

Supporting Information

A Protocol for Hydrogenation of Aldehydes and Ketones to Alcohols in Aqueous Media at Room Temperature in High Yields and Purity

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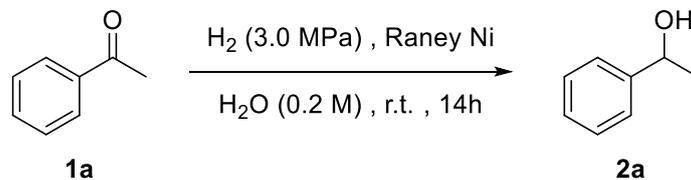
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1. General Information

All reactions were carried out in dried stainless-steel high pressure reactors, which were purchased from Anhui Kemi Instrument Co.,LTD., with built-in magnetic stirring or mechanical stirring paddle, and total volume of 20 ml (Six parallel reactors, dimensions 260 x 260 x 900 mm, wall-thickness of 100 mm), 50 ml (dimensions 550 x 550 x 700 mm, wall-thickness of 250 mm) or 100 ml (dimensions 550 x 550 x 850 mm, wall-thickness of 150 mm). A 50 μ m Raney nickel catalyst was used in this study. All reagents were purchased from commercial sources and used without further purification. Unless otherwise specified, Nuclear magnetic resonance (NMR) spectra were recorded in CDCl₃ or DMSO-*d*₆ on a 500 MHz (for ¹H) spectrometers. All chemical shifts were reported in ppm relative to tetramethylsilane (TMS) (¹H NMR, 0 ppm or ¹H NMR, 2.5 ppm) as the internal standard. The HPLC experiments were carried out on a Waters e2695 instrument (column: J&K, RP-C18, 5 μ m, 4.6 \times 150 mm), and the yields of the products were determined by using the corresponding pure compounds as the external standards. The coupling constants were reported in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet.

2. Optimization of the Reaction Conditions

Table S1. Screening the loading of Raney Ni^a

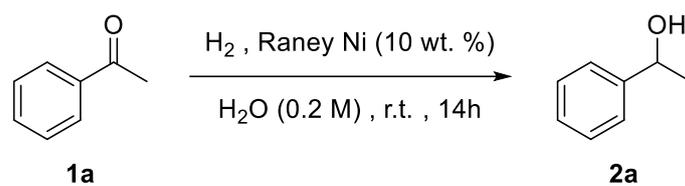


Entry	Raney Ni (wt. %)	Yield ^b /%
1	2	24
2	4	46
3	6	79
4	8	86
5	10	99
6	12	99

^aReaction conditions: acetophenone (**1a**, 120 mg, 1 mmol) and Raney Ni, H₂ (3.0 MPa) and H₂O (5 mL, 0.2 M) was stirred at room temperature for 14 h in dried stainless-steel high pressure reactor, with built-in magnetic stirring, and total volume of 20 ml.

^bThe yield was determined by HPLC using pure **2a** as the external standard ($t_{R,2a} = 5.3$ min, $\lambda_{max} = 209.9$ nm; water / methanol = 50 : 50 (v / v)).

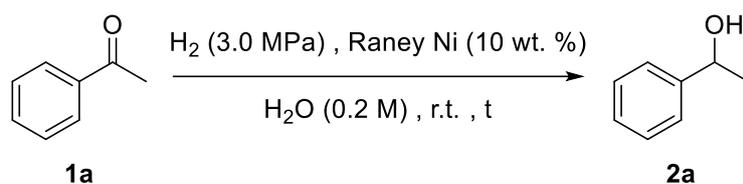
Table S2. Screening the H₂ pressures^a



Entry	H ₂ pressures (MPa)	Yield ^b /%
1	1.0	85
2	2.0	92
3	3.0	99

^aReaction conditions: acetophenone (**1a**, 360 mg, 3 mmol) and Raney Ni (10 wt. %), H₂ and H₂O (15 mL, 0.2 M) was stirred at room temperature for 14 h in dried stainless-steel high pressure reactor, with built-in magnetic stirring, and total volume of 100 ml.

^bThe yield was determined by HPLC using pure **2a** as the external standard ($t_{R,2a} = 5.3$ min, $\lambda_{max} = 209.9$ nm; water / methanol = 50 : 50 (v / v)).

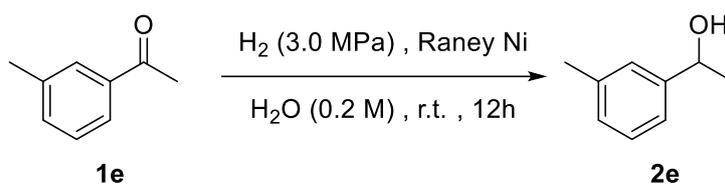
Table S3. Screening the reaction time^a

Entry	Time (h)	Yield ^b /%
1	4	63
2	6	82
3	8	90
4	10	94
5	12	99
6	14	99

^aReaction conditions: acetophenone (**1a**, 360 mg, 3 mmol) and Raney Ni (10 wt. %), H₂ (3.0 MPa) and H₂O (15 mL, 0.2 M) was stirred at room temperature in dried stainless-steel high pressure reactor, with built-in magnetic stirring, and total volume of 100 ml.

^bThe yield was determined by HPLC using pure **2a** as the external standard ($t_{R,2a} = 5.3$ min, $\lambda_{max} = 209.9$ nm; water / methanol = 50 : 50 (v / v)).

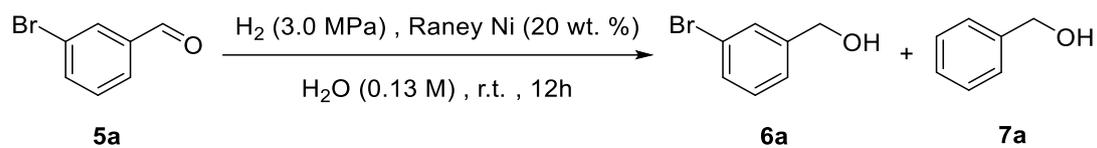
Table S4. further optimization of Raney Ni loading^a



Entry	Raney Ni (wt. %)	Yield ^b /%
1	10	88
2	15	94
3	20	99
4	25	99
5	30	99

^aReaction conditions: 3'-methylacetophenone (**1e**, 134 mg, 1 mmol) and Raney Ni, H₂ (3.0 MPa) and H₂O (5 mL, 0.2 M) was stirred at room temperature for 12 h in dried stainless-steel high pressure reactor, with built-in magnetic stirring, and total volume of 20 ml.

^bThe yield was determined by HPLC using pure **2e** as the external standard ($t_{R,2e} = 4.8$ min, $\lambda_{max} = 209.9$ nm; water / methanol = 40 : 60 (v / v)).

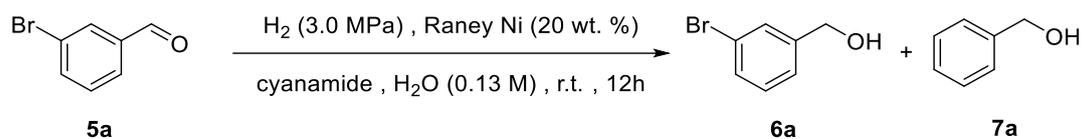
Table S5. Screening the different additives^a

Entry	additives ^b	Ratio [5a : 6a : 7a] ^c
1	Cyanamide	15:82:3
2	Ammonium hydroxide solution	3:48:49
3	Monoethanolamine	0:70:30
4	Thiourea	99:1:0
5	Morpholine	1:63:36
6	none	0:80:20

^aReaction conditions: 3'-Methylacetophenone (**5a**, 134 mg, 1 mmol) and Raney Ni (20 wt. %), additive (4 wt. %), H₂ (3.0 MPa) and H₂O (8 mL, 0.13 M) was stirred at room temperature for 12 h in dried stainless-steel high pressure reactor, with built-in mechanical stirring paddle, and total volume of 50 ml.

^bThe additive dosage is based on the mass ratio of additive to **5a**, and the mass ratio is 4 wt. %.

^cThe yield was determined by HPLC using pure **6a** and **7a** as the external standard ($t_{R,6a}$ = 5.7 min, λ_{max} = 219.3 nm; $t_{R,7a}$ = 3.7 min, λ_{max} = 209.9 nm; water / methanol = 40 : 60 (v / v)).

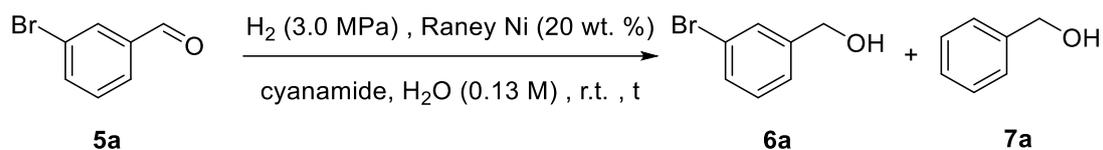
Table S6. Screening the loading of cyanamide^a

Entry	Cyanamide ^b (wt. %)	Ratio [5a : 6a : 7a] ^c
1	2	0:86:14
2	4	15:82:3
3	6	18:82:0

^a Reaction conditions: 3-bromobenzaldehyde (**5a**, 370 mg, 2 mmol), Raney Ni (20 wt. %) and cyanamide, H₂ (3.0 MPa) and H₂O (15 mL, 0.13 M) was stirred at room temperature for 12 h in dried stainless-steel high pressure reactor, with built-in mechanical stirring paddle, and total volume 50 ml.

^b Cyanamide dosage is based on the mass ratio of the cyanamide to **5a**.

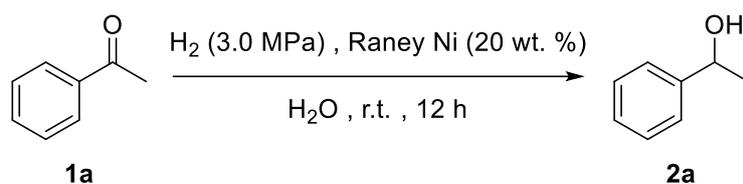
^c The yield was determined by HPLC using pure **6a** and **7a** as the external standard ($t_{R,6a}$ = 5.7 min, λ_{max} = 219.3 nm; $t_{R,7a}$ = 3.7 min, λ_{max} = 209.9 nm; water / methanol = 40 : 60 (v / v)).

