Supporting Information

Indium-Copper and Indium-Silver Mediated Barbier-Grignard-Type Alkylation Reaction of Aldehydes Using Unactivated Alkyl Halides in Water

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General Methods

Deionised water was used in all reactions. All aldehydes were purified before using. All commercially available alkyl iodides were used directly without purification.

Following commercial grade reagents were also used without further purification: indium (powder, -100 mesh, 99.99%), copper (I) iodide (98%), silver iodide (99.9%), and iodine (99%).

The stirrer was stirred at the largest speed of 1250 rpm. The stirring bar was egg-shaped in 1.6 cm x 0.6 cm (L x Diam.).

Analytical thin layer chromatography (TLC) was performed using precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm). Further visualization was possible by staining with acidic solution of ceric molybdate.

Flash chromatography was performed using silica gel with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use.

Infrared spectra were examined under neat conditions.

High Resolution Mass (HRMS) spectra were obtained using an EI mass spectrometer.

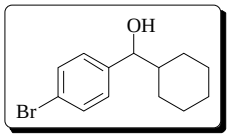
Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded at 300 MHZ or 400 MHZ spectrophotometer using $CDCl_3$ as solvent. Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-*d* (δ 7.2600, singlet). Multiplicities were given as: s (singlet); d (doublet); t (triplet); q (quartet); or m (multiplets). The number of protons (n) for a given resonance is indicated by nH. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-*d* (δ 77.03, triplet). The proportion of diastereomers was determined from the integration of ^{1}H NMR spectra.

Experimental Procedure

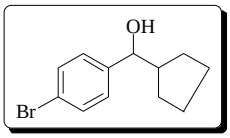
General procedure for the Barbier-Grignard-type alkylation reaction of benzaldehyde in water: To a 10 mL round-bottomed flask (RBF) was added 10 mL water, benzaldehyde (0.053g, 0.5 mmol), and I_2 (0.0253g, 0.1 mmol), stirred for several minutes, then indium

(0.344g, 3 mmol), and copper iodide (0.286g, 1.5 mmol) or silver iodide (0.587g, 2.5 mmol) were added to the RBF sequentially. After stirring for 10 minutes, cyclohexyl iodide (0.53g, 2.5 mmol) was introduced into the reaction system, and it was stirred vigorously at room temperature for 2 days. After reaction, 2 mL aq. HCl (1 M) was added, then it was extracted with ether (20 mL x 3), washed with brine, dried over sodium sulfate, filtered and evaporated solvent to give the oil residue. It was subjected to silica gel column chromatography using ethyl acetate and hexane as eluent to afford the desired product as colorless oil.

Spectroscopic Data of Products

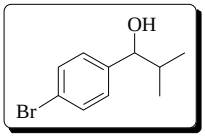


(*p*-Bromophenyl)-cyclohexyl-methanol (Table 2, entry 1): Colorless oil; R_f = 0.22 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3400 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85-1.26 (m, 5H), 1.35-1.39 (m, 1H), 1.51-2.00 (m, 6H), 4.33 (d, J = 6.92 Hz, 1H), 7.16 (d, J = 8.32 Hz, 2H), 7.44 (d, J = 8.40 Hz, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.5 (C), 131.2 (CHx2), 128.3 (CHx2), 121.1 (C), 78.6 (CH), 44.9 (CH), 29.2 (CH_2), 28.6 (CH_2), 26.3 (CH_2), 26.0 (CH_2), 25.9 (CH_2) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{13}H_{17}BrO$: 268.0463, found: 268.0452.

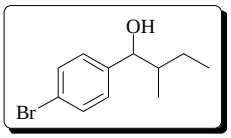


(*p*-Bromophenyl)-cyclopentyl-methanol (Table 2, entry 2): Colorless oil; R_f = 0.24 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3401 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.08-1.17 (m, 1H), 1.33-1.41 (m, 1H), 1.42-1.69 (m, 5H), 1.82-1.89 (m, 1H), 1.96 (s, br, 1H), 2.10-2.21 (m, 1H), 4.37 (d, J = 8.28 Hz, 1H), 7.21 (d, J = 8.32 Hz, 2H), 7.45 (d, J = 8.37 Hz, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.4 (C), 131.4 (CHx2), 128.2 (CHx2), 121.2 (C), 78.4 (CH), 47.7 (CH), 29.3 (CH_2), 29.3 (CH_2), 25.5 (CH_2), 25.4 (CH_2) ppm; HRMS (EI, m/z):

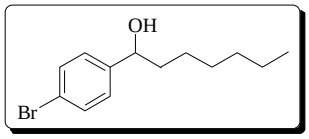
$[M]^+$, Calcd. for $C_{12}H_{15}BrO$: 254.0306, found: 254.0301.



1-(*p*-Bromophenyl)--2-methyl-1-propanol (Table 2, entry 3): Colorless oil; R_f = 0.26 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3406 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.79 (d, J = 6.80 Hz, 3H), 0.96 (d, J = 6.69 Hz, 3H), 1.86-1.94 (m, 1H), 2.02 (s, br, 1H), 4.32 (d, J = 6.64 Hz, 1H), 7.17 (d, J = 8.32 Hz, 2H), 7.45 (d, J = 8.40 Hz, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.5 (C), 131.2 (CHx2), 128.3 (CHx2), 121.1 (C), 79.2 (CH), 35.2 (CH), 18.8 (CH_3), 18.0 (CH_3) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{10}H_{13}BrO$: 228.0150, found: 228.0138.

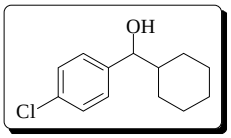


1-(*p*-Bromophenyl)-2-methyl-butan-1-ol (Table 2, entry 4): Colorless oil; R_f = 0.17 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3393 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): (two isomers) δ 0.74 (d, J = 6.81 Hz, 3H), 0.87-0.94 (m, 9H), 0.98-1.46 (m, 3H), 1.60-1.75 (m, 3H), 1.78 (s, br, 1H), 1.82 (s, br, 1H), 4.43 (d, J = 6.57 Hz, 1H), 4.53 (d, J = 5.04 Hz, 1H), 7.18-7.20 (m, 4H), 7.44-7.48 (m, 4H) ppm; ^{13}C NMR (75.4 MHz, $CDCl_3$): (two isomers) δ 142.9 (C), 142.5 (C), 131.2 (CHx2), 131.2 (CHx2), 128.4 (CHx2), 128.1 (CHx2), 121.1 (C), 120.9 (C), 78.1 (CH), 77.3 (CH), 42.0 (CH), 41.7 (CH), 25.8 (CH_2), 24.7 (CH_2), 15.0 (CH_3), 13.8 (CH_3), 11.7 (CH_3), 11.3 (CH_3) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{11}H_{15}BrO$: 242.0306, found: 242.0300.

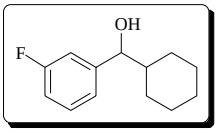


1-(*p*-Bromophenyl)-1-heptanol (Table 2, entry 5): Colorless oil; R_f = 0.17 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3400 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.87 (t,

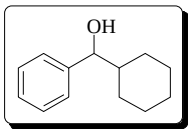
J = 6.72 Hz, 3H), 1.26-1.39 (m, 8H), 1.64-1.80 (m, 2H), 1.89 (s, 1H), 4.61-4.64 (m, 1H), 7.21 (d, *J* = 8.33 Hz, 2H), 7.46 (d, *J* = 8.40 Hz, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.9 (C), 131.5 (CHx2), 127.7 (CHx2), 121.2 (C), 74.0 (CH), 39.2 (CH_2), 31.7 (CH_2), 29.2 (CH_2), 25.6 (CH_2), 22.6 (CH_2), 14.1 (CH_3) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{13}H_{19}BrO$: 270.0619, found: 270.0616.



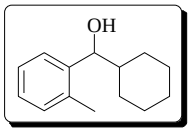
Cyclohexyl-(*p*-chlorophenyl)-methanol (Table 2, entry 6): Colorless oil; R_f = 0.28 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3400 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.86-1.26 (m, 5H), 1.36-1.39 (m, 1H), 1.52-1.68 (m, 3H), 1.74-1.77 (m, 1H), 1.86-1.94 (m, 2H), 4.36 (d, *J* = 6.92 Hz, 1H), 7.21-7.31 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.0 (C), 133.0 (C), 128.3 (CHx2), 128.0 (CHx2), 78.6 (CH), 45.0 (CH), 29.2 (CH_2), 28.6 (CH_2), 26.3 (CH_2), 26.0 (CH_2), 25.9 (CH_2) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{13}H_{17}ClO$: 224.0968, found: 224.0968.



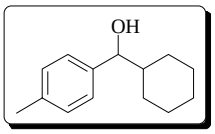
Cyclohexyl-(*m*-fluorophenyl)-methanol (Table 2, entry 7): Colorless oil; R_f = 0.30 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3401 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.96-1.42 (m, 6H), 1.57-1.94 (m, 5H), 1.96 (s, br, 1H), 4.37 (d, *J* = 6.87 Hz, 1H), 6.92-7.06 (m, 3H), 7.25-7.32 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.8 (d, *J* = 245.7 Hz, C), 146.3 (d, *J* = 6.5 Hz, C), 129.6 (d, *J* = 8.1 Hz, CH), 122.2 (d, *J* = 2.6 Hz, CH), 114.2 (d, *J* = 21.4 Hz, CH), 113.5 (d, *J* = 21.4 Hz, CH), 78.7 (CH), 45.0 (CH), 29.3 (CH_2), 28.5 (CH_2), 26.4 (CH_2), 26.1 (CH_2), 26.0 (CH_2) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{13}H_{17}OF$: 208.1250, found: 208.1263.



Cyclohexyl-phenyl-methanol (Table 2, entry 8): Colorless oil; R_f = 0.28 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3440 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 0.84-1.29 (m, 5H), 1.34-1.38 (m, 1H), 1.53-1.77 (m, 4H), 1.95-2.00 (m, 2H), 4.33 (d, J = 7.14 Hz, 1H), 7.22-7.34 (m, 5H) ppm; ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 143.6 (C), 128.1 (CHx2), 127.3 (CH), 126.6 (CHx2), 79.3 (CH), 44.9 (CH), 29.3 (CH_2), 28.8 (CH_2), 26.4 (CH_2), 26.1 (CH_2), 26.0 (CH_2) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{13}H_{18}O$: 190.1358, found: 190.1346.

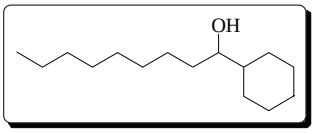


Cyclohexyl-(*o*-methylphenyl)-methanol (Table 2, entry 9): Colorless oil; R_f = 0.26 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3416 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.01-1.22 (m, 5H), 1.35-1.38 (m, 1H), 1.58-1.65 (m, 3H), 1.75-1.79 (m, 1H), 1.87 (s, 1H), 1.98-2.01 (m, 1H), 2.31 (s, 3H), 4.62 (d, J = 7.12 Hz, 1H), 7.10-7.23 (m, 3H), 7.37-7.39 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.9 (C), 135.0 (C), 130.2 (CH), 126.9 (CH), 126.2 (CH), 126.0 (CH), 75.0 (CH), 44.5 (CH), 29.5 (CH_2), 28.5 (CH_2), 26.4 (CH_2), 26.3 (CH_2), 26.0 (CH_2), 19.4 (CH_3) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{14}H_{20}O$: 204.1514, found: 204.1516.

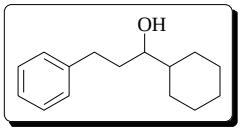


Cyclohexyl-(*p*-methylphenyl)-methanol (Table 2, entry 10): Colorless oil; R_f = 0.27 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3393 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85-1.28 (m, 5H), 1.35-1.38 (m, 1H), 1.55-1.66 (m, 3H), 1.74-1.81 (m, 2H), 1.98-2.01 (m, 1H), 2.34 (s, 3H), 4.31 (d, J = 7.00 Hz, 1H), 7.13-7.19 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.7 (C), 137.0 (C), 128.9 (CHx2), 126.6 (CHx2), 79.3 (CH), 44.9 (CH), 29.3

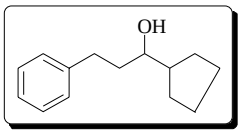
(CH_2), 29.0 (CH_2), 26.5 (CH_2), 26.1 (CH_2), 26.0 (CH_2), 21.1 (CH_3) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{14}H_{20}O$: 204.1514, found: 204.1505.



1-Cyclohexyl-1-nonanol (Table 2, entry 11): Colorless oil; R_f = 0.35 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3381 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 6.61 Hz, 3H), 0.95-1.48 (m, 21H), 1.66-1.82 (m, 5H), 3.33-3.36 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 76.2 (CH), 43.6 (CH), 34.1 (CH_2), 31.9 (CH_2), 29.8 (CH_2), 29.6 (CH_2), 29.3 (CH_2), 29.3 (CH_2), 27.7 (CH_2), 26.6 (CH_2), 26.4 (CH_2), 26.2 (CH_2), 26.0 (CH_2), 22.7 (CH_2), 14.1 (CH_3) ppm; HRMS (EI, m/z): $[M-H]^+$, Calcd. for $C_{15}H_{29}O$: 225.2218, found: 225.2208.

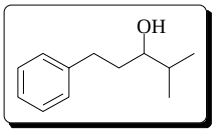


1-Cyclohexyl-3-phenyl-1-propanol (Table 2, entry 12): Colorless oil; R_f = 0.22 (Ethyl acetate/hexane, 1:8); ^1H NMR (300 MHz, $CDCl_3$): δ 0.97-1.44 (m, 7H), 1.64-1.87 (m, 7H), 2.59-2.69 (m, 1H), 2.79-2.88 (m, 1H), 3.35-3.41 (m, 1H), 7.15-7.30 (m, 5H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.4 (C), 128.4 (CHx2), 128.4 (CHx2), 125.8 (CH), 75.6 (CH), 43.8 (CH), 36.0 (CH_2), 32.4 (CH_2), 29.2 (CH_2), 27.8 (CH_2), 26.5 (CH_2), 26.3 (CH_2), 26.2 (CH_2) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{15}H_{22}O$: 218.1671, found: 218.1660.

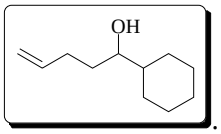


1-Cyclopentyl-3-phenyl-1-propanol (Table 2, entry 13): Colorless oil; R_f = 0.24 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3400 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.14-1.23 (m, 1H), 1.29-1.36 (m, 1H), 1.44 (s, 1H), 1.51-1.73 (m, 6H), 1.74-1.95 (m, 3H), 2.63-2.71 (m, 1H), 2.82-2.89 (m, 1H), 3.41-3.44 (m, 1H), 7.16-7.30 (m, 5H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.4 (C), 128.4 (CHx2), 128.4 (CHx2), 125.7 (CH), 75.4 (CH), 46.5

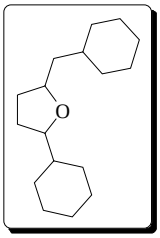
(CH), 37.9 (CH_2), 32.2 (CH_2), 29.1 (CH_2), 28.6 (CH_2), 25.7 (CH_2), 25.6 (CH_2) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{14}H_{20}O$: 204.1514, found: 204.1511.



4-Methyl-1-phenyl-pentan-3-ol (Table 2, entry 14): Colorless oil; R_f = 0.25 (Ethyl acetate/hexane, 1:8); FTIR (NaCl, neat): ν 3408 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 0.92 (d, J = 6.72 Hz, 6H), 1.38 (s, br, 1H), 1.60-1.85 (m, 3H), 2.60-2.70 (m, 1H), 2.80-2.89 (m, 1H), 3.37-3.41 (m, 1H), 7.16-7.31 (m, 5H) ppm; ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 142.4 (C), 128.4 (CHx2), 128.4 (CHx2), 125.8 (CH), 76.1 (CH), 36.0 (CH_2), 33.7 (CH), 32.5 (CH_2), 18.8 (CH_3), 17.2 (CH_3) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{12}H_{18}O$: 178.1358, found: 178.1350.



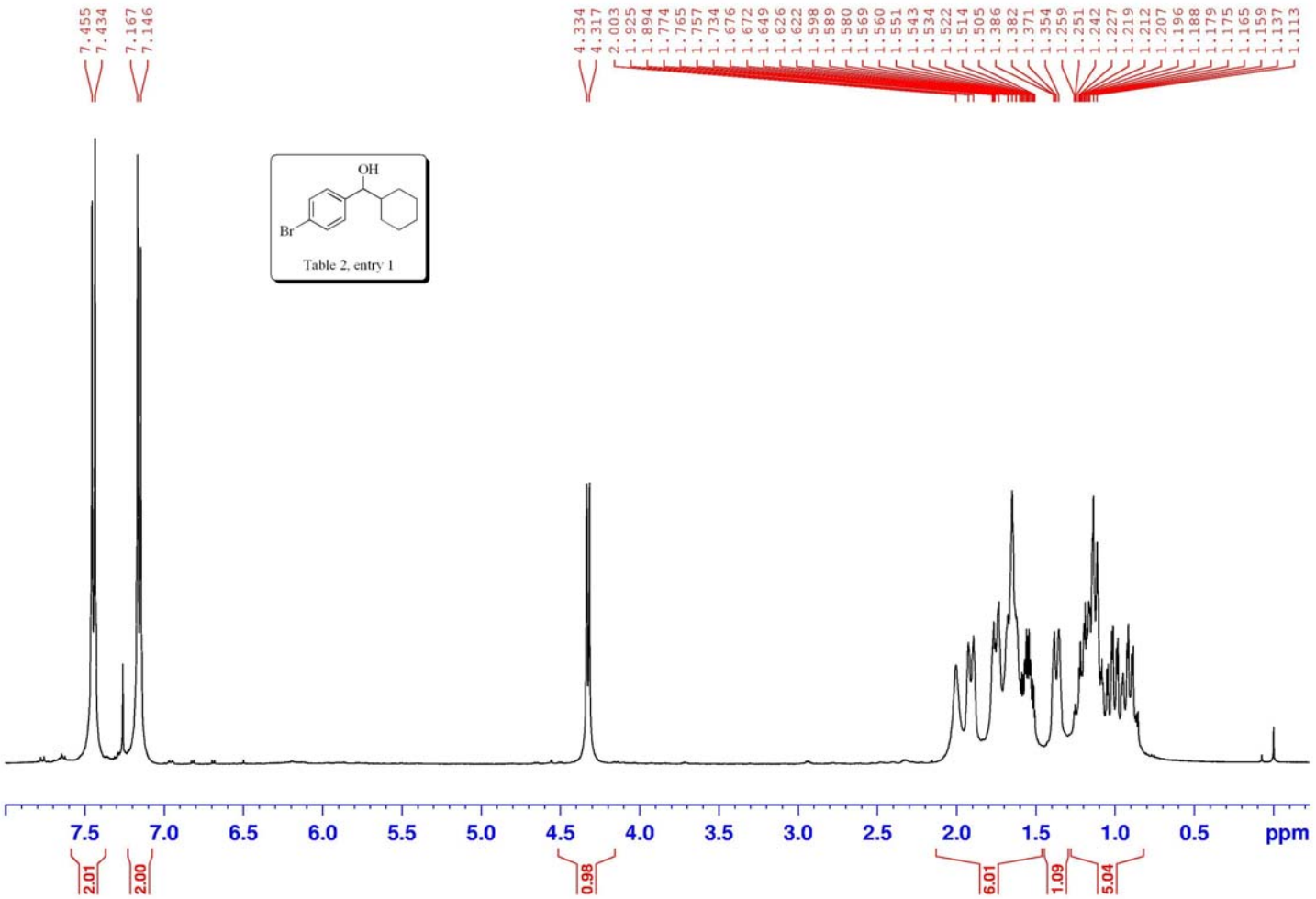
1-Cyclohexyl-pent-4-en-1-ol (Compound 1, in Scheme 1): Colorless oil; R_f = 0.35 (Ethyl acetate/hexane, 1:8); ^1H NMR (400 MHz, $CDCl_3$): δ 0.95-1.36 (m, 6H), 1.44-1.82 (m, 8H), 2.07-2.28 (m, 2H), 3.36-3.40 (m, 1H), 4.96-5.07 (m, 2H), 5.80-5.90 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.8 (CH), 114.7 (CH_2), 75.7 (CH), 43.7 (CH), 33.3 (CH_2), 30.4 (CH_2), 29.2 (CH_2), 27.8 (CH_2), 26.5 (CH_2), 26.3 (CH_2), 26.2 (CH_2) ppm; HRMS (EI, m/z): $[M-H]^+$, Calcd. for $C_{11}H_{19}O$: 167.1436, found: 167.1433.

