Supporting Information

A mild method for eliminating alkyl ethers to alkenes

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Experimental Section

1. General Remarks

NMR spectra were recorded on a *Bruker* AM 400 or a *Bruker* Avance 300 spectrometer as solutions at room temperature. Chemical shifts δ are expressed in parts per million (ppm) downfield from tetramethylsilane (TMS). References for ^{1}H NMR and ^{13}C NMR were the residual solvent peaks of chloroform (^{1}H: δ = 7.26 ppm), DMSO (^{1}H: δ = 2.50 ppm), D_1-chloroform (^{13}C: δ = 77.0 ppm) and D_6-DMSO (^{13}C: δ = 39.43 ppm). All coupling constants (*J*) are absolute values and are expressed in Hertz (Hz). The description of signals includes: s = singlet, d = doublet, t = triplet, quin = quintet, m = multiplet, m_c = centered multiplet, dd = doublet of doublets and ddd = double doublet of doublets and so forth. The spectra were analyzed according to first order. The assignments of the signal structure in ^{1}H NMR were made by the multiplicity and for ^{13}C NMR by DEPT 90- and DEPT 135-spectra (DEPT = distortionless enhancement by polarization transfer) and are described as follows: + = primary or tertiary C-atom (positive DEPT-signal), – = secondary C-atom (negative signal) and $C_{quart.}$ = quaternary C-atom (no signal).

IR spectra were recorded on a FT-IR *Bruker* IFS 88 spectrometer. Liquids and oils were measured between KBr plates and solids were measured as pure substances by ATR technique (ATR = attenuated total reflection). The position of the absorption band is given in wave numbers $\tilde{\nu}$ in cm^{-1}. The intensities of the bands were characterized as follows: vs = very strong (0-20% T), s = strong (21-40% T), m = medium (41-60% T), w = weak (61-80% T), vw = very weak (81-100% T).

Mass spectra were measured by EI-MS (electron impact mass spectrometry) and were recorded on a *Finnigan MAT 95*. The peaks are given as mass-to-charge-ratio (*m*/*z*). The molecule peak is given as $[M]^+$ and characteristic fragment peaks are given as $[M\text{-fragment}]^+$ or $[fragment]^+$. The signal intensities are given in percent, relatively to the intensity of the base signal (100%). For the high resolution mass, the following abbreviations were used: calcd. = calculated data, found = measured data.

The elemental analysis measurements were performed on an *Elementar vario MICRO* device using a *Sartorius* M2P precision balance. The values for carbon (C) and hydrogen (H) are expressed in mass percentages. The following abbreviations were used: calcd. = calculated data, found = measured data.

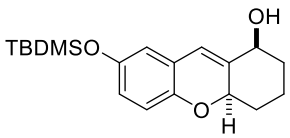
Melting points of solid substances were recorded on a Mel-Temp II (*Laboratory Devices Inc.*) and are not corrected.

Analytical thin layer chromatography (TLC) was carried out on Merck silica gel coated aluminum plates (silica gel 60, F_{254}), detected under UV-light at 254 nm or stained with "Seebach staining solution" (mixture of molybdato phosphoric acid, cerium(IV)-sulfate tetrahydrate, sulfuric acid and water) or basic potassium permanganate solution. Solvent mixtures are understood as volume/volume. Solvents, reagents and chemicals were purchased from *Sigma-Aldrich*, *ABCR* and *Acros Organics*. All solvents, reagents and chemicals were used as purchased unless stated otherwise.

Air- or moisture-sensitive reactions were carried out under argon atmosphere in oven-dried and previously evacuated glass ware. Liquids were transferred with plastic syringes and steel cannula. Reaction control was performed by thin layer chromatography. If the product was volatile, solvents were removed at room temperature at the rotary evaporator, otherwise at 40 °C. If not stated otherwise, crude products were purified by flash chromatography by the procedure of Still.[1] Silica gel 60 (0.040 × 0.063 mm, Geduran®, Merck) was used as stationary phase and as mobile phase, solvents of p.a. quality were used.

2. Experimental Procedures and Analytical Data[2]

(1,4a-*trans)*-7-((*tert*-Butyldimethylsilyl)oxy)-2,3,4,4a-tetrahydro-1*H*-xanthen-1-ol[3]

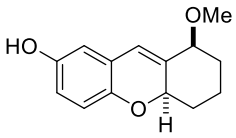


To a solution of (1,4a-*trans*)-2,3,4,4a-tetrahydro-1*H*-xanthene-1,7-diol[4] (750 mg, 3.44 mmol, 1.00 equiv.) in dichloromethane (30 mL), NEt_3 (0.720 mL, 522 mg, 5.16 mmol, 1.50 equiv.), DMAP (84.0 mg, 0.688 mmol, 0.200 equiv.) and TBDMSCl (570 mg, 3.78 mmol, 1.10 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 19 h. After addition of saturated aqueous $NaHCO_3$-solution, the mixture was extracted with dichloromethane (3 × 25 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 3:1) yielding the product as a yellow oil (759 mg, 2.28 mmol, 66%).

R_f (cyclohexane/ethyl acetate = 3:1) = 0.32. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.57–6.49 (m, 2 H, $H_{arom.}$), 6.43 (d, ^4J = 2.6 Hz, 1 H, 9-H), 6.29 (s, 1 H, $H_{arom.}$), 4.85 (dd, ^3J = 11.2 Hz, ^3J = 5.3 Hz, 1 H, 4a-H), 4.12–4.00 (m, 1 H, 1-H), 2.23–2.05 (m, 2 H, CH_2), 1.97–1.80 (m, 2 H, CH_2), 1.34–1.14 (m, 2 H, CH_2), 0.81 (s, 9 H, $C(CH_3)_3$), 0.00 (s, 6 H, 2 × CH_3) ppm . ^{13}C NMR (100 MHz, $CDCl_3$): δ = 149.3 ($C_{quart.}$), 146.9 ($C_{quart.}$), 140.2 ($C_{quart.}$), 121.4 ($C_{quart.}$) 119.5 (+, C-9), 117.7 (+, $C_{arom.}$), 115.0 (+, $C_{arom.}$), 113.8 (+, $C_{arom.}$), 75.9 (+, C-4a), 70.5 (+, C-1), 36.1 (–, CH_2), 34.1 (–, CH_2), 25.7 (+, $C(CH_3)_3$), 19.8 (–, CH_2), 18.1 ($C_{quart.}$), – 4.5 (+, 2 × CH_3) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3486 (m), 2929 (m), 1491 (m). MS (EI, 70 eV), *m*/*z* (%): 332 (100) $[M]^+$, 287 (5), 275 (7), 261 (5). HR-EIMS ($C_{19}H_{28}SiO_3$): calcd. 332.1808; found 332.1811. Elementary analysis ($C_{19}H_{28}SiO_3$): calcd. C 68.63, H 8.49; found C 68.57, H 8.34.

(1,4a-*trans*)-1-Methoxy-2,3,4,4a-tetrahydro-1*H*-xanthen-7-ol (1)[3]

To a solution of (1,4a-*trans)*-7-((*tert*-butyldimethylsilyl)oxy)-2,3,4,4a-tetrahydro-1*H*-xanthen-1-ol



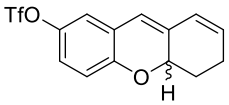
(750 mg, 2.26 mmol, 1.00 equiv.) in absolute THF (60 mL), NaH (60% in mineral oil, 117 mg, 2.94 mmol. 1.30 equiv.) and MeI (184 µL, 417 mg, 2.94 mmol. 1.30 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 3 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 25 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column

chromatography (cyclohexane/ethyl acetate = 10:1) yielding the product as a yellow oil (549 mg, 1.58 mmol, 70%). The instable product was converted as follows:

To a solution of (1,4a-*trans*)-7-((*tert*-butyldimethylsilyl)oxy)-2,3,4,4a-tetrahydro-1*H*-xanthen-1-ol (544 mg, 1.56 mmol, 1.00 equiv.) in absolute tetrahydrofuran (30 mL) under argon TBAF (1 M in THF, 3.14 mL, 820 mg, 3.14 mmol, 2.00 equiv.) was added and the reaction mixture was stirred at room temperature for 14 h. After addition of water, the aqueous phase was extracted with diethyl ether (3 × 25 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 3:1) yielding the product as a light yellow solid (320 mg, 1.38 mmol, 88%).

R_f (cyclohexane/ethyl acetate = 3:1) = 0.27. m.p.: 161–163 °C. ^{1}H NMR (400 MHz, D_6-DMSO): δ = 8.81 (s, 1 H, OH), 6.49–6.36 (m, 3 H, $H_{arom.}$), 6.14 (s, 1 H, 9-H), 4.79 (dd, ^3J = 11.3 Hz, ^3J = 5.3 Hz, 1 H, 4a-H), 3.62 (dd, ^3J = 11.3 Hz, ^3J = 4.9 Hz, 1 H, 1-H), 3.37 (s, 3 H, OCH_3), 2.19–2.07 (m, 1 H, CH_2), 2.03–1.92 (m, 1 H, CH_2), 1.82–1.68 (m, 1 H, CH_2), 1.64–1.48 (m, 1 H, CH_2), 1.44–1.27 (m, 1 H, CH_2), 1.19–1.03 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, D_6-DMSO): δ = 151.1 ($C_{quart.}$), 144.4 ($C_{quart.}$), 139.0 ($C_{quart.}$), 121.1 ($C_{quart.}$), 114.7 (+, C-9), 114.3 (+, $C_{arom.}$), 113.0 (+, $C_{arom.}$), 112.7 (+, $C_{arom.}$), 78.5 (+, C-4a), 75.2 (+, C-1), 56.6 (+, OCH_3), 34.2 (–, CH_2), 32.3 (–, CH_2), 19.2 (–, CH_2), ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3265 (m), 1492 (m), 1221 (m). MS (EI, 70 eV), *m*/*z* (%): 232 (100) $[M]^+$, 217 (29), 173 (68). HR-EIMS ($C_{14}H_{16}O_3$): calcd. 232.1099; found 232.1102. Elementary analysis ($C_{14}H_{16}O_3$): calcd. C 72.39, H 6.94; found C 72.42, H 7.30.

4,4a-Dihydro-3*H*-xanthen-7-yl trifluoromethanesulfonate (2)



To a solution of (1,4a-*trans*)-1-methoxy-2,3,4,4a-tetrahydro-1*H*-xanthen-7-ol (**1**) (500 mg, 2.15 mmol, 1.00 equiv.) in absolute dichloromethane (10 mL), NEt_3 (448 µL, 327 mg, 3.23 mmol. 1.50 equiv.) and triflic anhydride (533 µL, 911 mg, 3.32 mmol. 1.50 equiv.) were added under argon at 0 °C. The reaction mixture was stirred for 1.5 h. After addition of water, the aqueous phase was extracted with dichloromethane (3 × 10 mL). The combined organic layers were washed with saturated $NaHCO_3$-solution and brine, dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 40:1) yielding the product as a colorless solid (589 mg, 1.77 mmol, 82%).

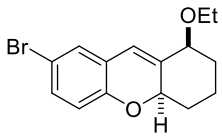
R_f (cyclohexane/ethyl acetate = 5/1) = 0.59. m.p.: 64–69 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.93 (dd, ^3J = 8.8 Hz, ^4J = 2.9 Hz, 1 H, 6-H), 6.87 (d, ^4J = 2.9 Hz 1 H, 8-H), 6.80 (d, ^3J = 8.8 Hz, 1 H, 5-H), 6.15 (d, ^3J = 9.5 Hz 1 H, 1-H), 6.10 (s, 1 H, 9-H), 6.03–5.94 (m, 1 H, 2-H), 5.02–4.98 (m, 1 H, 4a-H), 2.43–2.31 (m, 3 H, CH_2), 2.08–1.95 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.6 ($C_{quart.}$, C-10a), 143.6 ($C_{quart.}$, C-7), 134.6 ($C_{quart.}$, C-9a), 132.6 (+, CH), 125.6 (+, CH), 125.4 ($C_{quart.}$, C-8a), 120.4 (+, CH), 118.6 ($C_{quart.}$, q, $^1J_{CF}$ = 321.0 Hz, CF_3), 118.7 (+, CH), 117.0 (+, CH), 116.5 (+, CH), 74.8 (+, CH, C-4a), 28.5 (–, CH_2), 24.6 (–, CH_2) ppm. ^{19}F NMR (376 MHz, $CDCl_3$): δ = –72.7 (s, 3 F, CF_3) ppm. IR (ATR): $\tilde{\nu}$/cm^{-1} = 2947 (w), 2828 (vw), 1573 (vw), 1484 (m), 1416 (s), 1348 (w), 1247 (w), 1205 (s), 1134 (s), 1114 (s), 1047 (w), 1034 (m), 1001 (w), 952 (m), 897 (m), 870 (s), 858 (m), 817 (m), 771 (w), 746 (w), 733 (w), 712 (m), 667 (w), 630 (m), 597 (s), 573 (m), 533 (w), 508 (m), 490 (m), 466 (m), 441 (w), 409 (w) cm^{-1}. MS (EI, 70 eV), *m/z* (%): 332 (68) $[M]^+$, 200 (13), 199 (100) $[M–SO_2CF_3]^+$, 181.1 (29), 171 (12), 153 (18), 128 (15), 115 (11). HR-EIMS ($C_{14}H_{11}F_3O_4S$): calcd. 332.0330; found 332.0329.

General procedure for the elimination of alkyl ethers (GP1)

A solution of alkyl ether (1.00 equiv.) in absolute dichloromethane under argon atmosphere was cooled to 0 °C. Then, NEt_3 and Tf_2O were added successively. The reaction mixture was stirred for 1.5 h at that temperature. If the conversion was not completed, yet, the mixture was warmed up to room temperature and stirred for an additional time (considering table 1). The solution was quenched with water,[5] diluted with dichloromethane and washed successively with saturated sodium hydrogen carbonate solution and brine. The organic layers were dried over sodium sulfate, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate or pentane/diethyl ether).

(1,4a-*trans*)-7-Bromo-1-ethoxy-2,3,4,4a-tetrahydro-1*H*-xanthene (3-OEt)

To a solution of (1,4a-*trans*-7-bromo-2,3,4,4a-tetrahydro-1*H*-xanthen-1-ol[6] (300 mg, 1.07 mmol, 1.00 equiv.) in absolute THF (10 mL), NaH (60% in mineral oil, 59.9 mg, 1.50 mmol. 1.40 equiv.) and EtI (172 µL, 334 mg, 2.14 mmol. 2.00 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 4 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 10 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent

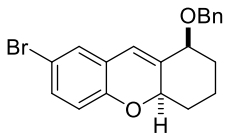


was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 20:1) yielding the product as a colorless solid (164 mg, 0.530 mmol, 50%).

R_f = 0.43 (cyclohexane/ethyl acetate = 10/1). – m.p.: 159 °C. – ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.09 (dd, ^3J = 8.5 Hz, ^4J = 2.4 Hz, 1 H, 6-H), 7.02 (d, ^4J = 2.4 Hz 1 H, 8-H), 6.53 (d, ^3J = 8.5 Hz, 1 H, 5-H), 6.22 (s, ^3J = 9.6 Hz, 1 H, 9-H), 5.87 (dd, ^3J = 11.4 Hz, ^3J = 5.2 Hz, 1 H, 4a-H), 3.68–3.58 (m, 3 H, OCH_2CH$_3$, 1-H), 2.19–1.06 (m, 2 H, CH_2), 1.92–1.85 (m, 1 H, CH_2), 1.80–1.68 (m, 1 H, CH_2), 1.46–1.28 (m, 2 H, CH_2), 1.28 (t, ^3J = 7.0 Hz, 3 H, CH_3) ppm. – ^{13}C NMR (100 MHz, $CDCl_3$): δ = 151.5 (C_{quart}, C-10a), 139.4 (C_{quart}, C-9a), 130.8 (+, CH), 128.7 (+, CH), 122.7 (C_{quart}, C-8a), 116.5 (+, CH), 112.5 (C_{quart}, C-7), 112.5 (+, CH), 77.8 (+, CH), 76.8 (+, CH), 65.3 (–, OCH_2CH$_3$), 34.8 (–, CH_2), 33.8 (–, CH_2), 20.0 (–, CH_2), 15.5 (+, CH_3) ppm. – IR (ATR): $\tilde{\nu}$/cm^{-1} = 2978 (vw), 2950 (w), 2858 (w), 1477 (w), 1441 (w), 1410 (w), 1369 (w), 1349 (w), 1240 (w), 1213 (w), 1201 (w), 1139 (w), 1115 (m), 1068 (w), 1043 (w), 1008 (w), 949 (w), 891 (w), 854 (w), 809 (m), 760 (vw), 701 (w), 628 (w), 569 (w), 554 (w), 515 (vw), 456 (w), 423 (w). – MS (EI, 70 eV), *m/z* (%): 310/308 (100/99) $[M]^+$, 281/279 (60/49) $[M–Et]^+$, 266 (40), 265 (57), 264 (76), 263 (66), 262 (65), 261 (39), 237 (80), 235 (71), 115 (46). – HR-EIMS ($C_{15}H_{17}O_2Br$): calcd. 308.0414; found 308.0412.

(1,4a-*trans*)-1-(Benzyloxy)-7-bromo-2,3,4,4a-tetrahydro-1*H*-xanthene (3-OBn)

To a solution of (1,4a-*trans*)-7-bromo-2,3,4,4a-tetrahydro-1*H*-xanthen-1-ol[6] (100 mg, 0.458 mmol, 1.00 equiv.) in absolute THF (1.5 mL), NaH (60% in mineral oil, 27.5 mg, 0.687 mmol. 1.50 equiv.) and BnBr (54 µL, 78.3 mg, 0.458 mmol. 1.00 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 18 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 3 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 100:1) yielding the product as a yellow oil (125 mg, 0.337 mmol, 74%).

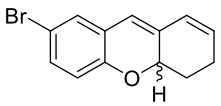


R_f = 0.42 (cyclohexane/ethyl acetate = 10/1). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.43–7.37 (m, 4 H, 4 × $H_{arom.}$), 7.36–7.29 (m, 1 H, $H_{arom.}$), 7.10 (dd, ^3J = 8.5 Hz, ^4J = 2.4 Hz, 1 H, 6-H), 7.03 (d, ^4J = 2.4 Hz, 1 H, 8-H), 6.54 (d, ^3J = 8.5 Hz, 1 H, 5-H), 6.54 (s, 1 H, 9-H), 4.87 (dd, ^3J = 11.3 Hz,

^3J = 5.4 Hz, 1 H, 4a-H), 4.75–4.58 (m, 2 H, OCH_2), 3.83–3.70 (m, 1 H, 1-H), 2.27–2.06 (m, 2 H, CH_2), 1.95–1.67 (m, 2 H, CH_2), 1.49–1.31 (m, 2 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 151.5 (C_{quart}, C-10a), 139.2 (C_{quart}, OCH_2C), 138.3 (C_{quart}, C-9a), 130.9 (+, C-6), 128.8 (+, C-8), 128.5 (+, 2 × $CH_{arom.}$), 127.7 (+, CH), 127.4 (+, 2 × $CH_{arom.}$), 122.6 (C_{quart}, C-8a), 116.5 (+, CH), 112.8 (+, CH), 112.5 (C_{quart}, C-7), 77.2 (+, CH), 76.8 (+, CH), 71.4 (–, OCH_2), 34.8 (–, CH_2), 33.6 (–, CH_2), 20.0 (–, CH_2) ppm. IR (ATR): $\tilde{\nu}$/cm^{-1} = 3029 (vw), 2939 (w), 2860 (w), 1698 (w), 1598 (w), 1477 (m), 1452 (w), 1410 (w), 1356 (w), 1240 (m), 1199 (m), 1110 (m), 1069 (m), 1026 (m), 885 (m), 810 (m), 735 (m), 697 (m), 638 (w), 510 (w), 457 (w). MS (EI, 70 eV), *m/z* (%): 373/371 (10/10) $[M+H]^+$, 372/370 (45/46) $[M]^+$, 282/280 (12/13), 281/279 (80/80) $[M–Bn]^+$, 265/263 (10/15) $[M–OBn]^+$, 264/262 (20/19) $[M–BnOH]^+$, 237/235 (46/40), 115 (12), 92 (11), 91 (100) $[Bn]^+$. HR-EIMS ($C_{20}H_{19}O_2Br$): calcd. 370.0563; found 370.0561.

7-Bromo-4,4a-dihydro-3*H*-xanthene (4)

a) Starting from (1,4a-*trans*)-7-bromo-1-methoxy-2,3,4,4a-tetrahydro-1*H*-xanthene[6] (**3-OMe**, 50 mg, 0.169 mmol), the product was synthesized according to GP1. The crude was purified by column chromatography (cyclohexane/ethyl acetate = 100/1) yielding the product as a light yellow solid (37.3 mg, 0.142 mmol, 84%).



b) Starting from (1,4a-*trans*)-7-bromo-1-ethoxy-2,3,4,4a-tetrahydro-1*H*-xanthene (**3-OEt**, 29 mg, 0.0938 mmol), the product was synthesized according to GP1. The crude was purified by column chromatography (cyclohexane/ethyl acetate = 100/1) yielding the product as a light yellow solid (14.4 mg, 0.0547 mmol, 58%).

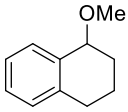
c) Starting from (1,4a-*trans*)-1-(benzyloxy)-7-bromo-2,3,4,4a-tetrahydro-1*H*-xanthene (**3-OBn**, 50 mg, 0.135 mmol), the product was synthesized according to GP1. The crude was purified by column chromatography (cyclohexane/ethyl acetate = 100/1) yielding the product as a light yellow solid (9.5 mg, 0.0361 mmol, 27%).

R_f (cyclohexane/ethyl acetate = 100/1) = 0.22. m.p.: 154 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.13 (dd, ^3J = 8.5 Hz, ^4J = 2.3 Hz, 1 H, 6-H), 7.08 (d, ^4J = 2.3 Hz, 1 H, 8-H), 6.66 (d, ^3J = 8.5 Hz, 1 H, 5-H), 6.14 (d, ^3J = 9.6 Hz 1 H, 1-H), 6.06 (s, 1 H, 9-H), 5.96–5.90 (m, 1 H, 2-H), 5.01–4.94 (m, 1 H, 4a-H), 2.45–2.30 (m, 3 H, CH_2), 2.07–1.93 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.2 (C_{quart}, C-10a), 133.8 (C_{quart}, C-9a), 131.9 (+, CH), 130.7 (+, CH), 128.7 (+, CH), 126.0 (C_{quart}, C-8a), 126.0 (+, CH), 117.4 (+, CH), 117.1 (+, CH), 113.5 (C_{quart}, C-7), 74.6 (+, CH, C-4a),

28.5 (–, CH_2), 24.6 (–, CH_2) ppm. IR (ATR): $\tilde{\nu}$ = 3032 (vw), 2921 (w), 2831 (w), 1673 (w), 1475 (m), 1427 (w), 1374 (w), 1346 (w), 1242 (m), 1219 (m), 1118 (m), 1070 (m), 1031 (m), 9626 (w), 897 (m), 885 (m), 860 (w), 815 (m), 789 (w), 716 (m), 670 (w), 639 (m), 604 (w), 563 (m), 538 (w), 496 (w), 459 (w), 415 (w) cm^{-1}. MS (EI, 70 eV), *m/z* (%): 264/262 (96/100) $[M]^+$, 261 (48), 183 (44) $[M–Br]^+$, 181 (45), 165 (60), 153 (54), 152 (67). HR-EIMS ($C_{13}H_{11}O_4Br$): calcd. 261.9997; found 261.9993.

1-Methoxy-1,2,3,4-tetrahydronaphthalene (5a)[7]

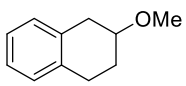
To a solution of 1,2,3,4-tetrahydronaphthalen-1-ol (500 mg, 3.37 mmol, 1.00 equiv.) in absolute THF (50 mL), NaH (60% in mineral oil, 176 mg, 4.39 mmol. 1.30 equiv.) and MeI (275 µL, 623 mg, 4.39 mmol. 1.30 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 3 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 50 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 10:1) yielding the product as a colorless oil (467 mg, 2.88 mmol, 85%).