Table S7. Screening the reaction time^a

Entry	Time (h)	Ratio [5a : 6a : 7a] ^b
1	12	18:82:0
2	16	4:95:1
3	20	0:83:17

^a Reaction conditions: 3-bromobenzaldehyde (**5a**, 185 mg, 1 mmol) and Raney Ni (20 wt. %), cyanamide (6 wt. %), H₂ (3.0 MPa) and H₂O (8 mL, 0.13 M) was stirred at room temperature in dried stainless-steel high pressure reactor, with built-in mechanical stirring paddle, and total volume of 50 ml.

^b The yield was determined by HPLC using pure **6a** and **7a** as the external standard ($t_{R,6a} = 5.7$ min, $\lambda_{max} = 219.3$ nm; $t_{R,7a} = 3.7$ min, $\lambda_{max} = 209.9$ nm; water / methanol = 40 : 60 (v / v)).

Table S8. The result of Raney Ni recycling^a

Entry	Number of reuses	Yield ^b /%			
		1	2 ^c	3 ^c	4 ^c
1	1	100	99	100	100
2	2	99	100	99	100
3	3	100	100	98	99
4	4	98	99	100	98
5	5	96	98	99	100
6	6	98	100	97	99
7	7	97	98	100	97
8	8	99	97	97	100
9	9	100	96	100	97
10	10	98	95	96	98
11	11	95	90	92	89
12	12	75	74	80	73
13	13	/	55	60	52
14	14	/	44	47	40
15	15	/	30	33	37
16	16	/	24	28	20

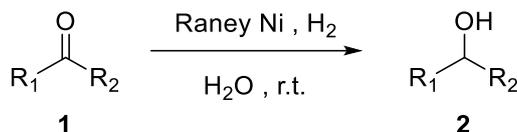
^a Reaction conditions: acetophenone (**1a**, 360 mg, 3 mmol) and Raney Ni (20 wt. %), H₂ (3.0 MPa) and H₂O (15 mL, 0.2 M) was stirred at room temperature in dried stainless-steel high pressure reactor, with built-in magnetic stirring, and total volume of 100 mL.

^b The yield was determined by HPLC using pure **2a** as the external standard ($t_{R,2a} = 5.3$ min, $\lambda_{max} = 209.9$ nm; water / methanol = 50 : 50 (v / v)).

^c Reaction conditions: acetophenone (**1a**, 3g) and Raney Ni (20 wt. %), H₂ (3.0 MPa) and H₂O (9 mL) was stirred at room temperature in dried stainless-steel high pressure reactors, with built-in magnetic stirring, and total volume of 100 mL.

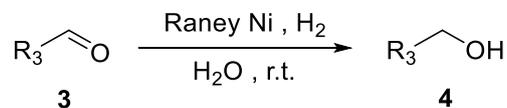
3. Experimental Procedures

3.1 General procedures for synthesis of the compounds 2



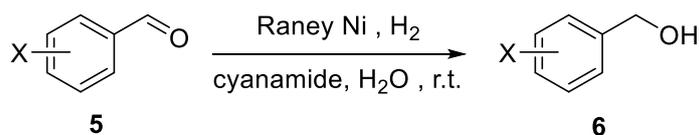
Ketones (3.0 mmol), Raney Ni (10 wt. % or 20 wt. %), H₂ (3.0 MPa), and H₂O (15 ml) were added to a high pressure reactor equipped with a magnetic stirrer, which total volume of 100 ml. Hydrogen was charged into the reactor at the inlet, and the gas was replaced three times. Reaction kettle filled with hydrogen 3.0 MPa, and the mixture was stirred strongly at room temperature for 12 h (aliphatic ketones was stirred strongly at room temperature for 24 h). Then hydrogen in the reactor was released in the fume hood upon completion. After that, the reaction filtered to remove Raney Ni, washed by ethyl acetate. Then the reaction mixture was extracted with ethyl acetate (3×15 mL) and the combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated to dryness to obtain product **2**. Raney Ni was recycled.

3.2 General procedures for synthesis of the compounds 4



Aldehyde (3.0 mmol), Raney Ni (20 wt. %), H₂ (3.0 MPa) and H₂O (15 ml) were added to a high pressure reactor equipped with a magnetic stirrer, which total volume of 100 ml. Hydrogen was charged into the reactor at the inlet, and the gas was replaced three times. Reaction kettle filled with hydrogen 3.0 MPa, and the mixture was stirred strongly at room temperature for 6 h (aliphatic aldehyde was stirred strongly at room temperature for 24 h). Then hydrogen in the reactor was released in the fume hood upon completion. After that, the reaction filtered to remove Raney Ni, washed by ethyl acetate. Then the reaction mixture was extracted with ethyl acetate (3×15 mL) and the combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated to dryness to obtain product **4**. Raney Ni was recycled.

3.3 General procedures for synthesis of the compounds 6



Halogenated aromatic aldehydes (2.0 mmol), Raney Ni (20 wt. %), H₂ (3.0 MPa), cyanamide (6 wt. %) and H₂O (15 ml) were added to a high pressure reactor that was equipped with a mechanical stirring paddle, which total volume of 50 ml. Hydrogen was charged into the reactor at the inlet, and the gas was replaced three times. Reaction kettle filled with hydrogen 3.0 MPa, and the mixture was stirred strongly at room temperature. Then hydrogen in the reactor was released in the fume hood upon completion. After that, the reaction filtered to remove Raney Ni, washed by ethyl acetate. Then the reaction mixture was extracted with ethyl acetate (3×15 mL) and combined organic layers. Product **6** was obtained through column chromatography purification. Starting material **5** and Raney Ni was recycled.

3.4 General procedures for synthesis of the compound 9

Oxcarbazepine (**8**, 3.0 mmol), Raney Ni (20 wt. %), H₂ (4.0 MPa), and H₂O (15 ml) were added to a high pressure reactor equipped with a magnetic stirrer, which total volume of 100 ml. Hydrogen was charged into the reactor at the inlet, and the gas was replaced three times. Reaction kettle filled with hydrogen 4.0 MPa, and the mixture was stirred strongly at 60°C for 48 h. Then hydrogen in the reactor was released in the fume hood upon completion. After that, the reaction filtered to remove Raney Ni, washed by ethyl acetate. Then the reaction mixture was extracted with ethyl acetate (3×15 mL) and the combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated to dryness to obtain product **9**. Raney Ni was recycled.

3.5 General procedures for synthesis of the compound 11

Estrone (**10**, 3.0 mmol), Raney Ni (30 wt. %), H₂ (4.0 MPa), and H₂O (15 ml) were added to a high pressure reactor equipped with a magnetic stirrer, which total volume of 100 ml. Hydrogen was charged into the reactor at the inlet, and the gas was replaced three times. Reaction kettle filled with hydrogen 4.0 MPa, and the mixture was stirred strongly at 60°C for 48 h. Then hydrogen in the reactor was released in the fume hood upon completion. After that, the reaction filtered to remove Raney Ni, washed by ethyl acetate. Then the reaction mixture was extracted with ethyl acetate (3×15 mL) and combined organic layers. Product **11** was obtained through column chromatography purification. Raney Ni was recycled.

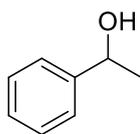
3.6 General procedures for synthesis of the compound 13

Tropinone (**12**, 3.0 mmol), Raney Ni (20 wt. %), H₂ (3.0 MPa), and H₂O (15 ml) were added to a high pressure reactor equipped with a magnetic stirrer, which total volume of 100 ml. Hydrogen was charged into the reactor at the inlet, and the gas was replaced three times. Reaction kettle filled with hydrogen 3.0 MPa, and the mixture was stirred strongly at room temperature for 24 h. Then hydrogen in the reactor was released in the fume hood upon completion. After that, the reaction filtered to remove Raney Ni, washed by ethyl acetate. Then the reaction mixture was extracted with ethyl acetate (3×15 mL) and the combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated to dryness to obtain product **13**. Raney Ni was recycled.

3.7 General procedures for synthesis of the compound 15

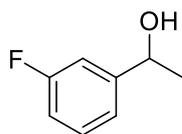
Vanillin (**14**, 3.0 mmol), Raney Ni (20 wt. %), H₂ (3.0 MPa), and H₂O (15 ml) were added to a high pressure reactor equipped with a magnetic stirrer, which total volume of 100 ml. Hydrogen was charged into the reactor at the inlet, and the gas was replaced three times. Reaction kettle filled with hydrogen 3.0 MPa, and the mixture was stirred strongly at room temperature for 12 h. Then hydrogen in the reactor was released in the fume hood upon completion. After that, the reaction filtered to remove Raney Ni, washed by ethyl acetate. Then the reaction mixture was extracted with ethyl acetate (3×15 mL) and combined organic layers. Product **15** was obtained through column chromatography purification. Raney Ni was recycled.

4. Characterization



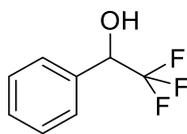
2a

1-phenylethanol (2a). Colorless liquid. (352 mg, 96%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.36-7.24 (m, 5H), 4.87 (q, *J* = 6.5 Hz, 1H), 1.96 (s, 1H), 1.48 (d, *J* = 6.5 Hz, 3H).



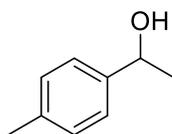
2b

3-fluoro-α-methylbenzenemethanol (2b). Colorless liquid. (407 mg, 97%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.32-7.27 (m, 1H), 7.13-7.08 (m, 2H), 6.97-6.93 (m, 1H), 4.88 (qd, *J*₁ = 6.5 Hz, *J*₂ = 2.5 Hz, 1H), 1.97 (s, 1H), 1.47 (dd, *J*₁ = 6.5 Hz, *J*₂ = 1.5 Hz, 3H).



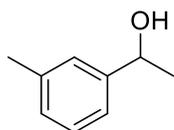
2c

1-phenyl-2,2,2-trifluoroethanol (2c). Colorless liquid. (517 mg, 98%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.46-7.45 (m, 2H), 7.41-7.39 (m, 3H), 5.01-4.96 (m, 1H), 2.88 (d, *J* = 4.0 Hz, 1H).



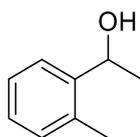
2d

1-(4-methylphenyl)ethanol (2d). Colorless liquid. (392 mg, 96%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.24 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 4.84 (q, *J* = 6.5 Hz, 1H), 2.33 (s, 3H), 1.93 (s, 1H), 1.46 (d, *J* = 6.5 Hz, 3H).



