Supporting Information for

# First Practical Cross-Alkylation of Primary Alcohols with a New and Recyclable Impregnated Iridium on Magnetite Catalyst

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## General Information

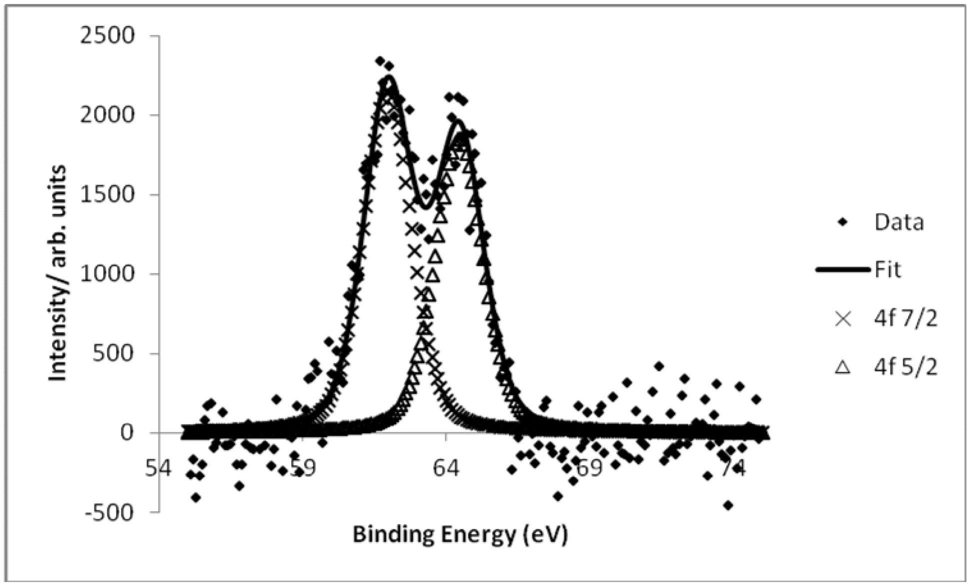
XPS analyses were carried out on a VG-Microtech Mutilab. XRD analyses were obtained on a BRUKER D-8 ADVANCE diffractometer with Göebel mirror, with a high temperature chamber (up to 900ºC), with a X-ray generator KRISTALLOFLEX K 760-80F (3KW, 20-60KV and 5-80mA). TEM images were obtained on a JEOL, model JEM-2010 equipped with an X-ray detector OXFORD INCA Energy TEM 100 for microanalysis (EDS). XRF analyses were obtained on a PHILIPS MAGIX PRO (PW2400) X-ray spectrometer equipped with a rhodium X-ray tube and a beryllium window. BET isotherms were carried out on a AUTOSORB-6 (Quantachrome), using $N_2$. Melting points were obtained with a Reichert Thermovar apparatus. NMR spectra were recorded on a Bruker AC-300 (300 MHz for $^1H$ and 75 MHz for $^{13}C$) using $CDCl_3$ as a solvent and TMS as internal standard for $^1H$ and $^{13}C$; chemical shifts are given in $\delta$ (parts per million) and coupling constants ($J$) in Hertz. FT-IR spectra were obtained on a JASCO 4100LE (Pike Miracle ATR) spectrophotometer. Mass spectra (EI) were obtained at 70 eV on a Himazdu QP-5000 spectrometer, giving fragment ions in *m/z* with relative intensities (%) in parentheses. Thin layer chromatography (TLC) was carried out on Schleicher & Schuell F1400/LS 254 plates coated with a 0.2 mm layer of silica gel; detection by $UV_{254}$ light, staining with phosphomolybdic acid [25 g phosphomolybdic acid, 10 g $Ce(SO_4)_2$ 4 $H_2O$, 60 mL of concentrated $H_2SO_4$ and 940 mL $H_2O$]. Column chromatography was performed using silica gel 60 of 35-70 mesh. All reagents were commercially available (Acros, Aldrich, Fluorochem) and were used as received. The ICP-MS analyses were carried out on a Thermo Elemental VGPQ-ExCell spectrometer. The Elemental Analysis were performed on a Elemental Microanalyzer Thermo Finningan Flash 1112 Series.

## General procedure for the preparation of $IrO_2$-$Fe_3O_4$ catalyst

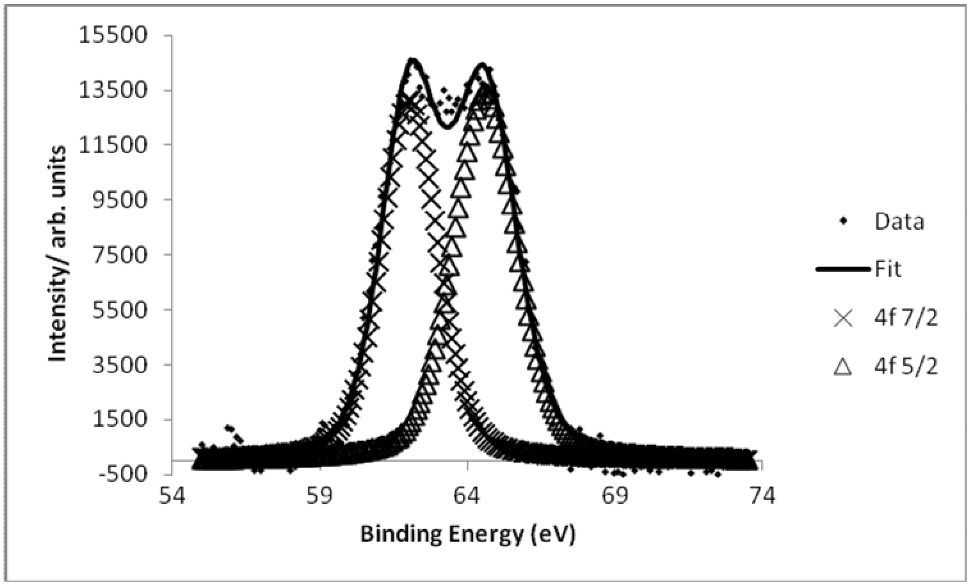
To a stirred solution of the metal salt $IrCl_3$ (1 mmol, 299 mg) in deionised water (150 mL) was added commercial available $Fe_3O_4$ (4 g, 17 mmol, powder < 5 μm, BET area: 9.86 $m^2/g$). After 10 minutes at room temperature, the mixture was slowly basified with NaOH (1M) until pH around 13. The mixture was stirred during one day at room temperature in air. After that, the catalyst was filtered and washed several times with deionized water (3 × 10 mL). The solid was dried at 100ºC during 24 h in a standard glassware oven obtaining the expected catalyst.

## XPS Data

### Before reaction

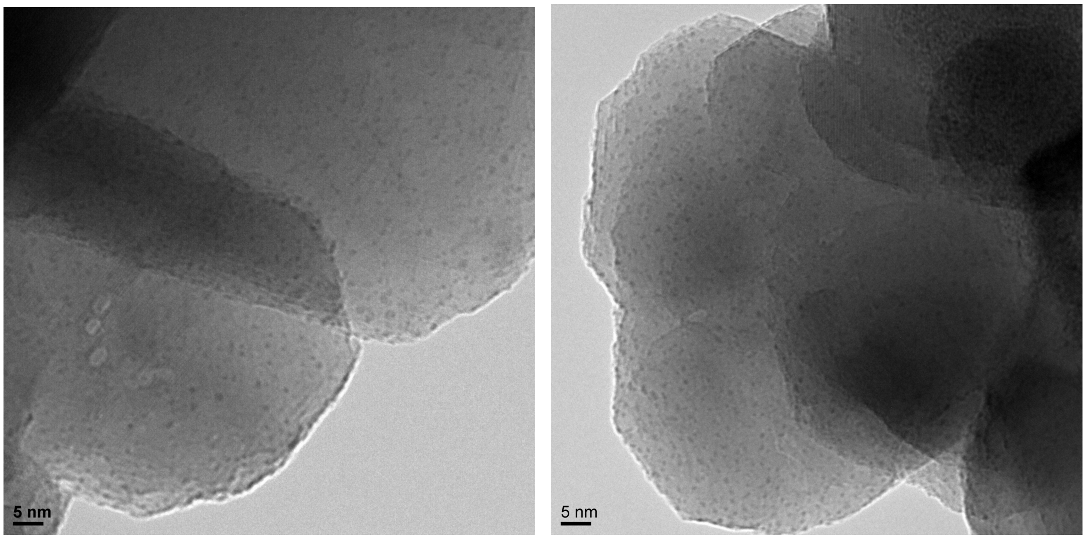


### After reaction

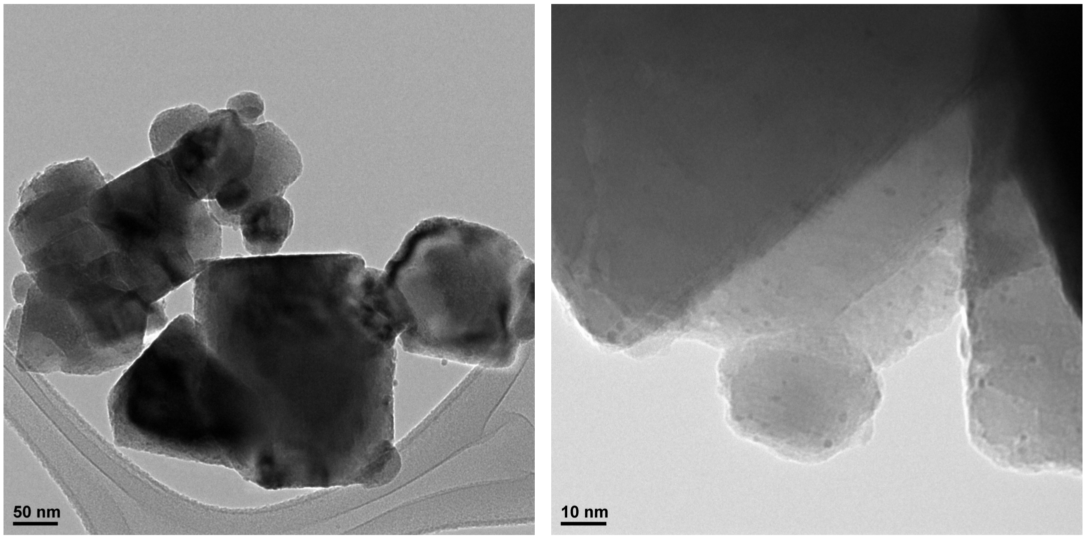


## TEM Images

### Before reaction

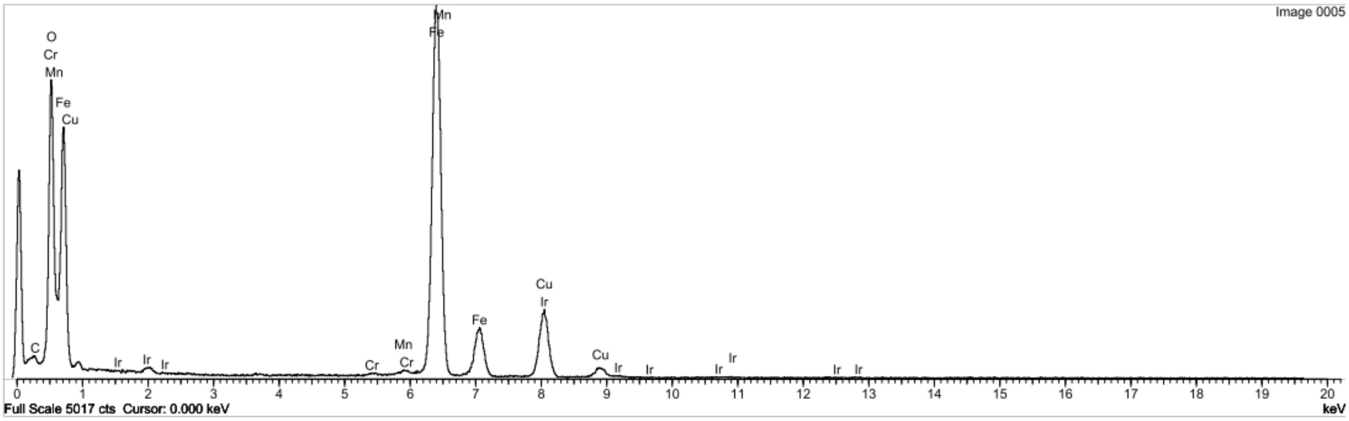


### After reaction

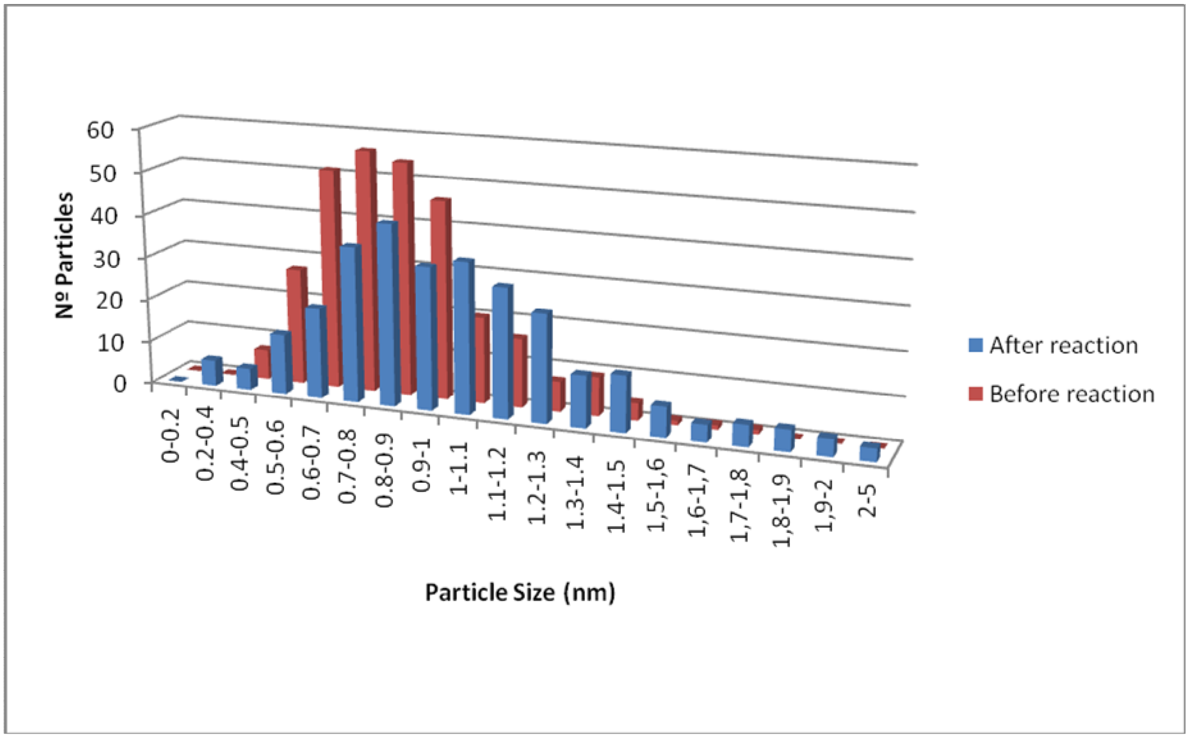


## EDS Analysis

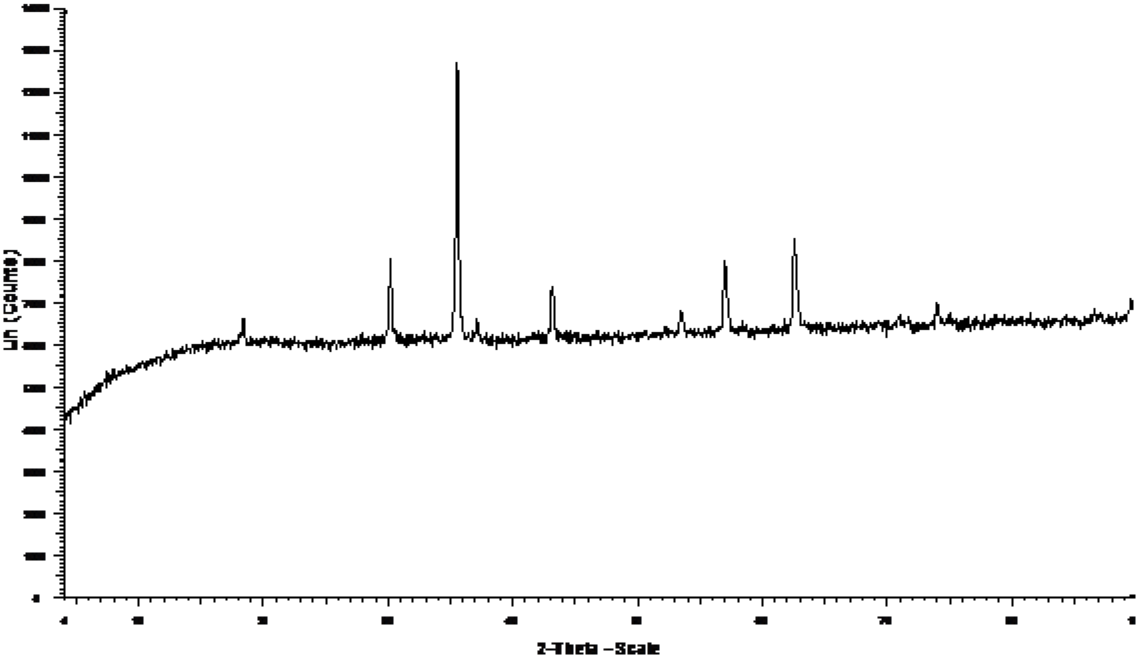
| Element | Peak Area | Area Sigma | k factor | Abs Corrn. | Weight% | Weight% Sigma | Atomic% |
|---|---|---|---|---|---|---|---|
| O K | 28972 | 531 | 1.810 | 1.000 | 33.05 | 0.43 | 63.46 |
| Cr K | 434 | 99 | 1.100 | 1.000 | 0.30 | 0.07 | 0.18 |
| Mn K | 673 | 120 | 1.153 | 1.000 | 0.49 | 0.09 | 0.27 |
| Fe K | 88664 | 506 | 1.170 | 1.000 | 65.37 | 0.45 | 35.96 |
| Ir L | 495 | 143 | 2.550 | 1.000 | 0.80 | 0.23 | 0.13 |
| Totals | | | | | 100.00 | | |



## Particle Size Distribution



## XRD Analysis

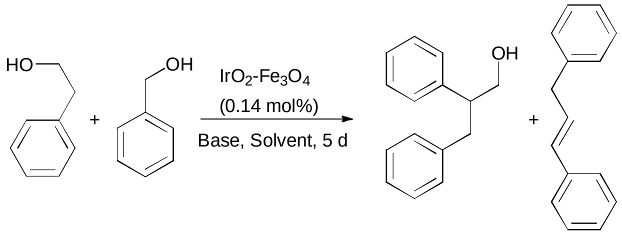


## General procedures for the preparation of the products

To a stirred solution of alcohol (**1**, 1 mmol) in toluene (1.5 mL) were added $IrO_2$-$Fe_3O_4$ (50 mg), KOH (1 mmol, 56 mg) and the corresponding alcohol (**2**, 2 mmol). The resulting mixture was stirred at 110ºC until the end of reaction. The catalyst was removed by a magnet and the resulting mixture was quenched with deionised water and extracted with $Et_2O$ (3 × 5 mL). The organic phases were dried over $MgSO_4$, followed by evaporation under reduced pressure to remove the solvent. The product was usually purified by chromatography on silica gel (hexane/ethyl acetate) to give the corresponding products **3**.

## Optimization reaction condition results

**Table** Optimization of reaction conditions[a]

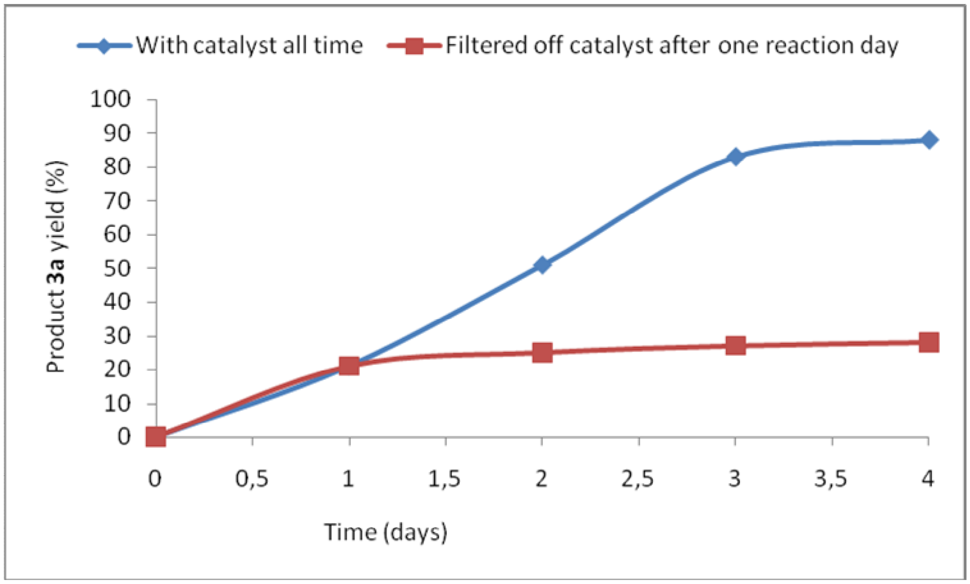


**1a** **2a** **3a** **4**

| Entry | Base (mol%) | solvent | T (ºC) | Yield[b] **3a** (%) | Yield[b] **4** (%) |
|---|---|---|---|---|---|
| 1 | *t*BuOK (100) | PhMe | 130 | 80 | 19 |
| 2 | *t*BuOK (100) | PhMe | 110 | 85 | 10 |
| 3 | *t*BuOK (100) | PhMe | 90 | 8 | 0 |
| 4 | *t*BuOK (100) | - | 110 | 74 | 18 |
| 5 | *t*BuOK (100) | $H_2O$ | 110 | 0 | 0 |
| 6 | *t*BuOK (100) | MeCN | 110 | 0 | 0 |
| 7 | *t*BuOK (100) | dioxane | 110 | 74 | 10 |
| 8 | *t*BuOK (100) | THF | 110 | 63 | 17 |
| 9 | *t*BuOK (100) | xylenes | 110 | 72 | 21 |
| 10 | - | PhMe | 110 | 0 | 0 |
| 11[c] | NaOH (100) | PhMe | 110 | 72 | 24 |
| 12[c] | KOH (100) | PhMe | 110 | 96 | 3 |
| 13 | $K_2CO_3$ (100) | PhMe | 110 | 0 | 0 |
| 14 | KOH (50) | PhMe | 110 | 10 | 12 |
| 15[c] | KOH (200) | PhMe | 110 | 71 | 26 |
| 16[c,d] | KOH (100) | PhMe | 110 | 2 | 0 |
| 17[c,e] | KOH (100) | PhMe | 110 | 69 | 3 |
| 18[f] | KOH (100) | PhMe | 110 | 55 | 11 |
| 19[c,g] | KOH (100) | PhMe | 110 | 91 | 4 |

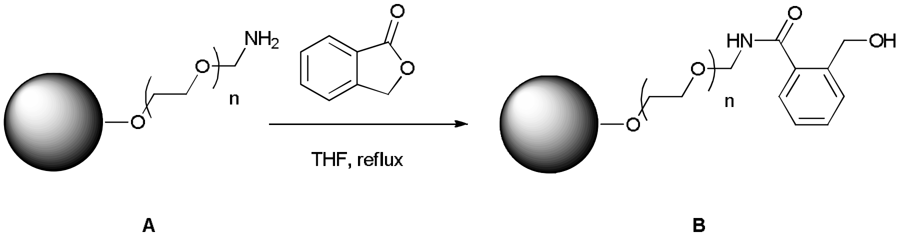
[a] Reaction carried out using **1a** (1.0 mmol) and **2a** (2.0 mmol), unless otherwise stated. [b] Isolated yield after column chromatography. [c] Reaction performed during 4 days. [d] Reaction performed using 1.0 mmol of **2a**. [e] Reaction performed using 3.0 mmol of **2a**. [f] Reaction performed using 0.03 mol% of $IrO_2$-$Fe_3O_4$. [g] Reaction performed using 0.28 mol% of $IrO_2$-$Fe_3O_4$.

## Reaction profiles for reaction in the presence of catalyst all time and for reaction filtered off the catalyst after the first day



## Three-phase test reaction

Phthalide (1 mmol) was anchored to a NovaSyn TG amino resin (**A**) by direct reaction in THF (2 mL). The mixture was refluxed 24h. Then, the resin was isolated by filtration. Only the 78% of the initial $NH_2$ groups were linked to phthalide according to elemental analysis. Elem. anal. for **A**: C, 64.77; H, 8.49; N, 0.13. Elem. anal. for **B**: C, 65.52; H, 8.54; N, 0.13;



To a stirred solution of alcohol (**1a**, 0.1 mmol) in toluene (0.1 mL) were added $IrO_2$-$Fe_3O_4$ (5 mg), KOH (0.1 mmol, 5.6 mg) and the corresponding resine **B** (0.5 g). The resulting three-phase mixture was stirred at 110ºC during 7 days. The mixture was filtered and the residue was treated with a mixture of trifluoroacetic acid/$H_2O$ (95:5; 1 mL) during 1 day. The resulting mixture was extracted with $Et_2O$ (3 × 5 mL). The organic phases were dried over $MgSO_4$, followed by evaporation under reduced pressure to remove the solvent. The $^1$H-NMR data, as well as MS-GC showed only the presence of 2-(hydroxymethyl)benzamide.

## Spectra data of products

### **2,3-Diphenylpropan-1-ol** (**3a**).[1]

Pale yellow oil; $R_f$ = 0.33 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3383, 1601, 1060, 1023 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.72 (s, br, 1H, OH), 2.9-3, 3.05-3.2 (2m, 1 and 2H, respectively, PhCH and $PhCH_2$), 3.8 (d, *J* = 6.2 Hz, 2H, $CH_2OH$), 7.1-7.4 (m, 10H, Ph × 2); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 38.6, 50.1, 66.2, 125.9, 126.7, 128 (2C), 128.1 (2C), 128.5 (2C), 129 (2C), 139.8, 141.9; EI-MS *m/z*: 212 ($M^+$, 1%), 195 (17), 194 (100), 193 (33), 181 (18), 180 (13), 179 (40), 178 (29), 166 (11), 165 (24), 163 (14), 121 (24), 116 (24), 115 (33), 104 (18), 103 (45), 92 (13), 91 (74), 89 (12), 78 (11), 77 (28), 65 (18).