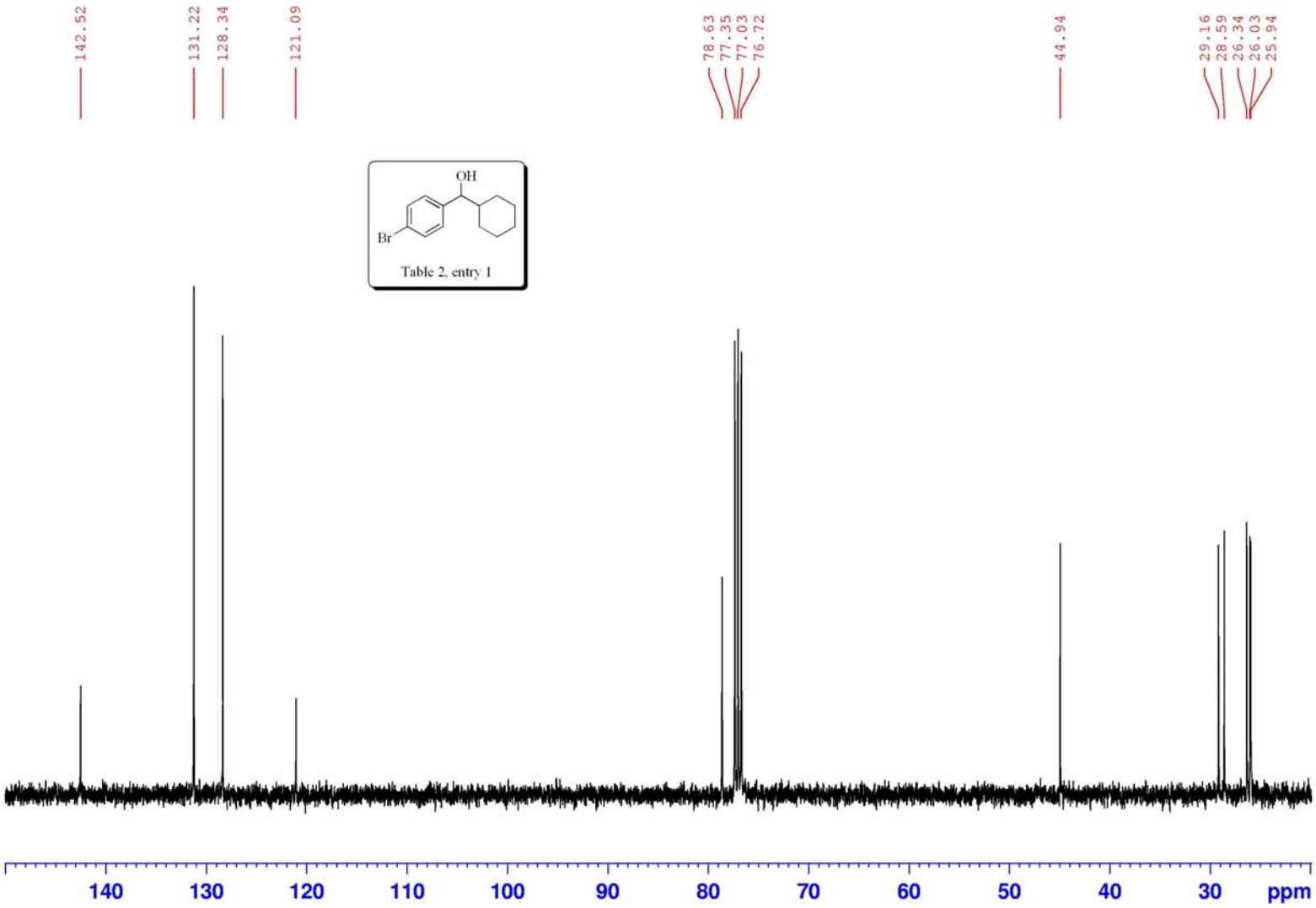


2-Cyclohexyl-5-[(1-cyclohexyl)-methyl]-tetrahydrofuran (Compound 2, in Scheme 1): Colorless oil; R_f = 0.72 (Ethyl acetate/hexane, 1:8); ^1H NMR (400 MHz, $CDCl_3$): (major

isomers) δ 0.88-1.00 (m, 4H), 1.11-1.41 (m, 10H), 1.48-1.94 (m, 14H), 3.48 (q, J = 7.28 Hz, 1H), 3.83-3.90 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): (major isomer) δ 83.6 (CH), 76.9 (CH), 43.9 (CH_2), 43.2 (CH), 35.1 (CH), 33.7 (CH_2), 33.6 (CH_2), 31.6 (CH_2), 29.9 (CH_2), 28.9 (CH_2), 28.5 (CH_2), 26.6 (CH_2x2), 26.3 (CH_2x2), 26.1 (CH_2), 26.0 (CH_2) ppm; HRMS (EI, m/z): $[M]^+$, Calcd. for $C_{17}H_{30}O$: 250.2297, found: 250.2276

