-diene-3,4-diol (*dl* and *meso*) (2j): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**

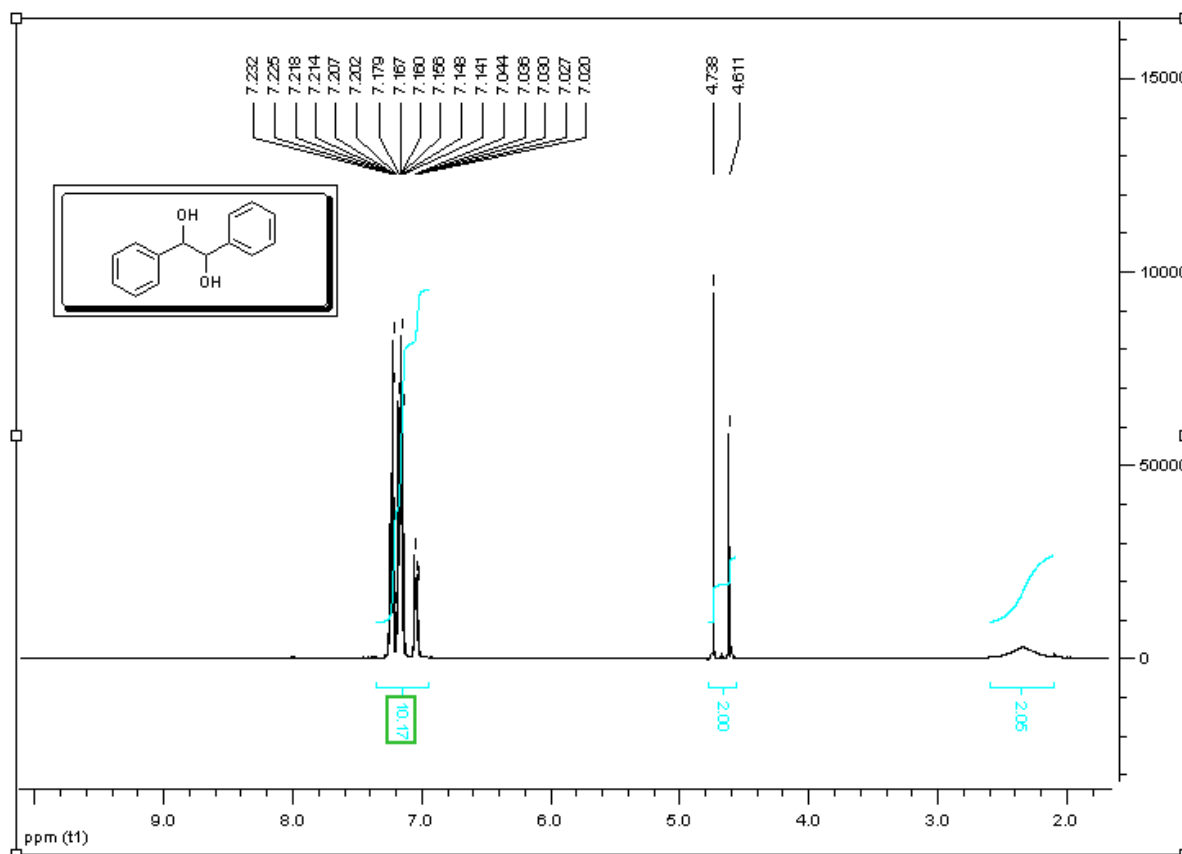


**(1E,5E)-1,6-diphenylhexa-1,5-diene-3,4-diol (*dl* and *meso*) (2j): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**