### **3-(4-Chlorophenyl)-2-phenylpropan-1-ol** (**3b**).

Pale yellow oil; $R_f$ = 0.07 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3364, 1599, 1491, 1452, 1403, 1091, 1062, 1014, 834, 820, 675 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.51 (s, br, 1H, OH), 2.75-2.85, 2.95-3.05 (2m, 1 and 2H, respectively, PhCH and $PhCHCH_2C$), 3.73 (d, *J* = 5.7 Hz, 2H, $CH_2OH$), 6.95 (d, *J* = 8.3 Hz, 2H, ClCCHCH × 2), 7.1-7.15, 7.15-7.2, 7.25-7.3 (3m, 4, 1 and 2H, respectively, ArH × 7); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 37.8, 50.1, 66.2, 126.9, 128 (2C), 128.2 (2C), 128.6 (2C), 130.3 (2C), 131.7, 138.3, 141.3; EI-MS *m/z*: 246 ($M^+$, 13%), 228 (15), 179 (16), 178 (20), 126 (11), 125 (22), 121 (100), 120 (19), 105 (10), 104 (15), 103 (58), 91 (26), 89 (13), 79 (10), 77 (22). HRMS calcd. (%) for $C_{15}H_{15}ClO$: 246.0811; found: 246.0814.

### **2-Phenyl-3-(*p*-tolyl)propan-1-ol** (**3c**).

Pale yellow oil; $R_f$ = 0.2 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3357, 1513, 1494, 1452, 1414, 1061, 1021, 808, 758 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.34 (s, br, 1H, OH), 2.28 (s, 3H, $CH_3$), 2.91, 2.93 (2 dd, $J^1$= 42.7 Hz, $J^2$= 17.8 Hz, 2H, $PhCHCH_2C$), 3-3.1 (m, 1H, PhCH), 3.76 (d, *J* = 5 Hz, 2H, $CH_2OH$), 6.97 (d, *J* = 8 Hz, 2H, $CH_3CCHCH$ × 2), 7.03 (d, *J* = 8 Hz, 2H, $CH_3CCH$ × 2), 7.2-7.25, 7.25-7.3 (2m, 3 and 2H, respectively, Ph); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 21, 38.2, 50.2, 66.4, 126.8, 128.1 (2C), 128.6 (2C), 128.9 (2C), 128.9 (2C), 135.4, 136.7, 142; EI-MS *m/z*: 226 ($M^+$, 13%), 224 (14), 223 (12), 208 (13), 207 (18), 195 (11), 194 (14), 193 (11), 179 (16), 178 (20), 165 (12), 129 (12), 121 (31), 120 (15), 119 (11), 117 (17), 115 (15), 106 (27), 105 (100), 104 (27), 103 (33), 92 (10), 91 (61), 79 (13), 77 (26). HRMS calcd. (%) for $C_{16}H_{18}O$: 226.1358; found: 226.1355.

### **3-[(4-*tert*-Butyl)phenyl]-2-phenylpropan-1-ol** (**3d**).[2]

Pale yellow oil; $R_f$ = 0.2 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3376, 1601, 1513, 1363, 1267, 1061, 1020, 833, 808, 758 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): δ 1.26 (s, br, 1H, OH), 1.28 (s, 9H, $^t$Bu), 2.92, 2.94 (2 dd, $J^1$= 26.6 Hz, $J^2$= 13.6 Hz, 2H, PhCHC$H_2$C), 3.05-3.1 (m, 1H, PhCH), 3.7-3.8 (m, 2H, C$H_2$OH), 7.04 (d, *J* = 7.9Hz, 2H, $^t$BuCCHC*H* × 2), 7.2-7.25 (m, 5H, Ph), 7.3 (d, *J* = 7.9Hz, 2H, $^t$BuCCH × 2); $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 31.3 (3C), 34.3, 38.1, 50, 66.3, 125.1 (2C), 126.8, 128 (2C), 128.6 (2C), 128.6 (2C), 136.8, 142.2, 148.8; EI-MS *m/z*: 268 (M$^+$, 19%), 164 (11), 149 (10), 148 (12), 147 (100), 132 (11), 121 (11), 120 (14), 117 (11), 103 (12), 91 (13).

### **3-(4-Methoxyphenyl)-2-phenylpropan-1-ol** (**3e**).

Pale yellow oil; $R_f$ = 0.17 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3399, 1610, 1583, 1509, 1299, 1242, 1062, 1031, 818, 751, 699 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): δ 1.54 (s, br, 1H, OH), 2.75-2.85, 2.9-3.05 (2m, 1 and 2H, respectively, PhCH and PhCHC$H_2$C), 3.70-3.75 (m, 2H, C$H_2$OH), 3.73 (s, 3H, OCH$_3$), 6.73 (d, *J* = 8.7 Hz, 2H, OCCH × 2), 6.97 (d, *J* = 8.7 Hz, 2H, OCCHC*H* × 2), 7.15-7.3 (m, 5H, Ph); $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 37.7, 50.3, 55.1, 66.2, 113.5 (2C), 126.7, 128 (2C), 128.5 (2C), 129.9 (2C), 131.9, 142, 157.7; EI-MS *m/z*: 242 (M$^+$, 11%), 121 (100). HRMS calcd. (%) for $C_{16}H_{18}O_2$: 242.1307; found: 242.1308.

### **3-(3,5-Dimethoxyphenyl)-2-phenylpropan-1-ol** (**3f**).

Pale yellow oil; $R_f$ = 0.03 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3386, 1594, 1458, 1428, 1203, 1147, 1055, 831, 759, 699 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): δ 1.65 (s, br, 1H, OH), 2.92, 2.95 (2 dd, $J^1$= 47.8 Hz, $J^2$= 17.8 Hz, 2H, PhCHC$H_2$C), 3.05-3.15 (m, 1H, PhCH), 3.73 (s, 6H, OCH$_3$ × 2), 3.75-3.85 (m, 2H, C$H_2$OH), 6.27 (d, *J* = 2.1 Hz, 2H, OCC*H*CCH$_2$ × 2), 6.31 (t, *J* = 2 Hz, 1H, OCCHCO), 7.2-7.3, 7.3-7.4 (2m, 3 and 2H, respectively, Ph); $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 38.9, 49.9, 55.1 (2C), 66.3, 98, 107 (2C), 126.7, 128 (2C), 128.5 (2C), 141.9, 142.2, 160.5 (2C); EI-MS *m/z*: 272 (M$^+$, 12%), 153 (11), 152 (100), 151 (16), 103 (10). HRMS calcd. (%) for $C_{17}H_{20}O_3$: 272.1412; found: 272.1387.

### **2-(4-Bromophenyl)-3-phenylpropan-1-ol** (**3g**).

Pale yellow oil; $R_f$ = 0.17 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3347, 1602, 1590, 1487, 1453, 1406, 1071, 1030, 1009, 818, 741, 716, 698 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): δ 1.42 (s, br, 1H, OH), 2.8-2.9, 2.95-3.1 (2m, 1 and 2H, respectively, PhCH$_2$C*H* and PhCH$_2$), 3.7-3.8 (m, 2H, C$H_2$OH), 7-7.1, 7.15-7.25 (2m, 4 and 3H, respectively, Ph and BrCCHC*H* × 2), 7.41 (d, *J* = 8.3 Hz, 2H, BrCCH × 2); $^{13}$C-

NMR (75 MHz, $CDCl_3$): δ 38.5, 49.6, 66.1, 120.5, 126.1, 128.3 (2C), 129 (2C), 129.8 (2C), 131.6 (2C), 139.4, 140.9; EI-MS *m/z*: 292 ($M^+$+2, 18%), 290 ($M^+$, 18), 201 (54), 200 (25), 199 (56), 198 (21), 180 (40), 179 (30), 178 (27), 165 (17), 120 (82), 102 (14), 92 (25), 91 (100), 89 (18), 77 (16). HRMS calcd. (%) for $C_{15}H_{15}BrO$: 290.0306; found: 290.0270.

**2-(4-Chlorophenyl)-3-phenylpropan-1-ol** (**3h**).[3]

Pale yellow oil; $R_f$ = 0.2 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3333, 1600, 1491, 1454, 1090, 1029, 1013, 822, 743, 698 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.68 (s, br, 1H, OH), 2.75-2.85, 2.95-3.05 (2m, 1 and 2H, respectively, $PhCH_2CH$ and $PhCH_2$), 3.71 (m, 2H, $CH_2OH$), 7-7.05, 7.05-7.1, 7.15-7.2, 7.2-7.3 (4m, 2,2,1 and 4H, respectively, ArH × 9); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 38.5, 49.5, 66, 126, 128.2 (2C), 128.6 (2C), 128.9 (2C), 129.3 (2C), 132.3, 139.4, 140.4; EI-MS *m/z*: 248 ($M^+$+2, 8%), 246 ($M^+$, 23), 228 (13), 215 (21), 180 (16), 179 (24), 178 (26), 165 (11), 157 (32), 156 (20), 155 (100), 154 (35), 139 (16), 138 (17), 137 (34), 125 (14), 103 (14), 102 (13), 101 (11), 92 (21), 91 (91), 89 (17), 77 (16).

**2,3-bis(4-Chlorophenyl)propan-1-ol** (**3i**).

Pale yellow oil; $R_f$ = 0.1 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3367, 1491, 1407, 1091, 1013, 906, 823, 731 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.39 (s, br, 1H, OH), 2.7-2.85, 2.95-3.05 (2m, 1 and 2H, respectively, $C_6H_4CH_2CH$ and $C_6H_4CH_2CH$), 3.76 (d, *J* = 6.2 Hz, 2H, $CH_2OH$), 6.96 (dt, $J^2$= 8.4 Hz, $J^3$= 2.2 Hz, 2H, $CH_2CCH$ × 2), 7.08 (dt, $J^2$= 8.4 Hz, $J^3$= 2.3 Hz, 2H, CHCC*H* × 2), 7.17 (dt, $J^2$= 8.4 Hz, $J^3$= 2.3 Hz, 2H, CHCCHC*H* × 2), 7.26 (dt, $J^2$= 8.4 Hz, $J^3$= 2.3 Hz, 2H, $CH_2CCHCH$ × 2); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 37.8, 49.5, 66.1, 128.4 (2C), 128.7 (2C), 129.4 (2C), 130.3 (2C), 131.9, 132.6, 137.9, 139.9; EI-MS *m/z*: 280 ($M^+$, 14%), 179 (10), 178 (20), 157 (32), 156 (13), 155 (100), 154 (15), 139 (13), 137 (28), 127 (14), 125 (37), 102 (11), 101 (11), 91 (30), 89 (18). HRMS calcd. (%) for $C_{15}H_{14}Cl_2O$: 280.0422; found: 280.0404.

**2-(4-Chlorophenyl)-3-(4-methoxyphenyl)propan-1-ol** (**3j**).

Pale yellow oil; $R_f$ = 0.1 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3383, 1611, 1583, 1510, 1492, 1463, 1300, 1243, 1177, 1091, 1034, 823, 731 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.48 (s, br, 1H, OH), 2.7-2.8, 2.9-3.05 (2m, 1 and 2H, respectively, $C_6H_4CH_2CH$ and $C_6H_4CH_2$), 3.7-3.8 (m, 5H, $CH_2OH$ and $OCH_3$), 6.74 (d, *J* = 8.6 Hz, 2H, OCCH × 2), 6.94 (d, *J* = 8.5 Hz, 2H, OCCHC*H* × 2), 7.09 (d, *J* = 8.4 Hz, 2H, ClCCHC*H* × 2), 7.25 (d, *J* = 8.4 Hz, 2H, ClCCH × 2); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 37.7, 49.7, 55.1, 66.1, 113.6 (2C), 128.6 (2C), 129.4 (2C), 129.9 (2C), 131.4, 132.3, 140.5, 157.8; EI-MS *m/z*: 276 ($M^+$, 6%), 121 (100). HRMS calcd. (%) for $C_{16}H_{17}ClO_2$: 276.0917; found: 276.0904.

**2-(2-Chlorophenyl)-3-phenylpropan-1-ol** (**3k**).

Pale yellow oil; $R_f$ = 0.2 (hexane/ethyl acetate:4/1); IR (ATR): ν 3335, 1494, 1474, 1454, 1440, 1055, 1037, 753, 698 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.81 (s, br, 1H, OH), 2.85-3.05, 3.65-3.8 (2m, 2 and 3H, respectively, C*H*$_2$OH, PhCH and PhCHC*H*$_2$), 7.05-7.35 (m, 9H, ArH); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 37.4, 44.9, 64.3, 126, 126.8, 127.6, 128.2 (2C), 128.2, 128.9 (2C), 129.6, 134.5, 139.3, 139.4; EI-MS *m/z*: 246 ($M^+$, 6%), 230 (17), 228 (53), 215 (16), 193 (16), 180 (13), 179 (25), 178 (31), 165 (10), 157 (11), 155 (34), 154 (18), 140 (10), 139 (13), 138 (31), 137 (24), 103 (11), 92 (31), 91 (100), 89 (12), 77 (12). HRMS calcd. (%) for $C_{15}H_{15}ClO$: 246.0811; found: 246.0807.

**2-(3-Chlorophenyl)-3-phenylpropan-1-ol** (**3l**).

Pale yellow oil; $R_f$ = 0.07 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3342, 1595, 1570, 1493, 1454, 1430, 1028, 782, 744, 695 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.62 (s, br, 1H, OH), 2.75-2.9, 2.95-3.05 (2m, 1 and 2H, respectively, PhCH and PhCHC*H*$_2$), 3.7-3.75 (m, 2H, C*H*$_2$OH), 7-7.05, 7.15-7.25 (2m, 3 and 6H, respectively, ArH); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 38.4, 49.8, 65.9, 126.1, 126.3, 126.9, 128.1, 128.3 (2C), 128.9 (2C), 129.7, 134.3, 139.3, 144.2; EI-MS *m/z*: 246 ($M^+$, 11%), 228 (23), 215 (10), 179 (16), 178 (19), 155 (22), 154 (17), 139 (10), 138 (28), 137 (16), 103 (10), 92 (27), 91 (100), 77 (11). HRMS calcd. (%) for $C_{15}H_{15}ClO$: 246.0811; found: 246.0816.

**2-(4-Methoxyphenyl)-3-phenylpropan-1-ol** (**3m**).

Pale yellow oil; $R_f$ = 0.13 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3407, 1609, 1583, 1509, 1245, 1177, 1109, 1029, 827, 806, 698 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.31 (s, br, 1H, OH), 2.8-2.9, 2.95-3.05 (2m, 1 and 2H, respectively, PhCH$_2$C*H* and PhCH$_2$), 3.7-3.8 (m, 5H, C*H*$_2$OH and OCH$_3$), 6.83 (d, *J* = 8.8 Hz, 2H, OCCH × 2), 7.05-7.15, 7.2-7.25 (2m, 5 and 2H, Ph and OCCHC × 2); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 38.9, 49.3, 55.2, 66.5, 114 (2C), 125.9, 128.2 (2C), 129 (2C), 129 (2C), 133.7, 140, 158.4; EI-MS *m/z*: 242 ($M^+$, 11%), 152 (13), 151 (100), 137 (14), 121 (11), 91 (32), 77 (13). HRMS calcd. (%) for $C_{16}H_{18}O_2$: 242.1307; found: 242.1308.

**3-(4-Chlorophenyl)-2-(4-Methoxyphenyl)propan-1-ol** (**3n**).

Pale yellow oil; $R_f$ = 0.07 (hexane/ethyl acetate: 4/1); IR (ATR): $\nu$ 3374, 1610, 1510, 1492, 1245, 1177, 1033, 1015, 907, 828, 729 cm$^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): $\delta$ 1.49 (s, br, 1H, OH), 2.7-2.85, 2.9-3 (2m, 1 and 2H, respectively, $C_6H_4CH_2C\mathit{H}$ and $C_6H_4C\mathit{H}_2CH$), 3.65-3.8 (m with s at 3.77, 5H, $C\mathit{H}_2OH$ and $OCH_3$), 6.83 (dt, $J^2$= 8.7 Hz, $J^3$= 2.3 Hz, 2H, OCCH × 2), 6.96 (dt, $J^2$= 8.4 Hz, $J^3$= 2.1 Hz, 2H, ClCCHC*H* × 2), 7.06 (dt, $J^2$= 8.7 Hz, $J^3$= 2.2 Hz, 2H, OCCHC*H* × 2), 7.15 (dt, $J^2$= 8.4 Hz, $J^3$= 2.1 Hz, 2H, ClCCH × 2); $^{13}$C-NMR (75 MHz, $CDCl_3$): $\delta$ 38, 49.3, 55.2, 66.4, 114 (2C), 128.2 (2C), 128.9 (2C), 130.3 (2C), 131.6, 133.1, 138.4, 158.4; EI-MS *m/z*: 276 ($M^+$, 6%), 152 (11), 151 (100), 121 (13), 91 (17). HRMS calcd. (%) for $C_{16}H_{17}ClO_2$: 276.0917; found: 276.0924.

**2,3-bis(4-Methoxyphenyl)propan-1-ol** (**3o**).

Pale yellow oil; $R_f$ = 0.03 (hexane/ethyl acetate: 4/1); IR (ATR): $\nu$ 3405, 1610, 1583, 1510, 1464, 1441, 1300, 1242, 1176, 1033, 908, 828, 729 cm$^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): $\delta$ 1.45 (s, br, 1H, OH), 2.7-2.85, 2.85-3.05 (2m, 1 and 2H, respectively, $C_6H_4CH_2C\mathit{H}$ and $C_6H_4C\mathit{H}_2CH$), 3.7-3.8 (m with 2s at 3.74 and 3.77, 2, 3 and 3H, respectively, $C\mathit{H}_2OH$ and $OCH_3$ × 2), 6.75 (dt, $J^2$= 8.7 Hz, $J^3$= 2.5 Hz, 2H, CHCCHC*H* × 2), 6.83 (dt, $J^2$= 8.7 Hz, $J^3$= 2.5 Hz, 2H, $CH_2CCHC\mathit{H}$ × 2), 6.97 (dt, $J^2$= 8.7 Hz, $J^3$= 2.5 Hz, 2H, $CH_2CCH$ × 2), 7.09 (dt, $J^2$= 8.7 Hz, $J^3$= 2.5 Hz, 2H, CHCCH × 2); $^{13}$C-NMR (75 MHz, $CDCl_3$): $\delta$ 37.9, 49.4, 55.1, 55.1, 66.4, 113.5 (2C), 113.9 (2C), 129 (2C), 129.9 (2C), 132, 133.8, 157.7, 158.2; EI-MS *m/z*: 272 ($M^+$, 20%), 152 (12), 151 (100), 121 (75), 91 (19). HRMS calcd. (%) for $C_{17}H_{20}O_3$: 272.1412; found: 272.1400.

**2-Benzylbutan-1-ol** (**3p**).[4]

Pale yellow oil; $R_f$ = 0.27 (hexane/ethyl acetate: 4/1); IR (ATR): $\nu$ 3347, 1494, 1454, 1032, 734, 698 cm$^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): $\delta$ 0.93 (t, *J* = 7.5 Hz, 3H, $CH_3$), 1.25-1.45 (m, 2H, $C\mathit{H}_2CH_3$), 1.54 (s, br, 1H, OH), 1.65-1.75 (m, 1H, CH), 2.6, 2.65 (2 dd, $J^1$= 18.2 Hz, $J^2$= 9.5 Hz, 2H, $PhCH_2$), 3.51 (d, *J* = 5.3 Hz, 2H, $C\mathit{H}_2OH$), 7.15-7.3 (m, 5H, Ph); $^{13}$C-NMR (75 MHz, $CDCl_3$): $\delta$ 11.2, 23.2, 37.2, 44.1, 64.4, 125.8, 128.2 (2C), 129.1 (2C), 140.8; EI-MS *m/z*: 164 ($M^+$, 17%), 146 (24), 131 (21), 117 (53), 103 (22), 92 (60), 91 (100).

**2-(4-Chlorobenzyl)butan-1-ol** (**3q**).

Pale yellow oil; $R_f$ = 0.27 (hexane/ethyl acetate: 4/1); IR (ATR): $\nu$ 3330, 1491, 1406, 1089, 1037, 1012, 837, 798 cm$^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): $\delta$ 0.92 (t, *J* = 7.54 Hz, 3H, $CH_2C\mathit{H}_3$), 1.3-1.45 (m, 3H, $C\mathit{H}_2CH_3$ and OH), 1.6-1.75 (m, 1H, $C\mathit{H}CH_2CH_3$), 2.5-2.7(m, 2H, $CC\mathit{H}_2CH$), 3.51 (d, *J* = 5.3 Hz, 2H, C*H*$_2$OH), 7.1 (d, *J* = 8.3 Hz, 2H, ClCCHC*H* × 2), 7.24 (d, *J* = 8.3 Hz, 2H, ClCCH × 2); $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 11.3, 23.1, 36.5, 44, 64.1, 128.2 (2C), 130.5 (2C), 133.3, 139.2; EI-MS *m/z*: 198 (M$^+$, 23%), 180 (17), 165 (12), 153 (18), 151 (56), 145 (20), 138 (25), 128 (10), 127 (35), 126 (25), 125 (100), 115 (13), 91 (36), 77 (10). HRMS calcd. (%) for $C_{11}H_{15}ClO$: 198.0811; found: 198.0800.

**2-(4-Methoxybenzyl)butan-1-ol** (**3r**).

Pale yellow oil; R$_f$ = 0.1 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3367, 1610, 1510, 1463, 1442, 1299, 1244, 1176, 1034, 834, 822 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): δ 0.93 (t, *J* = 7.5 Hz, 3H, CH$_2$C*H*$_3$), 1.3-1.45 (m, 3H, C*H*$_2$CH$_3$ and OH), 1.65-1.75 (m, 1H, C*H*CH$_2$CH$_3$), 2.57 (d, *J* = 7.1 Hz, 2H, CC*H*$_2$CH), 3.53 (d, *J* = 5.3 Hz, 2H, C*H*$_2$OH), 3.79 (s, 3H, OCH$_3$), 6.82 (dt, *J* $^2$= 8.6 Hz, *J* $^3$= 2.6 Hz, 2H, OCCH × 2), 7.1 (dt, *J* $^2$= 8.6 Hz, *J* $^3$= 2.6 Hz, 2H, OCCHC*H* × 2); $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 11.3, 23.2, 36.3, 44.2, 55.2, 64.5, 113.6 (2C), 130 (2C), 132.7, 157.7; EI-MS *m/z*: 194 (M$^+$, 13%), 122 (11), 121 (100). HRMS calcd. (%) for $C_{12}H_{18}O_2$: 194.1307; found: 194.1306.

**2-Benzyldodecan-1-ol** (**3s**).

Pale yellow oil; R$_f$ = 0.37 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3331, 1602, 1495, 1465, 1454, 1030, 739, 698 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): δ 0.92 (t, *J* = 6.6 Hz, 3H, CH$_3$), 1.25-1.45 (m, 18H, CH$_2$ × 9), 1.52 (s, br, 1H, OH), 1.75-1.9 (m, 1H, PhCH$_2$C*H*), 2.66 (d, *J* = 7.2 Hz, 2H, PhCH$_2$), 3.54 (d, *J* = 5.3 Hz, 2H, C*H*$_2$OH), 7.15-7.35 (m, 5H, Ph); $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 14.1, 22.7, 26.9, 29.3, 29.6 (3C), 29.9, 30.7, 31.9, 37.6, 42.5, 64.8, 125.8, 128.2 (2C), 129.1 (2C), 140.8; EI-MS *m/z*: 276 (M$^+$, 7%), 258 (25), 131 (17), 118 (14), 117 (40), 105 (15), 104 (100), 92 (74), 91 (79), 69 (11). HRMS calcd. (%) for $C_{19}H_{32}O$: 276.2453; found: 276.2459.

**(*E*)-Prop-1-ene-1,3-diyldibenzene** (**4**).

Pale yellow oil; R$_f$ = 0.8 (hexane/ethyl acetate: 4/1); IR (ATR): ν 1600, 1495, 1452, 1088, 1019 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): δ 3.54 (d, *J* = 3.4 Hz, 2H, CH$_2$), 6.3-6.5 (m, 2H, CH$_2$C*H*C*H*), 7.15-7.4 (m, 10H, Ph × 2); $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 39.3, 126.1 (2C), 126.2, 127.1, 128.5 (4C), 128.6 (2C), 129.2, 131, 137.4, 140.1; EI-MS *m/z*: 195 (M$^+$+1, 16%), 194 (M$^+$, 100), 193 (59), 179 (49), 178 (40), 165 (19), 117 (18), 116 (45), 115 (77), 103 (15), 91 (30), 89 (12), 77 (13).

**1,1,3,3-Tetradeuthero-2,3-diphenylpropan-1-ol** (**3a’**).

$R_f$ = 0.33 (hexane/ethyl acetate: 4/1); IR (ATR): ν 3357, 1494, 1447, 1078, 973, 696 $cm^{-1}$; $^1$H-NMR (300 MHz, $CDCl_3$): δ 1.31 (s, br, 1H, OH), 3.06 (s, 1H, CH), 7-7.5 (m, 10H, Ph × 2); $^{13}$C-NMR (75 MHz, $CDCl_3$): δ 37.9 (p, *J* = 17.7 Hz), 49.8, 65.6 (p, *J* = 20.1 Hz), 126, 126.8, 128.1 (2C), 128.2 (2C), 128.6 (2C), 129 (2C), 139.8, 141.8; EI-MS *m/z*: 216 ($M^+$, 13%), 213 (26), 198 (22), 197 (58), 196 (14), 184 (17), 183 (44), 182 (10), 181 (14), 180 (18), 179 (18), 168 (15), 167 (22), 166 (15), 124 (25), 123 (98), 122 (15), 121 (24), 107 (11), 106 (42), 105 (100), 104 (33), 94 (26), 93 (59), 92 (35), 80 (12), 79 (16), 78 (24), 77 (26).