The analytical data were in accordance with literature values.[7]

R_f = 0.65 (cyclohexane/ethyl acetate = 5/1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38–7.32 (m, 1 H, $H_{arom.}$), 7.22–7.14 (m, 2 H, 2 × $H_{arom.}$), 7.12–7.07 (m, 1 H, $H_{arom.}$), 4.32 (t, ^3J = 4.7 Hz, 1 H, 1-H), 3.44 (s, 3 H, OCH_3), 2.84 (dt, ^2J = 16.9 Hz, ^3J = 5.7 Hz, 1 H, CH_2), 2.77–2.67 (m, 1 H, CH_2), 2.06–1.85 (m, 3 H, CH_2), 1.80–1.69 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 137.5 (C_{quart}, C-8a), 136.6 (C_{quart}, C-4a), 129.3 (+, $CH_{arom.}$), 129.0 (+, $CH_{arom.}$), 127.5 (+, $CH_{arom.}$), 125.7 (+, $CH_{arom.}$), 76.9 (+, C*H*OMe), 56.3 (+, CH_3), 29.2 (–, CH_2), 27.5 (–, CH_2), 18.8 (–, CH_2) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3356 (vw), 3022 (vw), 2937 (w), 2818 (vw), 1683 (w), 1601 (vw), 1489 (vw), 1454 (w), 1349 (vw), 1286 (w), 1189 (vw), 1117 (w), 1085 (w), 945 (w), 901 (vw), 901 (vw), 767 (w), 740 (w), 553 (vw). MS (EI, 70 eV), *m/z* (%): 162 (23) $[M]^+$, 146 (42), 145 (28), 133 (21), 131 (45) $[M–OCH_3]^+$, 130 (55), 129 (73), 119 (21), 118 (100) $[C_9H_{10}]^+$, 117 (21), 115 (38), 91 (25), 90 (45), 89 (23). HR-EIMS ($C_{11}H_{14}O$): calcd. 162.1044; found 162.1045.

2-Methoxy-1,2,3,4-tetrahydronaphthalene (5b)



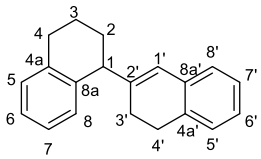
To a solution of 1,2,3,4-tetrahydronaphthalen-2-ol (450 µL, 500 mg, 3.37 mmol, 1.00 equiv.) in absolute THF (50 mL), NaH (60% in mineral oil, 176 mg, 4.39 mmol, 1.30 equiv.) and MeI (275 µL, 623 mg, 4.39 mmol. 1.30 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 3 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 50 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 10:1) yielding the product as a light orange oil (435 mg, 2.69 mmol, 80%).

The analytical data were in accordance with literature values.[8, 9]

R_f = 0.54 (cyclohexane/ethyl acetate = 5/1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.14–7.06 (m, 4 H, 4 × $H_{arom.}$), 3.71–3.63 (m, 1 H, 2-H), 3.44 (s, 3 H, OCH_3), 3.09 (dd, ^2J = 16.2 Hz, ^3J = 4.7 Hz, 1 H, CH_2), 2.95 (dt, ^2J = 16.9 Hz, ^3J = 5.9 Hz, 1 H, CH_2), 2.84–2.74 (m, 2 H, CH_2), 2.14–2.04 (m, 1 H, CH_2), 1.89–1.78 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 136.1 (C_{quart}, C-8a), 134.5 (C_{quart}, C-4a), 129.5 (+, $CH_{arom.}$), 128.6 (+, $CH_{arom.}$), 125.9 (+, $CH_{arom.}$), 125.8 (+, $CH_{arom.}$), 75.8 (+, C-2), 55.9 (+, CH_3), 35.2 (–, CH_2), 27.8 (–, CH_2), 27.0 (–, CH_2) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3448 (vw), 3018 (w), 2928 (m), 2822 (w), 1581 (vw), 1494 (w), 1454 (w), 1356 (w), 1238 (vw), 1188 (w), 1118 (w), 1098 (m), 981 (vw), 916 (vw), 813 (vw), 745 (m), 713 (vw), 434 (w). MS (EI, 70 eV), *m/z* (%): 162 (22) $[M]^+$, 161 (28), 131 (60) $[M–OCH_3]^+$, 130 (100) $[C_{10}H_{310}]^+$, 129 (39), 117 (52), 115 (33), 104 (52), 91 (40). HR-EIMS ($C_{11}H_{14}O$): calcd. 162.1046; found 162.1044.

1,2,3,3',4,4'-Hexahydro-1,2'-binaphthalene (6)

a) Starting from 1-methoxy-1,2,3,4-tetrahydronaphthalene (**5a**, 100 mg, 0.616 mmol), the product was synthesized according to GP1. The crude was purified by column chromatography (pentane/diethyl ether = 500/1) yielding the product as a light yellow oil (59.1 mg, 0.227 mmol, 74%).

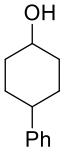


b) Starting from 2-methoxy-1,2,3,4-tetrahydronaphthalene (**5b**, 100 mg, 0.616 mmol), the product was synthesized according to GP1. The crude was purified by column chromatography (pentane/diethyl ether = 500/1) yielding the product as a light yellow solid (23.2 mg, 0.0892 mmol, 29%).

The analytical data were in accordance with literature values.[10, 11]

R_f = 0.83 (cyclohexane/ethyl acetate = 10/1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.20 (d, ^3J = 7.6 Hz, 1 H, $H_{arom.}$), 7.17–7.06 (m, 6 H, 6 × $H_{arom.}$), 7.00 (d, ^3J = 7.0 Hz, 1 H, $H_{arom.}$), 6.21 (s, 1 H, 1-H), 3.74–3.66 (m, 1 H, 1'-H), 2.86–2.74 (m, 4 H, CH_2), 2.26–2.08 (m, 2 H, CH_2), 2.00–1.90 (m, 2 H, CH_2), 1.88–1.68 (m, 2 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 145.6 (C_{quart}, C-2'), 137.9 (C_{quart}), 137.7 (C_{quart}), 134.9 (C_{quart}), 134.8 (C_{quart}), 129.4 (+, $CH_{arom.}$), 129.2 (+, $CH_{arom.}$), 127.3 (+, $CH_{arom.}$), 126.6 (+, $CH_{arom.}$), 126.4 (+, $CH_{arom.}$), 126.1 (+, $CH_{arom.}$), 125.8 (+, $CH_{arom.}$), 125.7 (+, $CH_{arom.}$), 125.3 (+, C-1') 47.4 (+, C-1), 30.0 (–, CH_2), 28.7 (–, CH_2), 28.7 (–, CH_2), 25.3 (–, CH_2), 21.6 (–, CH_2) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3059 (w), 3015 (m), 2930 (s), 2859 (m), 1908 (vw), 1644 (w), 1600 (w), 1573 (vw), 1488 (m), 1452 (m), 1434 (m), 1272 (w), 1202 (vw), 1139 (w), 1035 (w), 998 (vw), 937 (w), 879 (w), 848 (w), 804 (vw), 757 (m), 742 (m), 675 (vw), 599 (w), 557 (vw). MS (EI, 70 eV), *m/z* (%): 261 (22), 260 (100) $[M]^+$, 131 (32), 130 (100) $[C_{10}H_{10}]^+$, 129 (49), 128 (36), 115 (23). HR-EIMS ($C_{20}H_{20}$): calcd. 260.1567; found 260.1565.

4-Phenylcyclohexan-1-ol[12]



To a solution of 4-phenylcyclohexanon (5.00 g, 28.7 mmol, 1.00 equiv.) in acetonitrile (80 mL), $NaHSO_4$ (689 mg, 5.74 mmol, 0.200 equiv.) and $NaBH_4$ (2.17 g, 57.4 mmol, 2.00 equiv.) were added successively. The reaction mixture was heated under reflux for 1 h. Water (80 mL) was added and the mixture was stirred for 18 h. After the extraction with dichloromethane (3 × 80 mL), the combined organic phases were washed with brine, dried over sodium sulfate, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 10:1 to 5:1) yielding the product as a separable mixture of the *cis*-isomer (654 mg, 3.67 mmol, 17%) and the *trans*-isomer (3.52 g, 20.0 mmol, 70%) as colorless solids.

The analytical data were in accordance with literature values.[13, 14]

(1,4-*cis*)-4-Phenylcyclohexan-1-ol:

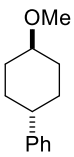
R_f = 0.15 (cyclohexane/ethyl acetate = 5/1). m.p.: 63 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.33–7.15 (m, 5 H, 5 × $H_{arom.}$), 4.14 (s, 1 H, 1-H), 2.60–2.48 (m, 1 H, 4-H), 1.97–1.83 (m, 4 H, CH_2), 1.74–1.63 (m, 4 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 147.3 (C_{quart}), 128.4 (+, 2 × $CH_{arom.}$), 126.9 (+, 2 × $CH_{arom.}$), 126.0 (+, $CH_{arom.}$), 65.6 (+, C-1), 43.9 (+, C-4), 33.1 (–, 2 × CH_2), 27.7 (–, 2 × CH_2) ppm. IR (ATR): $\tilde{\nu}$/cm^{-1} = 3282 (w), 3022 (vw), 3000 (vw), 2923 (w), 2889 (w), 2859 (w), 1601 (vw), 1579 (w), 1493 (w), 1371 (w), 1280 (vw), 1255 (w), 1146 (w), 1107

(w), 1063 (vw), 1032 (w), 992 (w), 949 (m), 894 (vw), 869 (w), 850 (vw), 822 (vw), 796 (vw), 760 (m), 704 (m), 635 (w), 603 (w), 531 (w). MS (EI, 70 eV), *m/z* (%): 176 (100) $[M]^+$, 158 (32), 143 (35), 130 (33), 129 (27), 118 (71), 117 (60), 104 (89), 91 (66). HR-EIMS ($C_{12}H_{16}O$): calcd. 176.1201; found 176.1201.

(1,4-*trans*)-4-Phenylcyclohexan-1-ol:

R_f = 0.10 (cyclohexane/ethyl acetate = 5/1). m.p.: 92–115 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.33–7.27 (m, 2 H, 2 × $H_{arom.}$), 7.23–7.16 (m, 3 H, 3 × $H_{arom.}$), 3.74–3.65 (m, 1 H, 1-H), 2.50 (tt, 1 H, 4-H), 2.15–2.06 (m, 2 H, CH_2), 1.99–1.89 (m, 2 H, CH_2), 1.61–1.37 (m, 4 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.5 (C_{quart}), 128.3 (+, 2 × $CH_{arom.}$), 126.8 (+, 2 × $CH_{arom.}$), 126.1 (+, $CH_{arom.}$), 70.6 (+, C-1), 43.4 (+, C-4), 35.9 (–, 2 × CH_2), 32.4 (–, 2 × CH_2) ppm. IR (ATR): $\tilde{\nu}$/cm^{-1} = 3272 (w), 3028 (w), 2920 (m), 2851 (w), 1560 (vw), 1493 (w), 1450 (m), 1344 (w), 1311 (w), 1224 (vw), 1117 (vw), 1060 (m), 1028 (w), 966 (w), 887 (w), 841 (w), 754 (m), 695 (m), 535 (m), 464 (w). MS (EI, 70 eV), *m/z* (%): 176 (18) $[M]^+$, 159 (12), 158 (100) $[M–H_2O]^+$, 143 (83), 130 (58), 129 (31), 117 (30), 115 (15), 104 (45), 91 (37). HR-EIMS ($C_{12}H_{16}O$): calcd. 176.1201; found 176.1204.

((1,4-*trans*)-4-Methoxycyclohexyl)benzene (7a-OMe)



To a solution of (1,4-*trans*)-4-phenylcyclohexanol (100 mg, 0.567 mmol, 1.00 equiv.) in absolute THF (2 mL), NaH (60% in mineral oil, 34.0 mg, 0.851 mmol. 1.50 equiv.) and MeI (46.3 µL, 105 mg, 0.737 mmol. 1.30 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 23 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 3 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 10:1) yielding the product as a colorless liquid (79.7 mg, 0.419 mmol, 74%).

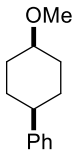
The analytical data were in accordance with literature values.[15]

R_f = 0.55 (cyclohexane/ethyl acetate = 5/1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32–7.27 (m, 2 H, 2 × $H_{arom.}$), 7.23–7.16 (m, 3 H, $H_{arom.}$), 3.39 (s, 3 H, OCH_3), 3.21 (tt, ^3J = 10.8 Hz, ^3J = 4.2 Hz, 1 H, 1-H), 2.51 (tt, ^3J = 12.1 Hz, ^3J = 3.5 Hz, 1 H, 1-H), 2.24–2.14 (m, 2 H, CH_2), 2.01–1.89 (m, 2 H, CH_2), 1.57–1.43 (m, 2 H, CH_2), 1.42–1.28 (m, 2 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.7 (C_{quart}), 128.3 (+, 2 × $CH_{arom.}$), 126.8 (+, 2 × $CH_{arom.}$), 126.0 (+, $CH_{arom.}$), 79.2 (+, OCH_3),

55.7 (+, C-1), 43.7 (+, C-4), 32.4 (–, 2 × CH_2), 32.2 (–, 2 × CH_2) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3025 (vw), 2926 (m), 2855 (w), 2818 (vw), 1600 (vw), 1492 (w), 1448 (w), 1373 (w), 1188 (w), 1142 (w), 1118 (w), 1089 (m), 1024 (w), 932 (w), 896 (w), 843 (vw), 755 (m), 698 (m), 535 (w), 515 (w), 463 (vw). MS (EI, 70 eV), *m/z* (%): 190 (6) $[M]^+$, 158 (100), 143 (34), 130 (23), 129 (15), 117 (11), 104 (25), 91 (23), 71 (12). HR-EIMS ($C_{13}H_{18}O$): calcd. 190.1358; found 190.1355.

((1,4-*cis*)-4-Methoxycyclohexyl)benzene (7b)

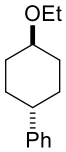
To a solution of (1,4-*cis*)-4-phenylcyclohexanol (100 mg, 0.567 mmol, 1.00 equiv.) in absolute THF (2 mL), NaH (60% in mineral oil, 34.0 mg, 0.851 mmol. 1.50 equiv.) and MeI (46.3 µL, 105 mg, 0.737 mmol. 1.30 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 21 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 3 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 10:1) yielding the product as a colorless liquid (87.5 mg, 0.460 mmol, 81%).



The analytical data were in accordance with literature values.[15]

R_f = 0.50 (cyclohexane/ethyl acetate = 10/1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32–7.22 (m, 4 H, 4 × $H_{arom.}$), 7.21–7.15 (m, 1 H, $H_{arom.}$), 3.52 (quin, ^3J = 2.9 Hz, 1 H, 1-H), 3.35 (s, 3 H, OCH_3), 2.53 (tt, ^3J = 12.0 Hz, ^4J = 3.5 Hz, 1 H, 4-H), 2.11–2.02 (m, 2 H, CH_2), 1.88–1.75 (m, 2 H, CH_2), 1.68–1.60 (m, 2 H, CH_2), 1.57–1.46 (m, 2 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 147.6 (C_{quart}), 128.3 (+, 2 × $CH_{arom.}$), 126.9 (+, 2 × $CH_{arom.}$), 125.8 (+, $CH_{arom.}$), 74.3 (+, OCH_3), 65.6 (+, C-1), 44.0 (+, C-4), 29.8 (–, 2 × CH_2), 28.1 (–, 2 × CH_2) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3443 (vw), 3060 (w), 3027 (w), 2930 (m), 2858 (m), 2820 (w), 1715 (vw), 1601 (w), 1493 (w), 1451 (w), 1374 (w), 1353 (w), 1328 (vw), 1279 (vw), 1227 (w), 1182 (w), 1153 (w), 1108 (m), 1090 (m), 1029 (w), 1005 (w), 929 (w), 918 (w), 895 (vw), 867 (vw), 792 (vw), 755 (w), 699 (m), 619 (vw), 528 (w). MS (EI, 70 eV), *m/z* (%): 191 (12) $[M+H]^+$, 190 (100) $[M]^+$, 158 (48), 143 (26), 130 (21), 129 (18), 119 (16), 117 (19), 115 (14), 104 (56), 103 (11), 91 (50), 78 (10), 71 (87). HR-EIMS ($C_{13}H_{18}O$): calcd. 190.1358; found 190.1356.

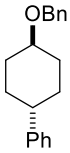
((1,4-*trans*)-4-Ethoxycyclohexyl)benzene (7a-OEt)[16]



To a solution of (1,4-*trans*)-4-phenylcyclohexanol (500 mg, 2.84 mmol, 1.00 equiv.) in absolute THF (10 mL), NaH (60% in mineral oil, 170 mg, 4.26 mmol. 1.50 equiv.) and EtI (297 µL, 576 mg, 3.69 mmol. 1.30 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 18 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 10 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 100:1) yielding the product as a colorless liquid (298 mg, 1.45 mmol, 51%).

R_f = 0.66 (cyclohexane/ethyl acetate = 5/1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32–7.27 (m, 2 H, 2 × $H_{arom.}$), 7.23–7.16 (m, 3 H, 3 × $H_{arom.}$), 3.56 (q, ^3J = 7.0 Hz, 2 H, OCH_2), 3.37–3.24 (m, 1 H, 1-H), 2.51 (tt, ^3J = 12.0 Hz, ^4J = 3.4 Hz, 1 H, 4-H), 2.23–2.11 (m, 2 H, CH_2), 2.01–1.88 (m, 2 H, CH_2), 1.57–1.45 (m, 2 H, CH_2), 1.44–1.31 (m, 2 H, CH_2), 1.23 (t, ^3J = 7.0 Hz, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.8 (C_{quart}), 128.3 (+, 2 × $CH_{arom.}$), 126.8 (+, 2 × $CH_{arom.}$), 126.0 (+, $CH_{arom.}$), 77.6 (+, C-1), 63.3 (–, OCH_2), 43.7 (+, C-4), 32.8 (–, 2 × CH_2), 32.5 (–, 2 × CH_2), 15.8 (+, CH_3) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3851 (vw), 3442 (vw), 3061 (vw), 3027 (vw), 2972 (w), 2931 (w), 2857 (vw), 1707 (vw), 1602 (vw), 1493 (vw), 1450 (w), 1373 (vw), 1356 (vw), 1105 (w), 1029 (vw), 897 (vw), 755 (vw), 699 (w), 532 (vw). MS (EI, 70 eV), *m/z* (%): 204 (7) $[M]^+$, 159 (11), 158 (100) $[M–EtOH]^+$, 143 (27), 130 (19), 129 (11), 104 (23), 91 (25), 85 (12), 57 (13). HR-EIMS ($C_{14}H_{20}O$): calcd. 204.1514; found 204.1513.

((1,4-*trans*)-4-(Benzyloxy)cyclohexyl)benzene (7a-OBn)



To a solution of (1,4-*trans*)-4-phenylcyclohexanol (200 mg, 1.13 mmol, 1.00 equiv.) in absolute THF (4 mL), NaH (60% in mineral oil, 63.3 mg, 1.58 mmol. 1.40 equiv.) and BnBr (174 µL, 251 mg, 1.47 mmol. 1.30 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 18 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 5 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 100:1) yielding the product as a colorless oil (154 mg, 0.578 mmol, 51%).

The analytical data were in accordance with literature values.[17]

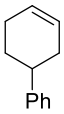
R_f = 0.65 (cyclohexane/ethyl acetate = 10/1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.45–7.35 (m, 4 H, 4 × $H_{arom.}$), 7.34–7.27 (m, 3 H, 3 × $H_{arom.}$), 7.25–7.15 (m, 3 H, 3 × $H_{arom.}$), 4.63 (s, 2 H, OCH_2), 3.53–3.37 (m, 1 H, 1-H), 2.62–2.47 (m, 1 H, 4-H), 2.23–2.17 (m, 2 H, CH_2), 2.03–1.87 (m, 2 H, CH_2), 1.66–1.37 (m, 4 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.7 (C_{quart}), 139.1 (C_{quart}), 128.3 (+, 2 × $CH_{arom.}$), 128.3 (+, 2 × $CH_{arom.}$), 127.5 (+, 2 × $CH_{arom.}$), 127.4 (+, $CH_{arom.}$), 126.7 (+, 2 × $CH_{arom.}$), 126.0 (+, $CH_{arom.}$), 77.2 (+, C-1), 69.9 (–, OCH_2), 43.7 (+, C-4), 32.6 (–, 2 × CH_2), 32.5 (–, 2 × CH_2) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3026 (vw), 2925 (w), 2851 (w), 1600 (vw), 1492 (w), 1449 (w), 1358 (w), 1308 (vw), 1210 (vw), 1097 (m), 1074 (m), 1029 (w), 953 (w), 899 (w), 756 (w), 736 (m), 696 (m), 651 (w), 612 (w), 532 (w), 452 (w). MS (EI, 70 eV), *m/z* (%): 266 (1) $[M]^+$, 175 (100) $[M–Bn]^+$, 158 (35) $[M–BnOH]^+$, 157 (27), 92 (11), 91 (100) $[Bn]^+$. HR-EIMS ($C_{19}H_{22}O$): calcd. 266.1665; found 266.1664.

4-Phenylcyclohexene (8a) and 3-phenylcyclohexene (8b)

Starting from either **7a-OMe**, **7b, 7a-OEt**, or **7a-OBn** (50 mg) the products were synthesized according to GP1. The crude was purified by column chromatography (pentane) yielding the products as colorless liquids, which could not always been completely separated.

4-Phenylcyclohexene (**8a**):

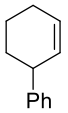
The analytical data were in accordance with literature values.[18]



R_f = 0.51 (pentane). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.35–7.28 (m, 2 H, 2 × $H_{arom.}$), 7.26–7.16 (m, 3 H, 3 × $H_{arom.}$), 5.82–5.72 (m, 2 H, 2 × H_{alkene}), 2.86–2.75 (m, 1 H, PhC*H*), 2.34–2.25 (m, 1 H, CH_2), 2.24–2.10 (m, 3 H, CH_2), 1.98–1.90 (m, 1 H, CH_2) , 1.82–1.70 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 147.3 (C_{quart}), 128.3 (+, 2 × CH), 127.0 (+, CH), 126.9 (+, 2 × CH), 126.8 (+, CH), 125.9 (+, CH), 40.2 (+, Ph*C*H), 33.4 (–, CH_2), 29.7 (–, CH_2), 25.9 (–, CH_2) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3443 (vw), 3062 (w), 3024 (m), 2916 (m), 2836 (w), 1651 (w), 1605 (w), 1493 (m), 1453 (w), 1434 (w), 1255 (w), 1184 (w), 1140 (w), 1115 (w), 1043 (w), 1029 (w), 969 (w), 942 (w), 914 (w), 889 (w), 756 (m), 721 (w), 699 (m), 596 (vw), 528 (w). MS (EI, 70 eV), *m/z* (%): 158 (39) $[M]^+$, 157 (27) $[M–H]^+$, 129 (14), 128 (10), 115 (14), 104 (100) $[C_8H_8]^+$, 91 (22), 78 (10).– HR-EIMS ($C_{12}H_{14}$): calcd. 158.1096; found 158.1094.

