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Zirconium-catalyzed cycloalumination of alkenes in a one-pot synthesis of 3-alkyl-1-(3-hydroxypropyl)cyclopentanols

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All chemicals were of commercial grade and used directly without purification. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AM-300 (at 300.13 and 75.13 MHz, respectively, Bruker Biospin, Germany) or Bruker AV 600 (at 600 and 150 MHz, respectively, Bruker Biospin, Germany) in $CDCl_3$. The *J* values are given in Hz. HRMS spectra were recorded on a Bruker micrOTOF II mass spectrometer using ESI. Elemental analysis data were obtained on a PerkinElmer Series II CHNS/O 2400 Analyzer.

Synthesis of 3-alkyl-1-(3-hydroxypropyl)cyclopentanols 4a-d.

A round-bottomed flask in a dry argon atmosphere was charged successively with stirring at 0°C with Cp_2ZrCl_2 (0.073 g, 0.25 mmol), alkene **1a-d** (5 mmol), and $AlEt_3$ (1.5 mL, 10 mmol). The temperature was brought to ambient and the mixture was stirred for 6 hours. Then solvent (10 mL) was added, the reaction mixture was cooled to -5 to -10 ºC, 1.5 equivalent of γ-butyrolactone (15 mmol) was added dropwise, and the mixture was stirred at room temperature for an additional 8 hours. Then the reaction mixture was hydrolyzed with 5% HCl in H_2O, the reaction products were extracted with diethyl ether. The organic layer was separated, and the aqueous layer was extracted twice with the diethyl ether (2x10 ml). The solvent was evaporated, and the residue was chromatographed on silica gel (SiO_2) (petroleum ether /EtOAc, 10:1) gave **4a-d** as a colorless oil.

3-Hexyl-1-(3-hydroxypropyl)cyclopentan-1-ol (*Z*/*E*, 1:2) (**4a**)

^{1}H NMR (300 MHz; δ, $CDCl_3$): 0.87 (t, 3H, CH_3, *J* = 6.0 Hz); 1.15-1.52 (m, 14H, CH_2); 1.60-2.19 (m, 5H, CH, CH_2 (ring)), 3.61 (t, 2H, -C<u>H</u>$_2$-OH, *J* = 6.0 Hz), 3.80 (s, 2H, -CH_2-O<u>H</u>).

^{13}C NMR (75MHz; δ, $CDCl_3$): 14.1, 22.7, 27.9 (27.7), 28.5 (28.6), 29.6 (29.5), 30.8 (31.3), 31.9 (2C), 36.4 (36.9), 38.0, 39.2 (2C) (39.1, 40.1), 47.0 (46.6), 62.9 (62.8), 82.1 (81.6).

MS (ESI): 229.2086 [M+H]. Calculated for $C_{14}H_{28}O_2$: 228.2089

Anal. calcd. For $C_{14}H_{28}O_2$: C, 73.63; H, 12.35%. Found: C, 73.61; H, 12.33%.

3-Heptyl-1-(3-hydroxypropyl)cyclopentan-1-ol (*Z*/*E*, 1:2) (**4b**)

^{1}H NMR (600 MHz; δ, $CDCl_3$): 0.88 (t, 3H, CH_3, *J* = 6.0 Hz); 1.25-1.39 (m, 18H, CH_2); 1.67-2.15 (m, 5H, CH, CH_2 (ring)), 3.00 (s, 2H, -CH_2-OH), 3.67 (t, 2H, -CH_2-OH, *J* = 6.0 Hz).

^{13}C NMR (150MHz; δ, $CDCl_3$): 14.1, 22.6, 28.0 (27.7), 28.5 (28.6), 29.3, 29.8 (29.7), 30.7 (31.3), 31.9, 36.3 (36.9), 38.0 (39.0), 39.2 (40.1), 39.3 (39.2), 47.0 (46.7), 63.1 (63.1), 82.1 (81.8).

MS (ESI): 243.2221 [M+H]. Calculated for $C_{15}H_{30}O_2$: 242.2225

Anal. calcd. For $C_{15}H_{30}O_2$: C, 74.32; H, 12.47%. Found: C, 74.31; H, 12.45%.

1-(3-Hydroxypropyl)-3-octylcyclopentan-1-ol (*Z*/*E*, 1:2) (**4c**)

^{1}H NMR (300 MHz; δ, $CDCl_3$): 0.86 (t, 3H, CH_3, *J* = 6.0 Hz); 1.13-1.50 (m, 20H, CH_2); 1.62-2.14 (m, 5H, CH, CH_2 (ring)), 3.04 (s, 2H, -CH_2-OH), 3.64 (t, 2H, -CH_2-OH, *J* = 6.0 Hz).

^{13}C NMR (75MHz; δ, $CDCl_3$): 14.2, 22.7, 28.1 (27.8), 28.6 (28.7), 29.4, 29.7, 30.0 (29.9), 30.8 (31.4), 31.9, 36.4 (36.9), 38.1, 39.3 (39.1, 39.2), 39.4 (40.2), 47.1 (46.8), 63.2 (63.1), 82.2 (81.7).

MS (ESI): 257.2398 [M+H]. Calculated for $C_{16}H_{32}O_2$: 256.2402

Anal. calcd. For $C_{16}H_{32}O_2$: C, 74.94; H, 12.58%. Found: C, 74.92; H, 12.57%.

3-Benzyl-1-(3-hydroxypropyl)cyclopentan-1-ol (*Z*/*E*, 1:1) (**4d**)

^{1}H NMR (300 MHz; δ, $CDCl_3$): 1.22-2.20 (m, 11H, CH_2), 2.55-2.59 (m, 2H, -CH_2-Ph), 2.86 (s, 2H, -CH_2-OH), 3.66 (t, 2H, -CH_2-OH, *J* = 6.0 Hz), 7.17-7-32 (m, 5H, Ph).