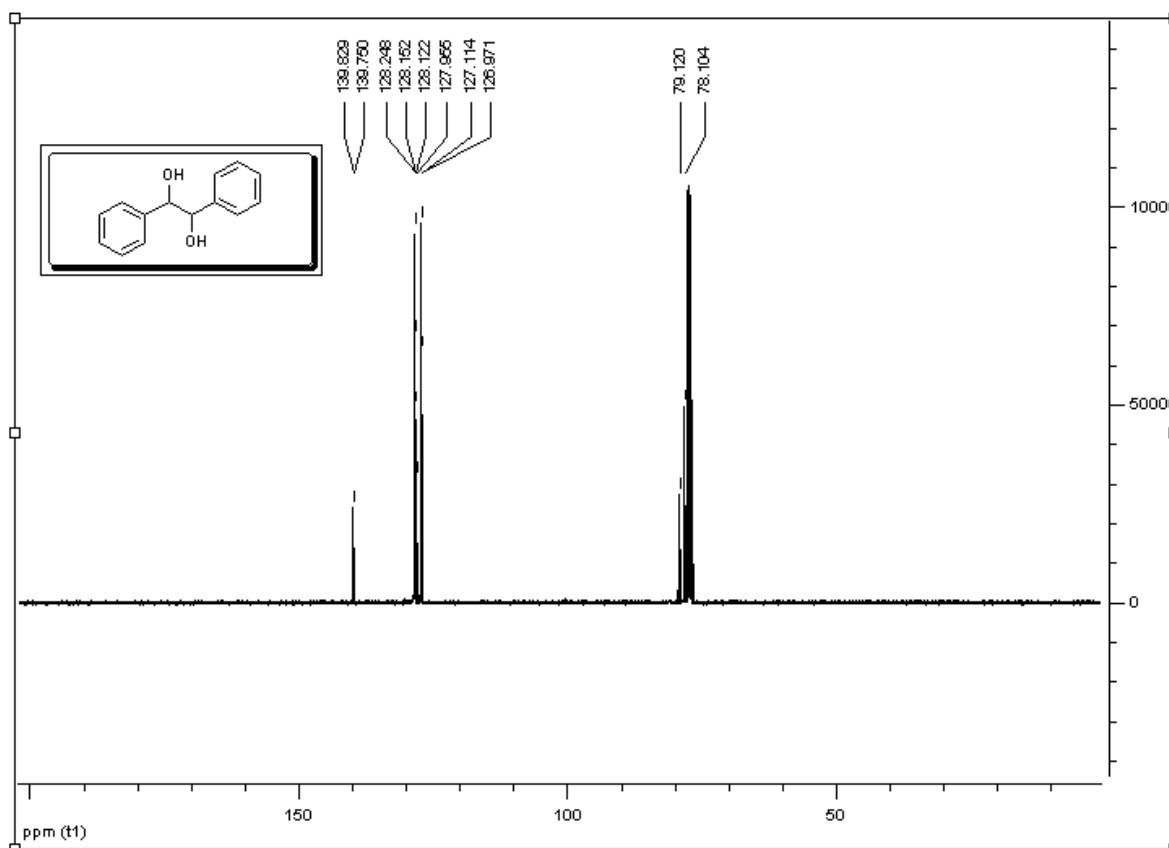




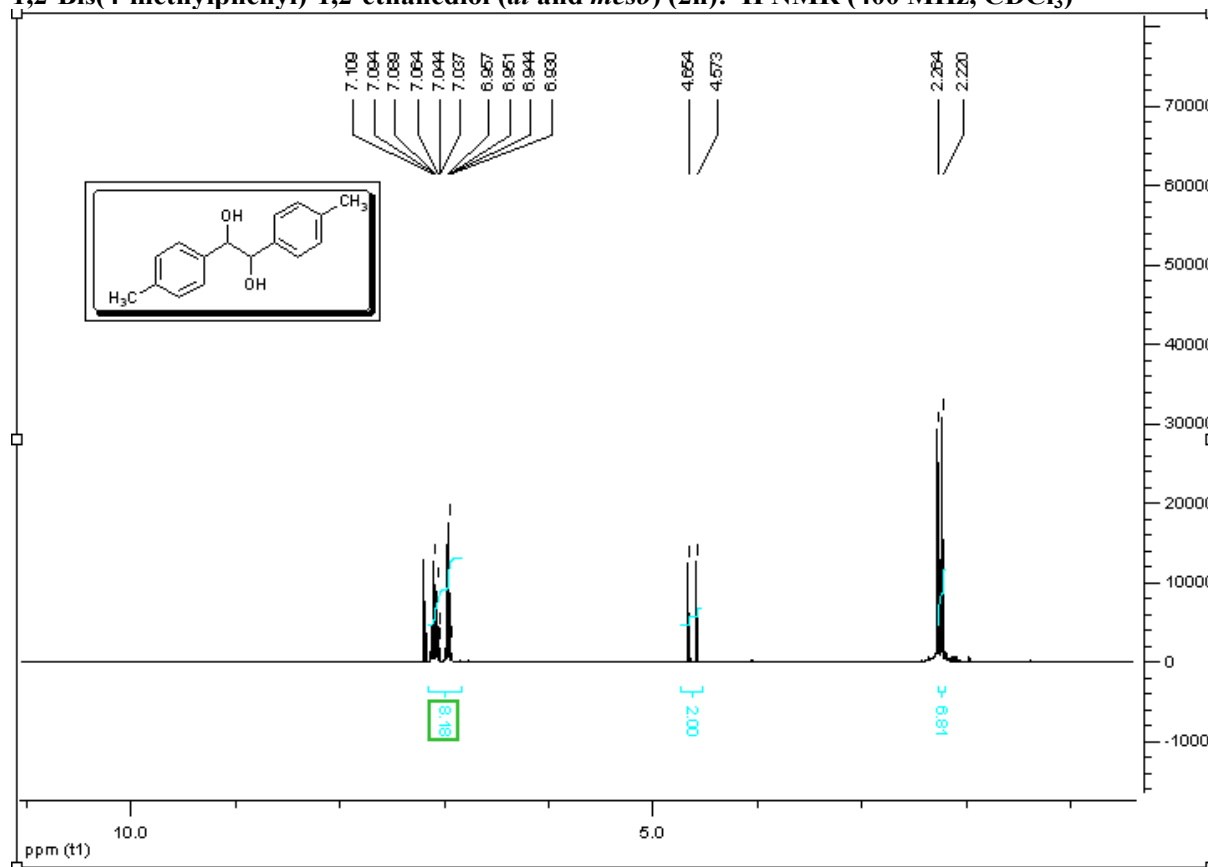
**1,2-Diphenyl-1,2-ethanediol (*dl* and *meso*) (2m): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



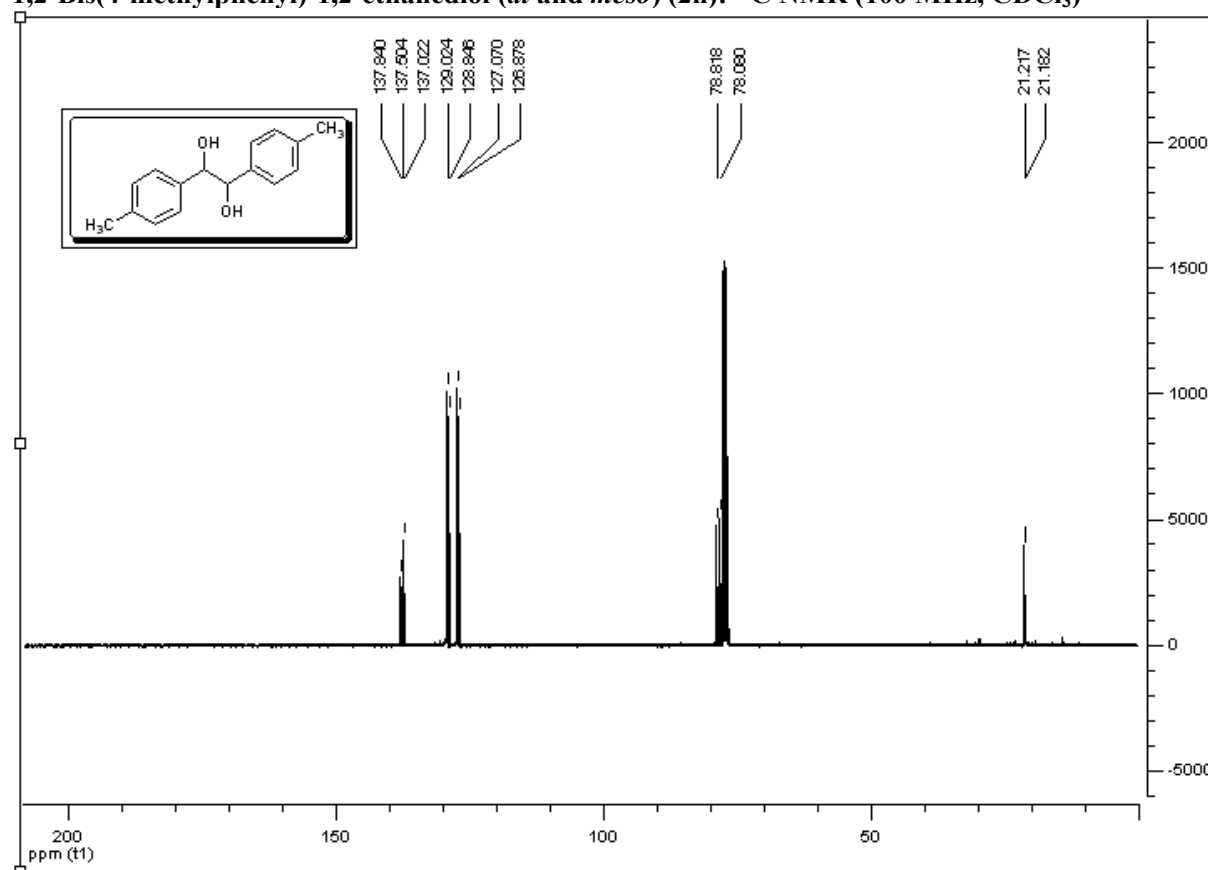
**1,2-Diphenyl-1,2-ethanediol (*dl* and *meso*) (2m): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