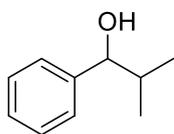
2e

1-(3-methylphenyl)ethanol (2e). Colorless liquid. (396 mg, 97%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.23 (t, $J = 7.0$ Hz, 1H), 7.18-7.14 (m, 2H), 7.08 (d, $J = 7.5$ Hz, 1H), 4.84 (q, $J = 6.5$ Hz, 1H), 2.35 (s, 3H), 1.92 (s, 1H), 1.48 (d, $J = 6.5$ Hz, 3H).



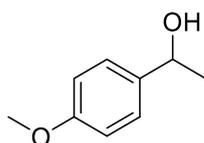
2f

1-(2-methylphenyl)ethanol (2f). Colorless liquid. (388 mg, 95%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.44 (d, $J = 7.5$ Hz, 1H), 7.19-7.16 (m, 1H), 7.13-7.07 (m, 2H), 5.01 (q, $J = 6.0$ Hz, 1H), 2.49 (s, 1H), 2.27 (s, 3H), 1.38 (d, $J = 6.5$ Hz, 3H).



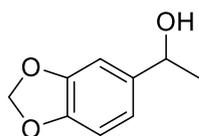
2g

2-methyl-1-phenyl-1-propanol (2g). Colorless liquid. (388 mg, 95%). The NMR data is identical to that reported in literature.² **¹H NMR (500 MHz, CDCl₃)** δ 7.34-7.24 (m, 5H), 4.34 (d, $J = 7.0$ Hz, 1H), 1.98-1.90 (m, 2H), 0.99 (d, $J = 6.5$ Hz, 3H), 0.79 (d, $J = 7.0$ Hz, 3H).



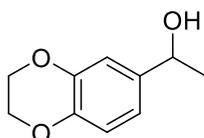
2h

1-(4-methoxyphenyl)ethanol (2h). Colorless oil. (447 mg, 98%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.29-7.26 (m, 2H), 6.87-6.85 (m, 2H), 4.82 (q, $J = 6.5$ Hz, 1H), 3.78 (s, 3H), 2.05 (s, 1H), 1.45 (d, $J = 6.0$ Hz, 3H).



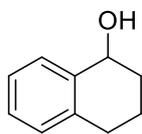
2i

1-(benzo[d][1,3]dioxol-5-yl)ethanol (2i). Colorless oil. (478 mg, 96%). The NMR data is identical to that reported in literature.³ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 6.88 (s, 1H), 6.81-6.75 (m, 2H), 5.93 (s, 2H), 4.81 (q, $J = 6.5$ Hz, 1H), 1.89 (s, 1H), 1.45 (d, $J = 6.5$ Hz, 3H).



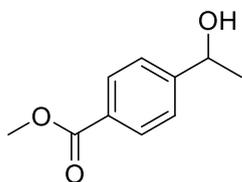
2j

1-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)ethanol (2j). Colorless oil. (478 mg, 96%). The NMR data is identical to that reported in literature.⁴ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 6.87 (s, 1H), 6.83-6.80 (m, 2H), 4.76 (q, $J = 6.5$ Hz, 1H), 4.22 (s, 4H), 2.13 (s, 1H), 1.43 (d, $J = 6.5$ Hz, 3H).



2k

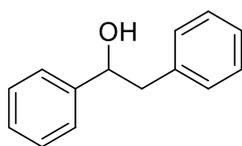
1,2,3,4-tetrahydro-1-naphthol (2k). clear liquid. (440 mg, 99%). The NMR data is identical to that reported in literature.⁵ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.39-7.36 (m, 1H), 7.20-7.14 (m, 2H), 7.07-7.04 (m, 1H), 4.69 (t, $J = 4.0$ Hz, 1H), 2.80-2.64 (m, 2H), 2.24 (s, 1H), 1.96-1.69 (m, 4H).



2l

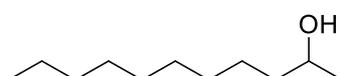
4-(methoxycarbonyl)-alpha-methylbenzyl alcohol (2l). white solid. (524 mg, 97%). The NMR data is identical to that reported in literature.⁶ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.99 (d, $J = 7.0$ Hz, 2H), 7.42 (d, $J = 7.0$ Hz, 2H), 4.94 (q, $J = 6.0$ Hz, 1H), 3.90 (s, 3H),

2.41 (s, 1H), 1.49 (d, $J = 6.5$ Hz, 3H).



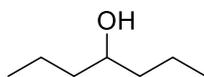
2m

1,2-diphenylethanol alcohol (2m). white solid. (588 mg, 99%). The NMR data is identical to that reported in literature.⁵ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.31-7.17 (m, 8H), 7.15 (d, $J = 7.5$ Hz, 2H), 4.83 (dd, $J_1 = 8.0$ Hz, $J_2 = 5.0$ Hz, 1H), 3.02-2.93 (m, 2H), 2.11 (s, 1H).



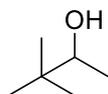
2n

2-undecanol (2n). clear liquid. (491 mg, 95%). The NMR data is identical to that reported in literature.⁷ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 3.81-3.75 (m, 1H), 1.56 (s, 1H), 1.50-1.39 (m, 3H), 1.28-1.26 (m, 13H), 1.18 (d, $J = 6.5$ Hz, 3H), 0.88 (t, $J = 7.0$ Hz, 3H).



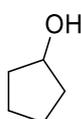
2o

4-heptanol (2o). pale yellow liquid. (271 mg, 78%). The NMR data is identical to that reported in literature.⁸ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 3.63-3.58 (m, 1H), 1.69 (s, 1H), 1.50-1.31 (m, 8H), 0.93 (t, $J = 7.5$ Hz, 6H).



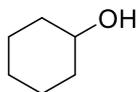
2p

3,3-dimethyl-2-butanol (2p). colorless liquid. (208 mg, 68%). The NMR data is identical to that reported in literature.⁵ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 3.49-3.45 (m, 1H), 1.73 (s, 1H), 1.11 (d, $J = 6.5$ Hz, 3H), 0.89 (s, 9H).



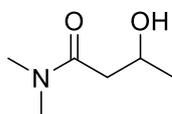
2q

cyclopentanol (2q). colorless liquid. (180 mg, 70%). The NMR data is identical to that reported in literature.⁹ **¹H NMR (500 MHz, CDCl₃)** δ 4.33 (s, 1H), 1.76-1.70 (m, 5H), 1.60-1.56 (m, 4H).



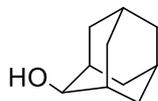
2r

cyclohexanol (2r). colorless liquid. (204 mg, 68%). The NMR data is identical to that reported in literature.⁶ **¹H NMR (500 MHz, CDCl₃)** δ 3.61-3.57 (m, 1H), 2.53 (s, 1H), 1.89-1.86 (m, 2H), 1.74-1.71 (m, 2H), 1.56-1.53 (m, 1H), 1.29-1.15 (m, 5H).



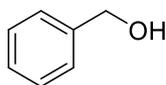
2s

3-hydroxy-N,N-dimethylbutanamide (2s). colorless liquid. (373 mg, 95%). The NMR data is identical to that reported in literature.¹⁰ **¹H NMR (500 MHz, CDCl₃)** δ 4.44 (s, 1H), 4.22-4.16 (m, 1H), 3.00 (s, 3H), 2.96 (s, 3H), 2.50-2.46 (m, 1H), 2.34-2.28 (m, 1H), 1.22 (d, *J* = 6.5 Hz, 3H).



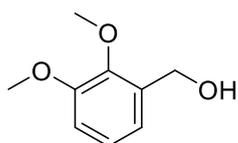
2t

2-adamantanol (2t). white crystalline powder. (433 mg, 95%). The NMR data is identical to that reported in literature.⁹ **¹H NMR (500 MHz, CDCl₃)** δ 3.87 (s, 1H), 2.07 (d, *J* = 12.5 Hz, 2H), 1.88-1.80 (m, 6H), 1.71-1.68 (m, 5H), 1.52 (d, *J* = 13.0 Hz, 2H).



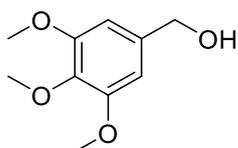
4a

benzyl alcohol (4a). colorless liquid. (321 mg, 99%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.33-7.24 (m, 5H), 4.58 (s, 2H), 2.89 (s, 1H).



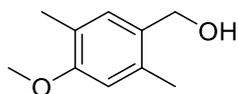
4b

2,3-dimethoxybenzyl alcohol (4b). white crystalline solid. (494 mg, 98%). The NMR data is identical to that reported in literature.¹¹ **¹H NMR (500 MHz, CDCl₃)** δ 7.04 (t, $J = 7.5$ Hz, 1H), 6.92 (d, $J = 7.5$ Hz, 1H), 6.87 (d, $J = 8.0$ Hz, 1H), 4.68 (s, 2H), 3.87 (d, $J = 7.0$ Hz, 6H), 2.38 (s, 1H).



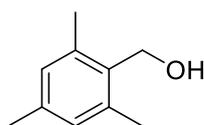
4c

(3,4,5-trimethoxyphenyl)methanol (4c). yellow oil. (588 mg, 99%). The NMR data is identical to that reported in literature.¹ **¹H NMR (500 MHz, CDCl₃)** δ 6.58 (s, 2H), 4.60 (s, 2H), 3.83 (d, $J = 9.5$ Hz, 9H), 2.45 (s, 1H).



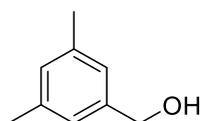
4d

2,5-dimethyl-4-methoxybenzyl alcohol (4d). colorless liquid. (473 mg, 95%). The NMR data is identical to that reported in literature.¹² **¹H NMR (500 MHz, CDCl₃)** δ 7.03 (s, 1H), 6.63 (s, 1H), 4.53 (s, 2H), 3.79 (s, 3H), 2.31 (s, 3H), 2.16 (s, 3H), 1.95 (s, 1H).



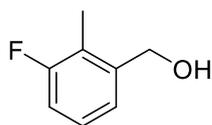
4e

2,4,6-trimethylbenzyl alcohol (4e). colorless liquid. (441 mg, 98%). The NMR data is identical to that reported in literature.¹³ **¹H NMR (500 MHz, CDCl₃)** δ 6.85 (s, 2H), 4.66 (s, 2H), 2.37 (s, 6H), 2.25 (s, 3H), 1.43 (s, 1H).