^{13}C NMR (75MHz; δ, $CDCl_3$): 27.8 (28.0), 31.2 (30.6), 39.3, 39.9 (39.3), 40.2 (40.8), 42.8 (42.2), 46.3 (46.8), 63.2 (63.1), 82.0 (81.7), 125.8, 128.7 (2C), 128.8 (2C), 141.9. (141.7).

MS (ESI): 235.1616 [M+H]. Calculated for $C_{15}H_{22}O_2$: 234.1619

Anal. calcd. For $C_{15}H_{22}O_2$: C, 76.88; H, 9.46%. Found: C, 76.85; H, 9.44%.

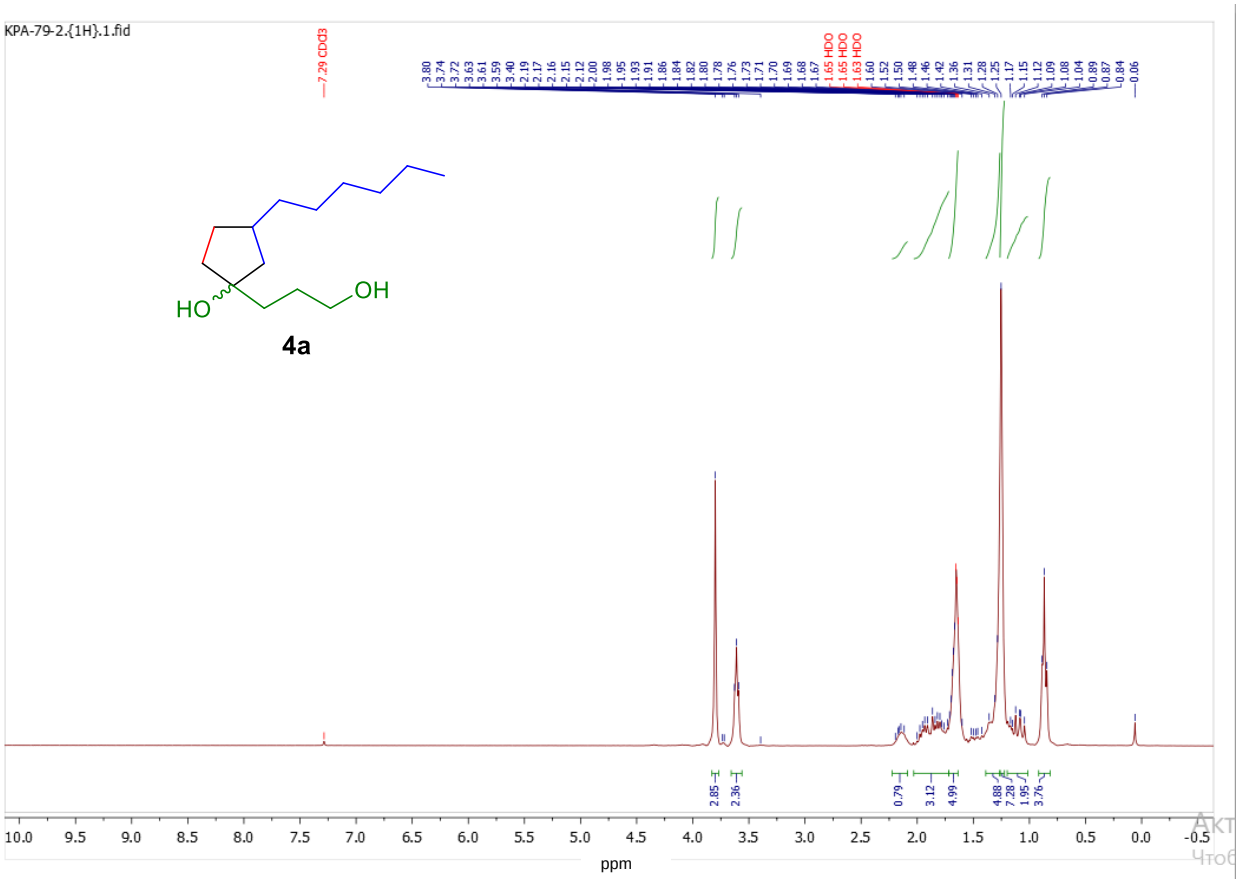

Figure S1 ^{1}H NMR spectrum of 3-hexyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4a**)