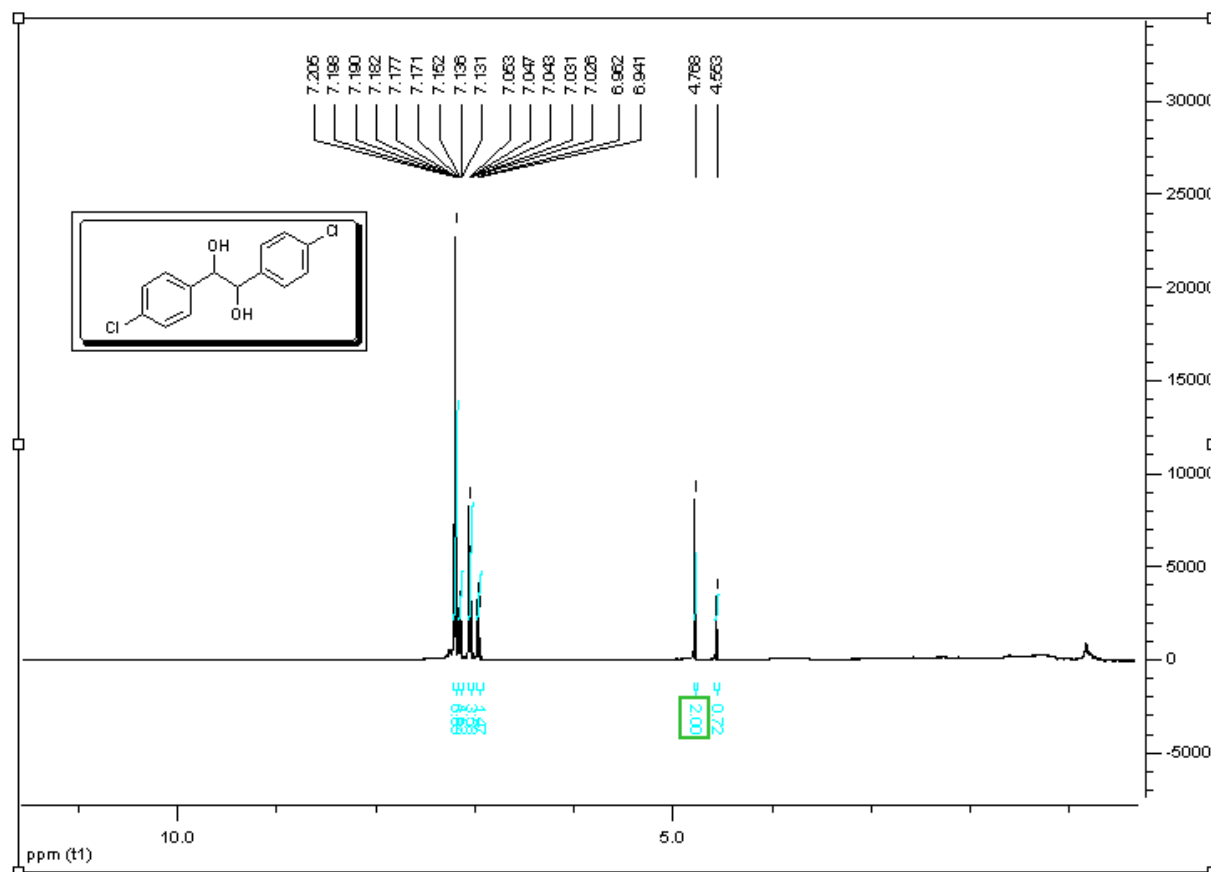
**1,2-Bis(4-methylphenyl)-1,2-ethanediol (*dl* and *meso*) (2n): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