Copies of ^{1}H NMR and ^{13}C NMR Data of Products

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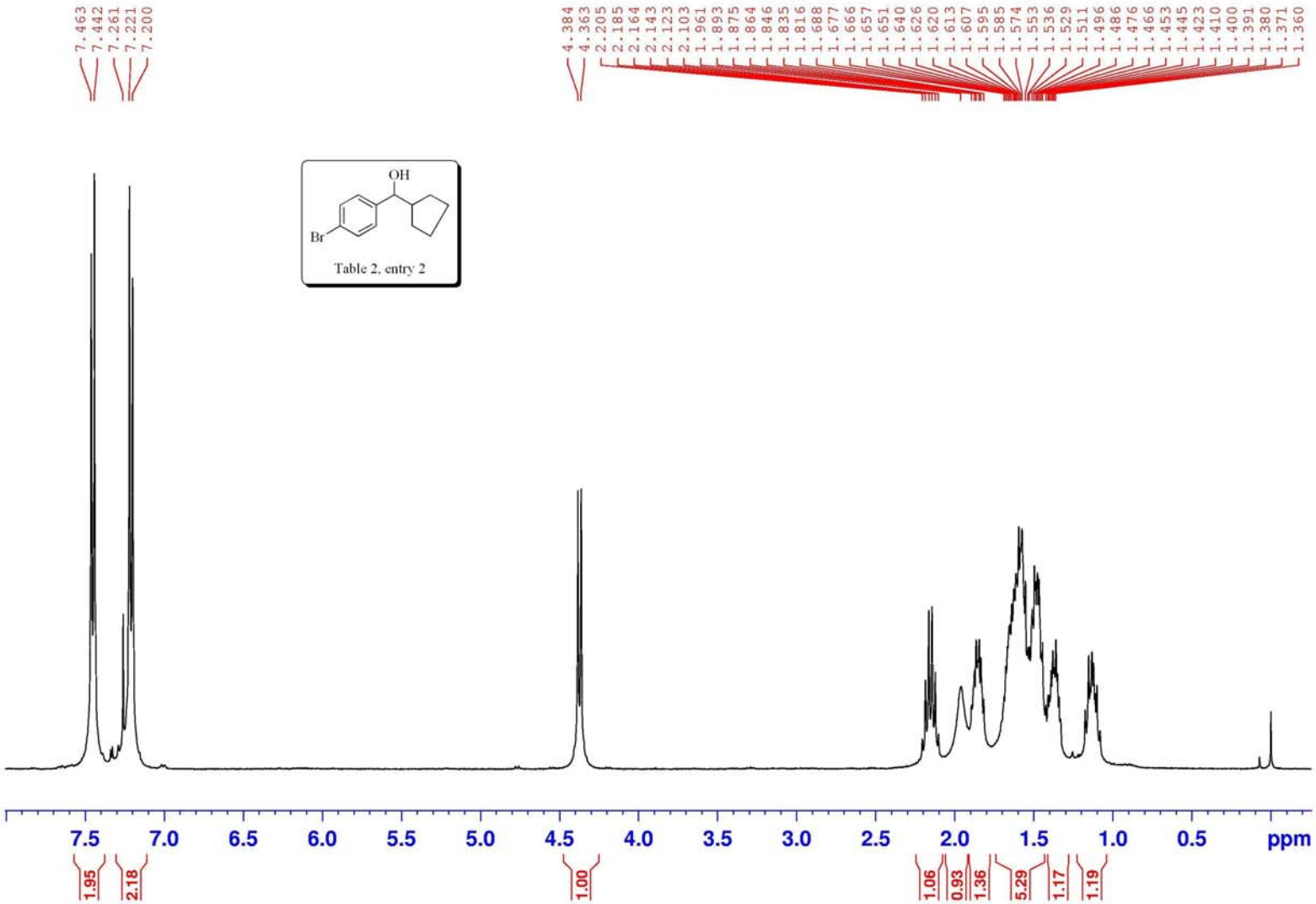
128.34
121.09
78.63
77.35
77.03
76.72
44.94
29.16
28.59
26.34
26.03
25.94
OH
Br
Table 2, entry 1
140
130
120
110
100
90
80
70
60
50
40
30
ppm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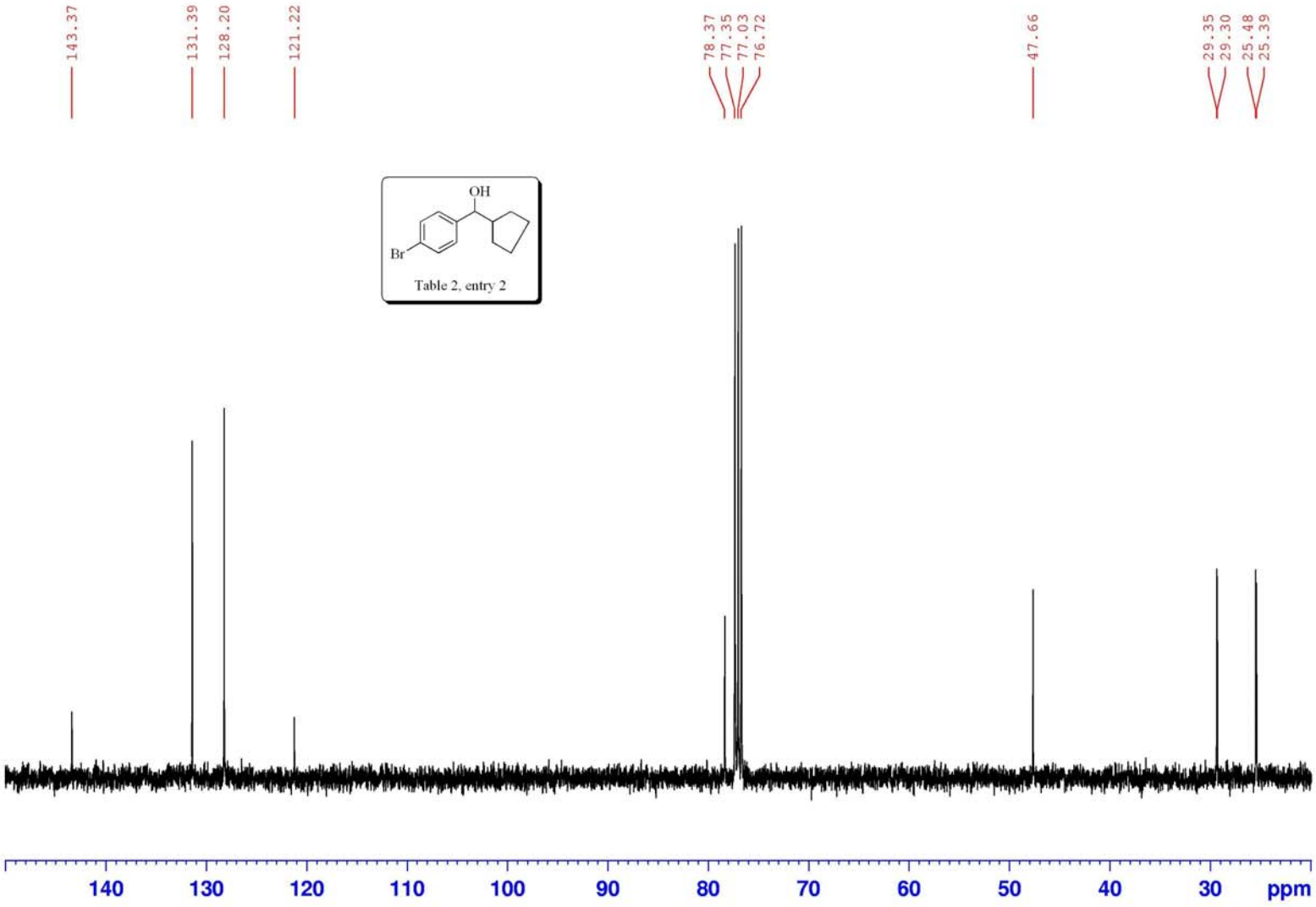
7.442
7.261
7.221
7.200
4.384
4.363
2.205
2.185
2.164
2.143
2.123
2.103
1.961
1.893
1.875
1.864
1.846
1.835
1.816
1.688
1.677
1.666
1.657
1.651
1.640
1.626
1.620
1.613
1.607
1.595
1.585
1.574
1.553
1.536
1.529
1.511
1.496
1.486
1.476
1.466
1.453
1.445
1.423
1.410
1.400
1.391
1.380
1.371
1.360
OH
Br
Table 2, entry 2
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.95
2.18
1.00
1.06
0.93
1.36
5.29
1.17
1.19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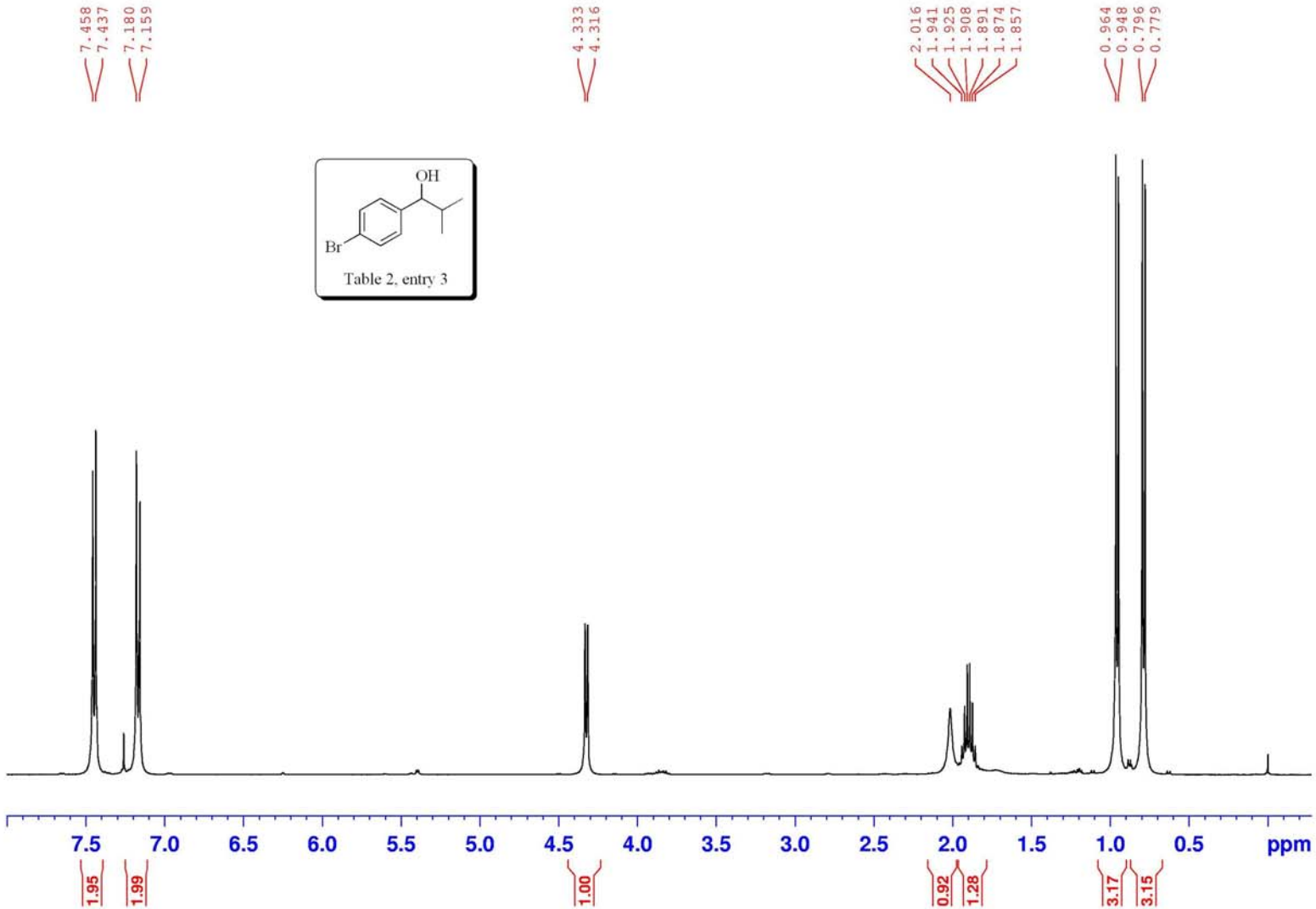
128.20
121.22
78.37
77.35
77.03
76.72
47.66
29.35
29.30
25.48
25.39
OH
Br
Table 2, entry 2
140
130
120
110
100
90
80
70
60
50
40
30
ppm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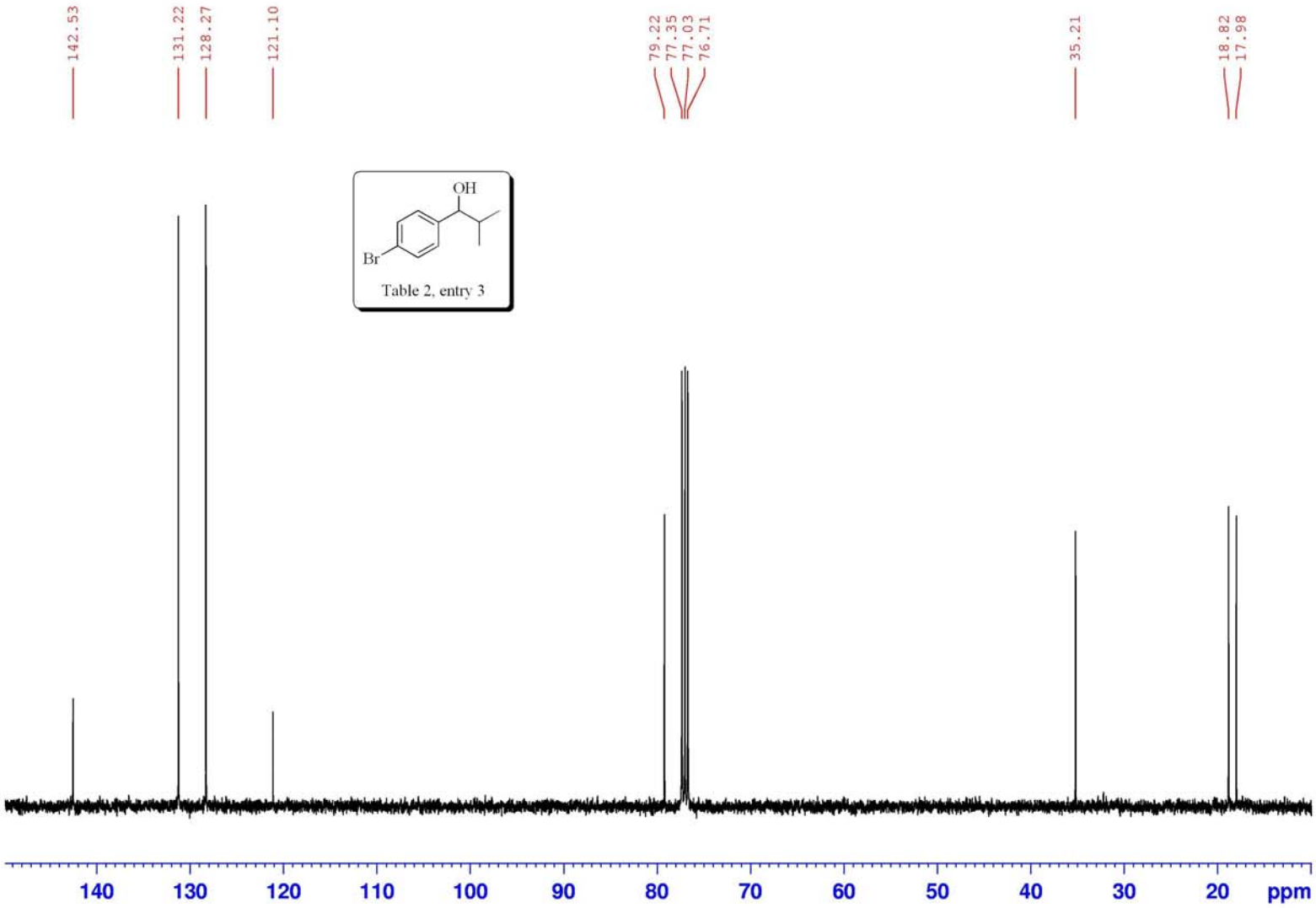
7.437
7.180
7.159
4.333
4.316
2.016
1.941
1.925
1.908
1.891
1.874
1.857
0.964
0.948
0.796
0.779
OH
Br
Table 2, entry 3
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.95
1.99
1.00
0.92
1.28
3.17
3.15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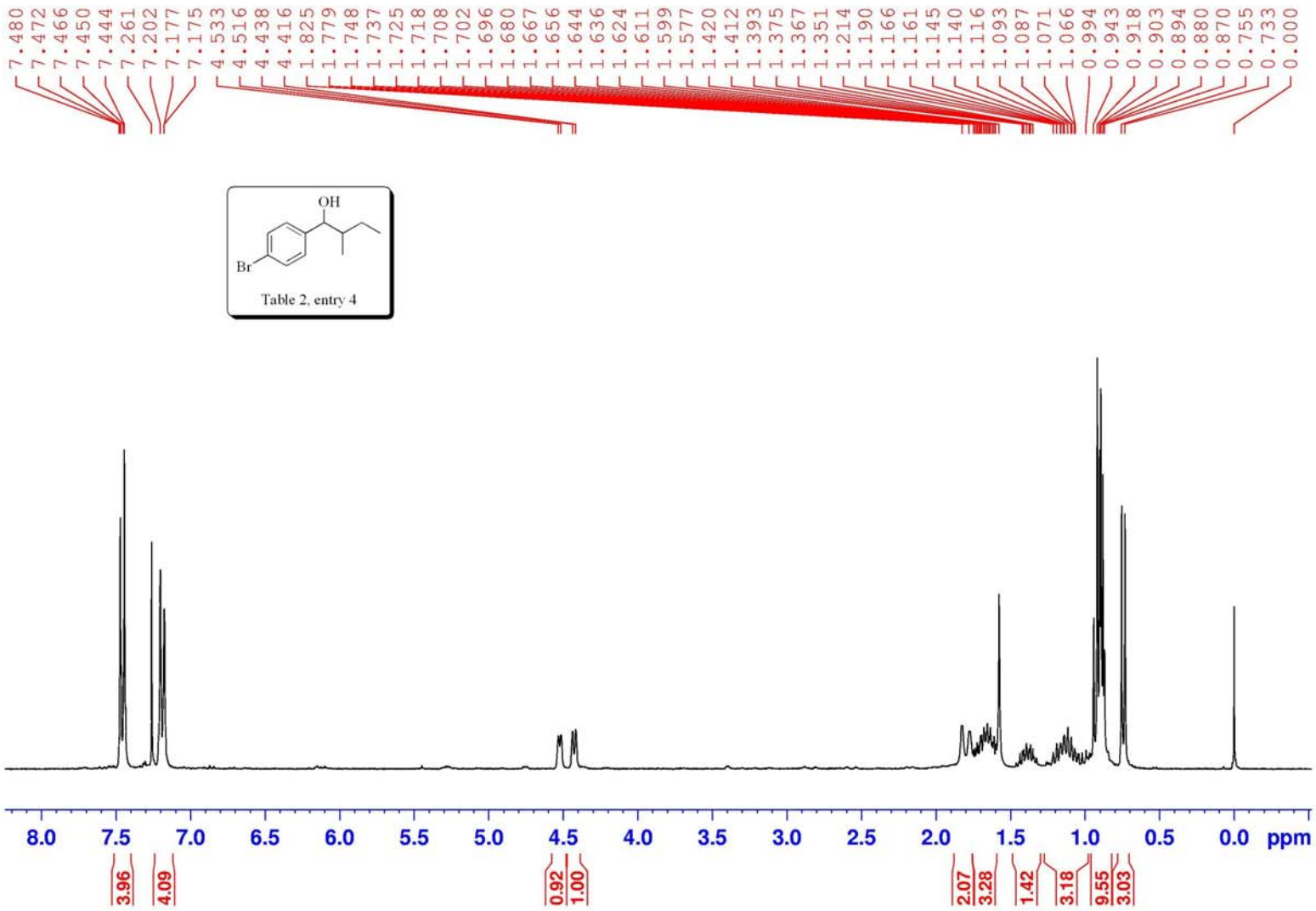
131.22
128.27
121.10
79.22
77.35
77.03
76.71
35.21
18.82
17.98
OH
Br
Table 2, entry 3
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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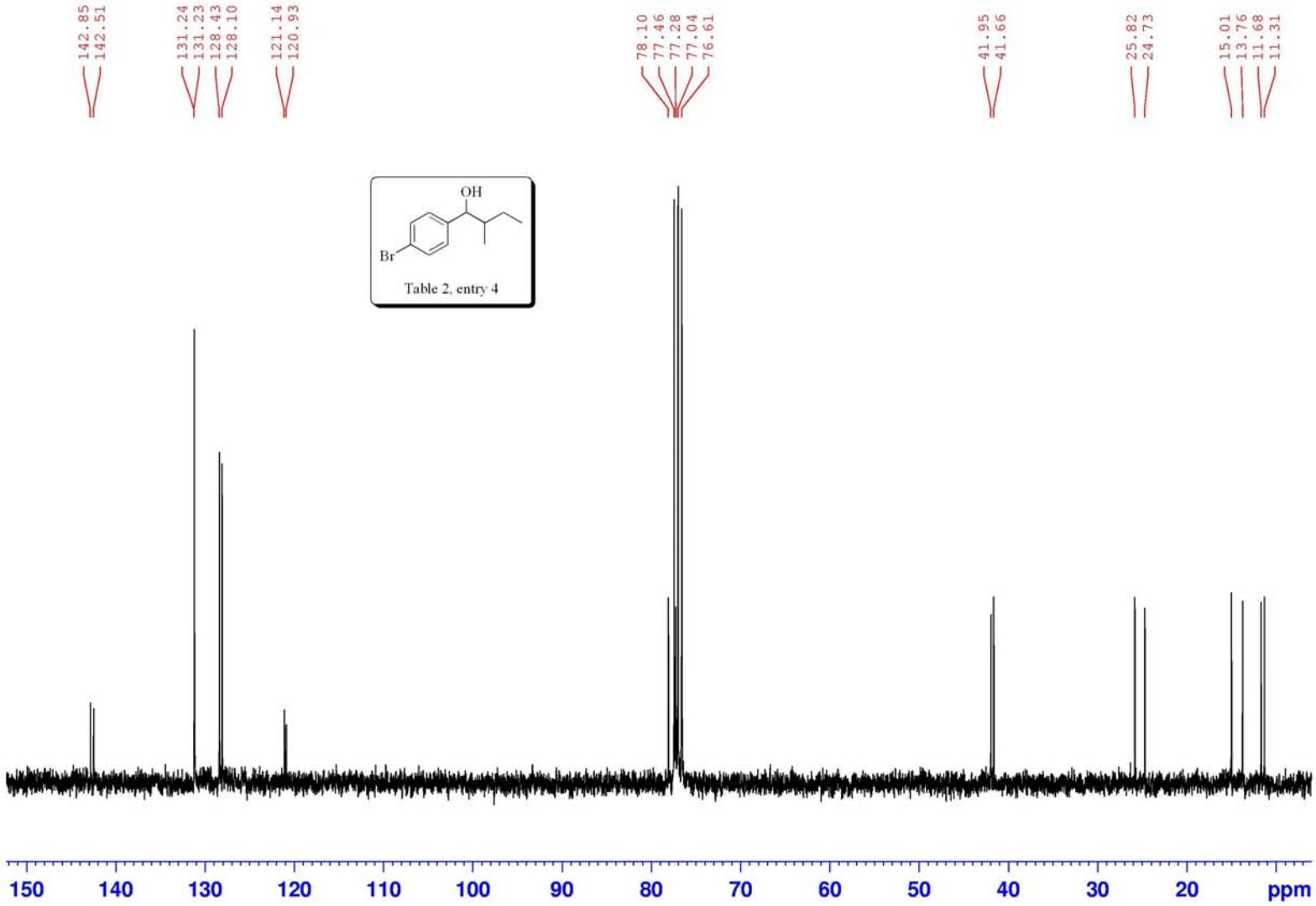
7.472
7.466
7.450
7.444
7.261
7.202
7.177
7.175
4.533
4.516
4.438
4.416
1.825
1.779
1.748
1.737
1.725
1.718
1.708
1.702
1.696
1.680
1.667
1.656
1.644
1.636
1.624
1.611
1.599
1.577
1.420
1.412
1.393
1.375
1.367
1.351
1.214
1.190
1.166
1.161
1.145
1.140
1.116
1.093
1.087
1.071
1.066
0.994
0.943
0.918
0.903
0.894
0.880
0.870
0.755
0.733
0.000
OH
Br
Table 2, entry 4