3-Phenylcyclohexene (**8b**):

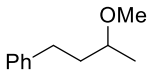
The analytical data were in accordance with literature values.[18-20]



R_f = 0.57 (pentane). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.34–7.27 (m, 2 H, 2 × $H_{arom.}$), 7.25–7.17 (m, 3 H, 3 × $H_{arom.}$), 5.93–5.85 (m, 1 H, H_{alkene}), 5.75–5.68 (m, 1 H, H_{alkene}), 3.44–3.37 (m, 1 H, PhC*H*), 2.12–1.97 (m, 3 H, CH_2), 1.79–1.70 (m, 1 H, CH_2), 1.67–1.57 (m, 2 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.7 (C_{quart}), 130.2 (+, CH), 128.4 (+, CH), 128.3 (+, 2 × CH), 127.7 (+, 2 × CH), 125.9 (+, CH), 41.9 (+, PhCH), 32.6 (–, CH_2), 25.0 (–, CH_2), 21.2 (–, CH_2) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3442 (vw), 3059 (vw), 3024 (vw), 2931 (w), 2859 (vw), 1642 (vw), 1602 (vw), 1493 (vw), 1448 (vw), 1328 (vw), 1159 (vw), 1121 (vw), 1072 (vw), 1037 (vw), 957 (vw), 911 (vw), 880 (vw), 845 (vw), 757 (w), 724 (vw), 700 (w), 674 (vw), 598 (vw), 530 (vw). MS (EI, 70 eV), *m/z* (%): 158 (45) $[M]^+$, 157 (100) $[M–H]^+$, 145 (24), 144 (15), 143 (23), 130 (37), 129 (42), 128 (29), 117 (14), 116 (17), 115 (44), 91 (51), 78 (10). HR-EIMS ($C_{12}H_{14}$): calcd. 158.1096; found 158.1097.

(3-Methoxybutyl)benzene

To a solution of 4-phenyl-2-butanol (2.06 mL, 2.00 g, 13.3 mmol, 1.00 equiv.) in absolute THF



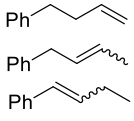
(30 mL), NaH (60% in mineral oil, 800 mg, 20.0 mmol. 1.50 equiv.) and MeI (1.08 mL, 2.46 g, 17.3 mmol. 1.30 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 4 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 30 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 10:1) yielding the product as a colorless liquid (2.03 g, 12.4 mmol, 93%).

The analytical data were in accordance with literature values.[21]

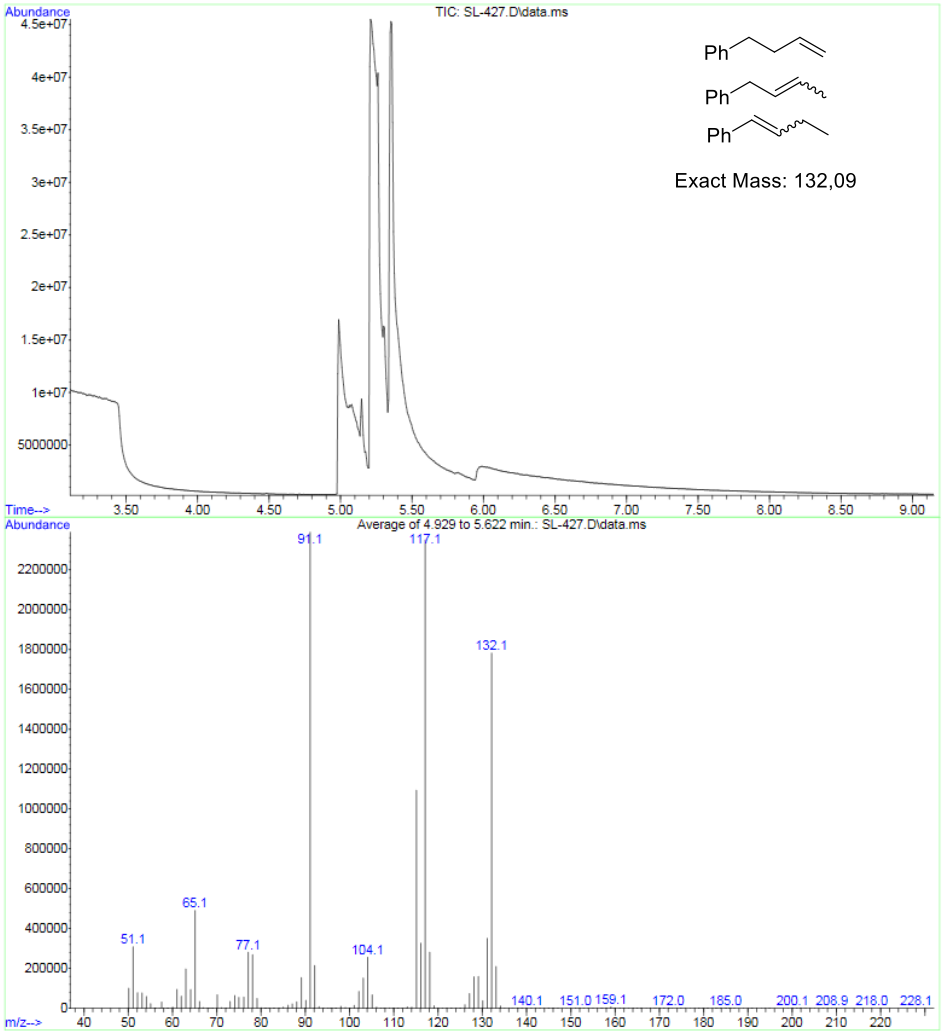
R_f = 0.19 (cyclohexane/ethyl acetate = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.33–7.26 (m, 2 H, 2 × $H_{arom.}$), 7.23–7.15 (m, 3 H, 3 × $H_{arom.}$), 3.34 (s, 3 H, OCH_3), 3.33–3.24 (m, 1 H, 3-H), 2.79–2.59 (m, 2 H, 2 × CH_2), 1.92–1.78 (m, 1 H, CH_2), 1.78–1.64 (m, 1 H, CH_2) , 1.17 (d, ^3J = 6.2 Hz, 3 H, CCH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 142.4 (C_{quart}), 128.4 (+, 2 × $CH_{arom.}$), 128.3 (+, 2 × $CH_{arom.}$), 125.7 (+, $CH_{arom.}$), 75.9 (+, C-3), 55.9 (+, OCH_3), 38.2 (–, CH_2), 31.7 (–, CH_2), 19.0 (+, CH_3) ppm. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3442 (vw), 3027 (vw), 2971 (w), 2926 (w), 2820 (vw), 1604 (vw), 1495 (vw), 1453 (w), 1373 (vw), 1135 (vw), 1088 (w), 744 (vw), 699 (w). MS (EI, 70 eV), *m/z* (%):

164 (1) $[M]^+$, 133 (12) $[M–OMe]^+$, 132 (31) $[M–MeOH]^+$, 131 (20), 118 (12), 117 (100) $[C_9H_9]^+$, 115 (19), 91 (66) $[C_7H_7]^+$, 89 (11), 65 (11). HR-EIMS ($C_{11}H_{16}O$): calcd. 164.1201; found 164.1200.

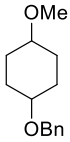
Buten-1-yl-benzene (mixture of isomers)



Starting from (3-methoxybutyl)benzene (250 mg, 1.52 mmol, 1.00 equiv.) the products were synthesized according to GP1 (1.50 equiv. of Tf_2O and NEt_3; 0 °C, 1.5 h). The crude was purified by column chromatography (pentane) yielding the products as an inseparable mixture of probably five isomers (74.1 mg, 0.561 mmol, 37%), which could not be separated and thus could not be analyzed *via* NMR. GC-MS measurement indicated the formation of several isomers (mass spectra of all peaks are similar, average is shown).



1-(Benzyloxy)-4-methoxycyclohexane (9)



To a solution of cyclohexane-1,4-diol (mixture of *cis/trans* isomers, 2.00 g, 17.2 mmol, 1.00 equiv.) in absolute THF (30 mL), NaH (454 mg, 18.9 mmol. 1.10 equiv.) and MeI (1.18 mL, 2.68 g, 18.9 mmol. 1.10 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 18 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 30 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 1:1, R_f = 0.25) yielding 4-methoxycyclohexan-1-ol as a colorless liquid (190 mg, 1.46 mmol, 8%).

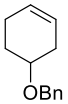
To a solution of 4-methoxycyclohexan-1-ol (130 mg, 0.999 mmol, 1.00 equiv.) in absolute THF (5 mL), NaH (72.0 mg, 3.00 mmol. 3.00 equiv.) and BnBr (356 µL, 513 mg, 3.00 mmol. 3.00 equiv.) were added under argon at 0 °C. The reaction mixture was warmed to room temperature and stirred for 18 h. After addition of water, the aqueous phase was extracted with ethyl acetate (3 × 5 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography (cyclohexane/ethyl acetate = 5:1) yielding the product as an inseparable mixture of *cis/trans* isomers (1:2.5) as a colorless liquid (190 mg, 0.862 mmol, 86%).

The analytical data were in accordance with literature values.[22]

R_f = 0.50 (cyclohexane/ethyl acetate = 5:1). ^{1}H NMR (300 MHz, $CDCl_3$): both isomers, δ = 7.31–7.15 (m, 5 H, 5 × $H_{arom.}$), 4.47 (s, 2 H, $OCH_{2(cis)}$), 4.45 (s, 2 H, $OCH_{2(trans)}$), 3.46–3.30 (m, 1 H, 4-H), 3.26 (s, 3 H, $OCH_{3(cis)}$), 3.25 (s, 3 H, $OCH_{3(trans)}$), 3.21–3.05 (m, 1 H, 1-H), 2.05–1.64 (m, 4 H, CH_2), 1.54–1.43 (m, 2 H, CH_2), 1.40–1.13 (m, 2 H, CH_2) ppm. ^{13}C NMR (75.0 MHz, $CDCl_3$): both isomers: δ = 139.2 ($C_{quart,cis}$), 139.0 ($C_{quart,trans}$), 128.33 (+, 2 × $CH_{arom.,cis}$), 128.27 (+, 2 × $CH_{arom.,trans}$), 127.5 (2 × $CH_{arom.,cis}$), 127.4 (2 × $CH_{arom.,trans}$, $CH_{arom.,cis}$), 127.3 ($CH_{arom.,trans}$), 78.0 (+, CH_{cis}), 76.5 (+, CH_{trans}), 76.2 (+, CH_{cis}), 74.3 (+, CH_{trans}), 70.2 (–, $OCH_{2,cis}$), 69.5 (–, $OCH_{2,trans}$), 55.9 (+, $OCH_{3,cis}$), 55.4 (+, $OCH_{3,trans}$), 29.1 (–, 2 × $CH_{2,cis}$), 28.7 (–, 2 × $CH_{2,cis}$), 27.5 (–, 2 × $CH_{2,trans}$), 27.1 (–, 2 × $CH_{2,trans}$) ppm.

((Cyclohex-3-en-1-yloxy)methyl)benzene (10)

Starting from 1-(Benzyloxy)-4-methoxycyclohexane (**9**) (50.0 mg, 0.227 mmol, 1.00 equiv.) the product were synthesized according to GP1. The crude was purified by column chromatography (cyclohexane/ethyl acetate = 10/1) yielding the product as colorless liquid (15.0 mg, 79.7 µmol, 35%, 73% brsm). The NMR spectrum of the reisolated starting material (ratio *cis/trans* 1:1) showed that only the *trans*-isomer had reacted.



The analytical data were in accordance with literature values.[23]

R_f = 0.50 (cyclohexane/ethyl acetate = 10:1). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.41–7.27 (m, 5 H, 5 × $H_{arom.}$), 5.70–5.53 (m, 2 H, 2 × H_{alkene}), 4.66–4.50 (m, 2 H, OCH_2Ph), 3.71–3.60 (m, 1 H, 1-H), 2.48–2.32 (m, 1 H, CH_2), 2.28–1.91 (m, 4 H, CH_2), 1.77–1.59 (m, 1 H, CH_2) ppm. ^{13}C NMR (75.0 MHz, $CDCl_3$): δ = 139.1 (C_{quart}), 128.2 (+, 2 × $CH_{arom.}$), 127.5 (+, 2 × $CH_{arom.}$), 127.4 (+, $CH_{arom.}$), 126.9 (+, CH_{alkene}), 124.3 (+, CH_{alkene}), 73.8 (–, OCH_2Ph), 69.9 (+, C-1), 31.7 (–, CH_2), 27.9 (–, CH_2), 24.1 (–, CH_2) ppm.