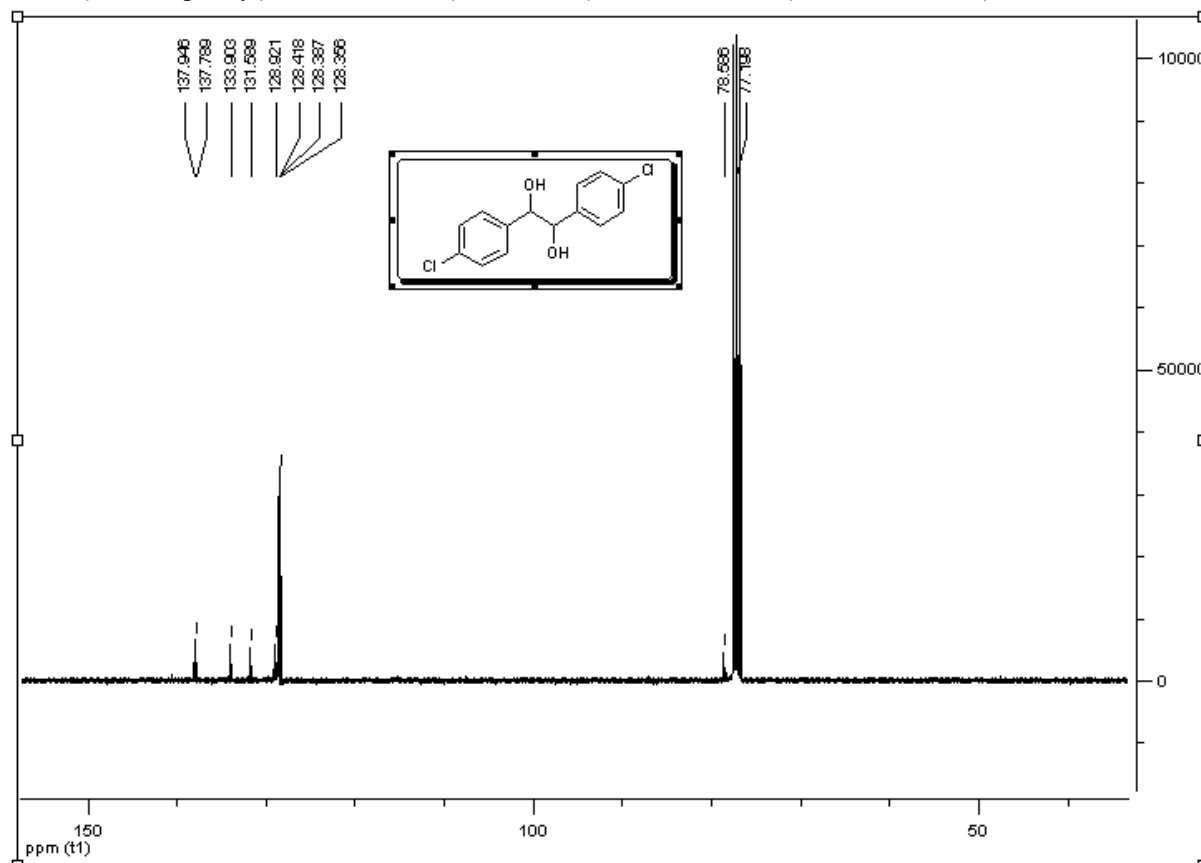
**1,2-Bis(4-methylphenyl)-1,2-ethanediol (*dl* and *meso*) (2n): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



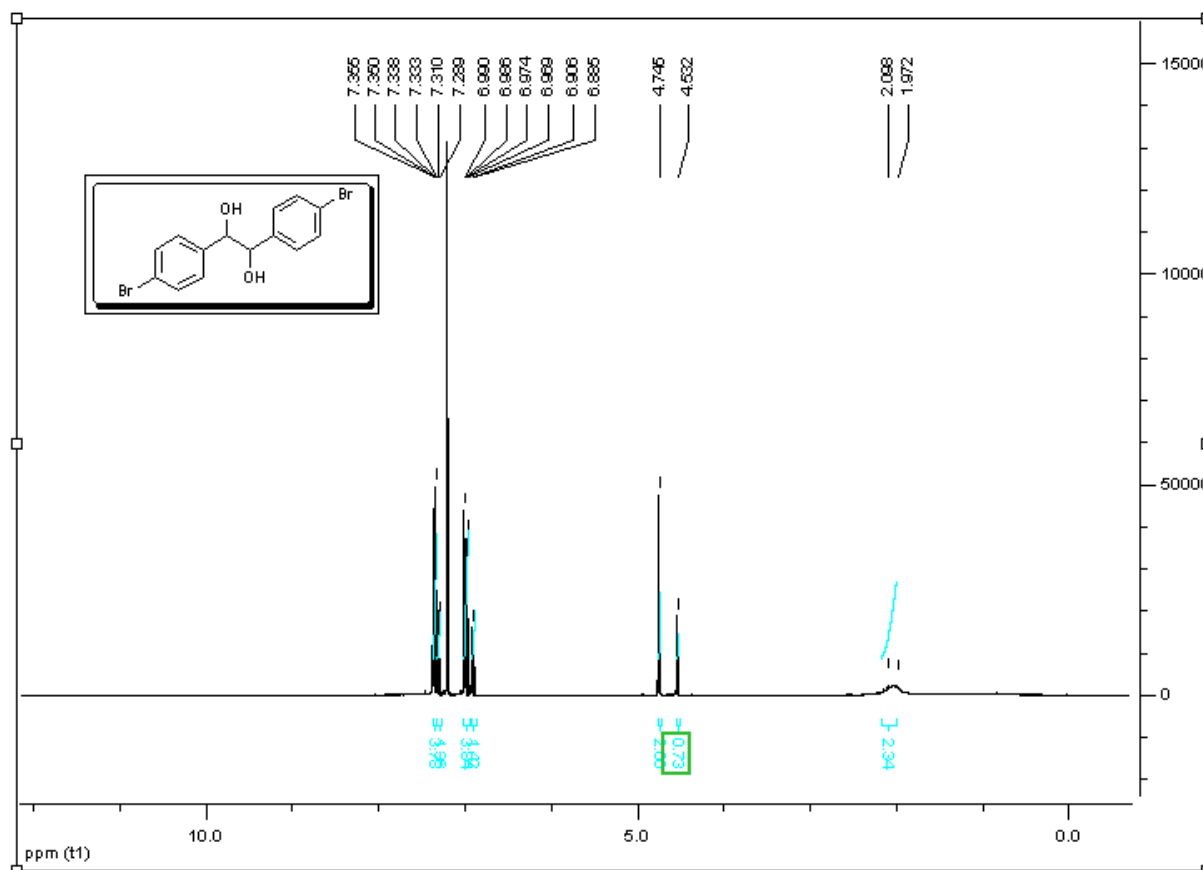
1,2-Bis(4-chlorophenyl)-1,2-ethanediol (*dl* and *meso*) (2o) :  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