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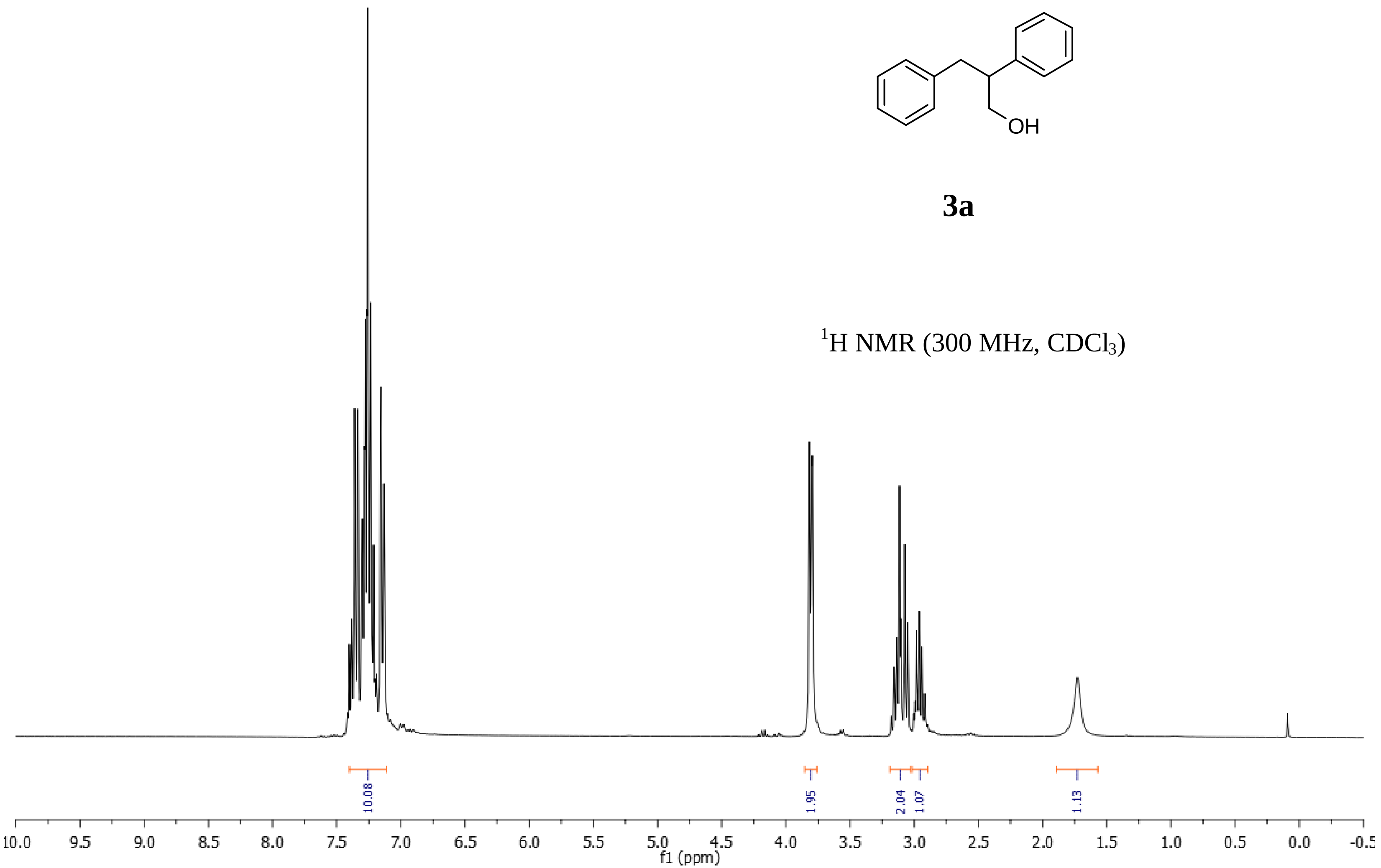
(4) Q. Zhu, Y. Lu, *Org. Lett.* 2008, **10**, 4803-4806.

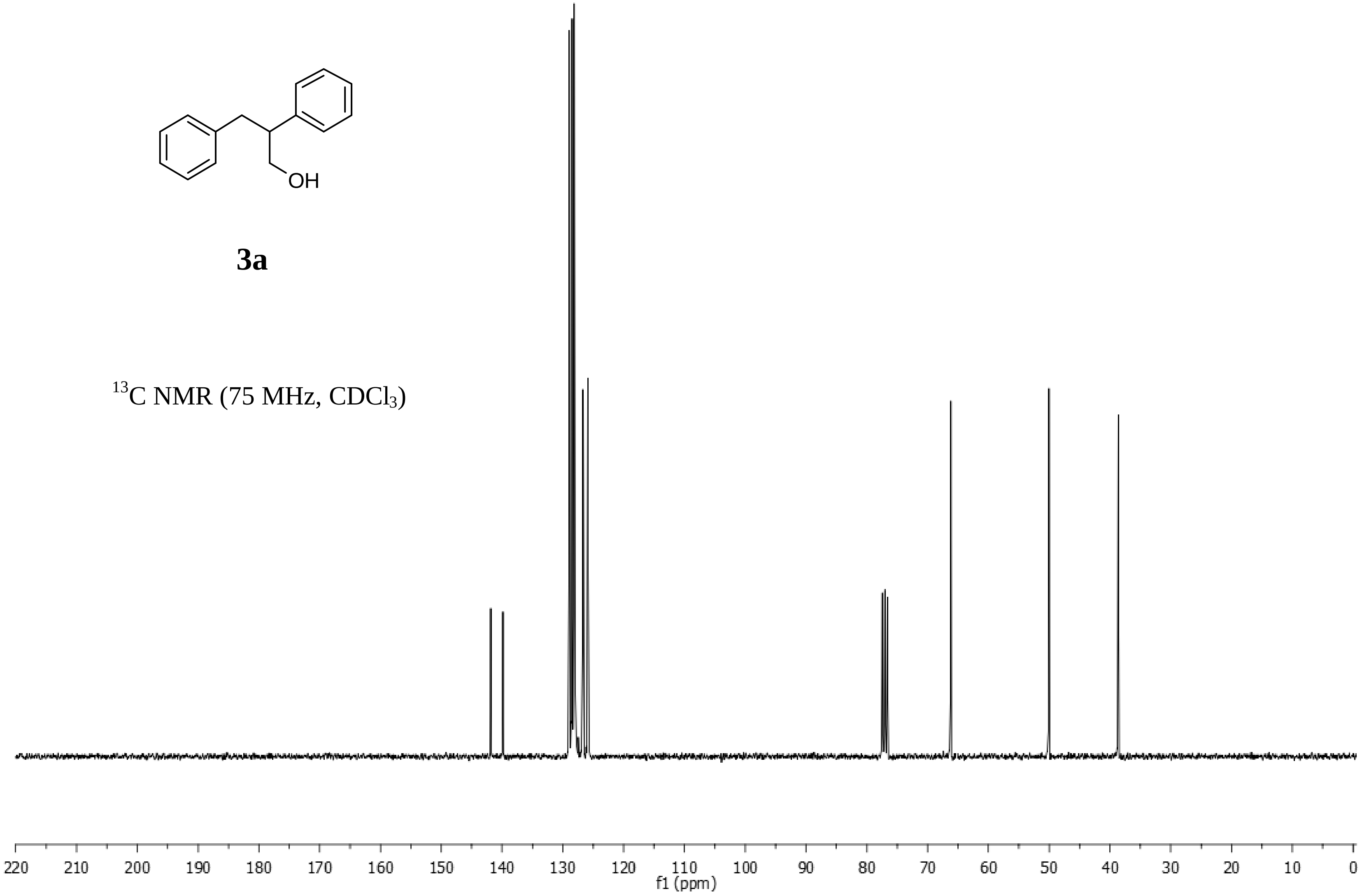


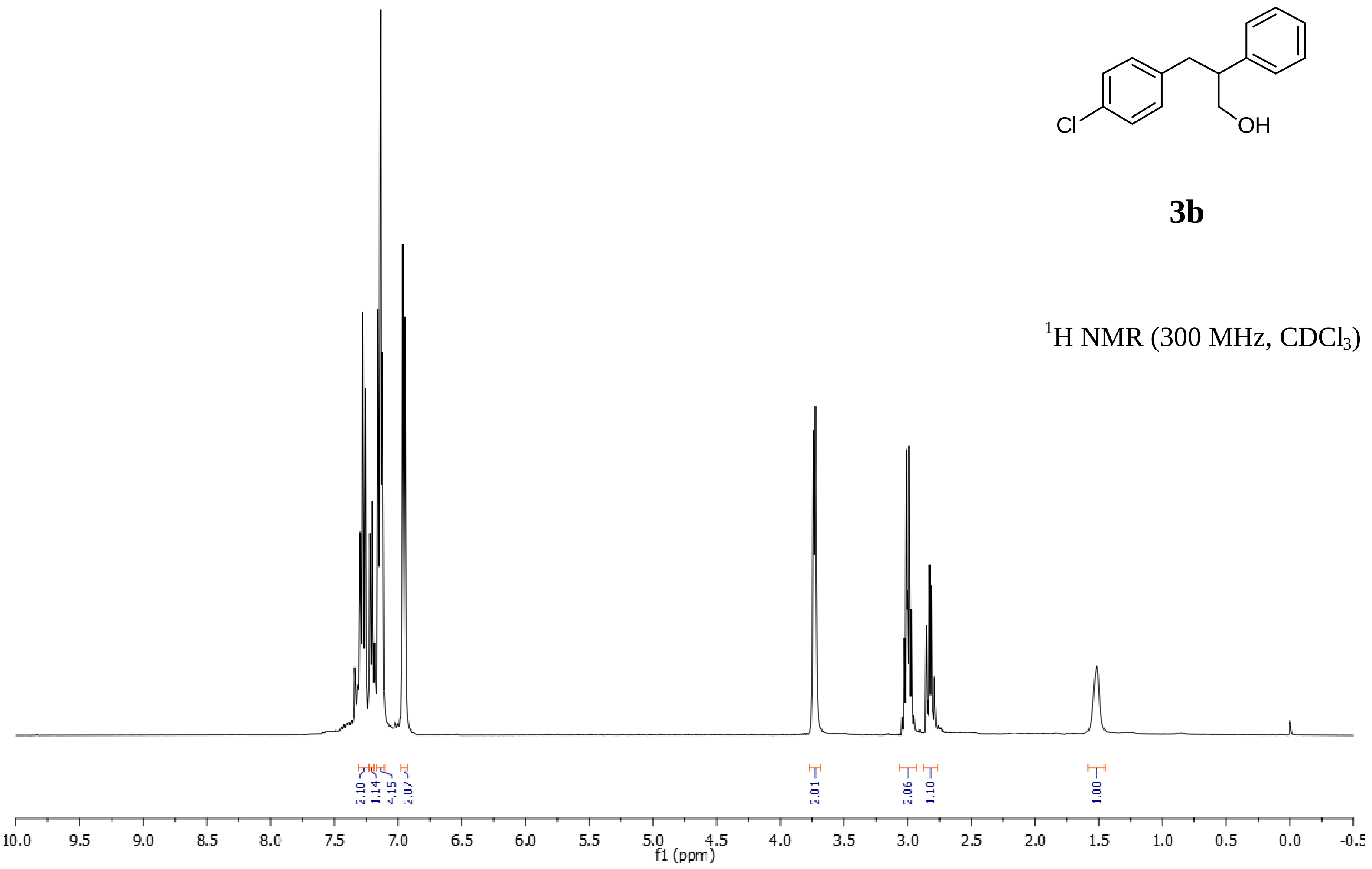
**3a**

$^1$H NMR (300 MHz, $CDCl_3$)

**3a**

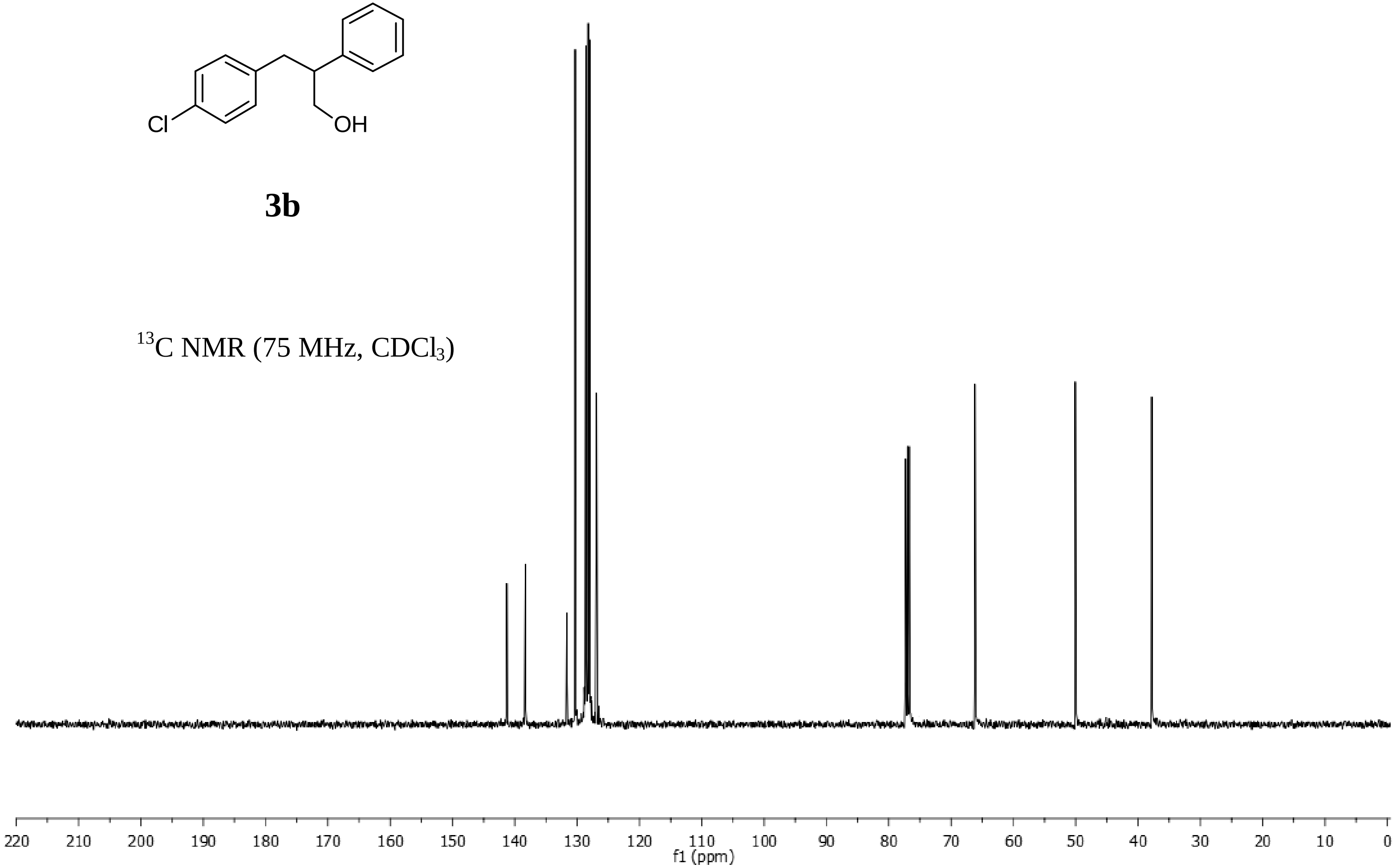
$^{13}$C NMR (75 MHz, CDCl$_3$)





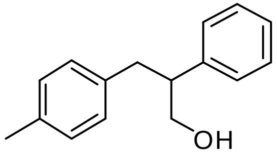
**3b**

$^1$H NMR (300 MHz, $CDCl_3$)



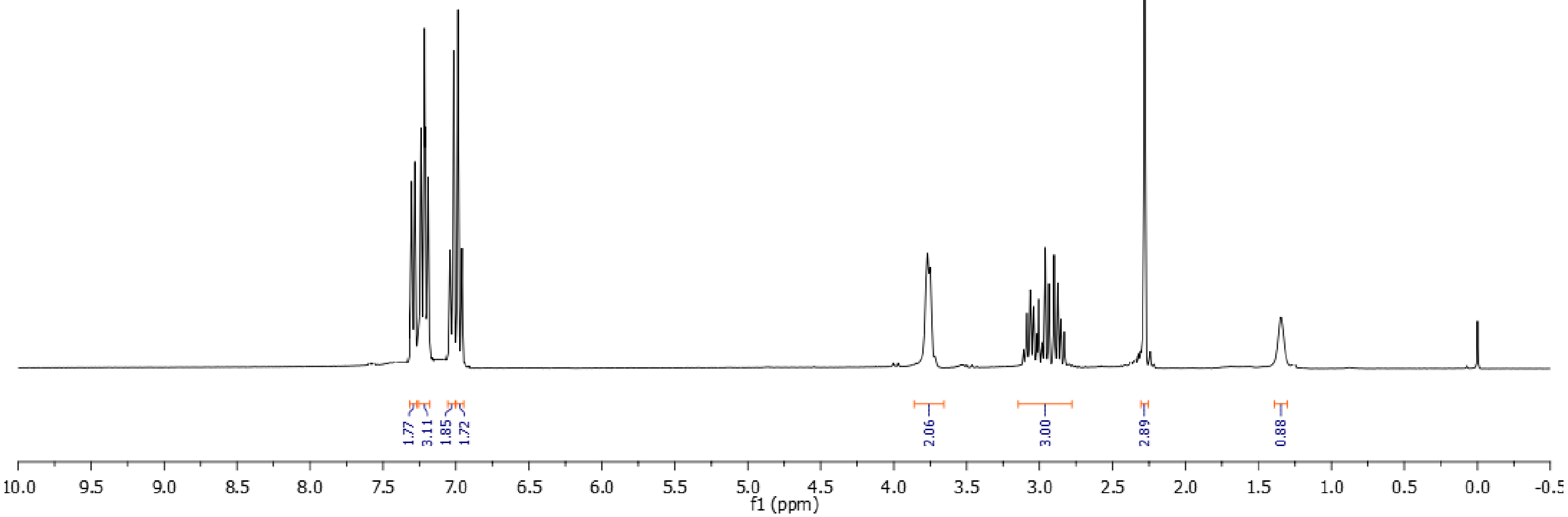
**3b**

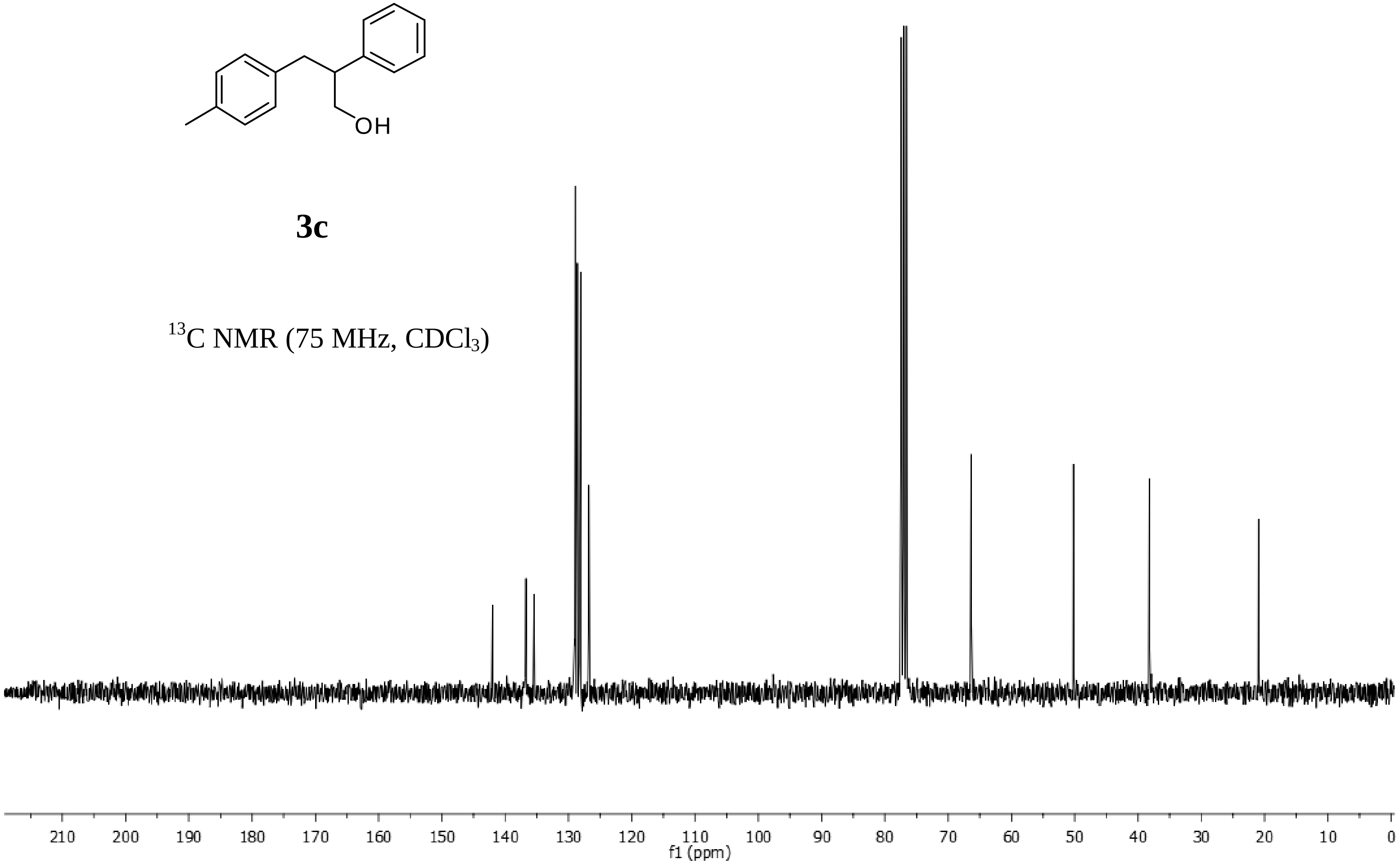
$^{13}$C NMR (75 MHz, $CDCl_3$)



**3c**

$^{1}$H NMR (300 MHz, $CDCl_3$)



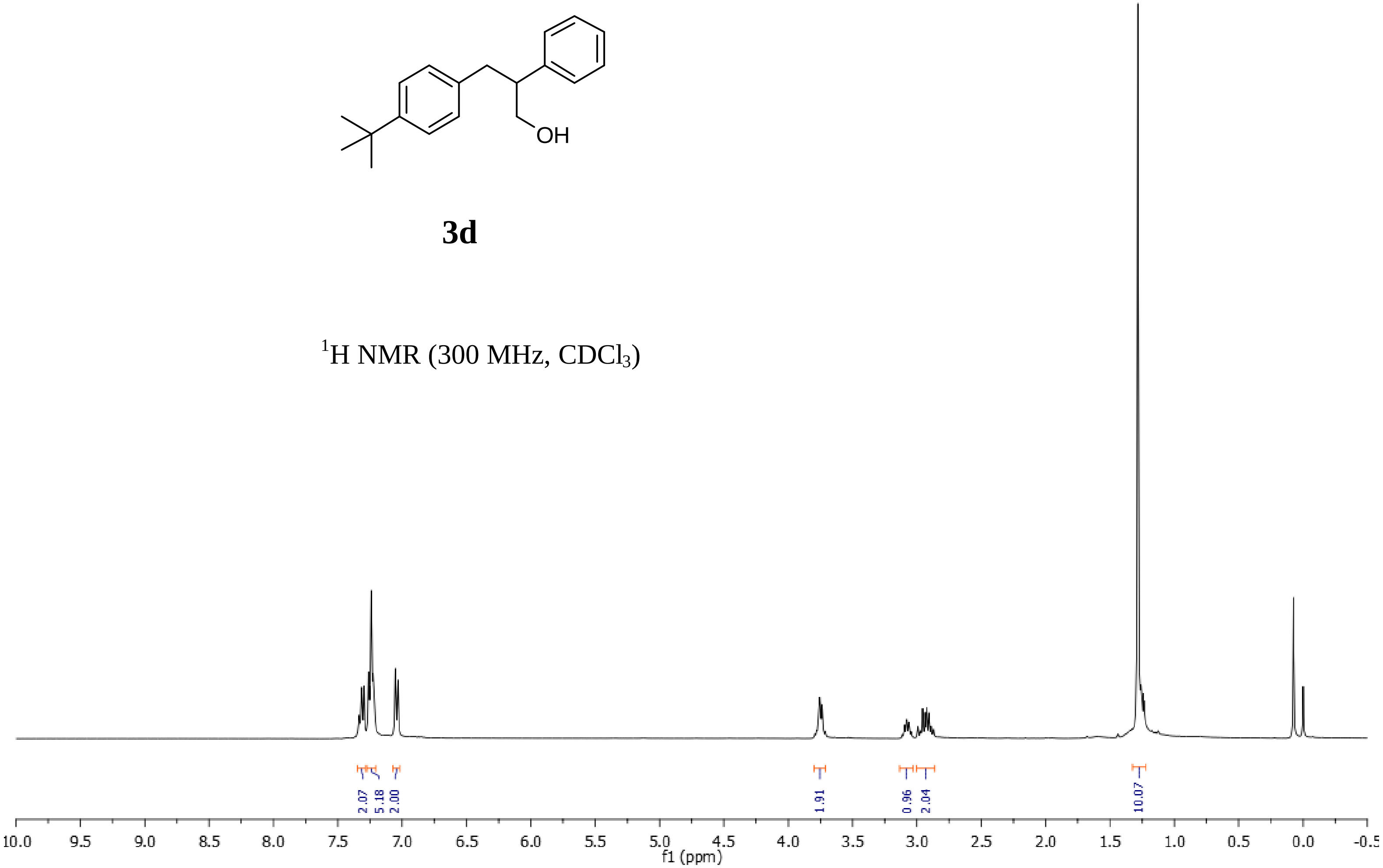


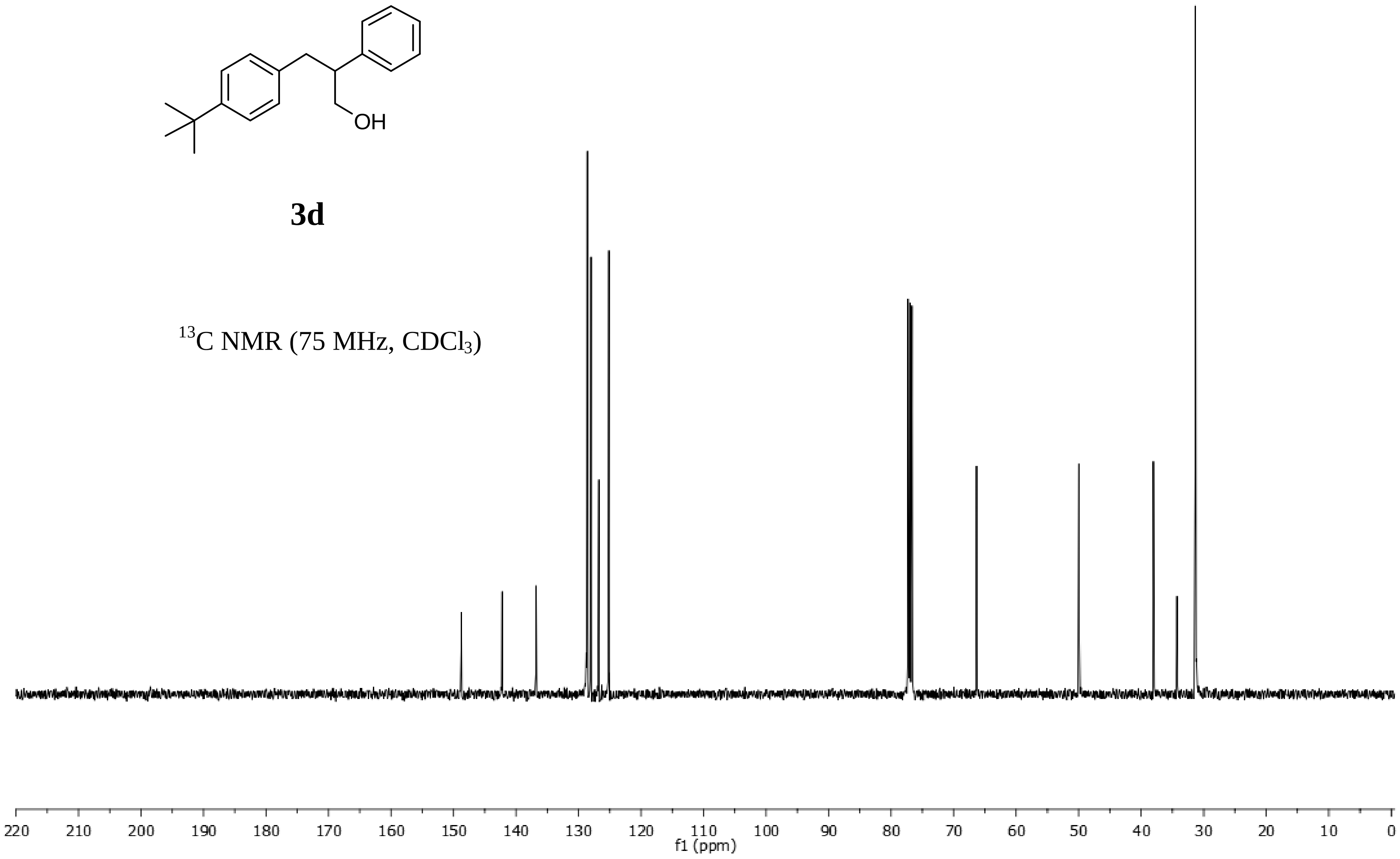
**3c**

$^{13}$C NMR (75 MHz, CDCl$_3$)