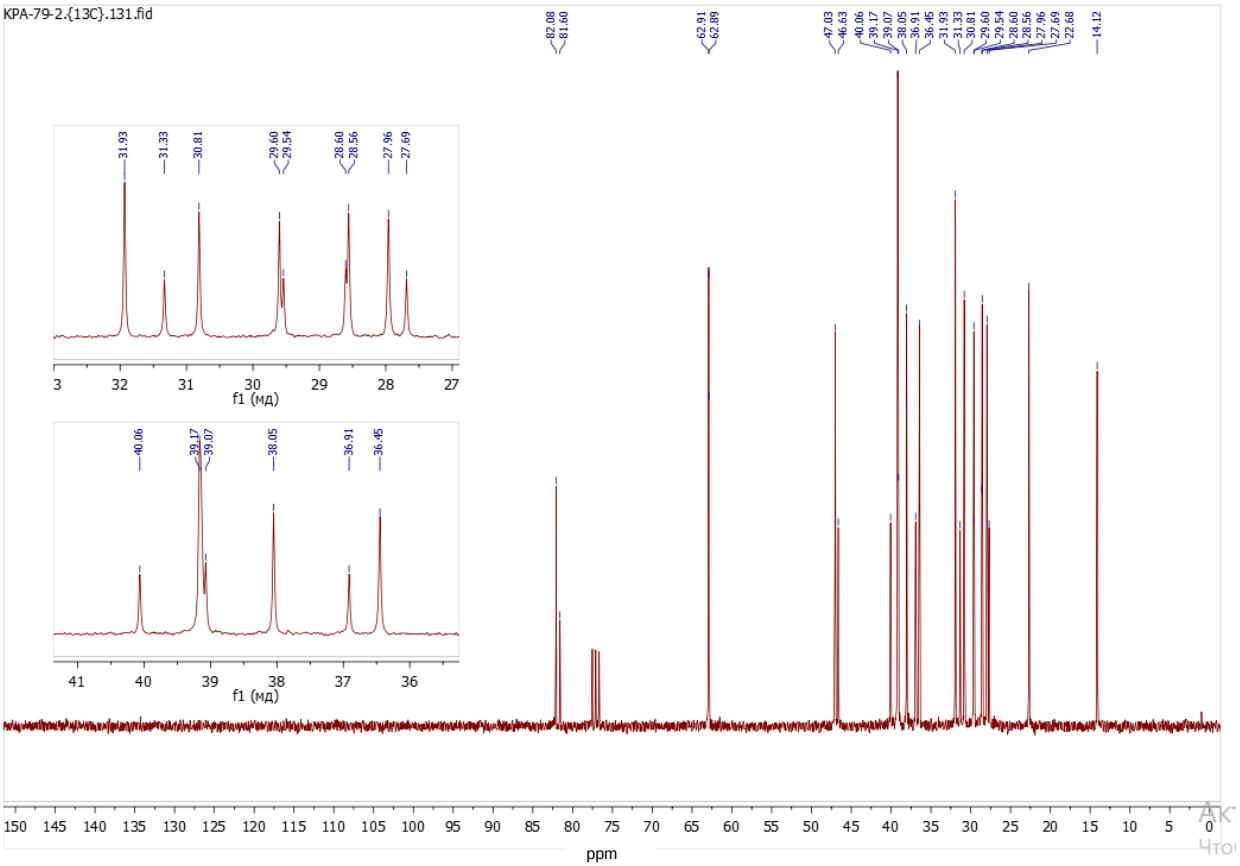

Figure S2 ^{13}C NMR spectrum of 3-hexyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4a**)

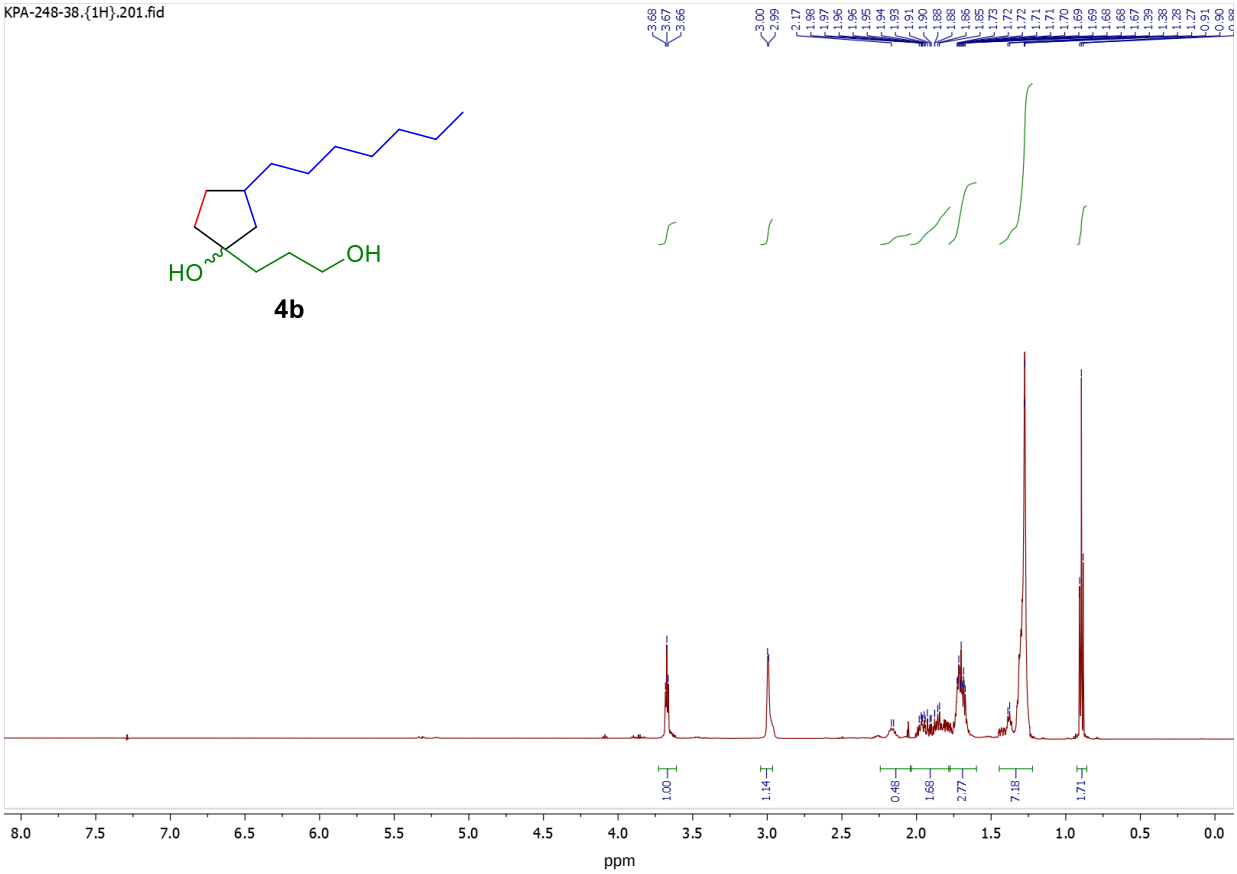

Figure S3 ^{1}H NMR spectrum of 3-heptyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4b**)

