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Supporting Information

Cross-Coupling Reaction between Alcohols through Sp^3 C-H Activation Catalyzed by Ruthenium/Lewis Acid System

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General Information and Procedure for the Coupling Reaction

General information:

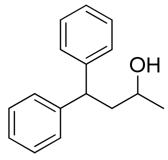
The materials were used as purchased. $Cl(CH_2)_2Cl$ and BF_3•OEt_2 were freshly distilled before use. All reactions were run under argon atmosphere using flame-dried glassware and magnetic stirring. Under standard conditions the reactions were monitored by thin-layer chromatography (TLC) on gel F_{254} plates. The silica gel (200-300 meshes) for column chromatography was from the Qingdao Marine Chemical Factory in China, and the distillation range of petroleum is 60-90°C.

^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ solution on the Varian Mercury-plus 300 or 400BB instruments, and spectral data are reported in *ppm* relative to tetramethylsilane (TMS) as internal standard. MS were measured on a HP-5988 spectrometer by direct inlet at 70 eV, and signals were given in m/z with relative intensity (%) in brackets. High-resolution mass spectral analysis (HRMS) data were measured on the Bruker ApexII by means of the ESI technique. Optical rotations were recorded in acetone at 20 °C by the DerkinElmer Model 341 Polarimeter. The $[\alpha]_D$ values were given in 10^{-1} deg cm^3 g^{-1}.

A general procedure for the coupling reaction:

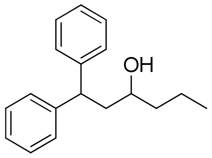
Typical procedure: To a flame-dried 25 mL flask were sequentially added $Cl(CH_2)_2Cl$ (4 mL), primary alcohol **2** (0.75 mmol) and $RuCl_2(PPh_3)_3$ (0.0125 mmol). It was stirred from room temperature to 40 °C for 10 min. The alcohol **1** (0.5 mmol) was added and stirred at 40 °C for 10 min under argon atmosphere, and then the freshly distilled BF_3•OEt_2 (0.15 mmol) was introduced into the above reaction mixture. The resulting mixture was stirred at 40 °C for 20 min, and another portion of BF_3•OEt_2 (0.45 mmol) was added further. The reaction was heated using oil bath to 50 °C, and stirred at 50 °C for 5h, After that, it was cooled to room temperature, and diluted with ethyl acetate (3 mL) followed by addition of saturated aqueous $NaHCO_3$ solution (2 mL). The organic layer was separated, and the aqueous phase was re-extracted with ethyl acetate (3 × 5 mL). The combined organic extracts were washed with H_2O (20 mL), and dried over anhydrous Na_2SO_4. After removal of the solvent, the residue was purified by the flash chromatography to afford the desired separable product **3**.

Analytic Data of Products



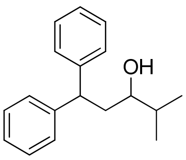
3aa

The compound ***3aa*** separated by flash chromatography (petraether: EtOAc=10:1)
[1]**H NMR** ($CDCl_3$, 400 MHz, ppm) δ: 1.18 (d, *J* = 6.0 Hz, 3 H), 2.02 (s, 1 H, -OH), 2.10 (dd, *J* = 8.4 and 6.4 Hz, 2 H), 3.63 (dd, *J* = 12.8 and 6.4 Hz, 1 H), 4.17 (dt, *J* = 8.0 Hz, 1 H), 7.13-7.18 (m, 2 H), 7.20-7.29 (m, 8 H); [13]**C NMR** ($CDCl_3$, 100 MHz, ppm) 23.9, 44.9, 47.6, 65.8, 126.1, 126.2, 127.6, 127.7, 128.0, 128.5, 144.3, 145.0; **MS m/z** (%) 226 (M^+, 3), 208 (76), 193 (46), 167 (100), 152 (32), 130 (37), 115 (24); **HRMS** (ESI): calculated for $C_{16}H_{22}NO$ $[M+NH_4]^+$: 244.1696; **found**: 244.1699.



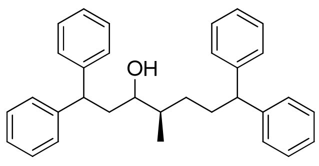
3ab

The compound ***3ab*** separated by flash chromatography (petraether: EtOAc=10:1).
[1]**H NMR** ($CDCl_3$, 400 MHz, ppm): δ 0.84-0.88 (m, 3 H), 1.29-1.31 (m, 1 H), 1.39-1.45 (m, 3 H, with -OH), 2.04-2.11 (m, 1 H), 2.19-2.26 (m, 1 H), 3.47 (dd, *J* = 6.0 and 3.2 Hz, 1 H), 4.23 (dd, *J* = 10.0 and 5.6 Hz, 1 H), 7.13-7.19 (m, 2H), 7.25-7.30 (m, 8 H); [13]**C NMR** ($CDCl_3$, 100 MHz, ppm) 14.0, 18.6, 40.1, 43.2, 47.5, 69.5, 126.1, 126.2, 127.7, 128.1, 128.4, 128.5, 144.1, 145.3; **MS m/z** (%) 254 (M^+, 1), 236 (49), 193 (100), 180 (22), 167 (89), 152 (23), 115 (22); **HRMS** (ESI): calculated for $C_{18}H_{26}NO$ $[M+NH_4]^+$: 272.2009; **found**: 272.2013.



3ac

The compound ***3ac*** separated by flash chromatography (petraether: EtOAc=10:1).
[1]**H NMR** ($CDCl_3$, 400 MHz, ppm): δ 0.84 (d, *J* = 7.2 Hz, 3 H), 0.91 (d, *J* = 6.8 Hz, 3 H), 1.43 (s, 1 H, -OH), 1.63-1.71 (m, 1 H), 2.00-2.08 (m, 1 H), 2.24-2.32 (m, 1 H), 3.25-3.29 (m, 1 H), 4.27 (dd, *J* = 10.8 and 5.2 Hz, 1 H), 7.14-7.23 (m, 2 H), 7.24-7.35 (m, 8 H); [13]**C NMR** ($CDCl_3$, 100 MHz, ppm) 17.1, 18.6, 34.0, 39.9, 47.7, 74.2, 126.1, 126.3, 127.7, 128.2, 128.4, 128.5, 144.0, 145.6; **MS m/z** (%) 254 (M^+, <1), 236 (32), 193 (79), 180 (52), 167 (100), 152 (24), 115 (17); **HRMS** (ESI): calculated for $C_{18}H_{26}NO$ $[M+NH_4]^+$: 272.2009; **found**: 272.2006.

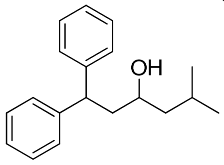


3ad

The ratio is 2:1 (Reaction time was 3 h)

The compound ***3ad*** separated by flash chromatography (petraether: EtOAc=10:1), but the mixture of diastereoiosmers could not be inseparable. The diastereoselectivity was determined from the integration resonances in the ^{13}**C NMR** spectrum of the mixture. The major isomer is defined as **A**, and the minor isomer as **B**. The chiral substrate purchased from the Prof. Wei-Sheng Tian, the Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences.

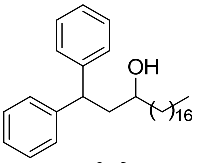
$[\alpha]_D^{20}$ = +4 (c = 1.0 in acetone, mix epimers ratio: 2:1); ^{1}**H NMR** ($CDCl_3$, 300 MHz, ppm): δ 0.86 (d, J = 6.9 Hz, 3 H_A+3 H_B), 1.05-1.37 (m, 4 H_A+4 H_B), 1.51 (s, 1 H, with -OH), 1.87-2.18 (m, 3 H_A+3 H_B), 3.13-3.18 (m, 1 H_A+1 H_B), 3.76-3.81 (m, 1 H_A+1 H_B), 4.13-4.19 (m, 1 H_A+1 H_B), 7.14-7.24 (m,20 H_A+20 H_B); ^{13}**C NMR** ($CDCl_3$, 75 MHz, ppm) 13.9, 15.2, 30.5, 31.4, 33.2, 33.3, 38.6, 38.9, 39.3, 40.1, 47.6, 47.7, 51.5, 51.6, 72.7 (**A**), 73.0 (**B**), 126.0, 126.2, 127.7, 127.8, 128.0, 128.1, 128.3, 128.4, 128.5, 143.8, 144.0, 144.7, 144.8, 145.1, 145.2, 145.3, 145.4; **MS m/z** (%) 434 (M^+, <1), 416 (1), 277 (16), 259 (100), 235 (10), 215 (8), 193 (12), 180 (46), 167 (74), 152 (28), 129 (14), 105 (22), 91 (16), 77 (24); **HRMS** (ESI): calculated for $C_{32}H_{38}NO$ $[M+NH_4]^+$: 452.2948; **found**: 452.2943.



3ae

The compound ***3ae*** separated by flash chromatography (petraether: EtOAc=10:1).

^{1}**H NMR** ($CDCl_3$, 400 MHz, ppm): δ 0.79 (d, J = 6.8 Hz, 3 H), 0.83 (d, J = 6.8 Hz, 3 H),1.24-1.30 (m, 1 H), 1.35-1.42 (m, 1 H), 1.55 (s, 1 H, -OH), 1.66-1.73 (m, 1 H), 2.01-2.09 (m, 1 H), 2.17-2.24 (m, 1 H), 3.48-3.55 (m, 1 H), 4.22 (dd, J =10.4 and 6.0 Hz, 1H), 7.12-7.18 (m, 2 H), 7.22-7.32 (m, 8 H); ^{13}**C NMR** ($CDCl_3$, 100 MHz, ppm) 22.2, 23.3, 24.6, 43.6, 47.2, 47.5, 67.8, 126.1, 126.2, 127.6, 127.7, 128.0, 128.1, 128.5, 144.1, 145.3; **MS m/z** (%) 268 (M^+, <1), 250 (38), 219 (12), 193 (52), 180 (42), 167 (100), 152 (24), 115 (14); **HRMS** (ESI): calculated for $C_{19}H_{28}NO$ $[M+NH_4]^+$: 286.2165; **found**: 286.2170.

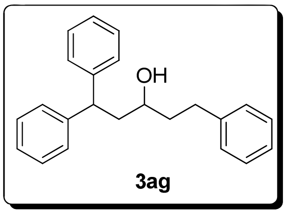


3af

(Reaction time was 16 h)

The compound ***3af*** separated by flash chromatography (petraether: EtOAc=10:1).

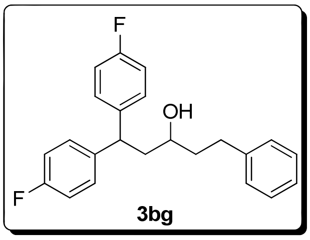
^{1}**H NMR** ($CDCl_3$, 400 MHz, ppm): δ 0.86-0.92 (m, 3 H), 1.21-1.32 (m, 30 H), 1.40 (s, 7.24-7.26 (m, 1 H, -OH), 1.47 (dd, J =10.4 and 5.2 Hz, 2H), 2.06-2.14 (m, 1 H), 2.22-2.29 (m, 1 H), 3.45-3.51 (m, 1 H), 4.26 (dd, J =10.4 and 6.0 Hz, 1H), 7.16-7.23 (m, 2 H), 7.25-7.35 (m, 8 H) ; ^{13}**C NMR** ($CDCl_3$, 100 MHz, ppm) 14.1, 22.7, 25.5, 29.3, 29.7, 31.9, 38.0, 43.2, 47.6, 69.8, 126.1, 126.3, 127.7, 128.1, 128.5, 144.2, 145.3; **MS m/z** (%) 450 (M^+, <1), 432 (6), 267 (6), 209 (20), 193 (64), 180 (45), 167 (100), 152 (20), 111 (30), 97 (40). **HRMS** (ESI): calculated for $C_{32}H_{54}NO$ $[M+NH_4]^+$: 468.4200; **found**: 468.4201.



(Reaction time was 4 h)

The compound ***3ag*** separated by flash chromatography (petraether: EtOAc=10:1).

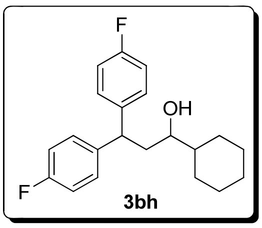
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 1.59 (s, 1H, -OH), 1.73-1.80 (m, 2 H), 2.09-2.16 (m, 1 H), 2.22-2.28 (m, 1 H), 2.53-2.60 (m, 1 H), 2.66-2.74 (m, 1 H),3.5 (m, 1 H), 4.19-4.23 (m, 1 H), 7.10-7.19 (m, 4 H), 7.22-7.29 (m, 11 H); **^{13}C NMR** ($CDCl_3$, 100 MHz, ppm) 31.9, 39.6, 43.1, 47.5, 69.3, 125.7, 126.1, 126.3, 127.6, 128.0, 128.3, 128.5, 141.9, 144.0, 145.0; **MS m/z** (%) 316 (M^+, <1), 298 (42), 220 (6), 207 (14), 193 (43), 180 (25), 167 (100), 152 (26), 91 (22); **HRMS** (ESI): calculated for $C_{23}H_{28}NO$ $[M+NH_4]^+$: 344.2165; **found**: 344.2164.



(Reaction time was 4 h)

The compound ***3bg*** separated by flash chromatography (petraether: EtOAc=10:1).

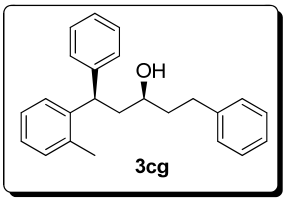
^{1}H NMR ($CDCl_3$, 300 MHz, ppm): δ 1.69-1.84 (m, 3 H, with -OH), 2.02-2.21 (m, 2 H), 2.54-2.82 (m, 2 H), 3.05-3.49 (m, 1 H), 4.20 (dd, J = 9.9 and 5.7 Hz, 1 H), 6.90-7.05 (m, 4 H), 7.10-7.27 (m, 9 H); **^{13}C NMR** ($CDCl_3$, 75 MHz, ppm) 31.9, 39.6, 43.3, 45.7, 69.0, 115.1, 115.2, 115.4, 115.5, 125.9, 128.2, 128.4, 128.9, 129.0, 129.3, 129.4, 139.5, 140.6, 141.7, 159.6, 159.7, 162.9, 163.0; **MS m/z** (%)352 (M^+, < 1), 334 (18), 229 (22), 216 (16), 203 (100), 183 (42), 131 (18), 91 (50); **HRMS** (ESI): calculated for $C_{23}H_{26}F_2NO$ $[M+NH_4]^+$: 370.1977; **found**: 370.1972.



(Reaction time was 3 h)

The compound ***3bh*** separated by flash chromatography (petraether: EtOAc=10:1)

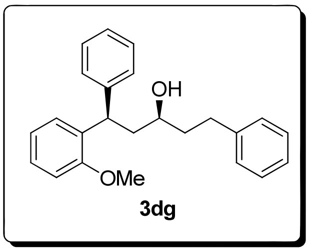
^{1}H NMR ($CDCl_3$, 300 MHz, ppm): δ 0.94-1.32 (m, 6 H, with -OH), 1.58-1.75 (m, 6 H), 1.91-2.02 (m, 1 H), 2.15-2.24 (m, 1 H), 3,13-3.19 (m, 1 H), 4.25 (dd, J = 11.1 and 4.5 Hz, 1 H), 6.90-7.00 (m, 4 H), 7.15-7.23 (m, 4 H); **^{13}C NMR** ($CDCl_3$, 75 MHz, ppm) 26.1, 26.2, 26.4, 27.7, 29.0, 40.1, 44.2, 45.8, 73.3, 115.0, 115.2, 115.3, 115.5, 128.8, 128.9, 129.4, 129.5, 139.4, 139.5, 141.2, 141.3, 159.7, 162.8, 163.0; **MS m/z** (%) 330 (M^+, <1), 312 (12), 229 (48), 216 (100), 203 (88), 183 (34), 133 (10), 95 (9); **HRMS** (ESI): calculated for $C_{21}H_{28}F_2NO$ $[M+NH_4]^+$: 348.2133; **found**: 348.2134.



syn : anti = 7 : 1 (Reaction time was 4 h)

The compound ***3cg*** separated by flash chromatography (petraether: EtOAc=10:1), but the mixture of diastereoiosmers could not be inseparable. The diastereoselectivity was determined from the integration resonances in the **^{13}C NMR** spectrum of the mixture. The major *syn* isomer is defined as **A**, and the minor *anti* isomer as **B**.

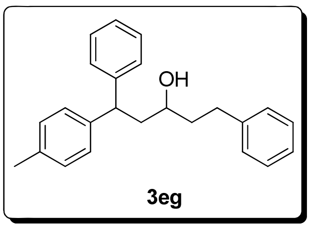
^{1}H NMR ($CDCl_3$, 300 MHz, ppm): δ 1.71-1.81 (m, 3 H, with -OH), 2.05-2.08 (m, 2H), 2.12-2.26 (m, 3 H), 2.54-2.70 (m, 2 H), 3.50-3.54 (m, 1 H), 4.42 (dd, *J* = 9.3 and 6 Hz, 1 H), 7.09-7.28 (m, 14 H); **^{13}C NMR** ($CDCl_3$, 75 MHz, ppm) 19.9, 31.9, 39.6, 42.8, 43.6, 69.3 (**A**), 69.0 (**B**), 125.7, 125.9, 126.1, 126.6, 127.9, 128.2, 128.3, 128.4, 130.6, 136.7, 141.4, 141.9, 144.7; **MS m/z** (%) 330 (M^+, < 1), 312 (32), 297 (3), 207 (28), 181 (100), 166 (50), 129 (20), 105 (22), 91 (58), 77 (10); **HRMS** (ESI): calculated for $C_{24}H_{30}NO$ $[M+NH_4]^+$: 348.2323; **found**: 348.2320.



syn : anti = 3 : 2 (Reaction time was 4 h)

The compound ***3dg*** separated by flash chromatography (petraether: EtOAc=10:1), but the mixture of diastereoiosmers could not be inseparable. The diastereoselectivity was determined from the integration resonances in the **^{13}C NMR** spectrum of the mixture. The major *syn* isomer is defined as **A**, and the minor *anti* isomer as **B**.

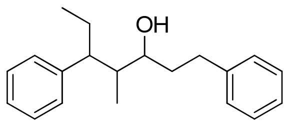
^{1}H NMR ($CDCl_3$, 300 MHz, ppm): δ 1.73-1.81 (m, 2 H_A+2 H_B,), 2.01-2.24 (m, 3 H_A+3 $H_{B,}$ with -OH), 2.55-2.80 (m, 2 H_A +2 H_B), 3.43-3.60 (m, 1 H_A+1 H_B), 3.73 (s, 3 H_B), 3.79 (s, 3 H_A), 3.62-3.78 (m, 1 H_A+1 H_B), 6.77-6.90 (m, 2 H_A+2 H_B), 7.04-7.28 (m, 12 H_A+12 H_B); **^{13}C NMR** ($CDCl_3$, 75 MHz, ppm) 31.9, 32.0, 38.7, 39.0, 39.5, 39.6, 42.5, 42.8, 55.3 (**B**), 55.6 (**A**), 69.1 (**A**), 69.6 (**B**), 110.6, 110.7, 120.5, 121.1, 125.5, 125.6, 125.9, 127.1, 127.2, 127.6, 128.0, 128.2, 128.3, 128.3, 128.5, 132.7, 133.4, 142.1, 142.3, 144.0, 156.6, 156.7; **MS m/z** (%) 346 (M^+, < 1), 328 (10), 314 (5), 223 (18), 197 (62), 183 (35), 165 (20), 152 (12), 129 (14), 91 (100), 77 (8); **HRMS** (ESI): calculated for $C_{24}H_{30}NO_2$ $[M+NH_4]^+$: 364.2271; **found**: 364.2266.



syn : anti = 1 : 1 (Reaction time was 4 h)

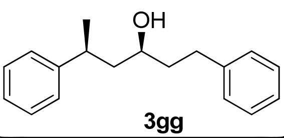
The compound ***3eg*** separated by flash chromatography (petraether: EtOAc=10:1), but the mixture of diastereoiosmers could not be inseparable. The diastereoselectivity was determined from the integration

resonances in the ^{13}**C NMR** spectrum of the mixture. The *syn* isomer is defined as **A**, and the *anti* isomer as **B**.
^{1}**H NMR** ($CDCl_3$, 300 MHz, ppm): δ 1.38 (s, 1 H_A+1 H_B, with -OH), 1.75-1.84 (m, 2 H_A+2 H_B), 2.13-2.25 (m, 2 H_A+2 H_B), 2.28-2.30 (m, 3 H_A+3 H_B), 2.60-2.74 (m, 2 H_A+2 H_B), 3.54-3.55 (m, 1 H_A+1 H_B), 4.15-4.20 (m, 1 H_A+1 H_B), 7.06-7.28 (m, 14 H_A+14 H_B); ^{13}**C NMR** ($CDCl_3$, 75 MHz, ppm) 20.91, 31.9, 39.6, 43.1, 47.0, 47.1, 69.38, 69.42, 125.7, 126.1, 126.2, 127.5, 127.6, 127.8, 127.9, 128.2, 128.3, 128.4, 128.5, 129.1, 129.2, 135.6, 135.7, 140.9, 142.0, 144.2, 145.3; **MS m/z** (%) 330 (M^+, < 1), 312 (28), 297 (4), 207 (35), 181 (100), 166 (48), 129 (16), 105 (18), 91 (50), 77 (10); **HRMS** (ESI): calculated for $C_{24}H_{30}NO$ $[M+NH_4]^+$: 348.2323; **found**: 348.2324.

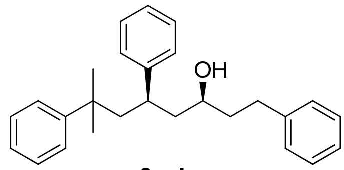

3fg

syn* : *anti = 5 : 1 (Reaction time was 6 h)

The compound ***3fg*** separated by flash chromatography (petraether: EtOAc=10:1), but we just received two diastereoiosmers and could not be inseparable, the other isomers were trace and could not be received. The major isomer is defined as **A**, and the minor isomer as **B**, and the ratio was determined from the integration resonances in the ^{13}**C NMR** spectrum of the mixture.
^{1}**H NMR** ($CDCl_3$, 300 MHz, ppm): δ 0.61-0.70 (m, 3 H_A+3 H_B,), 0.93 (d, *J* = 7.2 Hz, 3 H_A), 0.99 (d, *J* = 6.3 Hz, 3 H_B), 1.40-1.88 (m, 5 H_A+5 H_B), 2.29-2.47 (m, 2 H_A+2 H_B), 2.47-2.76 (m, 1 H_A+1 H_B), 3.29-3.33 (m, 1 H_A+1 H_B), 7.04-7.27 (m, 10 H_A+10 H_B); ^{13}**C NMR** ($CDCl_3$, 75 MHz, ppm) 10.7 (**B**), 11.6 (**A**), 12.2, 12.3, 25.0 (**A**), 26.3 (**B**), 32.7, 33.4 (**A**), 37.3 (**B**), 42.9 (**B**), 45.4 (**A**), 50.0 (**A**), 50.7 (**B**), 71.7 (**B**), 72.7, 125.7, 125.9, 126.0, 128.1, 128.2, 128.3, 129.0, 142.1, 142.3, 144.1, 144.3; **MS m/z** (%) 282 (M^+, < 1), 264 (4), 235 (3), 173 (4), 163 (7), 145 (12), 131 (14), 119 (30), 105 (23), 91 (100), 77 (5); **HRMS** (ESI): calculated for $C_{20}H_{26}ONa$ $[M+Na]^+$: 305.1876; **found**: 305.1873.

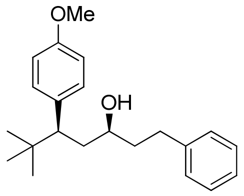

3gg

syn* : *anti > 99 : 1 (Reaction time was 4 h)


3gg'

syn* : *anti > 99 : 1 (Reaction time was 4 h)

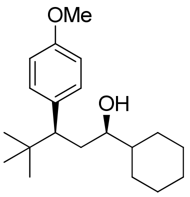
The compound ***3gg*** separated by flash chromatography (petraether: EtOAc=10:1), and the *anti* isomer could not be detected by ^{13}**C NMR** spectrum, but the other byproduct ***3gg'***could be received in 10% yields.
^{1}**H NMR** ($CDCl_3$, 400 MHz, ppm): δ 1.29 (d, *J* = 5.2 Hz, 3 H), 1.35 (s, 1 H, with -OH), 1.70-1.86 (m, 4 H), 2.61-2.69 (m, 1 H), 2.75-2.81 (m, 1 H), 2.89-2.94 (m, 1 H), 3.65-3.69 (m, 1 H), 7.13-7.24 (m, 6 H), 7.28-7.33 (m, 4 H); ^{13}**C NMR** ($CDCl_3$, 100 MHz, ppm) 22.0, 31.9, 36.8, 39.3, 46.2, 69.7, 125.7, 126.1, 126.8, 128.4, 128.5, 142.1, 147.3; **MS m/z** (%) 254 (M^+, < 1), 236 (28), 194 (5), 158 (6), 145 (32), 131 (42), 118 (50), 105 (100), 91 (92), 77 (16); **HRMS** (ESI): calculated for $C_{18}H_{26}NO$ $[M+NH_4]^+$: 272.2009; **found**: 272.2012.