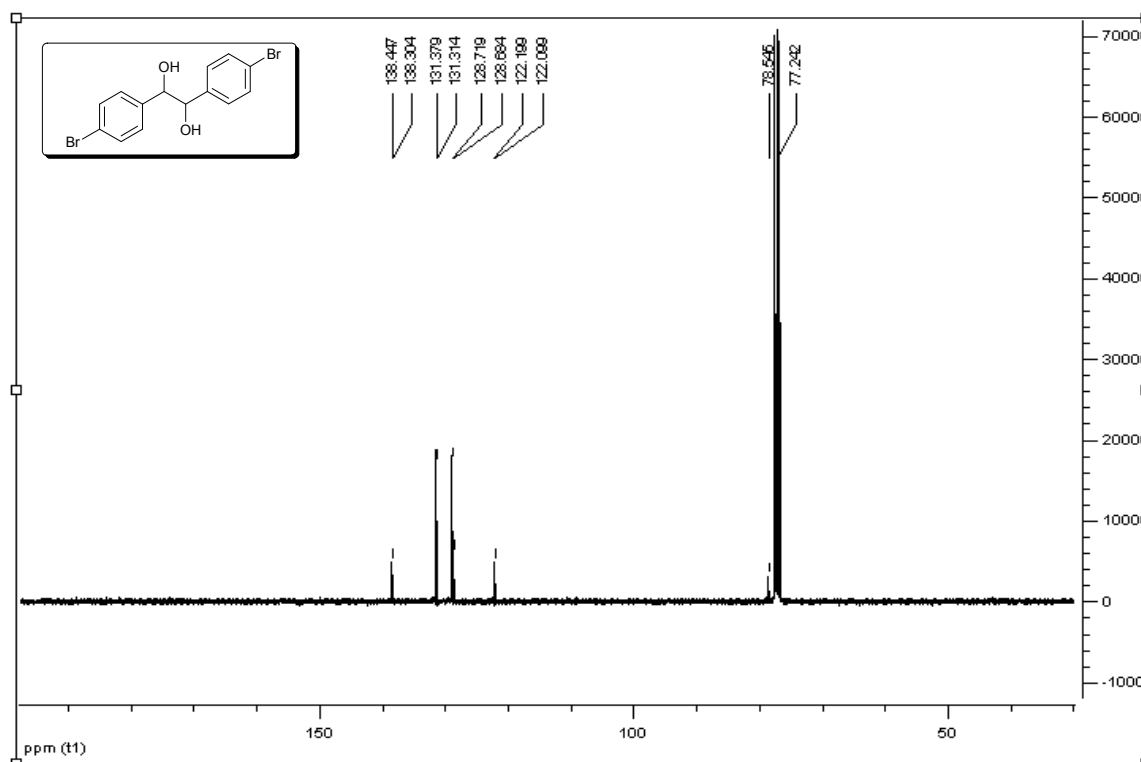
1,2-Bis(4-chlorophenyl)-1,2-ethanediol (*dl* and *meso*) (2o) :  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



1,2-Bis(4-bromophenyl)-1,2-ethanediol (*dl* and *meso*) (2p) :  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )

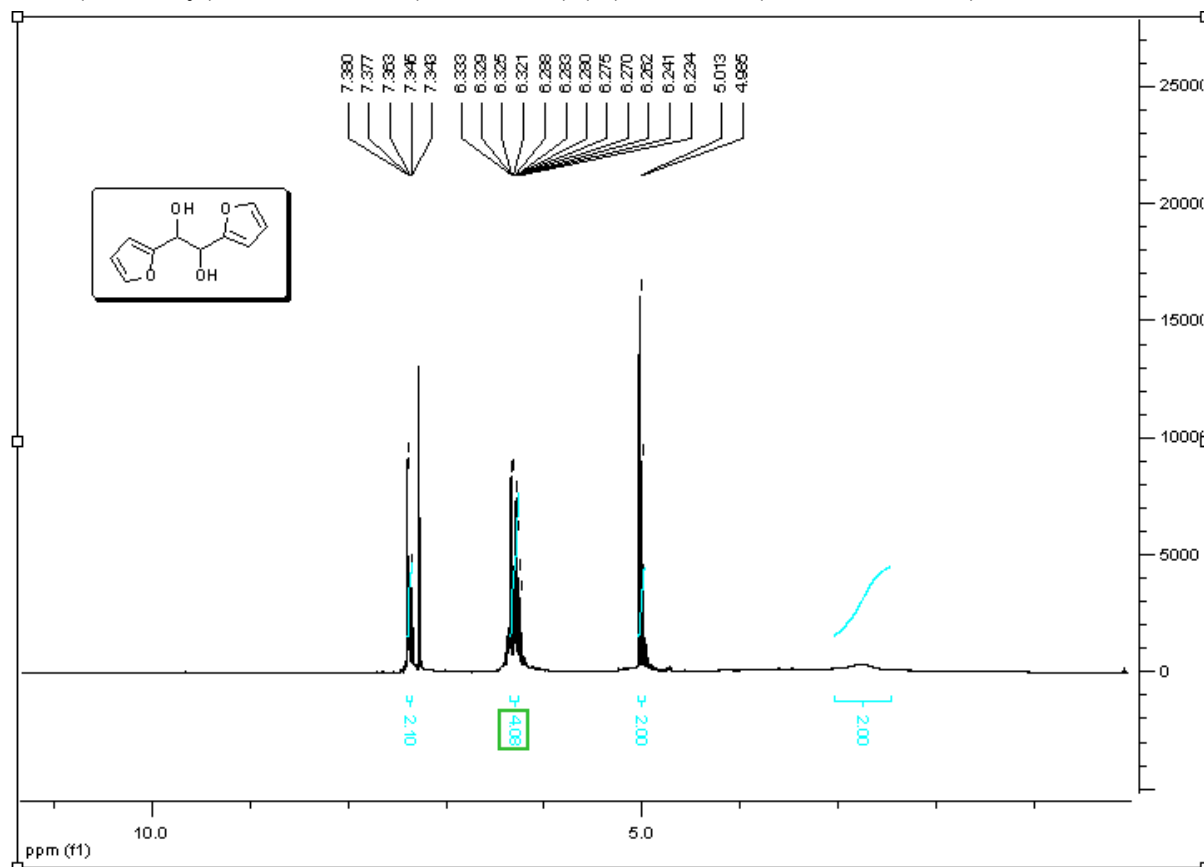


1,2-Bis(4-bromophenyl)-1,2-ethanediol (*dl* and *meso*) (2p) :  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