**3d**

$^1$H NMR (300 MHz, $CDCl_3$)



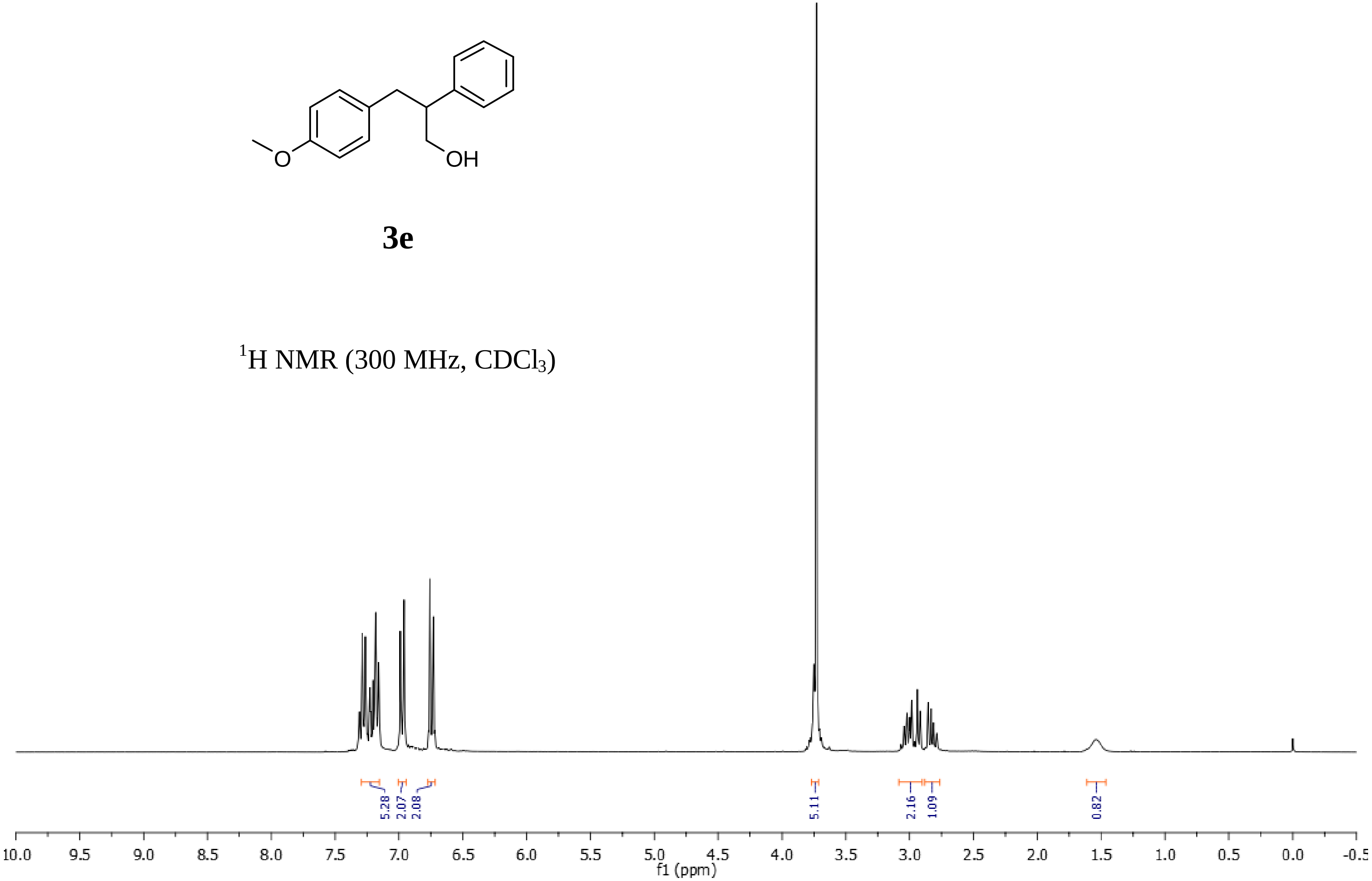


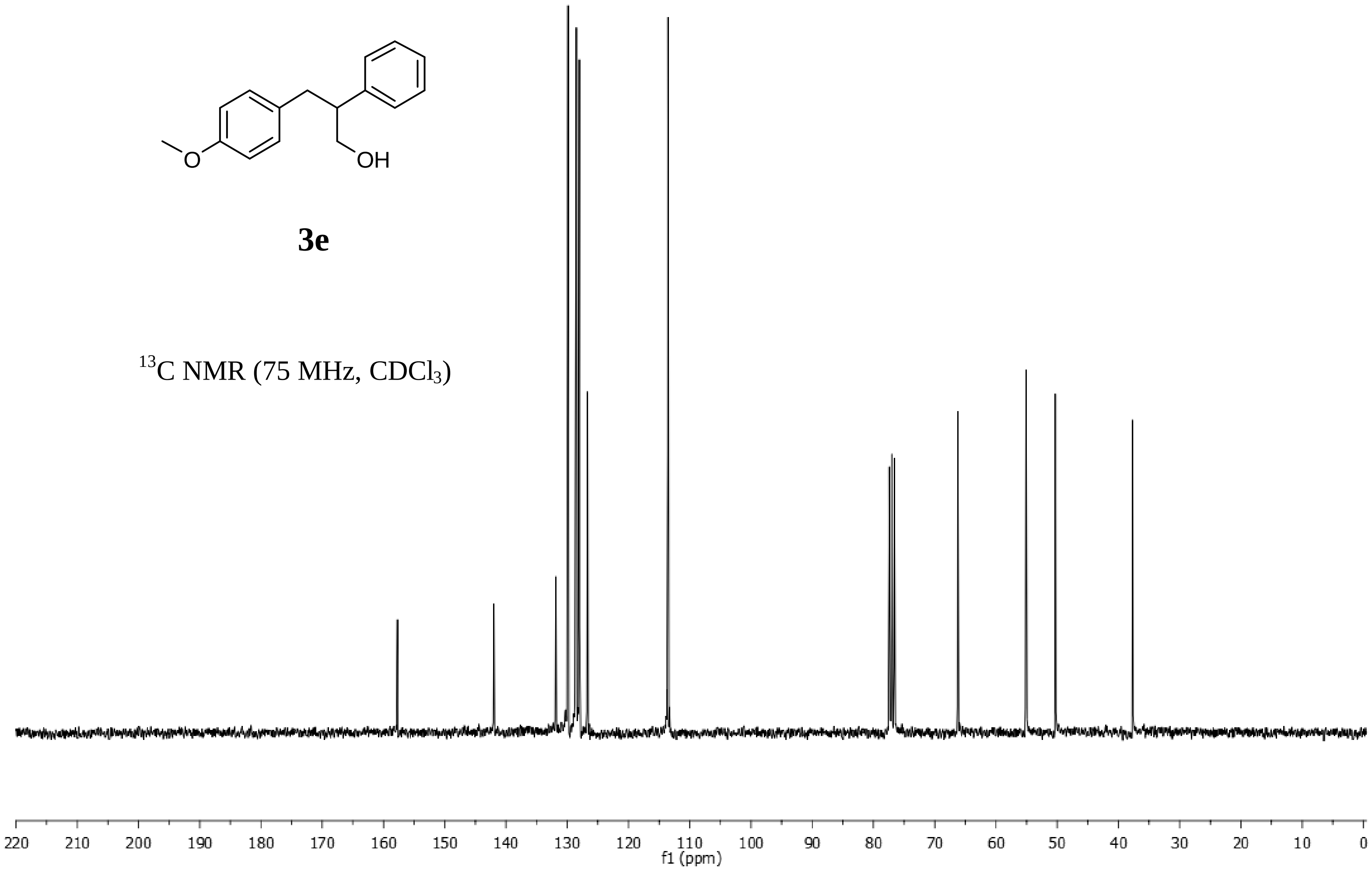
**3d**

$^{13}$C NMR (75 MHz, $CDCl_3$)

**3e**

$^{1}$H NMR (300 MHz, CDCl$_3$)



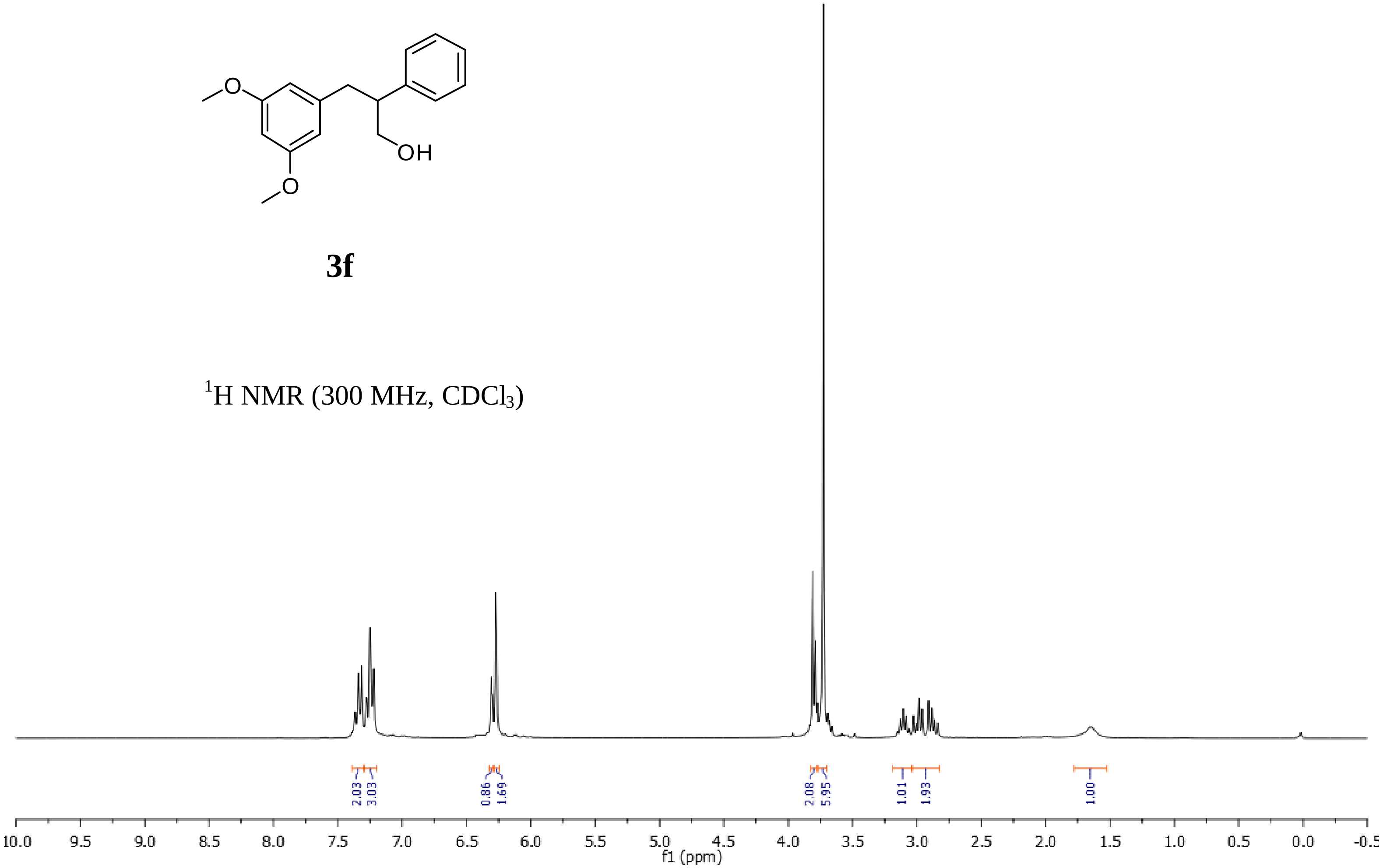


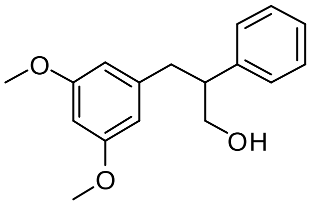
**3e**

$^{13}$C NMR (75 MHz, $CDCl_3$)

**3f**

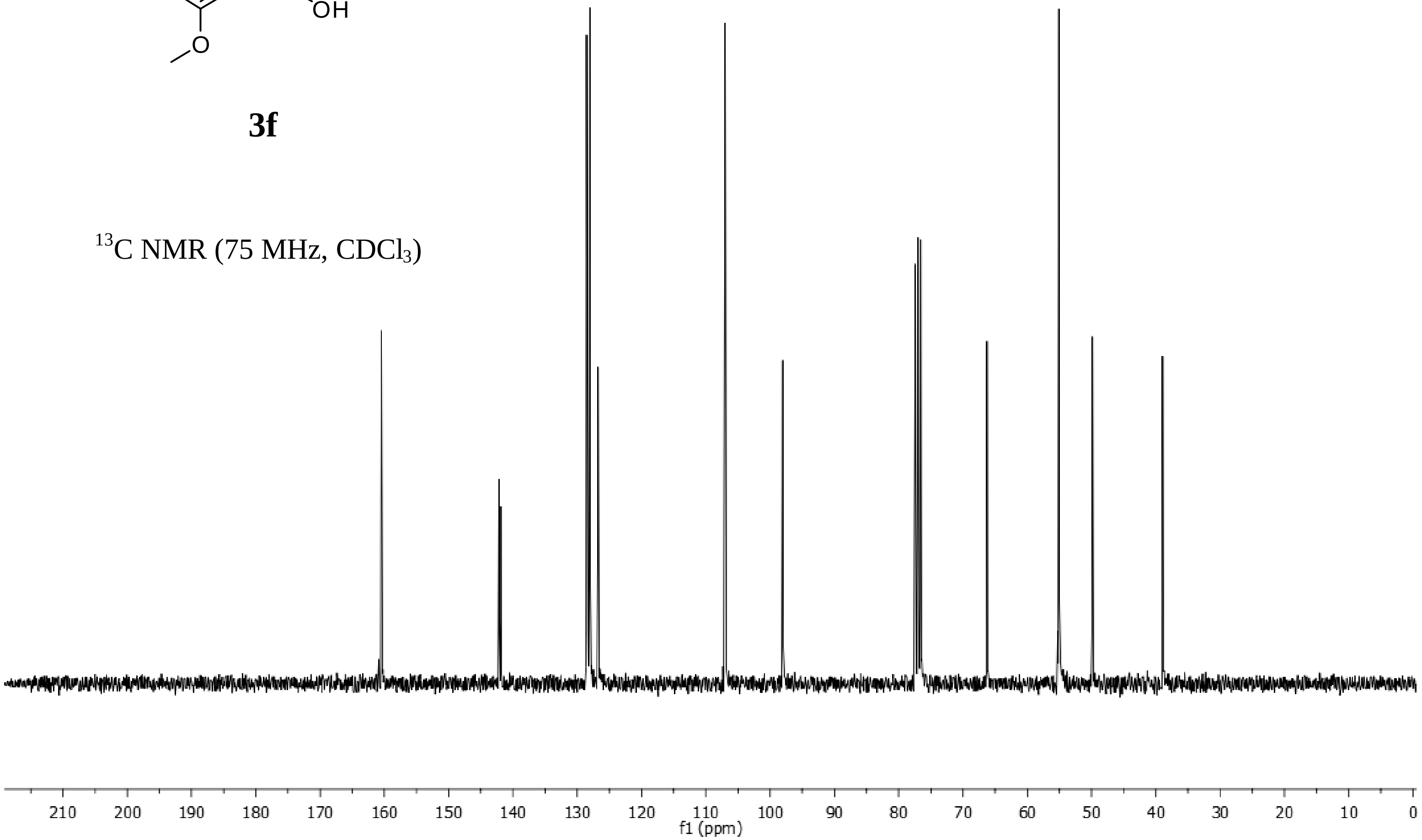
$^1$H NMR (300 MHz, $CDCl_3$)

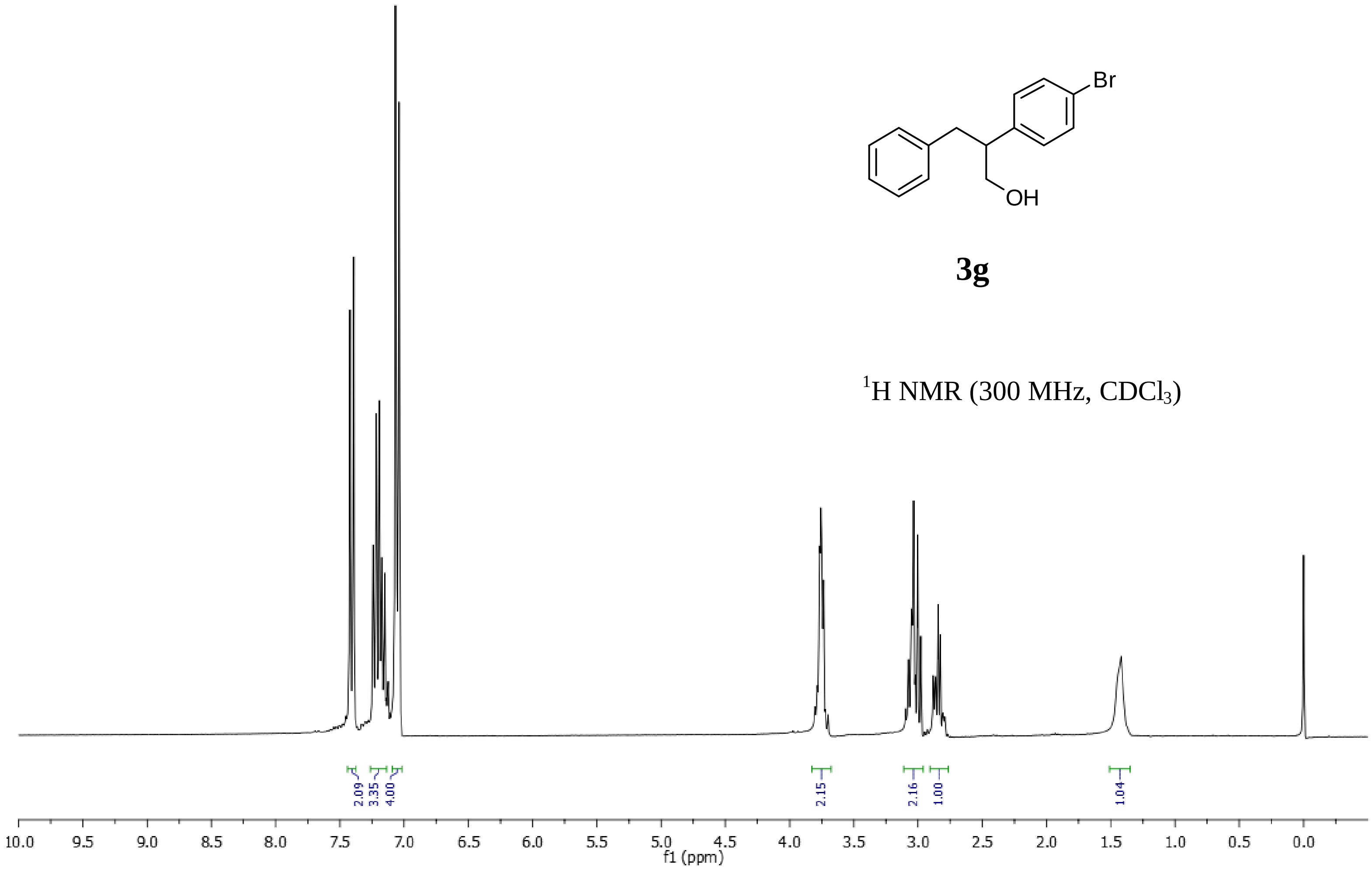




**3f**

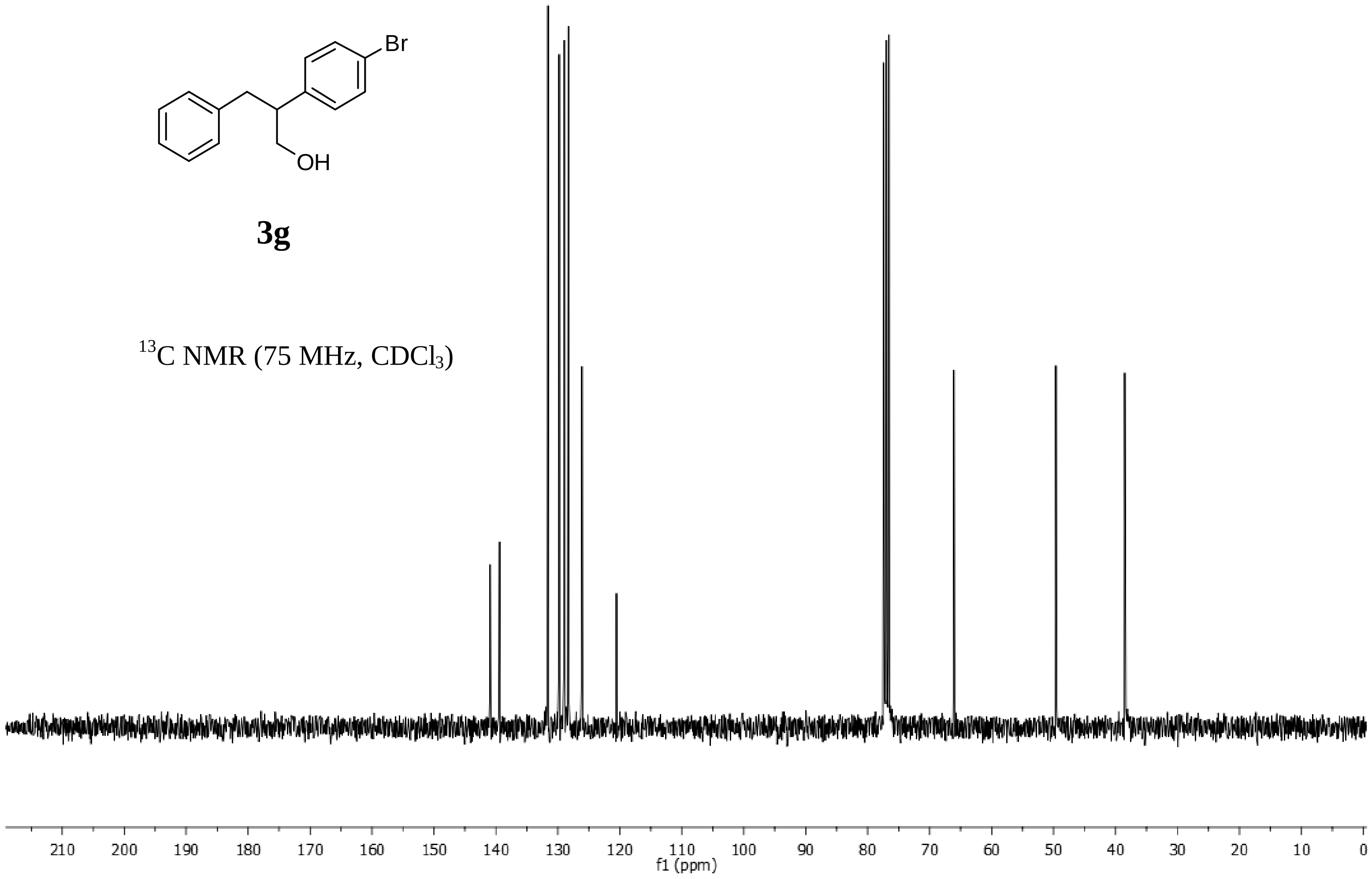
$^{13}$C NMR (75 MHz, CDCl$_3$)