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.96
4.09
0.92
1.00
2.07
3.28
1.42
3.18
9.55
3.03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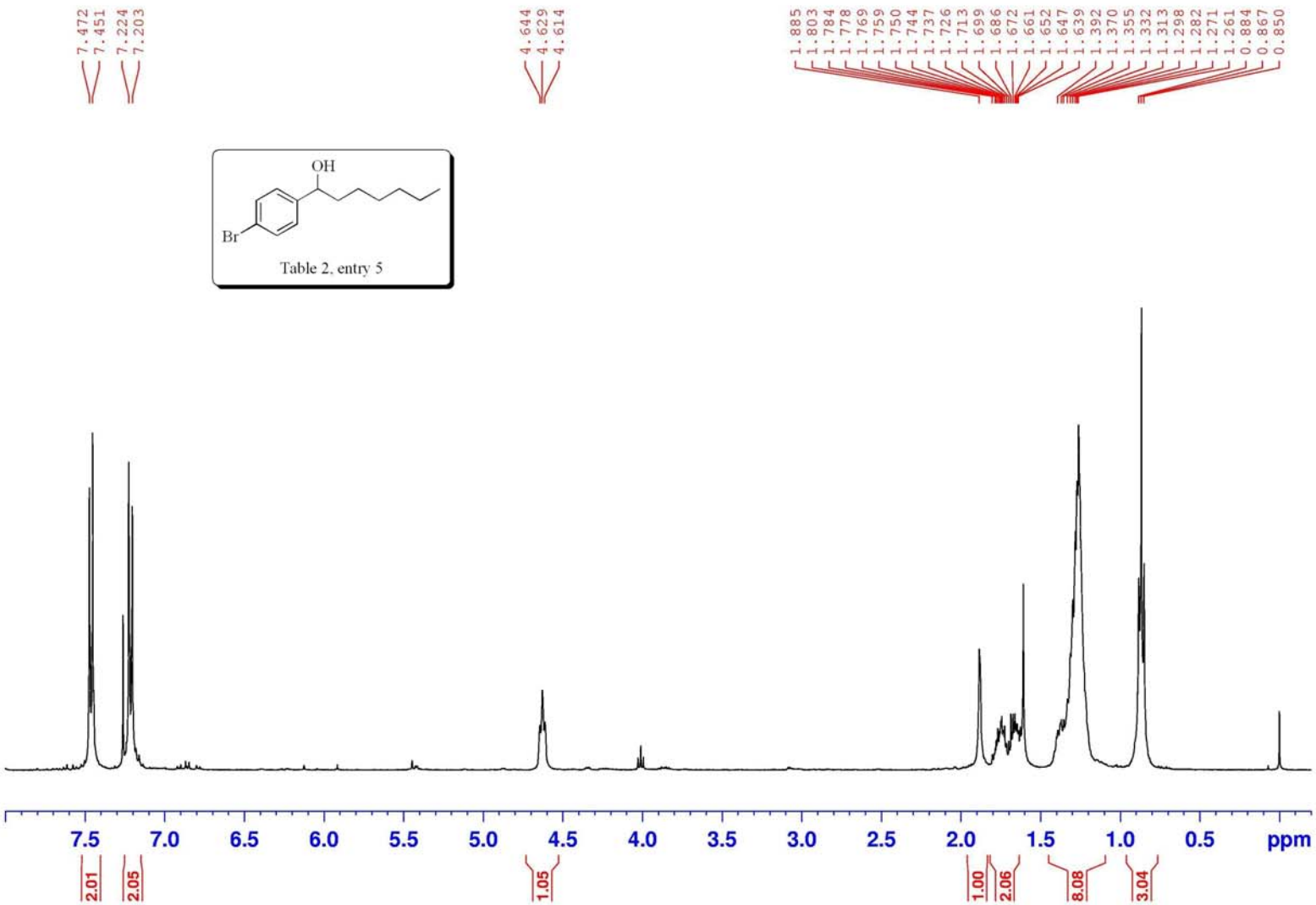
142.51
131.24
131.23
128.43
128.10
121.14
120.93
78.10
77.46
77.28
77.04
76.61
41.95
41.66
25.82
24.73
15.01
13.76
11.68
11.31
OH
Br
Table 2, entry 4
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.451
7.224
7.203
4.644
4.629
4.614
1.885
1.803
1.784
1.778
1.769
1.759
1.750
1.744
1.737
1.726
1.713
1.699
1.686
1.672
1.661
1.652
1.647
1.639
1.392
1.370
1.355
1.332
1.313
1.298
1.282
1.271
1.261
0.884
0.867
0.850
OH
Br
Table 2, entry 5
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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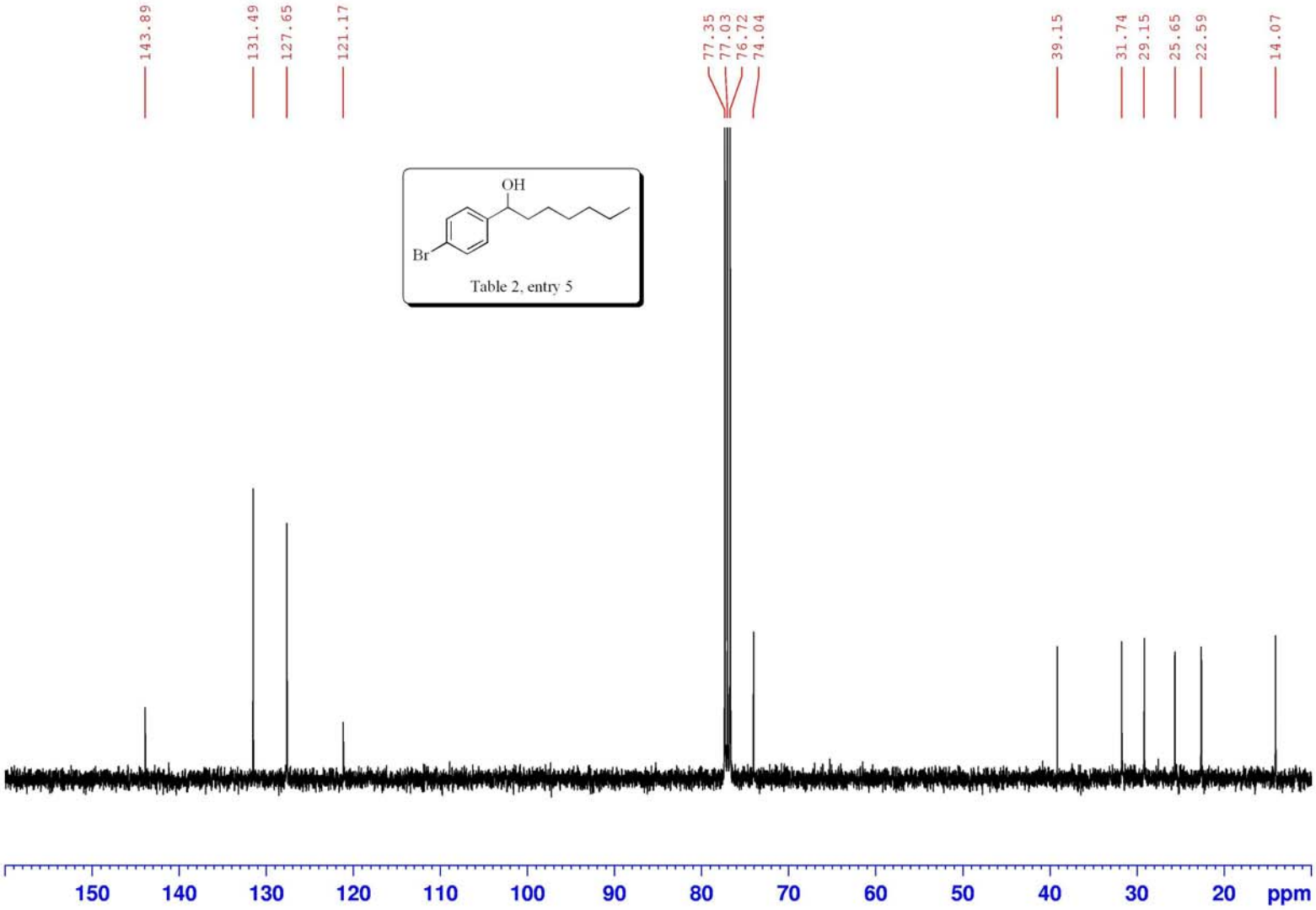
2.05
1.05
1.00
2.06
8.08
3.04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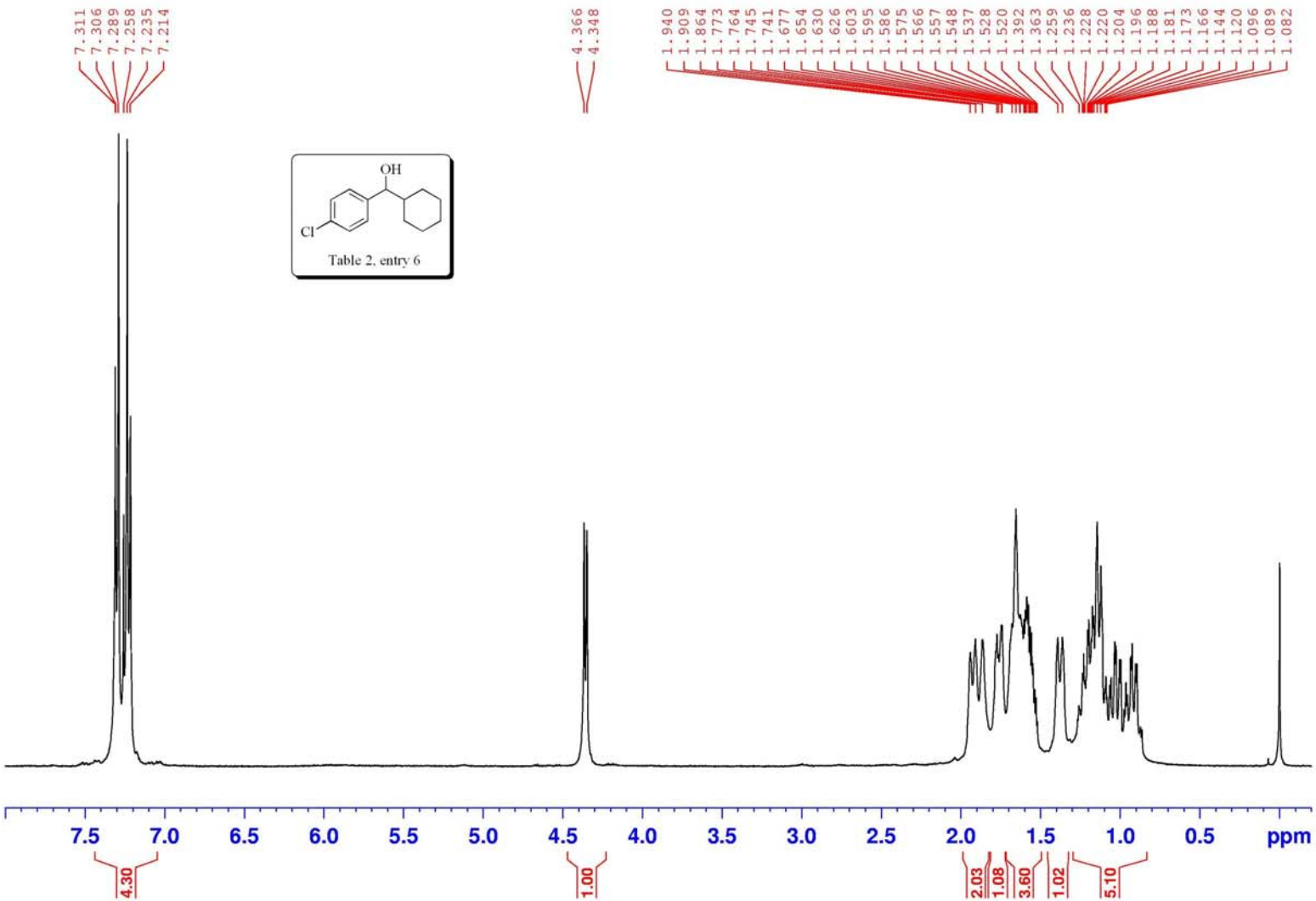
131.49
127.65
121.17
77.35
77.03
76.72
74.04
39.15
31.74
29.15
25.65
22.59
14.07
OH
Br
Table 2, entry 5
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.289
7.258
7.235
7.214
4.366
4.348
1.940
1.909
1.864
1.773
1.764
1.745
1.741
1.677
1.654
1.630
1.626
1.603
1.595
1.586
1.575
1.566
1.557
1.548
1.537
1.528
1.520
1.392
1.363
1.259
1.236
1.228
1.220
1.204
1.196
1.188
1.181
1.173
1.166
1.144
1.120
1.096
1.089
1.082
OH
Cl
Table 2, entry 6
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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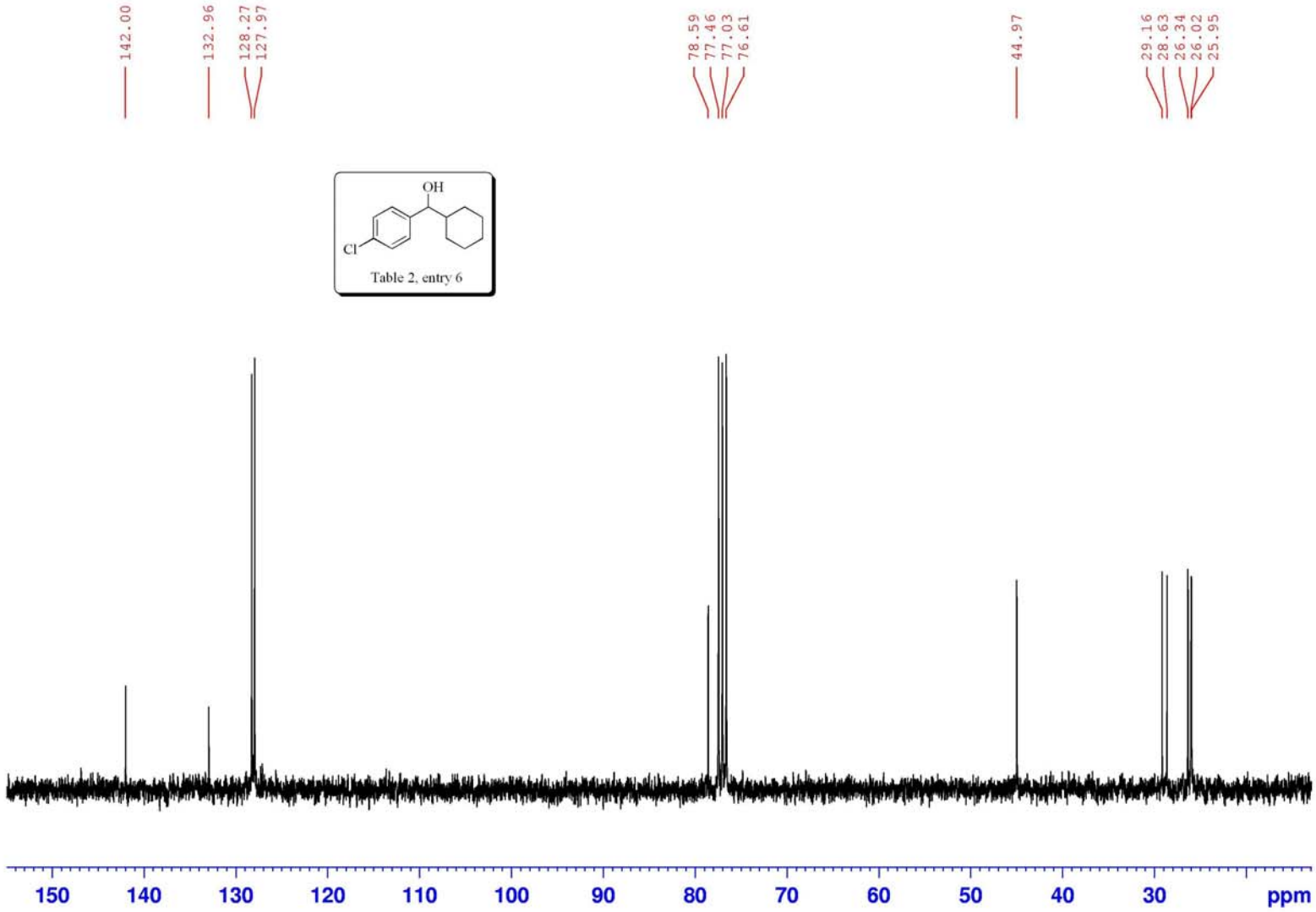
1.00
2.03
1.08
3.60
1.02
5.10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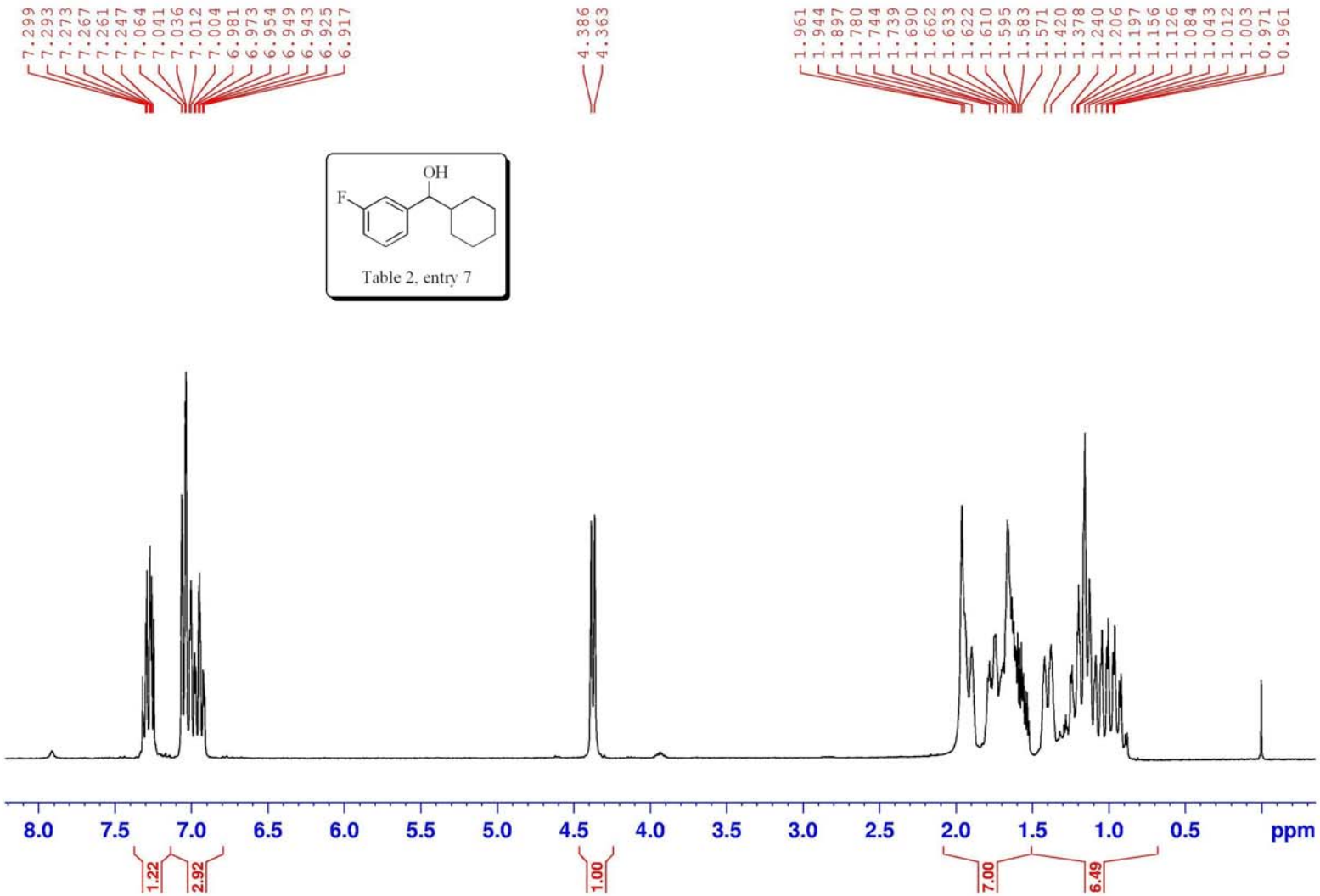
128.27
127.97
78.59
77.46
77.03
76.61
44.97
29.16
28.63
26.34
26.02
25.95
OH
Cl
Table 2, entry 6
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm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7.273
7.267
7.261
7.247
7.064
7.041
7.036
7.012
7.004
6.981
6.973
6.954
6.949
6.943
6.925
6.917
4.386
4.363
1.961
1.944
1.897
1.780
1.744
1.739
1.690
1.662
1.633
1.622
1.610
1.595
1.583
1.571
1.420
1.378