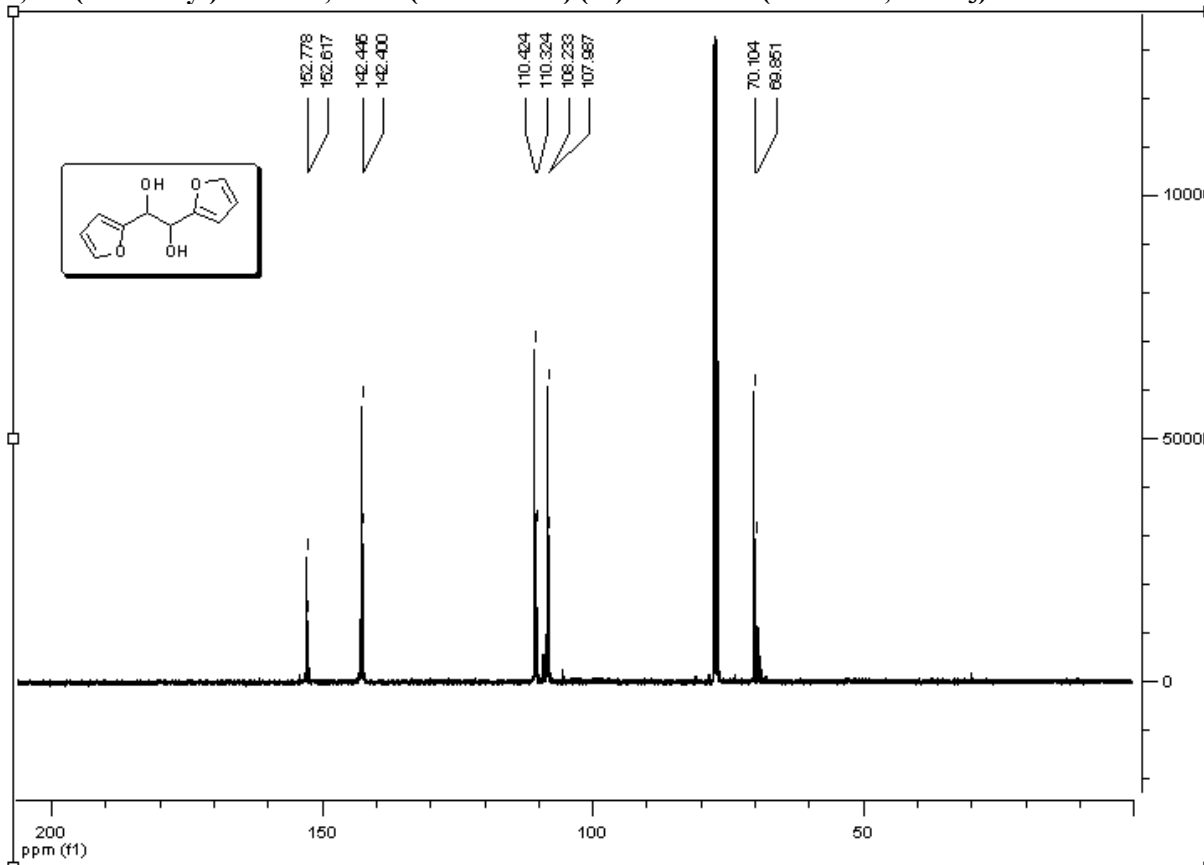




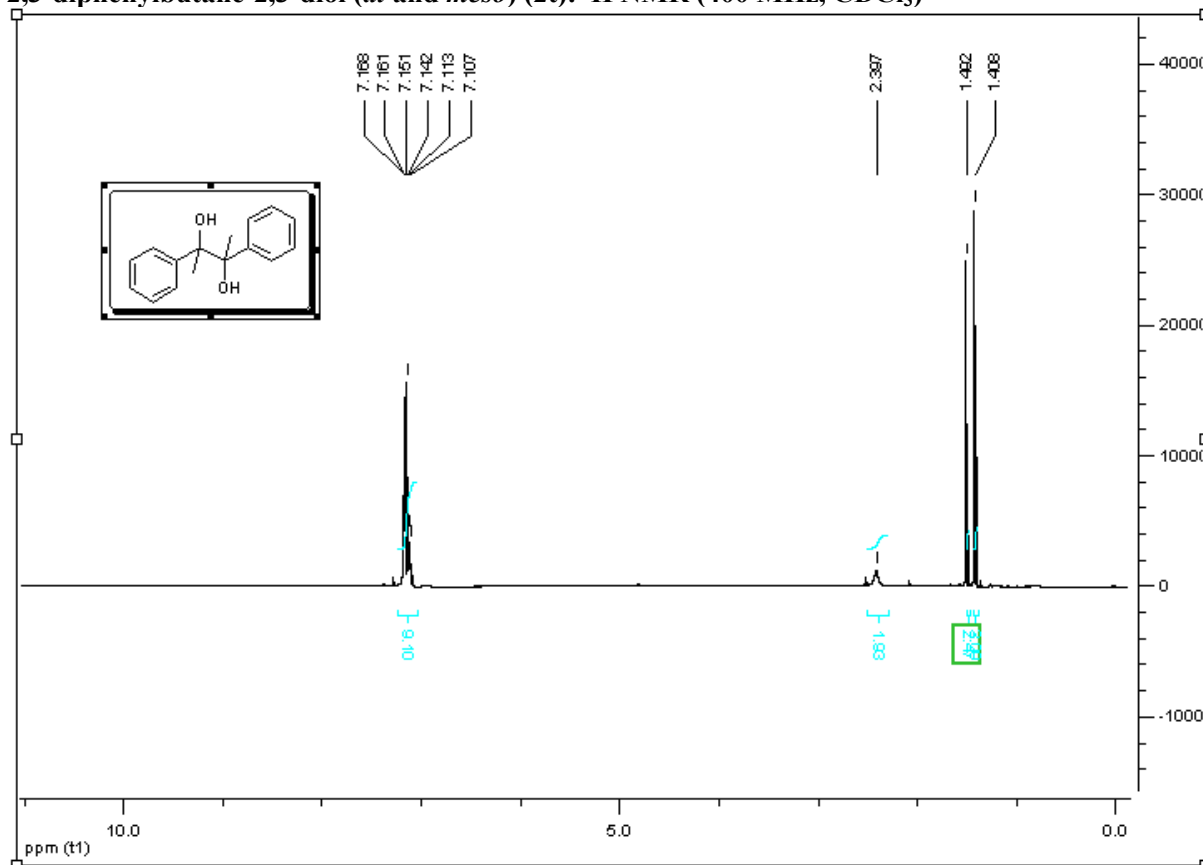
**1,2-di(furan-2-yl)ethane-1,2-diol (*dl* and *meso*) (2s): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