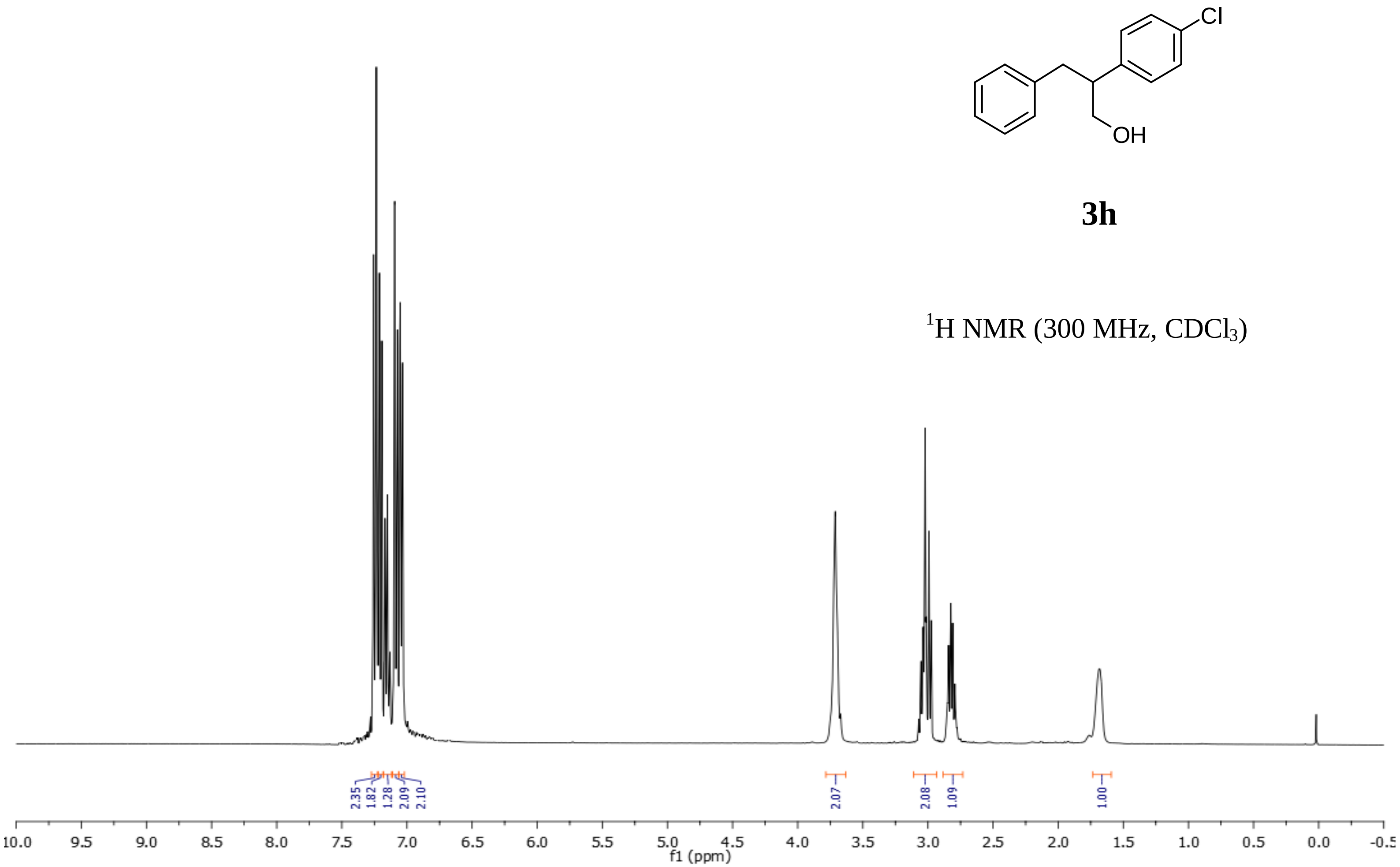
**3g**

$^1$H NMR (300 MHz, $CDCl_3$)



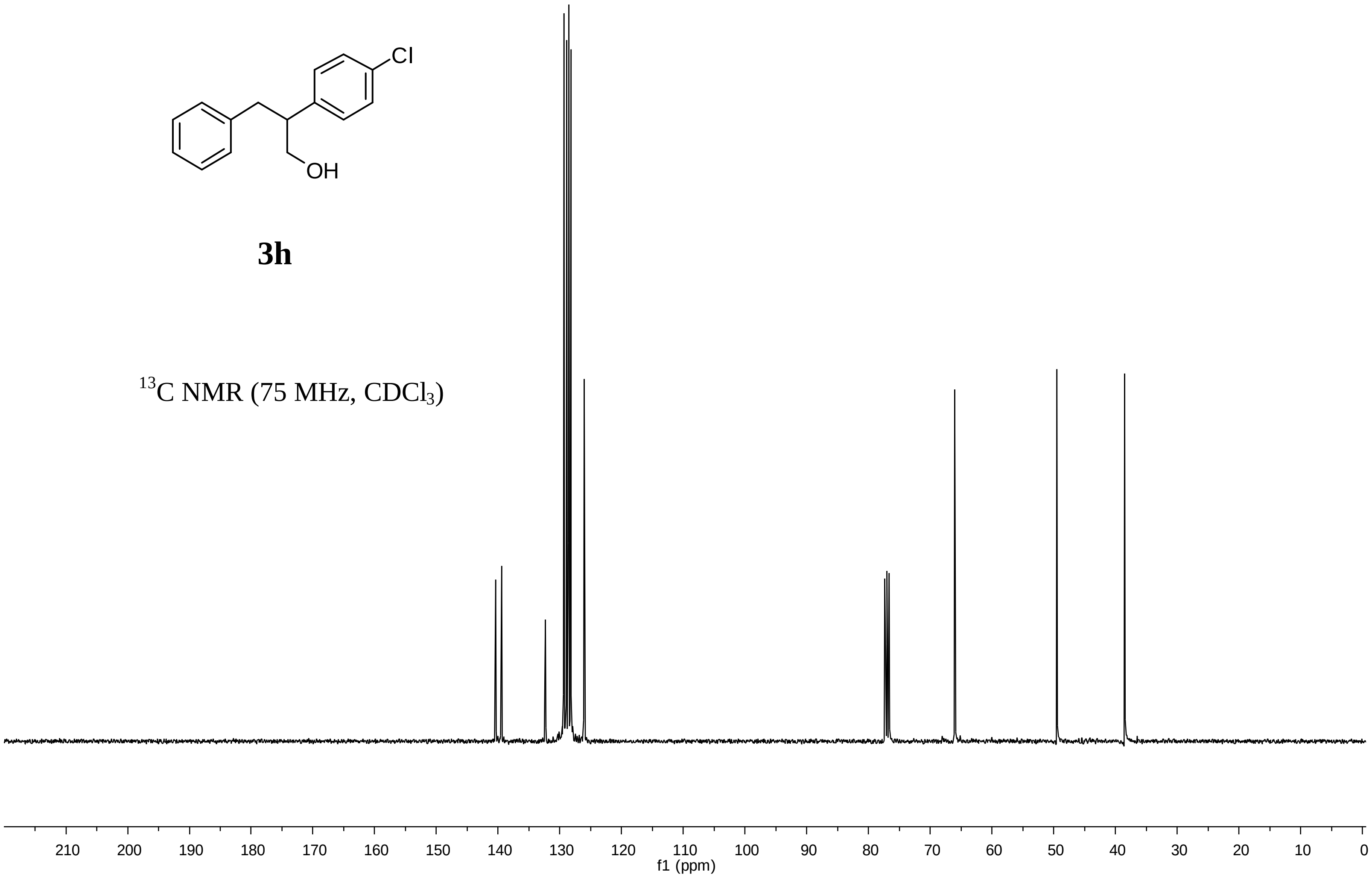
**3g**

$^{13}$C NMR (75 MHz, $CDCl_3$)



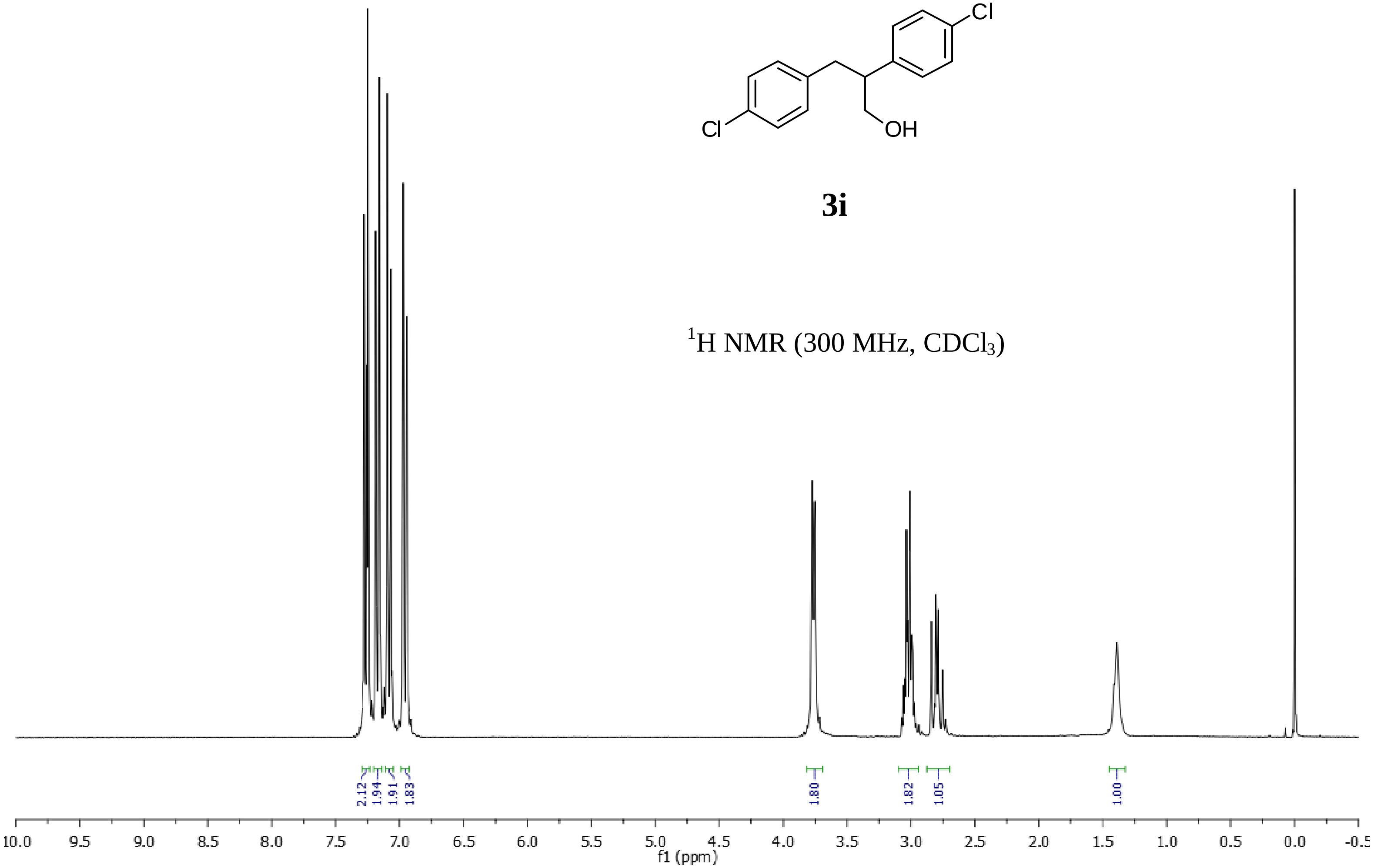
**3h**

$^1$H NMR (300 MHz, CDCl$_3$)



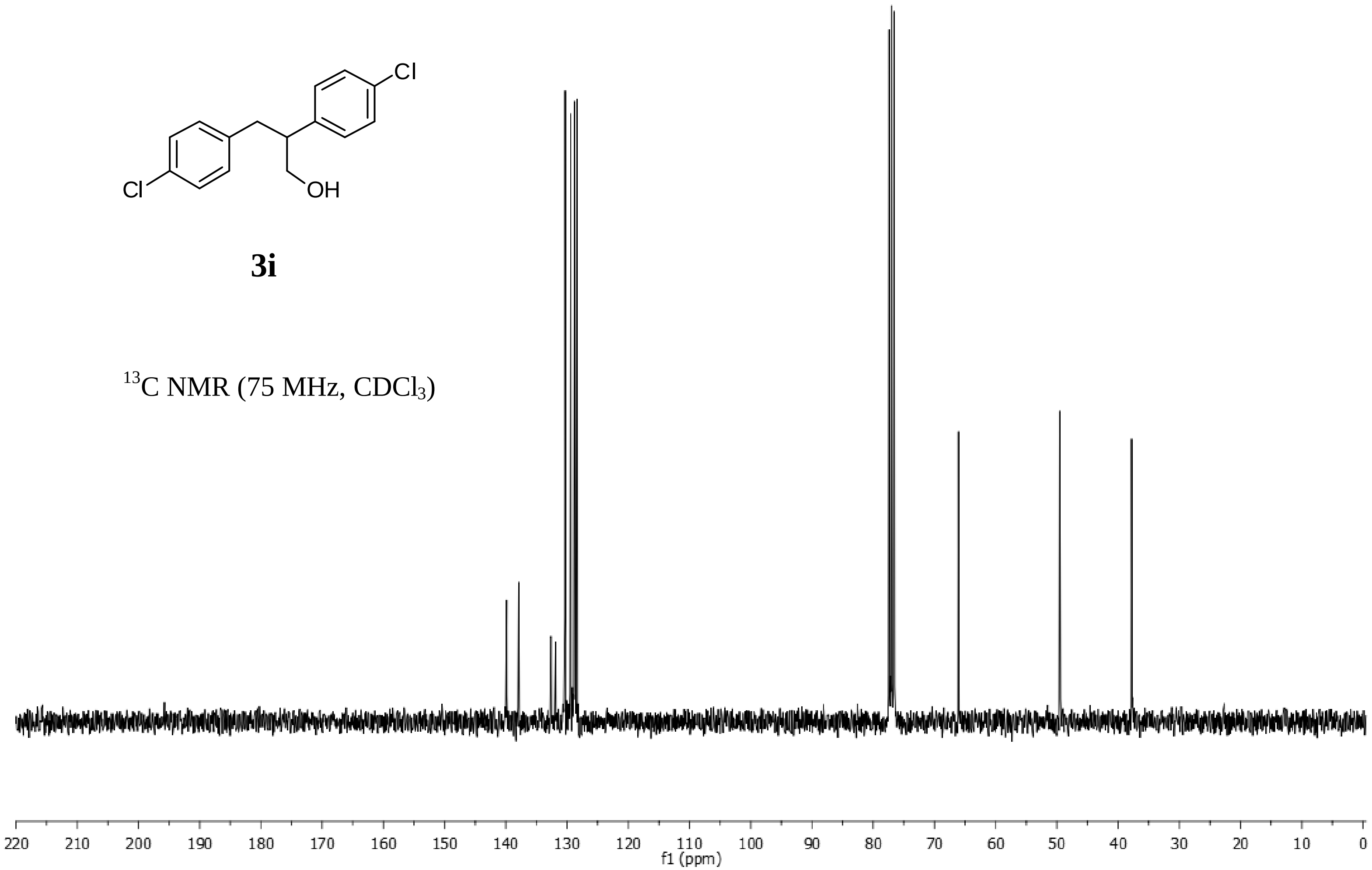
**3h**

$^{13}$C NMR (75 MHz, $CDCl_3$)



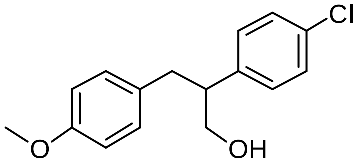
**3i**

$^1$H NMR (300 MHz, $CDCl_3$)



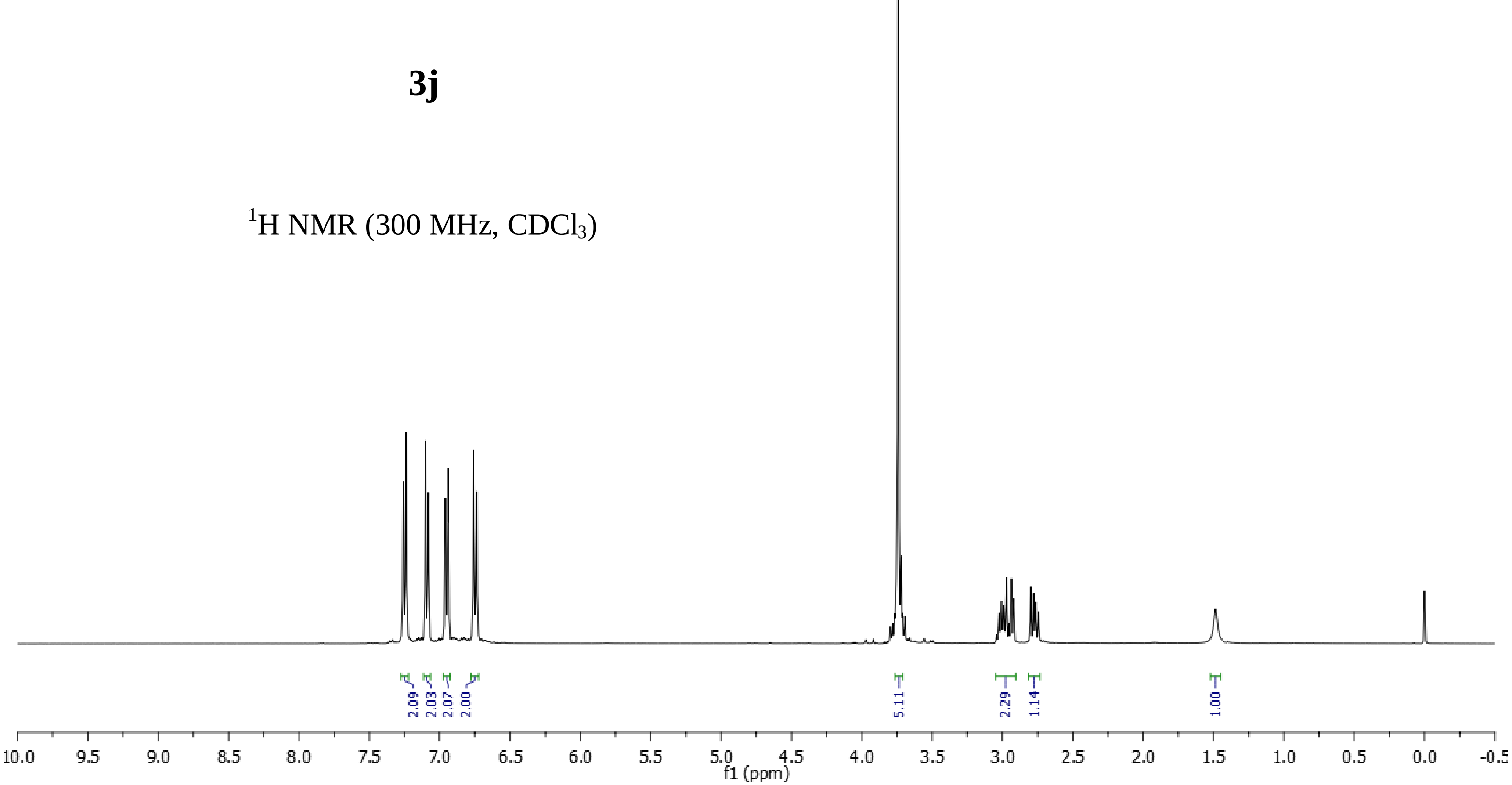
**3i**

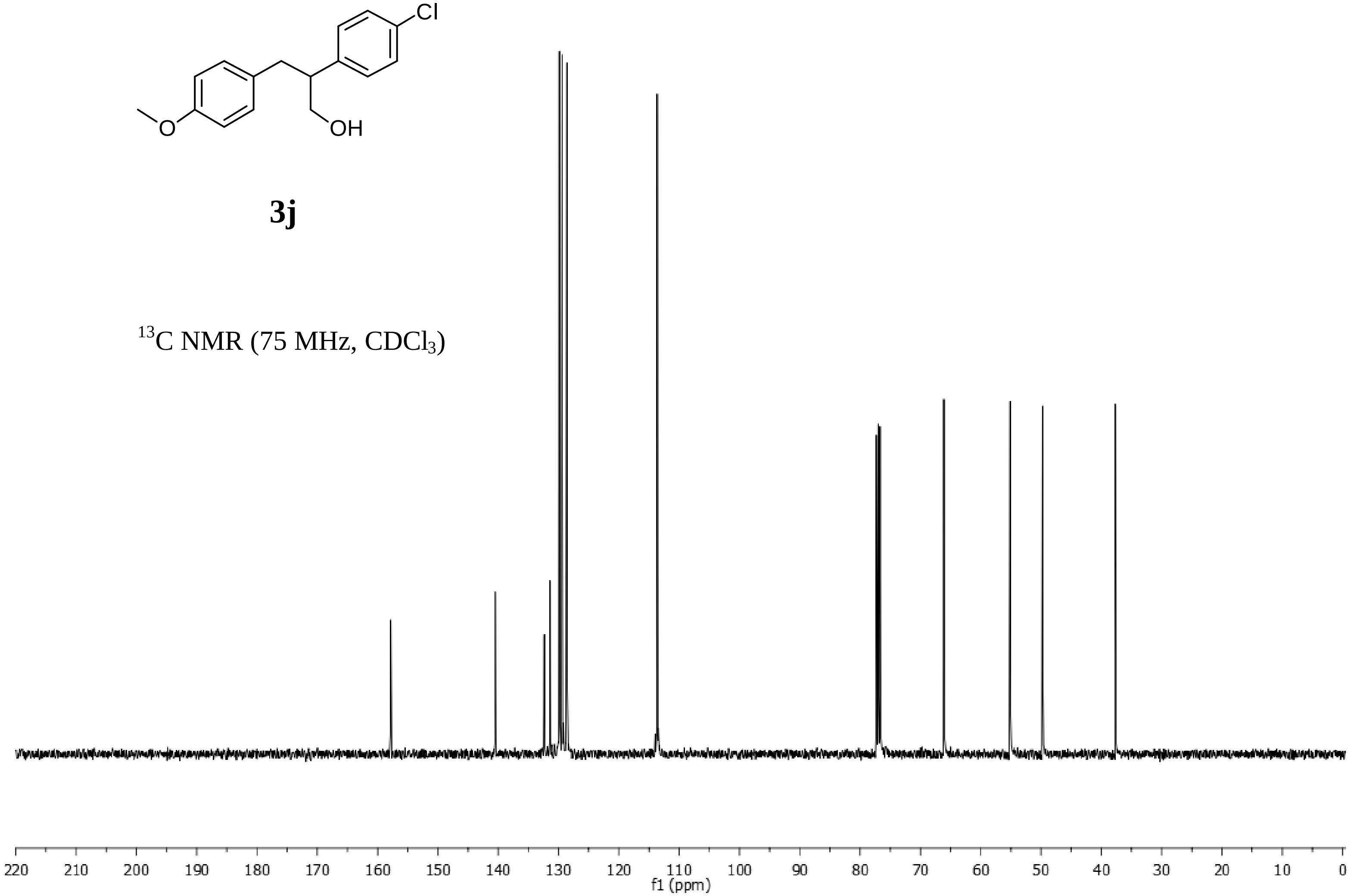
$^{13}$C NMR (75 MHz, $CDCl_3$)



**3j**

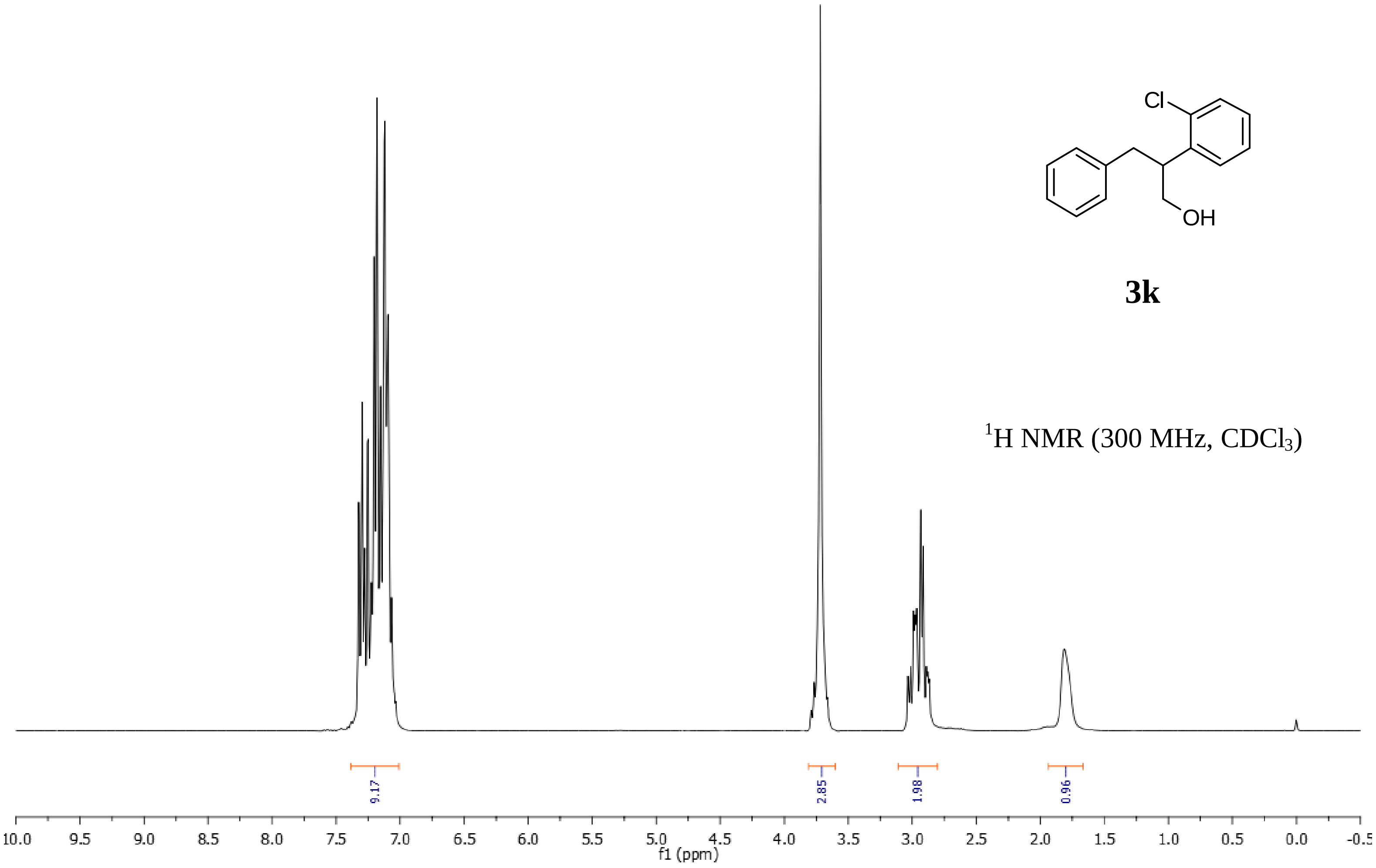
$^1$H NMR (300 MHz, CDCl$_3$)