1.240
1.206
1.197
1.156
1.126
1.084
1.043
1.012
1.003
0.971
0.961
OH
F
Table 2, entry 7
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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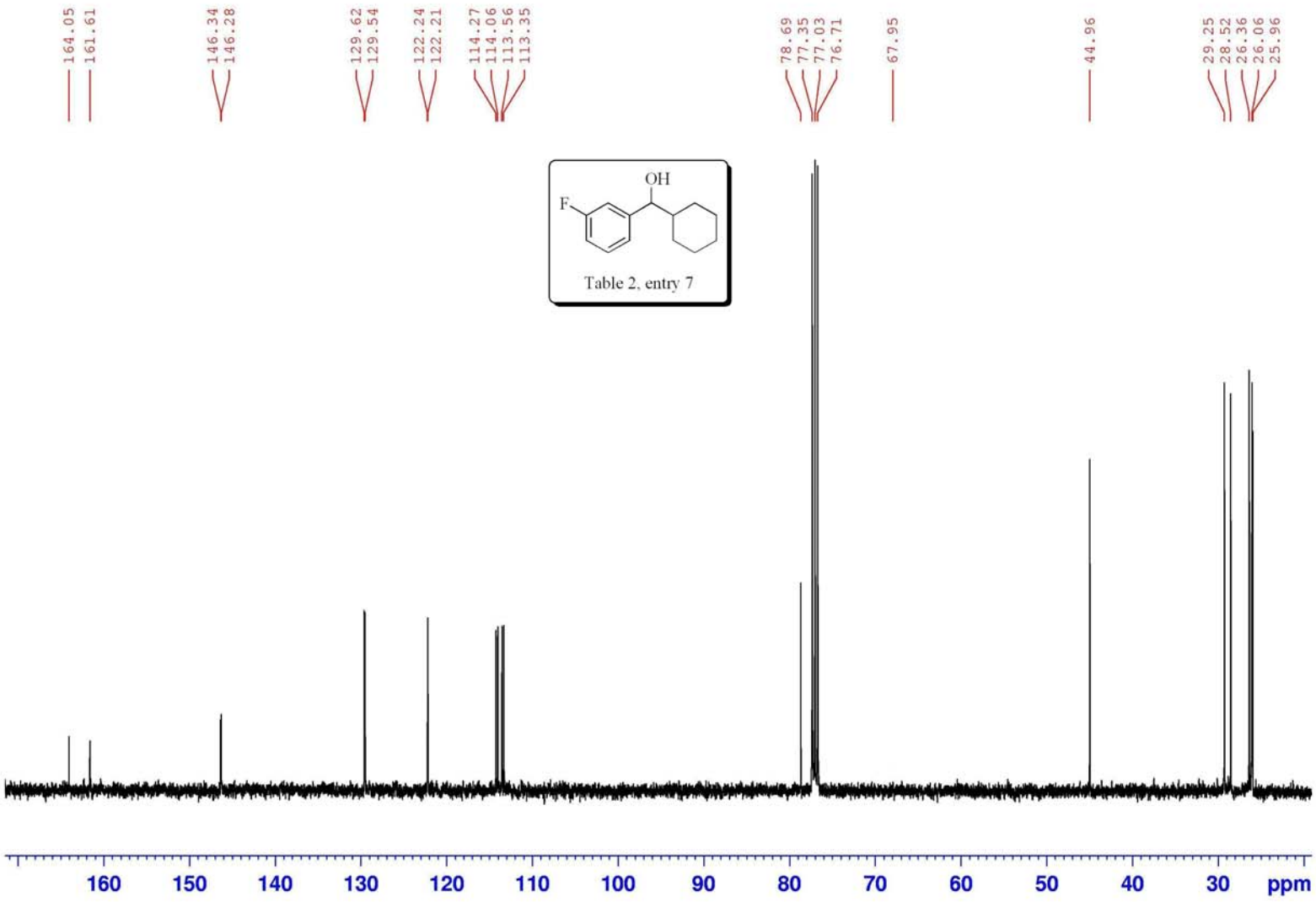
1.00
7.00
6.49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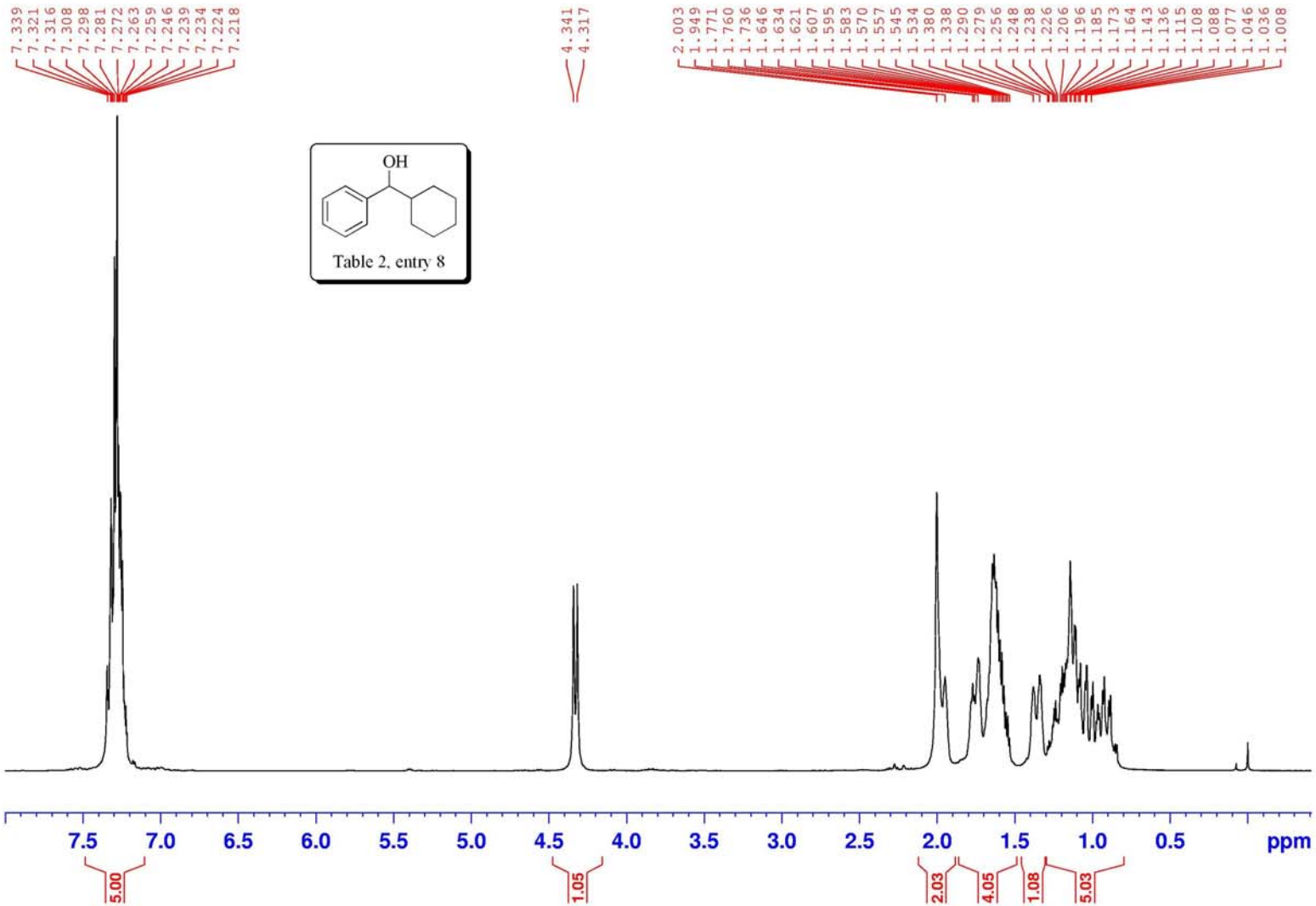
113.56
113.35
78.69
77.35
77.03
76.71
67.95
44.96
29.25
28.52
26.36
26.06
25.96
OH
F
Table 2, entry 7
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm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7.281
7.272
7.263
7.259
7.246
7.239
7.234
7.224
7.218
4.341
4.317
2.003
1.949
1.771
1.760
1.736
1.646
1.634
1.621
1.607
1.595
1.583
1.570
1.557
1.545
1.534
1.380
1.338
1.290
1.279
1.256
1.248
1.238
1.226
1.206
1.196
1.185
1.173
1.164
1.143
1.136
1.115
1.108
1.088
1.077
1.046
1.036
1.008
OH
Table 2, entry 8
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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1.5
1.0
0.5
ppm
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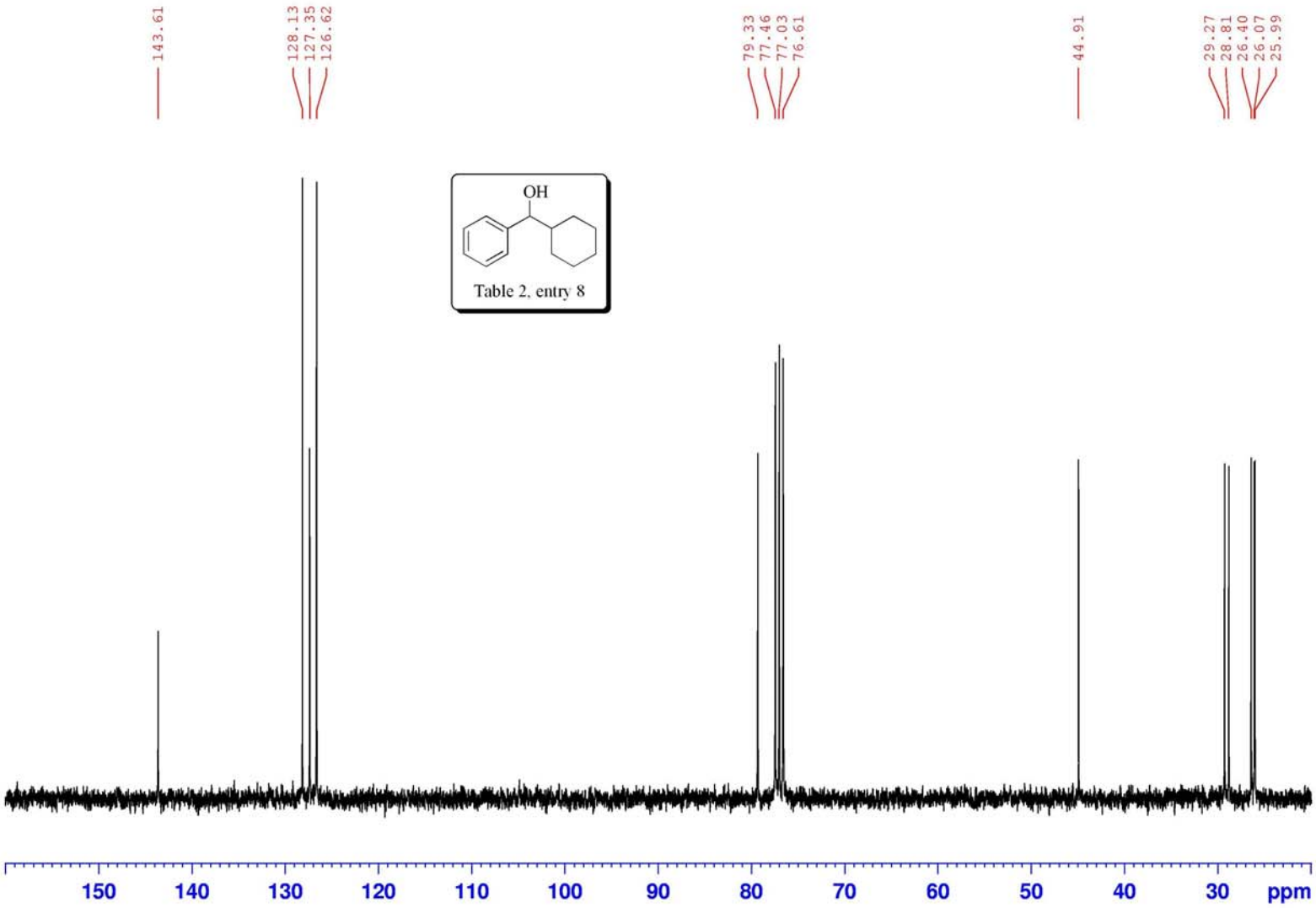
1.05
2.03
4.05
1.08
5.03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126.62
79.33
77.46
77.03
76.61
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29.27
28.81
26.40
26.07
25.99
OH
Table 2, entry 8
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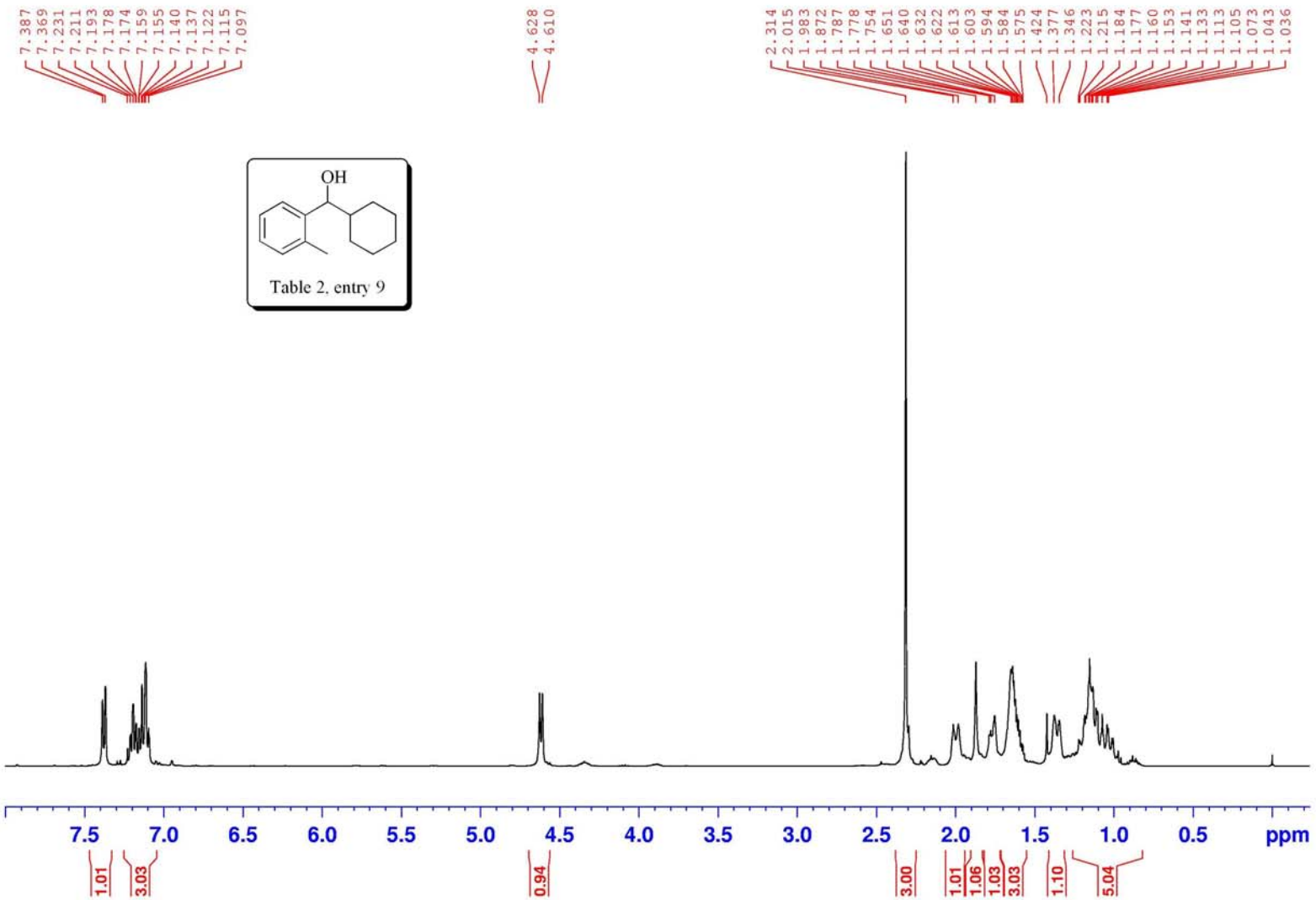
140
130
120
110
100
90
80
70
60
50
40
30
ppm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7.159
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7.137
7.122
7.115
7.097
4.628
4.610
2.314
2.015
1.983
1.872
1.787
1.778
1.754
1.651
1.640
1.632
1.622
1.613
1.603
1.594
1.584
1.575
1.424
1.377
1.346
1.223
1.215
1.184
1.177
1.160
1.153
1.141
1.133
1.113
1.105
1.073
1.043
1.036
OH
Table 2, entry 9
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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3.03
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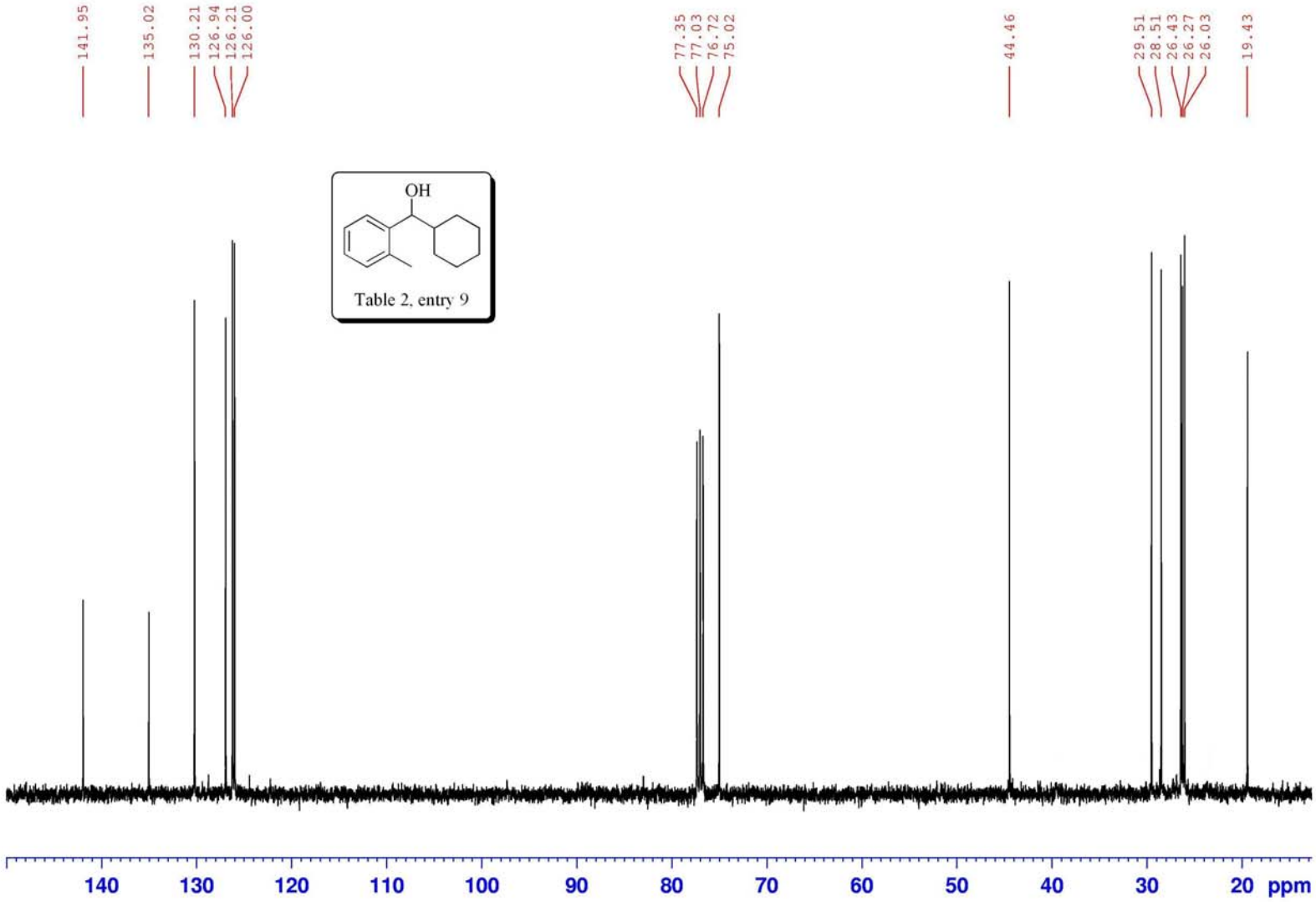
1.01
1.06
1.03
3.03
1.10
5.04