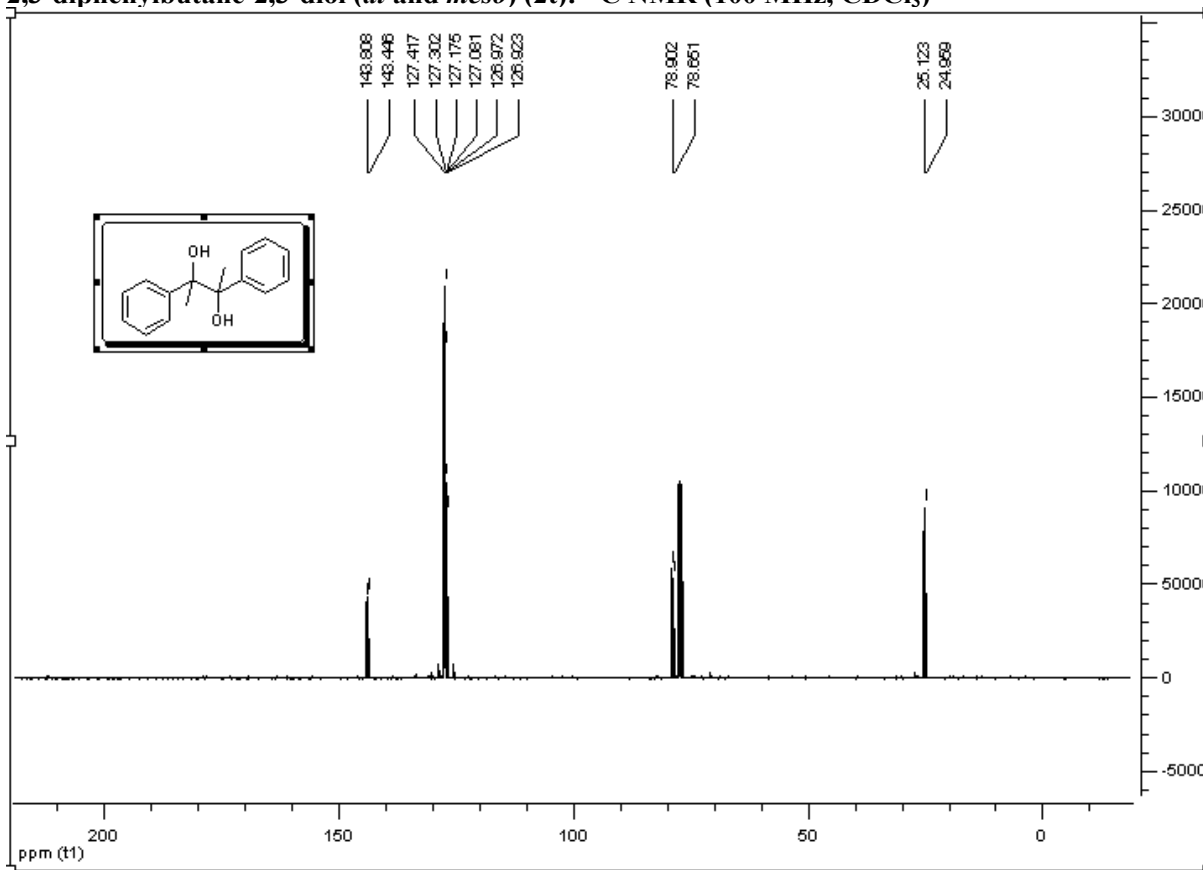
**1,2-di(furan-2-yl)ethane-1,2-diol (*dl* and *meso*) (2s): <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