Notes and references

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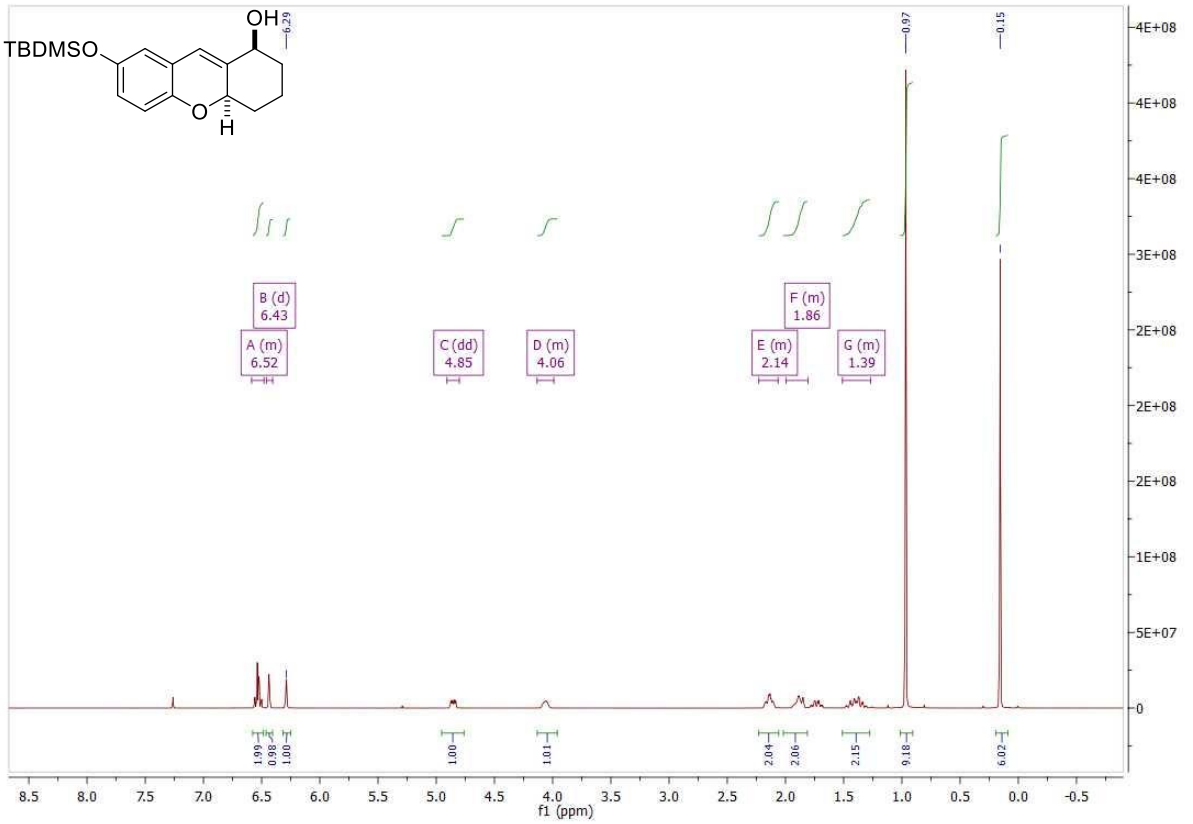
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D (m)
4.06
F (m)
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E (m)
2.14
G (m)
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2.06
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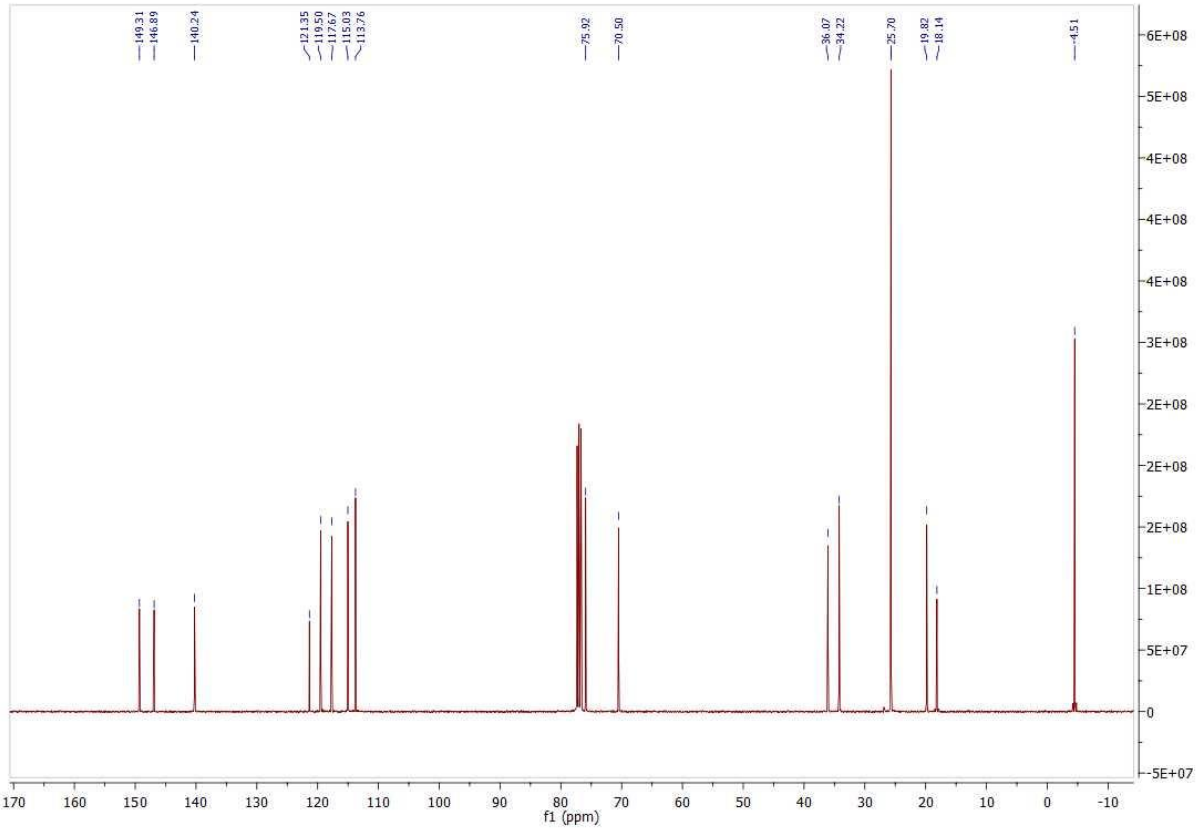
f1 (ppm)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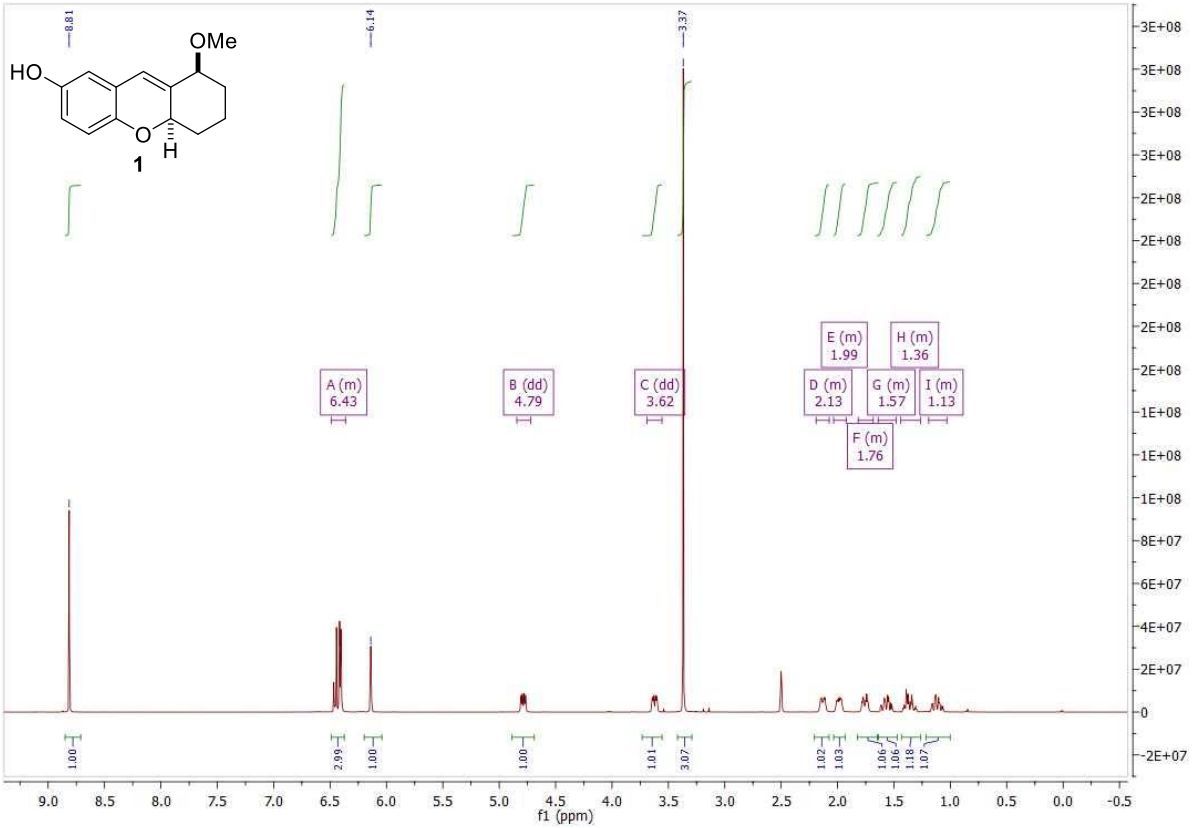
f1 (ppm)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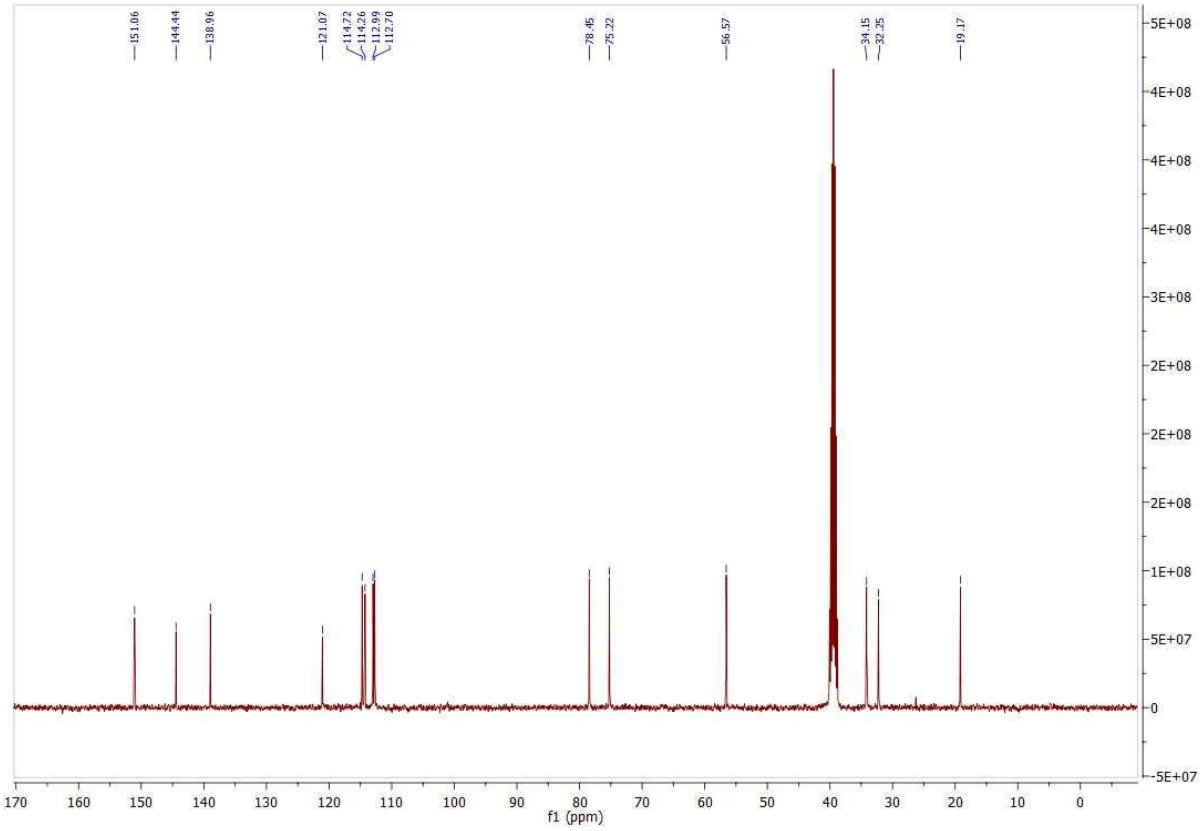
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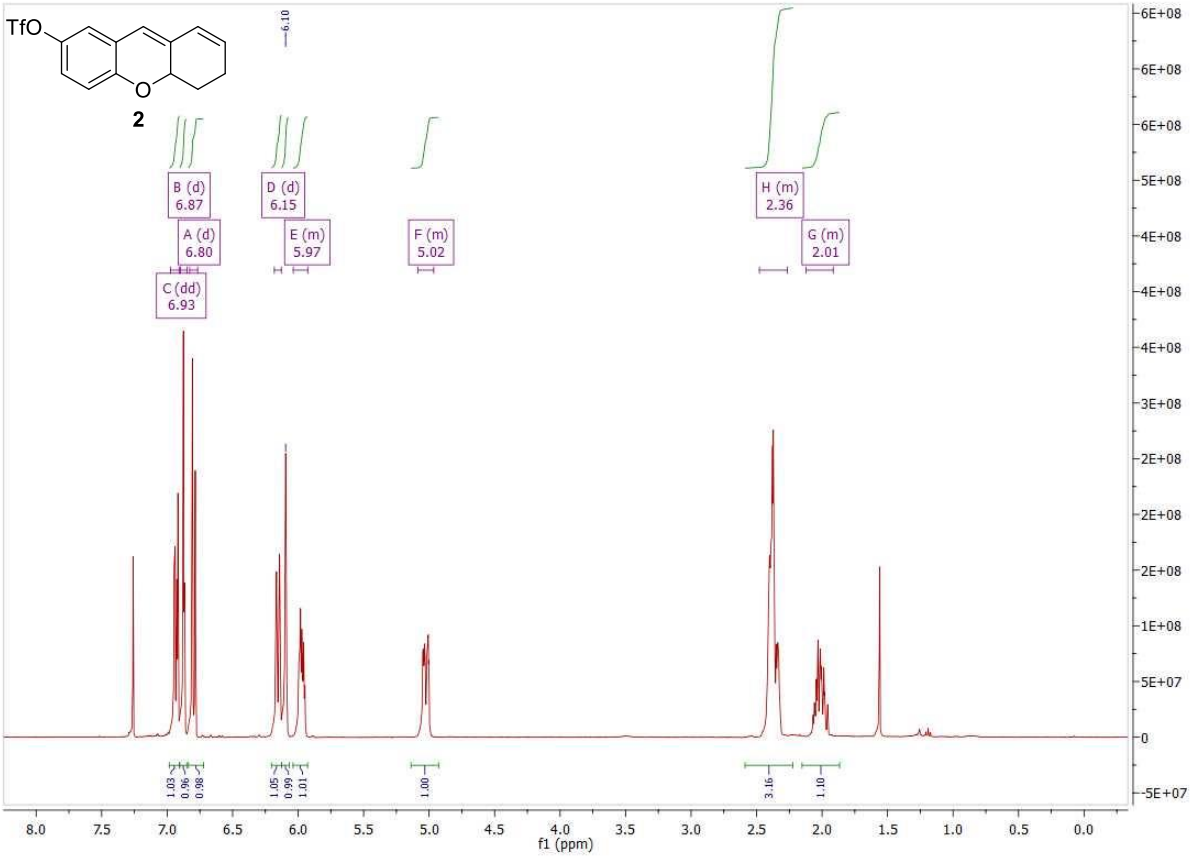
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D (d)
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5.97
F (m)
5.02
H (m)
2.36
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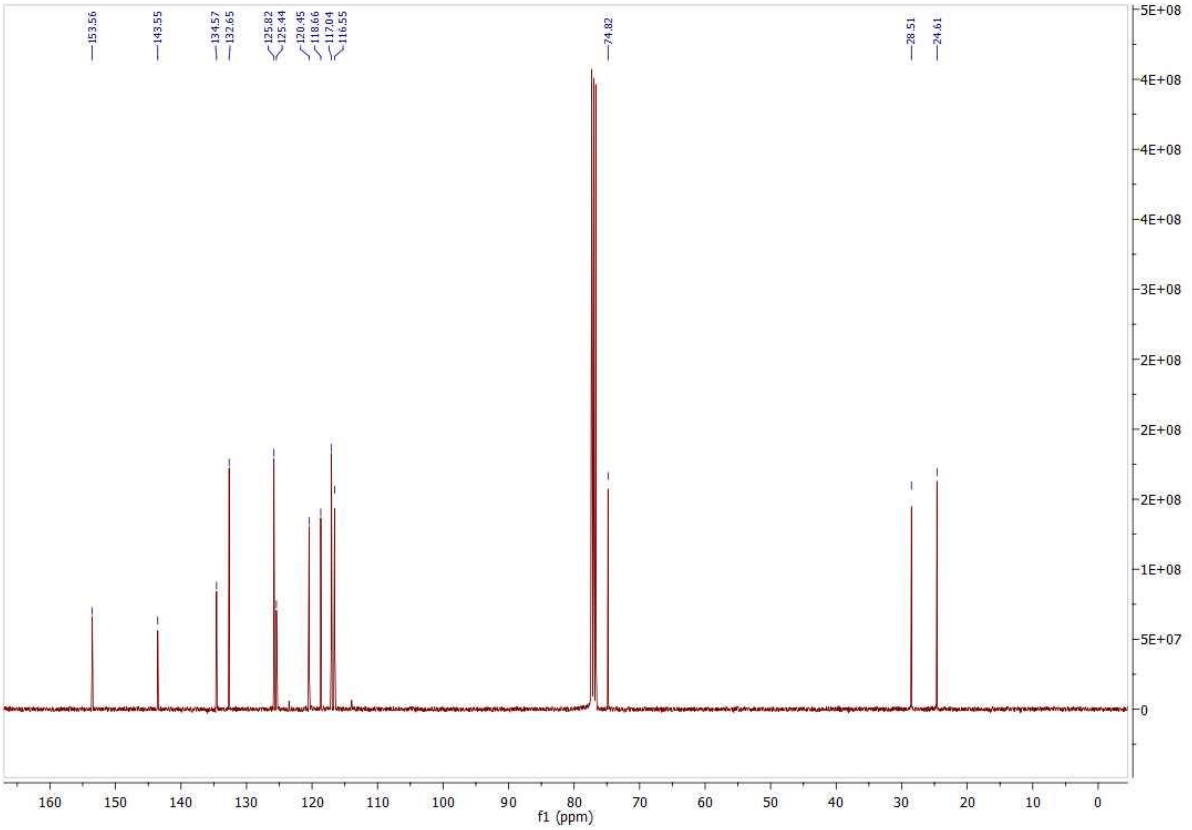
f1 (ppm)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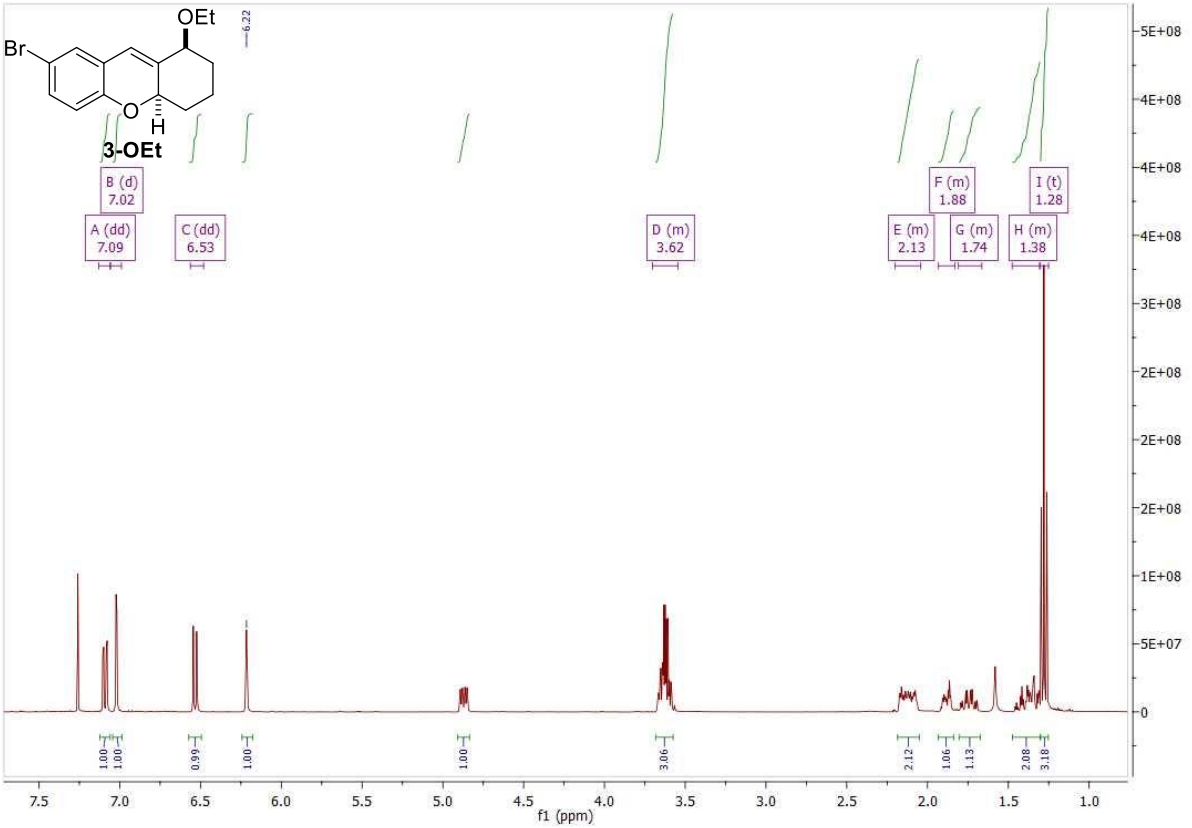
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2.12
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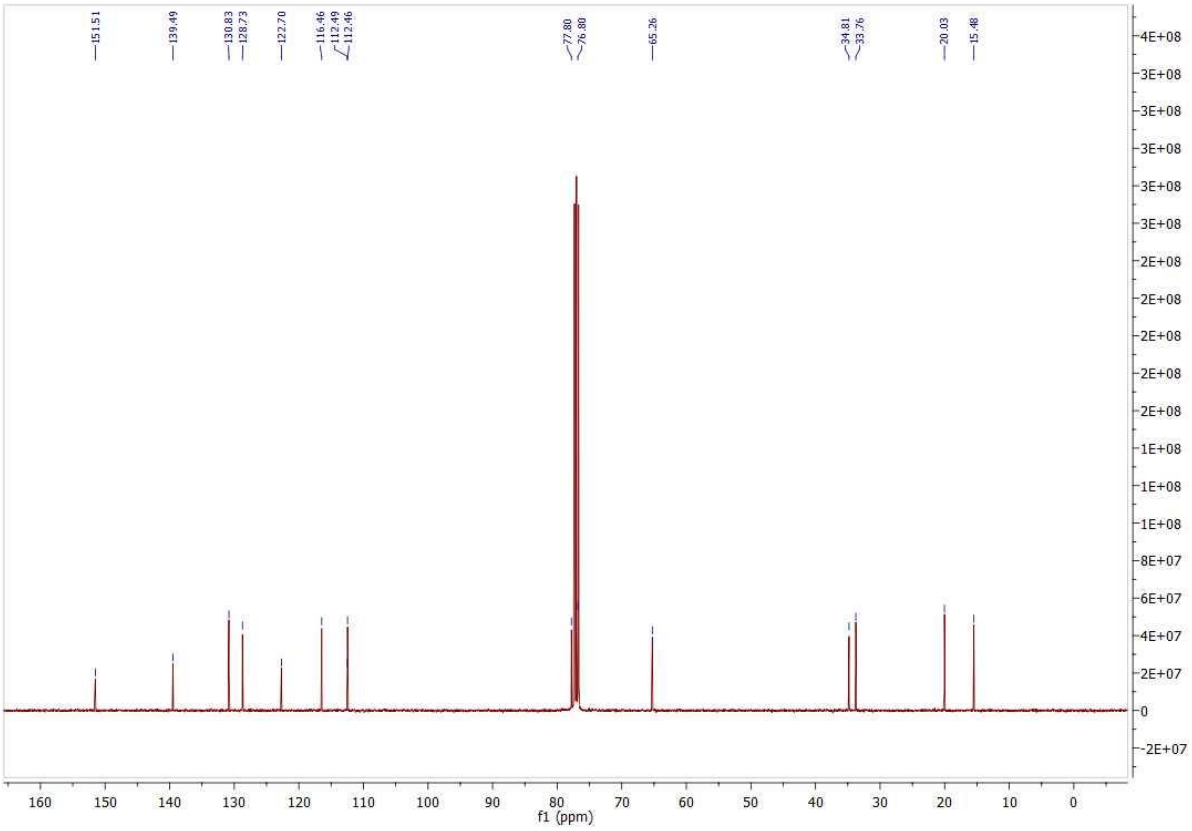
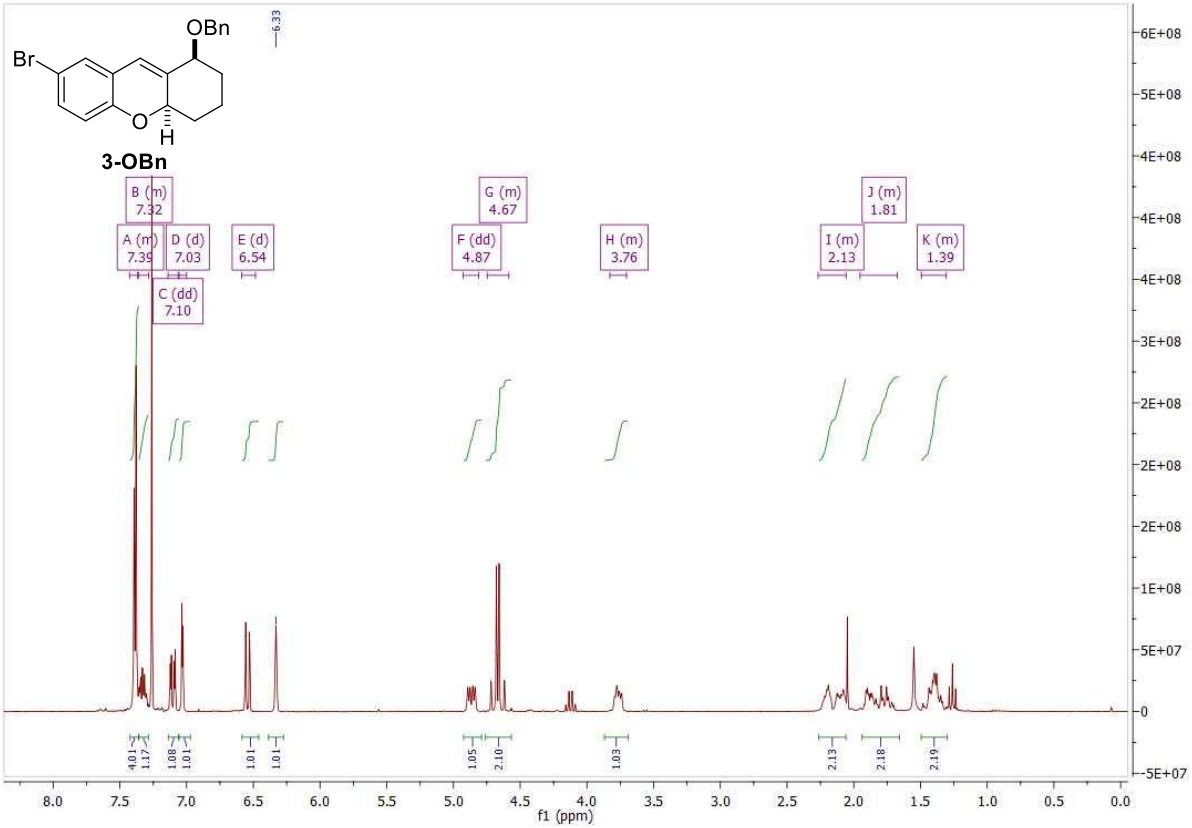
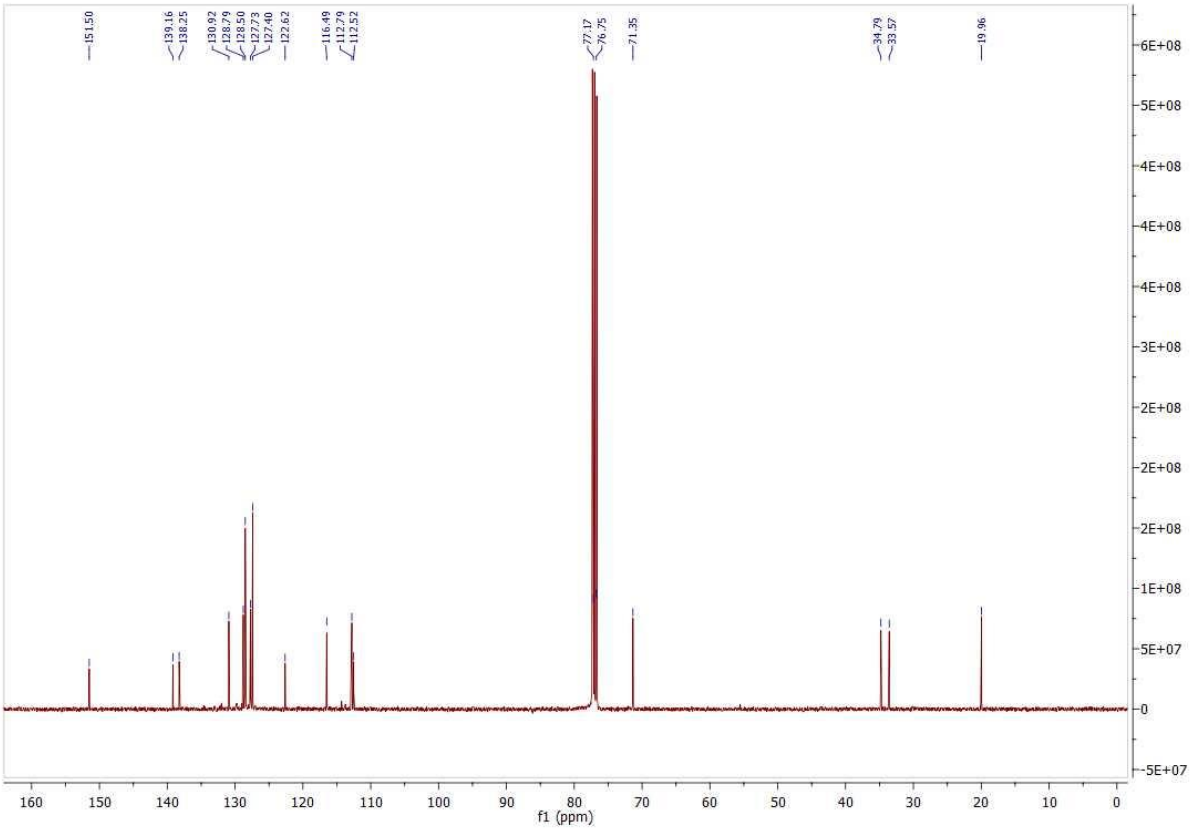
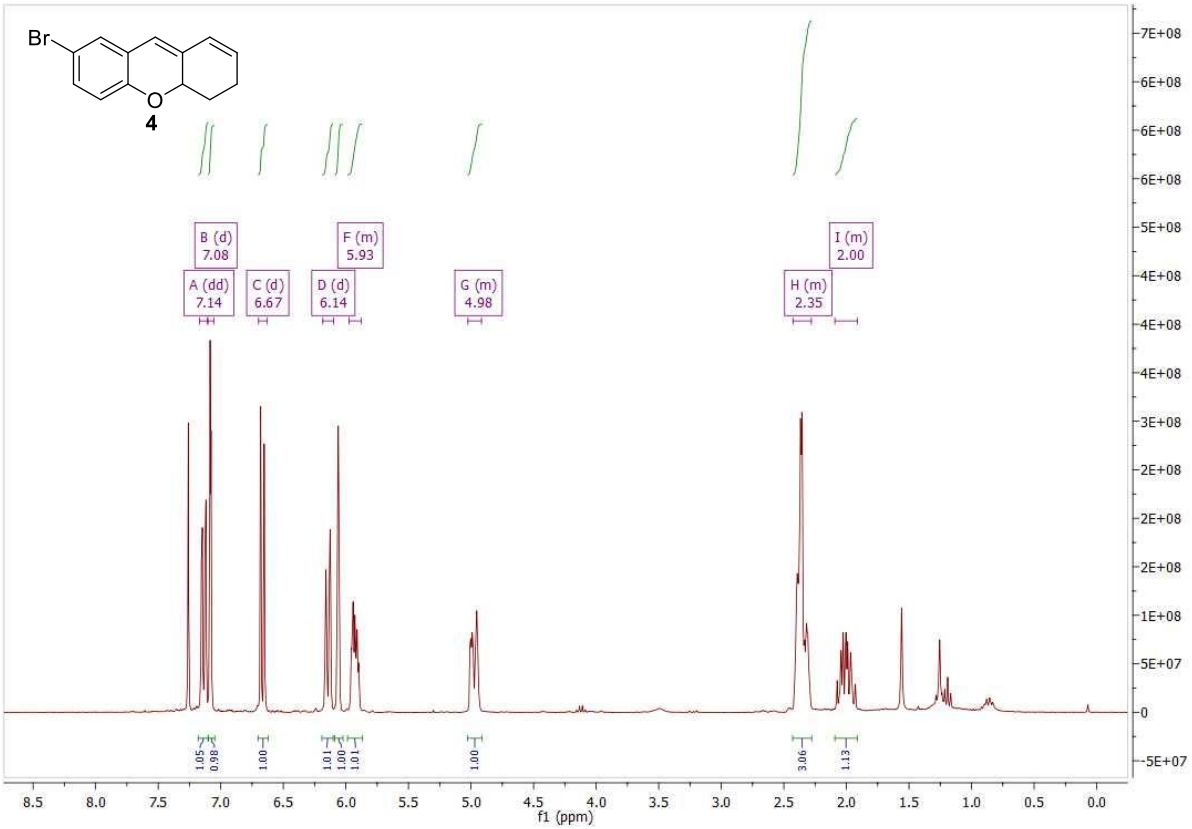
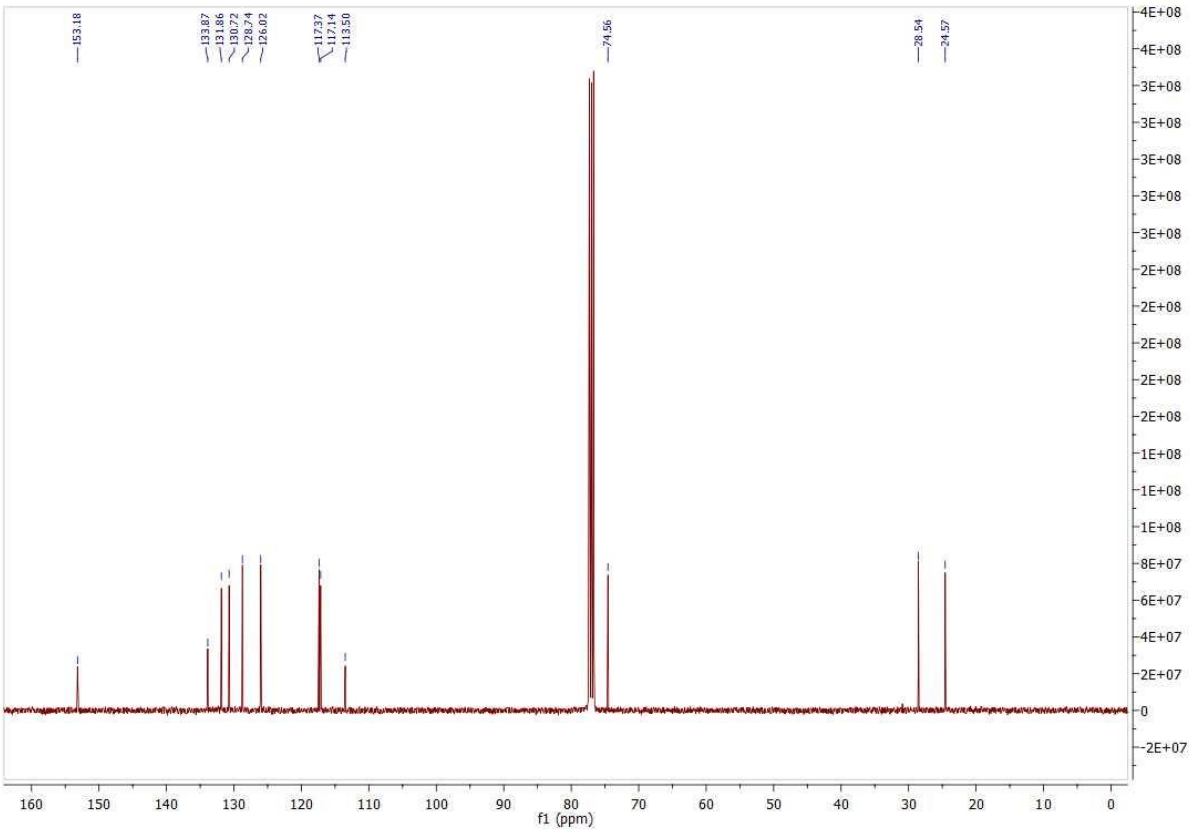
3.18
f1 (ppm)