3hg ***syn : anti*** > 99 : 1 (Reaction time was 3 h)

The compound ***3hg*** separated by flash chromatography (petraether: EtOAc=10:1), and the *anti* isomer could not be detected by **^{13}C NMR** spectrum.

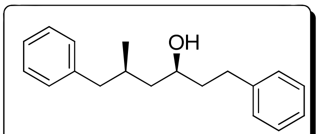
^{1}H NMR ($CDCl_3$, 300 MHz, ppm): δ 0.83 (s, 9 H), 1.34 (s, 1 H, with -OH), 1.67-1.84 (m, 4 H), 2.51-2.69 (m, 3 H), 3.26 (dt, *J* = 3.3 Hz, 1 H), 3.80 (s, 3 H), 6.82 (d, *J* = 9.0 Hz, 2 H), 7.04-7.26 (m, 7 H); **^{13}C NMR** ($CDCl_3$, 75 MHz, ppm) 28.2, 32.2, 33.6, 37.1, 40.1, 51.4, 55.1, 69.6, 113.1, 125.7, 128.2, 128.3, 130.0, 134.3, 142.2, 157.9; **MS m/z** (%) 326 (M^+, 3), 293 (1), 269 (10), 251(5), 177 (5), 147 (7), 135 (100), 121 (15), 105 (10), 91 (47); **HRMS** (ESI): calculated for $C_{22}H_{34}NO_2$ $[M+NH_4]^+$: 344.2584; **found**: 344.2587.



3hh ***syn : anti*** > 99 : 1 (Reaction time was 3 h)

The compound ***3hh*** separated by flash chromatography (petraether: EtOAc=10:1), and the *anti* isomer could not be detected by **^{13}C NMR** spectrum.

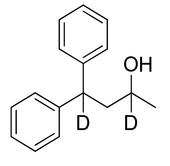
^{1}H NMR ($CDCl_3$, 300 MHz, ppm): δ 0.86 (s, 9 H), 0.91-1.23 (m, 7 H, with -OH), 1.62-1.83 (m, 7 H), 2.64 (dd, *J* = 12.0 and 2.4 Hz, 1 H), 2.89-2.94 (m, 1 H), 3.79 (s, 3 H), 6.81 (d, *J* = 6.3 Hz, 2 H), 7.05 (d, *J* = 7.8 Hz, 2 H); **^{13}C NMR** ($CDCl_3$, 75 MHz, ppm) 26.1, 26.2, 26.5, 28.1, 28.2, 29.0, 33.6, 44.5, 51.4, 55.1, 73.7, 112.9, 130.5, 134.4, 157.7; **MS m/z** (%) 304 (M^+, 1), 247 (6), 177 (2), 149(4), 135 (100), 121 (8), 105 (4), 95 (18); **HRMS** (ESI): calculated for $C_{20}H_{36}NO_2$ $[M+NH_4]^+$: 322.2741; **found**: 322.2741.



3ig ***syn : anti*** = 8 : 1 (Reaction time was 16 h)

The compound ***3ig*** separated by flash chromatography (petraether: EtOAc=10:1), but the mixture of diastereoiosmers could not be inseparable. Furthermore, other isomers were trace and could not be received. The diastereoselectivity was determined from the integration resonances in the **^{13}C NMR** spectrum of the mixture. The major *syn* isomer is defined as **A**, and the minor *anti* isomer as **B**.

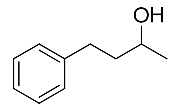
^{1}H NMR ($CDCl_3$, 300 MHz, ppm): δ 0.89, (d, *J* = 6.6 Hz, 3 H), 1.21-1.30 (m, 1 H), 1.40-1.58 (m, 2 H), 1.65-1.77 (m, 2 H), 2.00 (s, 1H, with –OH), 2.33-2.82 (m, 4 H), 3.75 (m, 1 H), 7.12-7.27 (m, 10 H); **^{13}C NMR** ($CDCl_3$, 75 MHz, ppm) 19.1, 20.3, 31.3, 31.9, 32.1, 39.2, 40.0, 44.3, 44.5, 69.1 (**A**), 69.4 (**B**), 125.8, 128.2, 128.3, 128.4, 129.2, 141.0, 142.1; **MS m/z** (%) 268 (M^+, < 1), 250 (5), 208 (3), 172 (2), 159 (13), 145 (15), 131 (10), 118 (90), 103 (12), 91 (100), 65 (8); **HRMS** (ESI): calculated for $C_{19}H_{28}NO$ $[M+NH_4]^+$: 286.2165; **found**: 286.2168.



3aa-d2

The compound ***3aa-d*$_2$** separated by flash chromatography (petraether: EtOAc=10:1), The CH_3CD_2OH (min 98 atom % D, from the Sigma-Aldrich Trading Co., Ltd.).

^{1}H NMR ($CDCl_3$, 300 MHz, ppm) δ: 1.20 (s, 3 H), 1.36 (s, 1 H, -OH), 2.16 (s, 2 H), 7.14-7.21 (m, 2 H), 7.25-7.29 (m, 8 H); **^{13}C NMR** ($CDCl_3$, 75 MHz, ppm) 23.9, 44.7, 126.2, 126.3, 127.7, 128.0, 128.5, 128.5, 144.2, 144.9; **MS m/z** (%) 228 (M^+, < 1), 210 (32), 209 (46), 194 (38), 179 (16), 168 (100), 166 (52), 153 (28), 130 (34), 116 (24); **HRMS** (ESI): calculated for $C_{16}H_{16}D_2ONa$, $[M+Na]^+$: 251.1375; **found**: 251.1377.



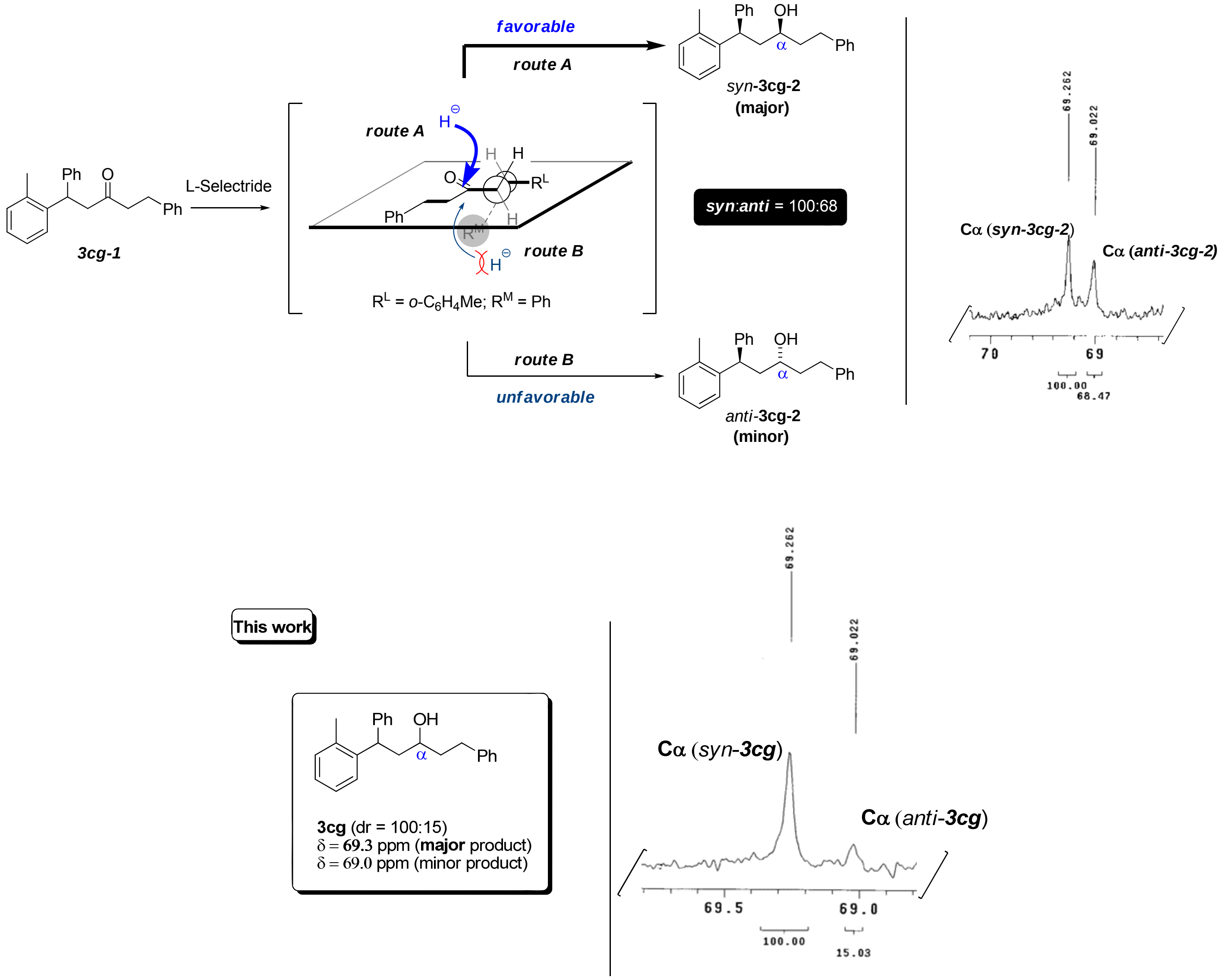
4-phenyl-2-butanol

The compound ***4-phenyl-2-butanol*** as a major product was separated by flash chromatography (petraether: EtOAc=10:1). The coupling reactionan used the general procedure, and stirred at 55 ^oC for 24h.

^{1}H NMR ($CDCl_3$, 400 MHz, ppm) δ: 1.24 (d, *J* = 6.4 Hz, 3 H), 1.75 (s, 1 H, -OH), 1.76-1.82 (m, 2 H), 2.67-2.79 (m, 2 H), 3.84 (dd, *J* = 12.2 and 6.6 Hz, 1 H), 7.18-7.22 (m, 3 H), 7.26-7.32 (m, 2 H); **^{13}C NMR** ($CDCl_3$, 100 MHz, ppm) 29.6, 32.1, 40.8, 67.5, 125.8, 128.4, 142.0; **MS m/z** (%) 150 (M^+, 6), 132 (48), 117 (84), 105 (68), 91 (100), 77 (18).

Stereo-configuration Analytic of Products

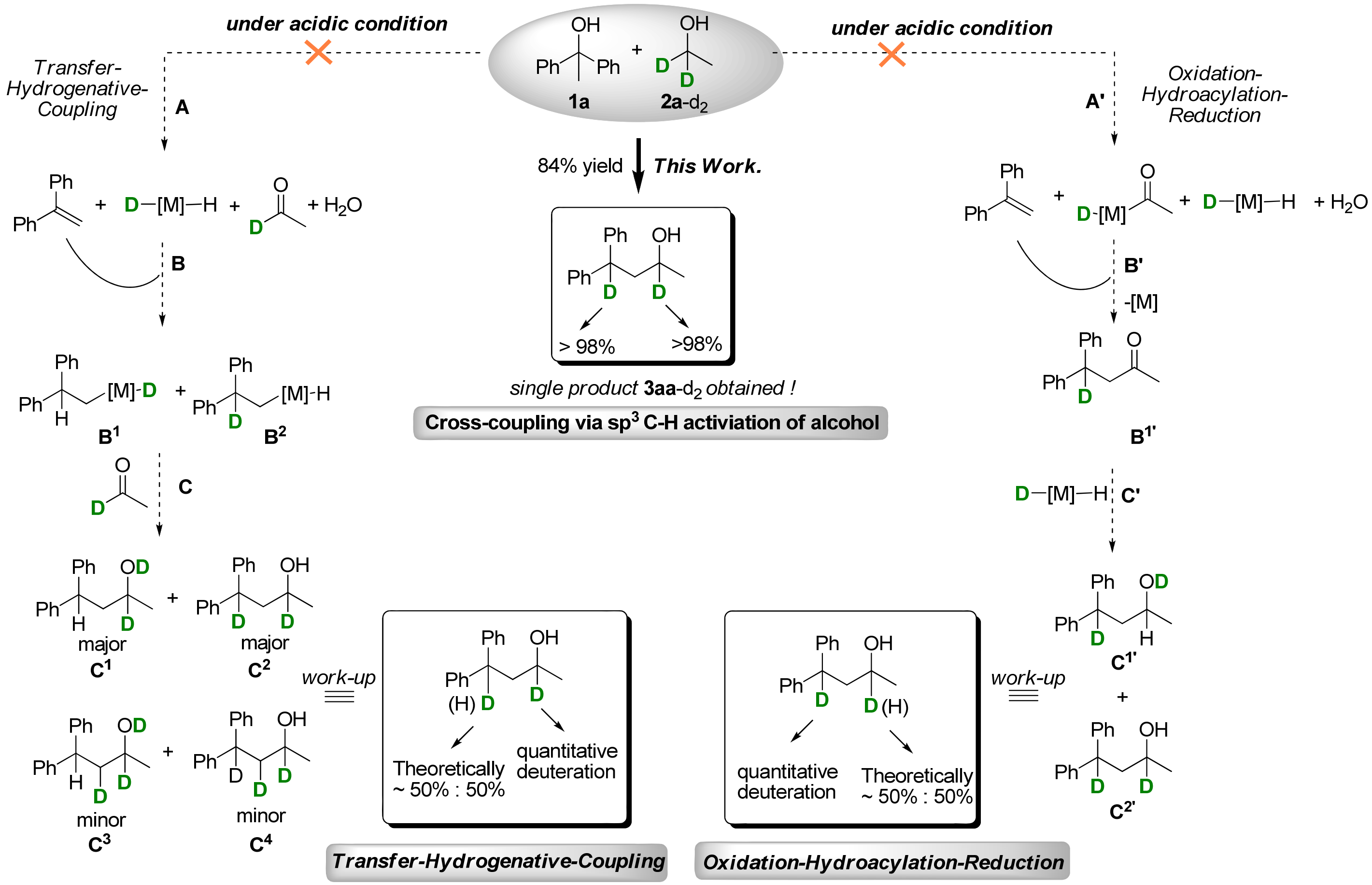
For determination of the relative configuration, the Hydride with bulky substituents L-selectride was especially useful for this stereoselective reduction of carbonyl compounds ***3cg-1*.** Through the configuration analytic that the preferential conformation was ***syn-3cg-2***, and the *syn: anti* products were received with the ratio 3:2. Compared with the chemical shifts of ***Cα***, we assured that the major reaction product of our work is *syn* configuration (scheme 1)..



SI-Scheme 1. *Stereo-configuration Analytic of Products*

The Comparison of different pathway in deuterium experiment

—The “Transfer-Hydrogenative-Coupling”, “Oxidation-Hydroacylation-Reduction”, and our “Sp^3- C-H Activiation”

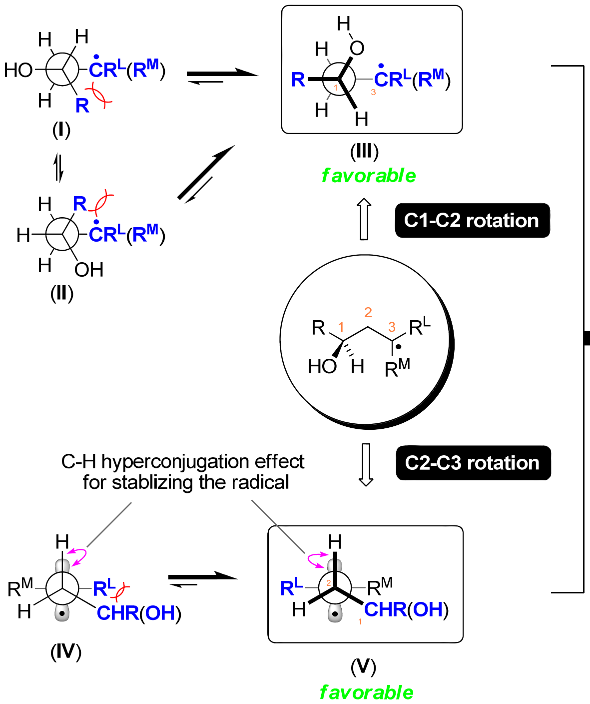


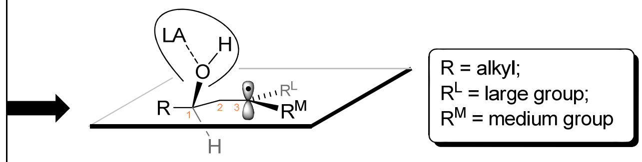
For the recent examples on the *"Transfer-Hydrogenative-Coupling"* under ***basic*** condition, see: 1) *J. Am. Chem. Soc.* **2007**. *129*, 15134-15135; 2) *J. Am. Chem. Soc.* **2008**. *130*, 6338-6339; 3) *J. Am. Chem. Soc.* **2008**. *130*. 6340-6341.

For the recent review on "*Oxidation-Hydroacylation-Reduction*", *see: Acc. Chem. Res.* **2008**, *41*, 222-234.

SI-Scheme 2. *Deuterium Experiment*

Preferred conformation of the radical intermediate D

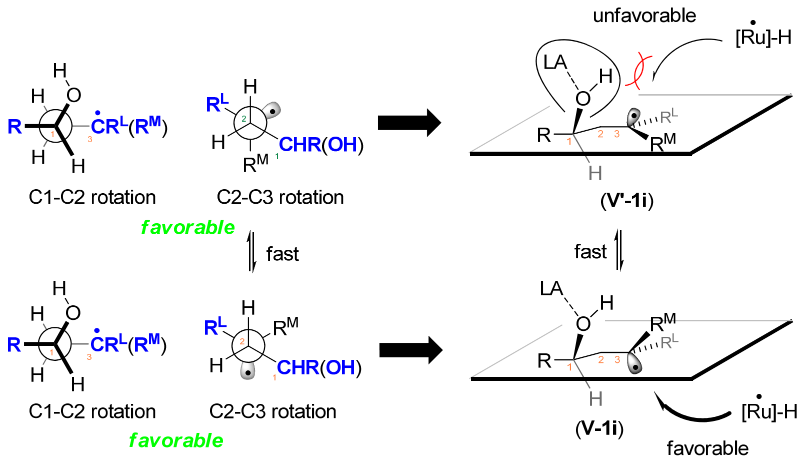




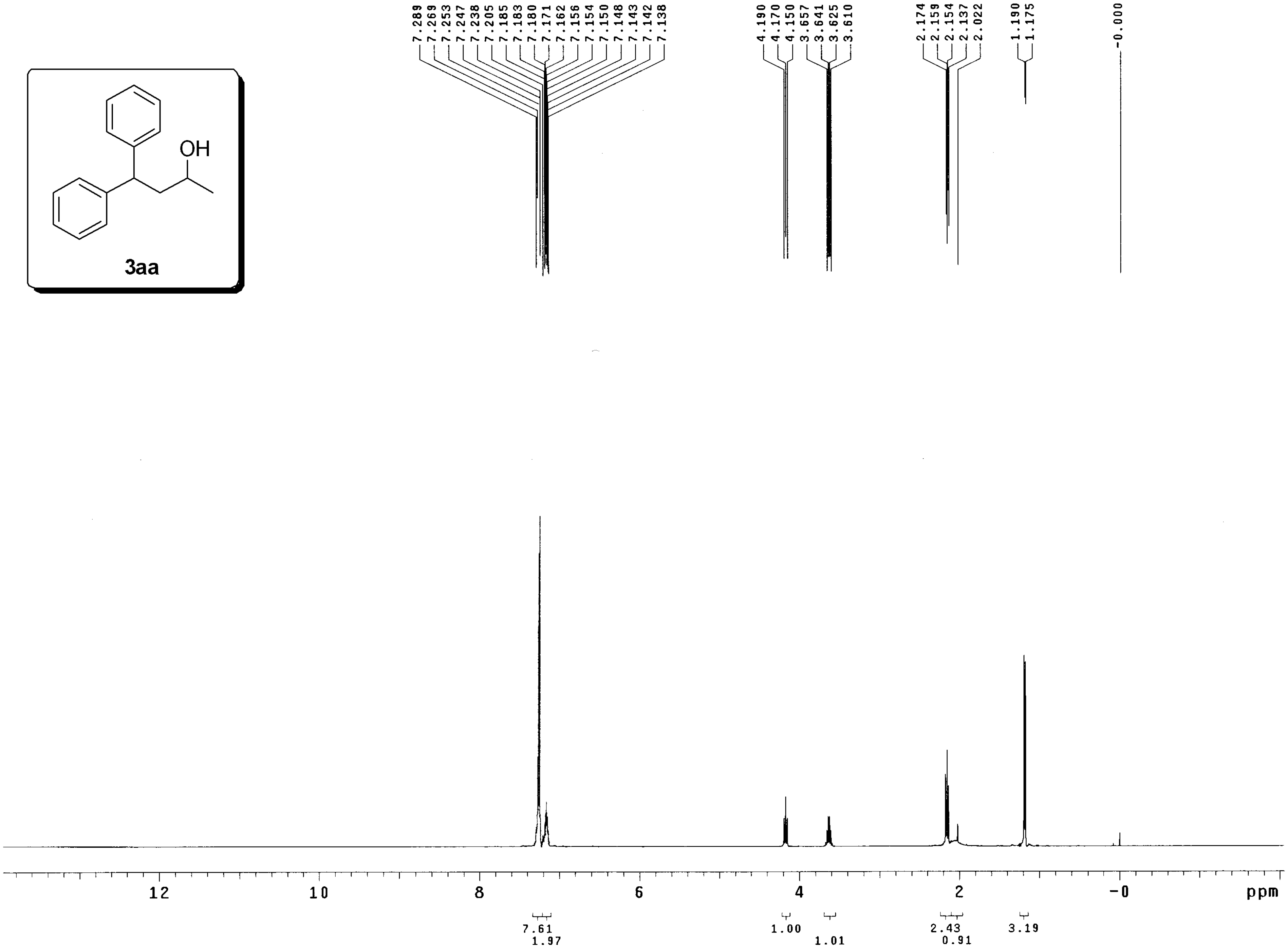
In this stable conformation of the radical intermediate in the transition state **D** of Scheme 3, "**R-C1-C2-C3**" with an energetically stable staggered conformation (**III**) aligned in the one plane, in which "**OH**" is arranged on the upside of the plane and "**H**" is on its downside. "**R^L-R^M-C2-C3**" with a preferred conformation (**V**) alomst located in this plane, wherein "**R^M**" is in front and "**R^L**" is at backside.

For all examples except entry 8 of Table 3, either of **R^L** and **R^M** is aromatic group . So, the active specie in **D** of Scheme 3 should be the planar benzyl radical because of the p-π conjugation.

In the case of entry 8 of Table 3 (R^L = CH_2Ph, R^M = Me, R = CH_2CH_2Ph), the tertiary radical intermediate in **D** should be the rapidly inverting pyramidal structure (see **V-1i** and **V'-1i**), but the stereochemical outcome of the final reductive product is equivalent to that through the mentioned above reaction model of the planar radicals.