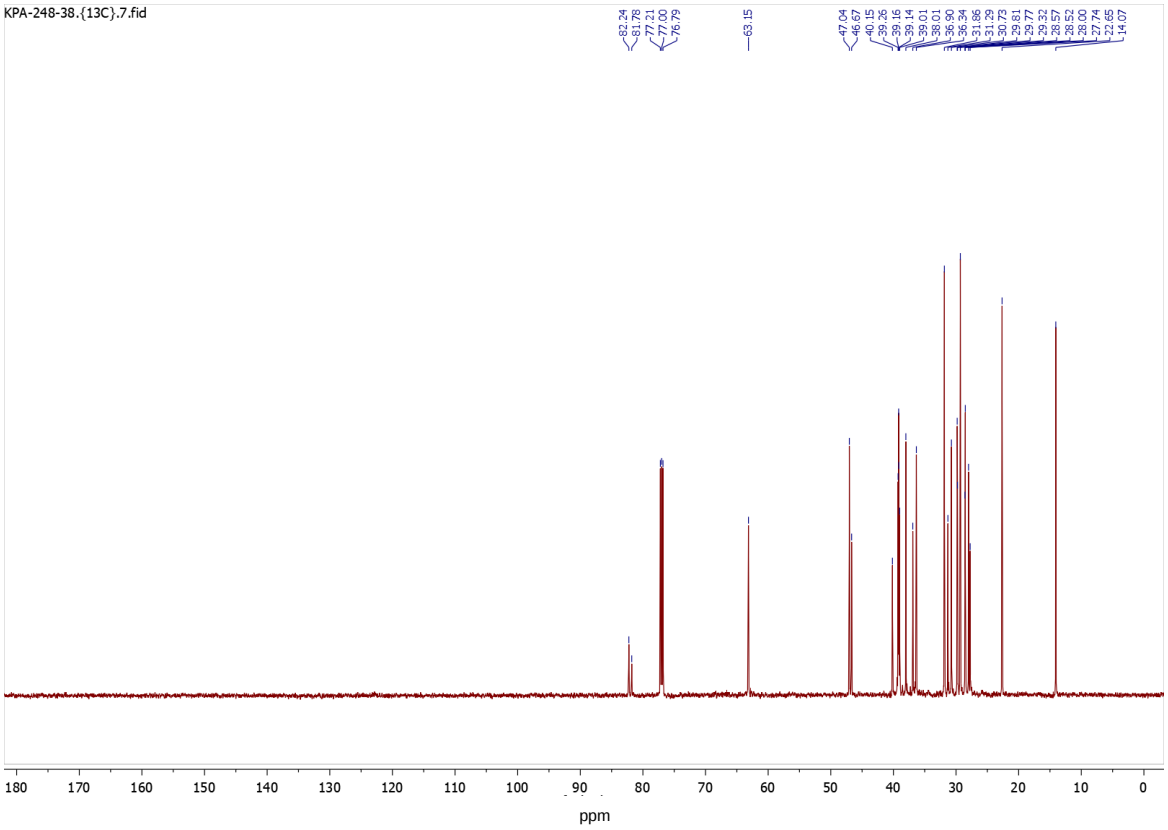

Figure S4 ^{13}C NMR spectrum of 3-heptyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4b**)

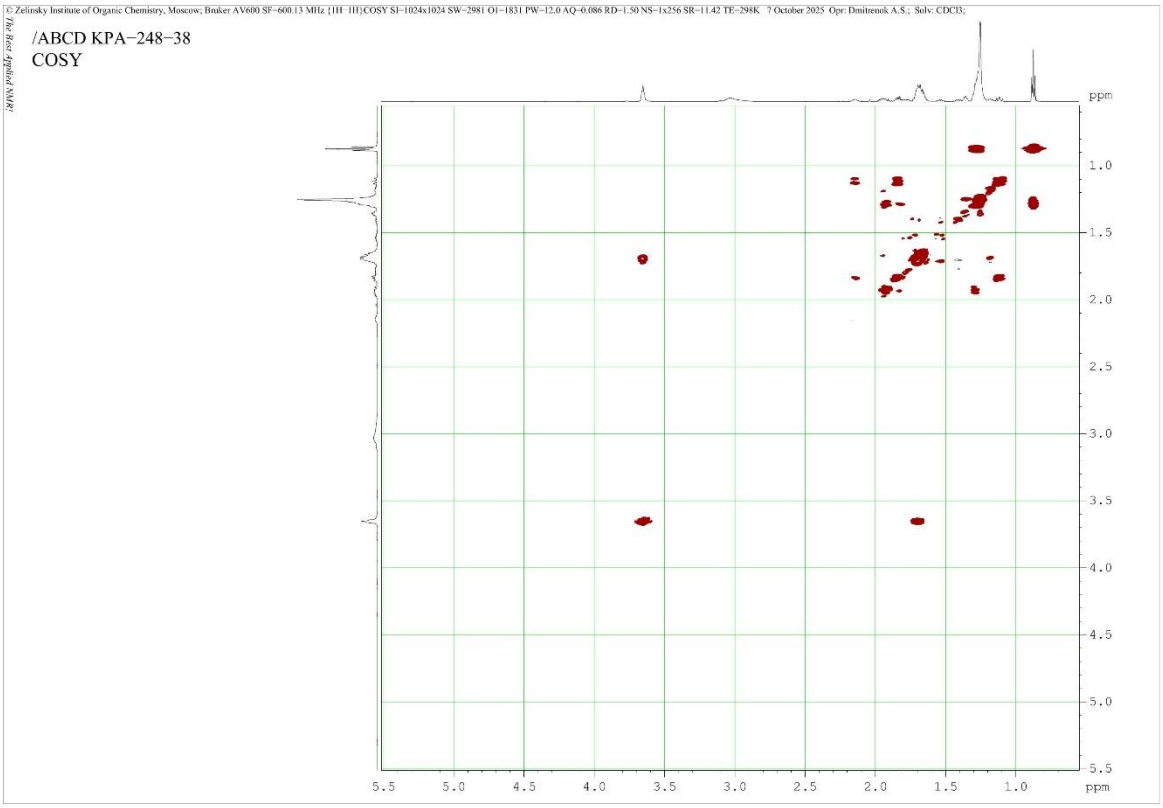

Figure S5 COSY HH 2D NMR spectrum of 3-heptyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4b**)