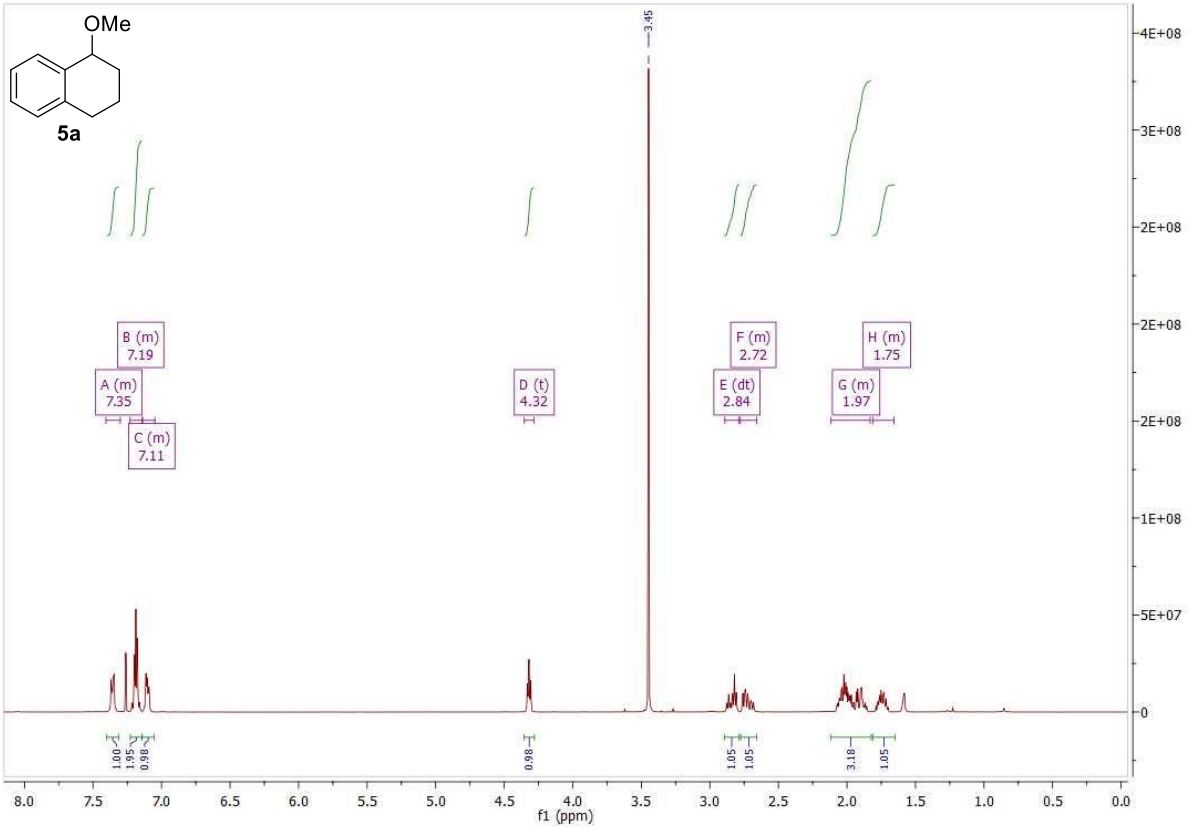

151.51
139.49
130.83
128.73
122.70
116.46
112.49
112.46
77.80
76.80
65.26
34.81
33.76
20.03
15.48
f1 (ppm)

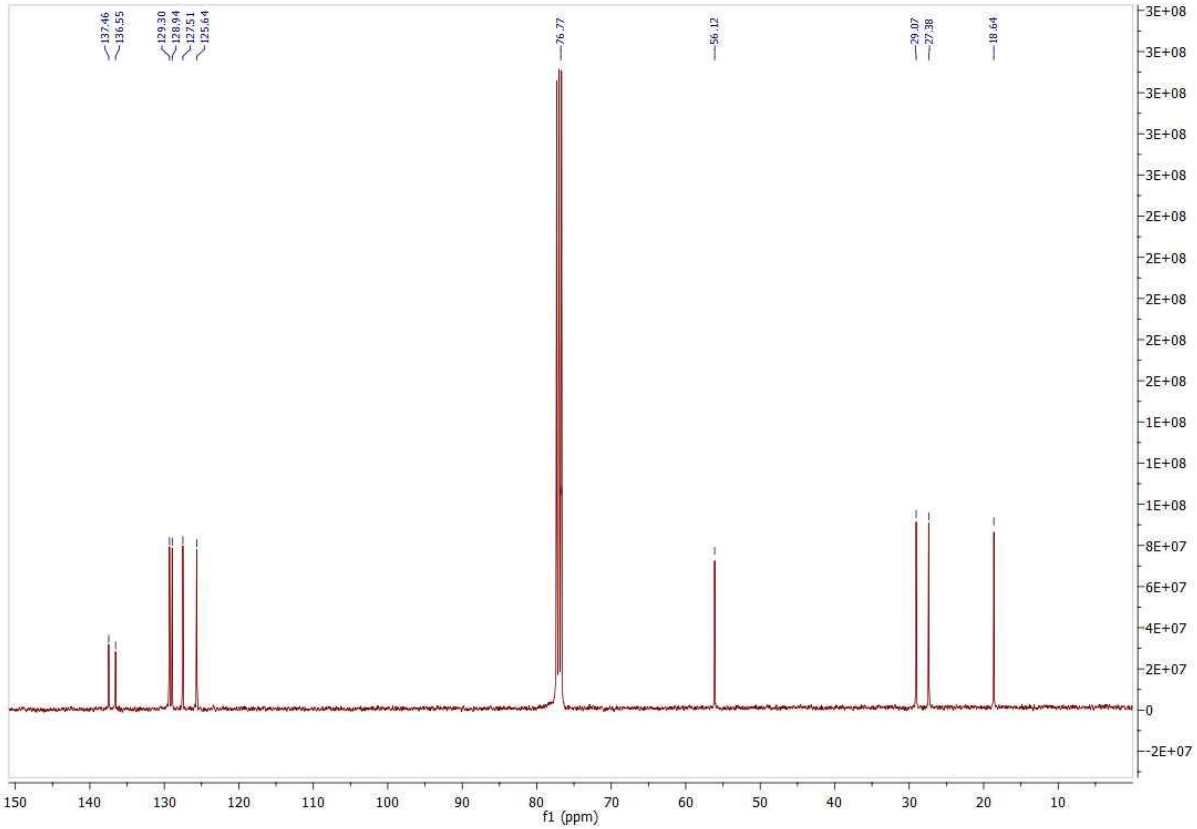

OBn
Br
O
H
3-OBn
6.33
B (m) 7.32
A (m) 7.39
D (d) 7.03
C (dd) 7.10
E (d) 6.54
G (m) 4.67
F (dd) 4.87
H (m) 3.76
J (m) 1.81
I (m) 2.13
K (m) 1.39
4.01
1.17
1.08
1.01
1.01
1.01
1.05
2.10
1.03
2.13
2.18
2.19
f1 (ppm)

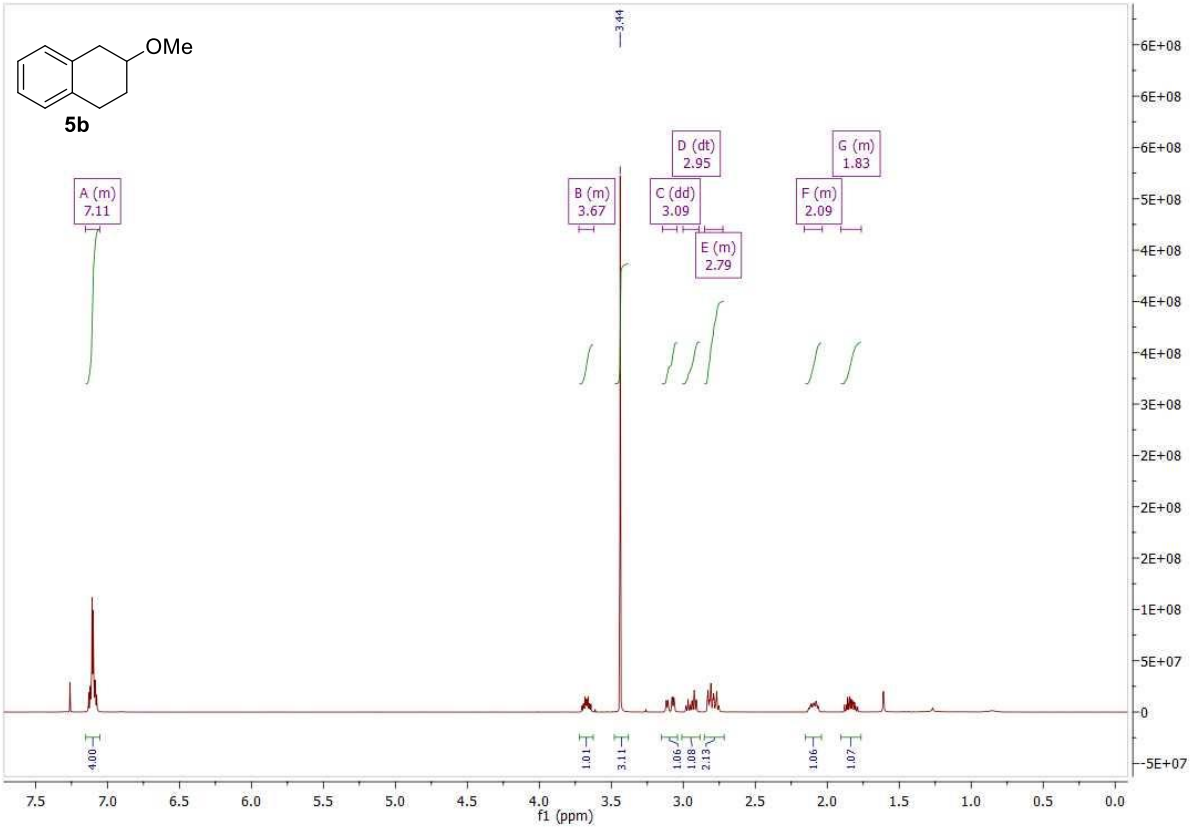

151.50
139.16
138.25
130.92
128.79
128.50
127.73
127.40
122.62
116.49
112.79
112.52
77.17
76.75
71.35
34.79
33.57
19.96
f1 (ppm)

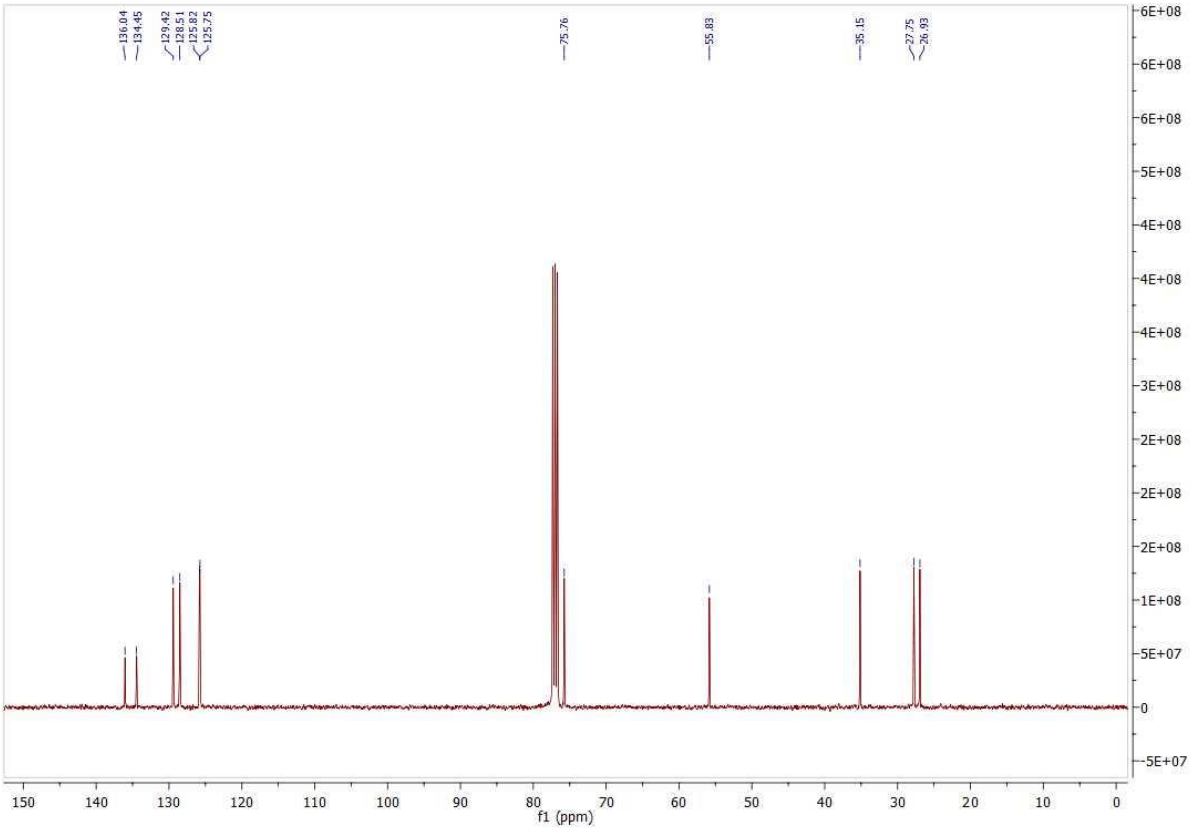

Br
O
4
B (d)
7.08
A (dd)
7.14
C (d)
6.67
F (m)
5.93
D (d)
6.14
G (m)
4.98
I (m)
2.00
H (m)
2.35
1.05
0.98
1.00
1.01
1.00
1.01
1.00
3.06
1.13
f1 (ppm)

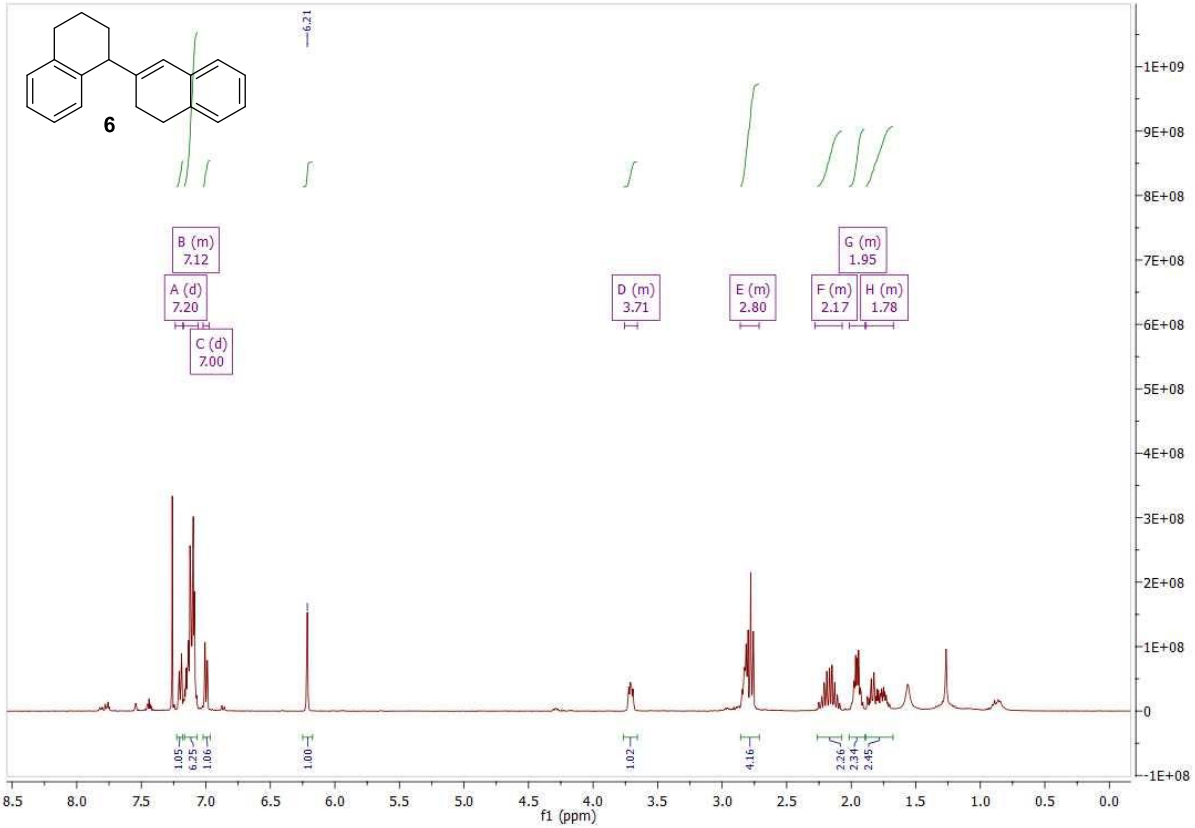

153.18
133.87
131.86
130.72
128.74
126.02
117.37
117.14
113.50
74.56
28.54
24.57
f1 (ppm)

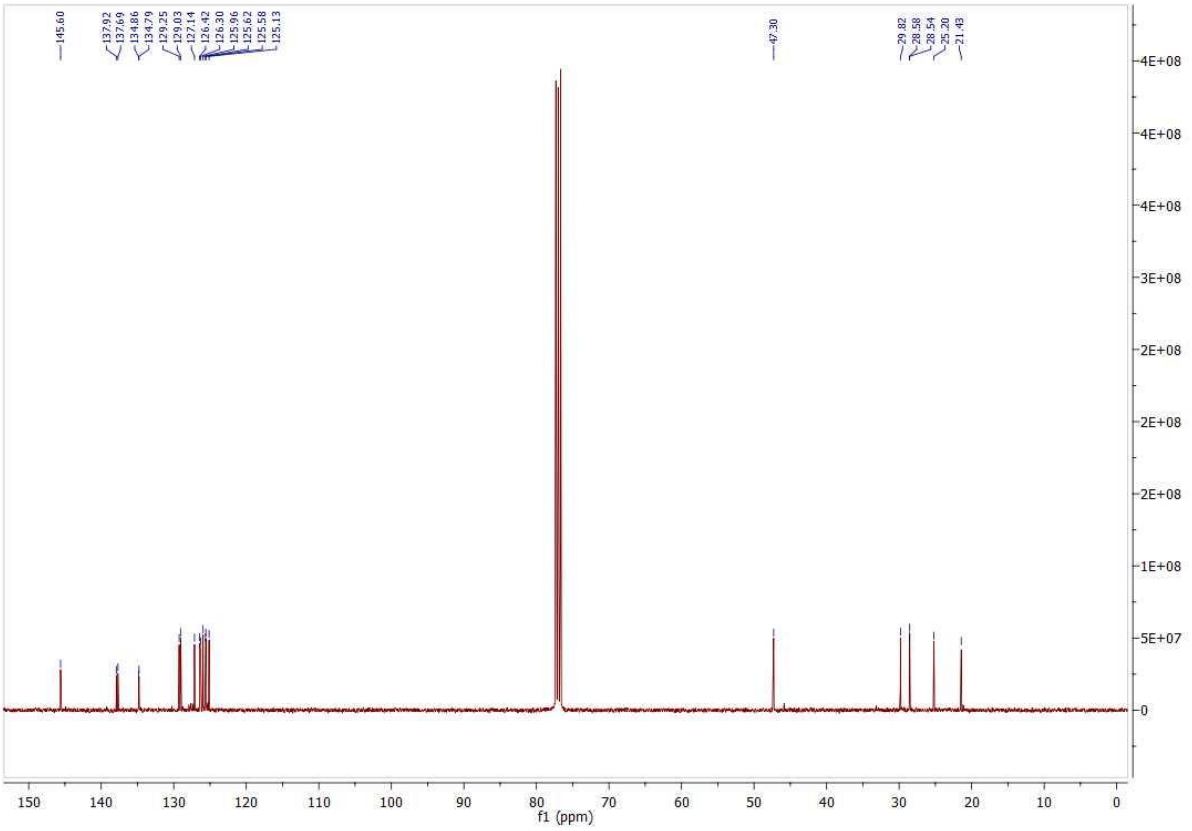

OMe
5a
B (m) 7.19
A (m) 7.35
C (m) 7.11
D (t) 4.32
F (m) 2.72
E (dt) 2.84
H (m) 1.75
G (m) 1.97
3.45
f1 (ppm)

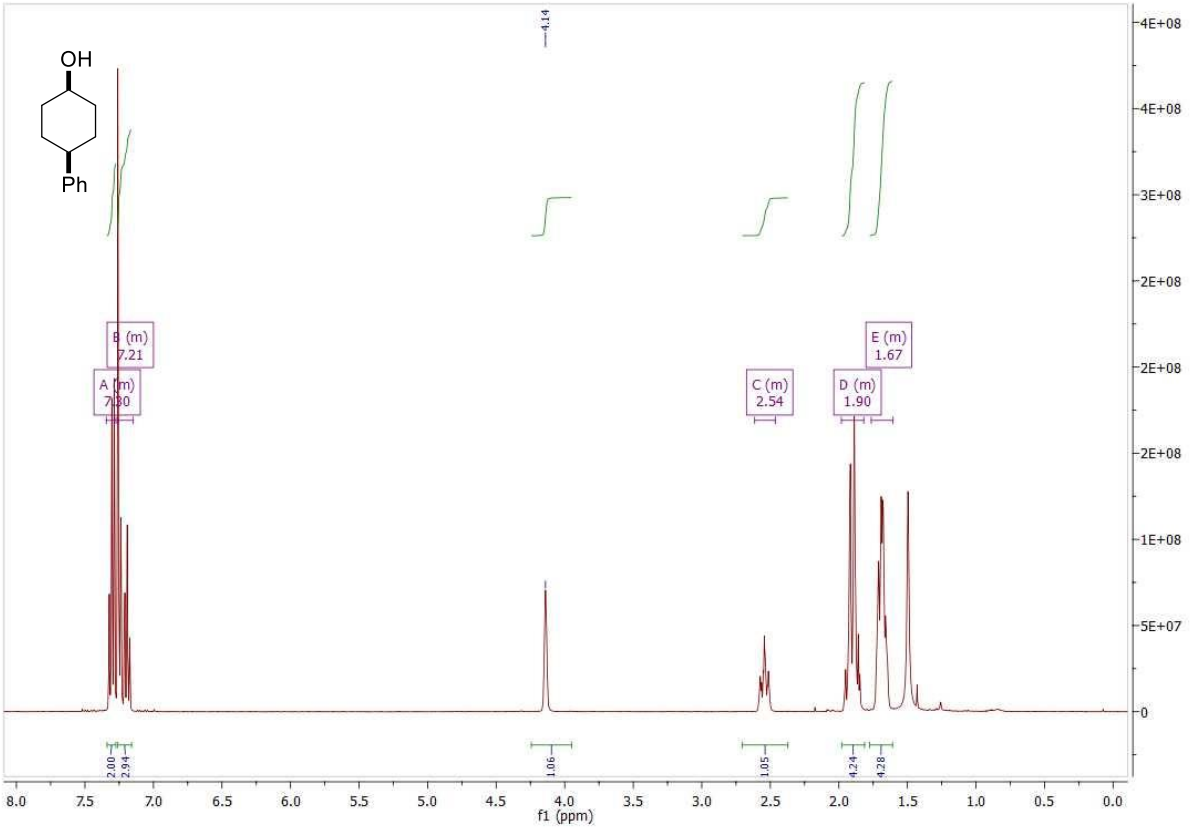

137.46
136.55
129.30
128.94
127.51
125.64
76.77
56.12
29.07
27.38
18.64
f1 (ppm)