SI-Scheme 3. *Preferred conformation of the radical intermediate in D of Scheme 3.*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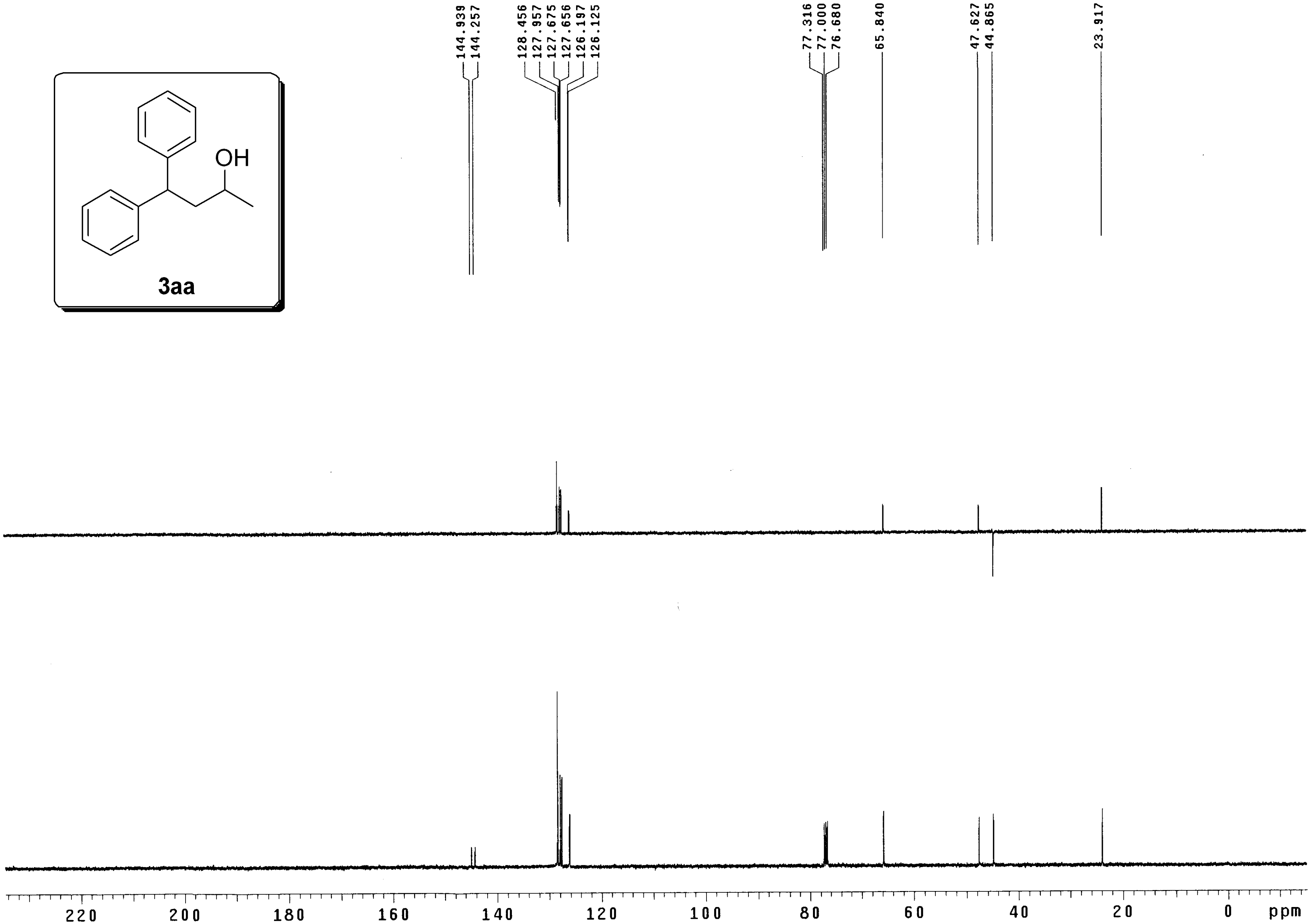
4.190 4.170 4.150 3.657 3.641 3.625 3.610
2.174 2.159 2.154 2.137 2.022
1.190 1.175
-0.000
OH
3aa
12
10
8
6
4
2
-0
ppm
7.61
1.97
1.00
1.01
2.43
0.91
3.19



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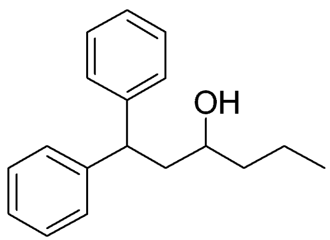
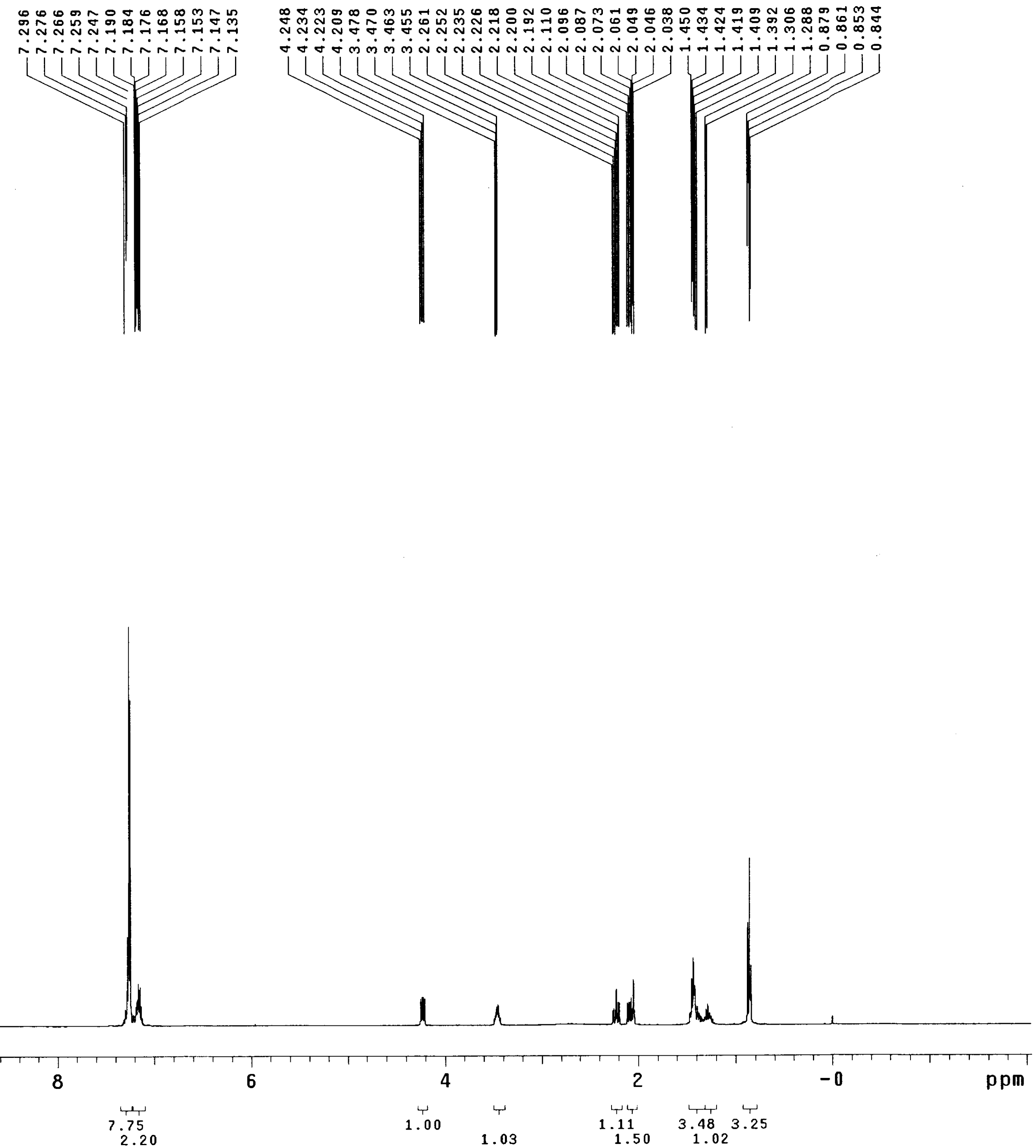
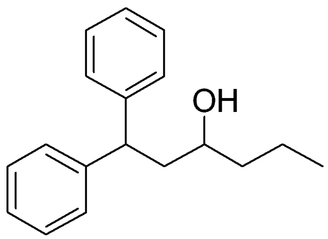
144.257
128.456
127.957
127.675
127.656
126.197
126.125
77.316
77.000
76.680
65.840
47.627
44.865
23.917
OH
3aa
220 200 180 160 140 120 100 80 60 40 20 0 ppm

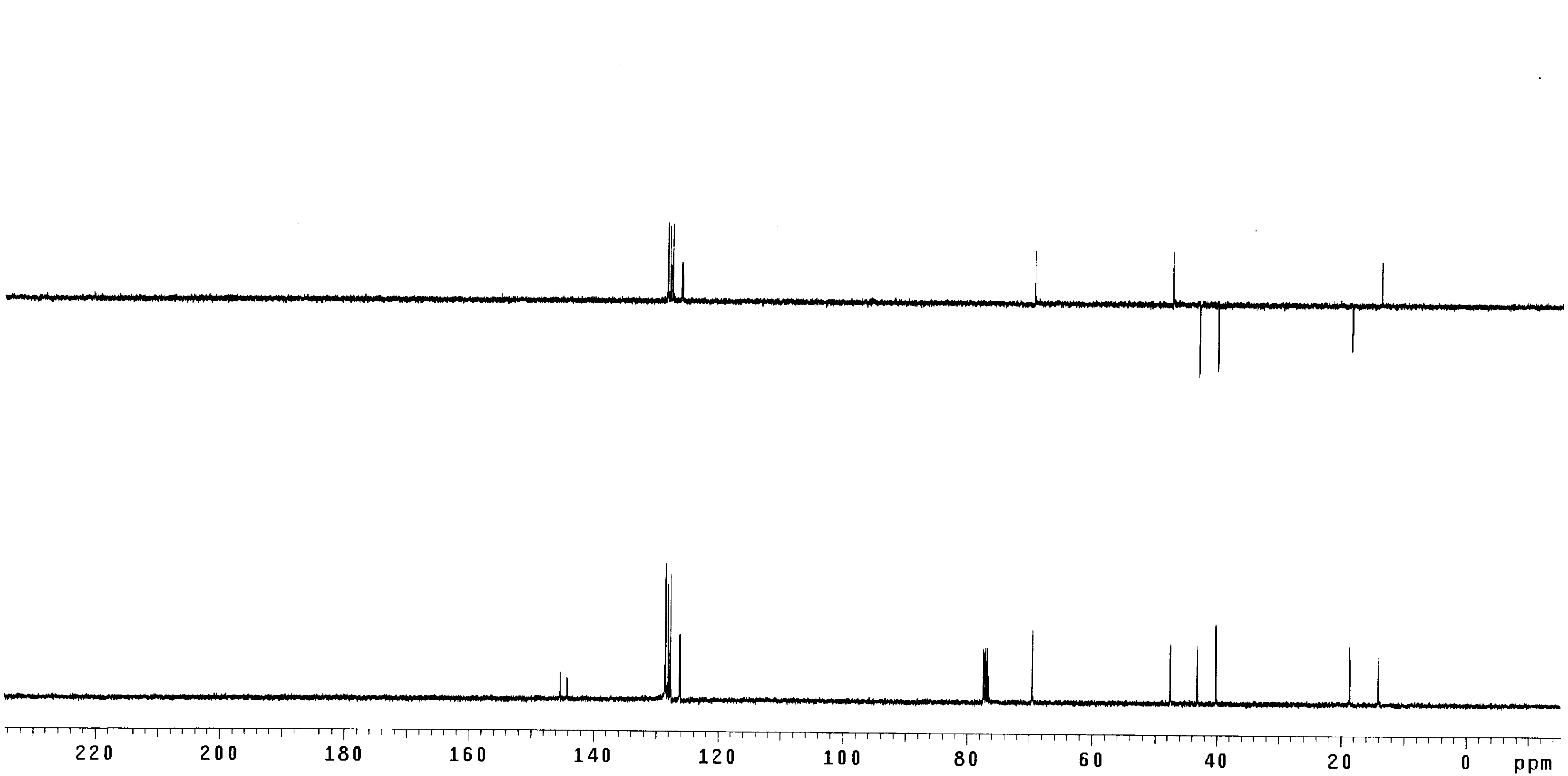


3ab


7.296 7.276 7.266 7.259 7.247 7.190 7.184 7.176 7.168 7.158 7.153 7.147 7.135
4.248 4.234 4.223 4.209 3.478 3.470 3.463 3.455 2.261 2.252 2.235 2.226 2.218 2.200 2.192 2.110 2.096 2.087 2.073 2.061 2.049 2.046 2.038 1.450 1.434 1.424 1.419 1.409 1.392 1.306 1.288 0.879 0.861 0.853 0.844
12 10 8 6 4 2 -0 ppm
7.75 2.20 1.00 1.03 1.11 1.50 3.48 1.02 3.25


OH

3ab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127.672
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126.094
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77.000
76.676
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47.498
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120
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60
40
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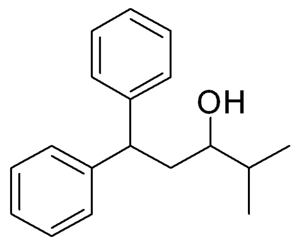
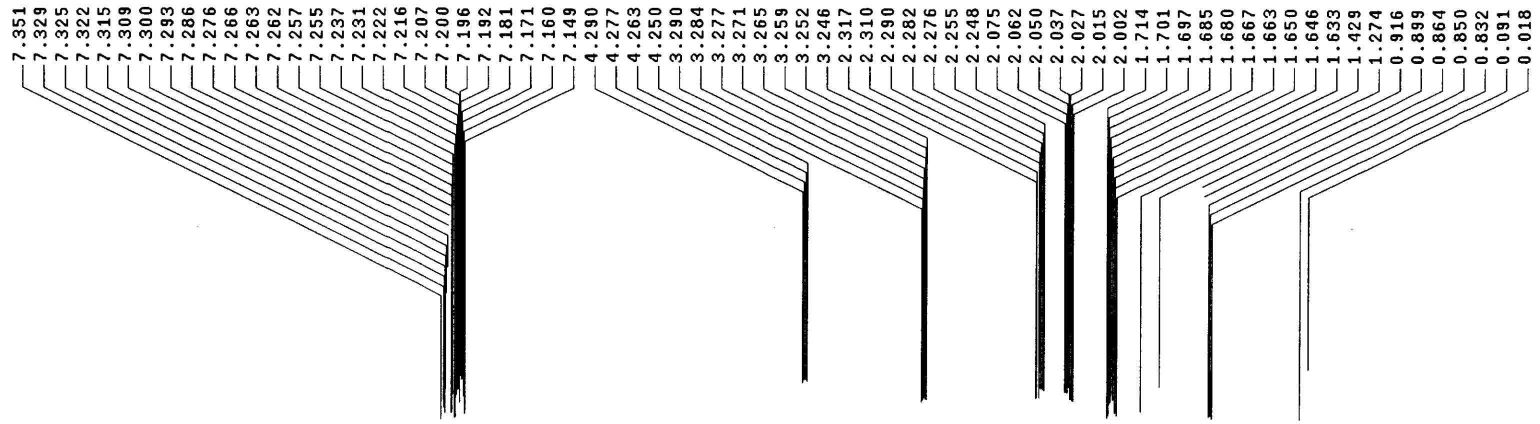
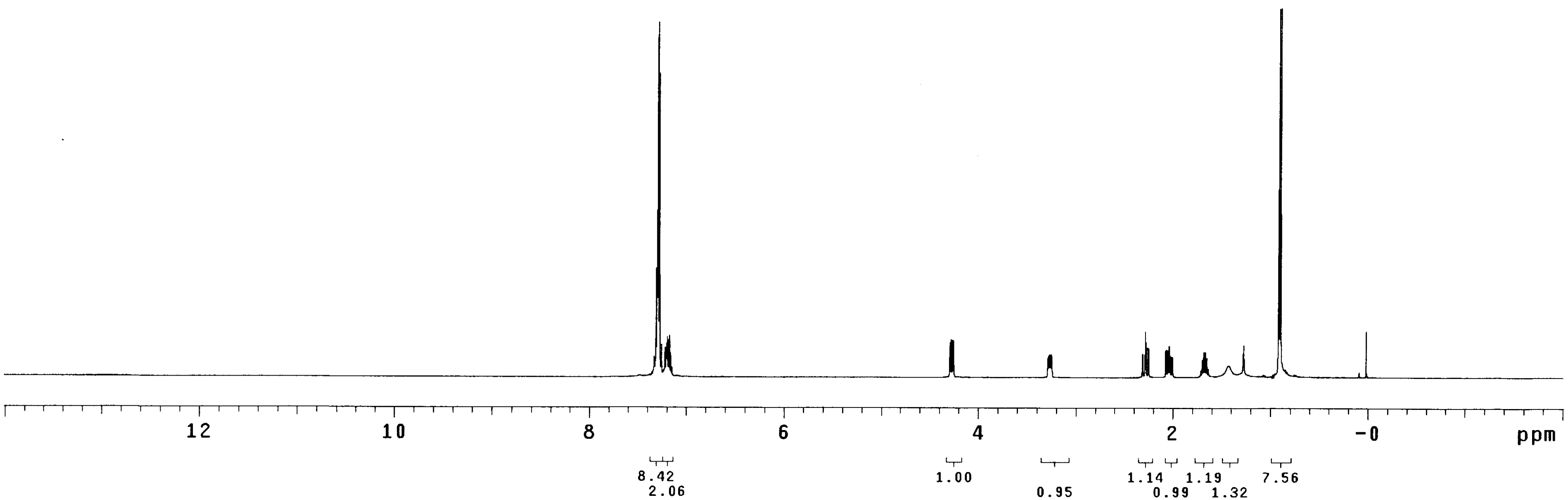
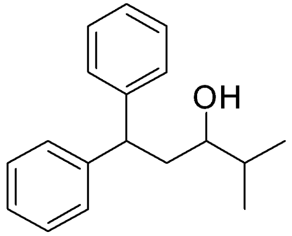
ppm



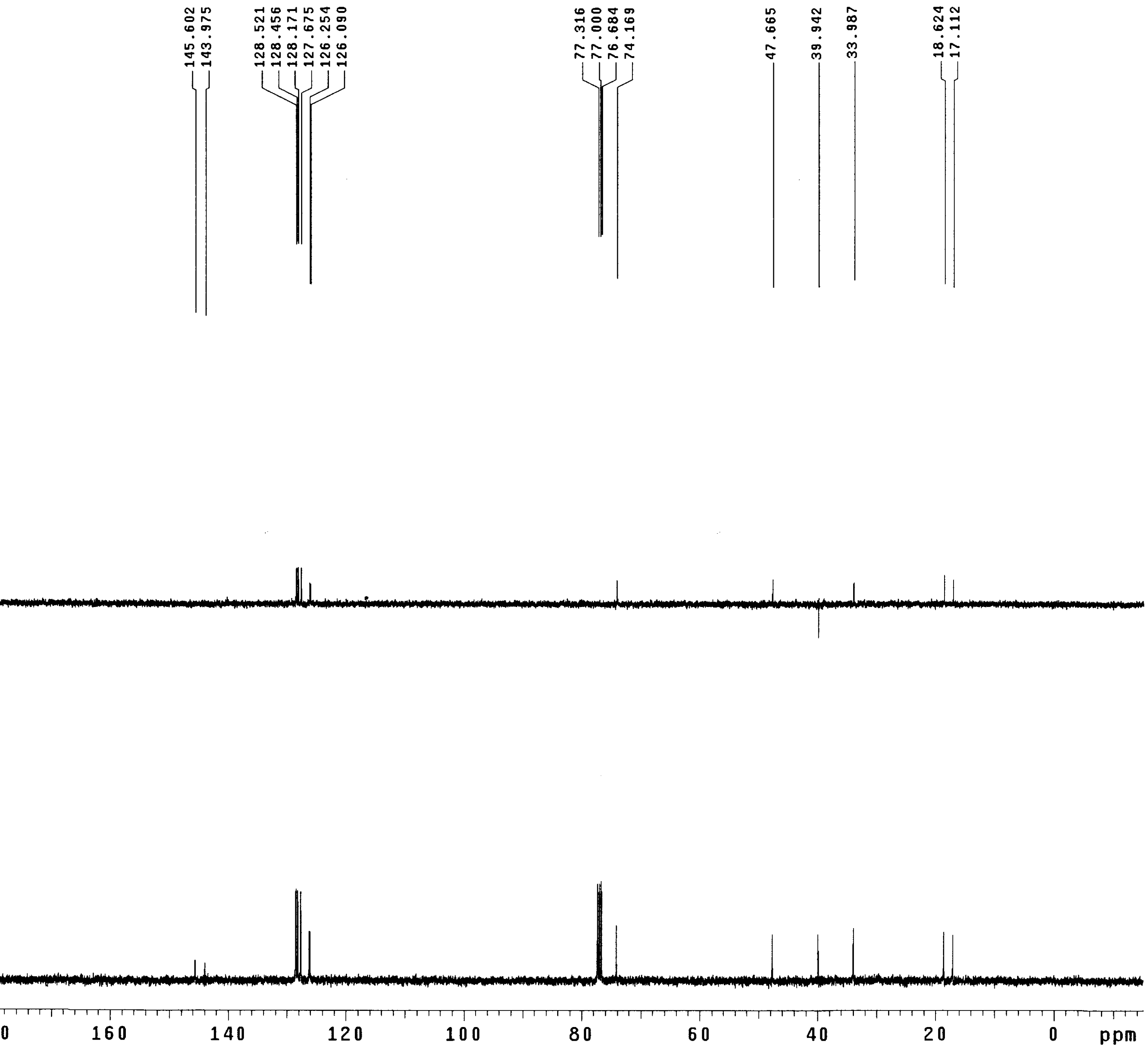
3ac







3ac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47.665
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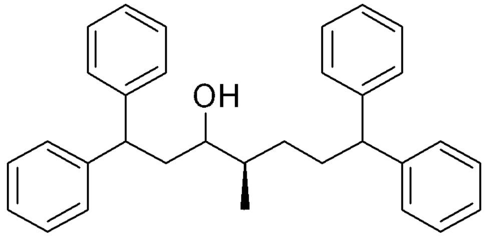
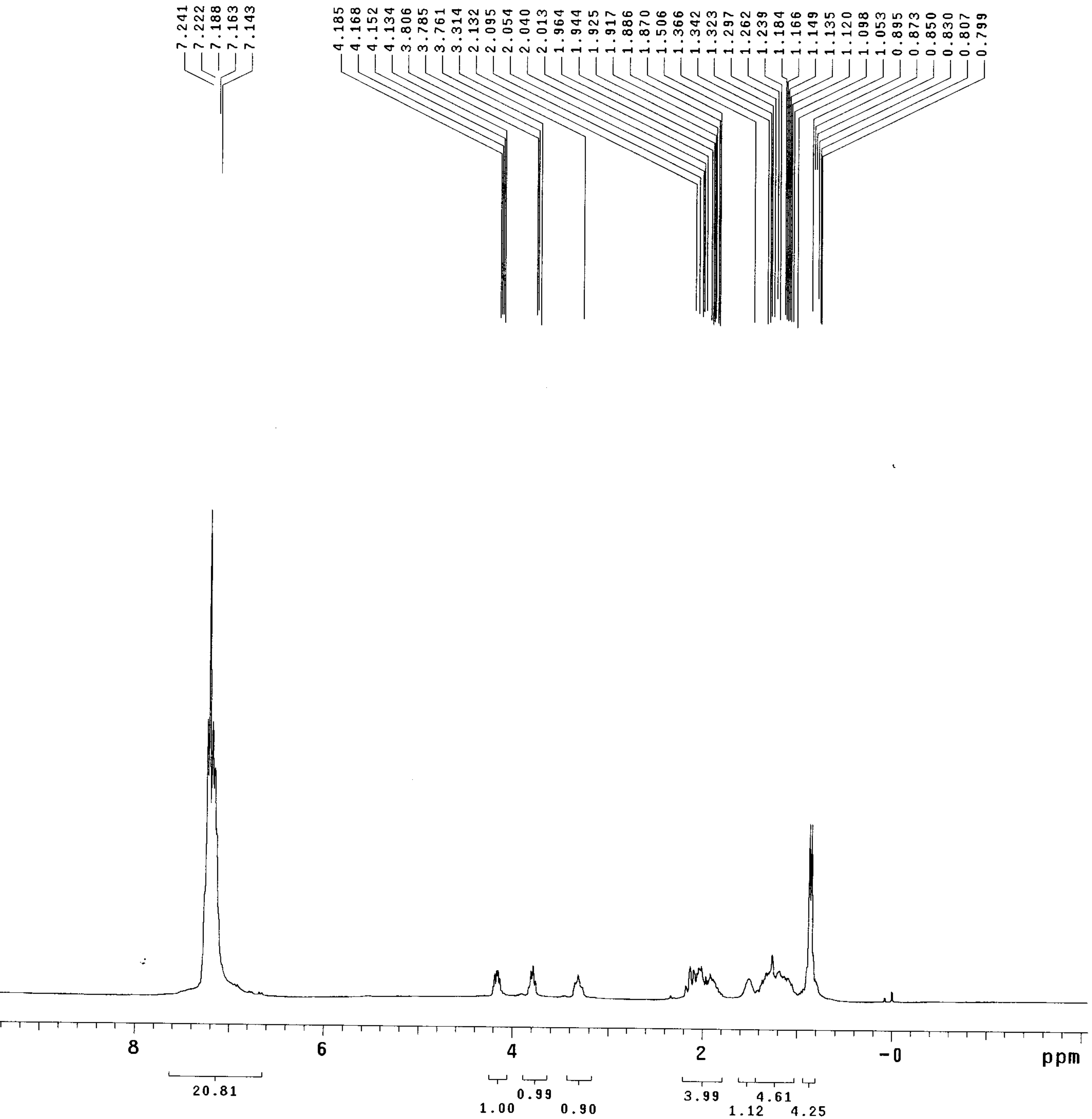
ppm