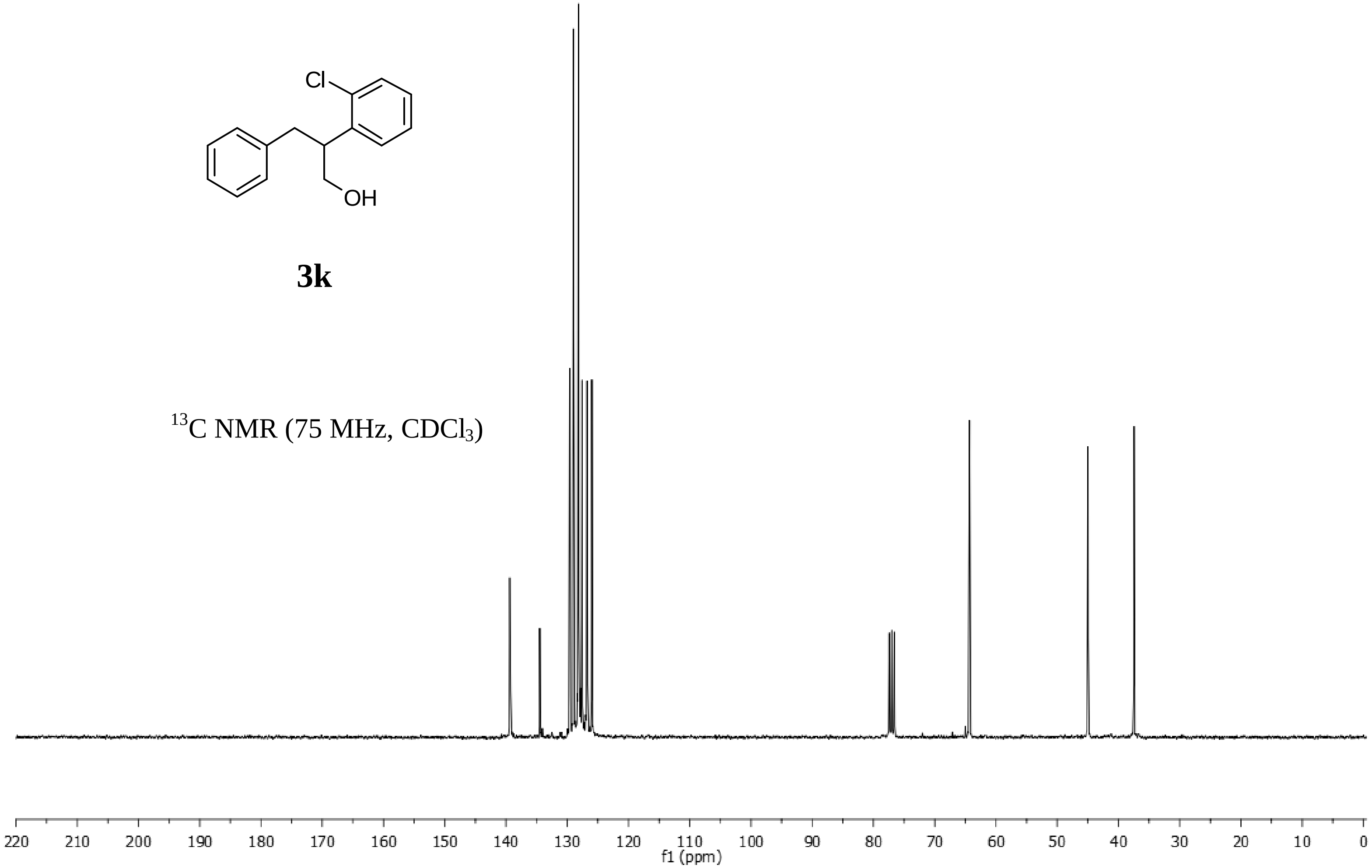
**3j**

$^{13}$C NMR (75 MHz, $CDCl_3$)



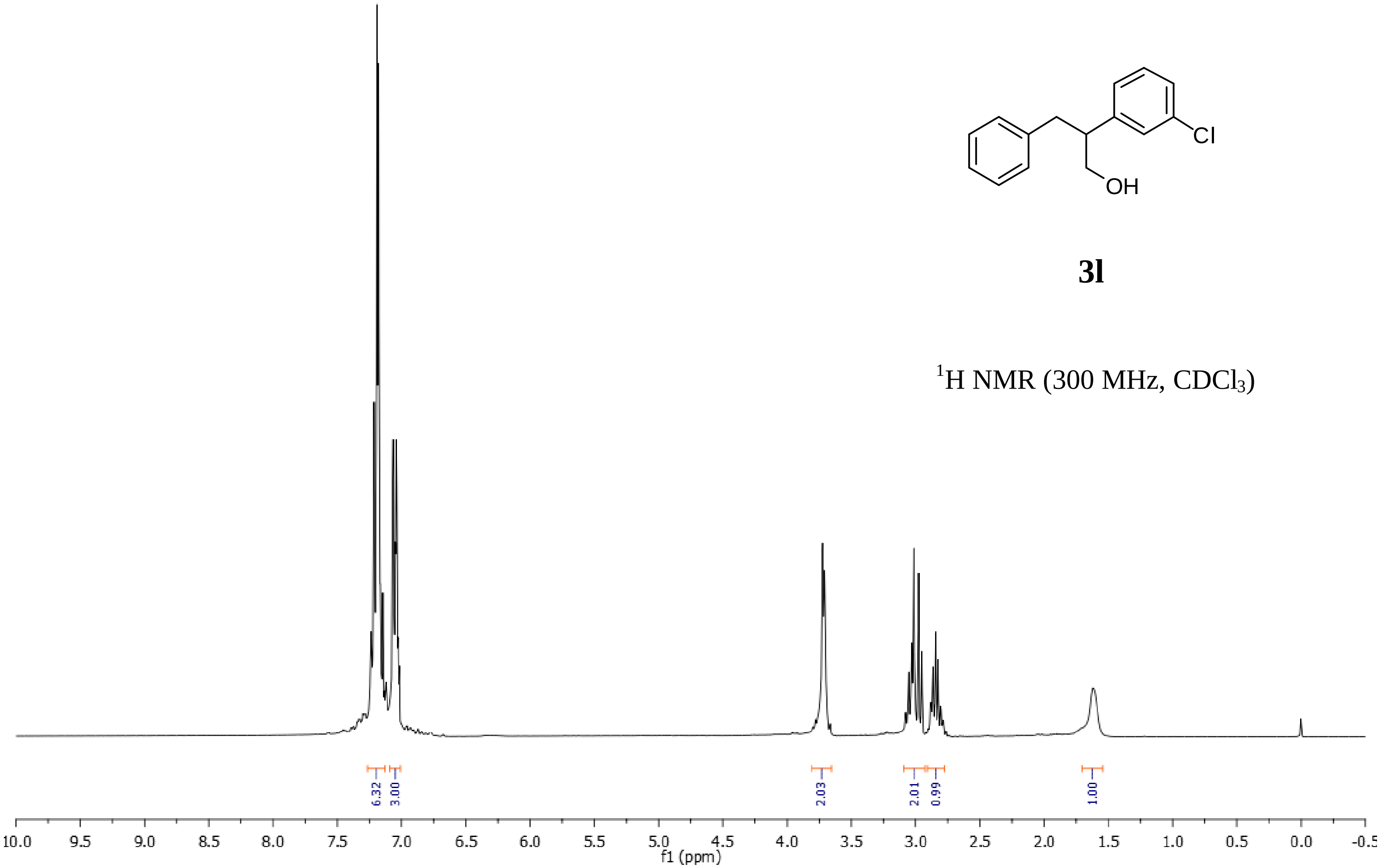
**3k**

$^1$H NMR (300 MHz, $CDCl_3$)



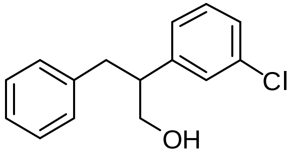
**3k**

$^{13}$C NMR (75 MHz, $CDCl_3$)



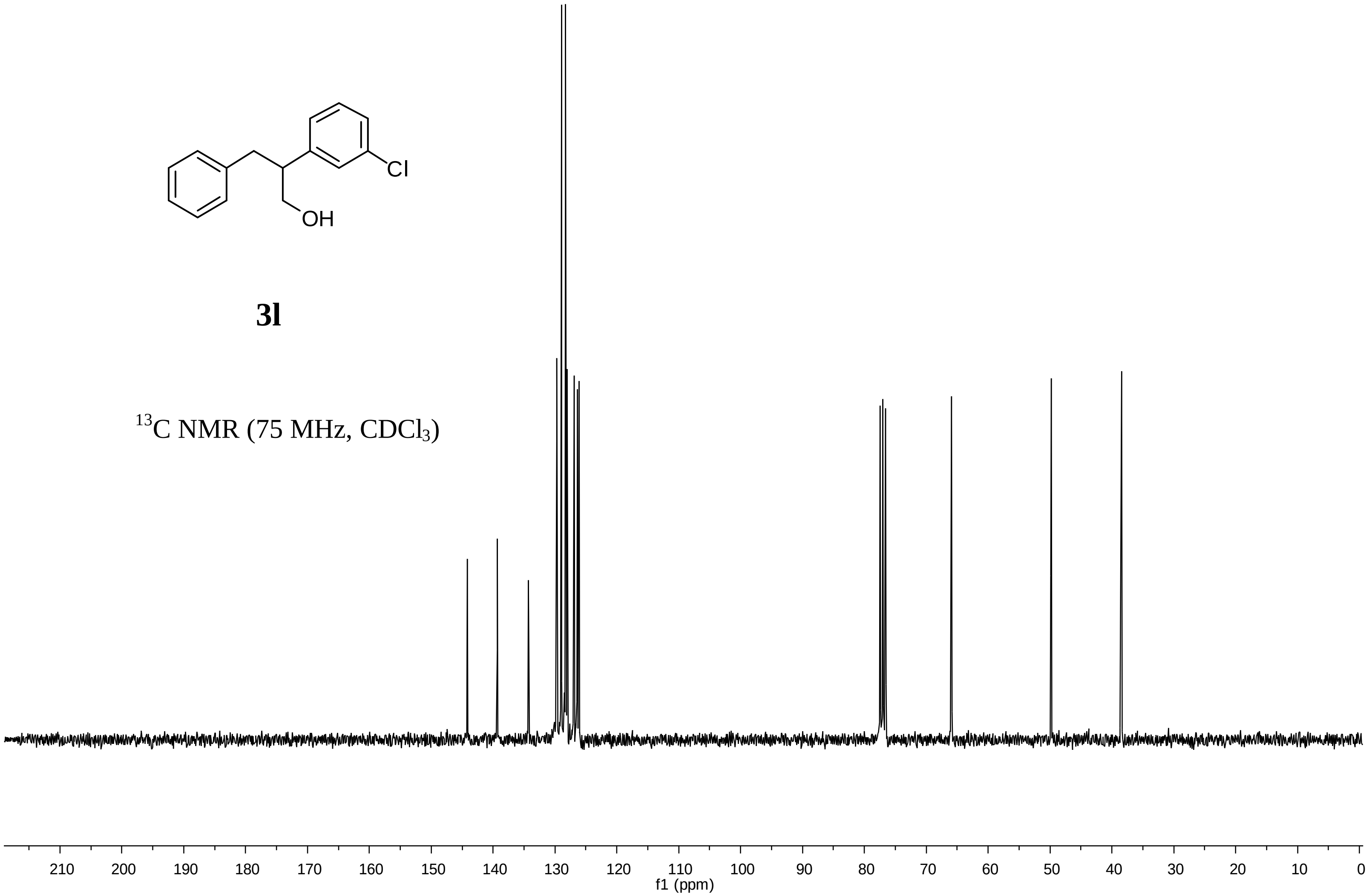
**3l**

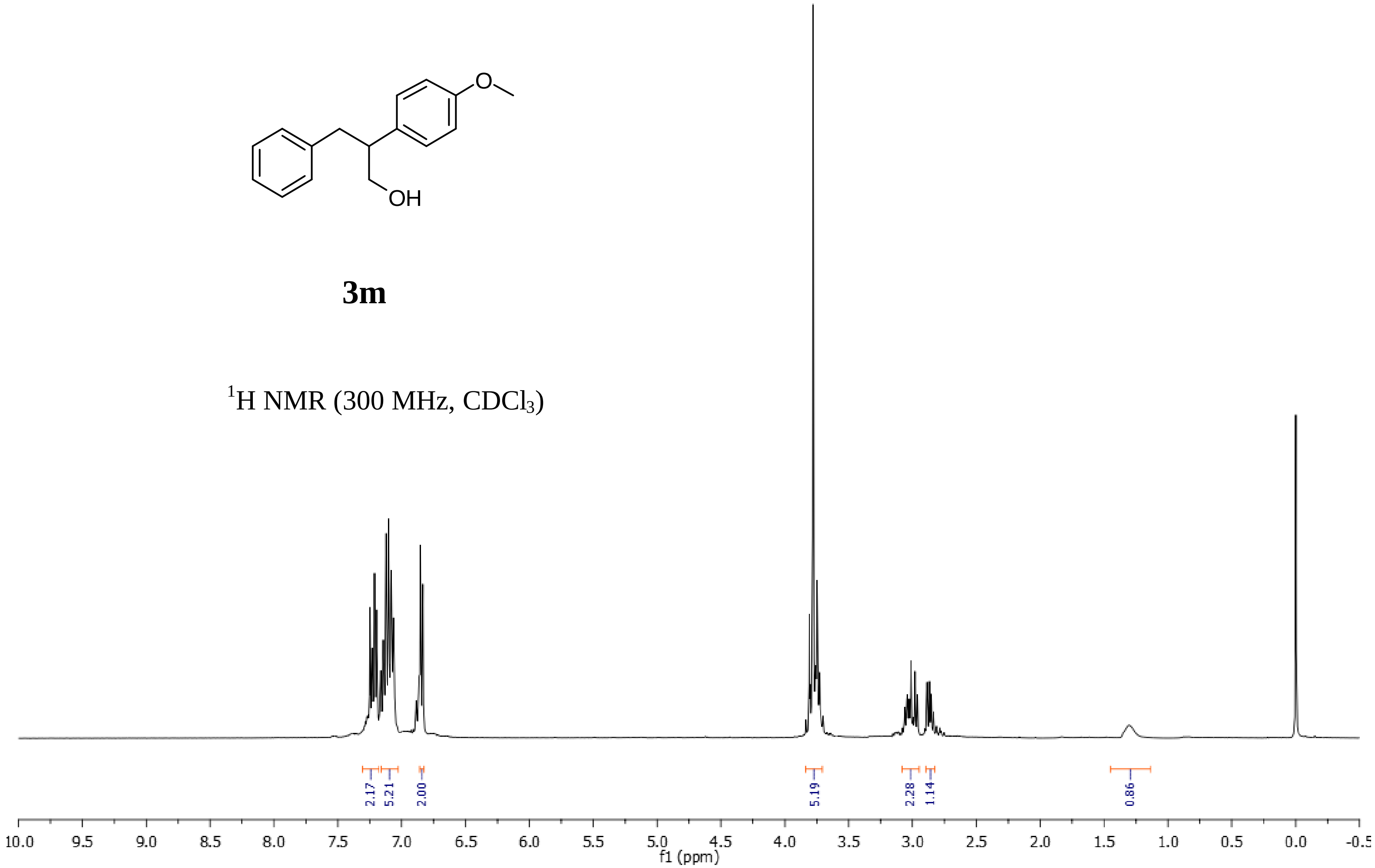
$^1$H NMR (300 MHz, $CDCl_3$)



**3l**

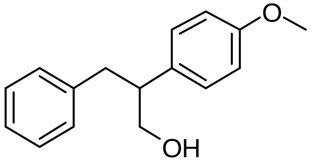
$^{13}$C NMR (75 MHz, $CDCl_3$)





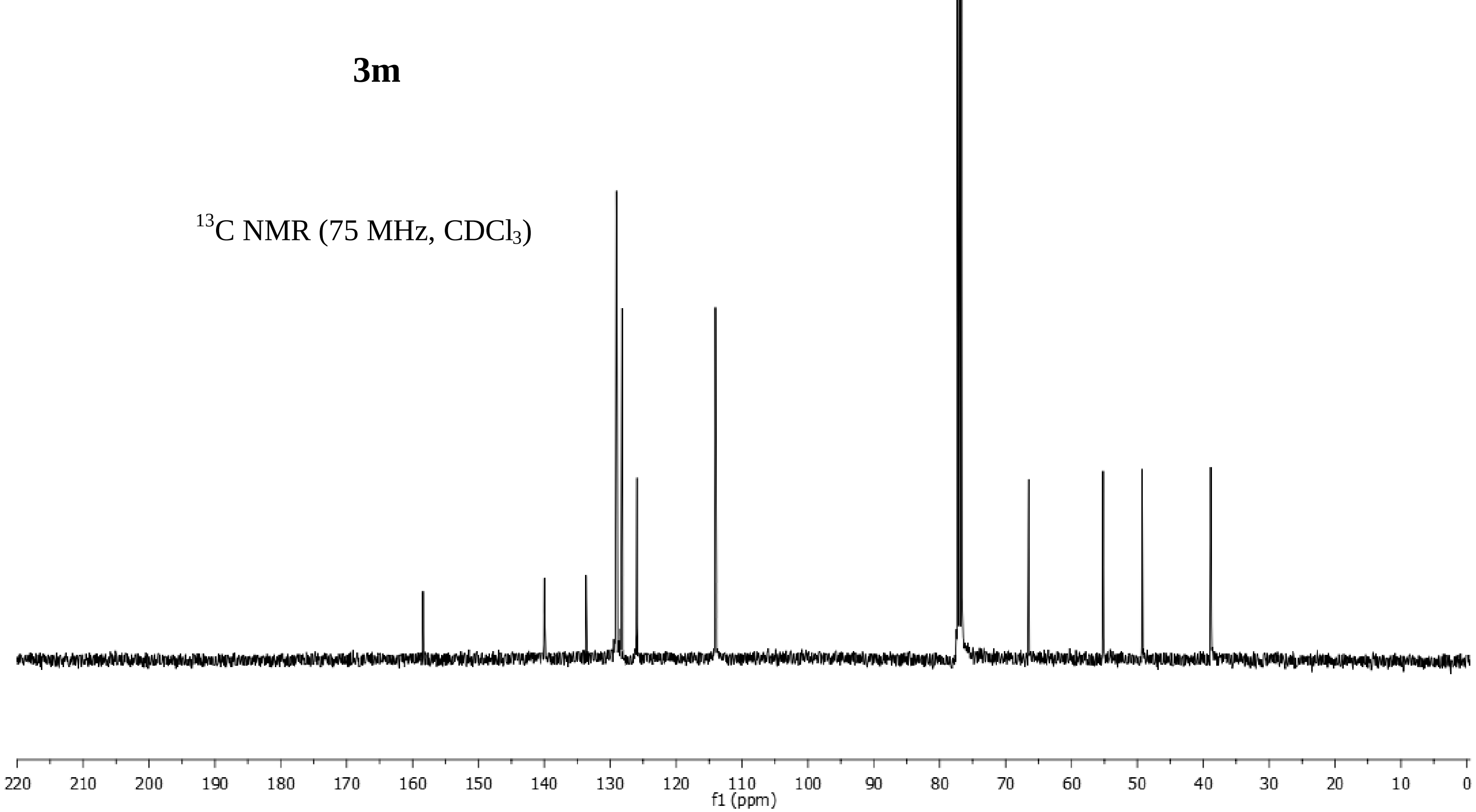
**3m**

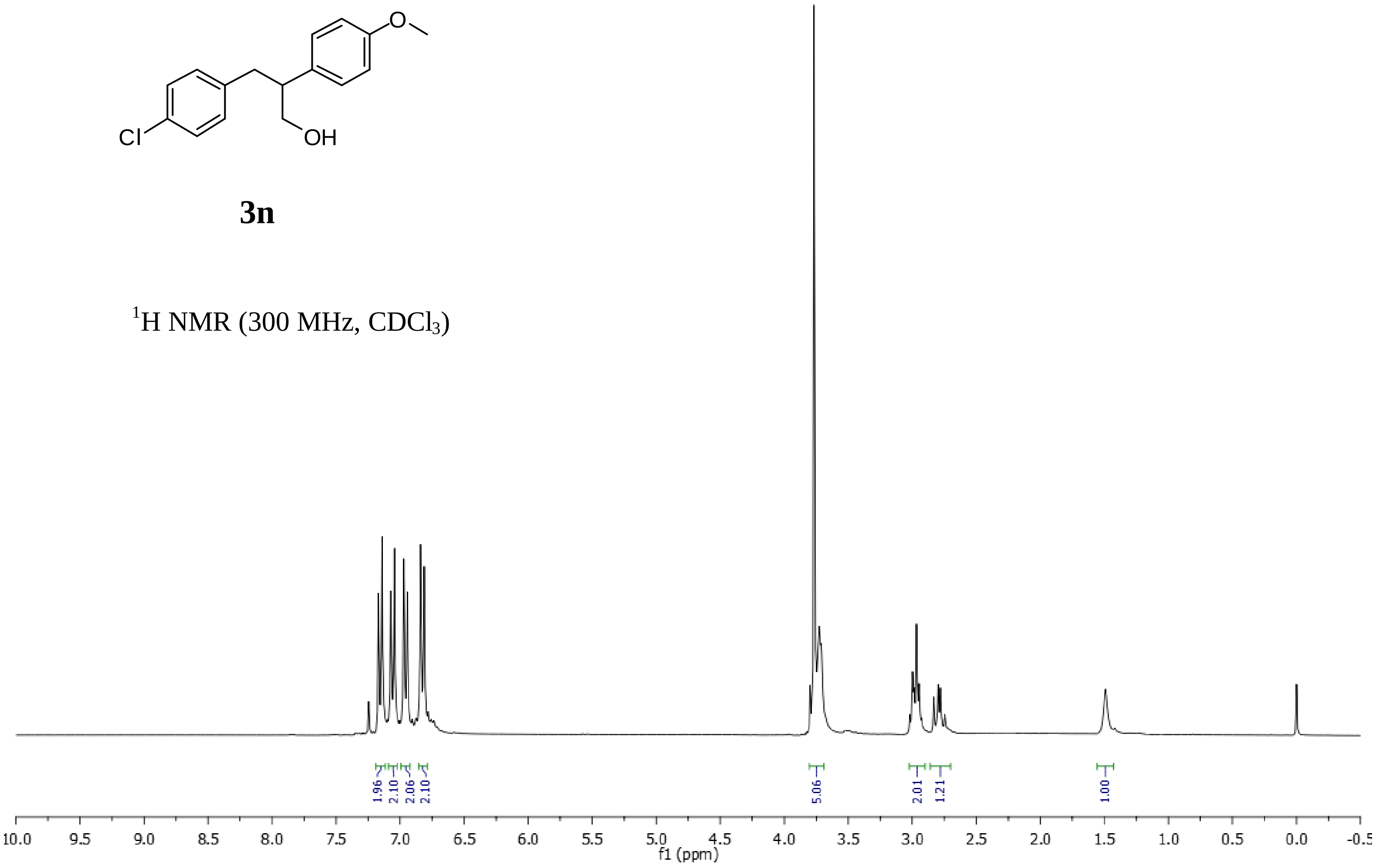
$^1$H NMR (300 MHz, $CDCl_3$)



**3m**

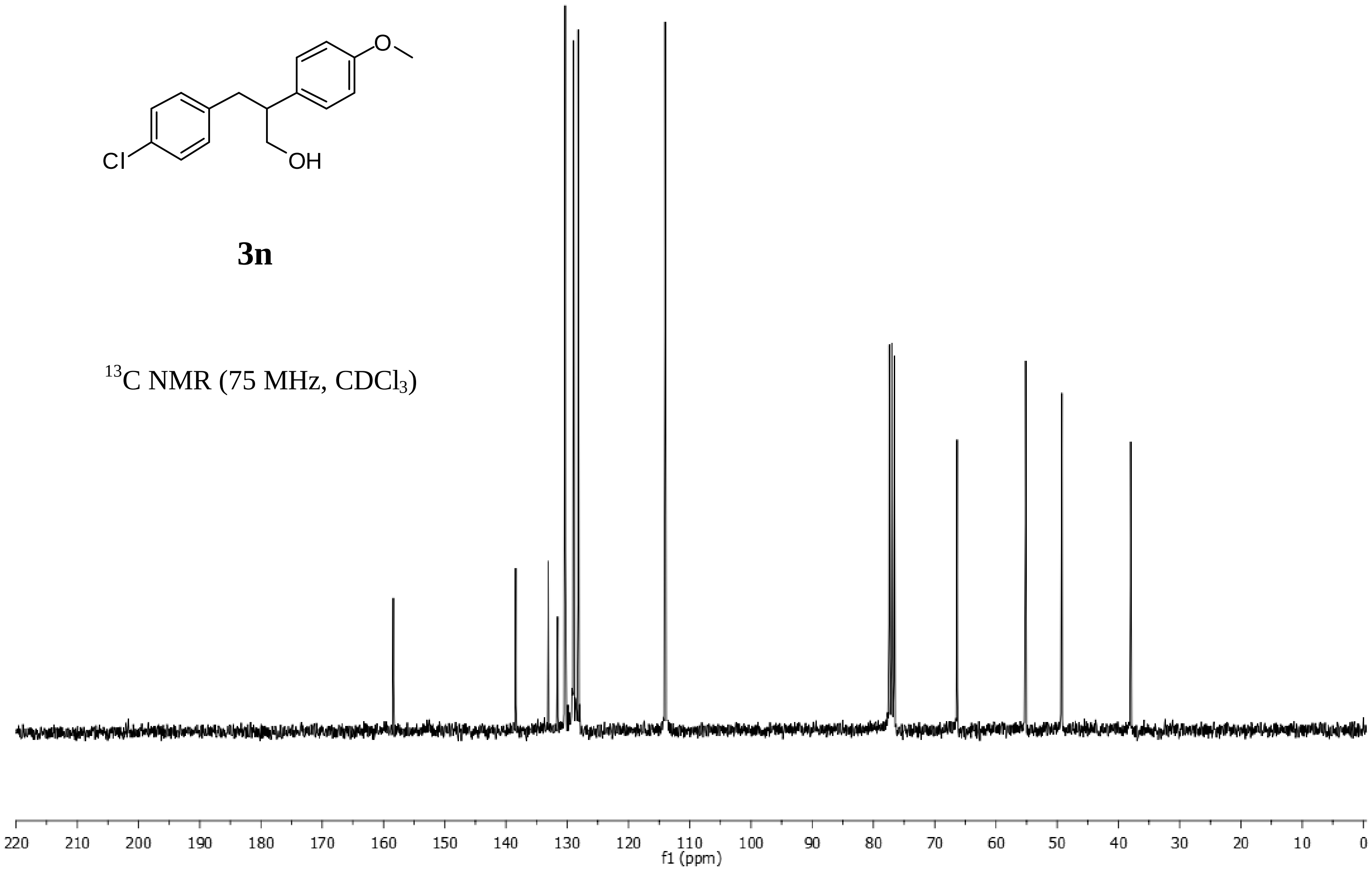
$^{13}$C NMR (75 MHz, $CDCl_3$)





**3n**

$^1$H NMR (300 MHz, $CDCl_3$)