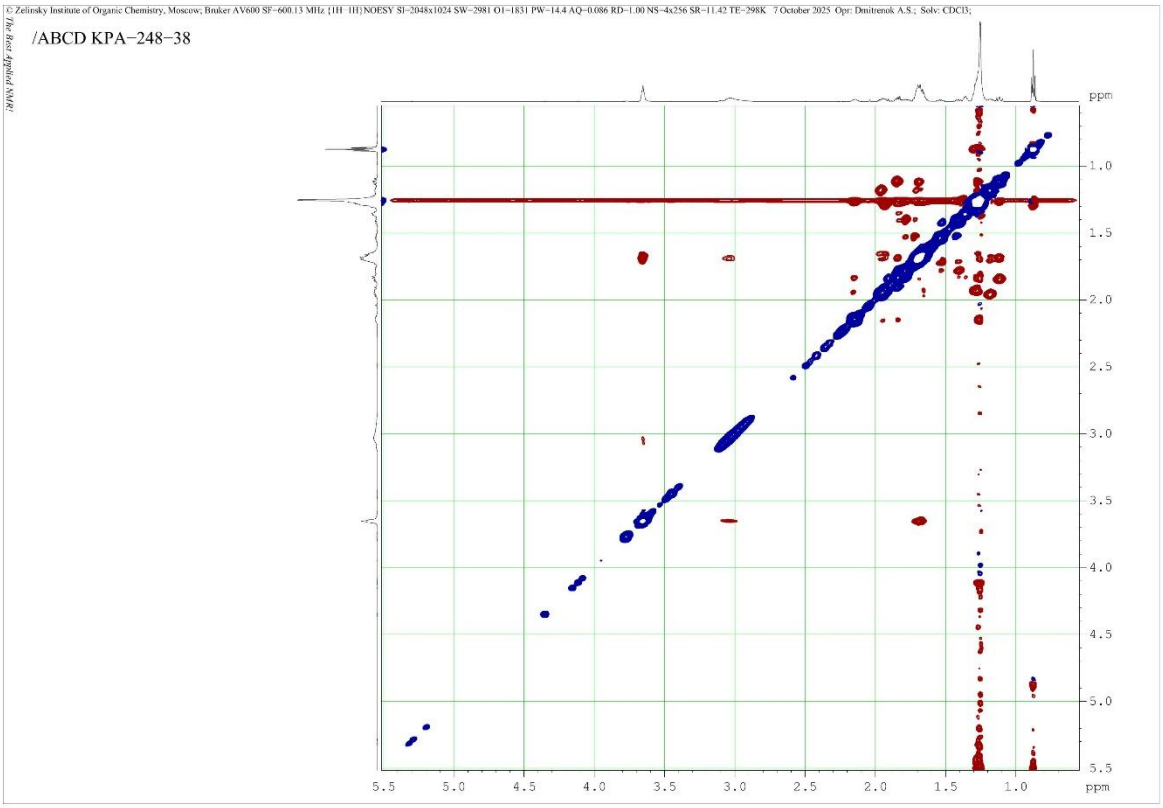

Figure S6 NOESY 2D NMR spectrum of 3-heptyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4b**)

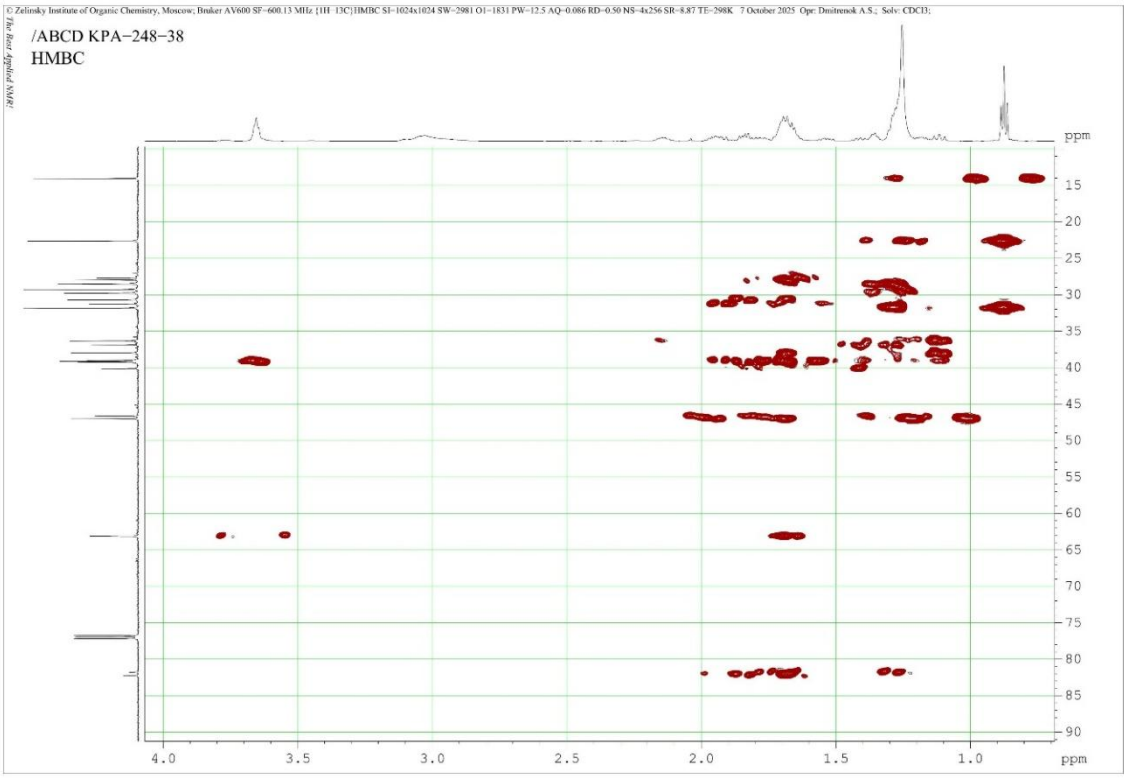

Figure S7 HMBC 2D NMR spectrum of 3-heptyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4b**)