OH

3ad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4.152
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3.314
2.132
2.095
2.054
2.040
2.013
1.964
1.944
1.925
1.917
1.886
1.870
1.506
1.366
1.342
1.323
1.297
1.262
1.239
1.184
1.166
1.149
1.135
1.120
1.098
1.053
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0.850
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0.807
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0.90
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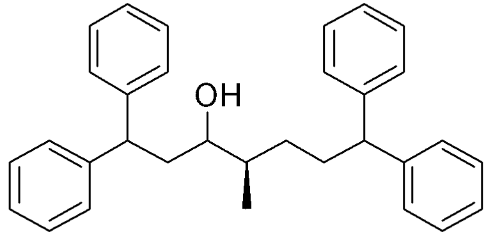
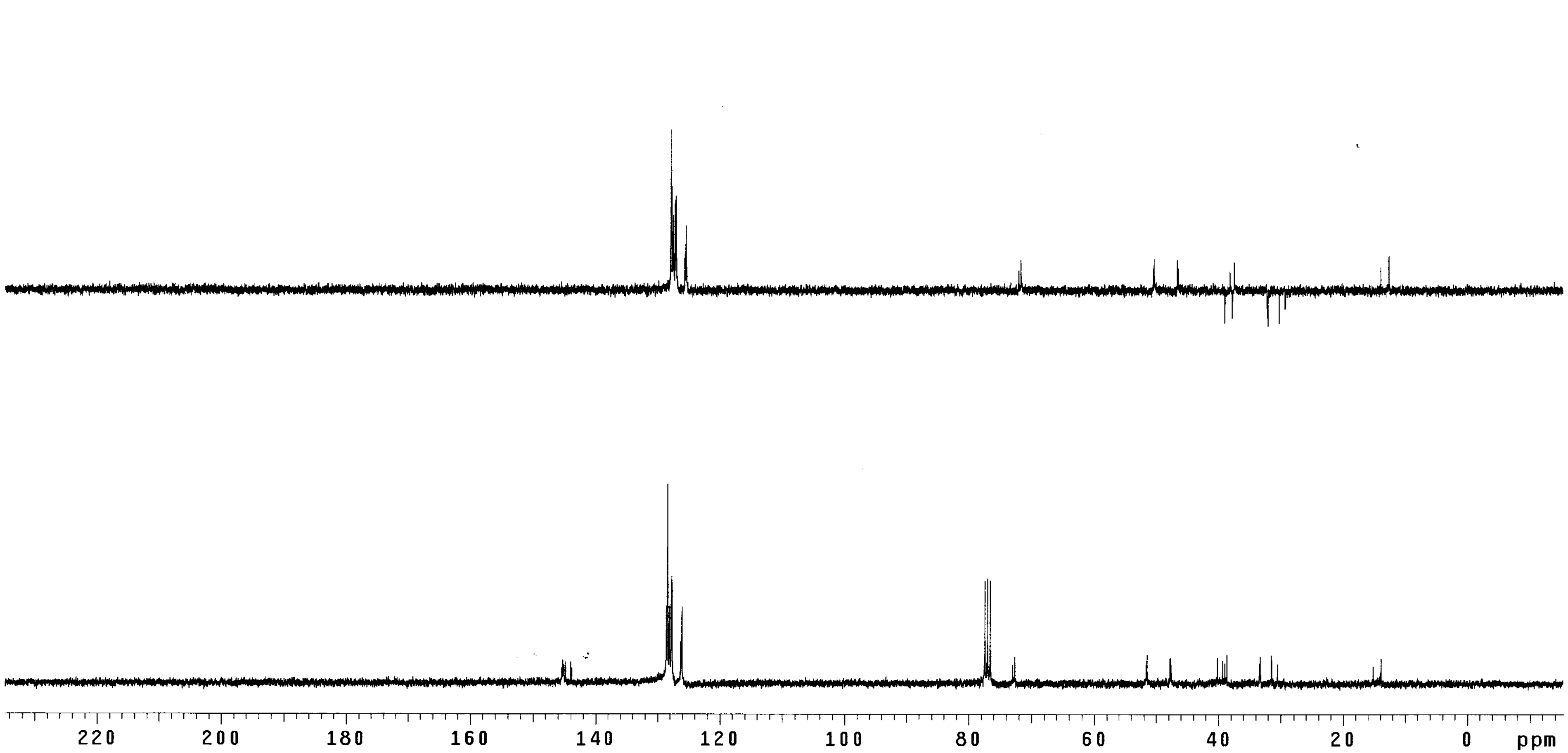
4.25


OH

3ad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73.036
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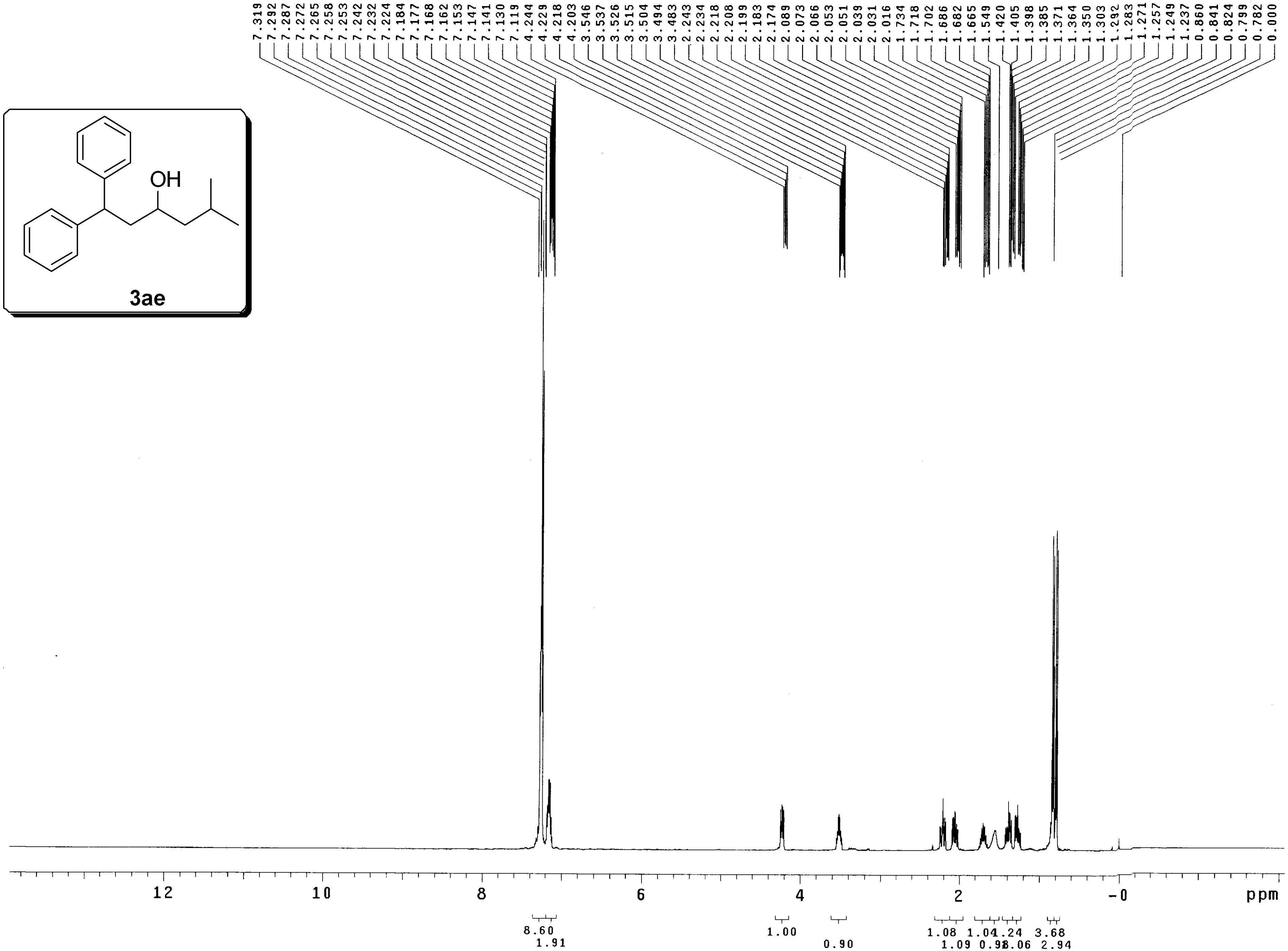
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7.162
7.153
7.147
7.141
7.130
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4.229
4.218
4.203
3.546
3.537
3.526
3.515
3.504
3.494
3.483
2.243
2.234
2.218
2.208
2.199
2.183
2.174
2.089
2.073
2.066
2.053
2.051
2.039
2.031
2.016
1.734
1.718
1.702
1.686
1.682
1.665
1.549
1.420
1.405
1.398
1.385
1.371
1.364
1.350
1.303
1.292
1.283
1.271
1.257
1.249
1.237
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0.824
0.799
0.782
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1.09
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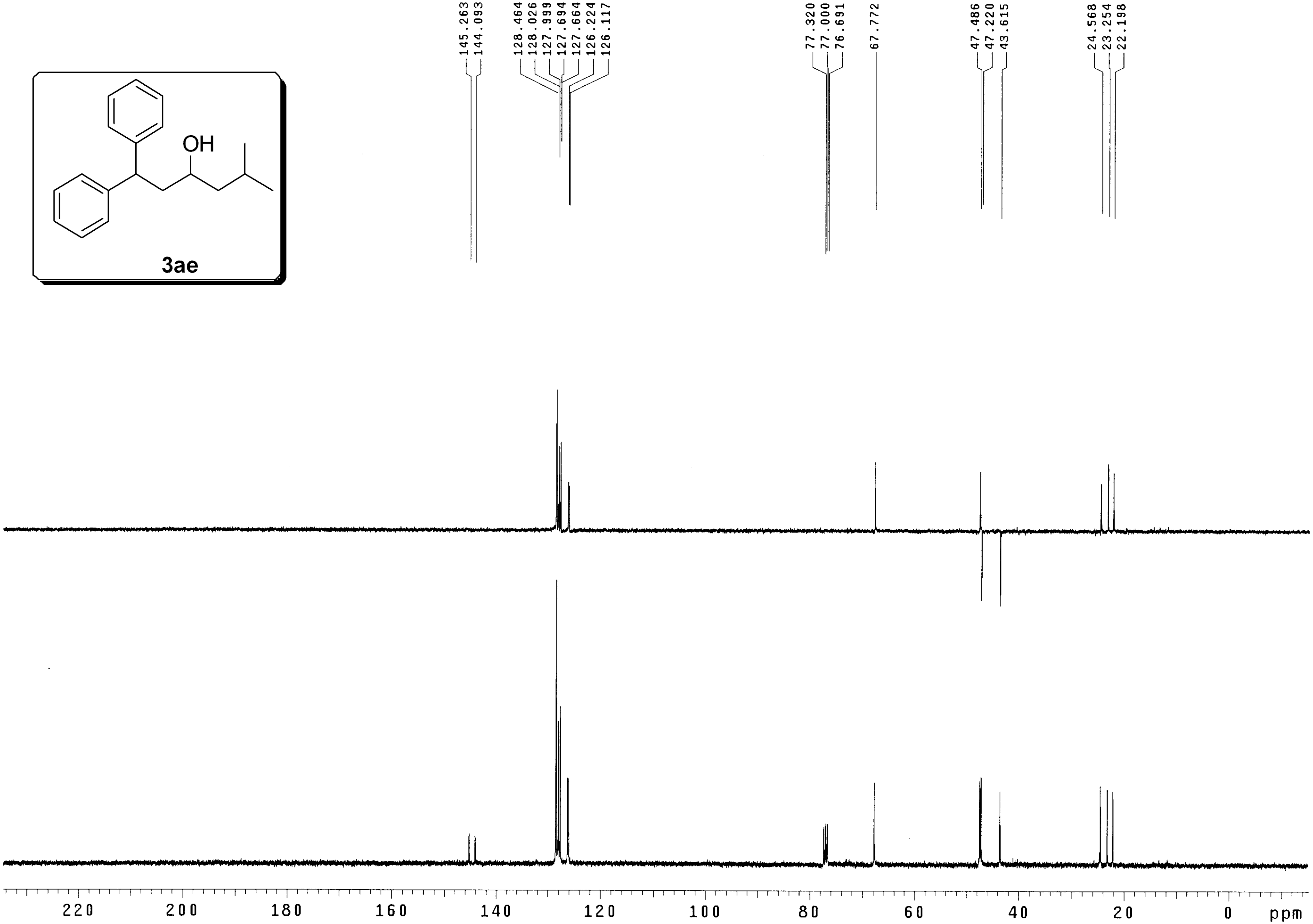
2.94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47.220
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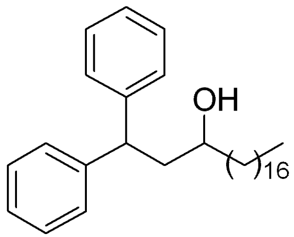
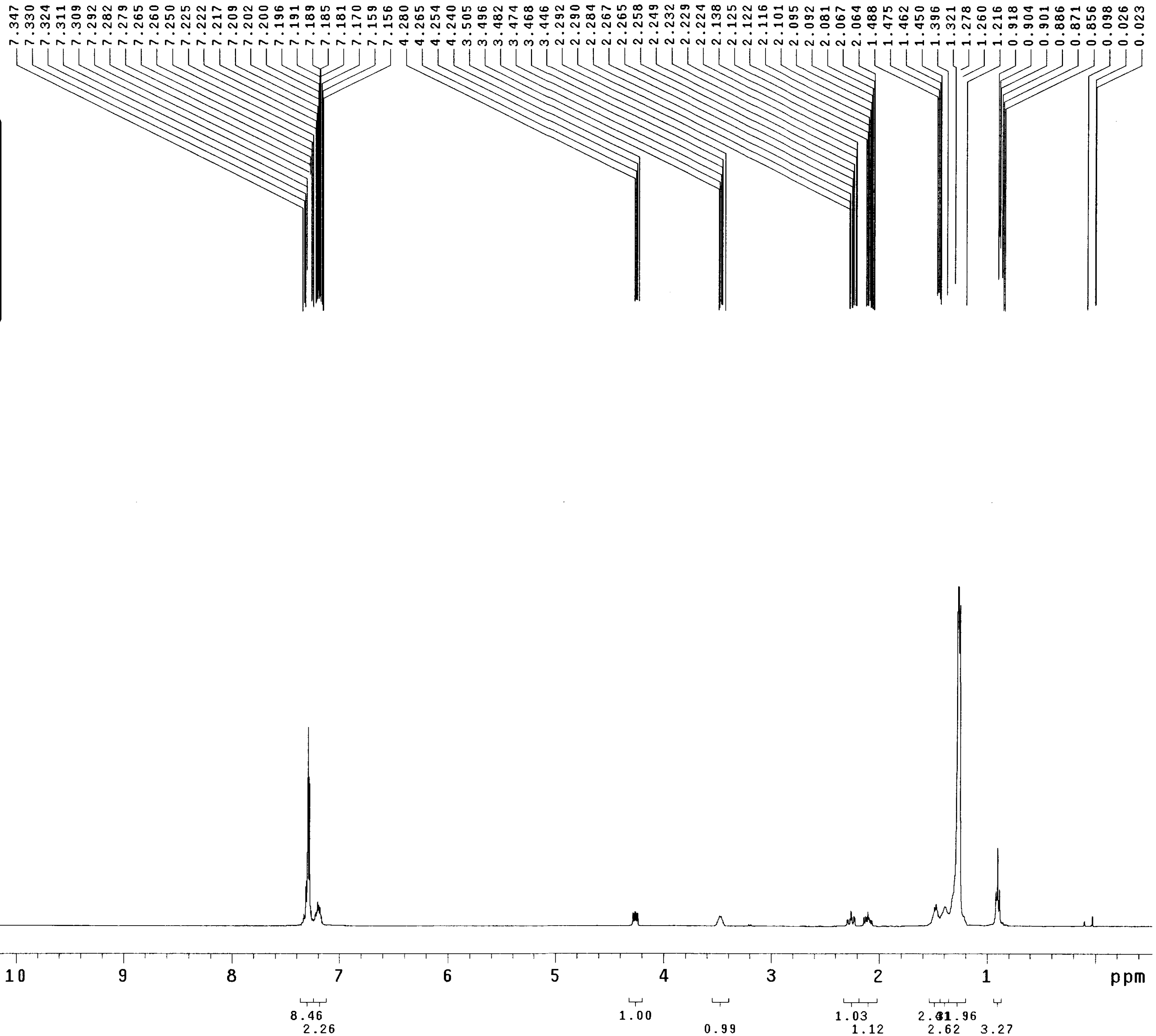

OH
16

3af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1.260
1.216
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0.901
0.886
0.871
0.856
0.098
0.026
0.023
12
11
10
9
8
7
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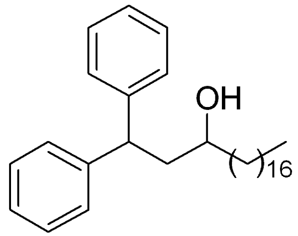
OH
16

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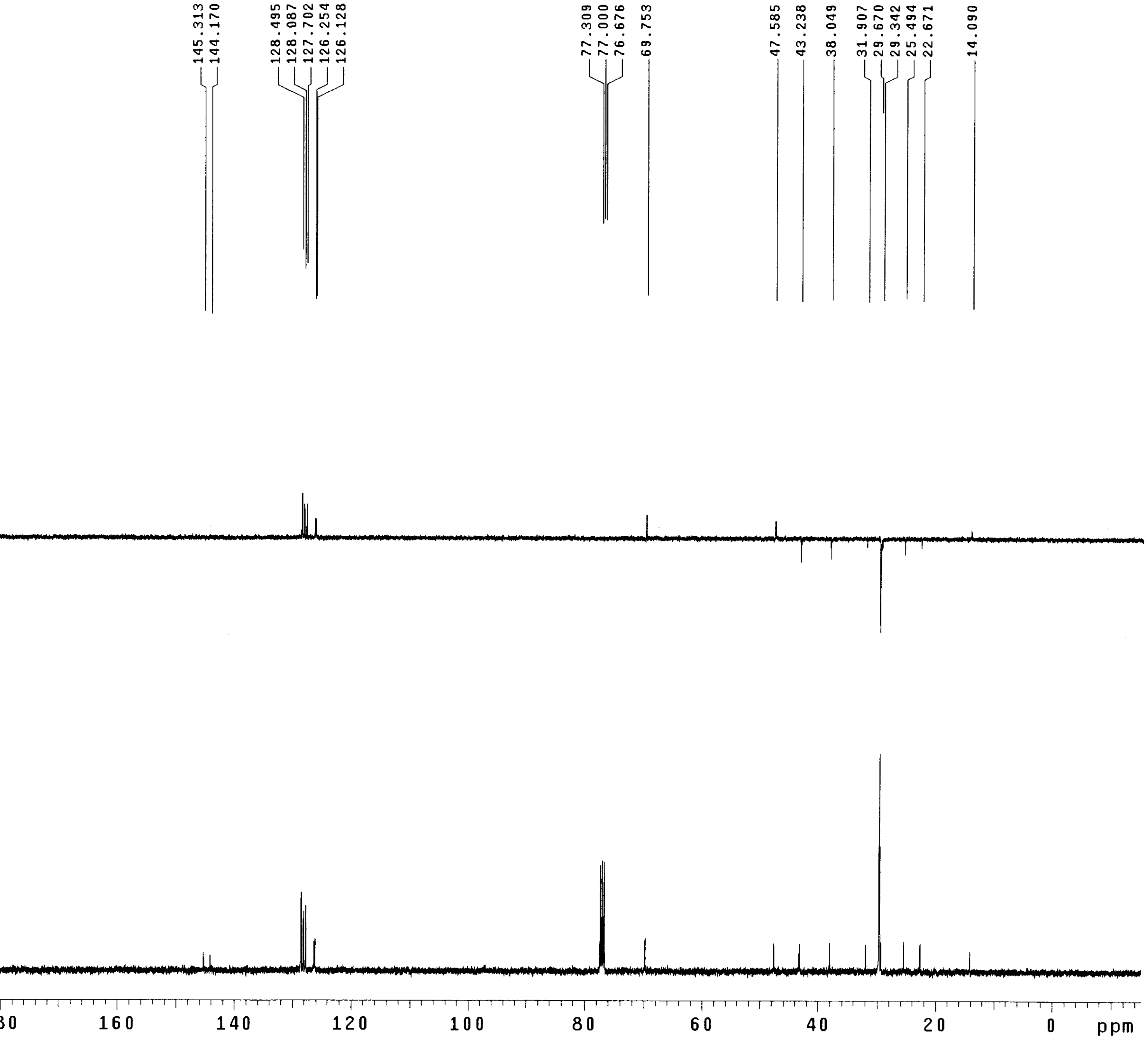
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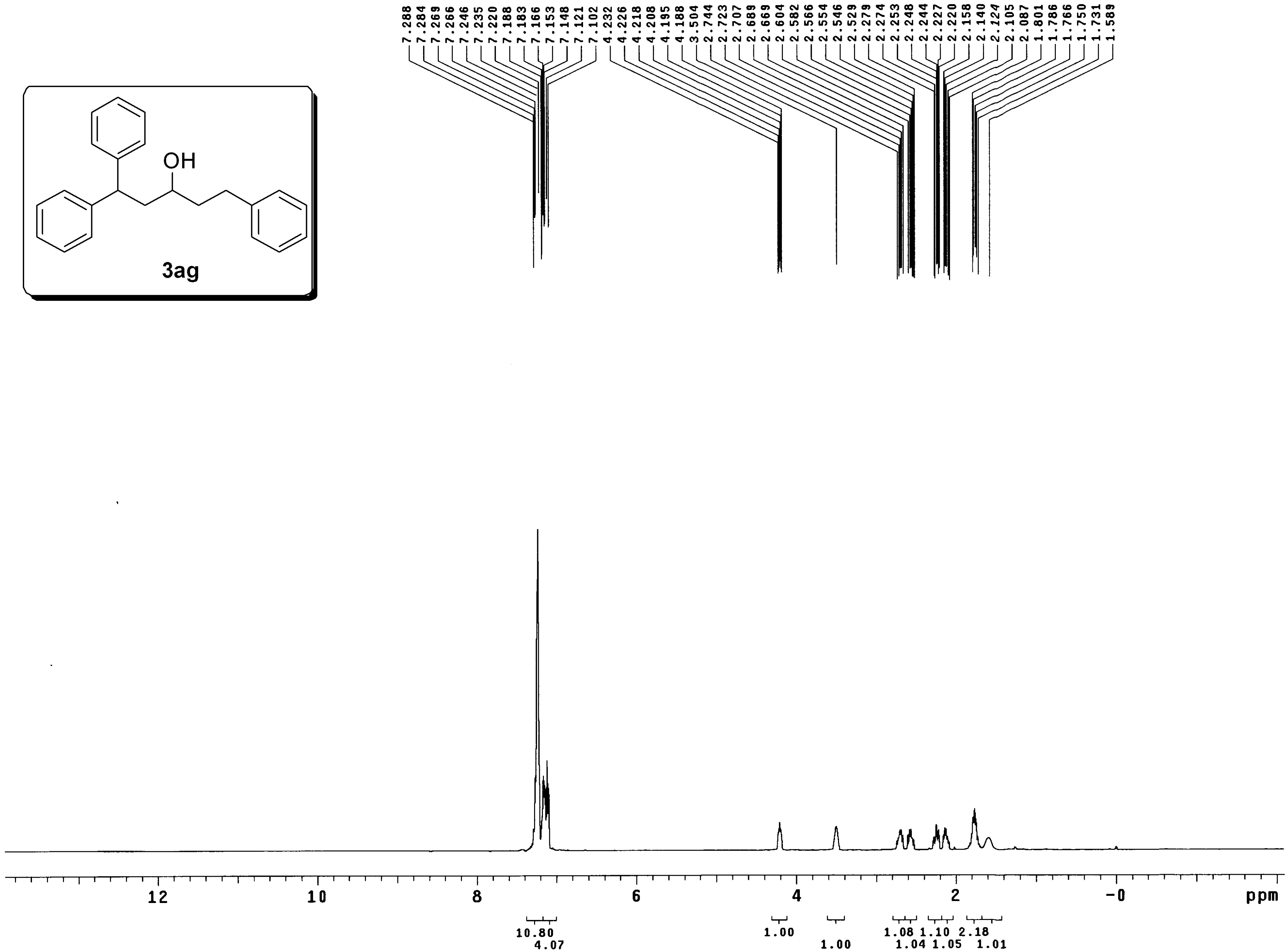
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2.566
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2.244
2.227
2.220
2.158
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2.124
2.105
2.087
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1.766
1.750
1.731
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3ag
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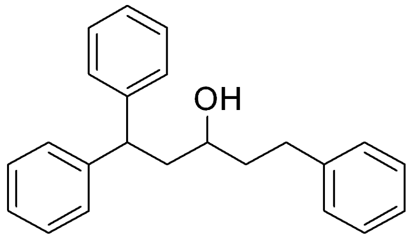