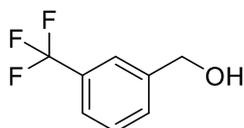
4f

3,5-dimethylbenzyl alcohol (4f). colorless liquid. (400 mg, 98%). The NMR data is identical to that reported in literature.¹³ ¹H NMR (500 MHz, CDCl₃) δ 6.95 (s, 2H), 6.91 (s, 1H), 4.57 (s, 2H), 2.30 (s, 6H), 2.17 (s, 1H).



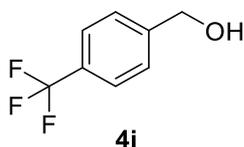
4g

3-fluoro-2-methyl-benzenemethanol (4g). colorless liquid. (400 mg, 95%). The NMR data is identical to that reported in literature.¹⁴ ¹H NMR (500 MHz, CDCl₃) δ 7.13-7.11 (m, 2H), 6.97-6.93 (m, 1H), 4.63 (s, 2H), 2.22 (d, *J* = 2.0 Hz, 3H).



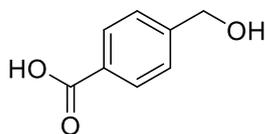
4h

3-(trifluoromethyl)benzyl alcohol (4h). clear liquid. (507 mg, 96%). The NMR data is identical to that reported in literature.¹⁵ ¹H NMR (500 MHz, CDCl₃) δ 7.59 (s, 1H), 7.53-7.42 (m, 3H), 4.68 (s, 2H), 2.71 (s, 1H).



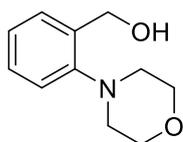
4i

4-(trifluoromethyl)benzyl alcohol (4i). colorless liquid. (512 mg, 97%). The NMR data is identical to that reported in literature.¹ ¹H NMR (500 MHz, CDCl₃) δ 7.59 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 7.5 Hz, 2H), 4.70 (s, 2H), 2.52 (s, 1H).



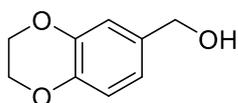
4j

4-(hydroxymethyl)benzoic acid (4j). white coarse fibers. (433 mg, 95%). The NMR data is identical to that reported in literature.¹⁵ ¹H NMR (500 MHz, DMSO-*d*₆) δ 12.84 (s, 1H), 7.91 (d, *J* = 8.5 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 5.33 (s, 1H), 4.58 (s, 2H).



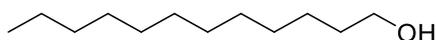
4k

(2-morpholinophenyl)methanol (**4k**). white powder. (556 mg, 96%). The NMR data is identical to that reported in literature.¹⁶ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.28 (t, $J = 7.5$ Hz, 1H), 7.23 (d, $J = 7.0$ Hz, 1H), 7.18 (d, $J = 8.0$ Hz, 1H), 7.12 (t, $J = 7.5$ Hz, 1H), 4.94 (s, 1H), 4.79 (s, 2H), 3.85 (t, $J = 4.5$ Hz, 4H), 2.97 (t, $J = 4.5$ Hz, 4H).



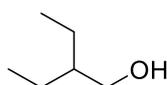
4l

2,3-dihydro-1,4-benzodioxin-6-methanol (**4l**). brown oil. (483 mg, 97%). The NMR data is identical to that reported in literature.¹⁷ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 6.83-6.77 (m, 3H), 4.49 (d, $J = 4.0$ Hz, 2H), 4.20 (d, $J = 3.5$ Hz, 4H), 2.44 (s, 1H).



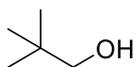
4m

1-dodecanol (**4m**). colorless liquid. (547 mg, 98%). The NMR data is identical to that reported in literature.¹⁸ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 3.61 (t, $J = 6.5$ Hz, 2H), 2.25 (s, 1H), 1.58-1.53 (m, 2H), 1.33-1.26 (m, 18H), 0.88 (t, $J = 6.5$ Hz, 3H).



4n

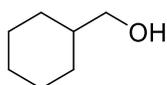
2-ethyl-1-butanol (**4n**). clear liquid. (459 mg, 75%). The NMR data is identical to that reported in literature.¹⁹ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 3.48 (d, $J = 4.5$ Hz, 2H), 2.02 (s, 1H), 1.35-1.23 (m, 5H), 0.87-0.81 (m, 6H).



4o

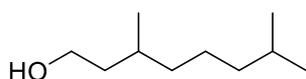
neopentyl alcohol (**4o**). colorless waxy crystalline solid. (317 mg, 60%). The NMR data is identical to that reported in literature.²⁰ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 3.29 (s, 2H),

2.41 (s, 1H), 0.91 (s, 9H).



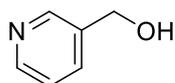
4p

cyclohexanemethanol (4p). colorless oil. (582 mg, 85%). The NMR data is identical to that reported in literature.⁶ ¹H NMR (500 MHz, CDCl₃) δ 3.35 (d, *J* = 6.5 Hz, 2H), 2.31 (s, 1H), 1.69-1.58 (m, 5H), 1.44-1.36 (m, 1H), 1.20-1.05 (m, 3H), 0.90-0.82 (m, 2H).



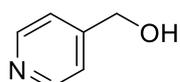
4q

3,7-dimethyl-1-octanol (4q). colorless liquid. (441 mg, 93%). The NMR data is identical to that reported in literature.²¹ ¹H NMR (500 MHz, CDCl₃) δ 3.71-3.62 (m, 2H), 1.81 (s, 1H), 1.68-1.08 (m, 11H), 0.91-0.86 (m, 8H).



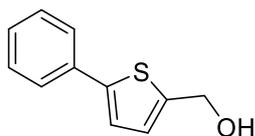
4r

3-pyridinemethanol (4r). light yellow liquid. (320 mg, 98%). The NMR data is identical to that reported in literature.¹³ ¹H NMR (500 MHz, CDCl₃) δ 7.19-7.17 (m, 1H), 7.08-7.05 (m, 1H), 6.45-6.43 (m, 1H), 5.99-5.96 (m, 1H), 5.00 (s, 1H), 3.42-3.38 (m, 2H).



4s

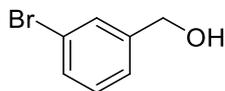
4-pyridinemethanol (4s). white crystalline powder. (320 mg, 98%). The NMR data is identical to that reported in literature.²² ¹H NMR (500 MHz, CDCl₃) δ 8.54-8.44 (m, 2H), 7.30-7.28 (m, 2H), 4.73 (s, 2H), 3.84 (s, 1H).



4t

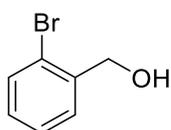
(5-phenyl-2-thienyl)methanol (4t). white crystalline powder. (542 mg, 95%). The NMR

data is identical to that reported in literature.²³ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.57 (d, $J = 7.5$ Hz, 2H), 7.38-7.34 (m, 2H), 7.28-7.24 (m, 1H), 7.16 (d, $J = 3.5$ Hz, 1H), 6.96 (d, $J = 3.5$ Hz, 1H), 4.80 (s, 2H), 2.03 (s, 1H).



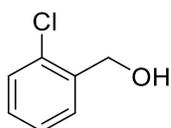
6a

3-bromobenzyl alcohol (6a). light yellow liquid. (325 mg, 87%). The NMR data is identical to that reported in literature.⁶ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.52 (s, 1H), 7.41 (d, $J = 8.0$ Hz, 1H), 7.27 (d, $J = 7.5$ Hz, 1H), 7.22 (t, $J = 8.0$ Hz, 1H), 4.66 (s, 2H), 1.95 (s, 1H).



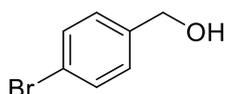
6b

2-bromobenzyl alcohol (6b). white solid. (299 mg, 80%). The NMR data is identical to that reported in literature.¹⁶ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.55 (d, $J = 8.0$ Hz, 1H), 7.49 (d, $J = 7.5$ Hz, 1H), 7.34 (t, $J = 7.5$ Hz, 1H), 7.16 (td, $J_1 = 8.0$ Hz, $J_2 = 2.0$ Hz, 1H), 4.76 (s, 2H), 2.01 (s, 1H).



6c

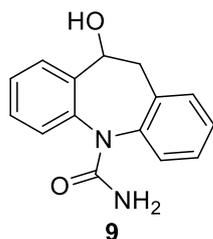
2-chlorobenzyl alcohol (6c). white crystalline powder. (242 mg, 85%). The NMR data is identical to that reported in literature.⁶ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.48 (dd, $J_1 = 7.5$ Hz, $J_2 = 1.5$ Hz, 1H), 7.36 (dd, $J_1 = 8.0$ Hz, $J_2 = 1.0$ Hz, 1H), 7.30-7.22 (m, 2H), 4.79 (s, 2H), 1.98 (s, 1H).



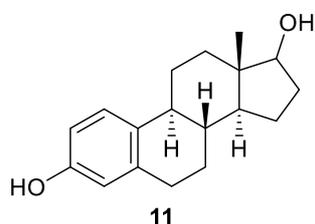
6d

4-bromobenzyl alcohol (6d). white crystalline powder. (261 mg, 70%). The NMR data is identical to that reported in literature.⁶ $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.48 (d, $J =$

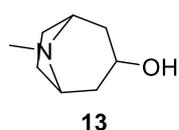
8.5 Hz, 2H), 7.23 (d, $J = 8.5$ Hz, 2H), 4.65 (s, 2H), 1.77 (s, 1H).



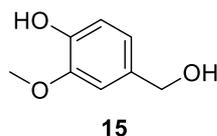
Licarbazepine (**9**). Light yellow solid powder. (719 mg, 95%). The NMR data is identical to that reported in literature.²⁴ **¹H NMR (500 MHz, DMSO-*d*₆)** δ 7.51 (d, $J = 7.0$ Hz, 1H), 7.33-7.28 (m, 3H), 7.25-7.18 (m, 4H), 5.73 (d, $J = 7.0$ Hz, 2H), 5.63 (s, 1H), 5.10 (s, 1H), 3.32 (s, 2H).



β -Estradiol (**11**). White powder. (694 mg, 85%). The NMR data is identical to that reported in literature.²³ **¹H NMR (500 MHz, CDCl₃)** δ 8.95 (s, 1H), 7.03 (d, $J = 8.5$ Hz, 1H), 6.50 (dd, $J_1 = 8.5$ Hz, $J_2 = 2.0$ Hz, 1H), 6.43 (s, 1H), 4.47 (d, $J = 4.5$ Hz, 1H), 3.53-3.49 (m, 1H), 2.75-2.65 (m, 2H), 2.24-2.20 (m, 1H), 2.08-2.03 (m, 1H), 1.88-1.74 (m, 2H), 1.60-1.54 (m, 1H), 1.40-1.06 (m, 8H), 0.66 (s, 3H).