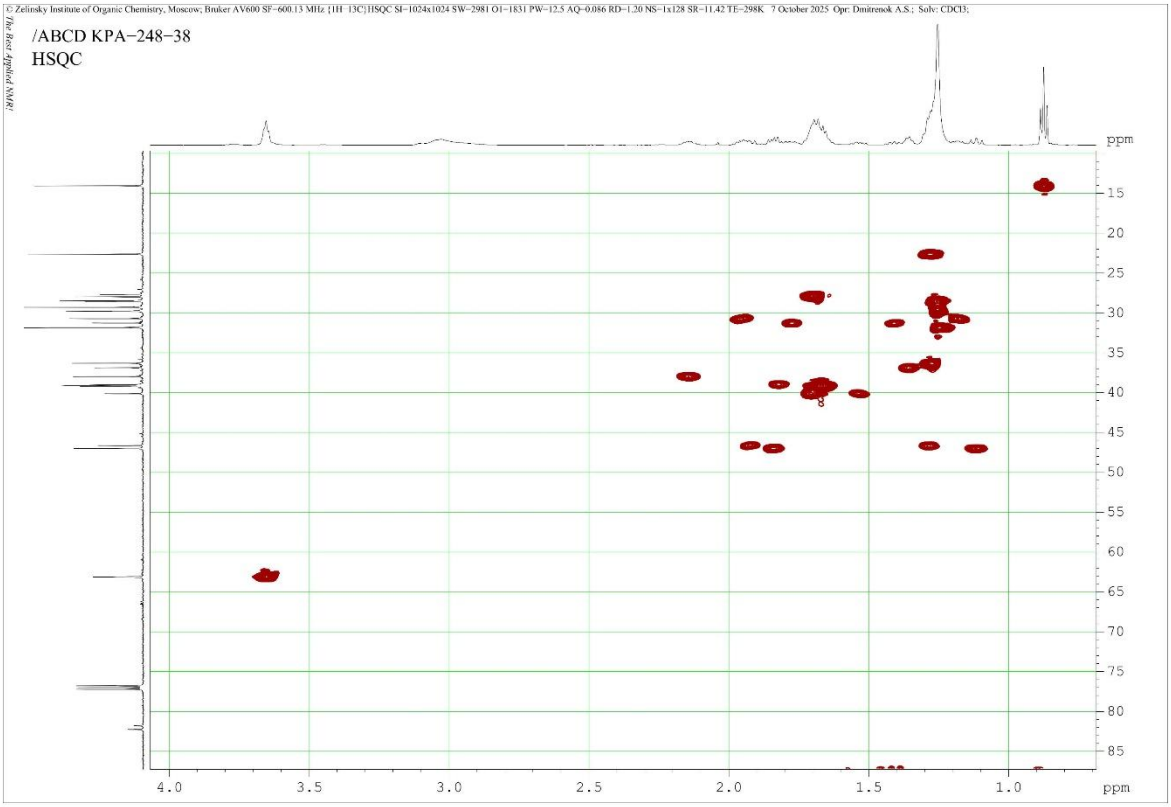

Figure S8 HSQC 2D NMR spectrum of 3-heptyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4b**)