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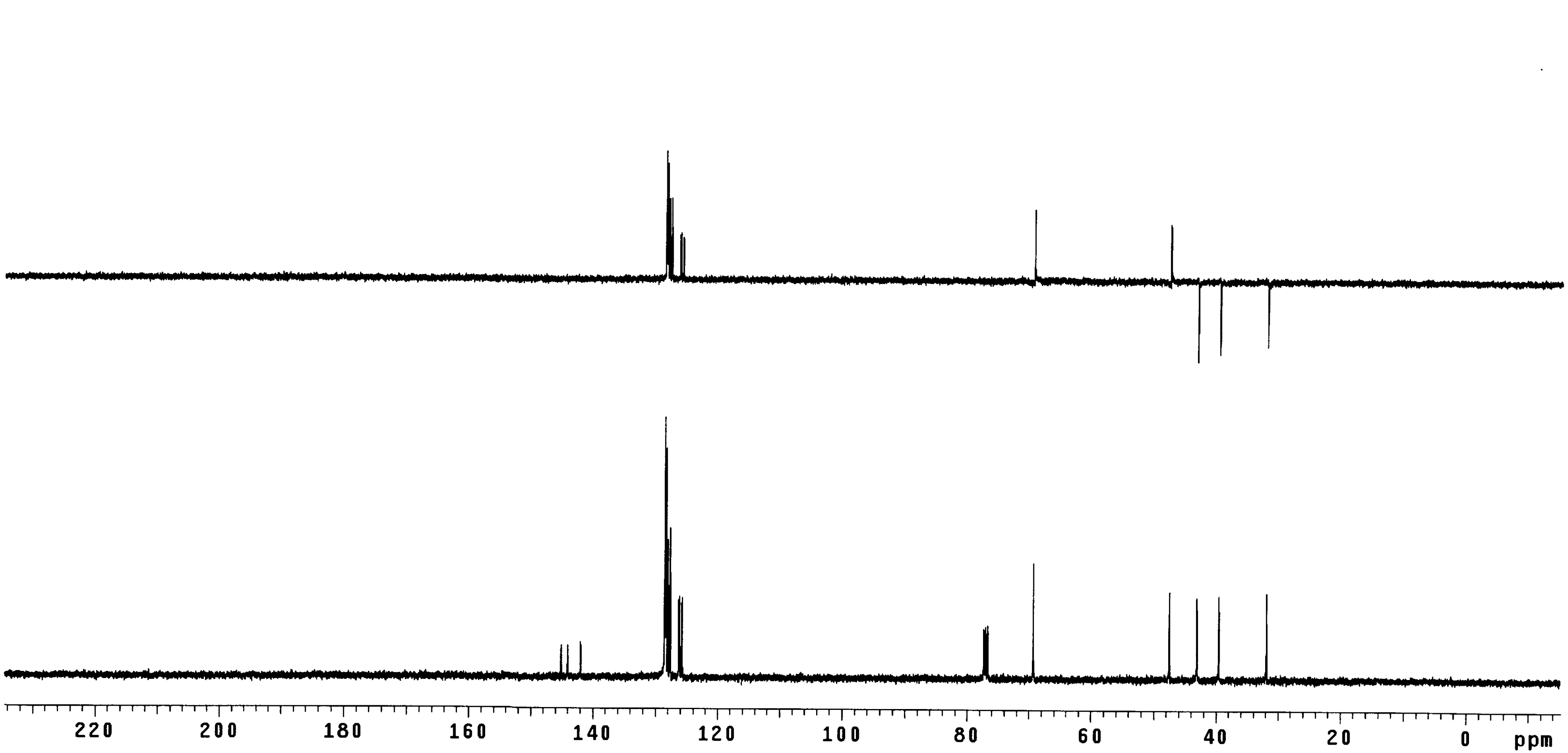
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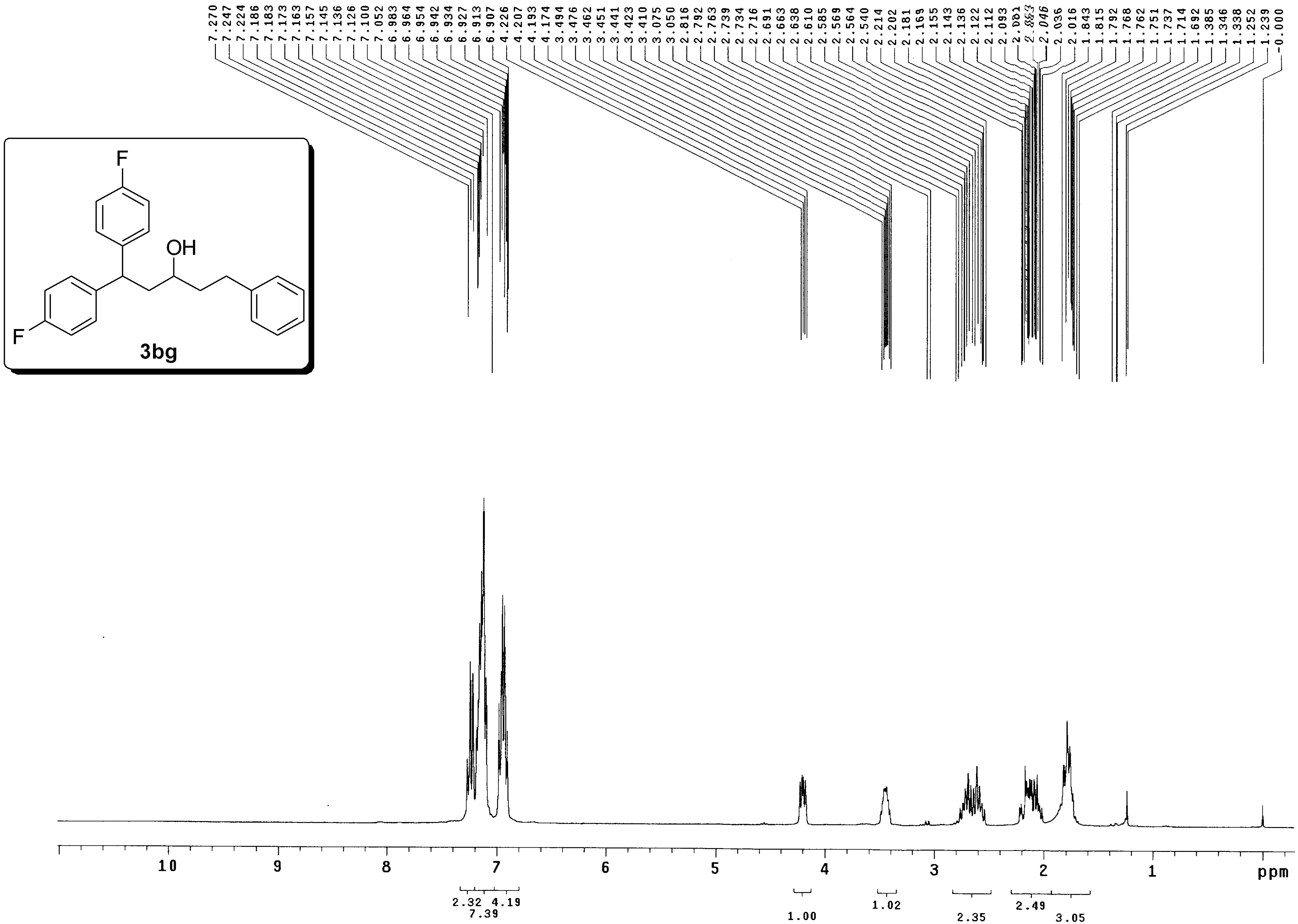
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F
OH
F
3bg
10
9
8
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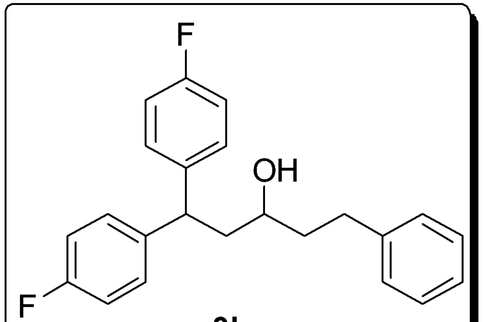
2.49
3.05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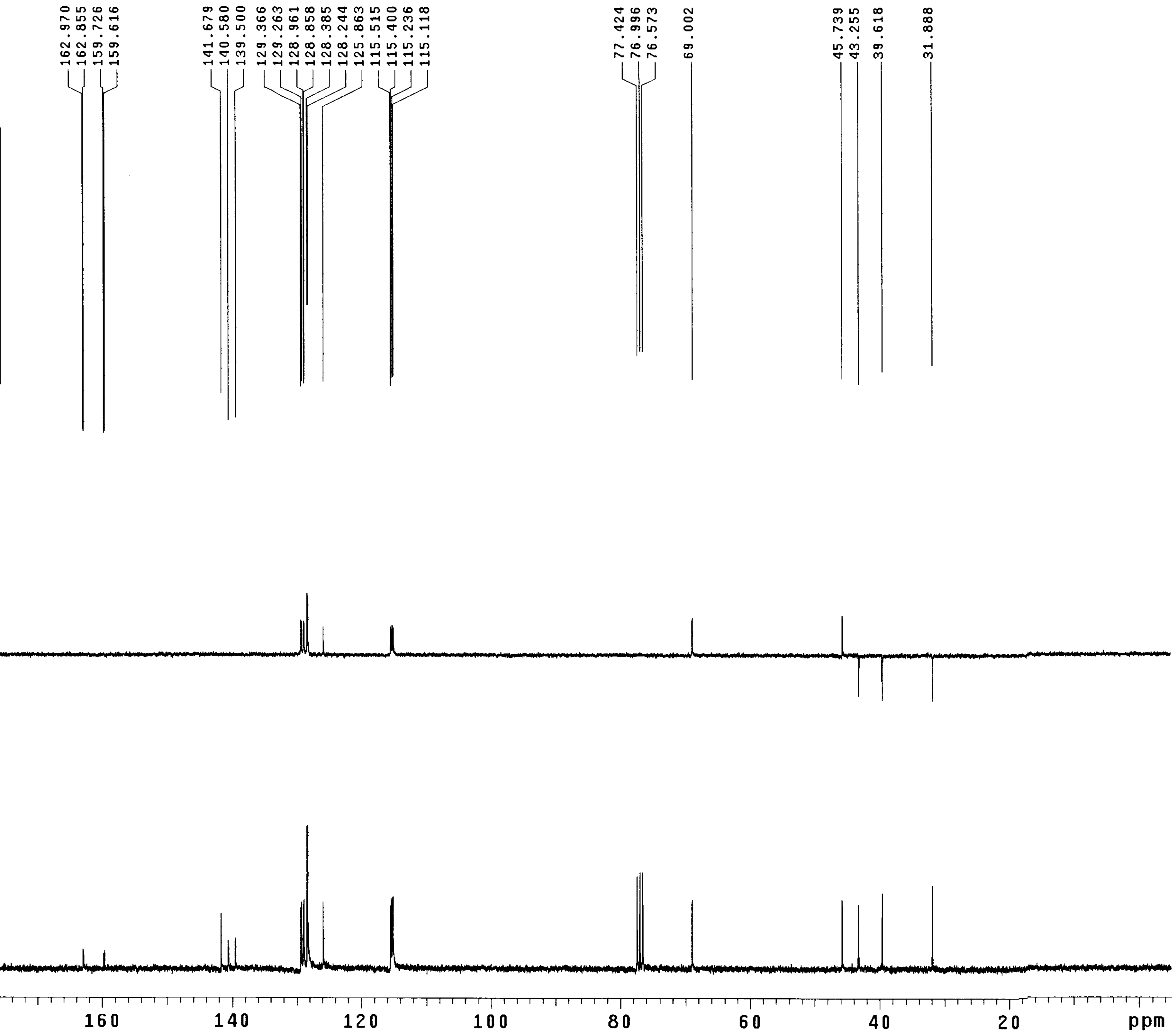
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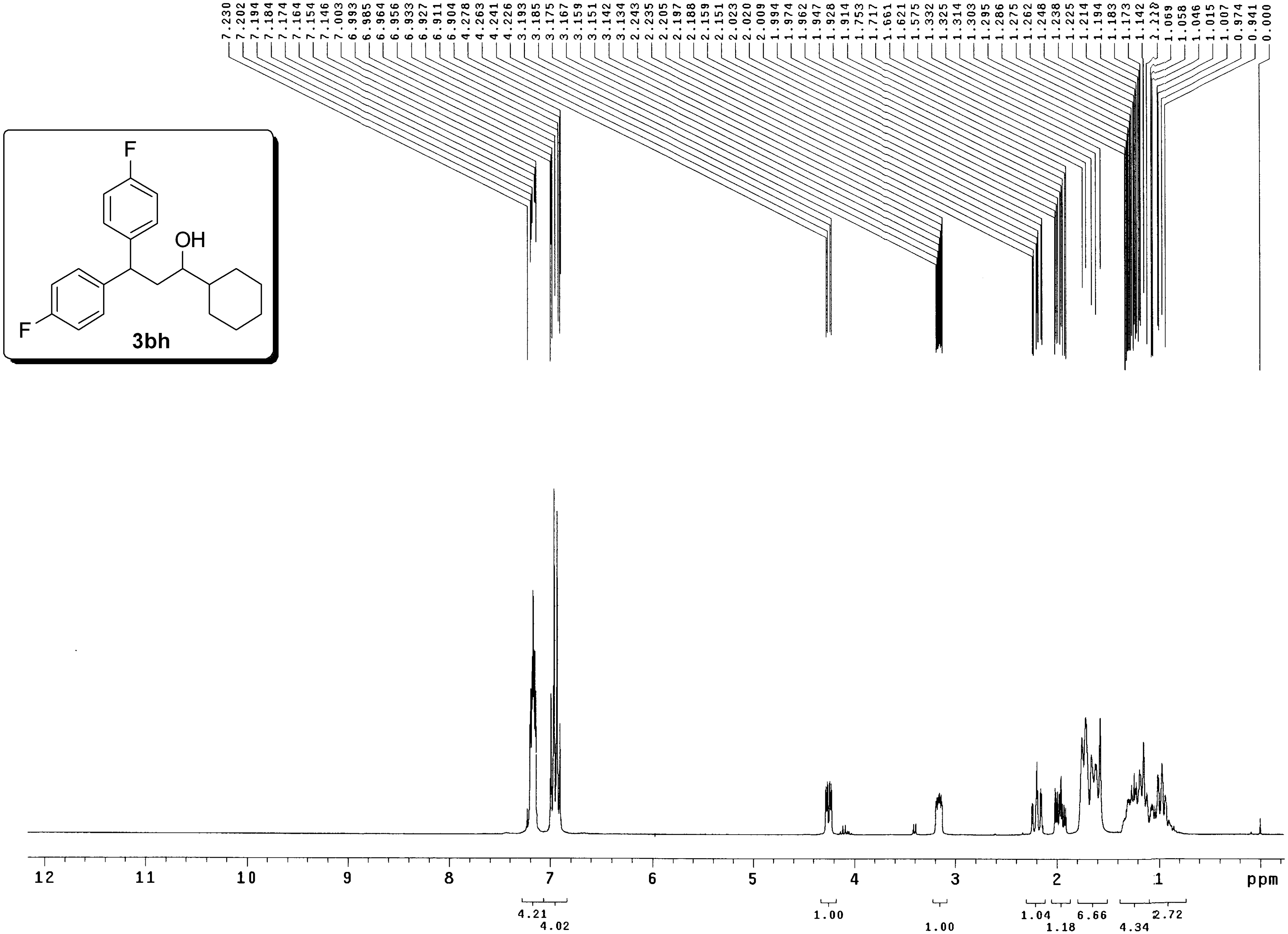
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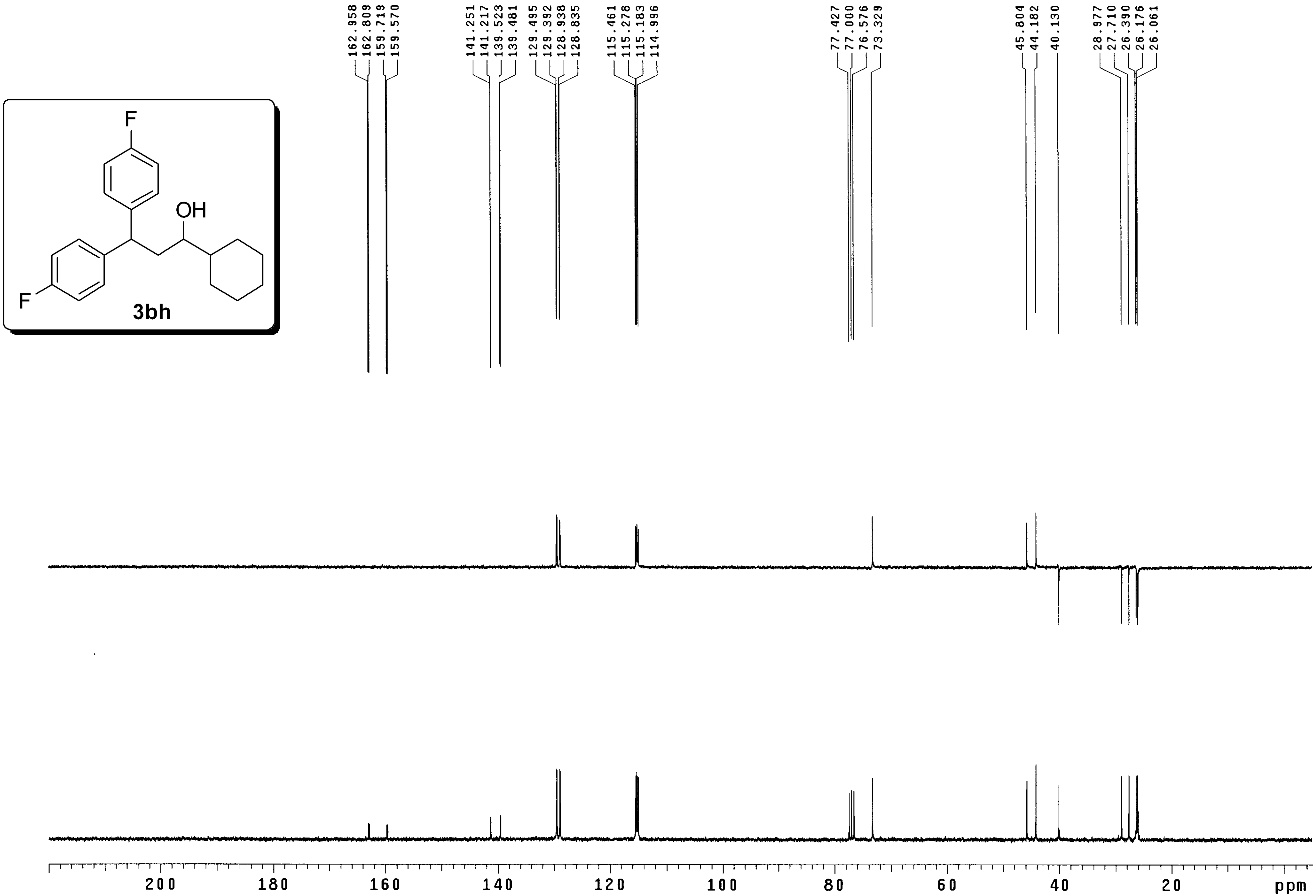
12 11 10 9 8 7 6 5 4 3 2 1 ppm
4.21 4.02 1.00 1.00 1.04 1.18 6.66 4.34 2.72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115.183
114.996
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77.000
76.576
73.329
45.804
44.182
40.130
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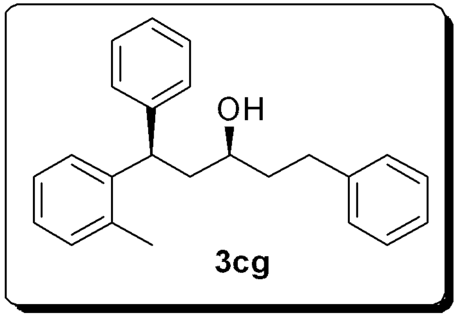
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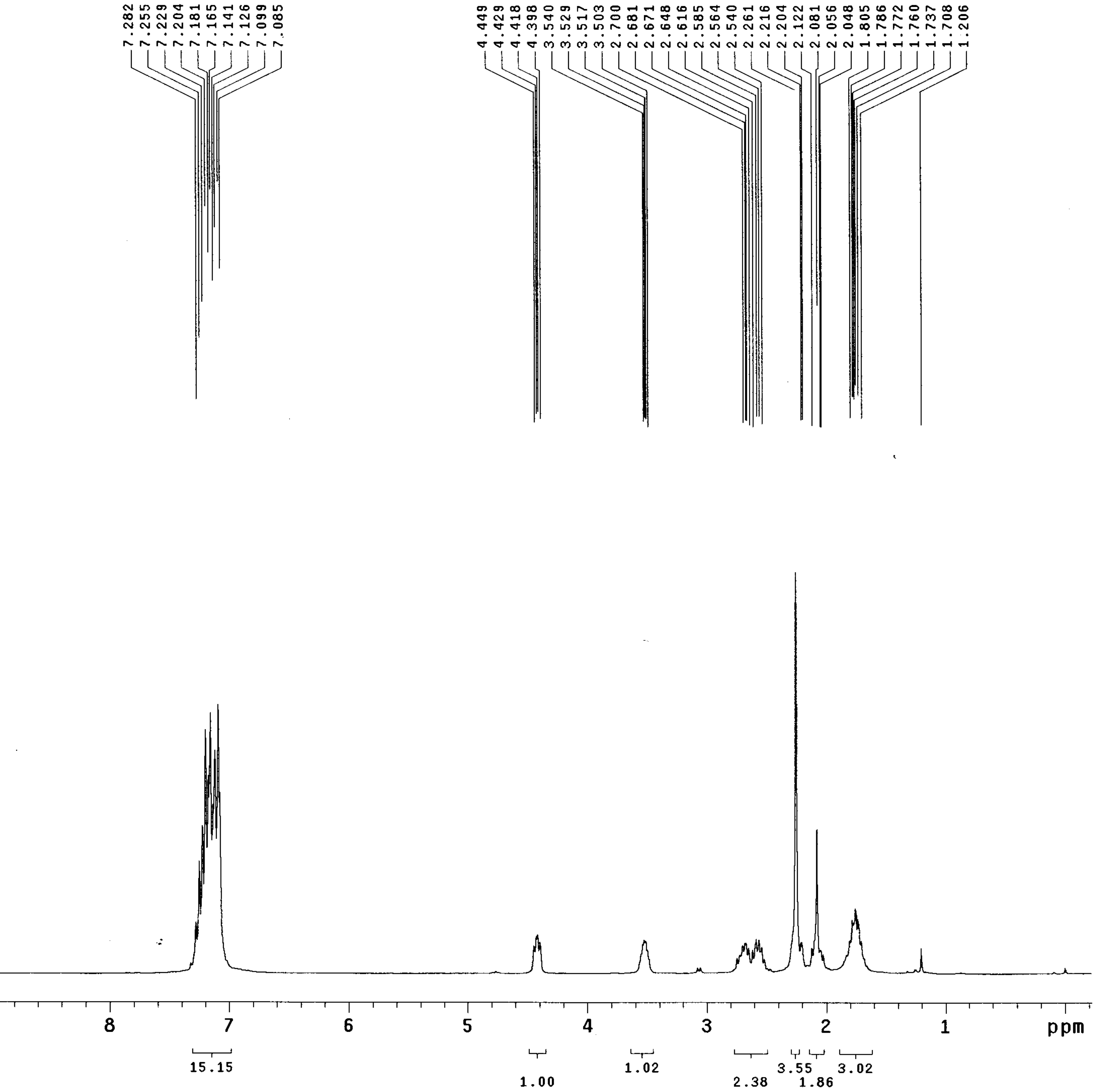
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3.517
3.503
2.700
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2.671
2.648
2.616
2.585
2.564
2.540
2.261
2.216
2.204
2.122
2.081
2.056
2.048
1.805
1.786
1.772
1.760
1.737
1.708
1.206
12
11
10
9
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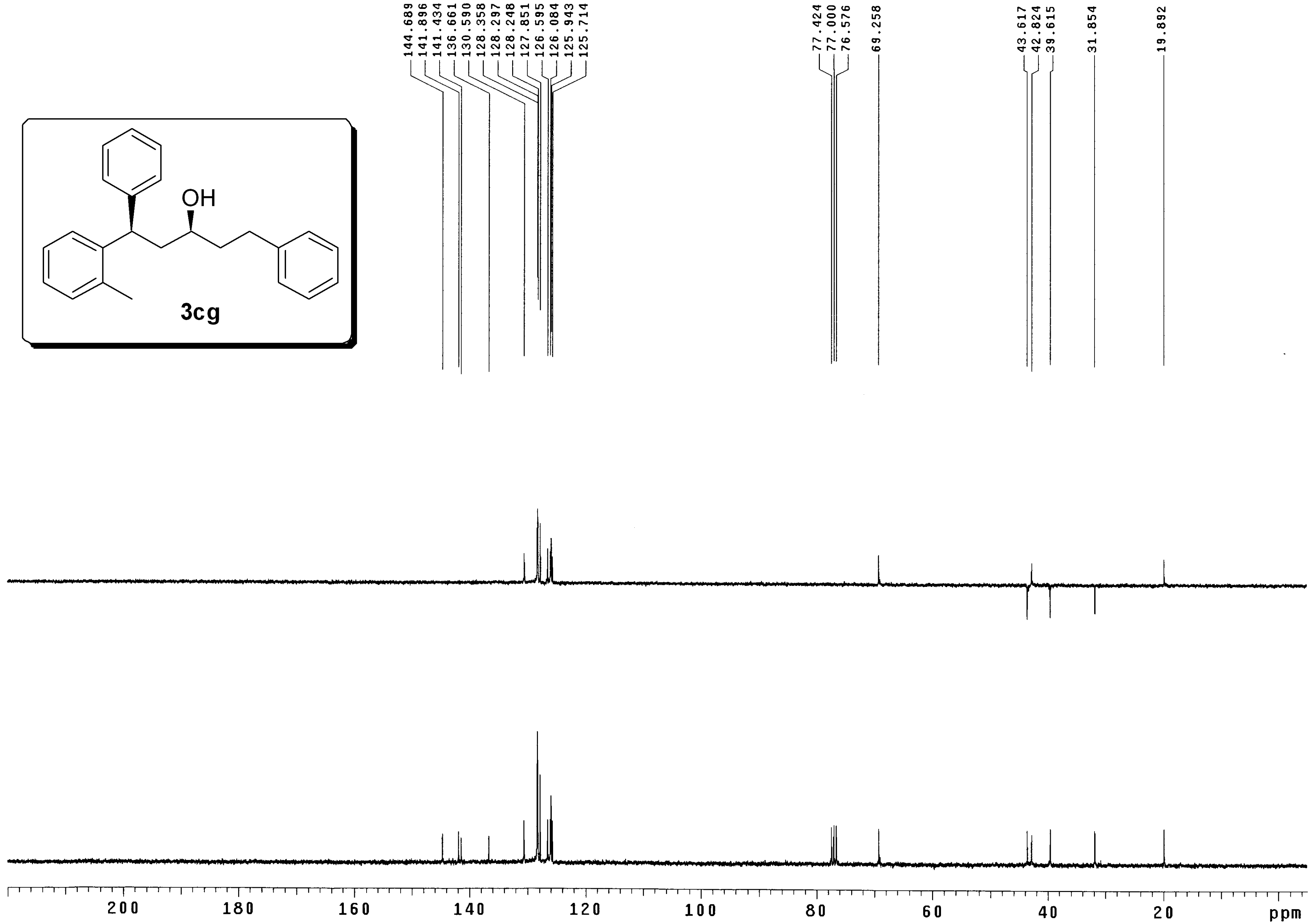
1.86
3.02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76.576
69.258
43.617
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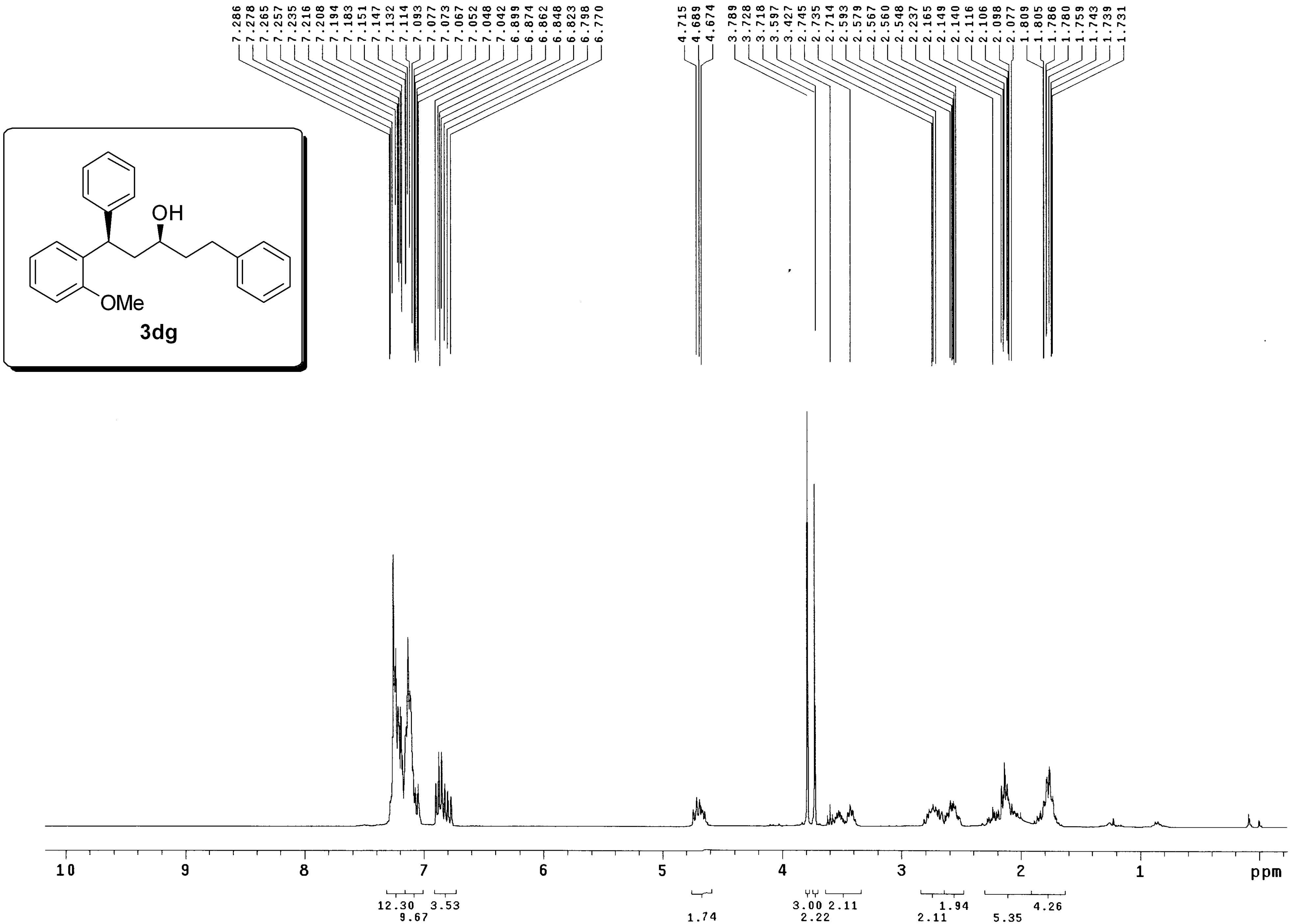
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4.715 4.689 4.674
3.789 3.728 3.718 3.597 3.427 2.745 2.735 2.714 2.593 2.579 2.567 2.560 2.548 2.237 2.165 2.149 2.140 2.116 2.106 2.098 2.077 1.809 1.805 1.786 1.780 1.759 1.743 1.739 1.731
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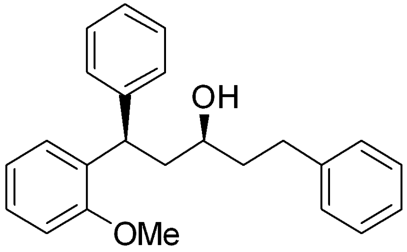
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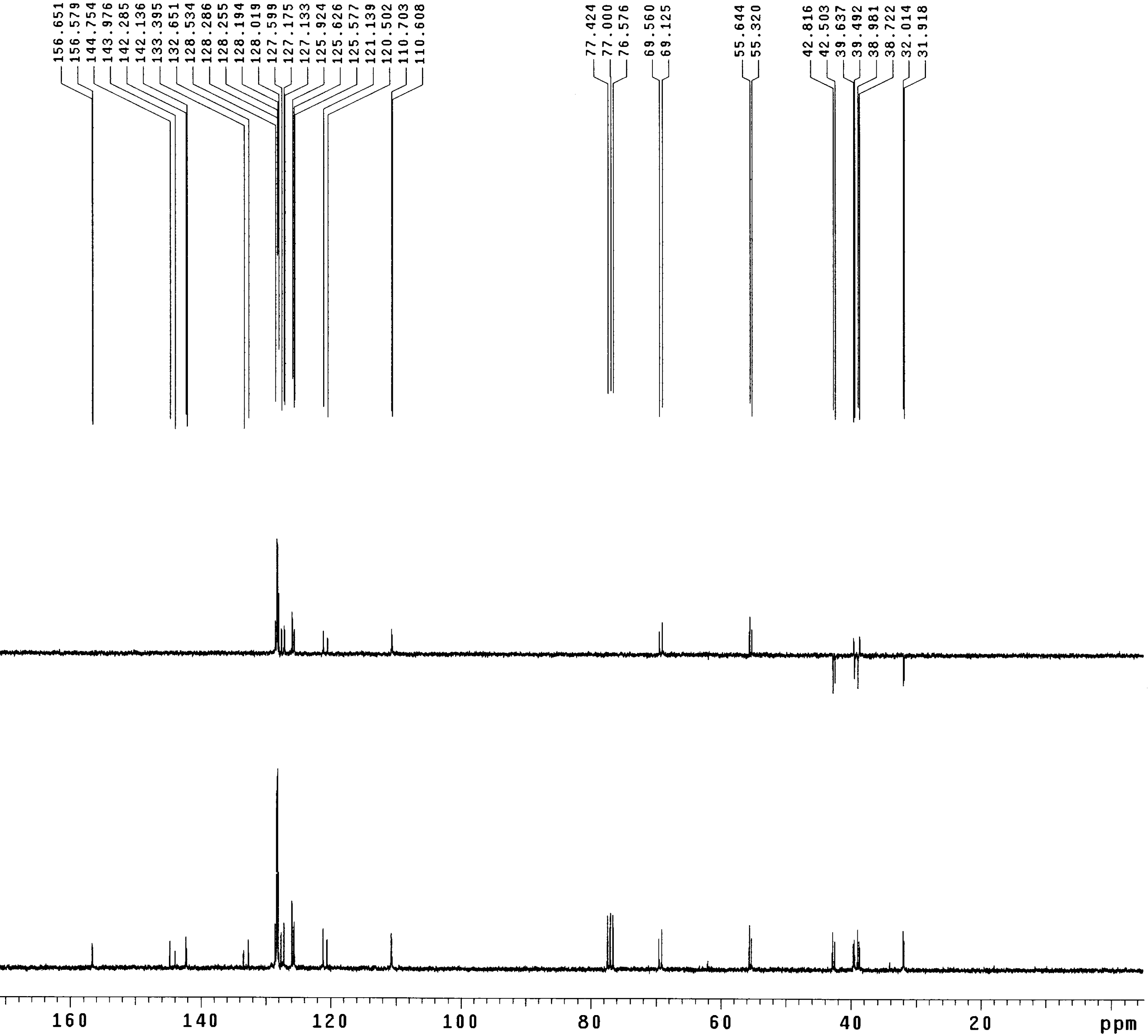
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77.000
76.576
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69.125
55.644
55.320
42.816
42.503
39.637
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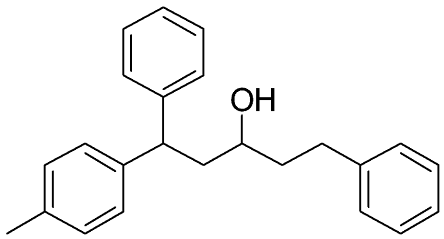