141.95
135.02
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126.21
126.00
77.35
77.03
76.72
75.02
44.46
29.51
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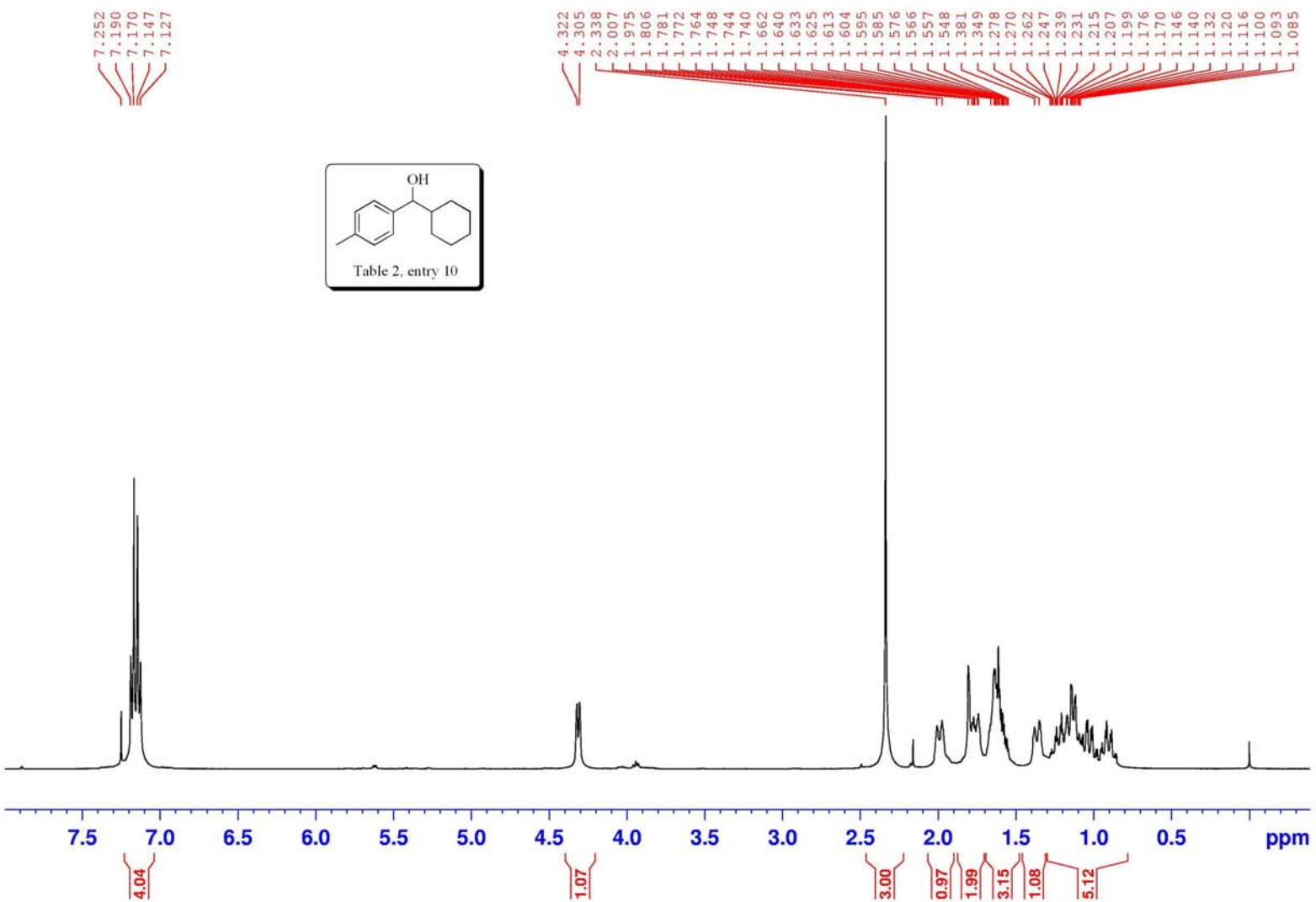
26.43
26.27
26.03
19.43
OH
Table 2, entry 9
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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4.322
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2.338
2.007
1.975
1.806
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1.772
1.764
1.748
1.744
1.740
1.662
1.640
1.633
1.625
1.613
1.604
1.595
1.585
1.576
1.566
1.557
1.548
1.381
1.349
1.278
1.270
1.262
1.247
1.239
1.231
1.215
1.207
1.199
1.176
1.170
1.146
1.140
1.132
1.120
1.116
1.100
1.093
1.085
OH
Table 2, entry 10
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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ppm
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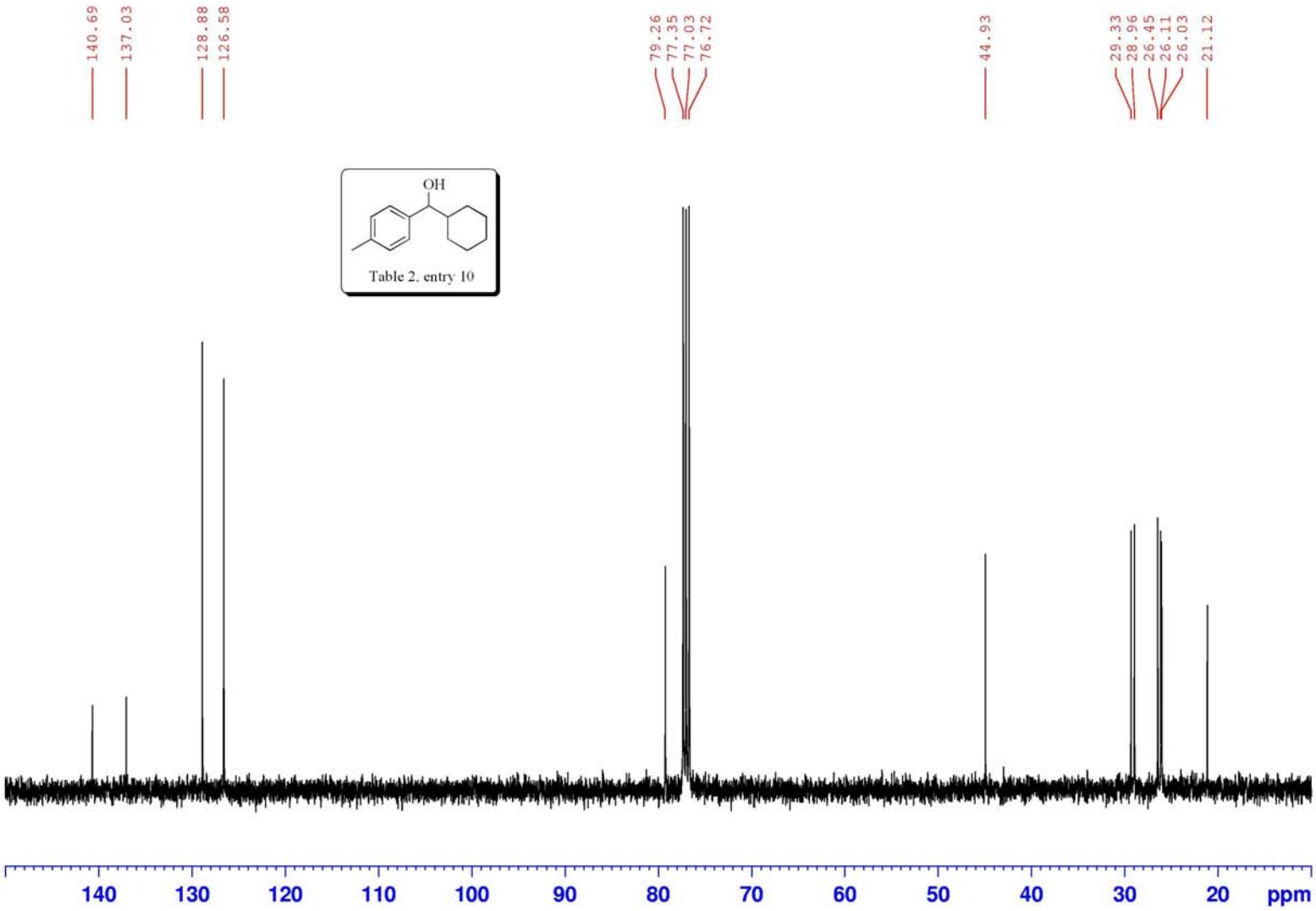
5.12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128.88
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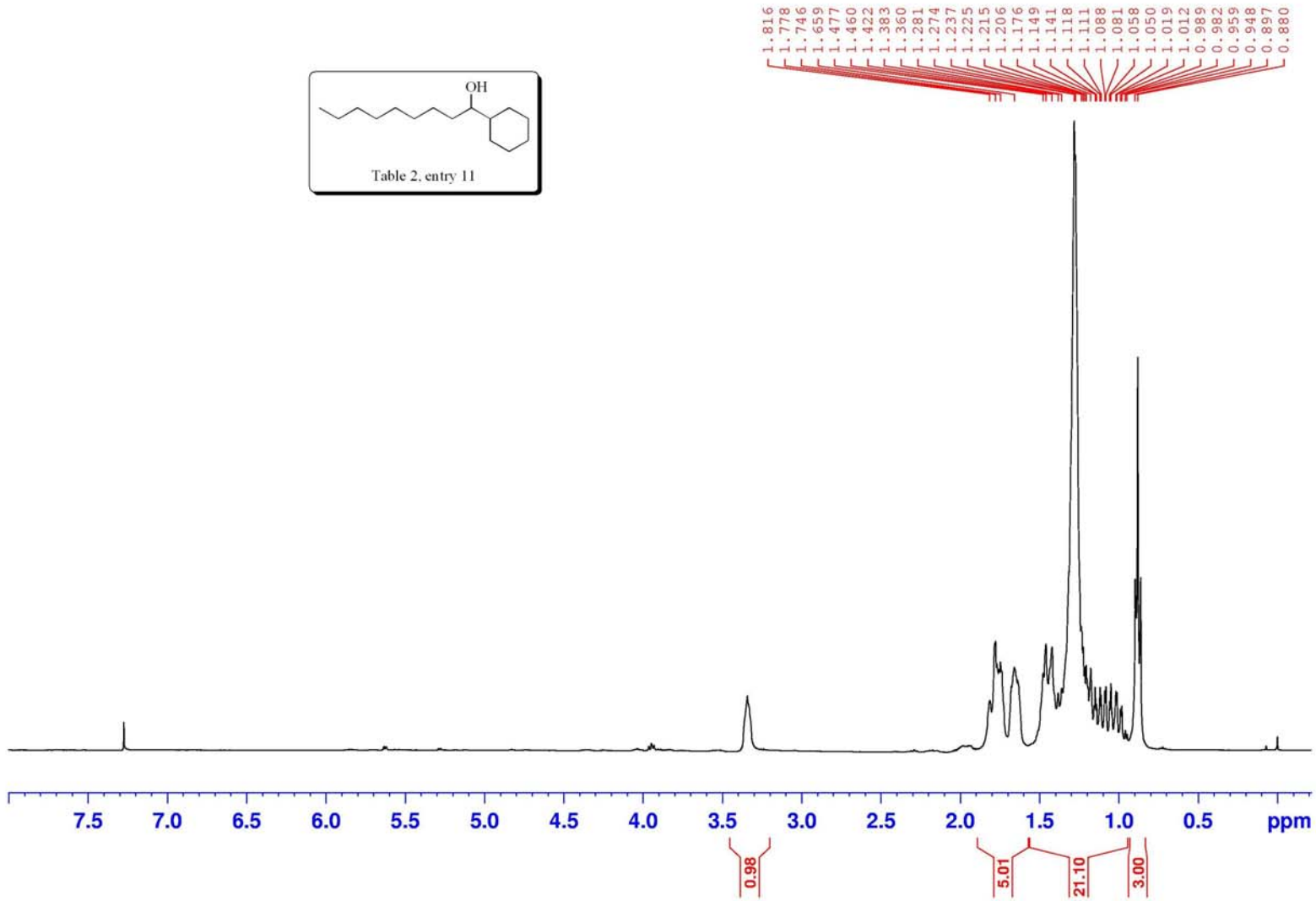
79.26
77.35
77.03
76.72
44.93
29.33
28.96
26.45
26.11
26.03
21.12
OH
Table 2, entry 10
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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1.659
1.477
1.460
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1.383
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1.237
1.225
1.215
1.206
1.176
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1.141
1.118
1.111
1.088
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1.058
1.050
1.019
1.012
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0.982
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0.948
0.897
0.880
7.5
7.0
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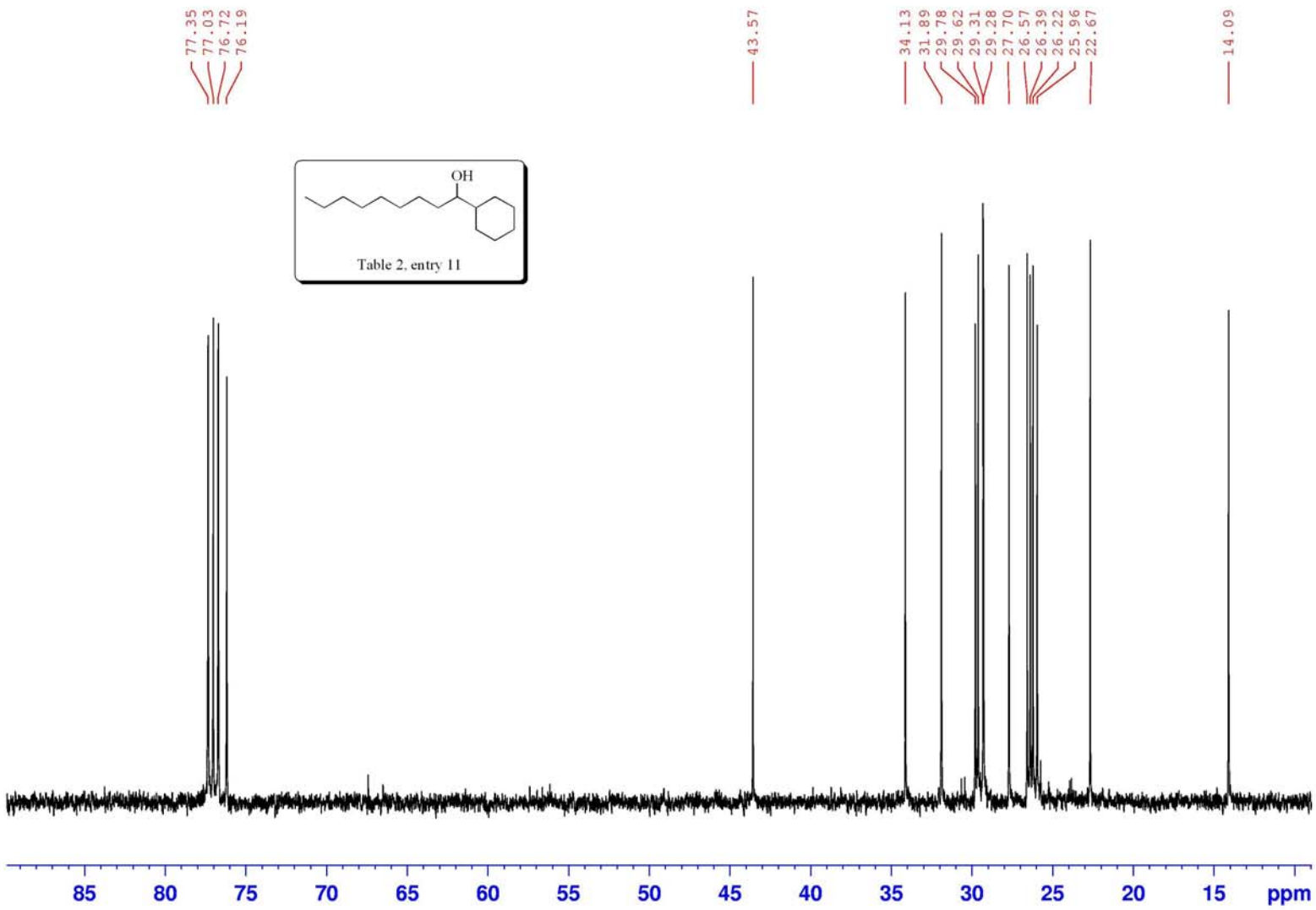
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.98
5.01
21.10
3.00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77.03
76.72
76.19
43.57
34.13
31.89
29.78
29.62
29.31
29.28
27.70
26.57
26.39
26.22
25.96
22.67
14.09
OH
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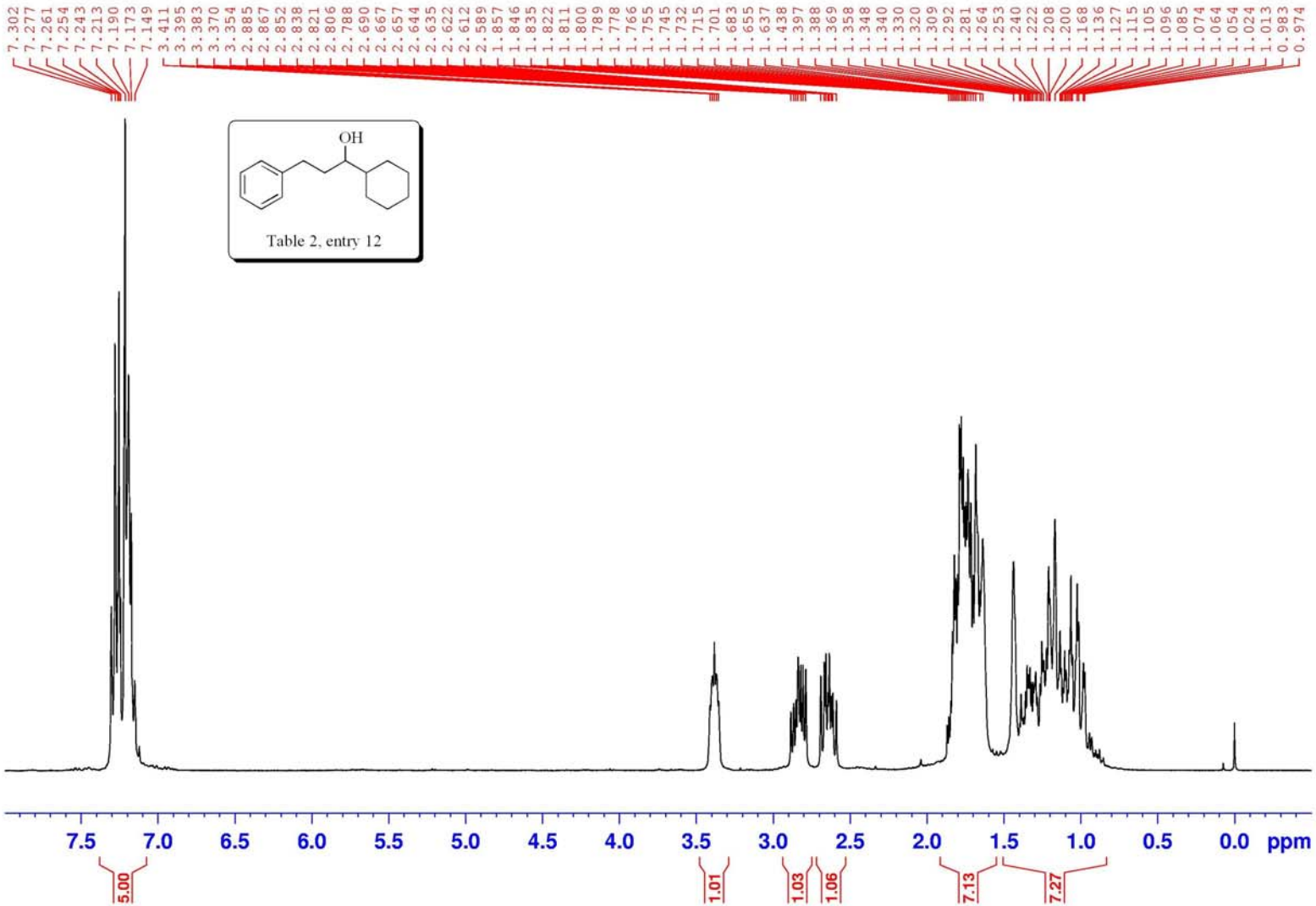
80
75
70
65
60
55
50
45
40
35
30
25
20
15
ppm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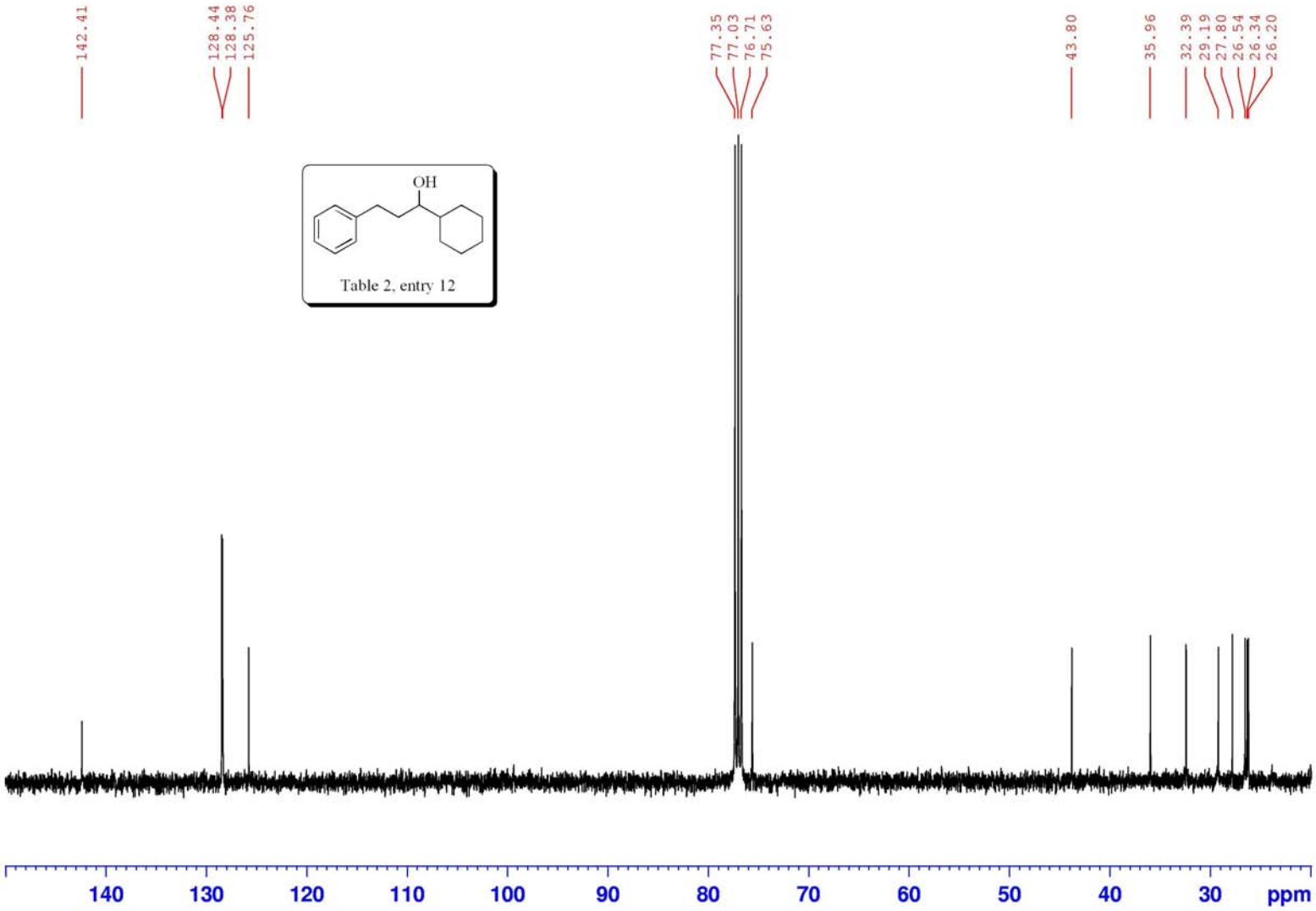
7.277
7.261
7.254
7.243
7.213
7.190
7.173
7.149
3.411
3.395
3.383
3.370
3.354
2.885
2.867
2.852
2.838
2.821
2.806
2.788
2.690
2.667
2.657
2.644
2.635
2.622
2.612
2.589
1.857
1.846
1.835
1.822
1.811
1.800
1.789
1.778
1.766
1.755
1.745
1.732
1.715
1.701
1.683
1.655
1.637
1.438
1.397
1.388
1.369
1.358
1.348
1.340
1.330
1.320
1.309
1.292
1.281
1.264
1.253
1.240
1.222
1.208
1.200
1.168
1.136
1.127
1.115
1.105
1.096
1.085
1.074
1.064
1.054
1.024
1.013
0.983
0.974
OH
Table 2, entry 12
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
5.00
1.01
1.03
1.06
7.13
7.27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128.38
125.76
77.35
77.03
76.71
75.63
43.80
35.96
32.39
29.19
27.80
26.54
26.34
26.20
OH
Table 2, entry 12
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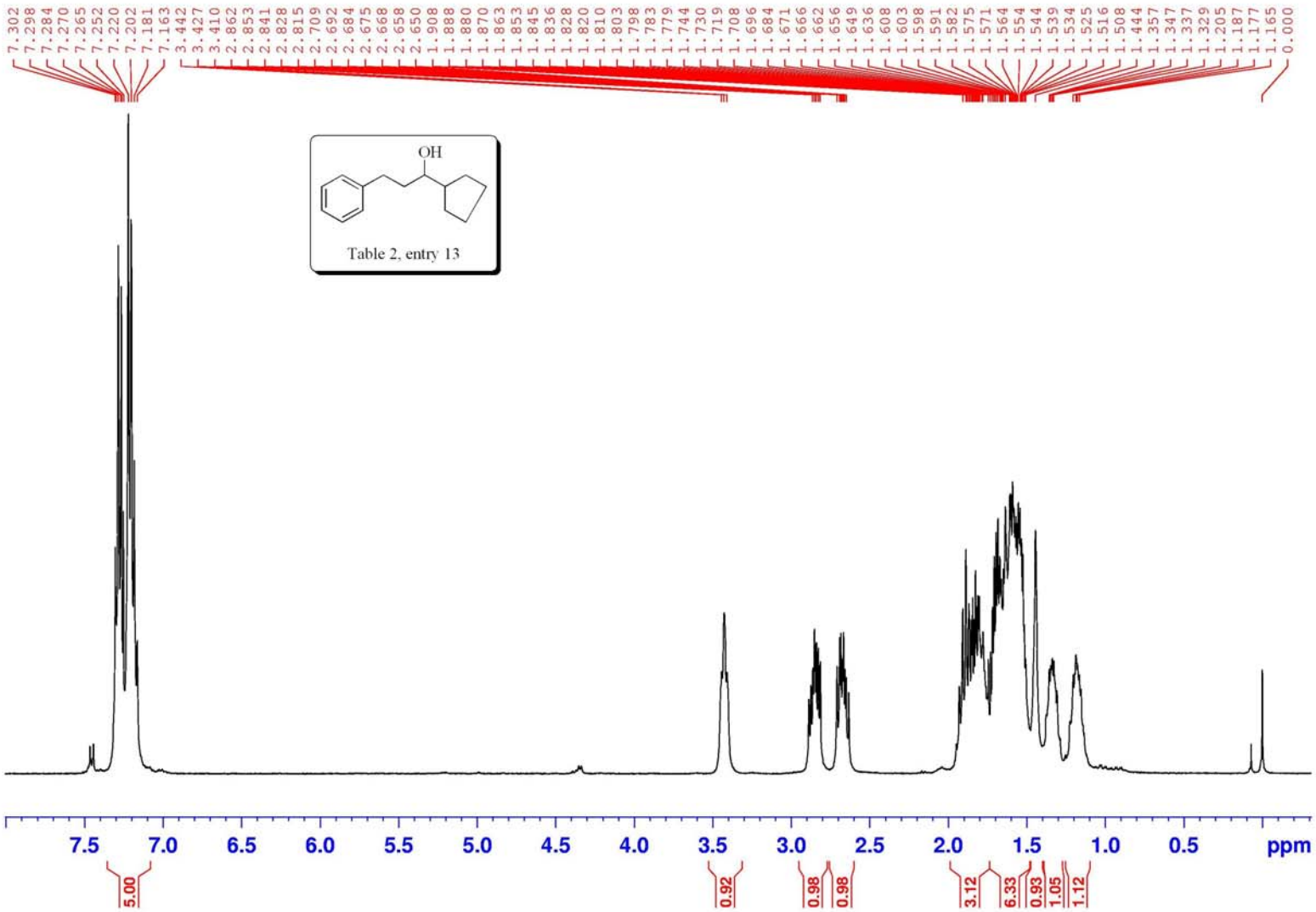
130
120
110
100
90
80
70
60
50
40
30
ppm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7.284
7.270
7.265
7.252
7.220
7.202
7.181
7.163
3.442
3.427
3.410
2.862
2.853
2.841
2.828
2.815
2.709
2.692
2.684
2.675
2.668
2.658
2.650
1.908
1.888
1.880
1.870
1.863
1.853
1.845
1.836
1.828
1.820
1.810
1.803
1.798
1.783
1.779
1.744
1.730
1.719
1.708
1.696
1.684
1.671
1.666
1.662
1.656
1.649
1.636
1.608
1.603
1.598
1.591
1.582
1.575
1.571
1.564
1.554
1.544
1.539
1.534
1.525
1.516
1.508
1.444
1.357
1.347
1.337
1.329
1.205
1.187
1.177
1.165
0.000
OH
Table 2, entry 13
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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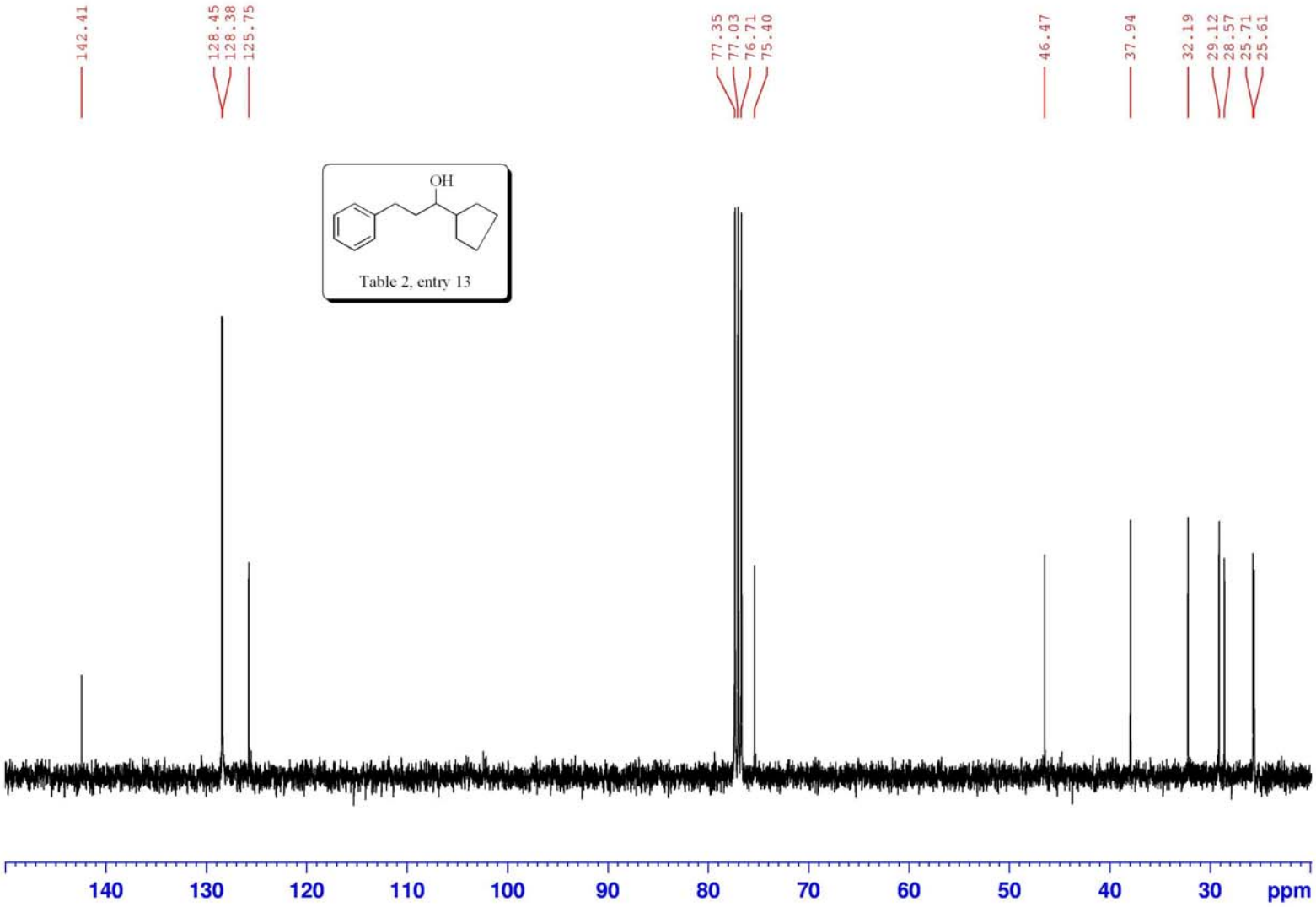
0.98
0.98
3.12
6.33
0.93
1.05
1.12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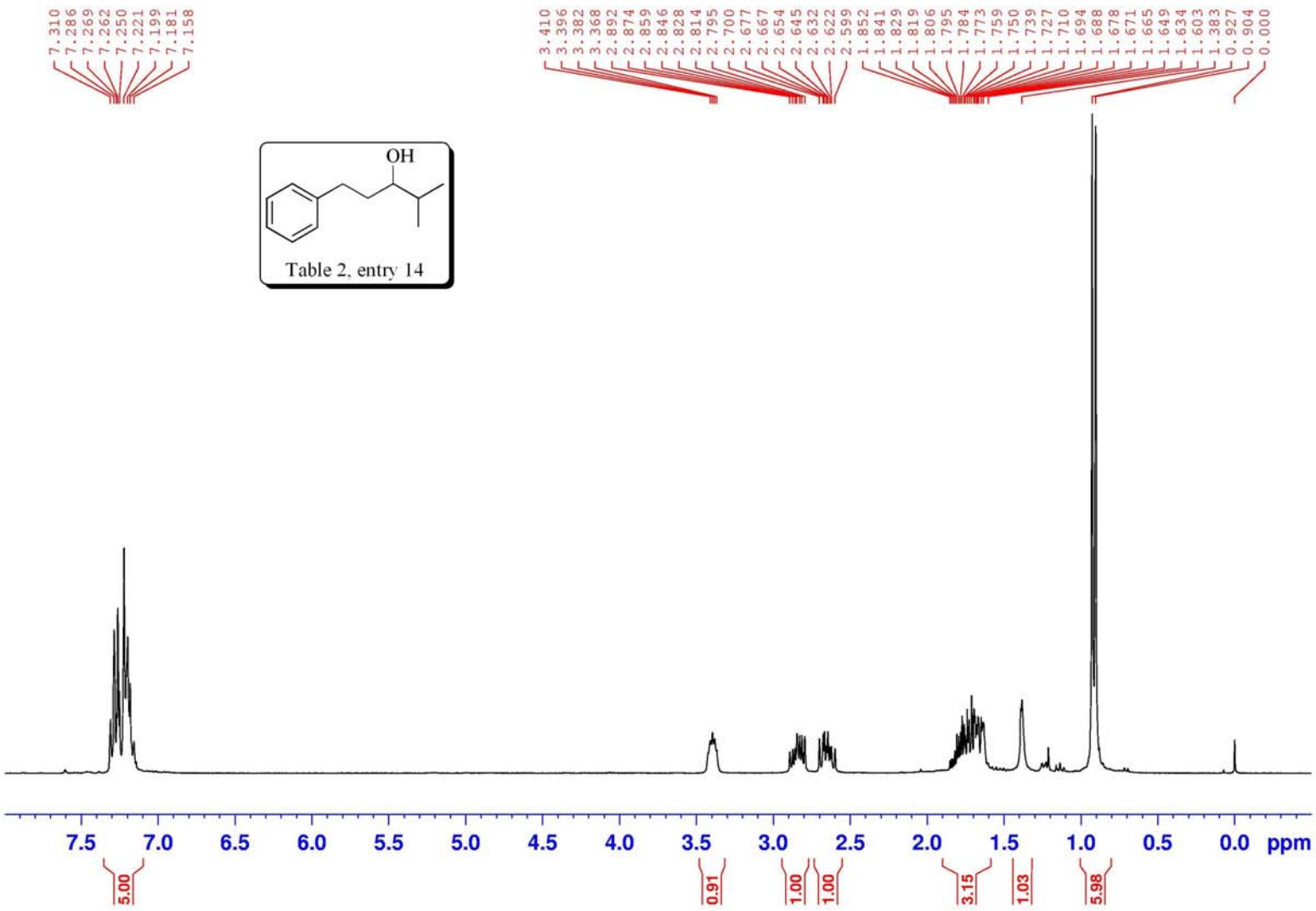
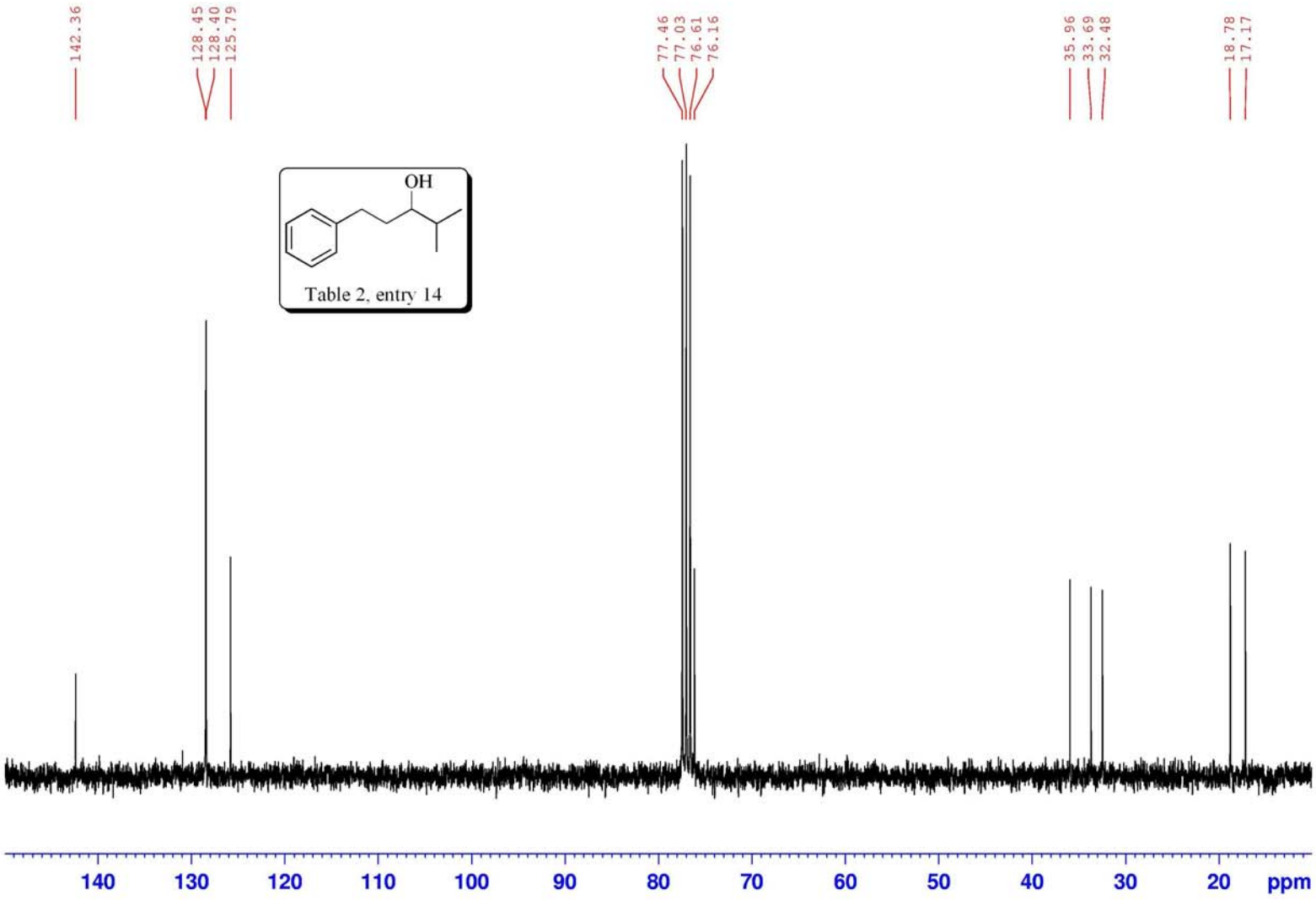
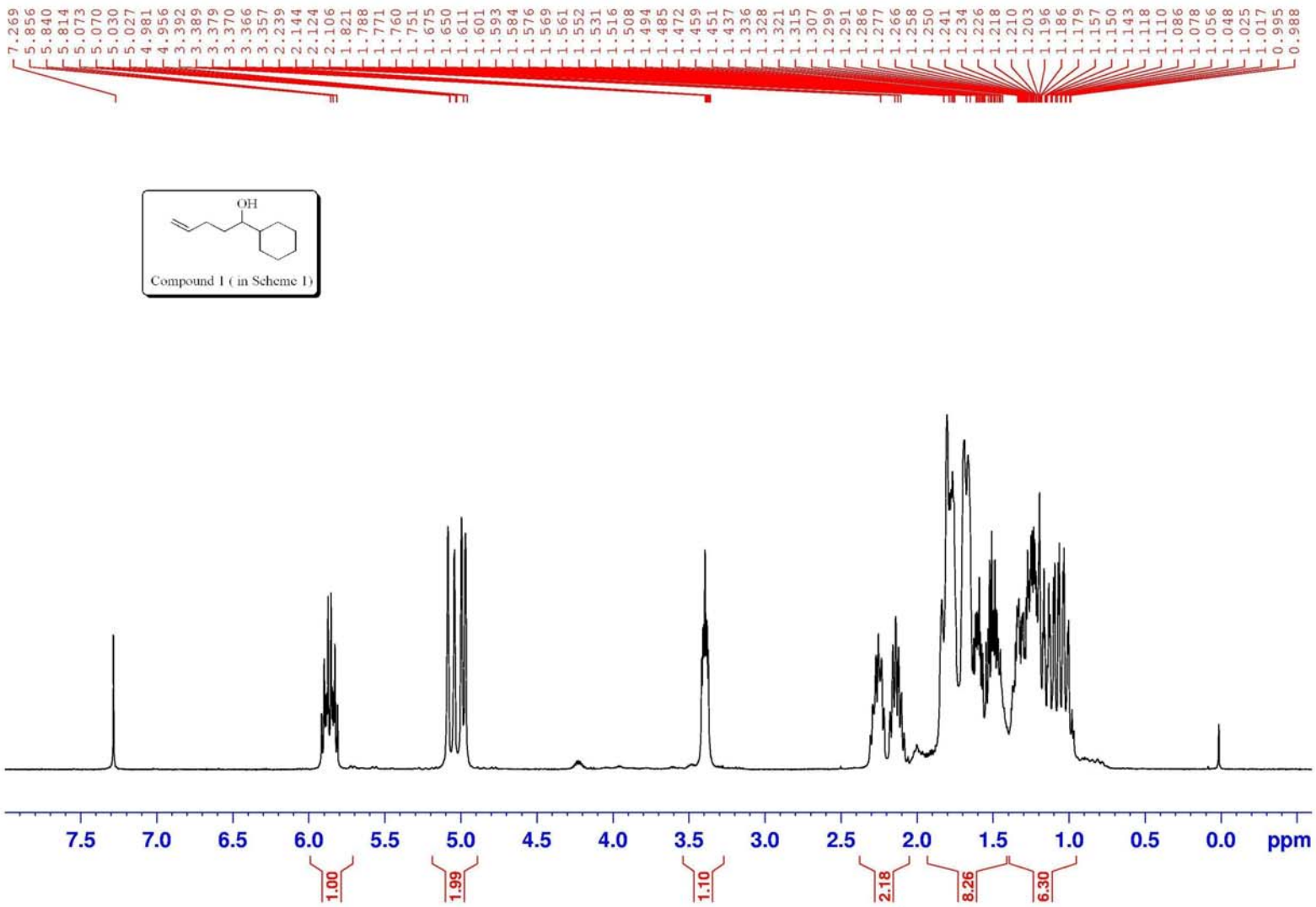
128.45
128.38
125.75
77.35
77.03
76.71
75.40
46.47
37.94
32.19
29.12
28.57
25.71
25.61
OH
Table 2, entry 13
140
130
120
110
100
90
80
70
60
50
40
30
ppm