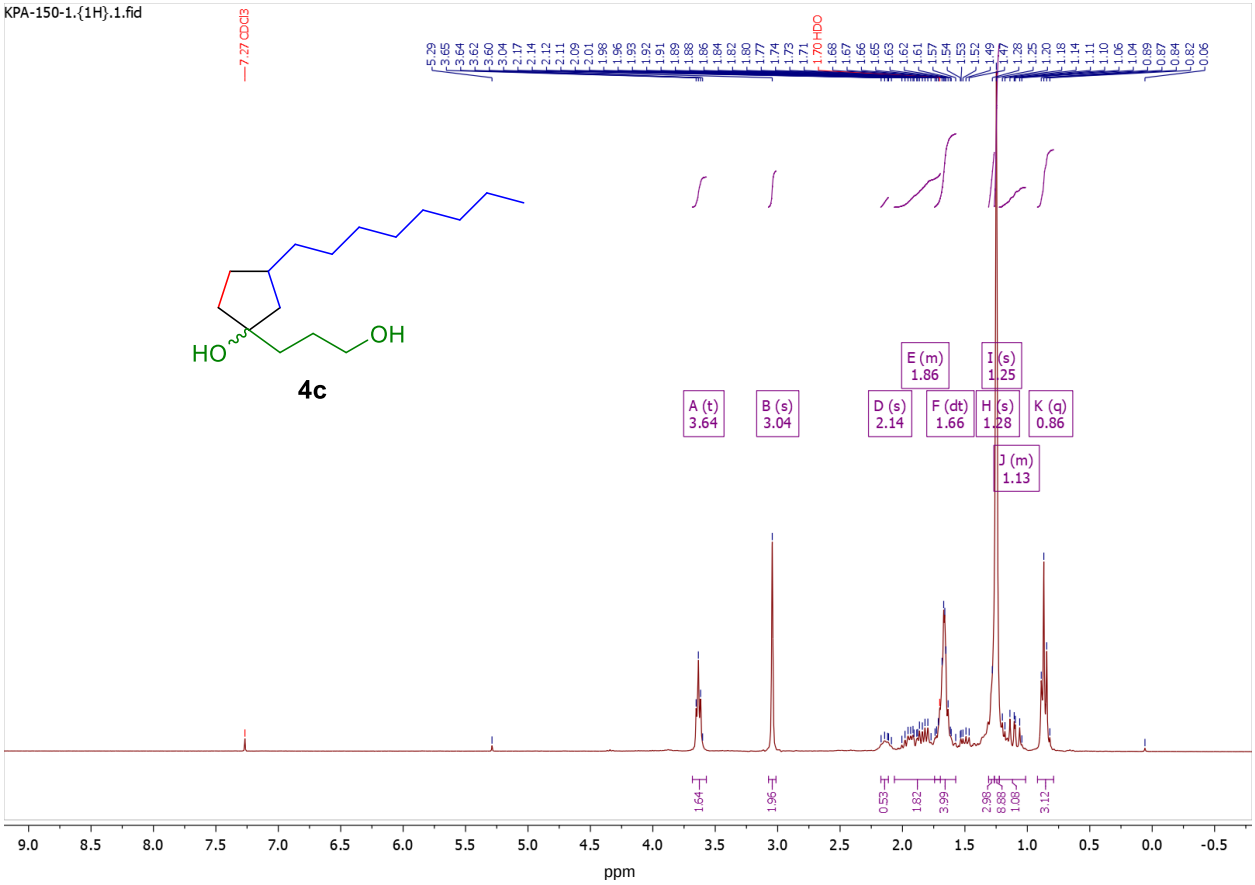

Figure S9 ^{1}H NMR spectrum of 1-(3-hydroxypropyl)-3-octyl-cyclopentan-1-ol (**4c**)

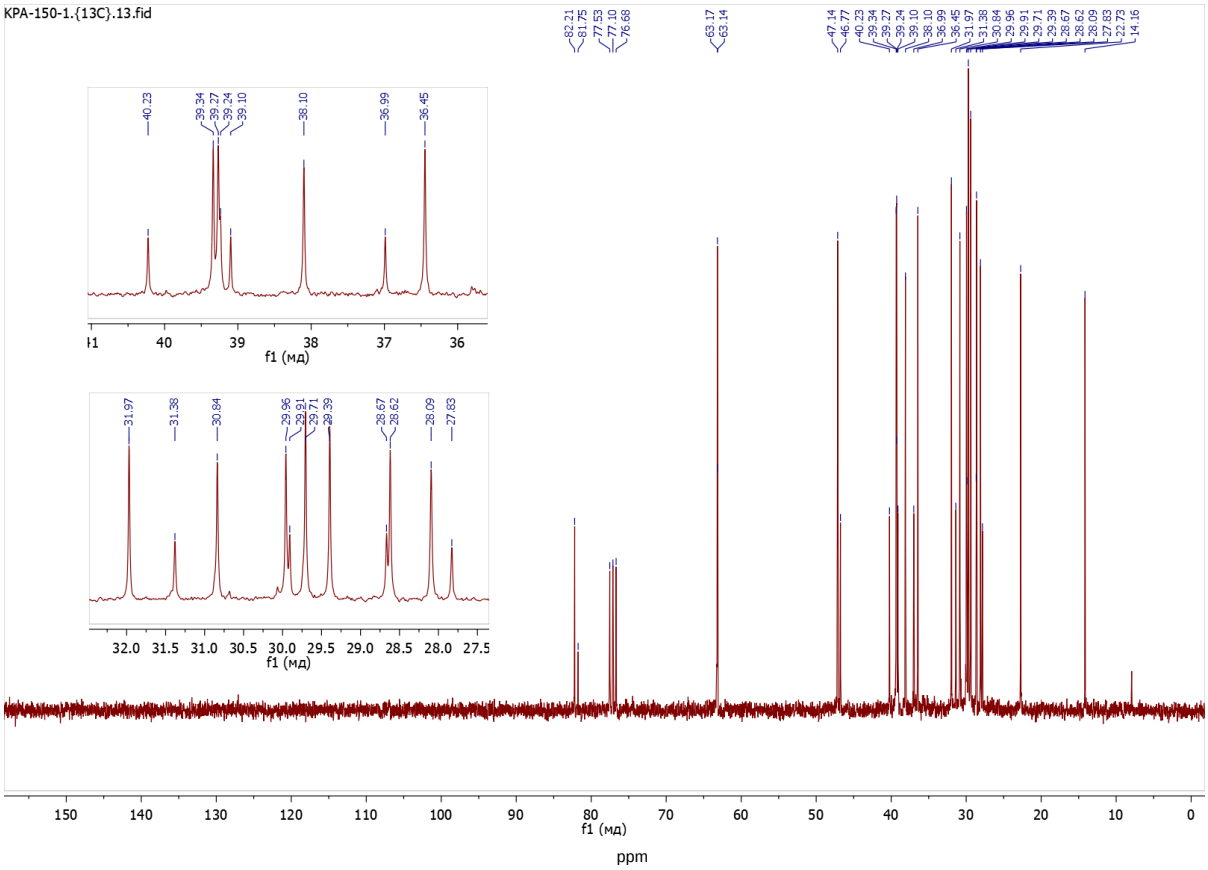

Figure S10 ^{13}C NMR spectrum of 1-(3-hydroxypropyl)-3-octyl-cyclopentan-1-ol (**4c**)