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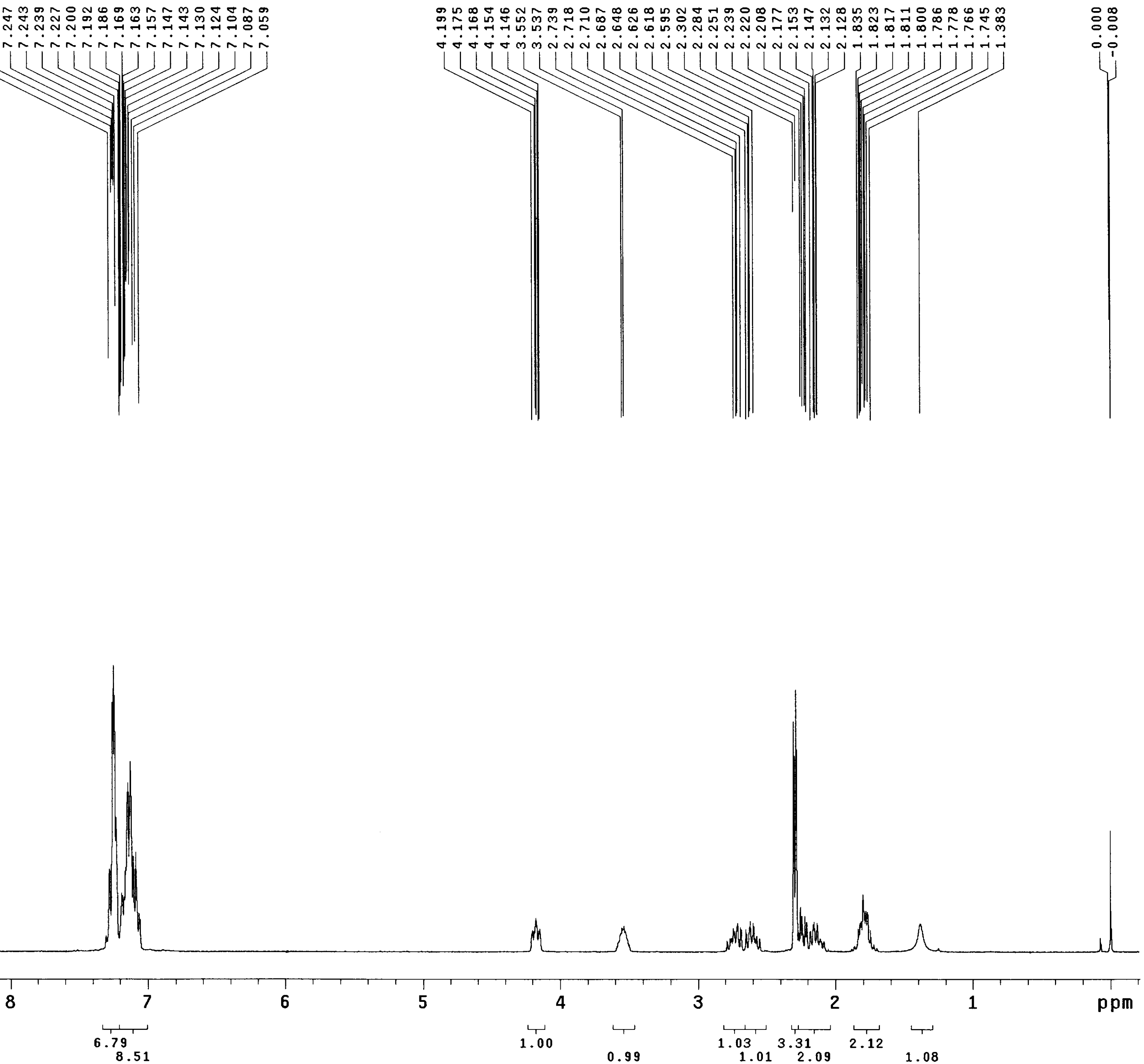
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4.154
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2.687
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2.208
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2.147
2.132
2.128
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1.823
1.817
1.811
1.800
1.786
1.778
1.766
1.745
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0.000
-0.008
10
9
8
7
6
5
4
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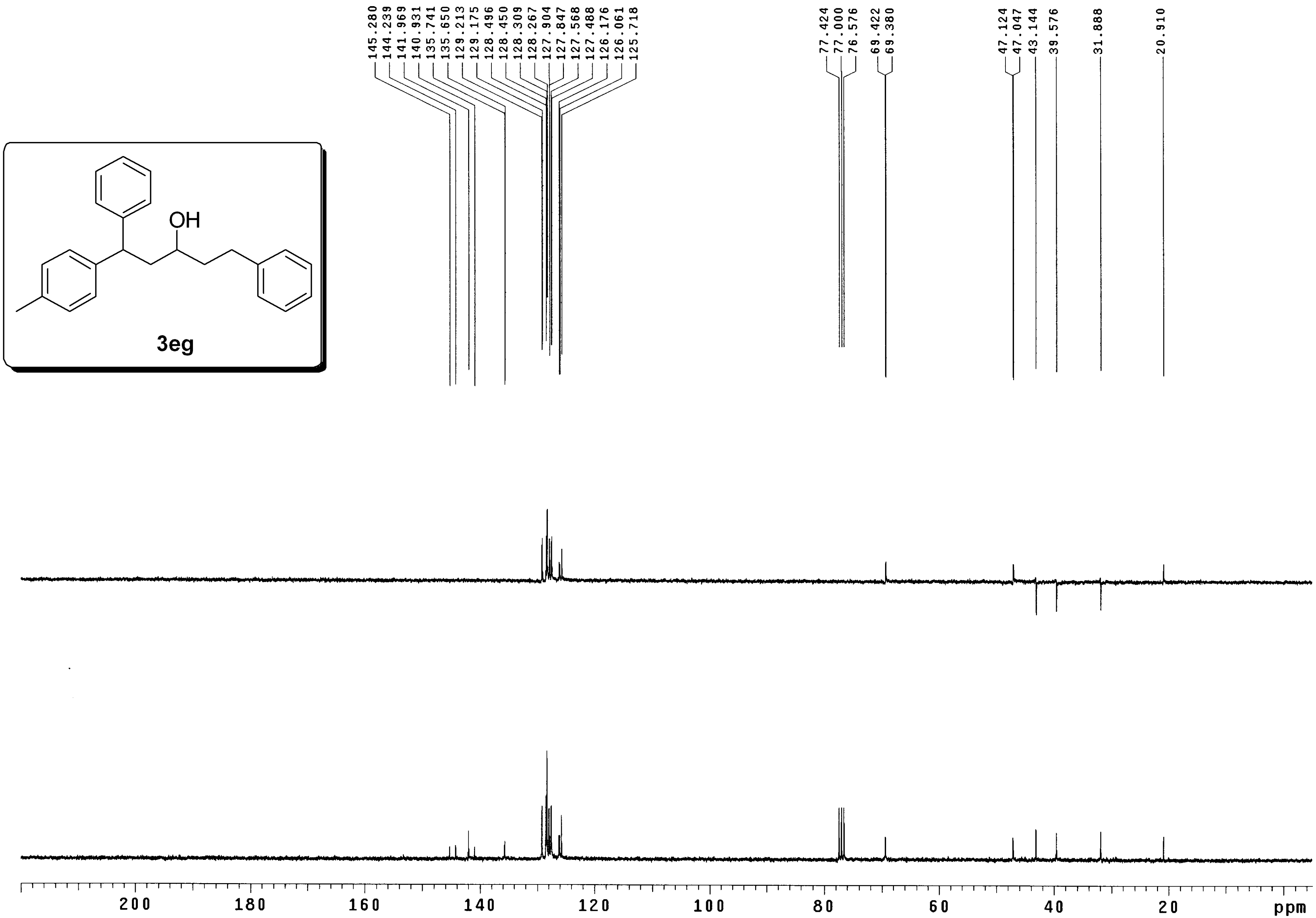
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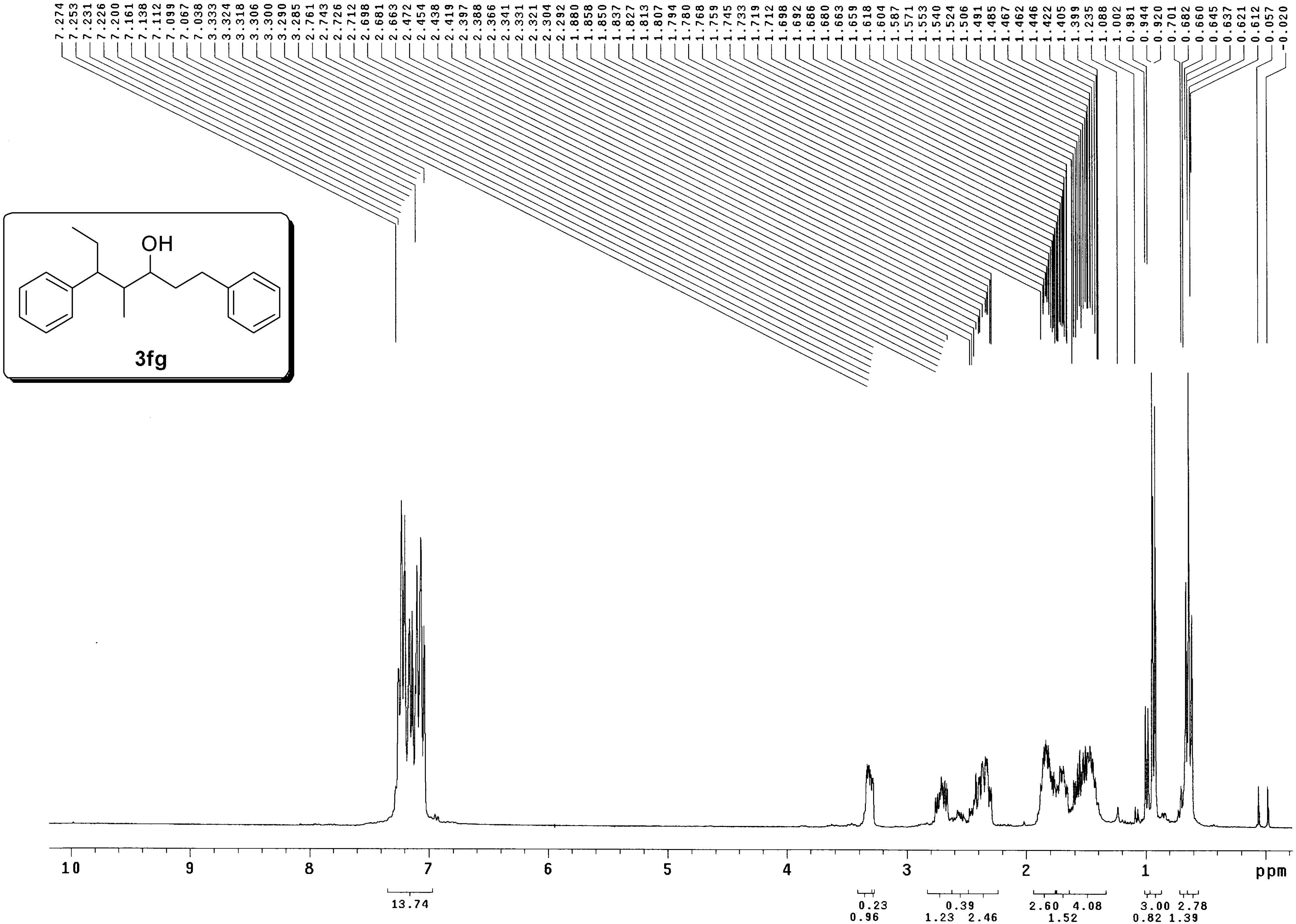
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7
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4
3
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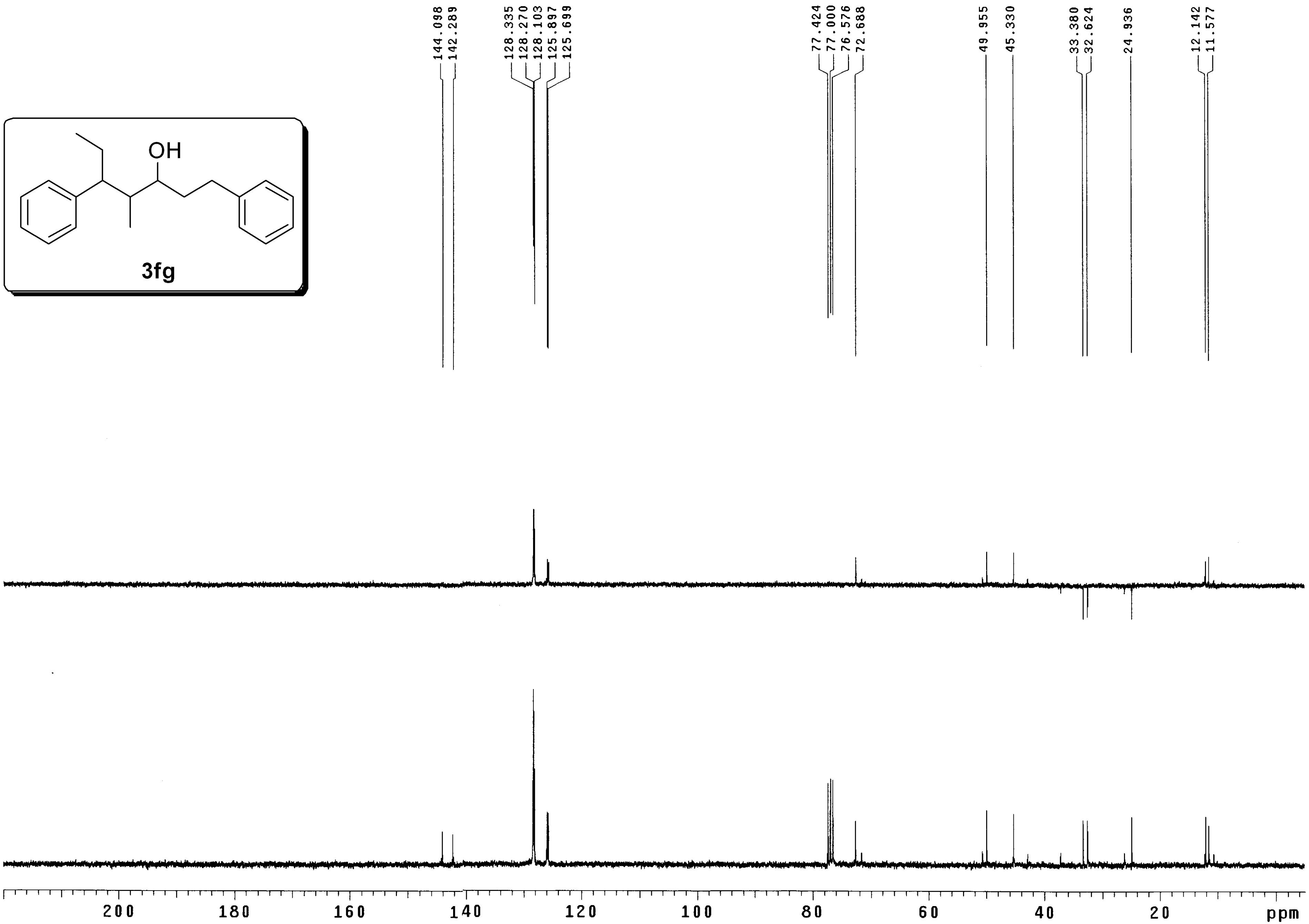
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33.380
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11.577
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20
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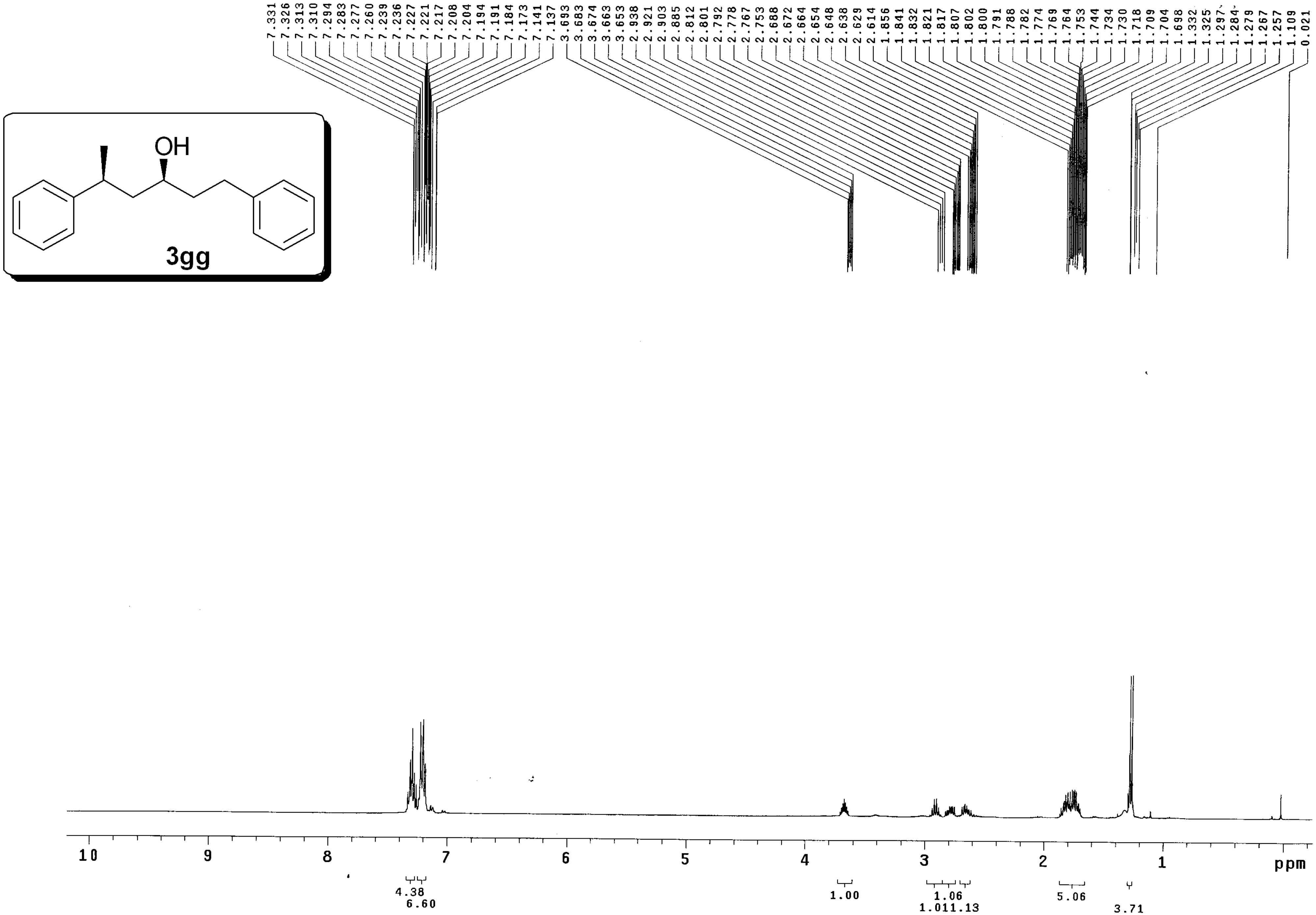
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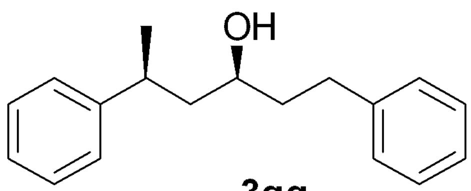
OH