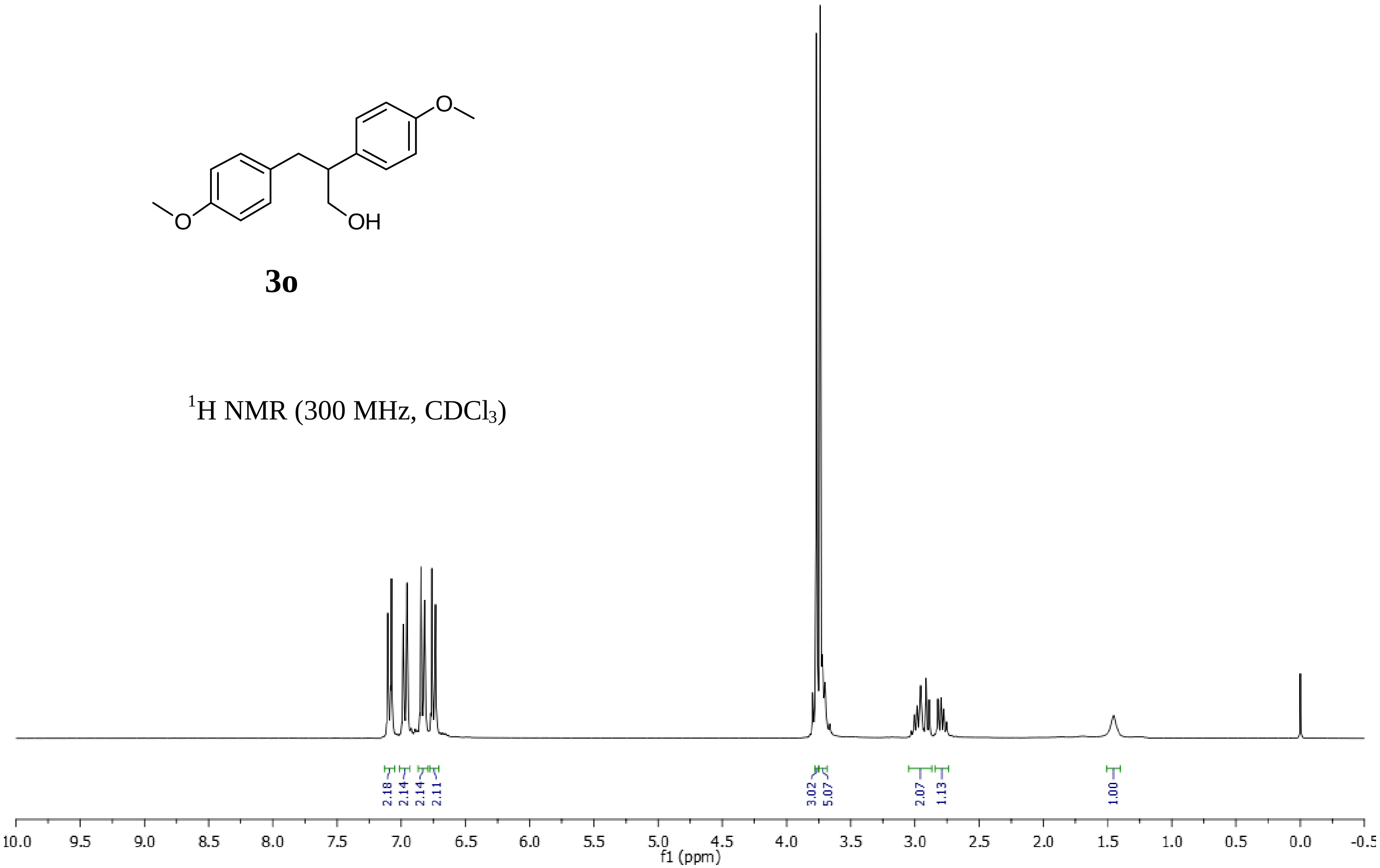


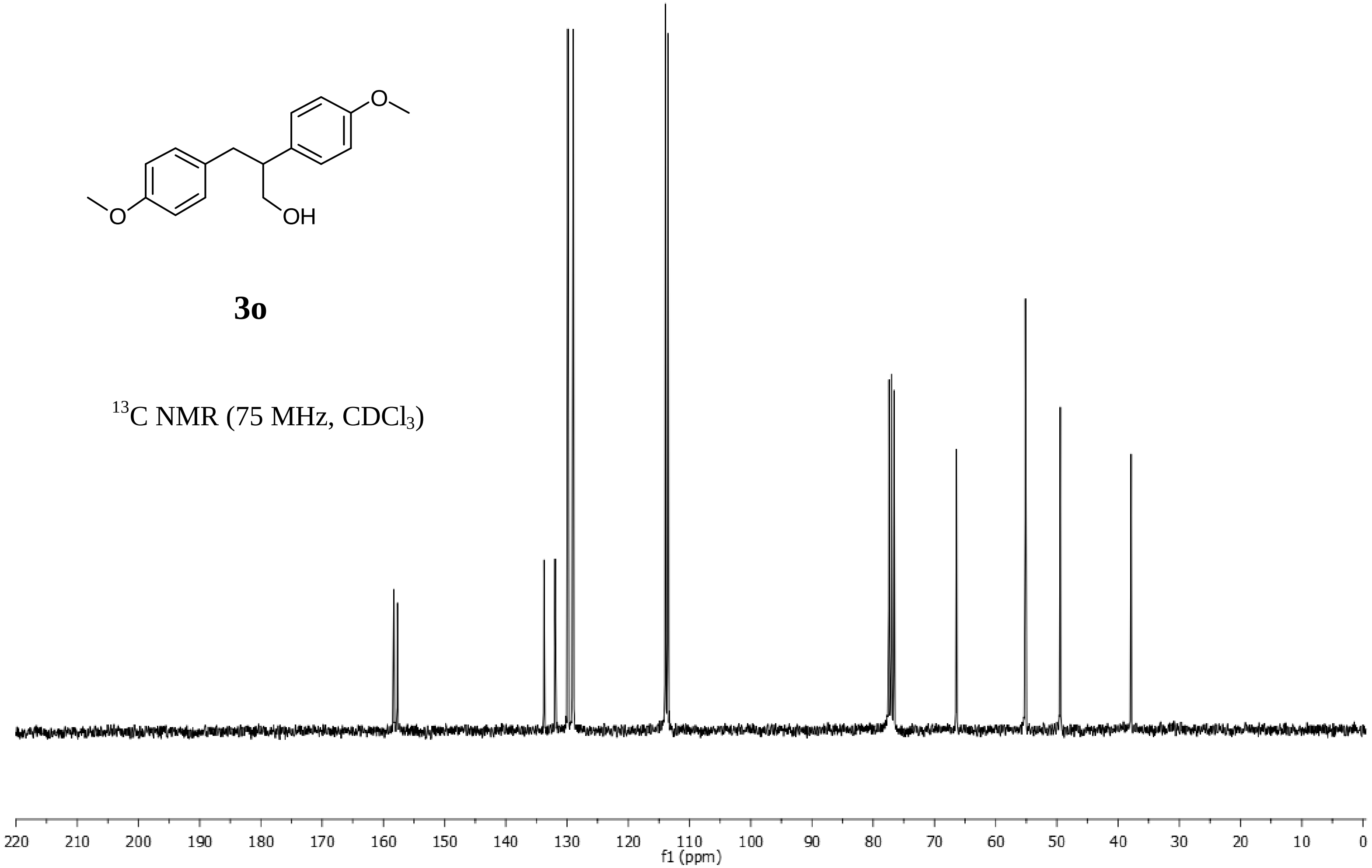
**3n**

$^{13}$C NMR (75 MHz, CDCl$_3$)

**3o**

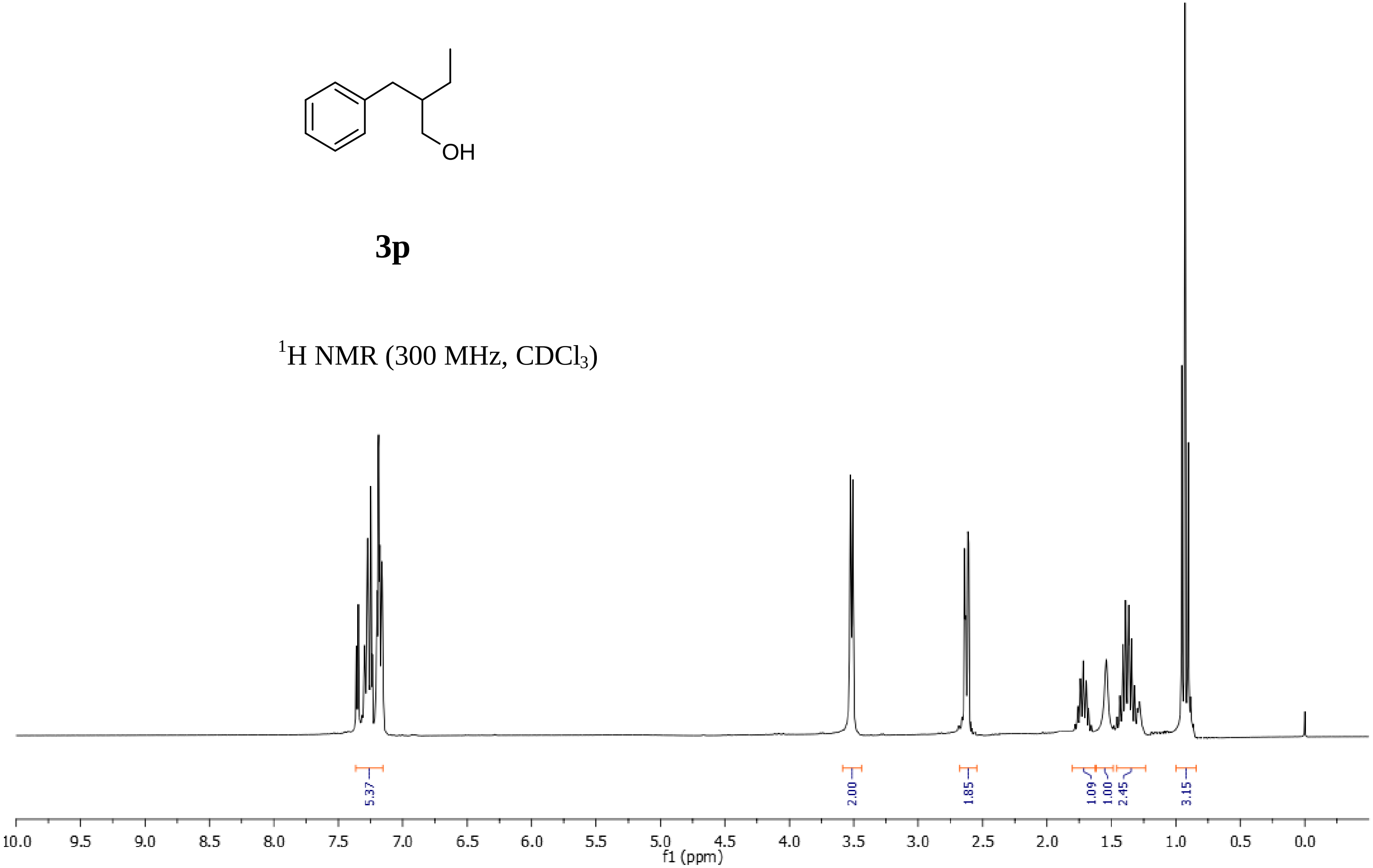
$^1$H NMR (300 MHz, $CDCl_3$)





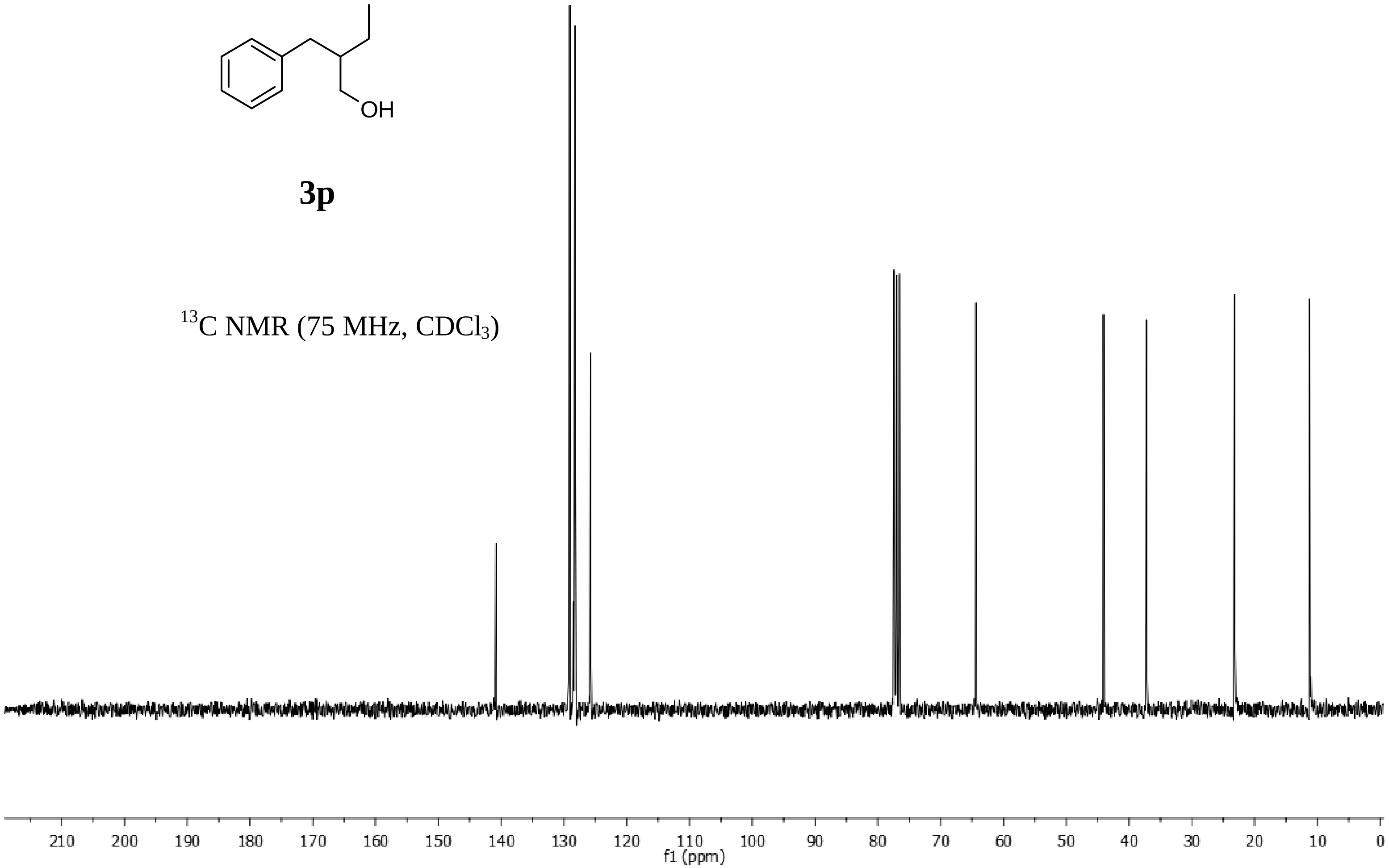
**3o**

$^{13}$C NMR (75 MHz, $CDCl_3$)



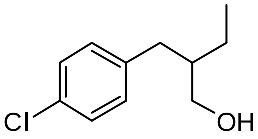
**3p**

$^1$H NMR (300 MHz, $CDCl_3$)



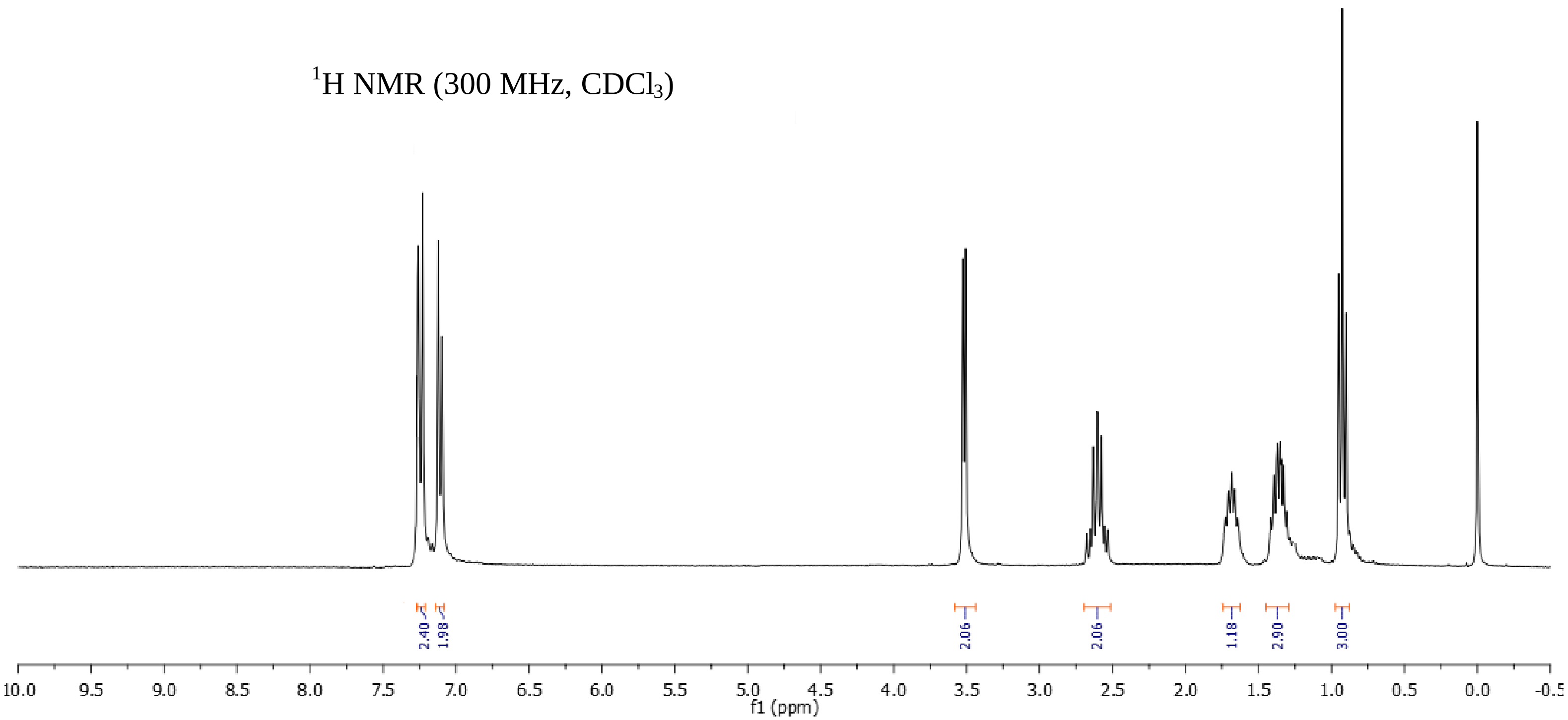
**3p**

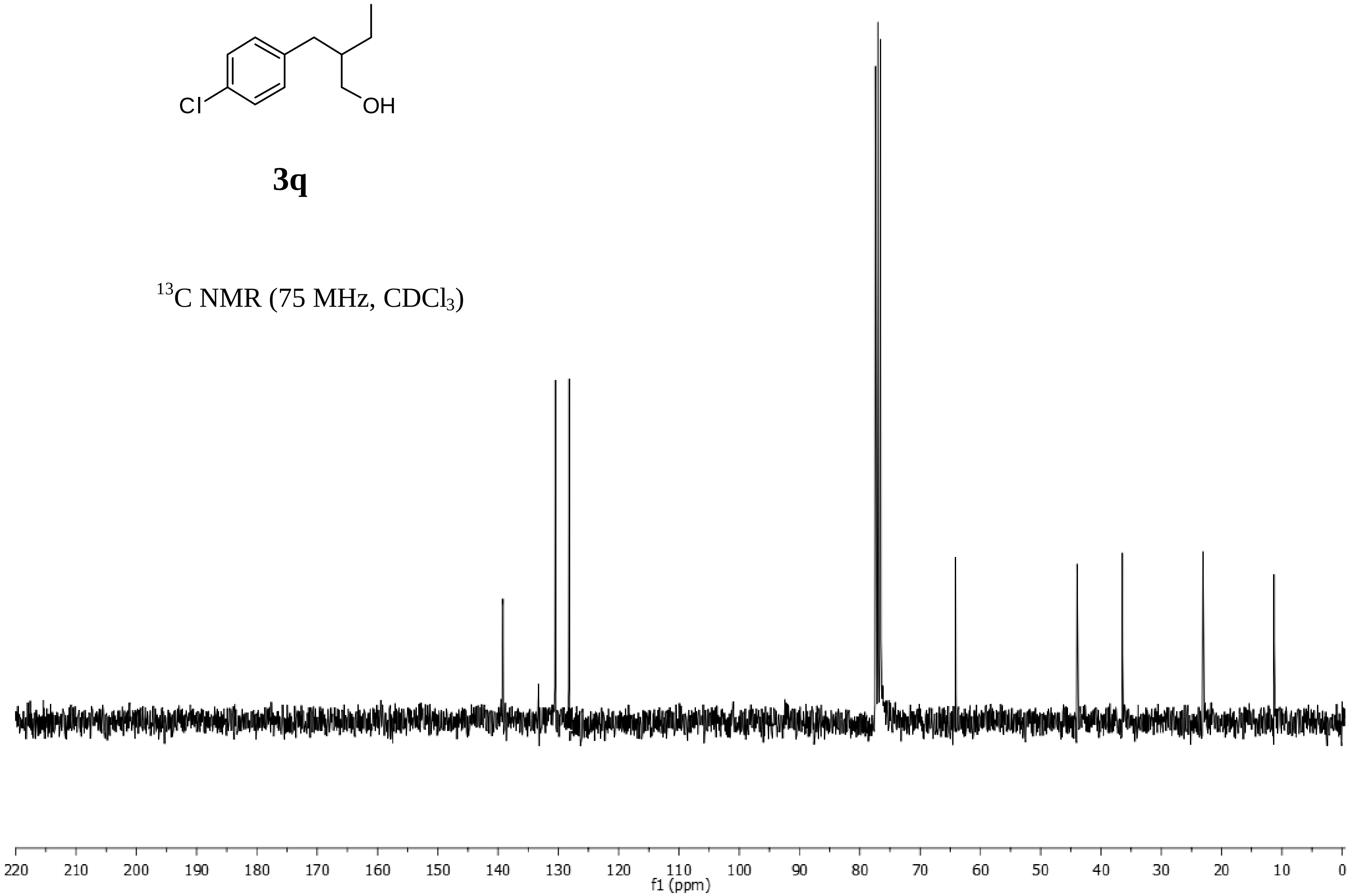
$^{13}$C NMR (75 MHz, $CDCl_3$)



**3q**

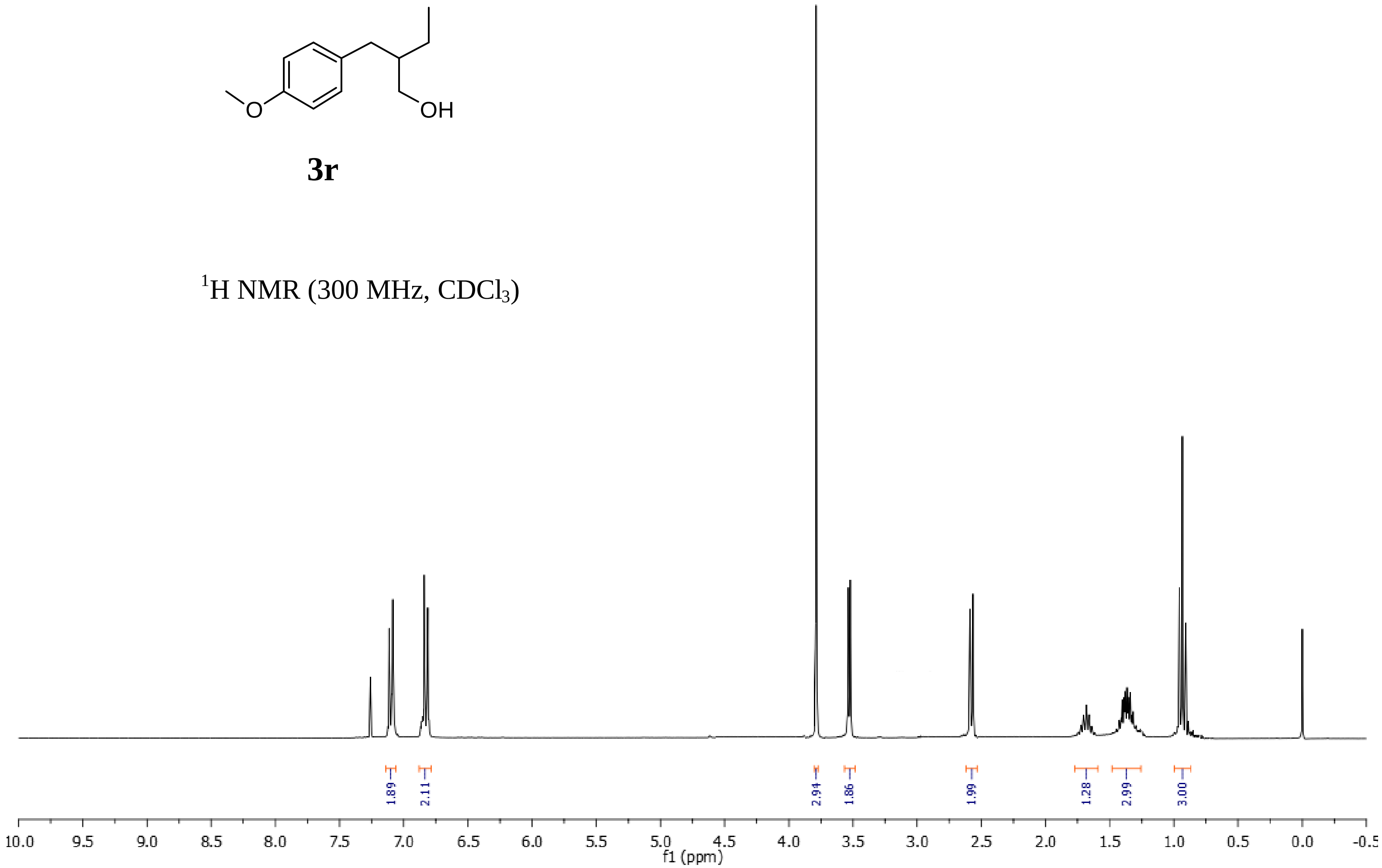
$^1$H NMR (300 MHz, CDCl$_3$)