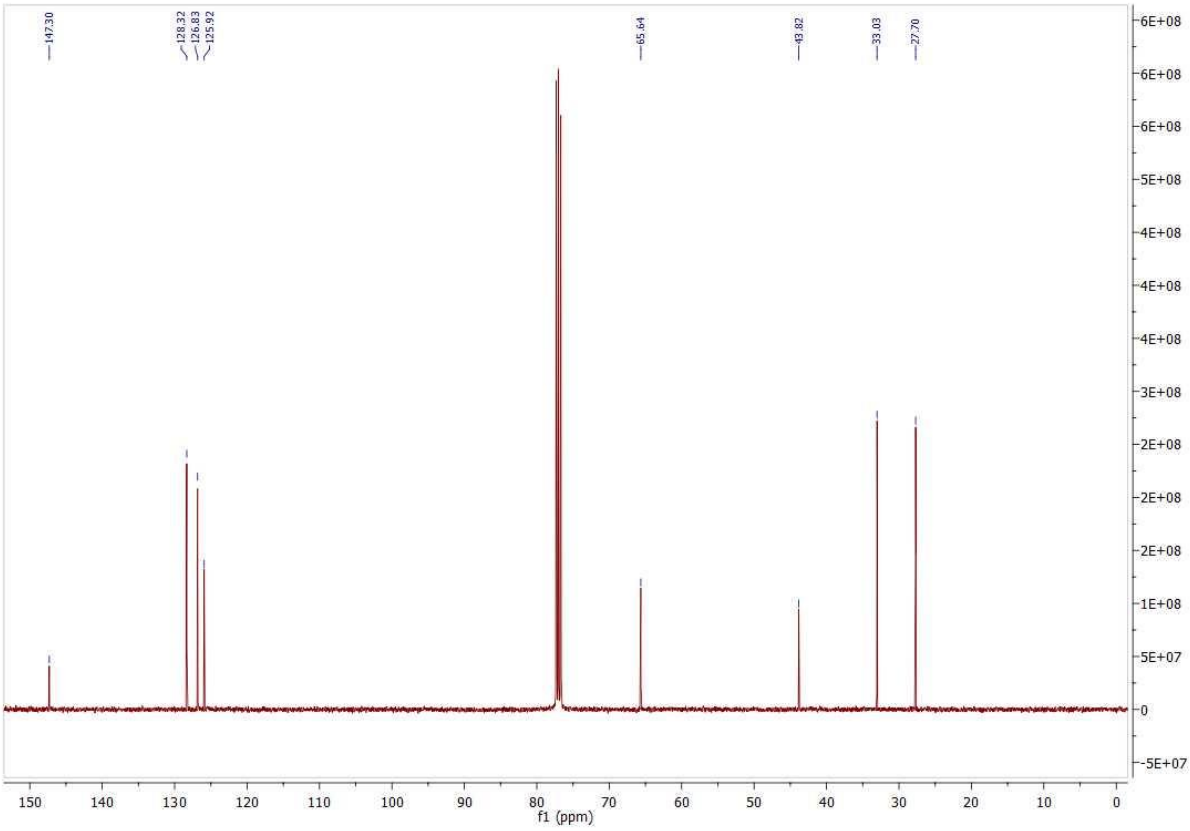

OMe
5b
3.44
A (m)
7.11
B (m)
3.67
C (dd)
3.09
D (dt)
2.95
E (m)
2.79
F (m)
2.09
G (m)
1.83
4.00
1.01
3.11
1.06
1.08
2.13
1.06
1.07
f1 (ppm)

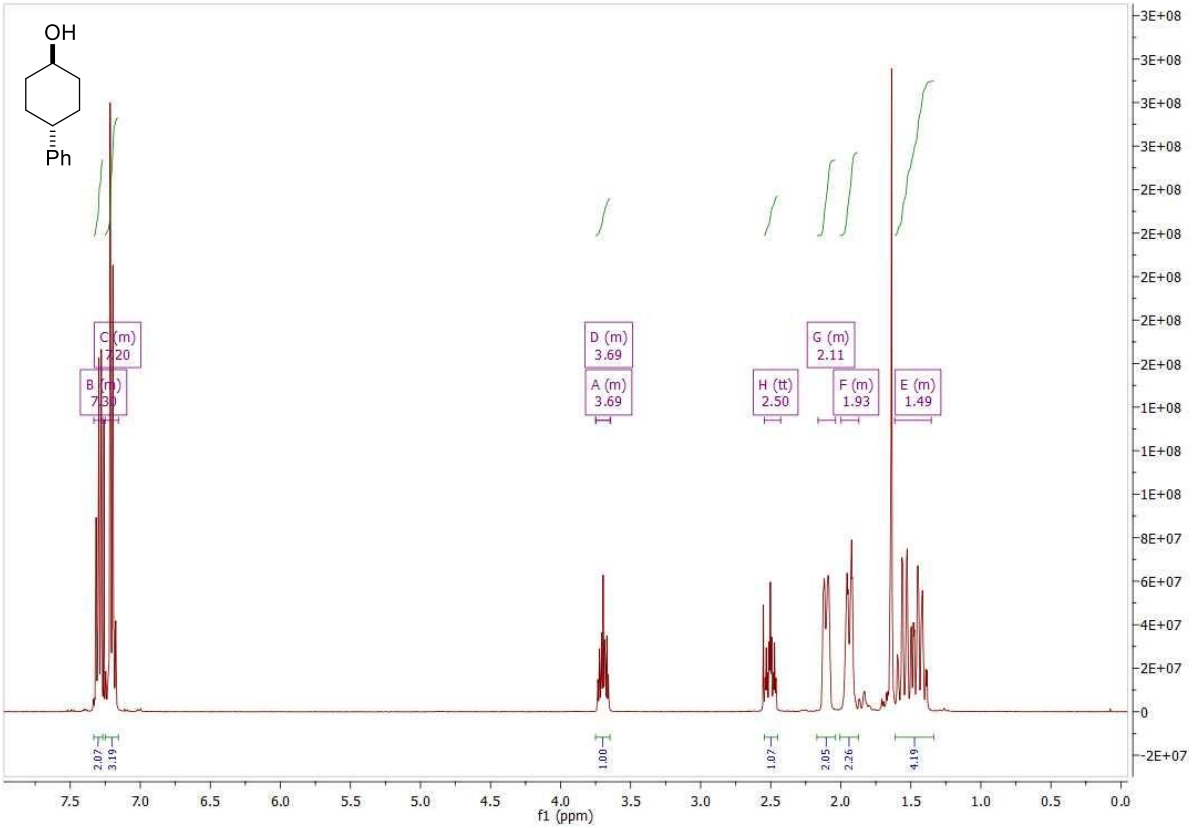

136.04
134.45
129.42
128.51
125.82
125.75
75.76
55.83
35.15
27.75
26.93
f1 (ppm)

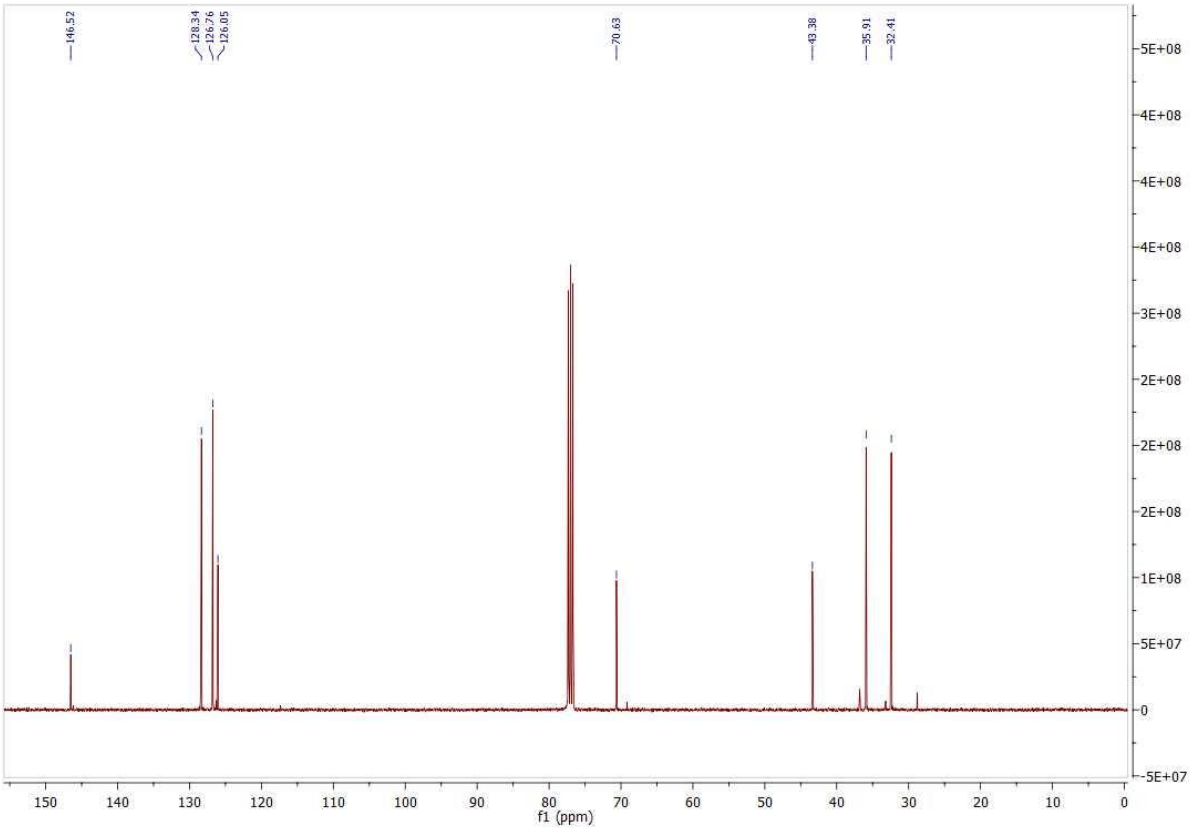

6
6.21
B (m)
7.12
A (d)
7.20
C (d)
7.00
D (m)
3.71
E (m)
2.80
G (m)
1.95
F (m)
2.17
H (m)
1.78
1.05
6.25
1.06
1.00
1.02
4.16
2.26
2.34
2.45
f1 (ppm)

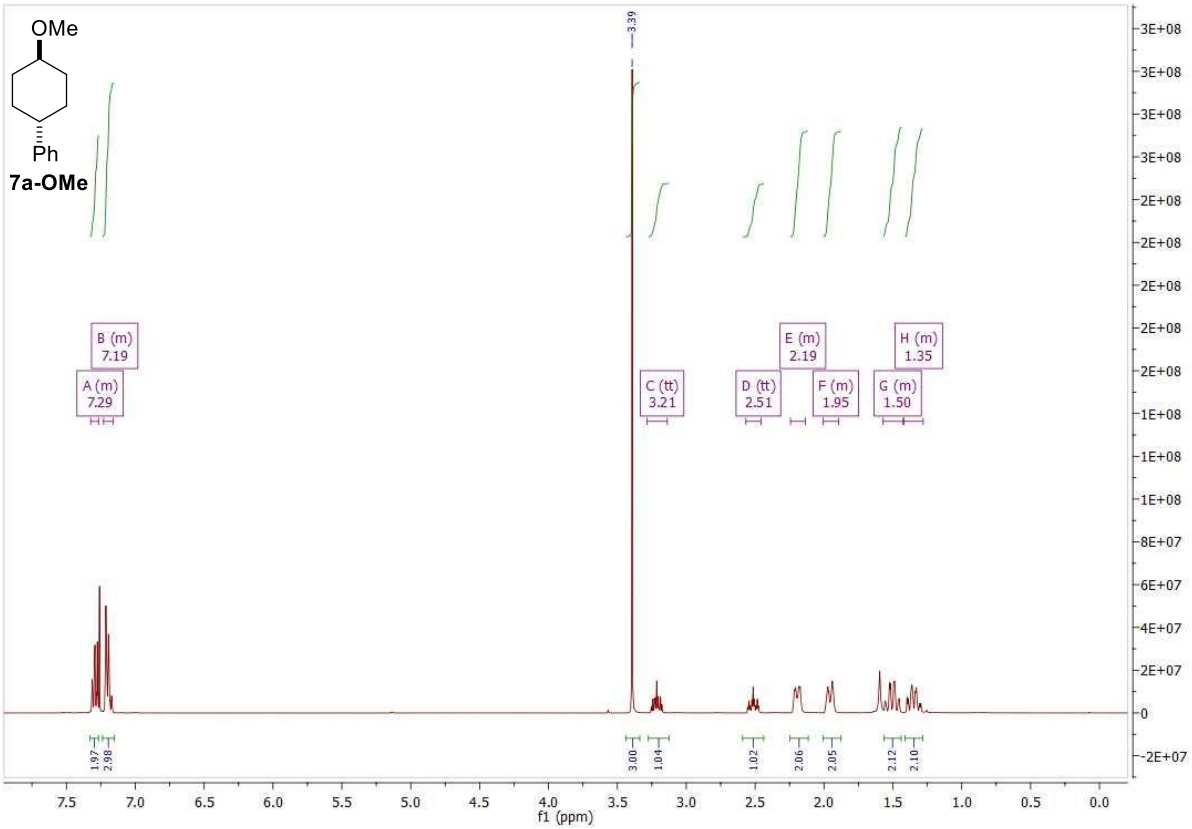

145.60
137.92
137.69
134.86
134.79
129.25
129.03
127.14
126.42
126.30
125.96
125.62
125.58
125.13
47.30
29.82
28.58
28.54
25.20
21.43
f1 (ppm)

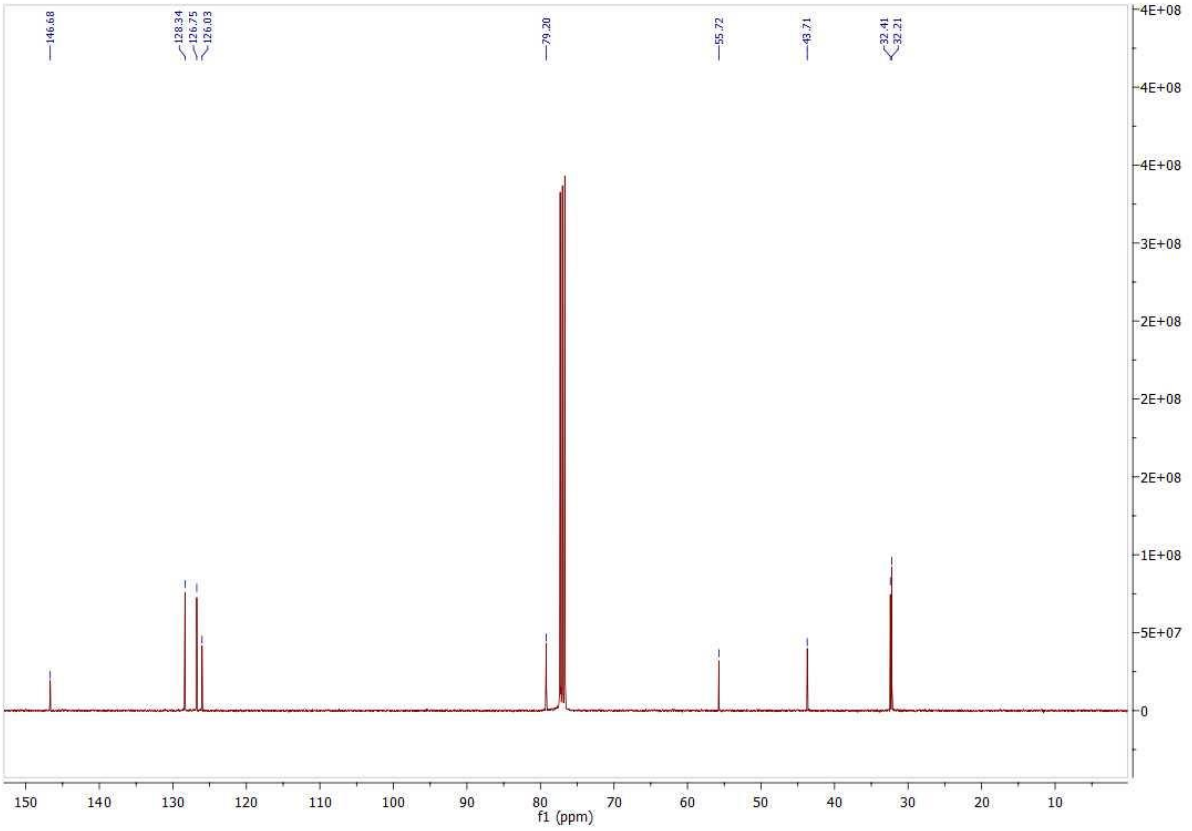

OH
Ph
B (m)
7.21
A (m)
7.30
C (m)
2.54
D (m)
1.90
E (m)
1.67
4.14
2.00
2.94
1.06
1.05
4.24
4.28
f1 (ppm)

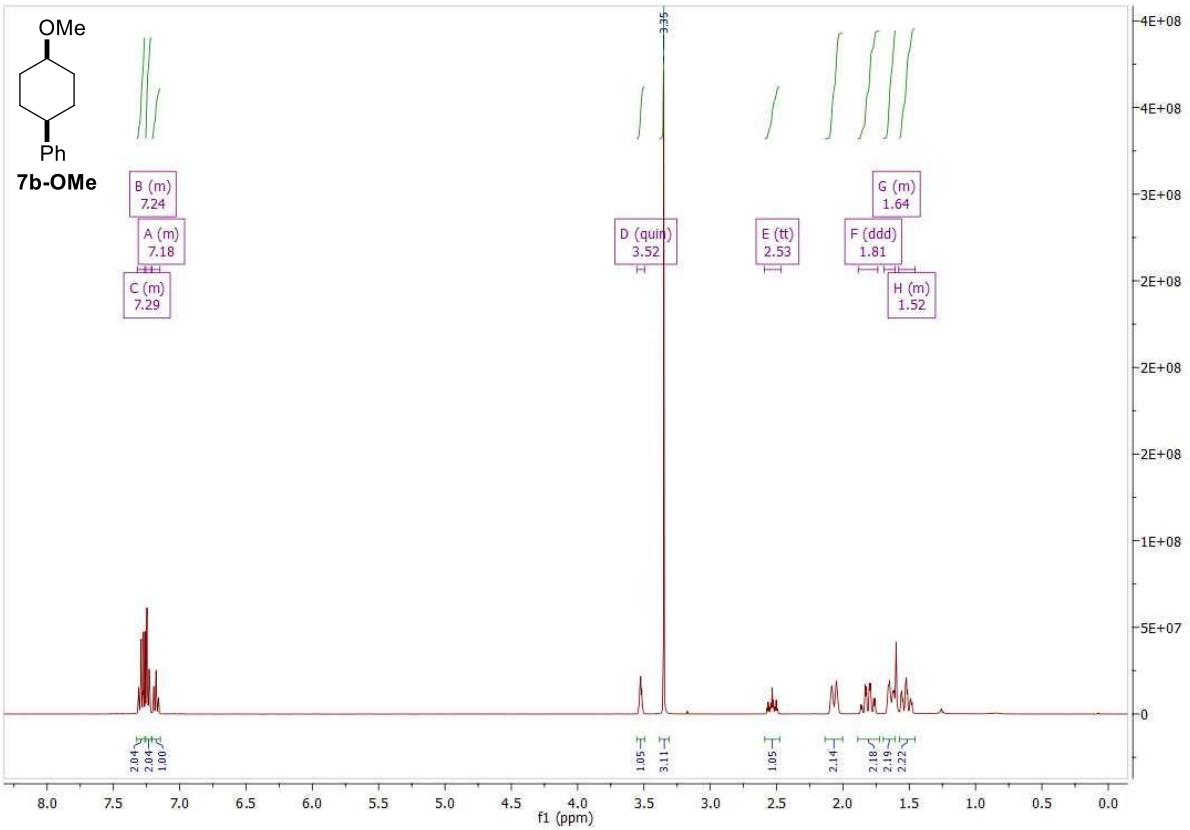

147.30
128.32
126.83
125.92
65.64
43.82
33.03
27.70
f1 (ppm)

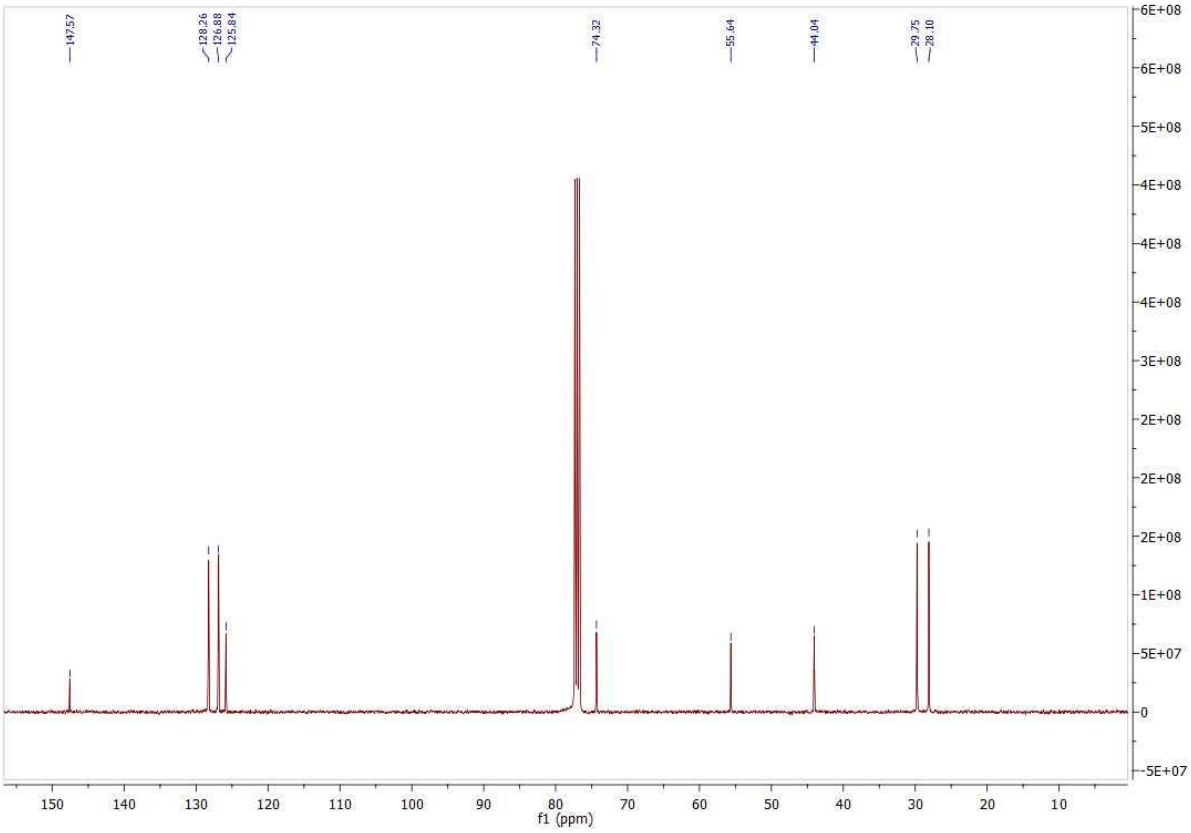

OH
Ph
C (m)
7.20
B (m)
7.30
D (m)
3.69
A (m)
3.69
H (tt)
2.50
G (m)
2.11
F (m)
1.93
E (m)
1.49
2.07
3.19
1.00
1.07
2.05
2.26
4.19
f1 (ppm)