7.310
7.286
7.269
7.262
7.250
7.221
7.199
7.181
7.158
3.410
3.396
3.382
3.368
2.892
2.874
2.859
2.846
2.828
2.814
2.795
2.700
2.677
2.667
2.654
2.645
2.632
2.622
2.599
1.852
1.841
1.829
1.819
1.806
1.795
1.784
1.773
1.759
1.750
1.739
1.727
1.710
1.694
1.688
1.678
1.671
1.665
1.649
1.634
1.603
1.383
0.927
0.904
0.000
OH
Table 2, entry 14
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
5.00
0.91
1.00
1.00
3.15
1.03
5.98


142.36
128.45
128.40
125.79
77.46
77.03
76.61
76.16
35.96
33.69
32.48
18.78
17.17
OH
Table 2, entry 14
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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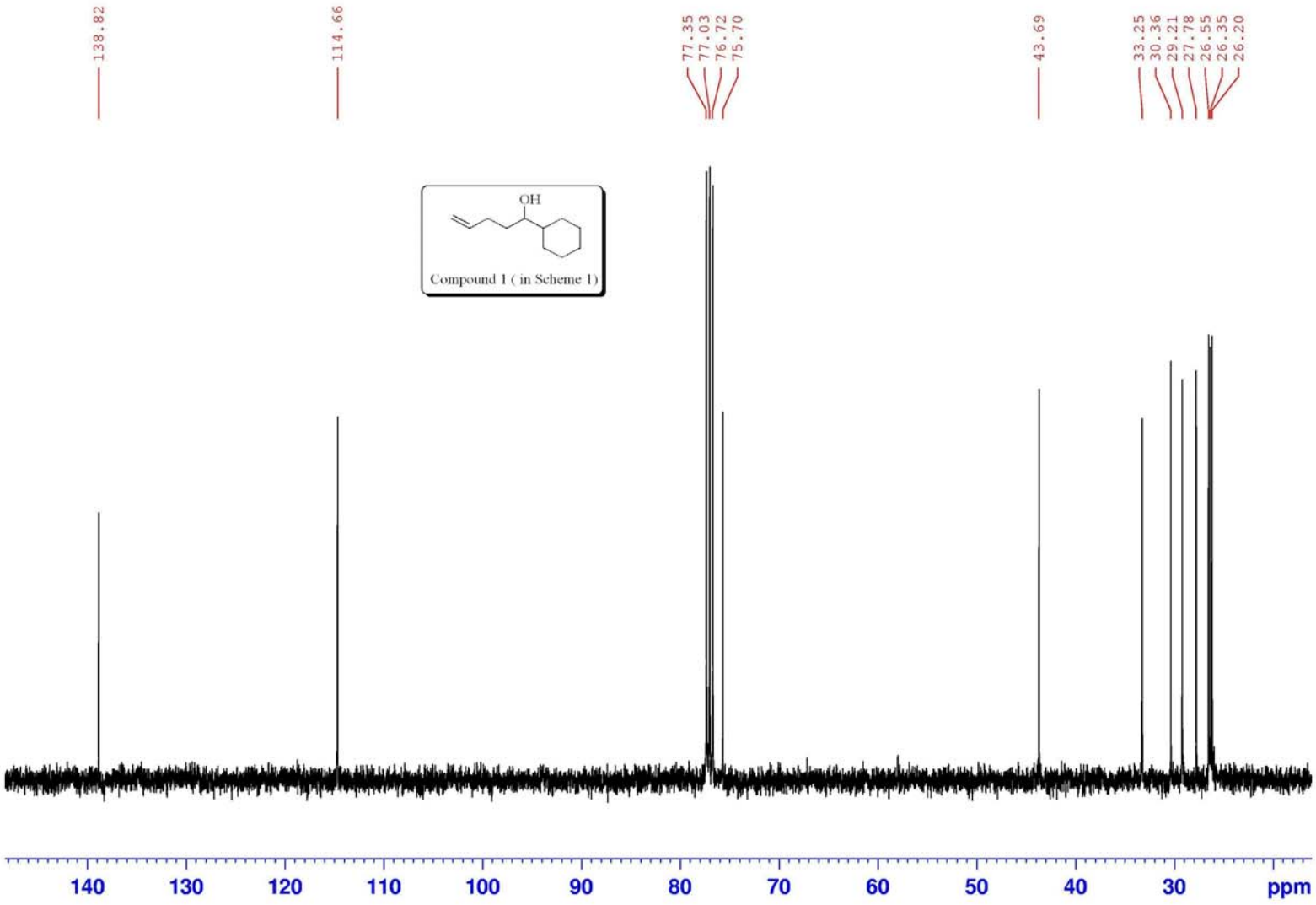
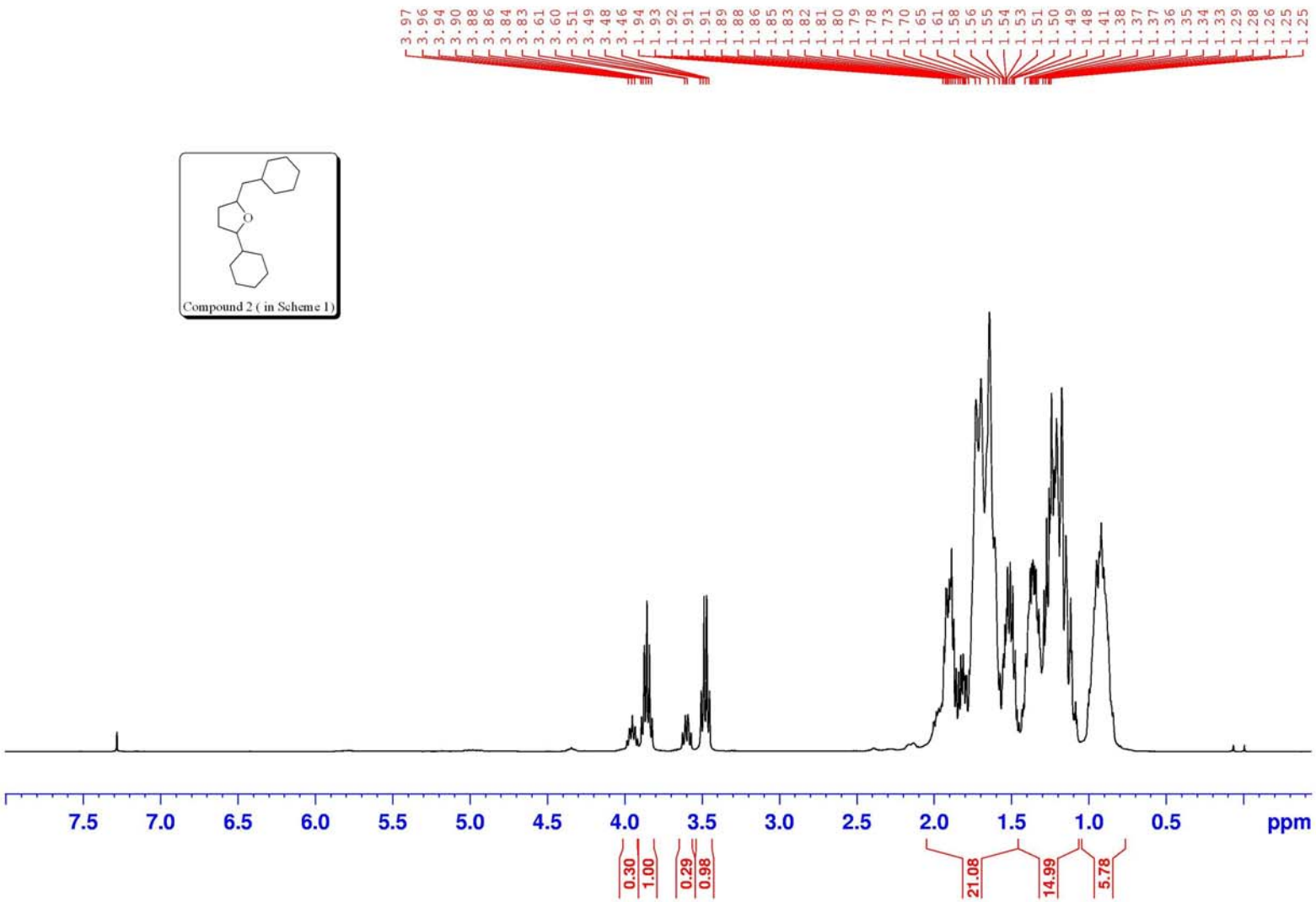
5.840
5.814
5.073
5.070
5.030
5.027
4.981
4.956
3.392
3.389
3.379
3.370
3.366
3.357
2.239
2.144
2.124
2.106
1.821
1.788
1.771
1.760
1.751
1.675
1.650
1.611
1.601
1.593
1.584
1.576
1.569
1.561
1.552
1.531
1.516
1.508
1.494
1.485
1.472
1.459
1.451
1.437
1.336
1.328
1.321
1.315
1.307
1.299
1.291
1.286
1.277
1.266
1.258
1.250
1.241
1.234
1.226
1.218
1.210
1.203
1.196
1.186
1.179
1.157
1.150
1.143
1.118
1.110
1.086
1.078
1.056
1.048
1.025
1.017
0.995
0.988
OH
Compound 1 (in Scheme 1)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.99
1.10
2.18
8.26
6.30