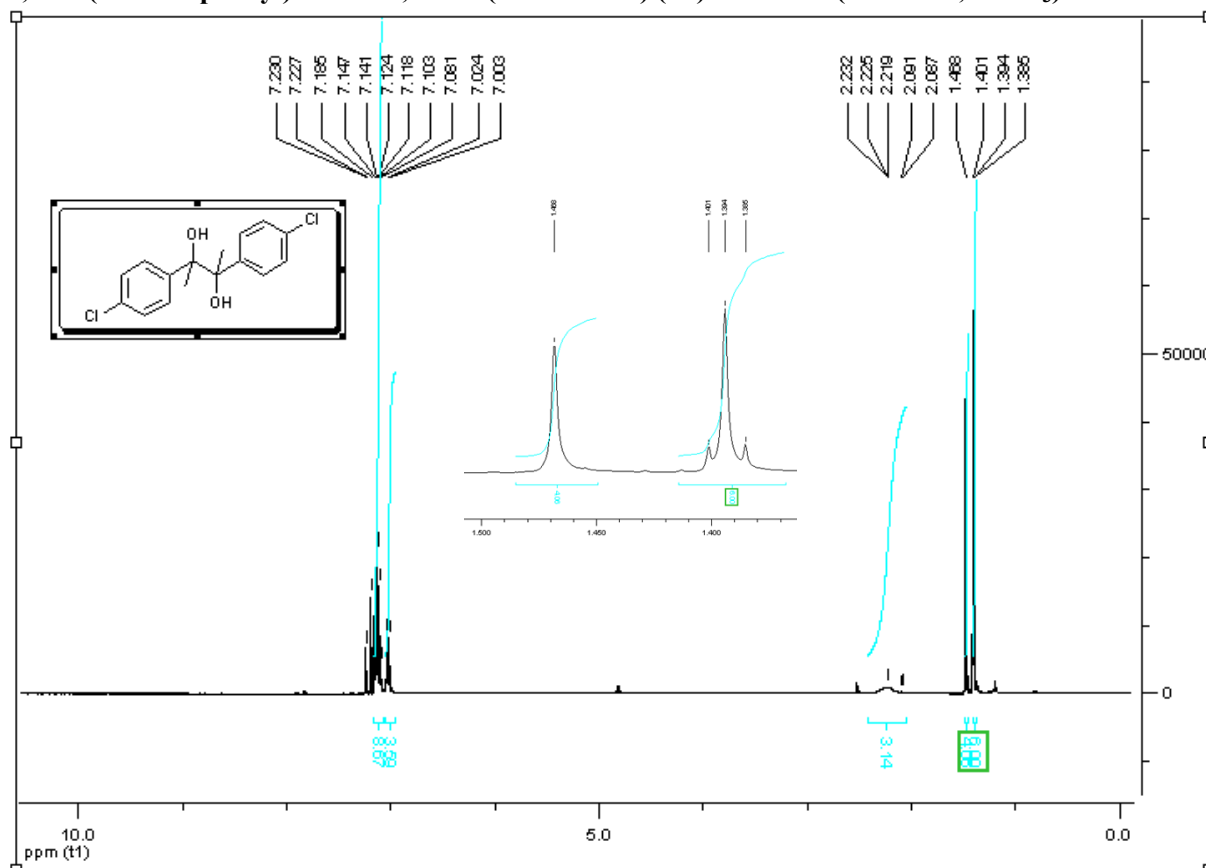
2,3-diphenylbutane-2,3-diol (*dl* and *meso*) (2t):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



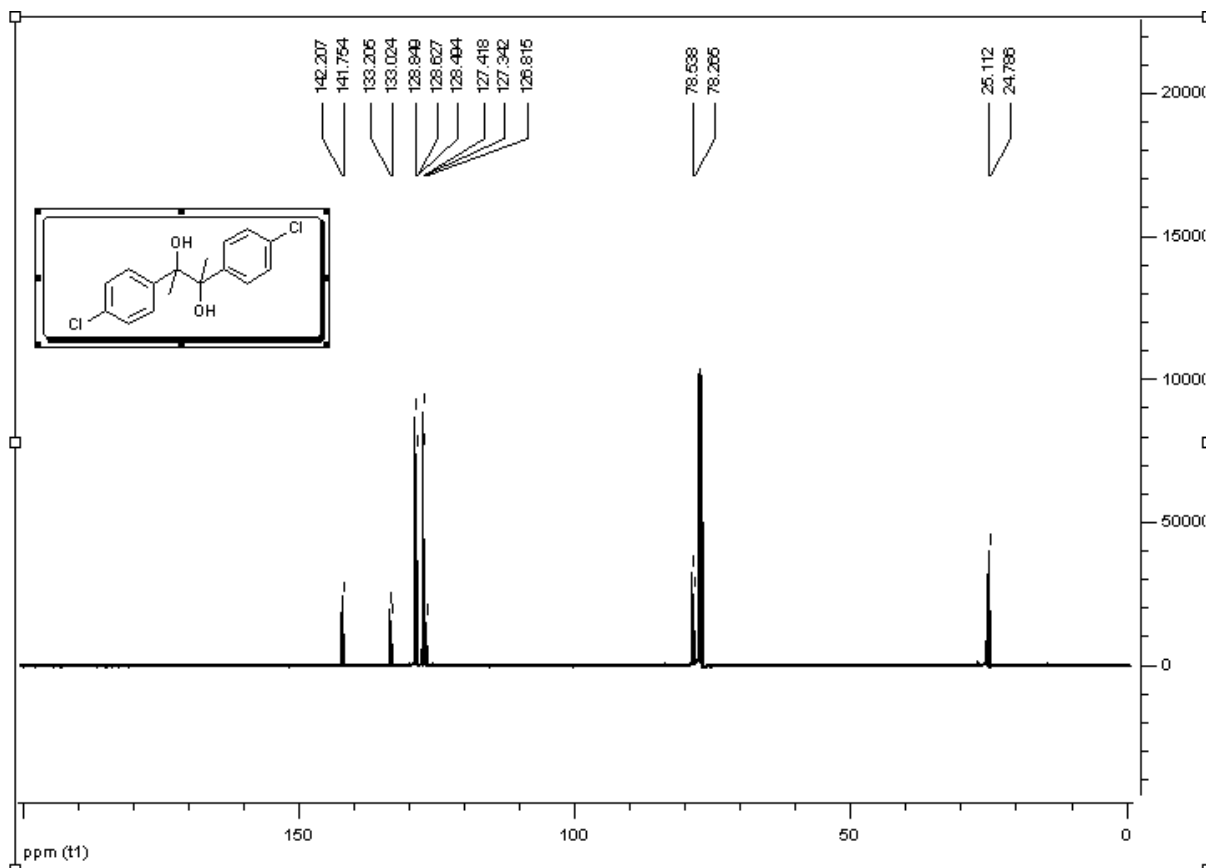
2,3-diphenylbutane-2,3-diol (*dl* and *meso*) (2t):  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



2,3-bis(4-chlorophenyl)butane-2,3-diol (*dl* and *meso*) (2u):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



2,3-bis(4-chlorophenyl)butane-2,3-diol (*dl* and *meso*) (2u):  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )





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<sup>i</sup> (a) M. Yasuda, K. Tsuruwa, T. Azuma, S. Arulananda Babu, A. Baba, *Tetrahedron* 2009, **65**(46), 9569; (b) Y. Handa, J. Inanaga, *Tet. Let.* 1987, **28**(46), 5717.

<sup>ii</sup> M. Shimizu, H. Goto and R. Hayakawa, *Org. Lett.*, 2002, **4**, 4097.

<sup>iii</sup> M.M.V. Ramana, B.K.D., Singh, J.A., Parihar, *Journal of Chemical Research* 2004, **11**, 760.