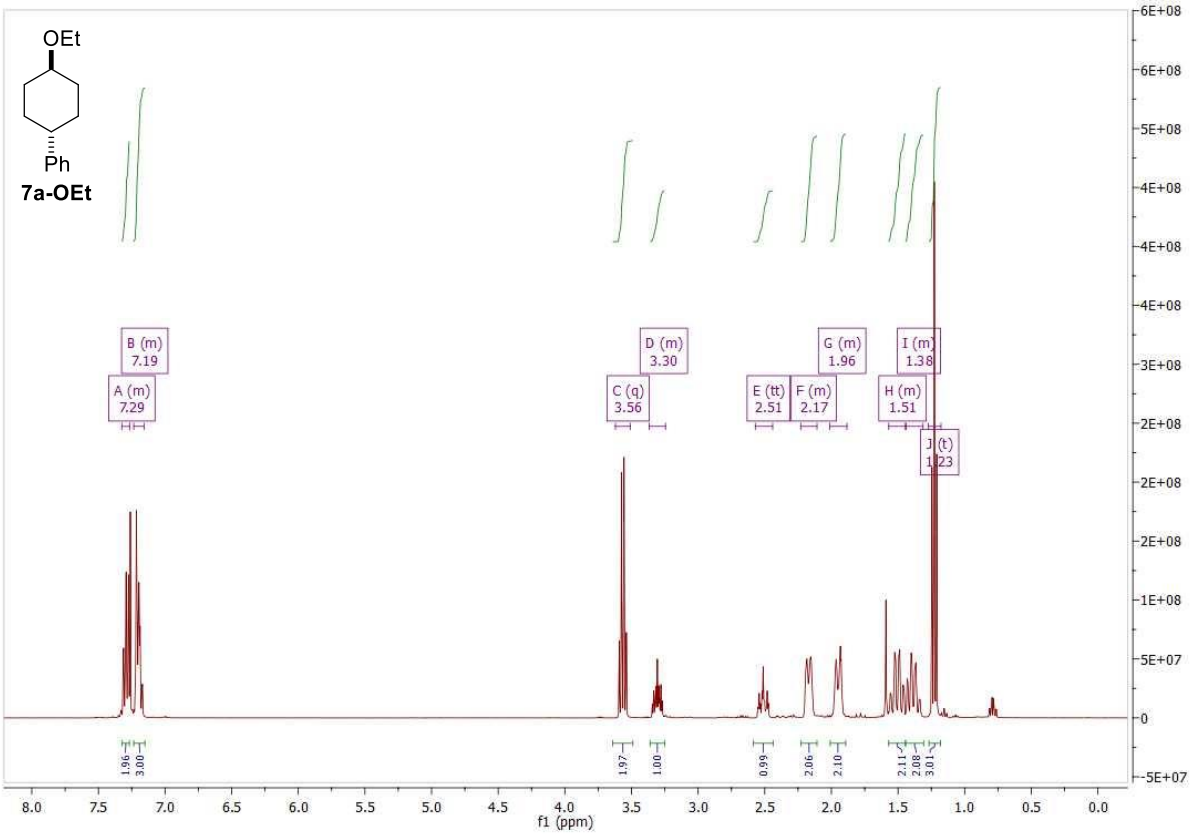

146.52
128.34
126.76
126.05
70.63
43.38
35.91
32.41
f1 (ppm)

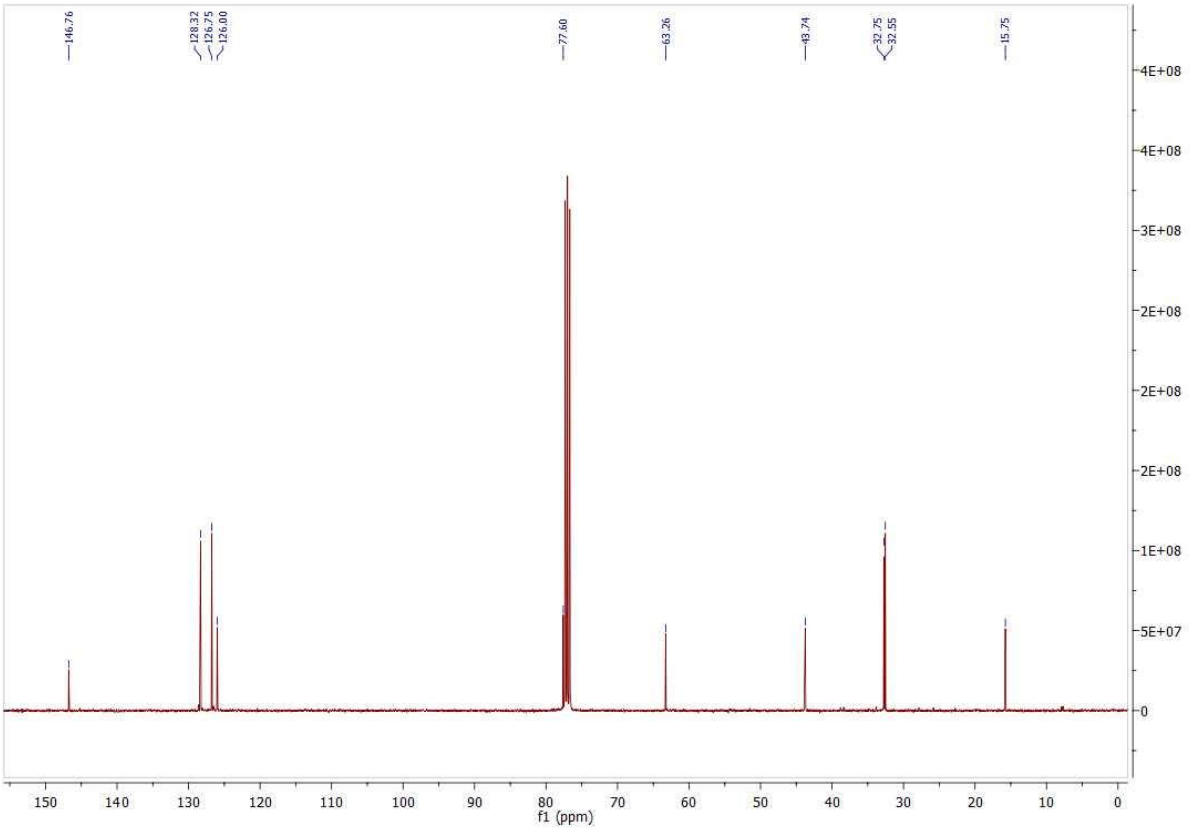

OMe
Ph
7a-OMe
3.39
B (m) 7.19
A (m) 7.29
C (tt) 3.21
D (tt) 2.51
E (m) 2.19
F (m) 1.95
G (m) 1.50
H (m) 1.35
1.97
2.98
3.00
1.04
1.02
2.06
2.05
2.12
2.10
f1 (ppm)

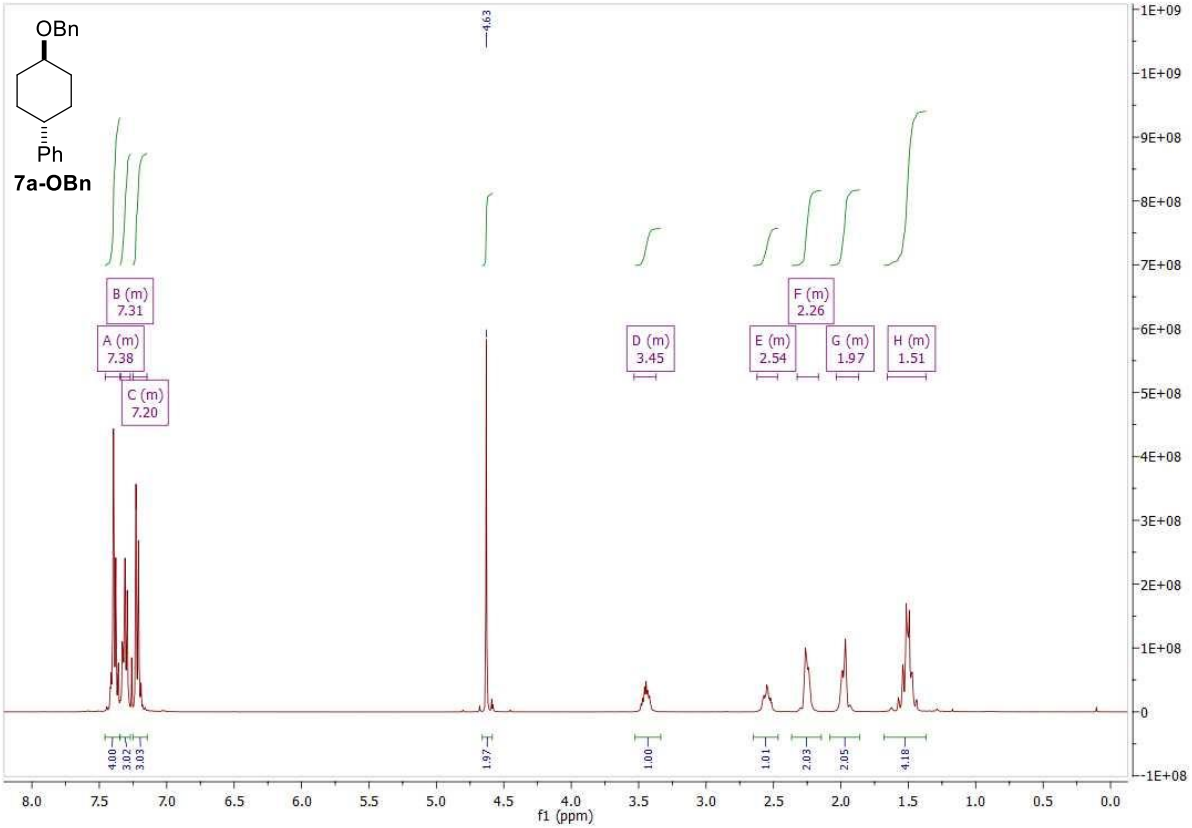

146.68
128.34
126.75
126.03
79.20
55.72
43.71
32.41
32.21
f1 (ppm)

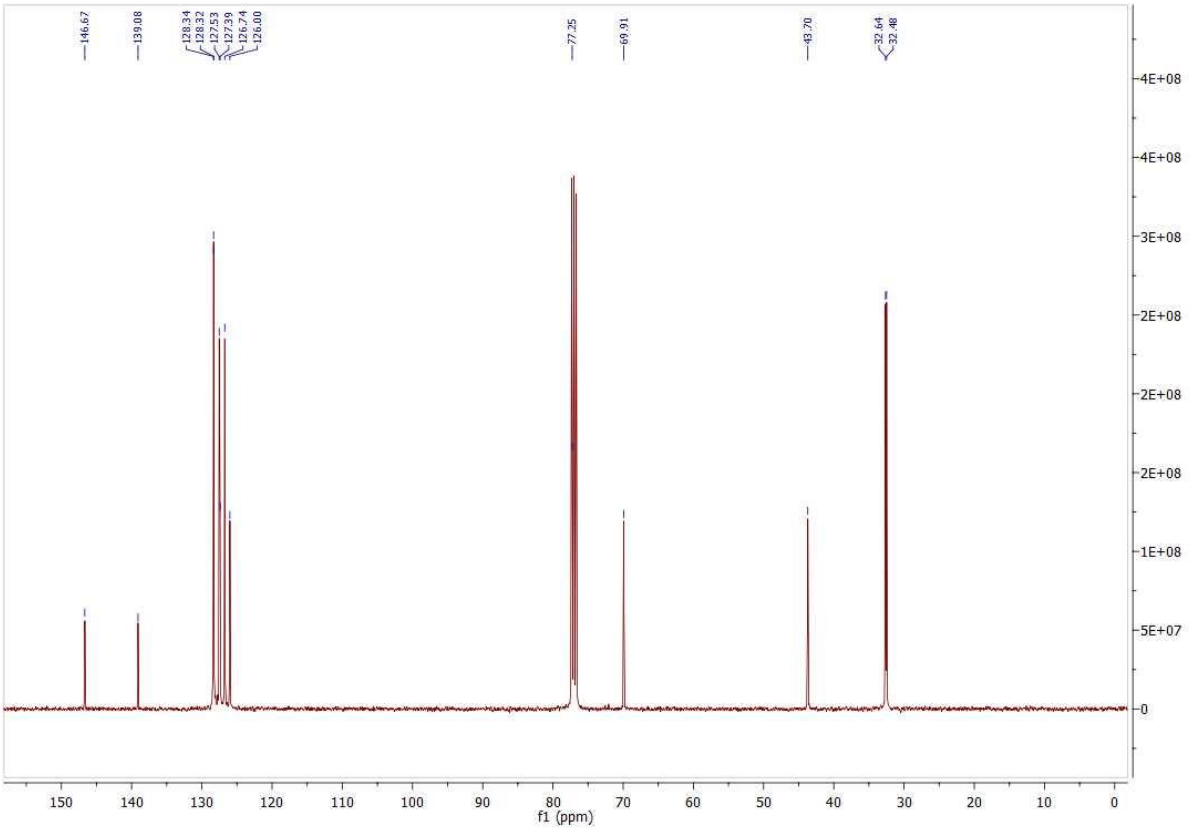

OMe
Ph
7b-OMe
B (m) 7.24
A (m) 7.18
C (m) 7.29
D (quin) 3.52
E (tt) 2.53
F (ddd) 1.81
G (m) 1.64
H (m) 1.52
f1 (ppm)

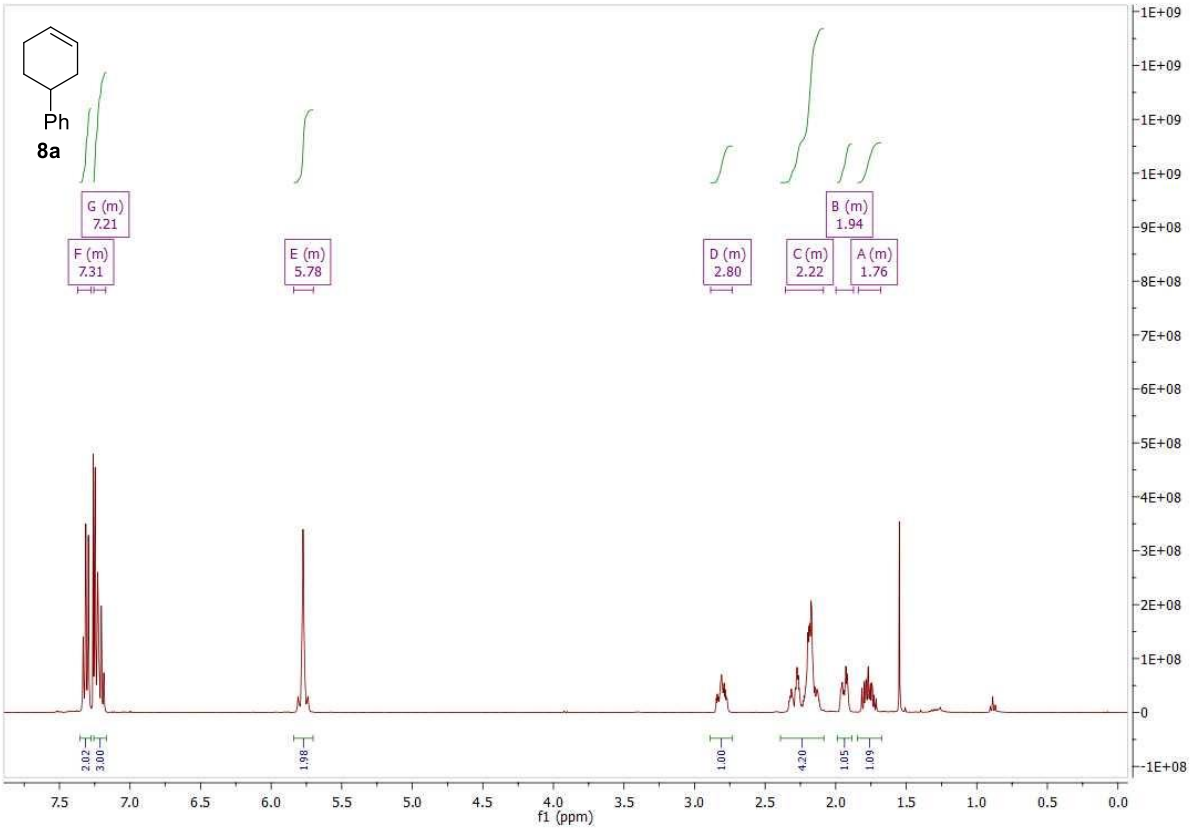

147.57
128.26
126.88
125.84
74.32
55.64
44.04
29.75
28.10
f1 (ppm)

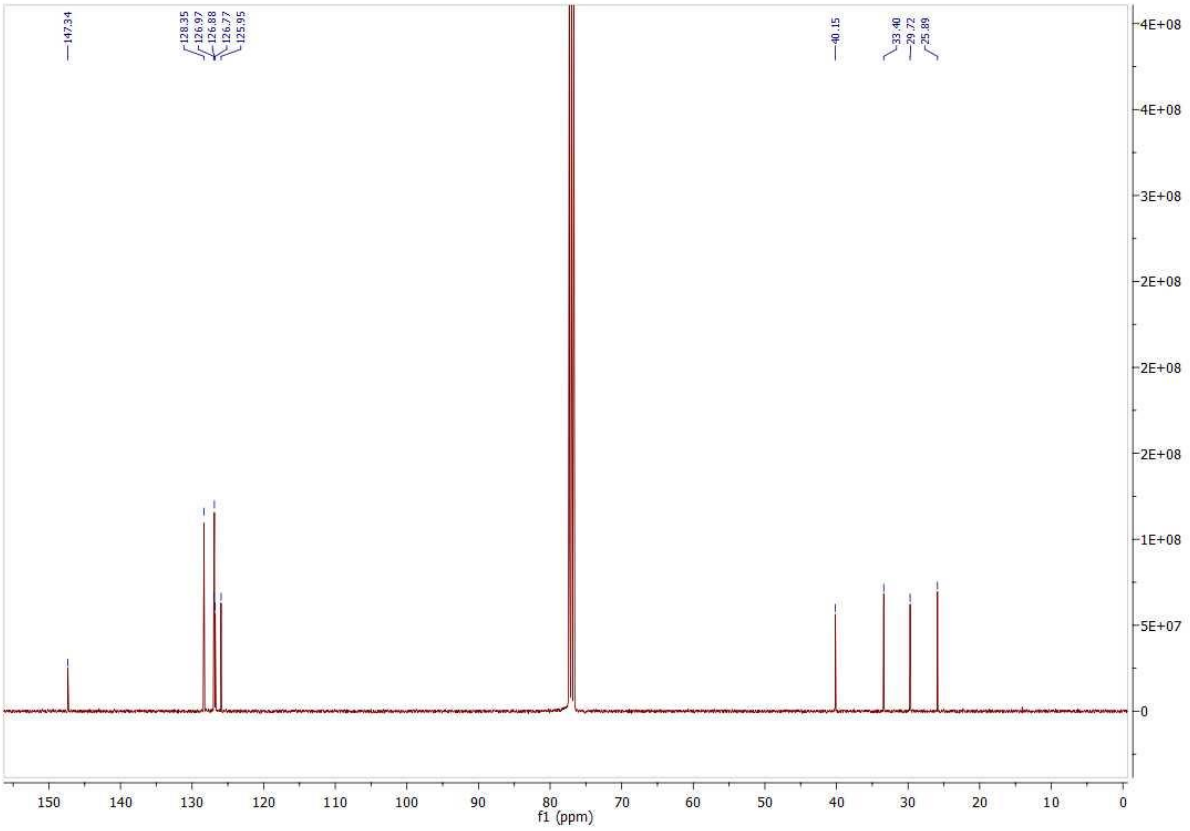

OEt
Ph
7a-OEt
B (m) 7.19
A (m) 7.29
D (m) 3.30
C (q) 3.56
E (tt) 2.51
F (m) 2.17
G (m) 1.96
H (m) 1.51
I (m) 1.38
J (t) 1.23
f1 (ppm)

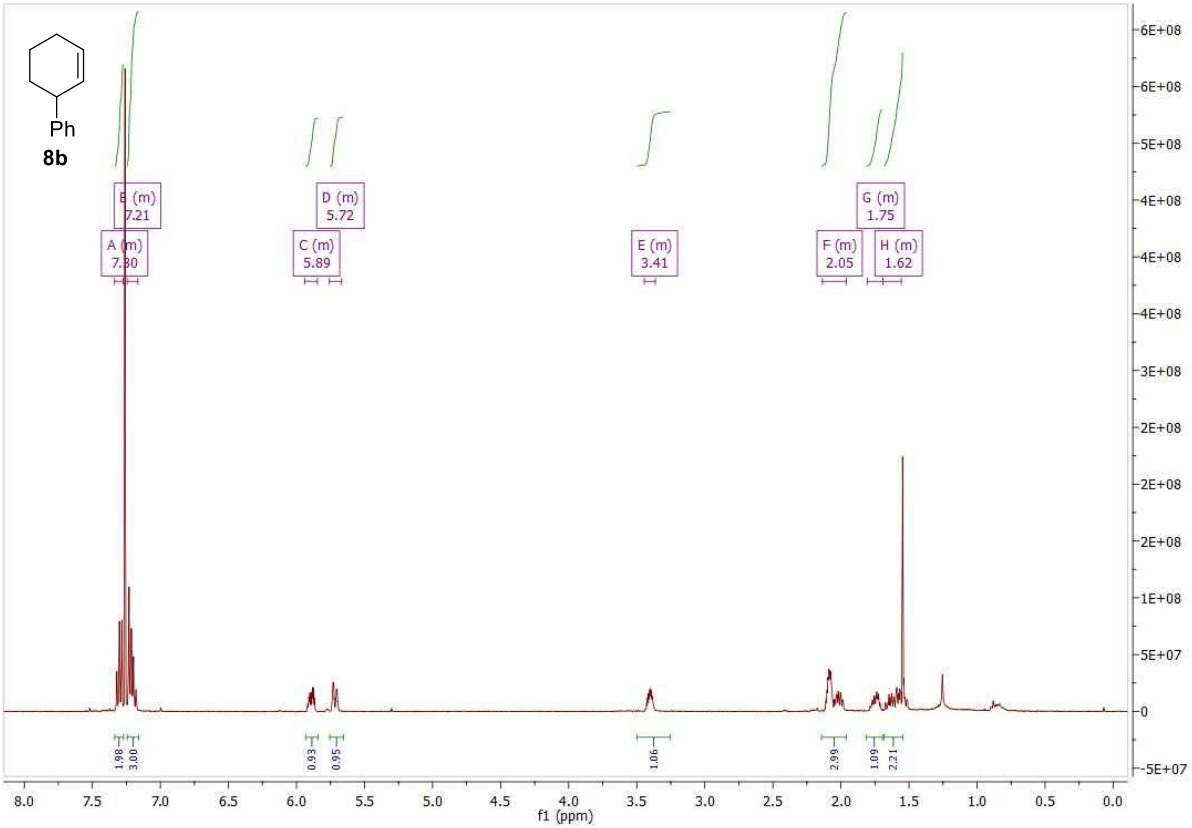

146.76
128.32
126.75
126.00
77.60
63.26
43.74
32.75
32.55
15.75
f1 (ppm)