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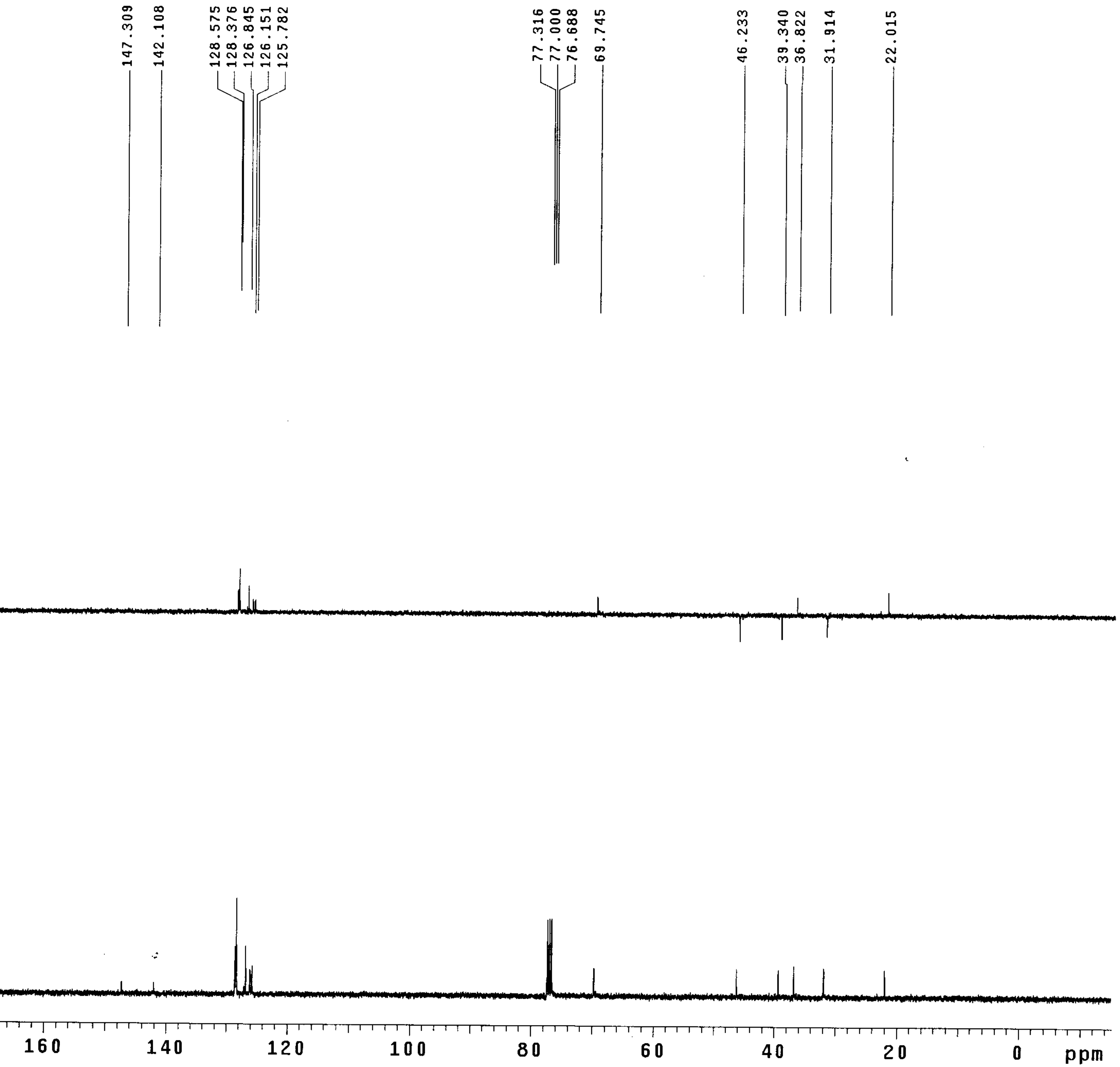
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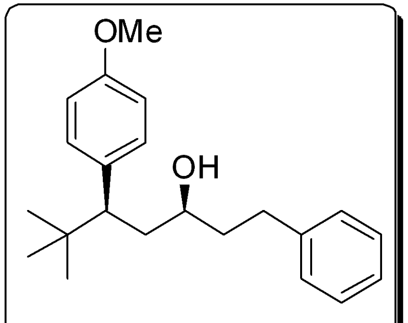

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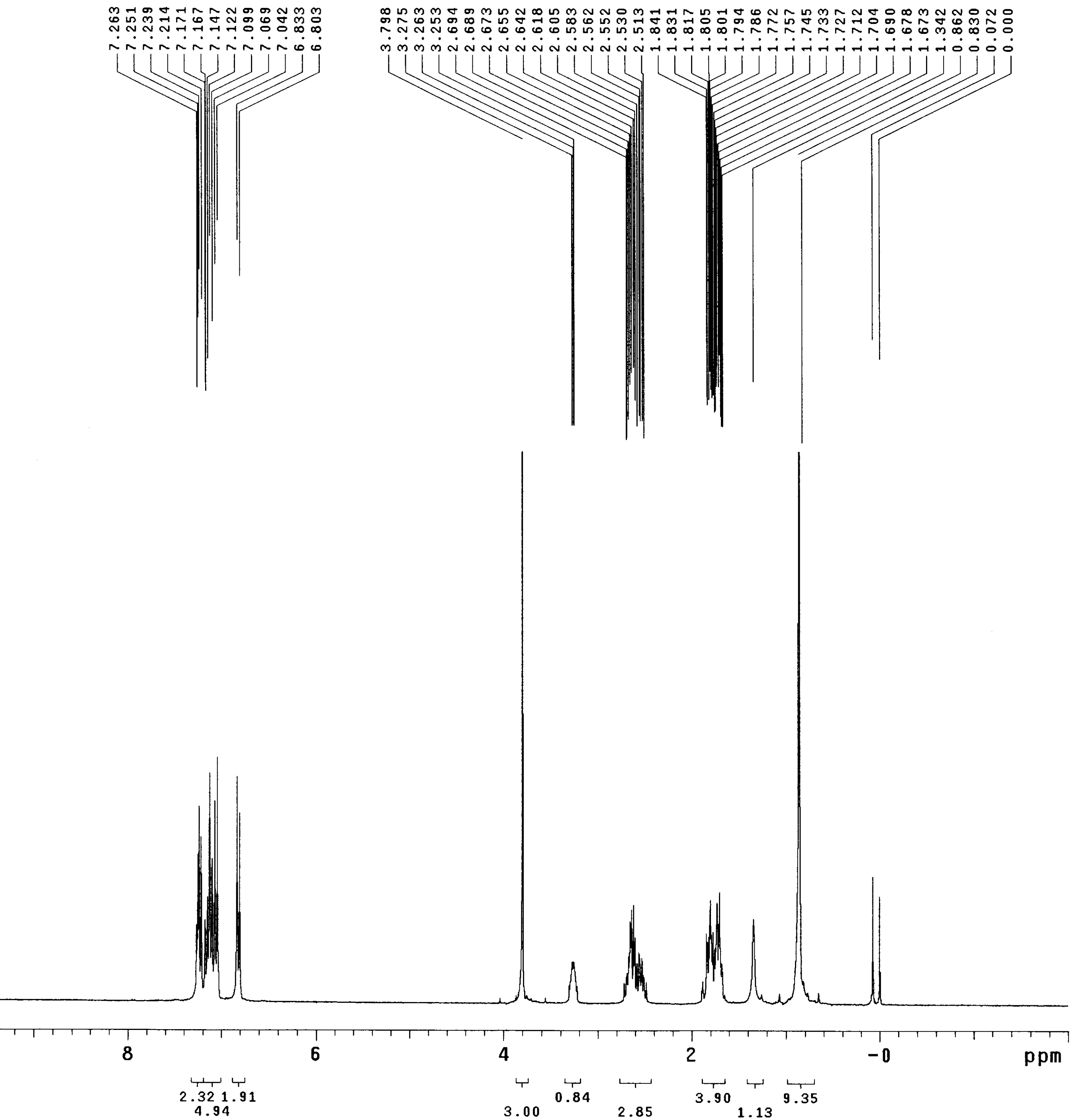
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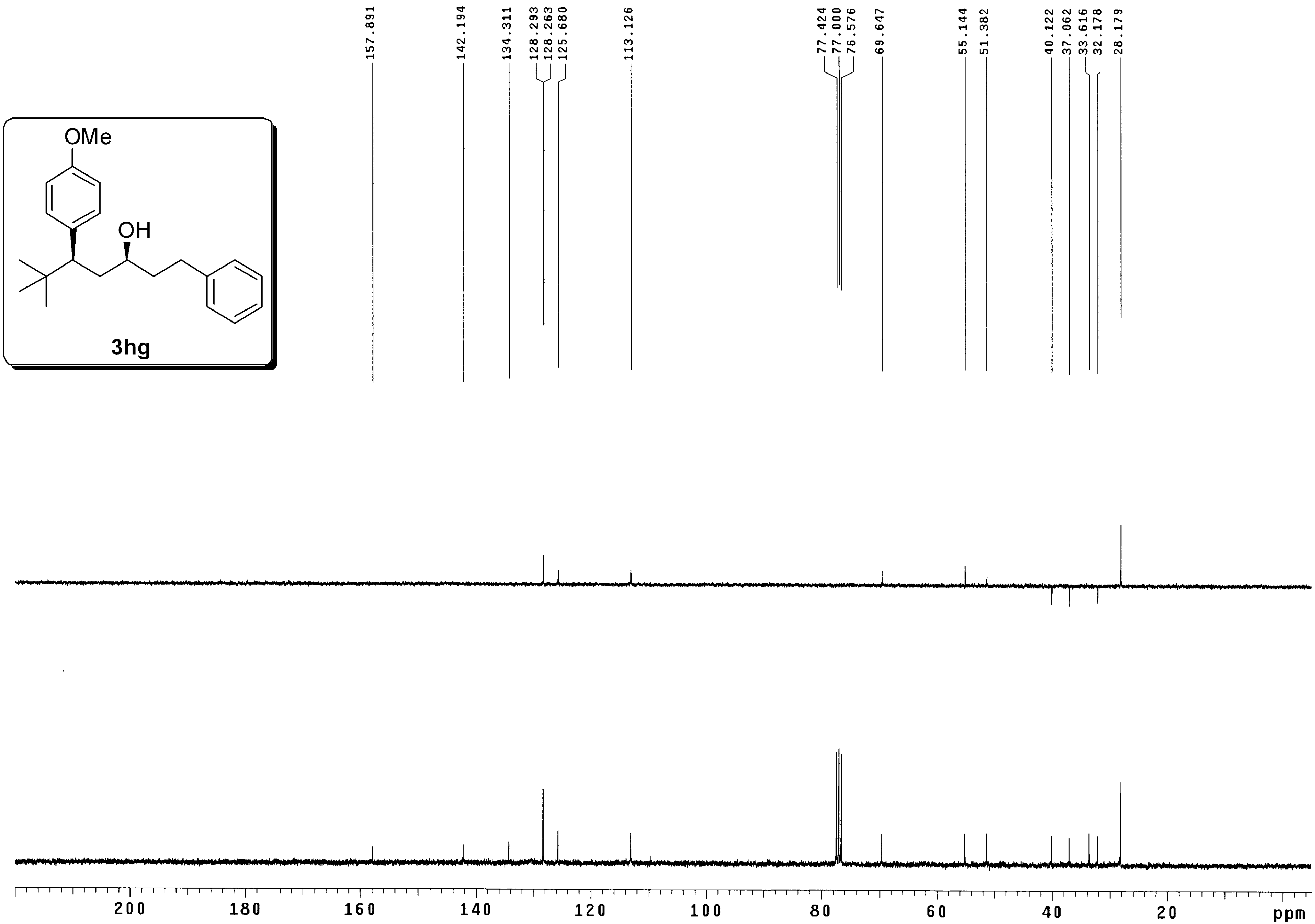
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37.062
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OMe
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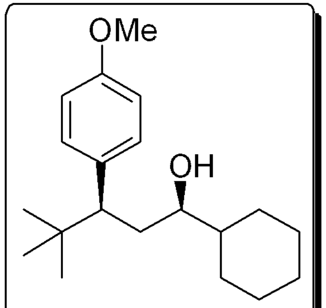
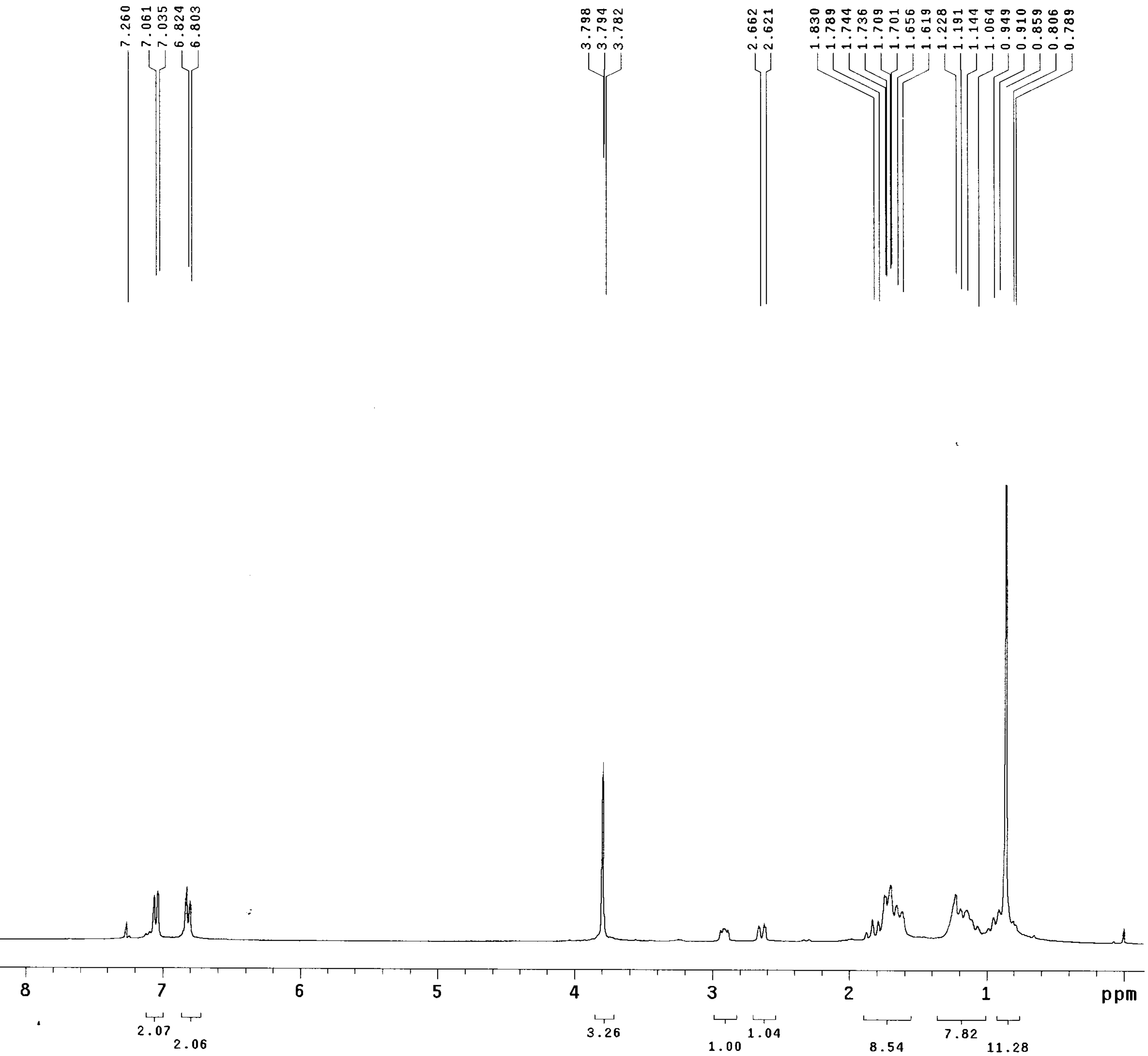

OMe
OH

3hh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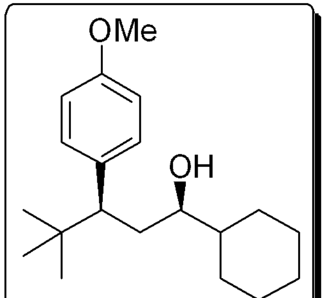
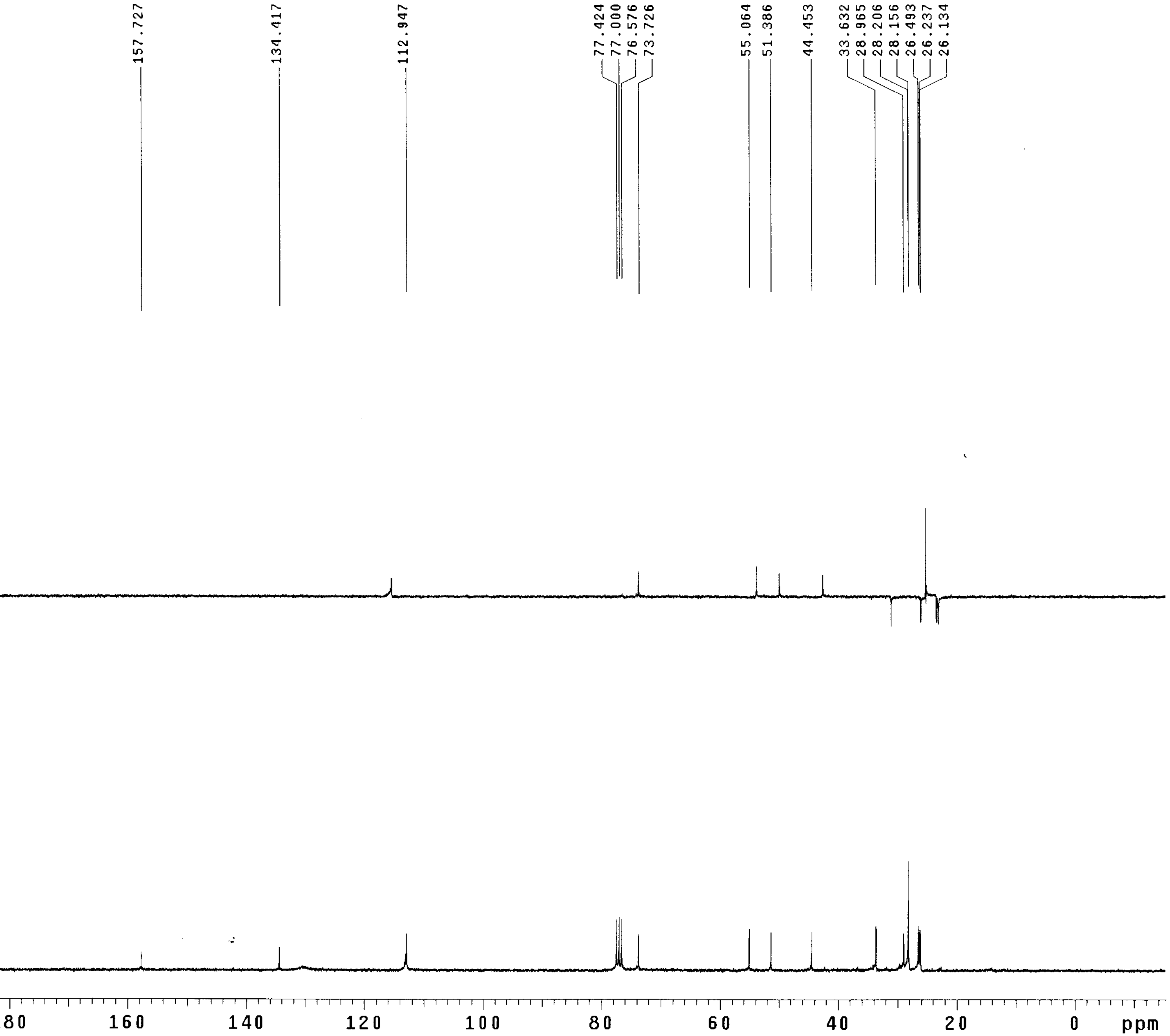
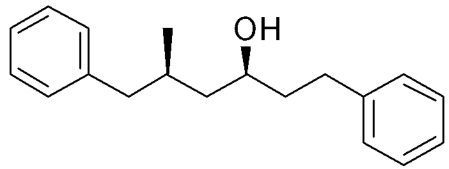
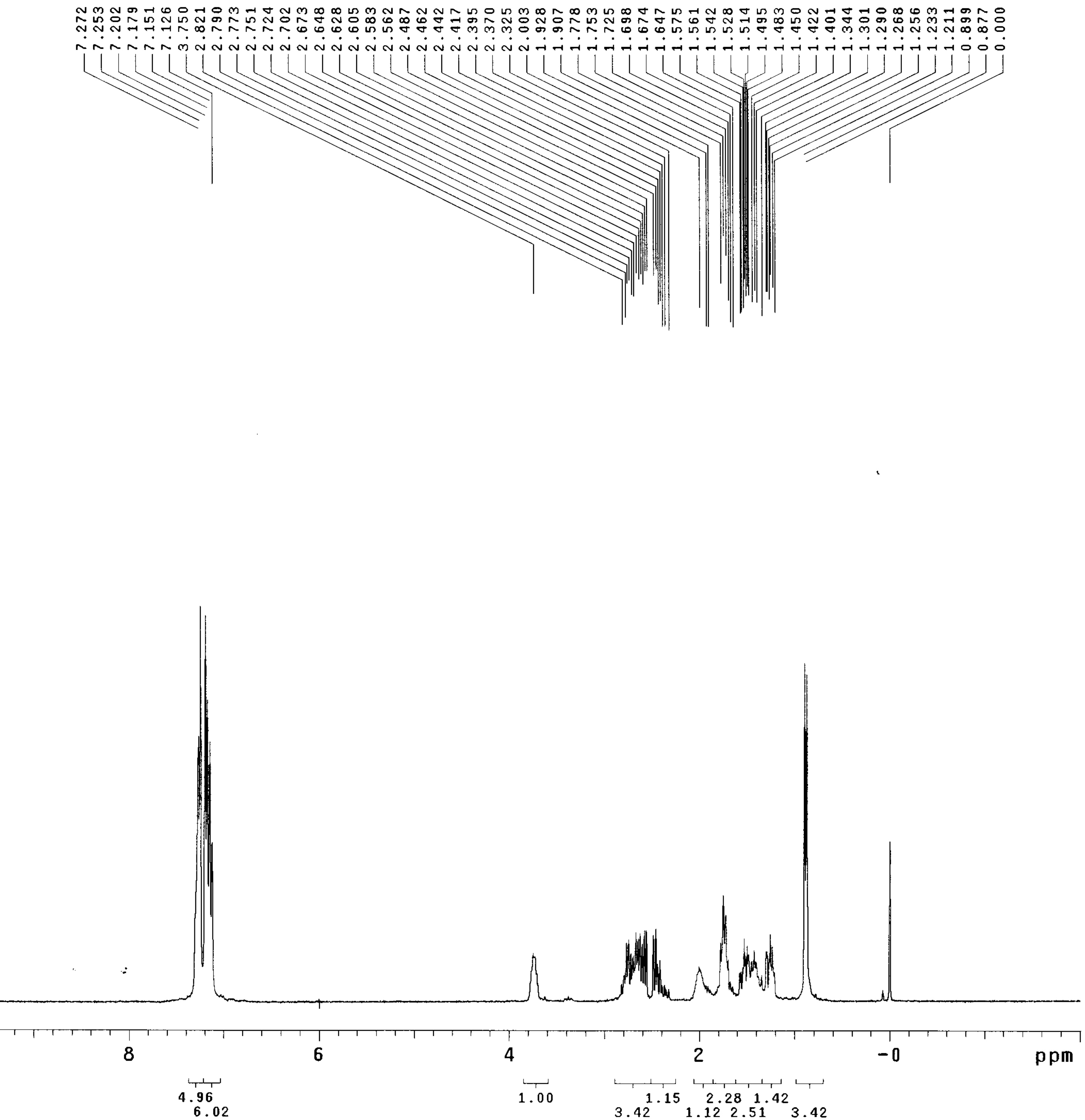