Tropine (**13**). white crystalline powder. (415 mg, 98%). The NMR data is identical to that reported in literature.²⁵ **¹H NMR (500 MHz, CDCl₃)** δ 4.24 (s, 1H), 3.77 (s, 1H), 2.91 (s, 2H), 2.11 (s, 3H), 2.01-1.98 (m, 2H), 1.86-1.80 (m, 4H), 1.52 (d, $J = 13.5$ Hz, 2H).



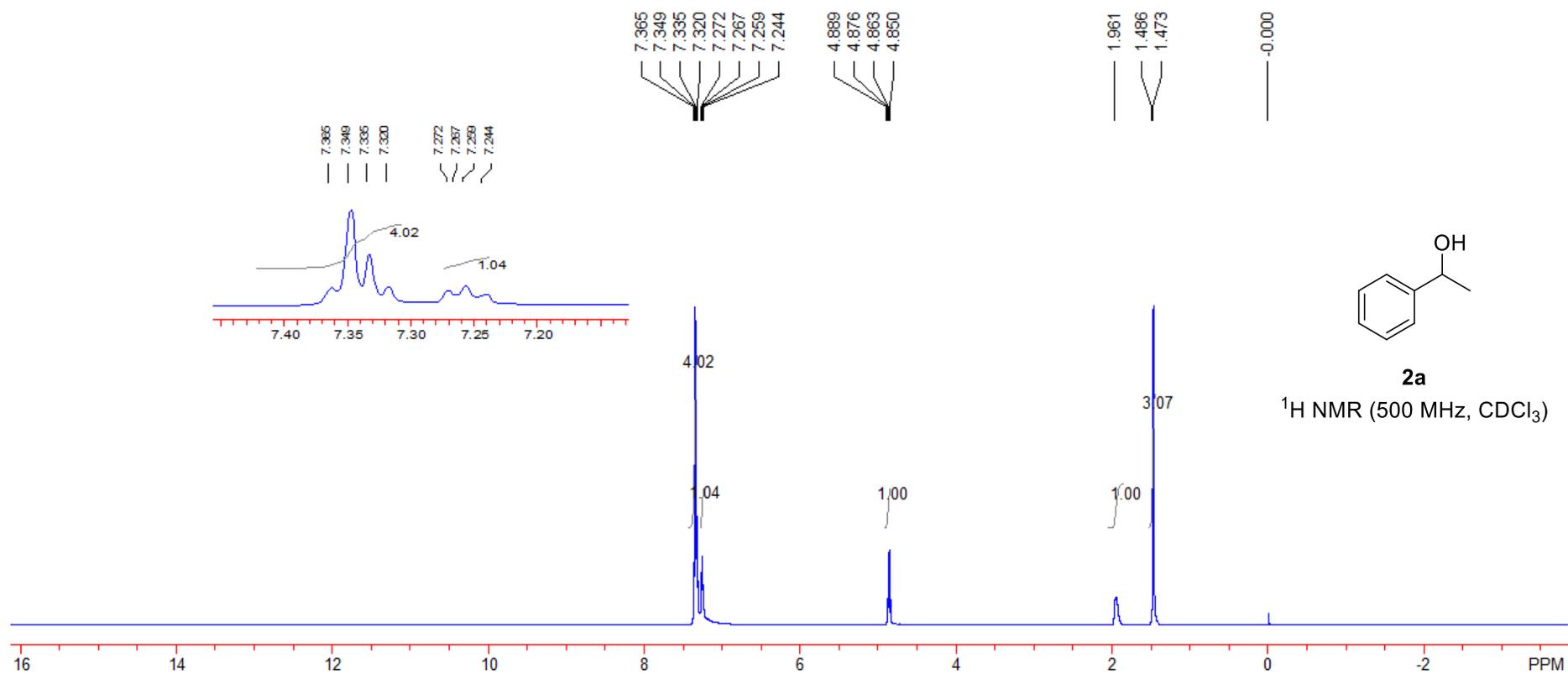
Vanillyl alcohol (**15**). white crystalline powder. (416 mg, 90%). The NMR data is identical to that reported in literature.²⁶ **¹H NMR (500 MHz, CDCl₃)** δ 8.73 (s, 1H), 6.88 (s, 1H), 6.70 (t, $J = 8.5$ Hz, 2H), 4.97 (t, $J = 5.5$ Hz, 1H), 4.37 (d, $J = 5.5$ Hz, 2H), 3.75 (s, 3H).