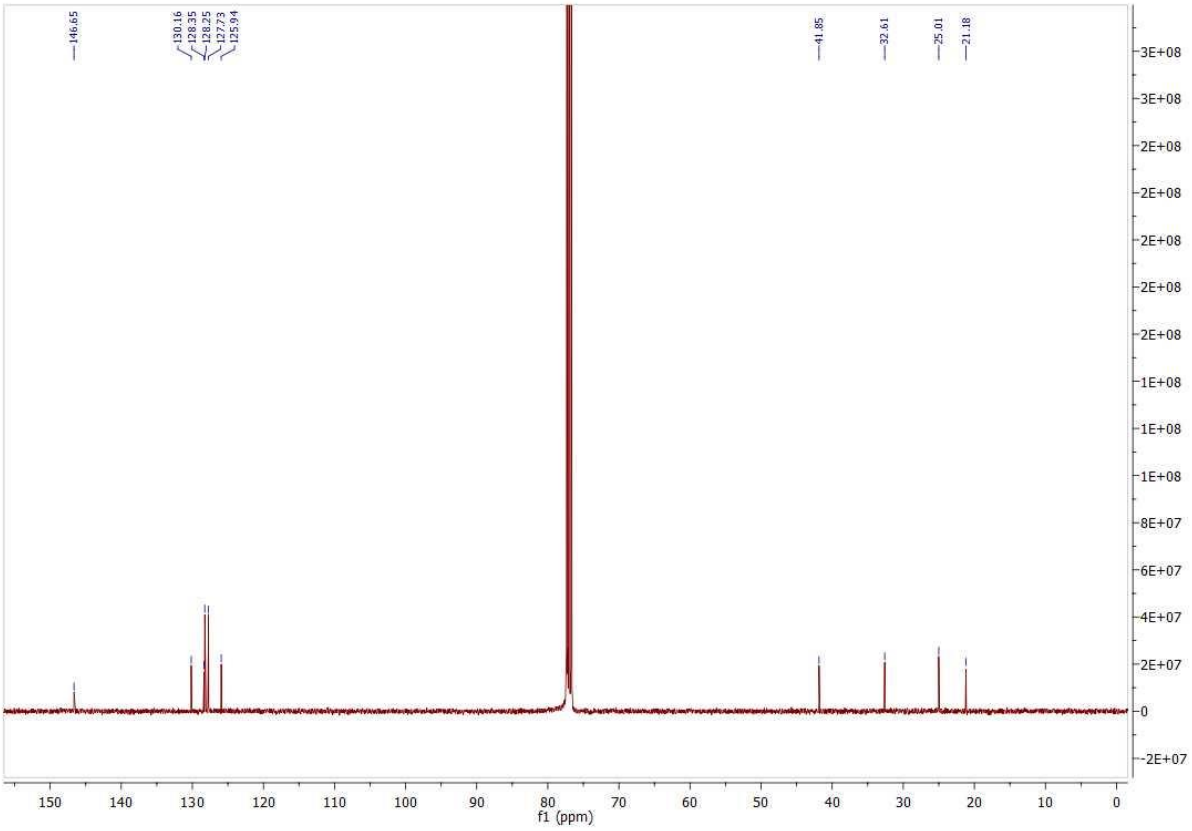

OBn
Ph
7a-OBn
4.63
B (m) 7.31
A (m) 7.38
C (m) 7.20
D (m) 3.45
F (m) 2.26
E (m) 2.54
G (m) 1.97
H (m) 1.51
4.00
3.02
3.03
1.97
1.00
1.01
2.03
2.05
4.18
f1 (ppm)

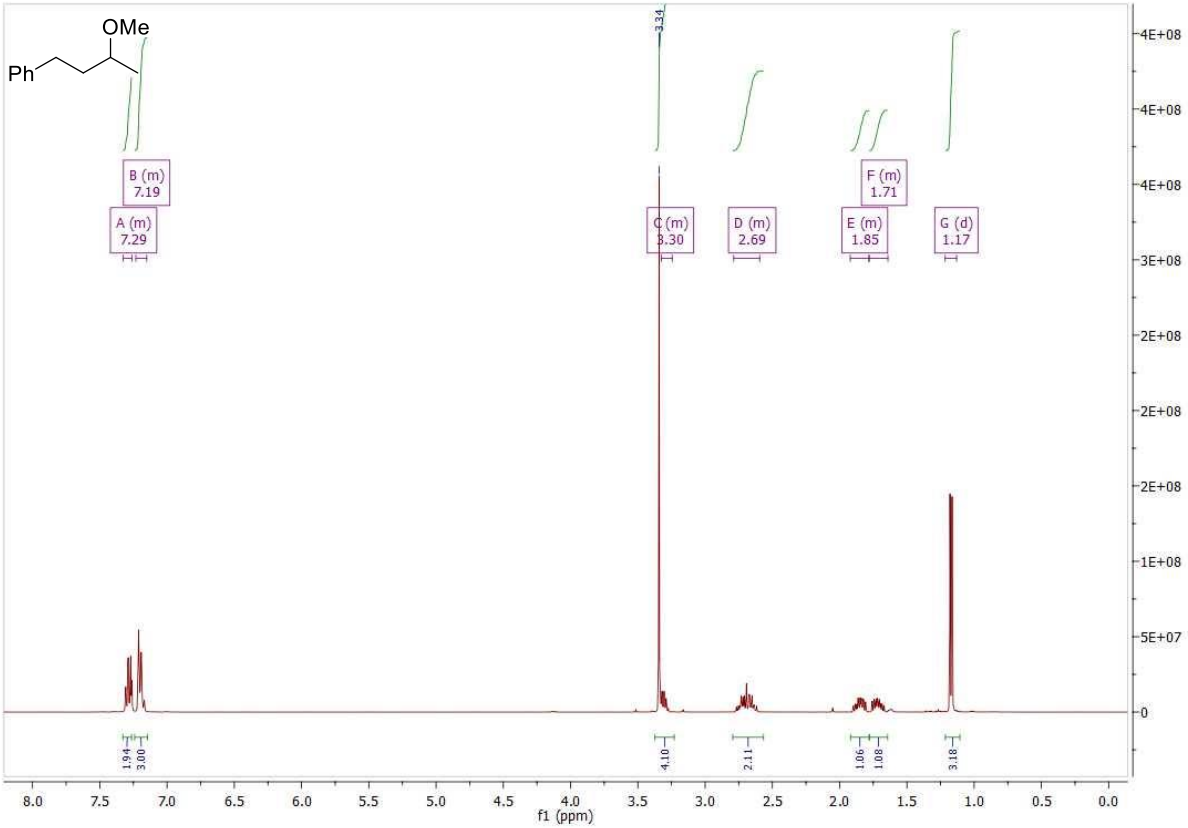

146.67
139.08
128.34
128.32
127.53
127.39
126.74
126.00
77.25
69.91
43.70
32.64
32.48
f1 (ppm)

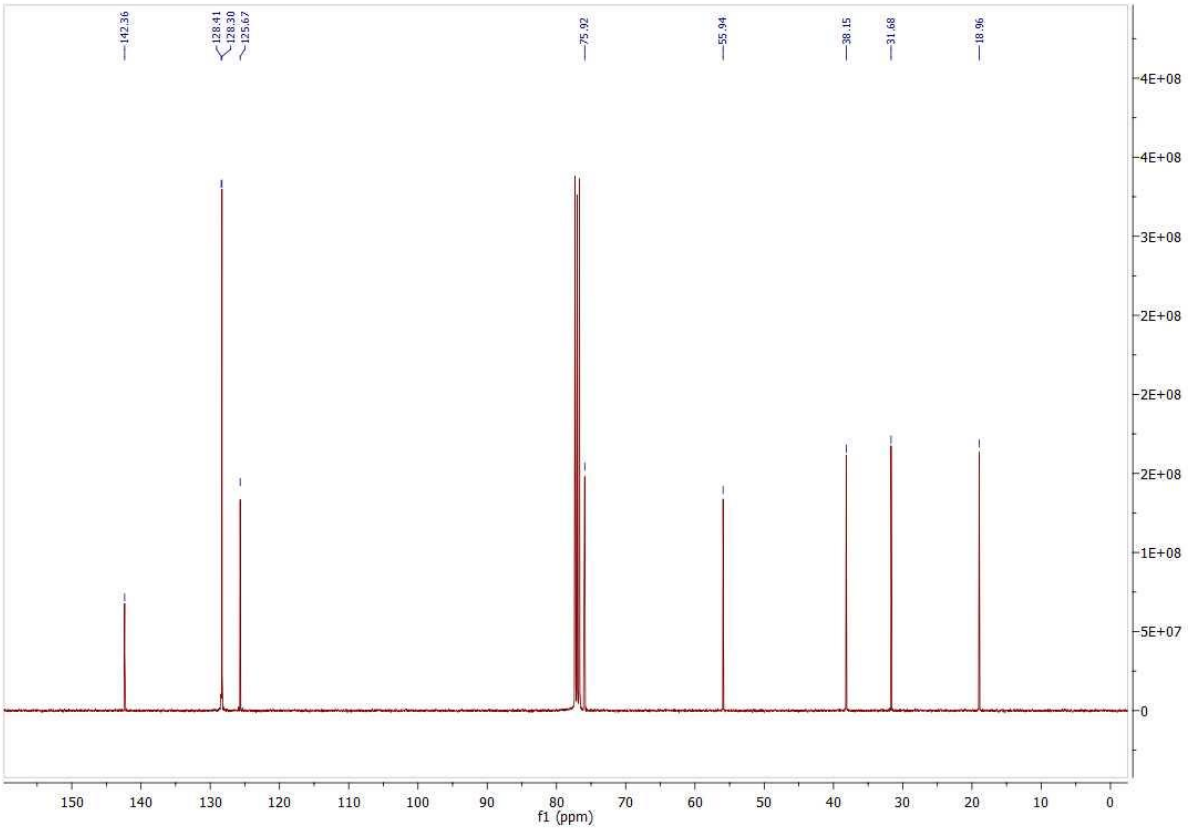

Ph
8a
G (m)
7.21
F (m)
7.31
E (m)
5.78
D (m)
2.80
C (m)
2.22
B (m)
1.94
A (m)
1.76
2.02
3.00
1.98
1.00
4.20
1.05
1.09
f1 (ppm)

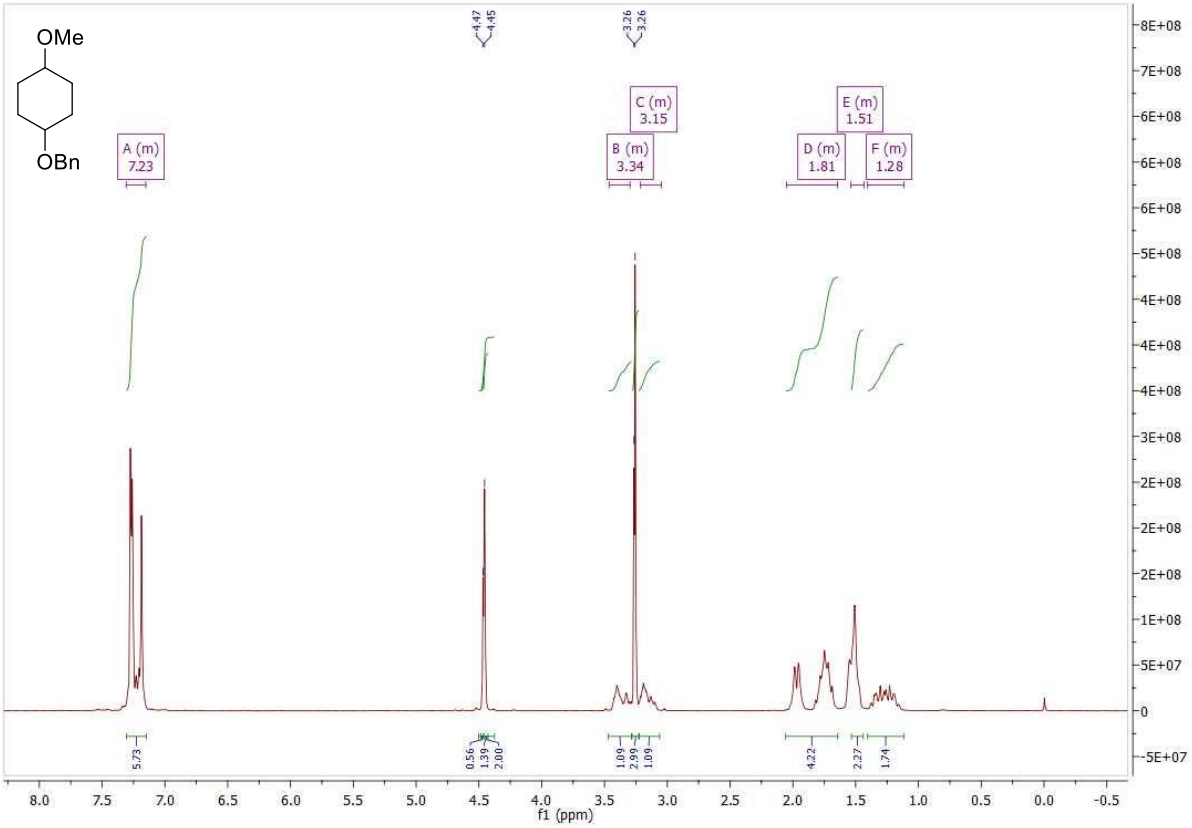

147.34
128.35
126.97
126.88
126.77
125.95
40.15
33.40
29.72
25.89
f1 (ppm)

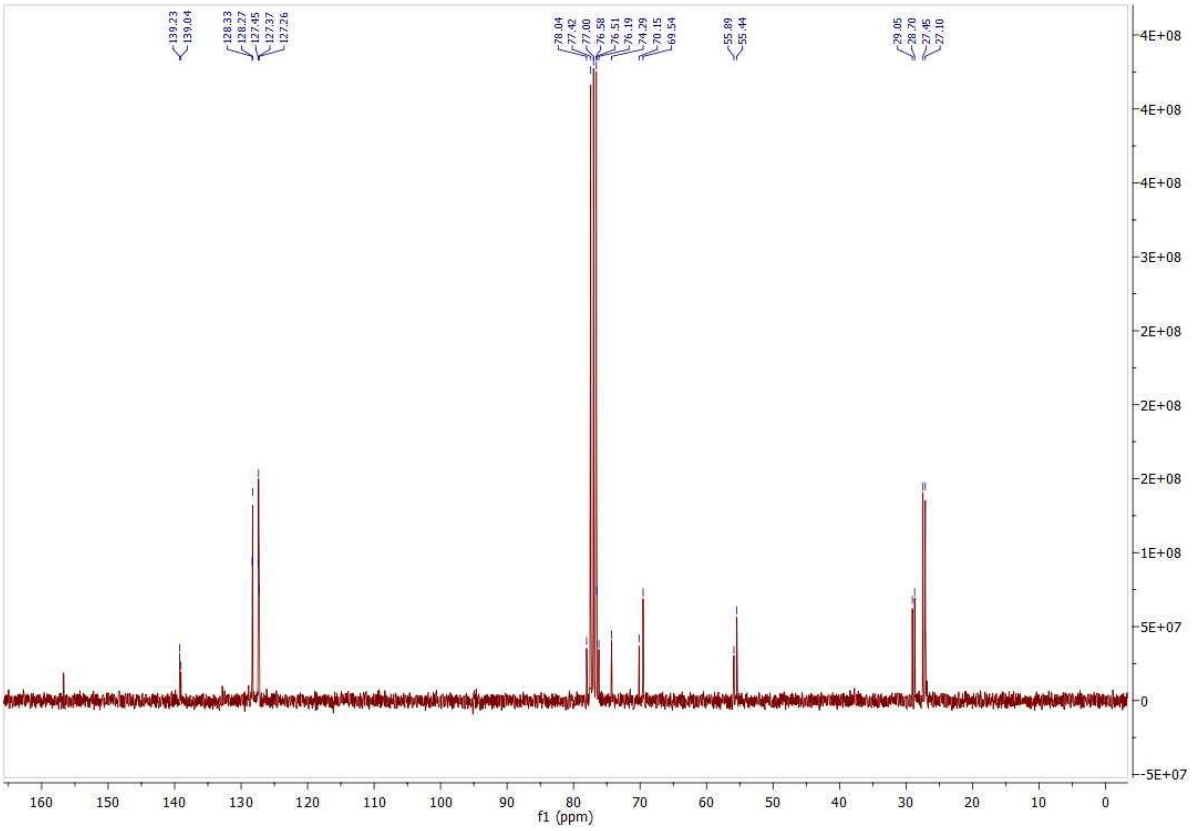

Ph
8b
B (m)
7.21
A (m)
7.30
D (m)
5.72
C (m)
5.89
E (m)
3.41
G (m)
1.75
F (m)
2.05
H (m)
1.62
1.98
3.00
0.93
0.95
1.06
2.99
1.09
2.21
f1 (ppm)

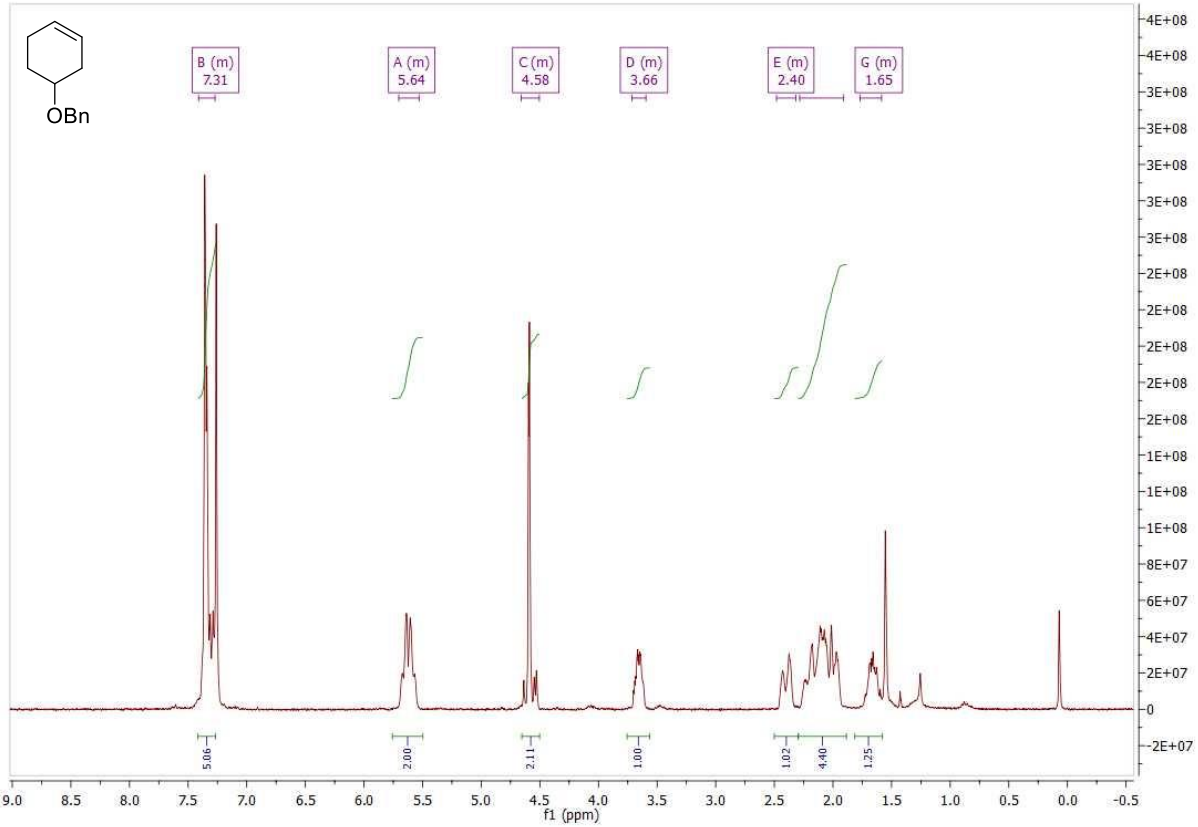

146.65
130.16
128.35
128.25
127.73
125.94
41.85
32.61
25.01
21.18
f1 (ppm)

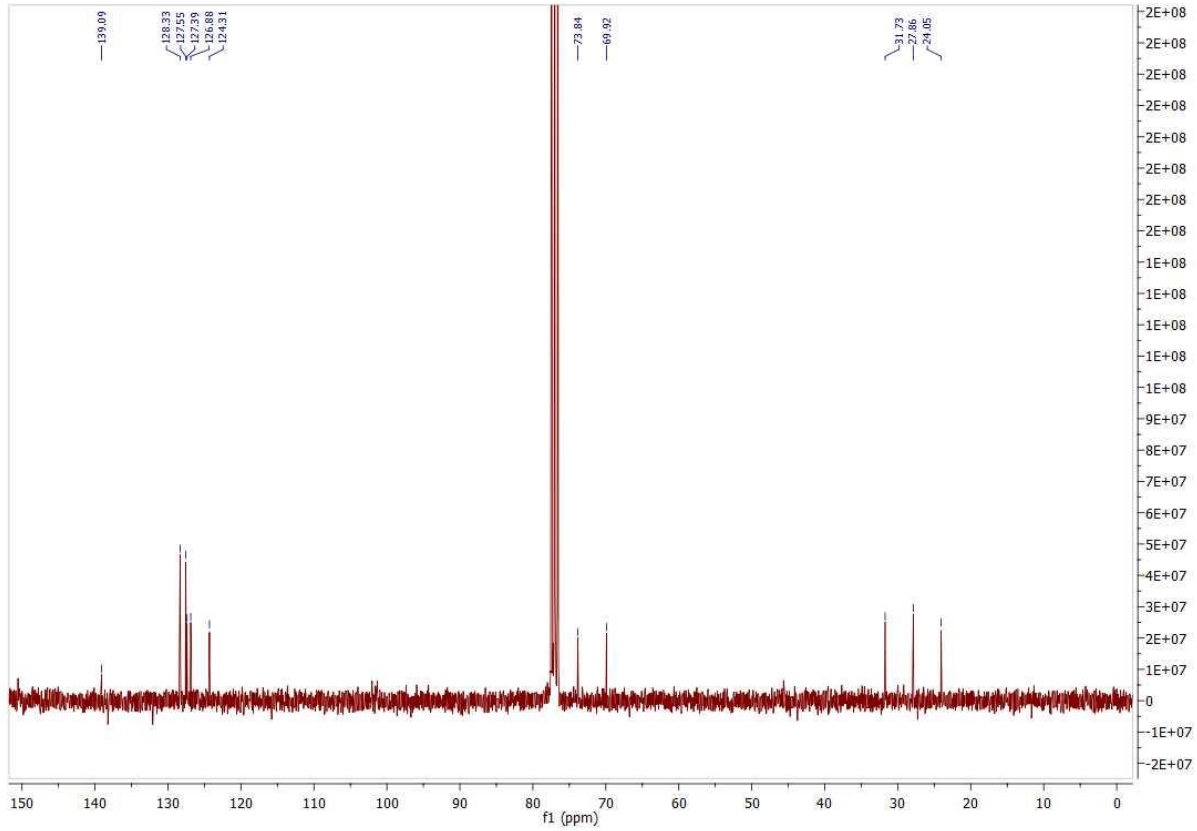

OMe
Ph
B (m)
7.19
A (m)
7.29
C (m)
3.30
D (m)
2.69
F (m)
1.71
E (m)
1.85
G (d)
1.17
f1 (ppm)


142.36
128.41
128.30
125.67
75.92
55.94
38.15
31.68
18.96
f1 (ppm)


OMe
OBn
A (m)
7.23
B (m)
3.34
C (m)
3.15
D (m)
1.81
E (m)
1.51
F (m)
1.28
4.47
4.45
3.26
3.26
5.73
0.56
1.39
2.00
1.09
2.99
1.09
4.22
2.27
1.74
f1 (ppm)


139.23
139.04
128.33
128.27
127.45
127.37
127.26
78.04
77.42
77.00
76.58
76.51
76.19
74.29
70.15
69.54
55.89
55.44
29.05
28.70
27.45
27.10
f1 (ppm)


OBn
B (m) 7.31
A (m) 5.64
C (m) 4.58
D (m) 3.66
E (m) 2.40
G (m) 1.65
5.06
2.00
2.11
1.00
1.02
4.40
1.25
f1 (ppm)


139.09
128.33
127.55
127.39
126.88
124.31
73.84
69.92
31.73
27.86
24.05
f1 (ppm)