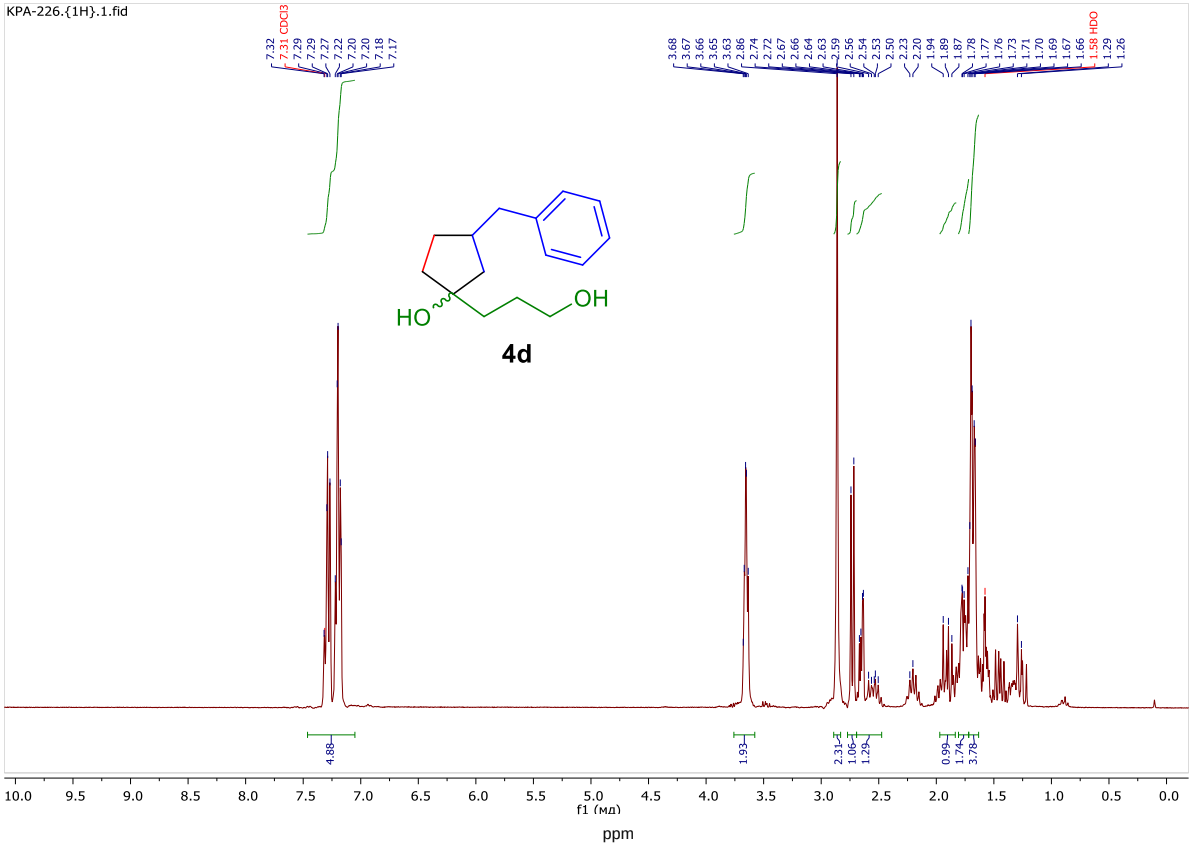

Figure S11 ^{1}H NMR spectrum of 3-benzyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4d**)

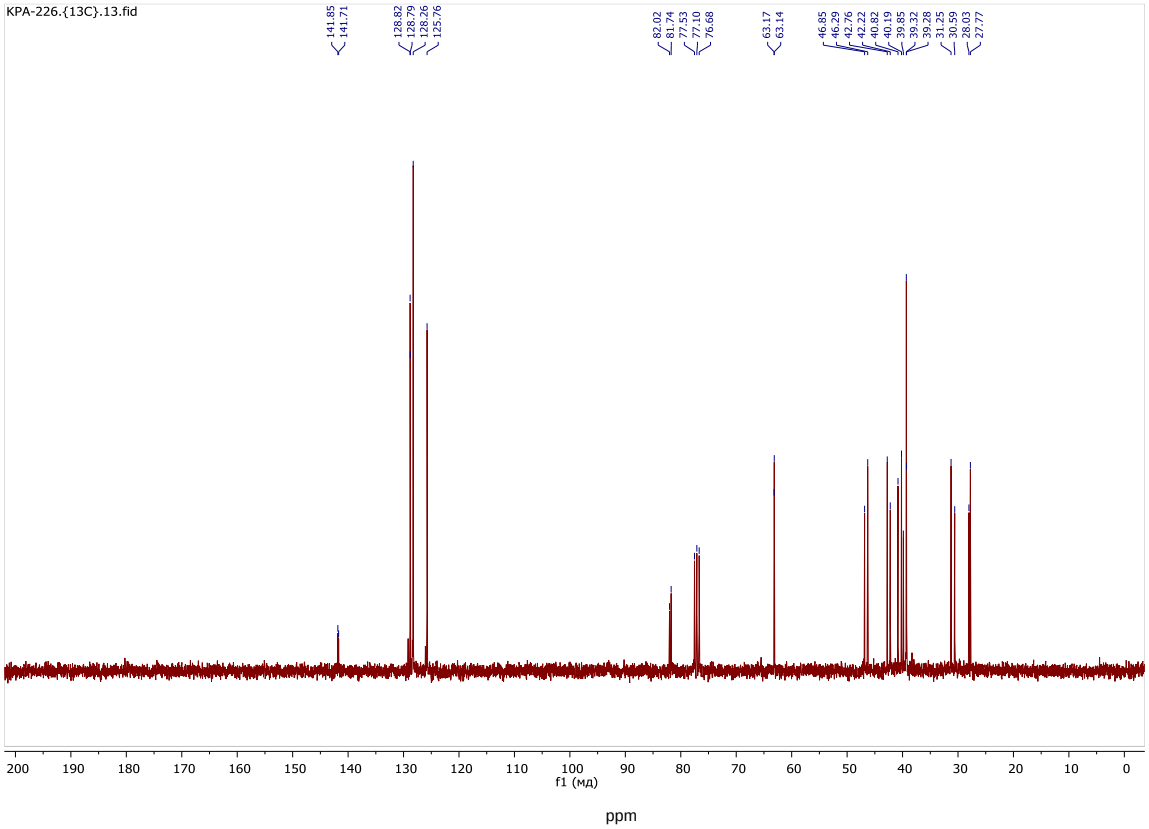

Figure S12 ^{13}C NMR spectrum of 3-benzyl-1-(3-hydroxypropyl)cyclopentan-1-ol (**4d**)