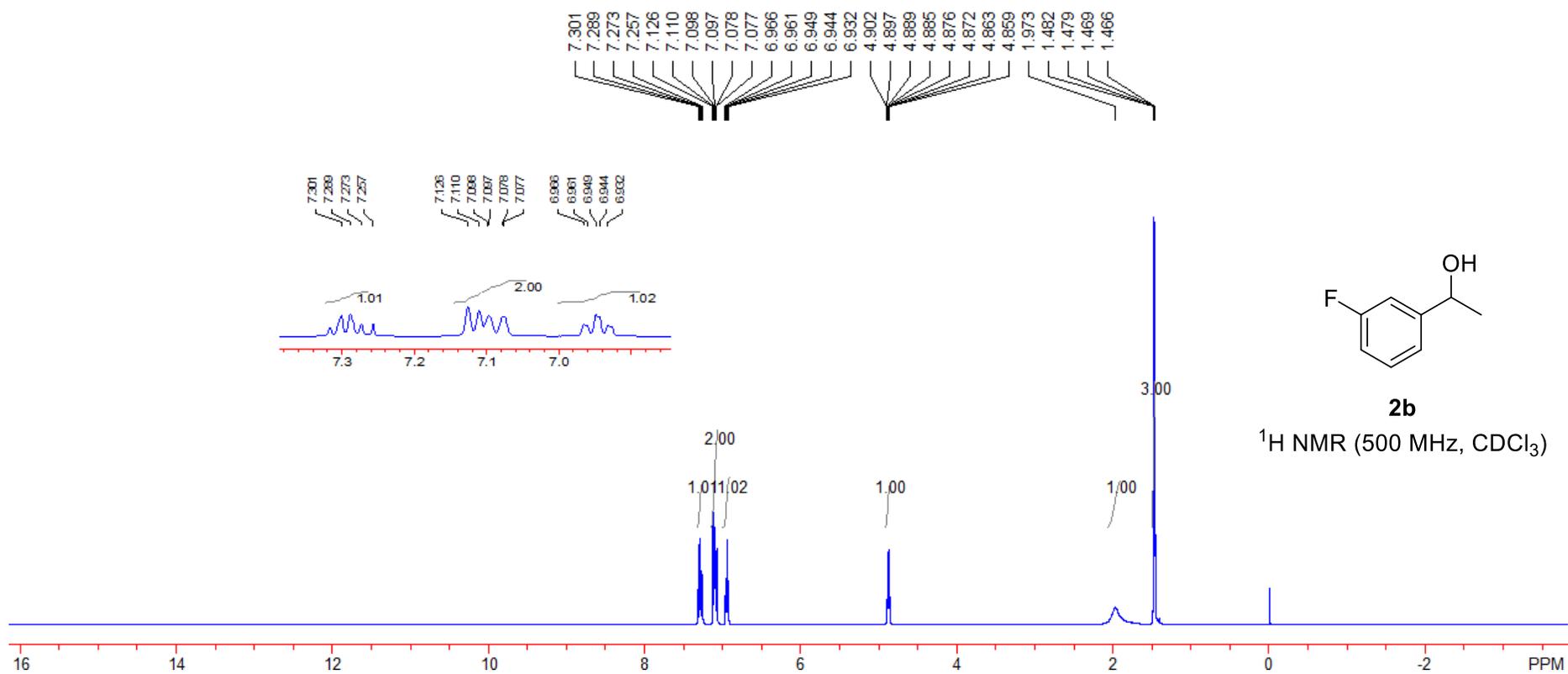
5. References

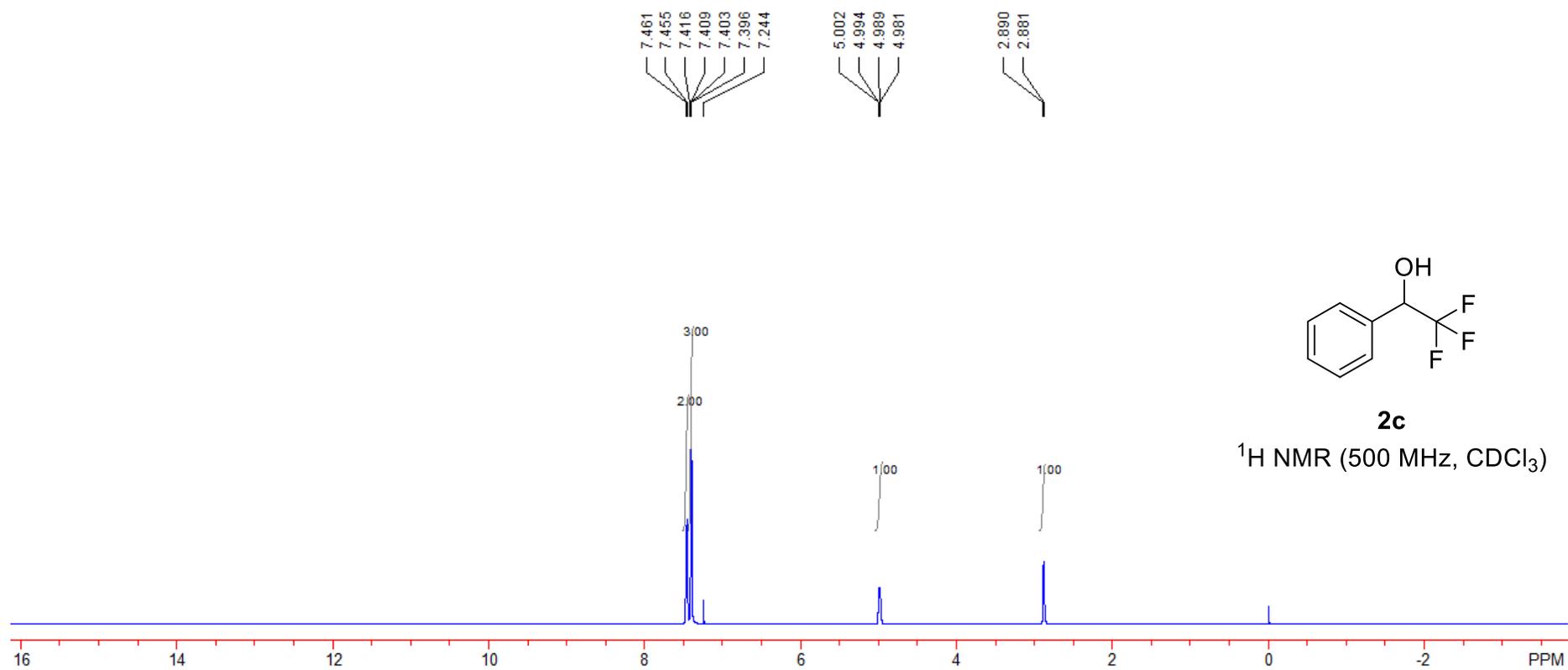
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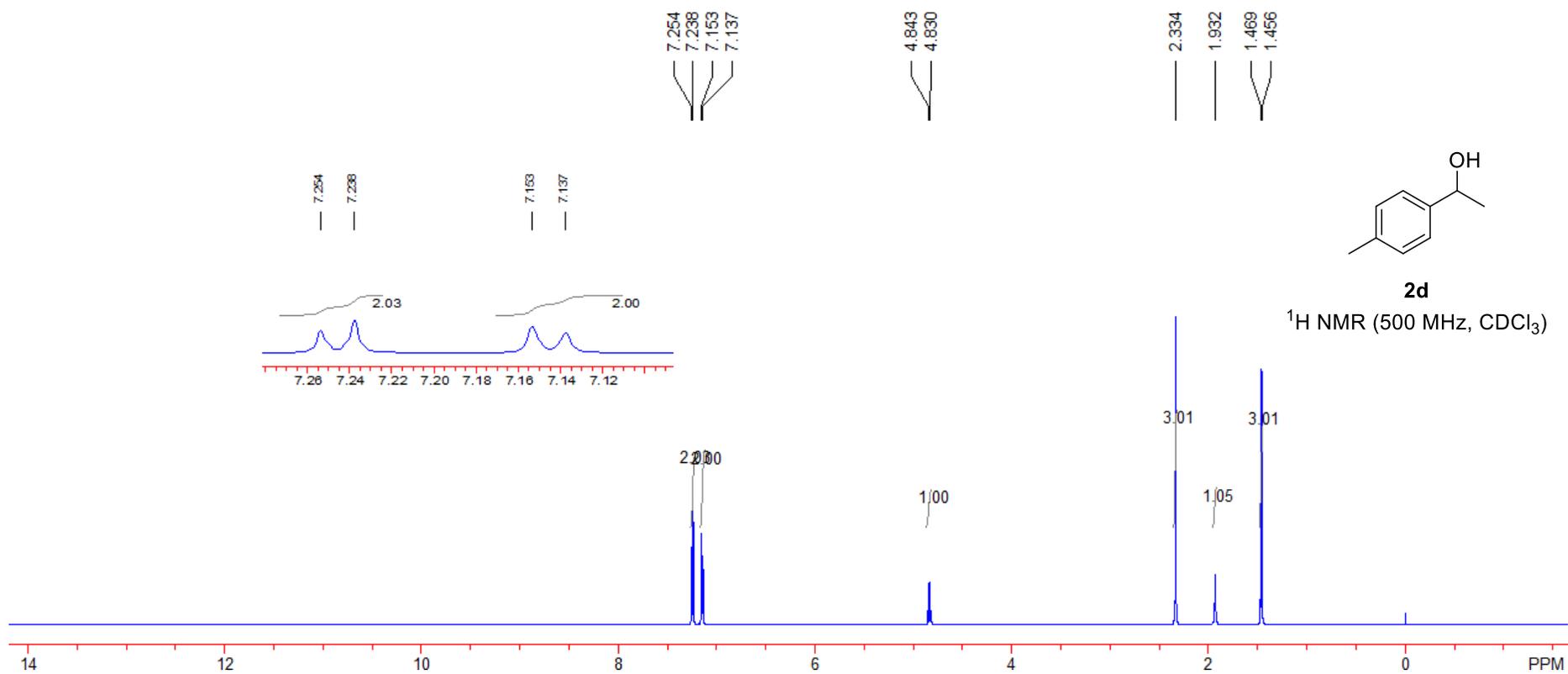
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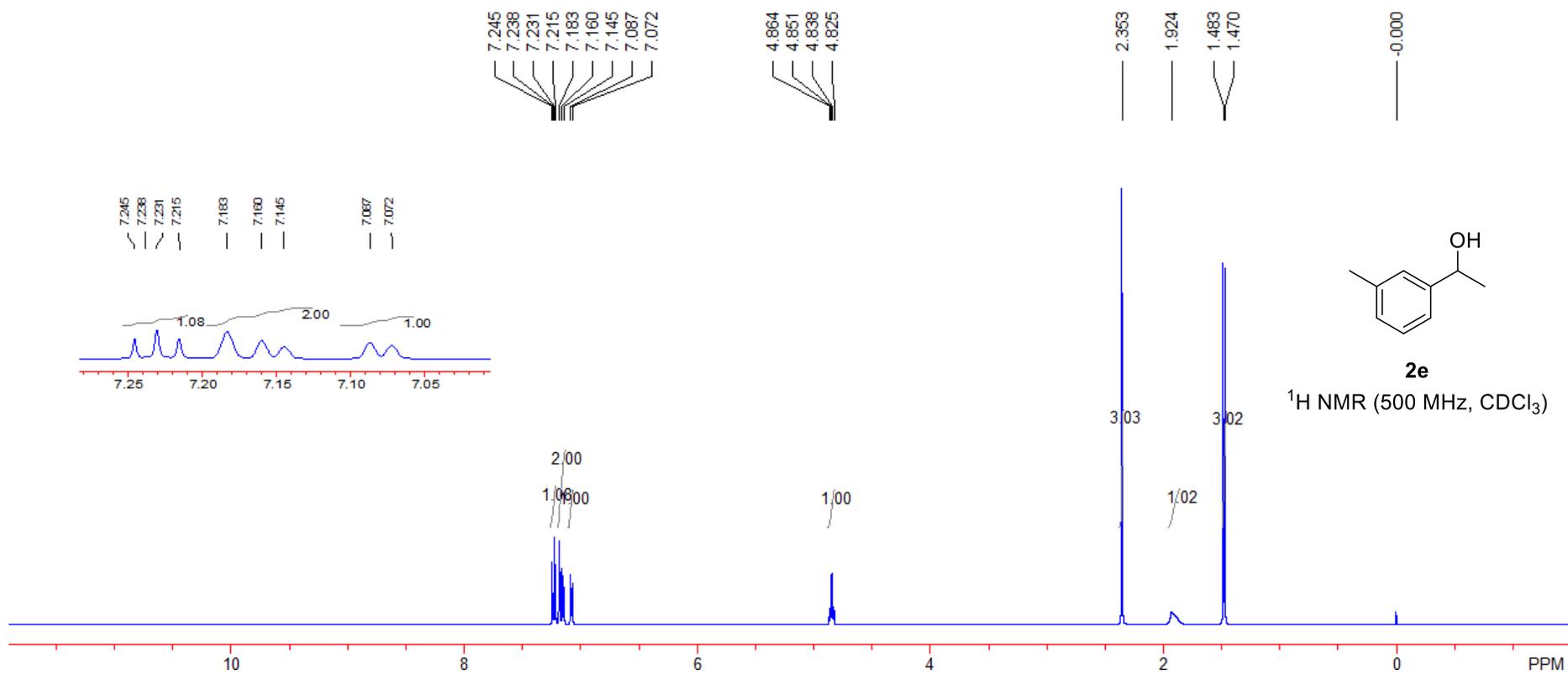
6. NMR Spectra