138.82
114.66
77.35
77.03
76.72
75.70
43.69
33.25
30.36
29.21
27.78
26.55
26.35
26.20
OH
Compound 1 (in Scheme 1)
140
130
120
110
100
90
80
70
60
50
40
30
ppm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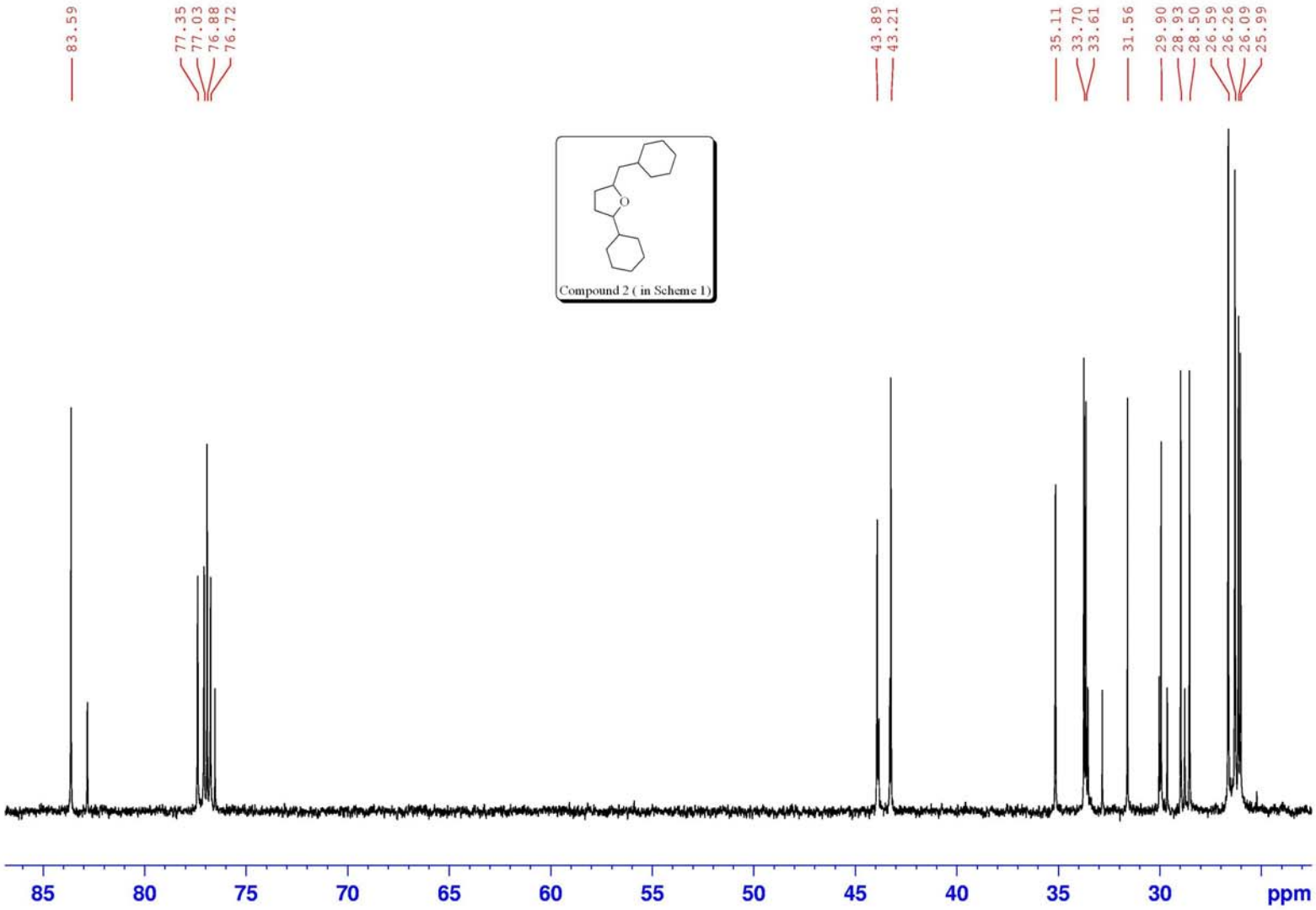
3.96
3.94
3.90
3.88
3.86
3.84
3.83
3.61
3.60
3.51
3.49
3.48
3.46
1.94
1.93
1.92
1.91
1.91
1.89
1.88
1.86
1.85
1.83
1.82
1.81
1.80
1.79
1.78
1.73
1.70
1.65
1.61
1.58
1.56
1.55
1.54
1.53
1.51
1.50
1.49
1.48
1.41
1.38
1.37
1.37
1.36
1.35
1.34
1.33
1.29
1.28
1.26
1.25
1.25
O
Compound 2 (in Scheme 1)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.30
1.00
0.29
0.98
21.08
14.99
5.78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77.03
76.88
76.72
43.89
43.21
35.11
33.70
33.61
31.56
29.90
28.93
28.50
26.59
26.26
26.09
25.99
O
Compound 2 (in Scheme 1)
85
80
75
70
65
60
55
50
45
40
35
30
ppm