OMe
OH

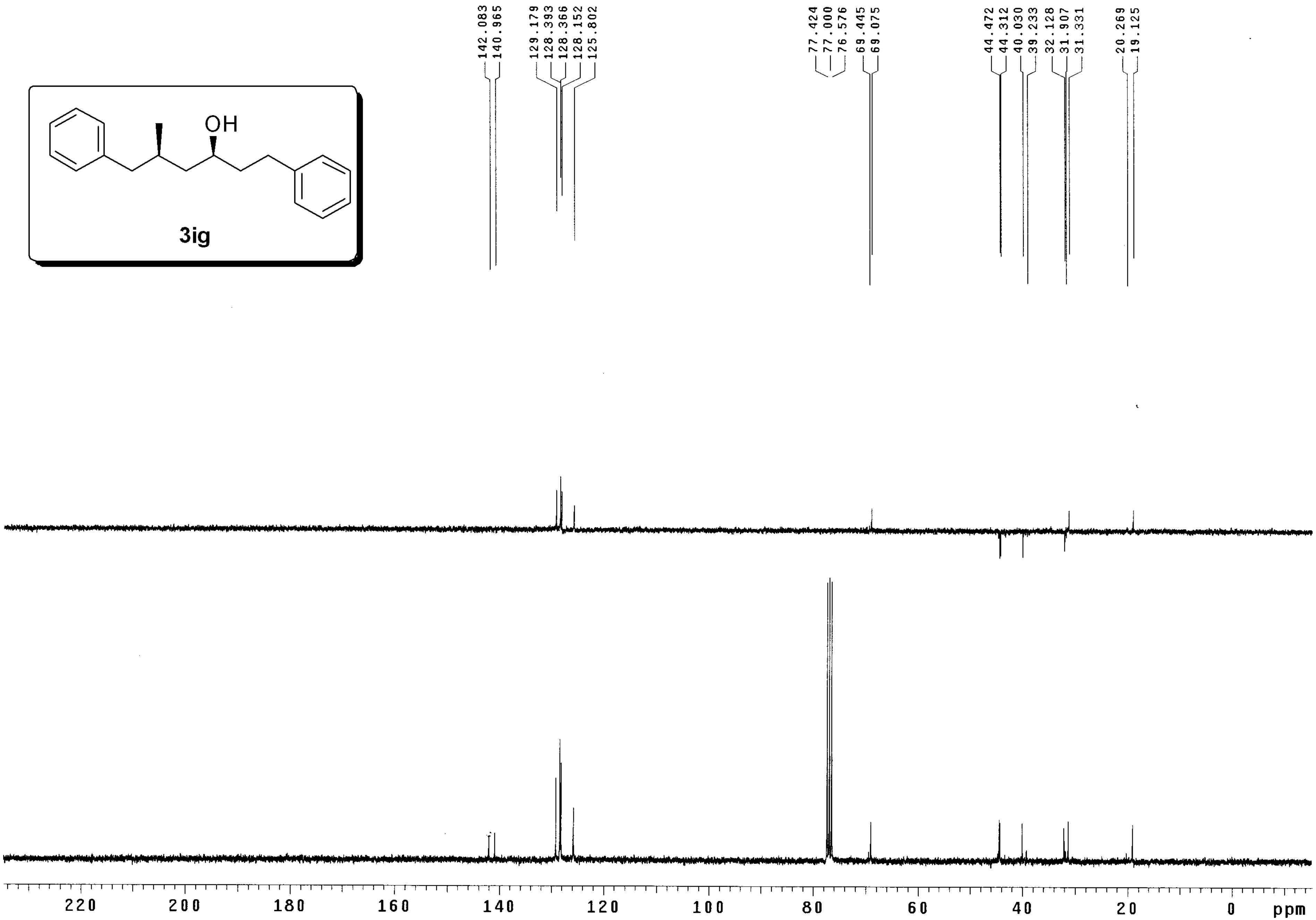
3hh

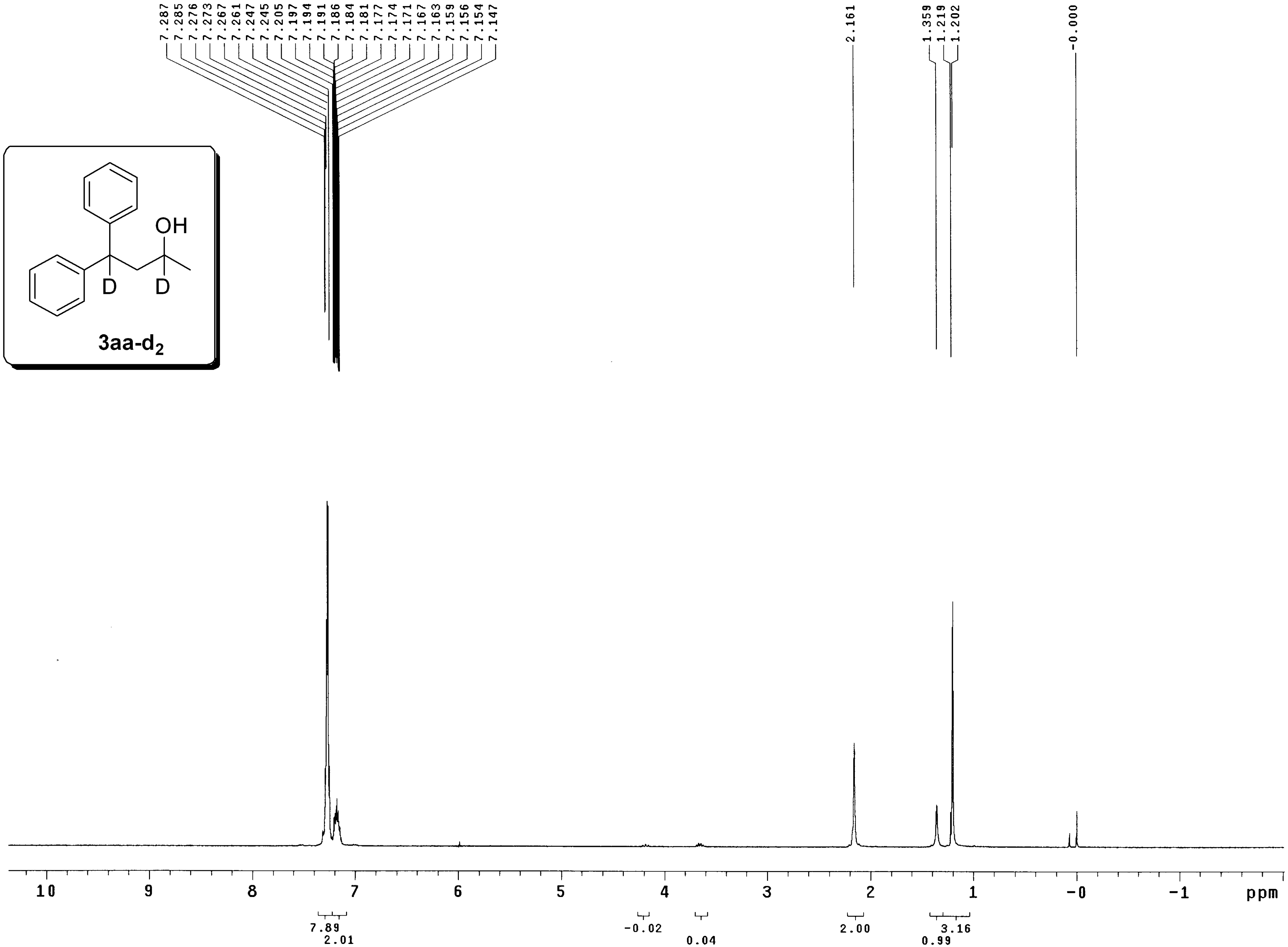

157.727
134.417
112.947
77.424
77.000
76.576
73.726
55.064
51.386
44.453
33.632
28.965
28.206
28.156
26.493
26.237
26.134
220
200
180
160
140
120
100
80
60
40
20
0
ppm


OH

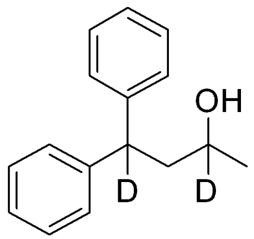
3ig


7.272
7.253
7.202
7.179
7.151
7.126
3.750
2.821
2.790
2.773
2.751
2.724
2.702
2.673
2.648
2.628
2.605
2.583
2.562
2.487
2.462
2.442
2.417
2.395
2.370
2.325
2.003
1.928
1.907
1.778
1.753
1.725
1.698
1.674
1.647
1.575
1.561
1.542
1.528
1.514
1.495
1.483
1.450
1.422
1.401
1.344
1.301
1.290
1.268
1.256
1.233
1.211
0.899
0.877
0.000
12
10
8
6
4
2
-0
ppm
4.96
6.02
1.00
1.15
3.42
2.28
1.42
1.12
2.51
3.42

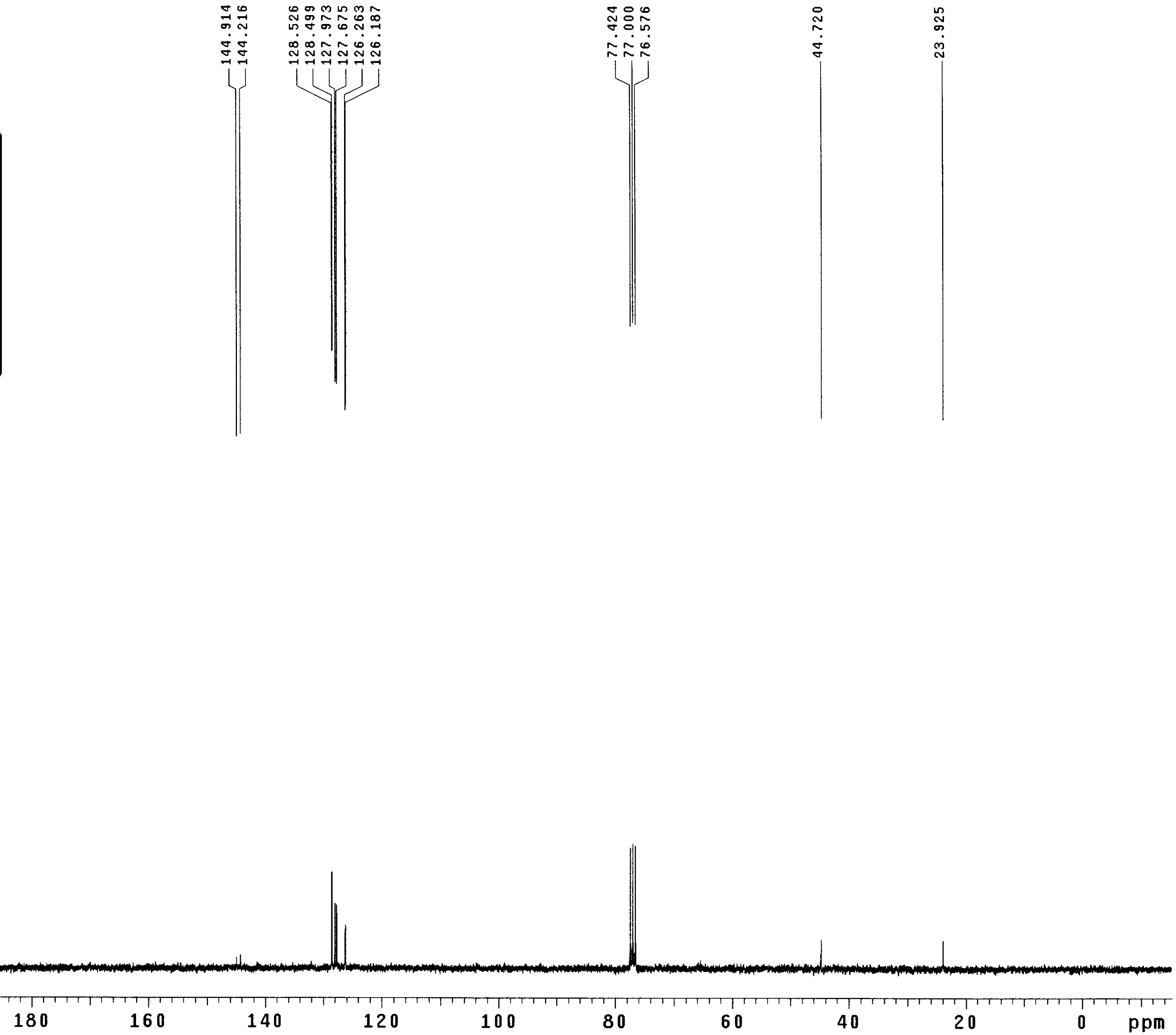

142.083
140.965
129.179
128.393
128.366
128.152
125.802
77.424
77.000
76.576
69.445
69.075
44.472
44.312
40.030
39.233
32.128
31.907
31.331
20.269
19.125
OH
3ig
220
200
180
160
140
120
100
80
60
40
20
0
ppm

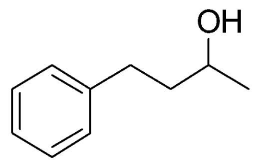


CH_3CD_2OH (min 98 atom % D, from the Sigma-Aldrich Trading Co., Ltd.).

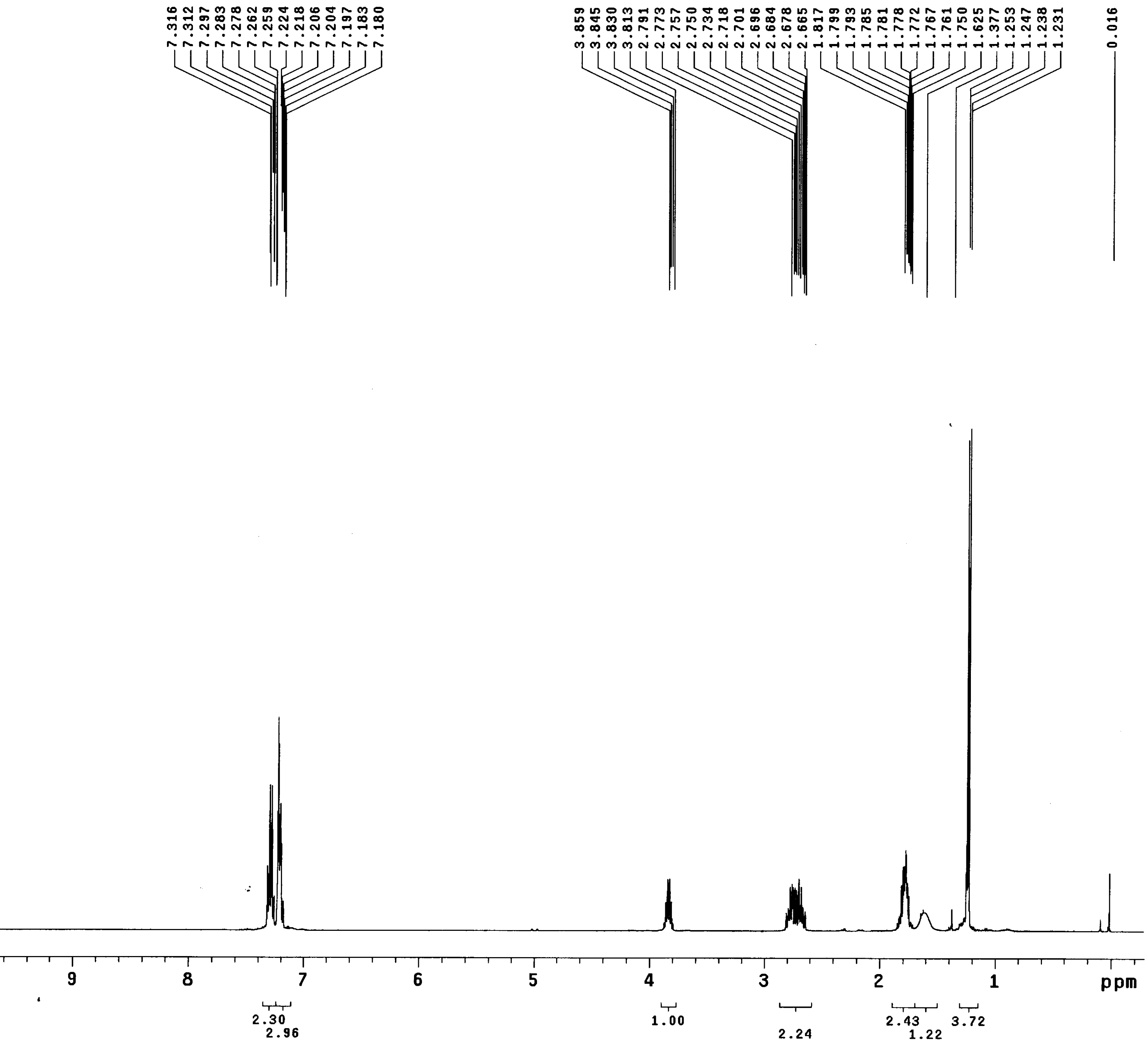


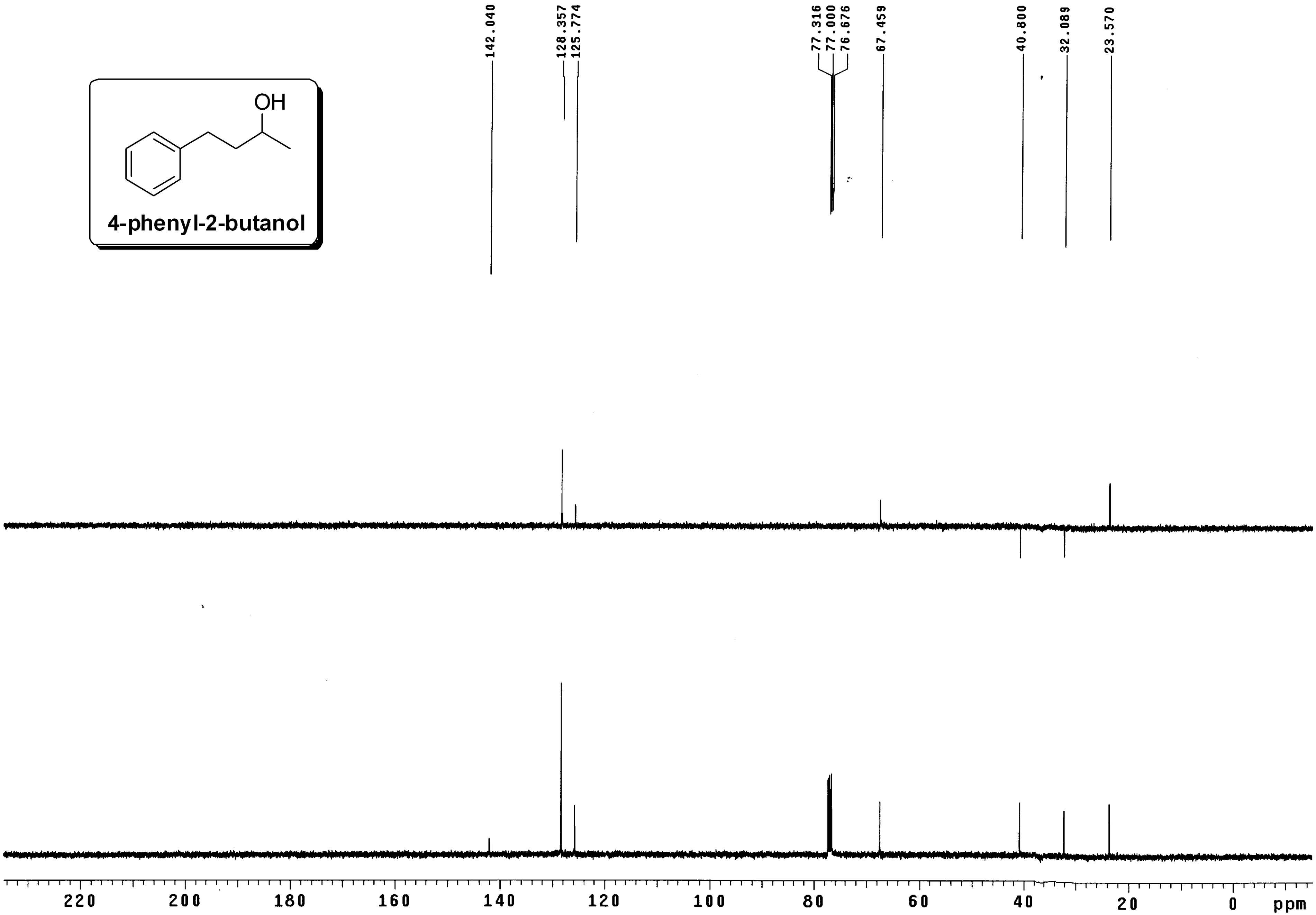
3aa-d$_2$


144.914
144.216
128.526
128.499
127.973
127.675
126.263
126.187
77.424
77.000
76.576
44.720
23.925
220
200
180
160
140
120
100
80
60
40
20
0
ppm



4-phenyl-2-butanol




142.040
128.357
125.774
77.316
77.000
76.676
67.459
40.800
32.089
23.570
OH
4-phenyl-2-butanol
220
200
180
160
140
120
100
80
60
40
20
0
ppm