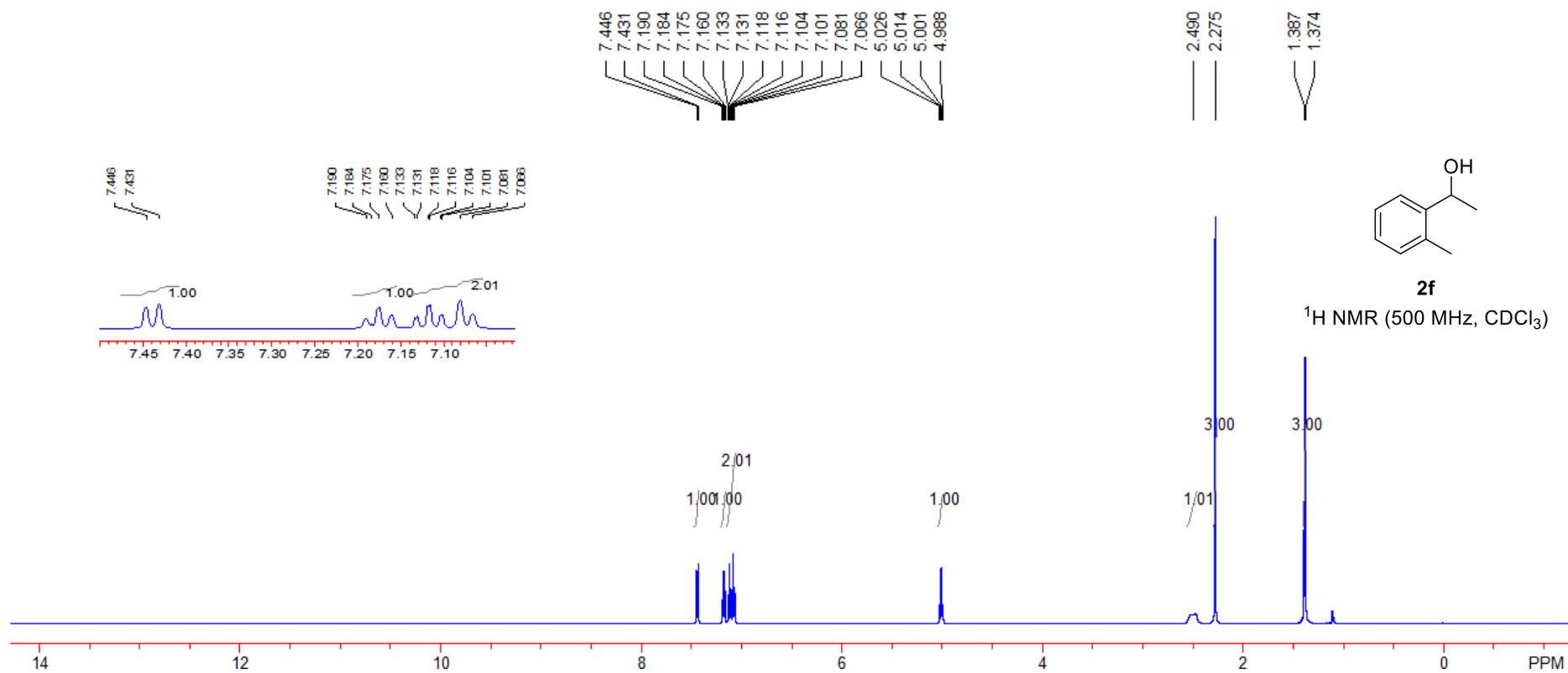


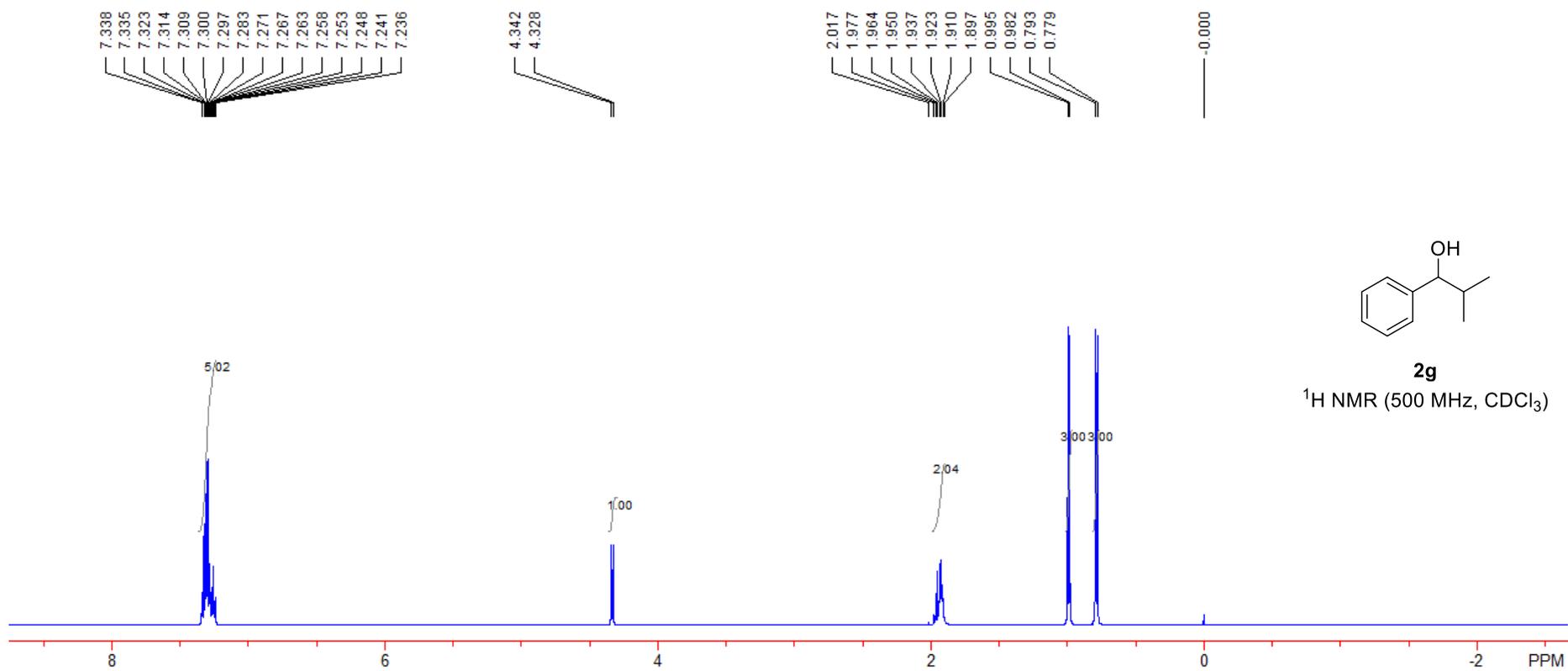


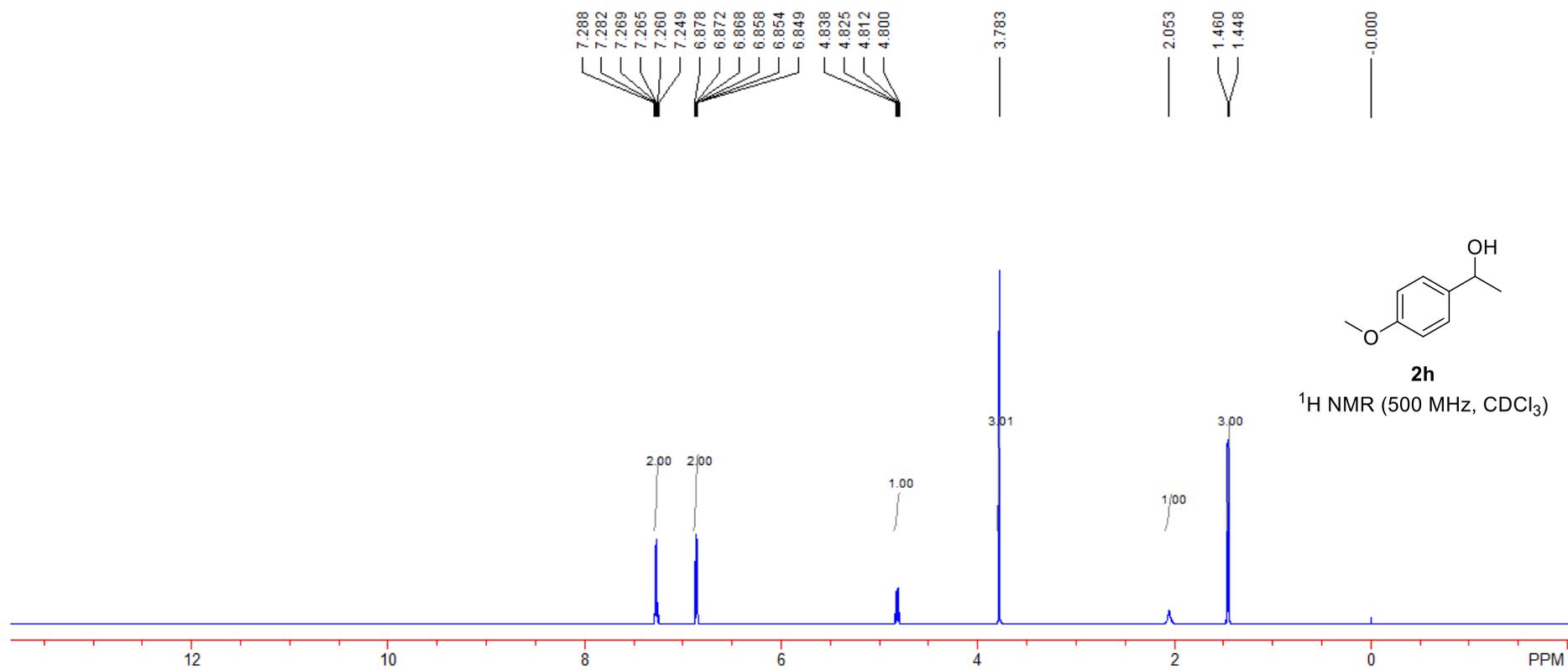