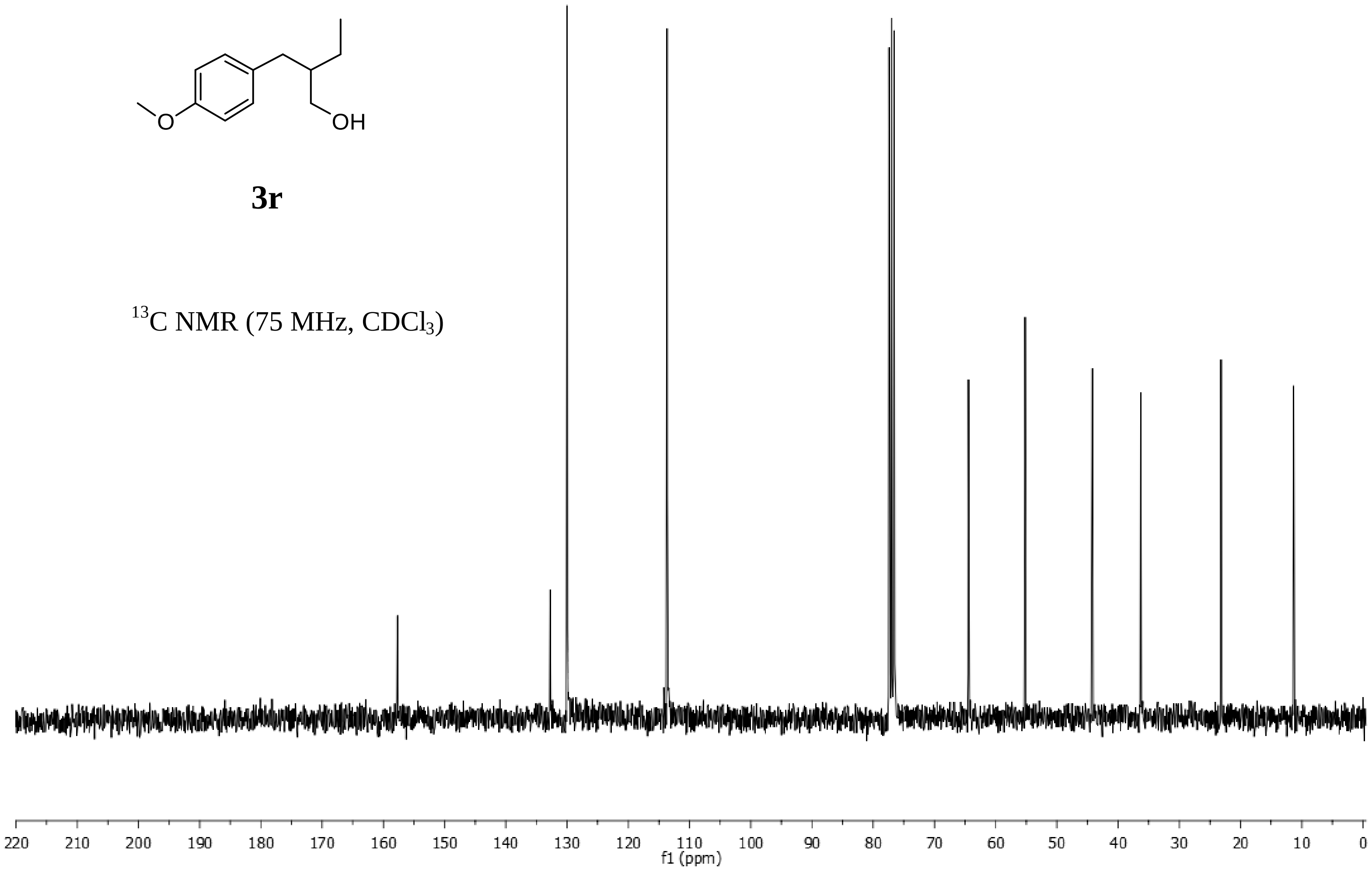
**3q**

$^{13}$C NMR (75 MHz, $CDCl_3$)



**3r**

$^1$H NMR (300 MHz, CDCl$_3$)

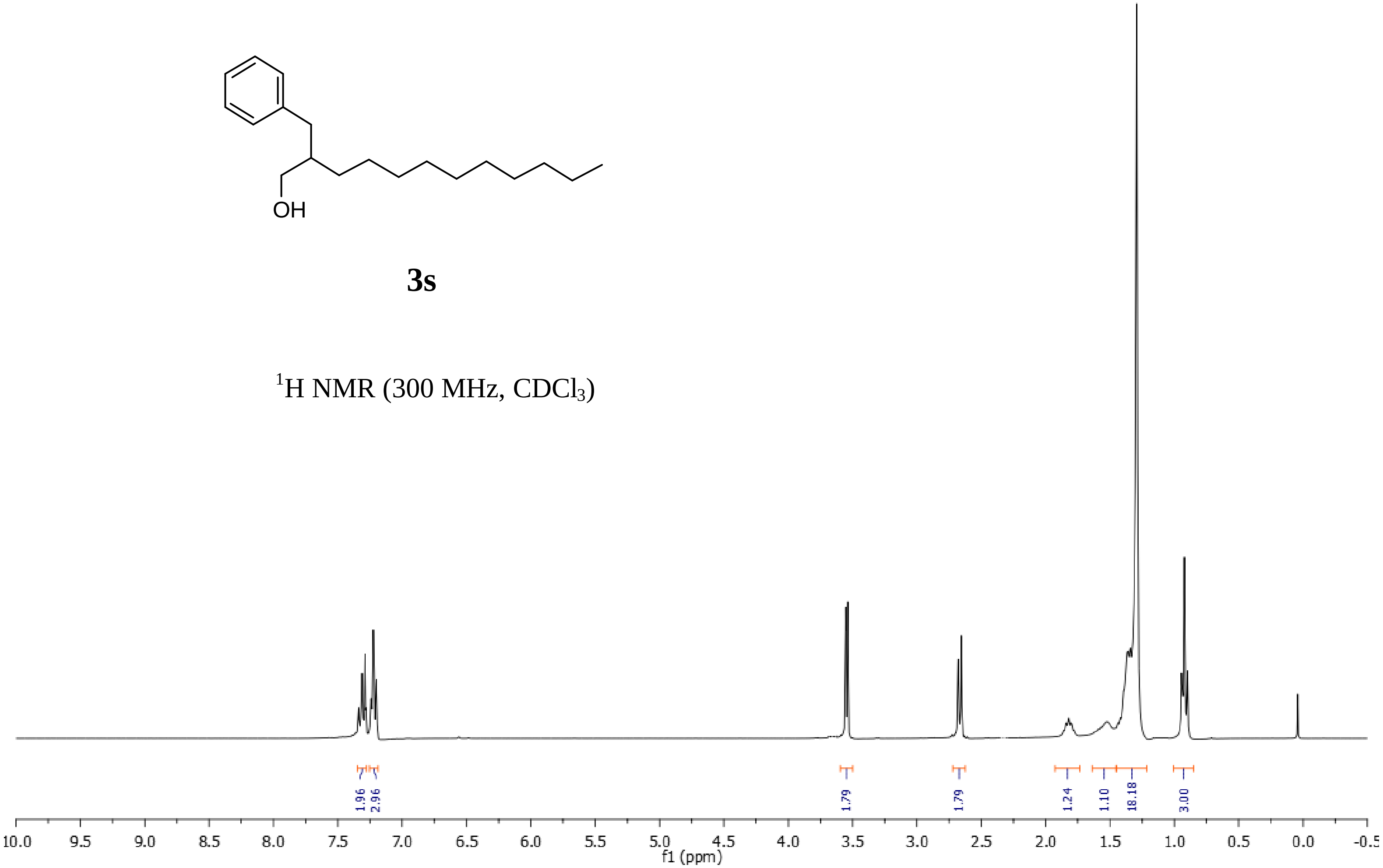


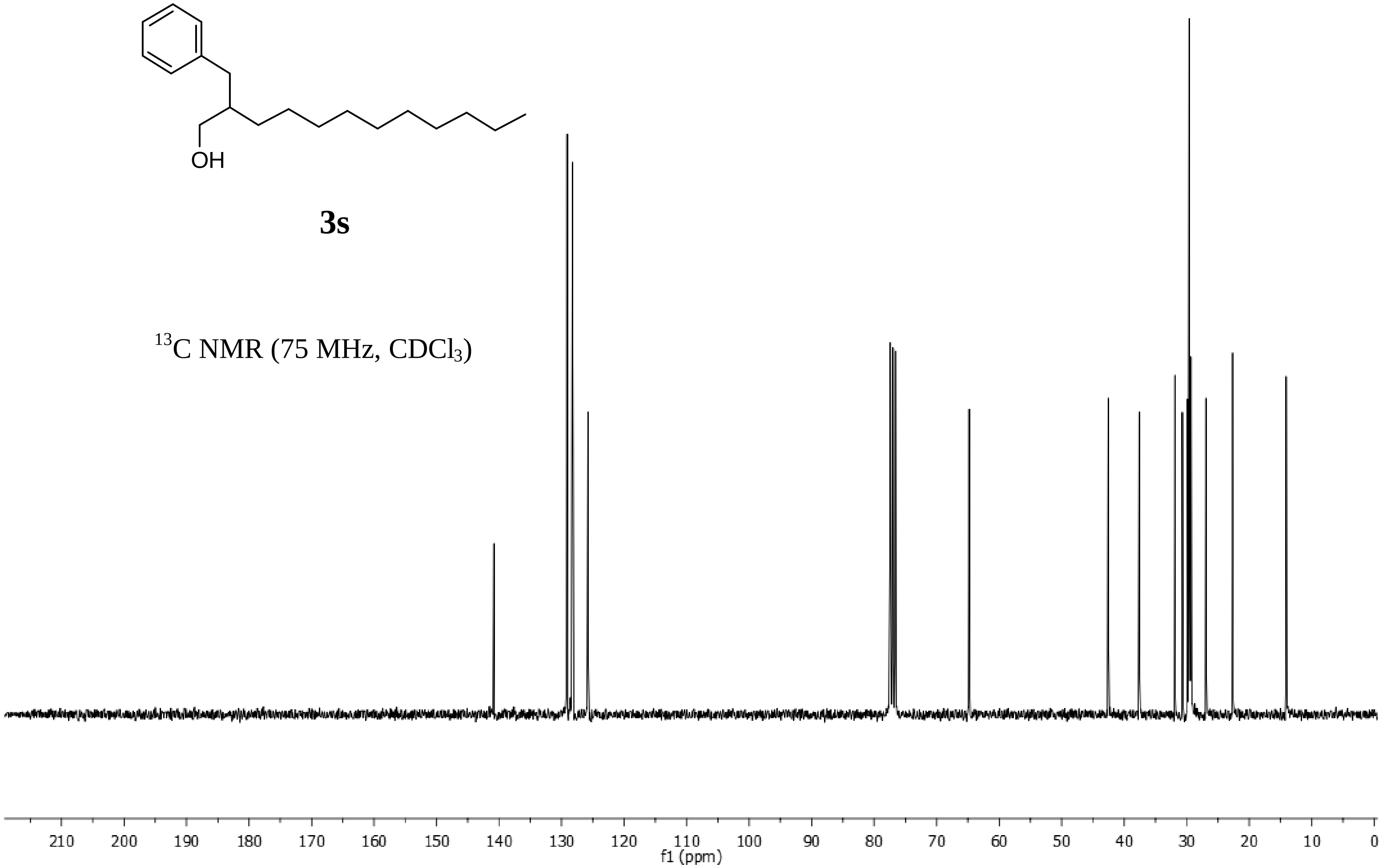
**3r**

$^{13}$C NMR (75 MHz, $CDCl_3$)

**3s**

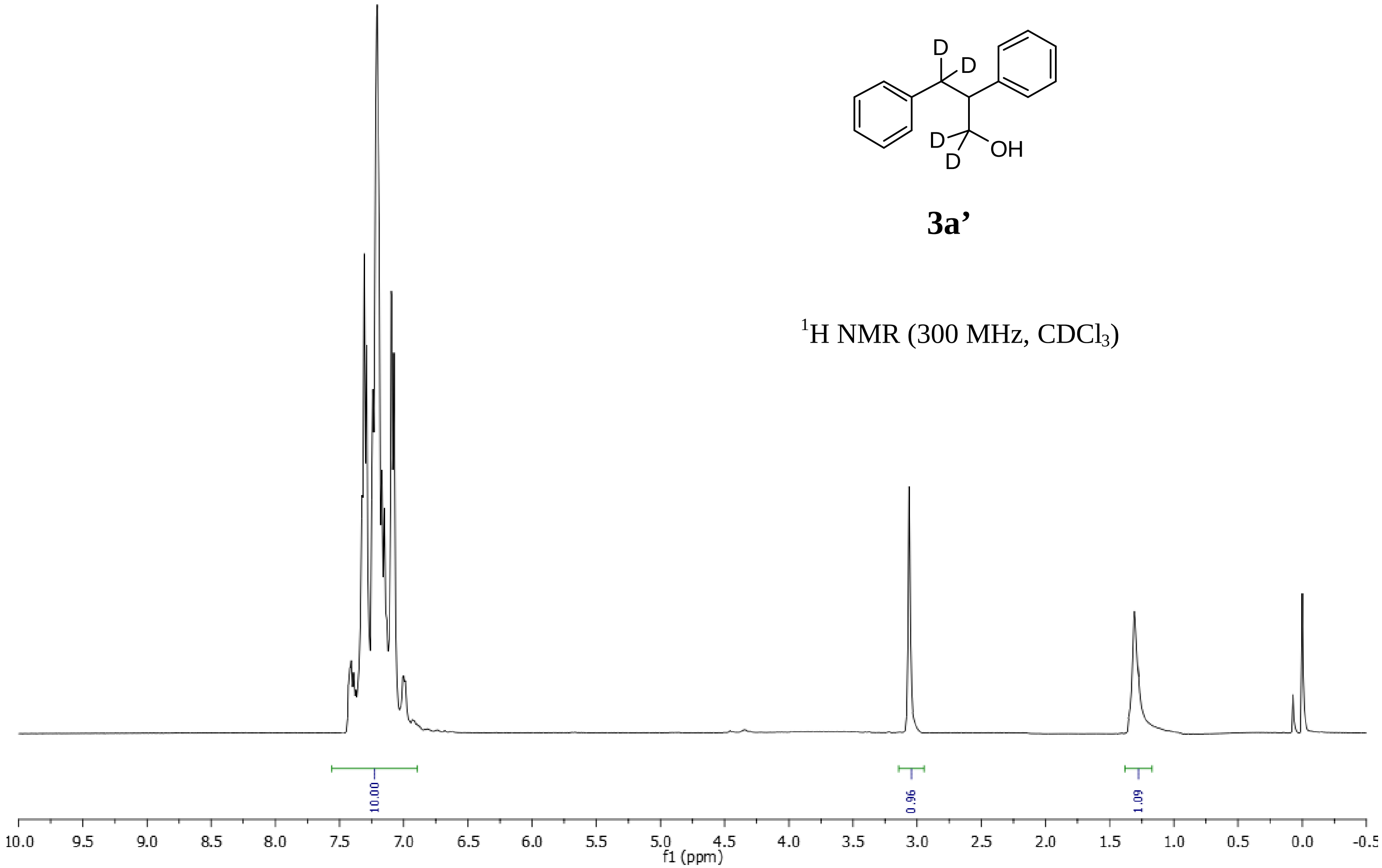
$^1$H NMR (300 MHz, $CDCl_3$)





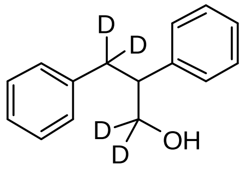
**3s**

$^{13}$C NMR (75 MHz, $CDCl_3$)



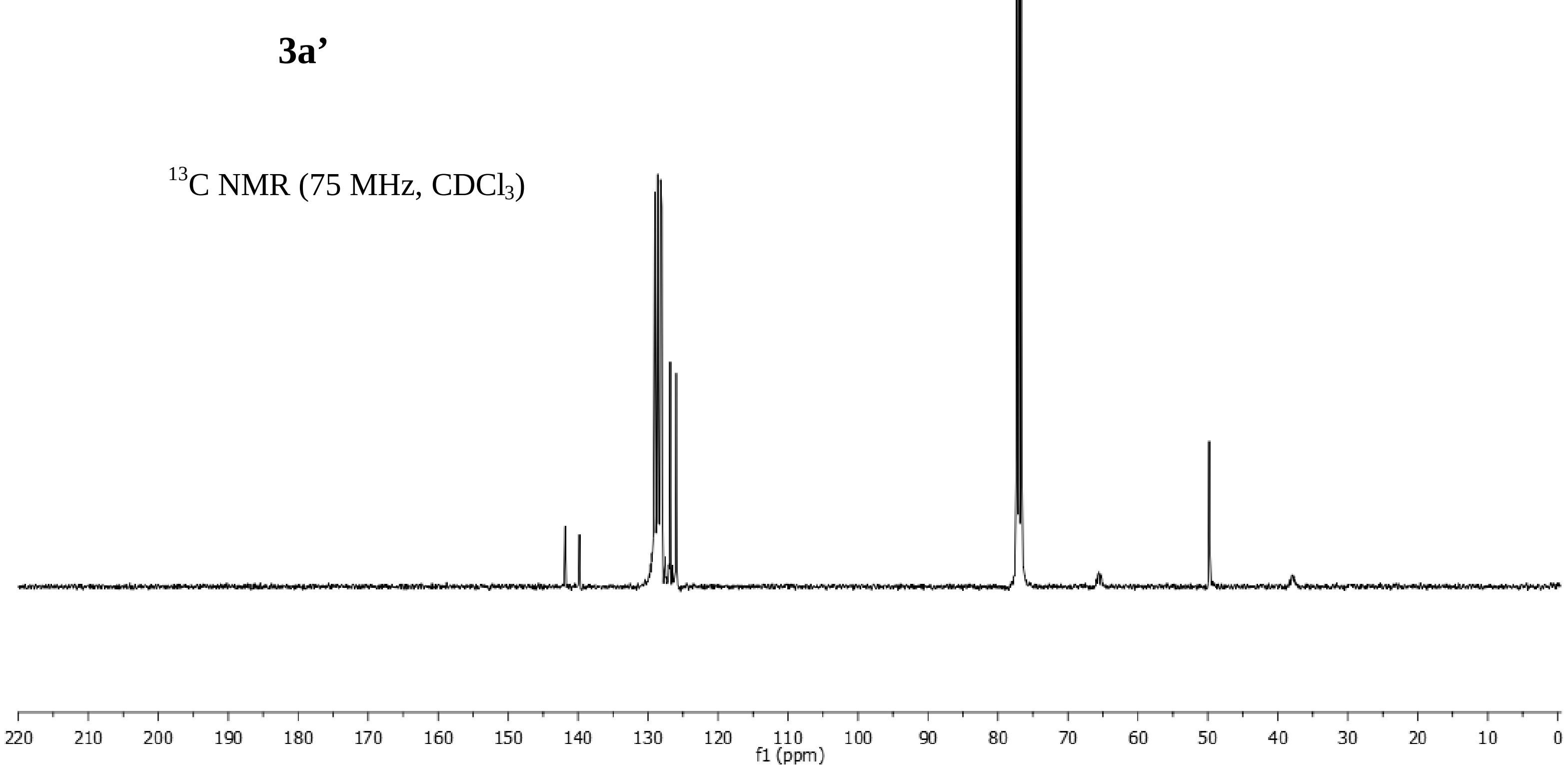
**3a’**

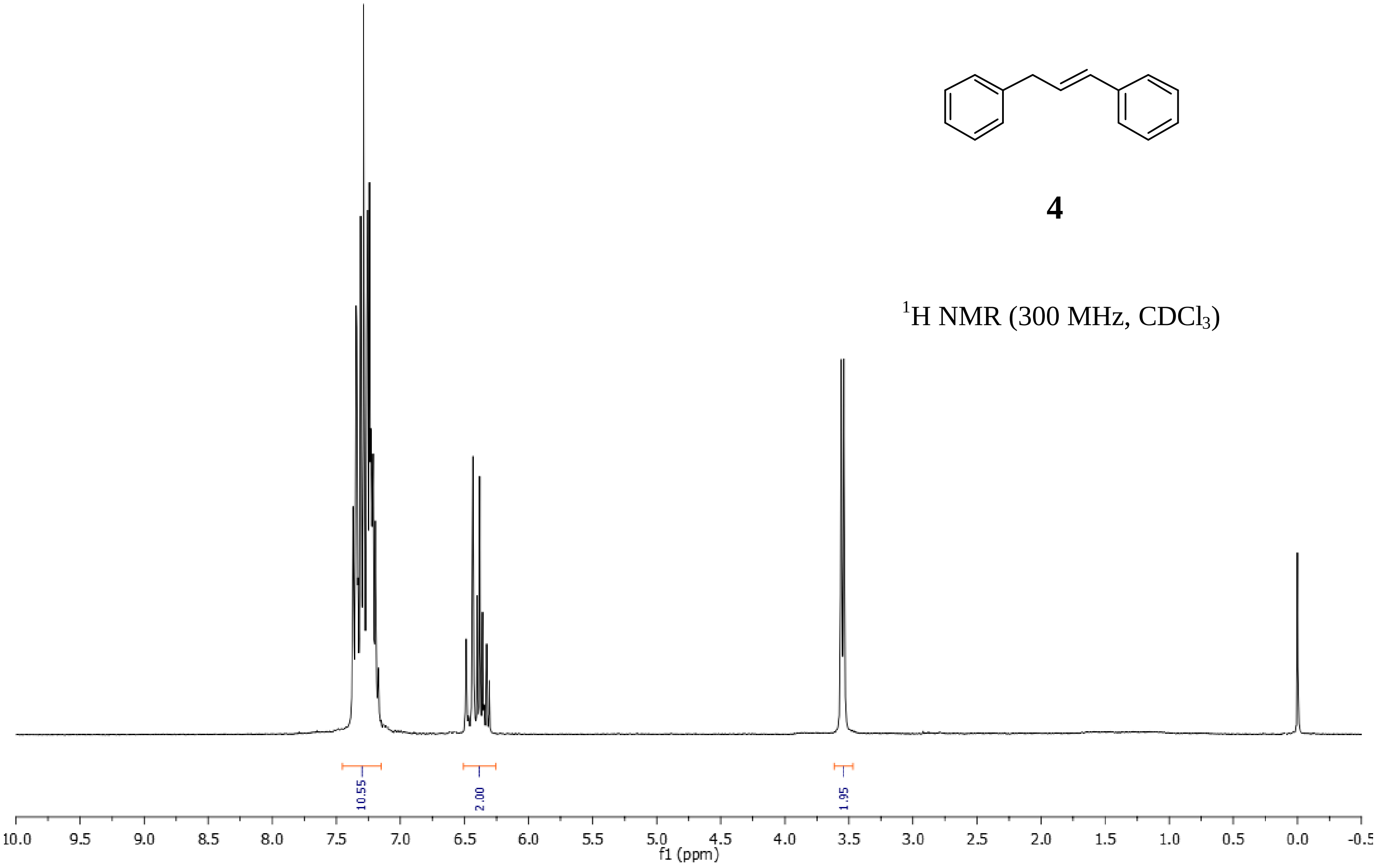
$^1$H NMR (300 MHz, $CDCl_3$)



**3a’**

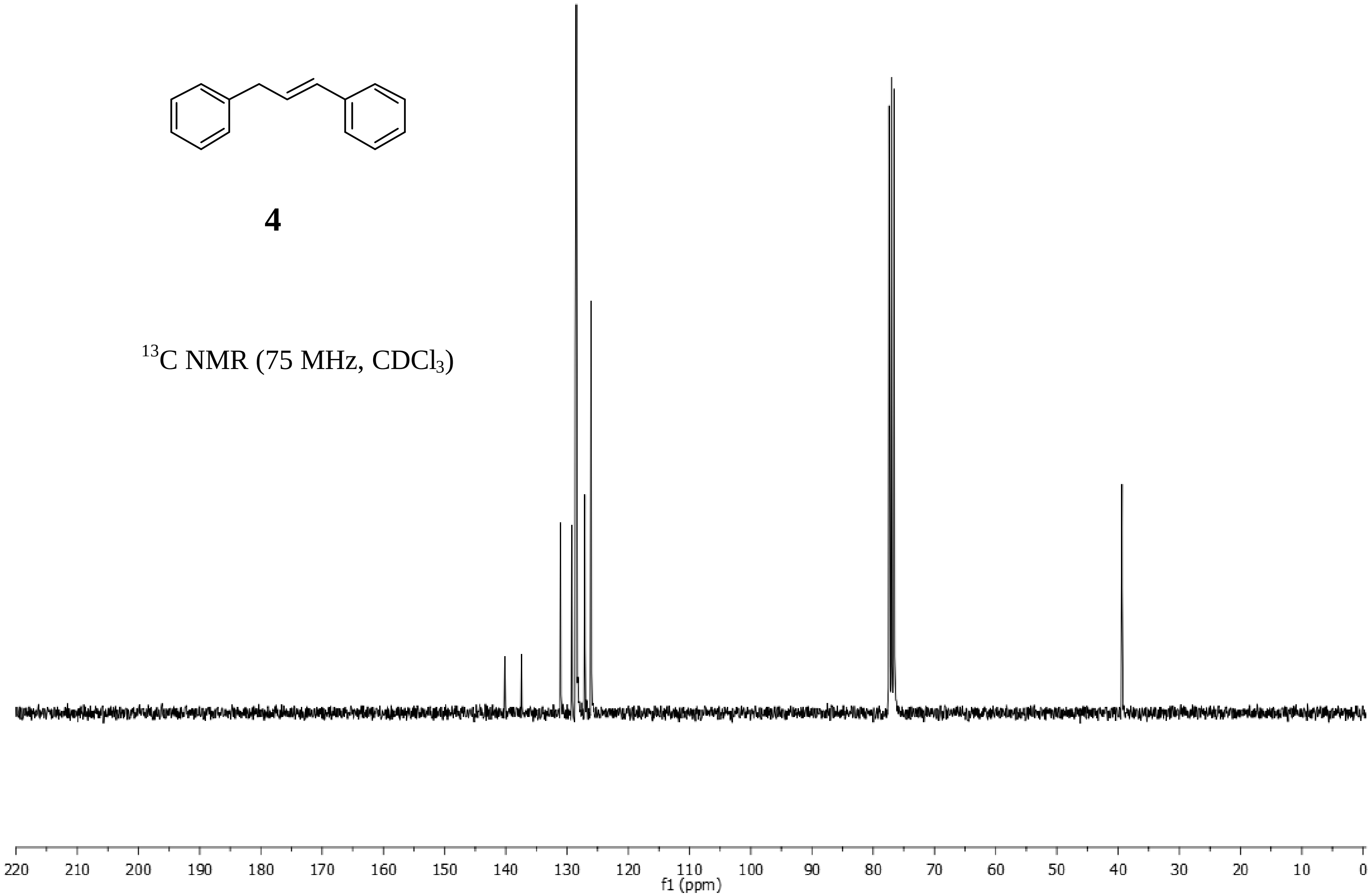
$^{13}$C NMR (75 MHz, $CDCl_3$)





**4**

$^{1}$H NMR (300 MHz, $CDCl_3$)



**4**

$^{13}$C NMR (75 MHz, $CDCl_3$)