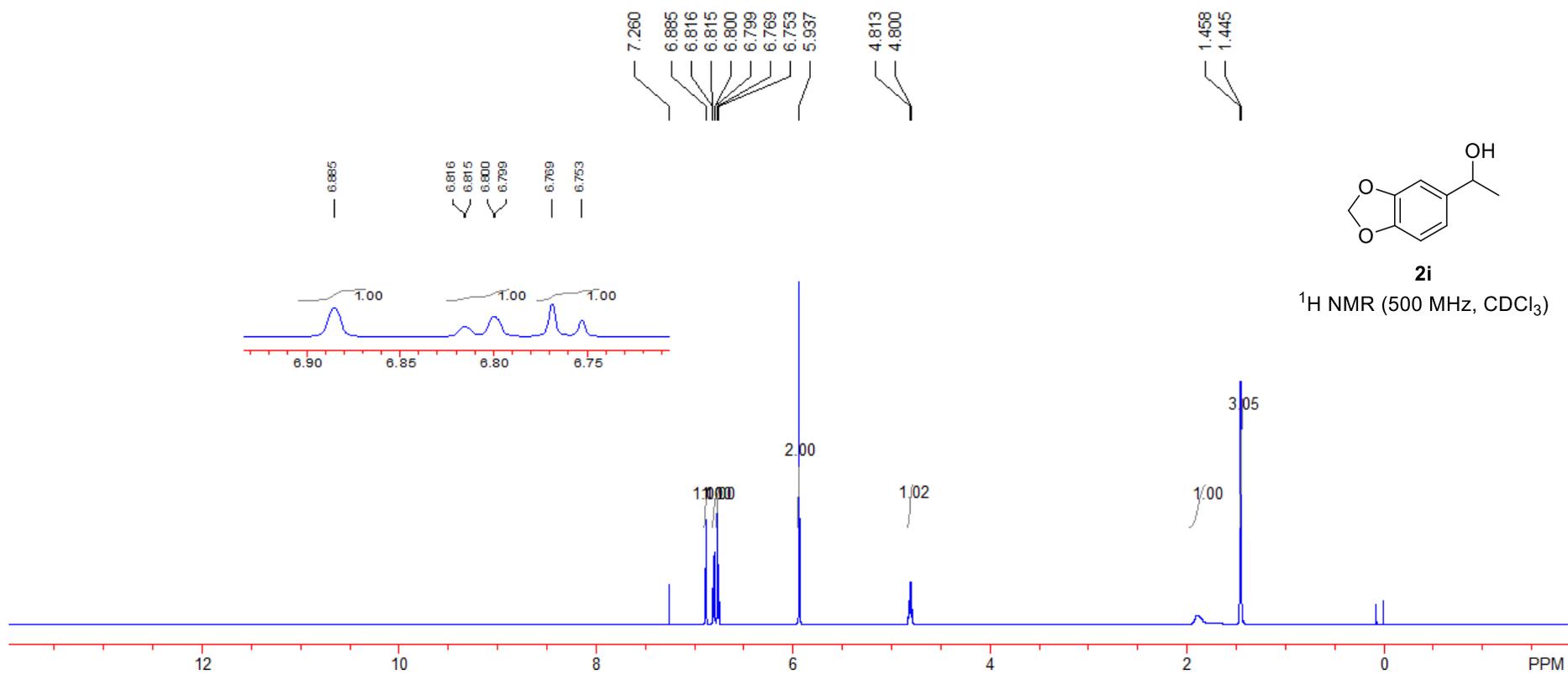


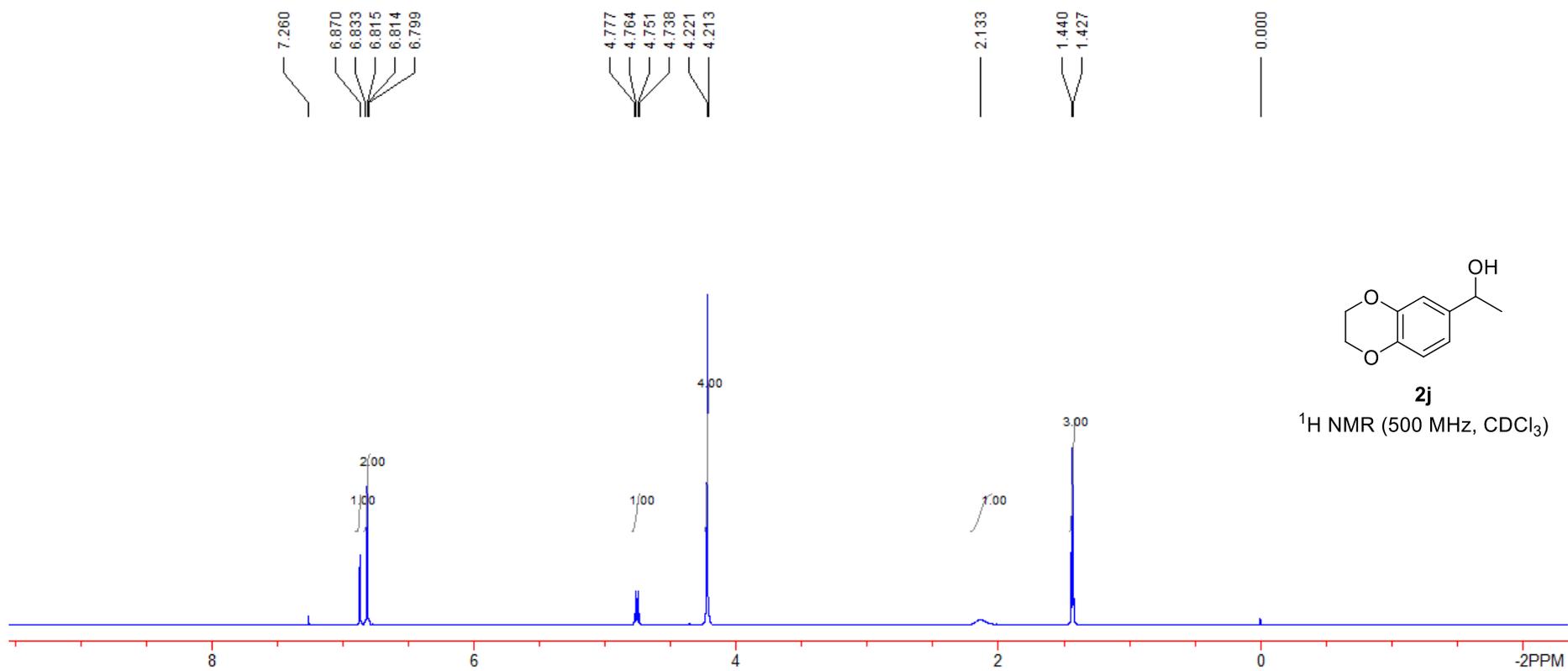


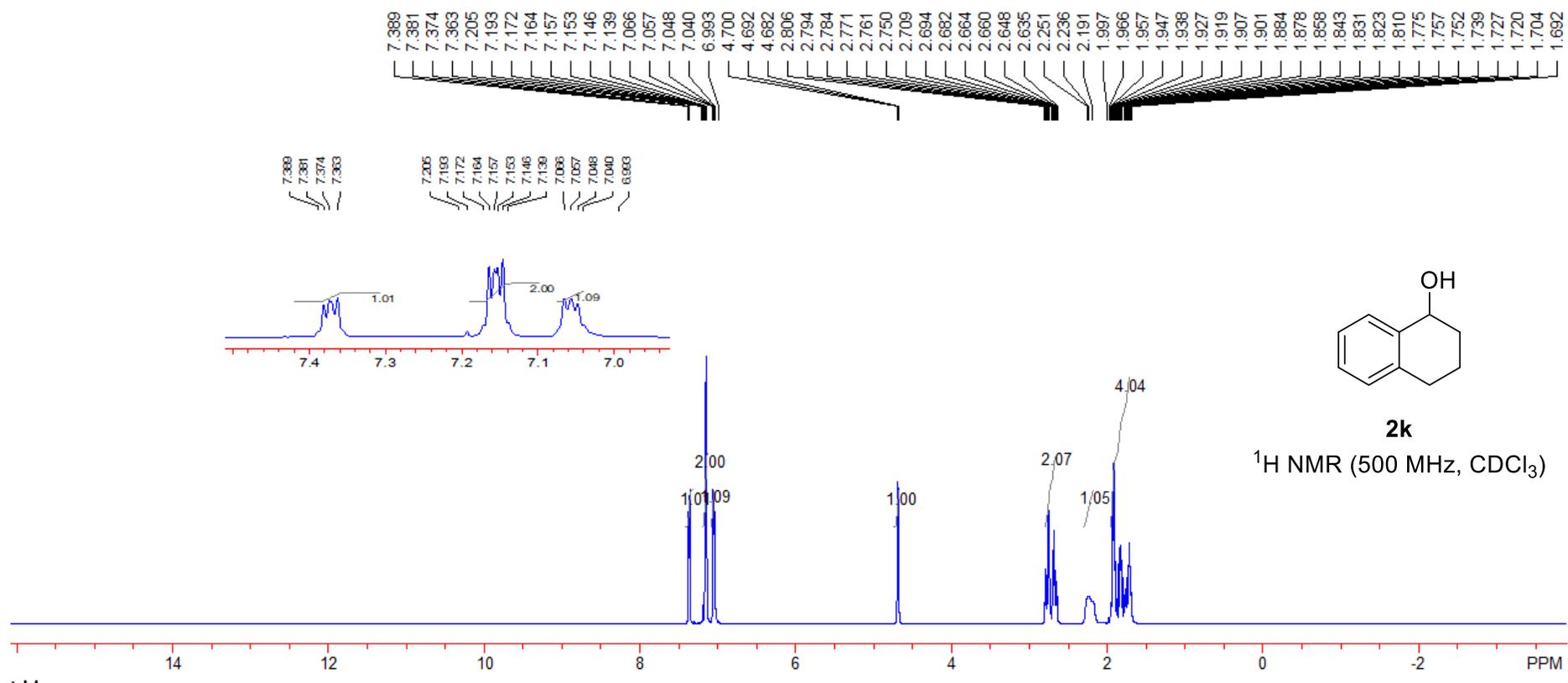


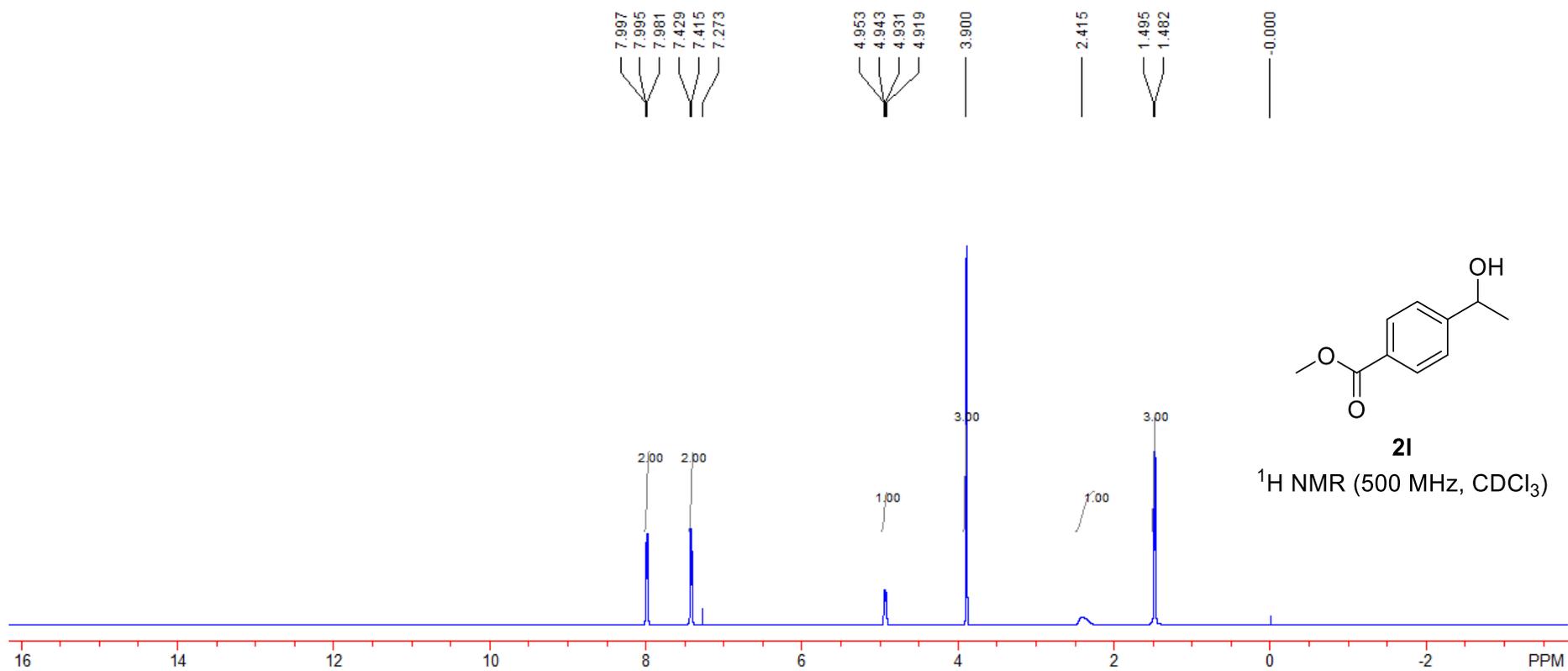


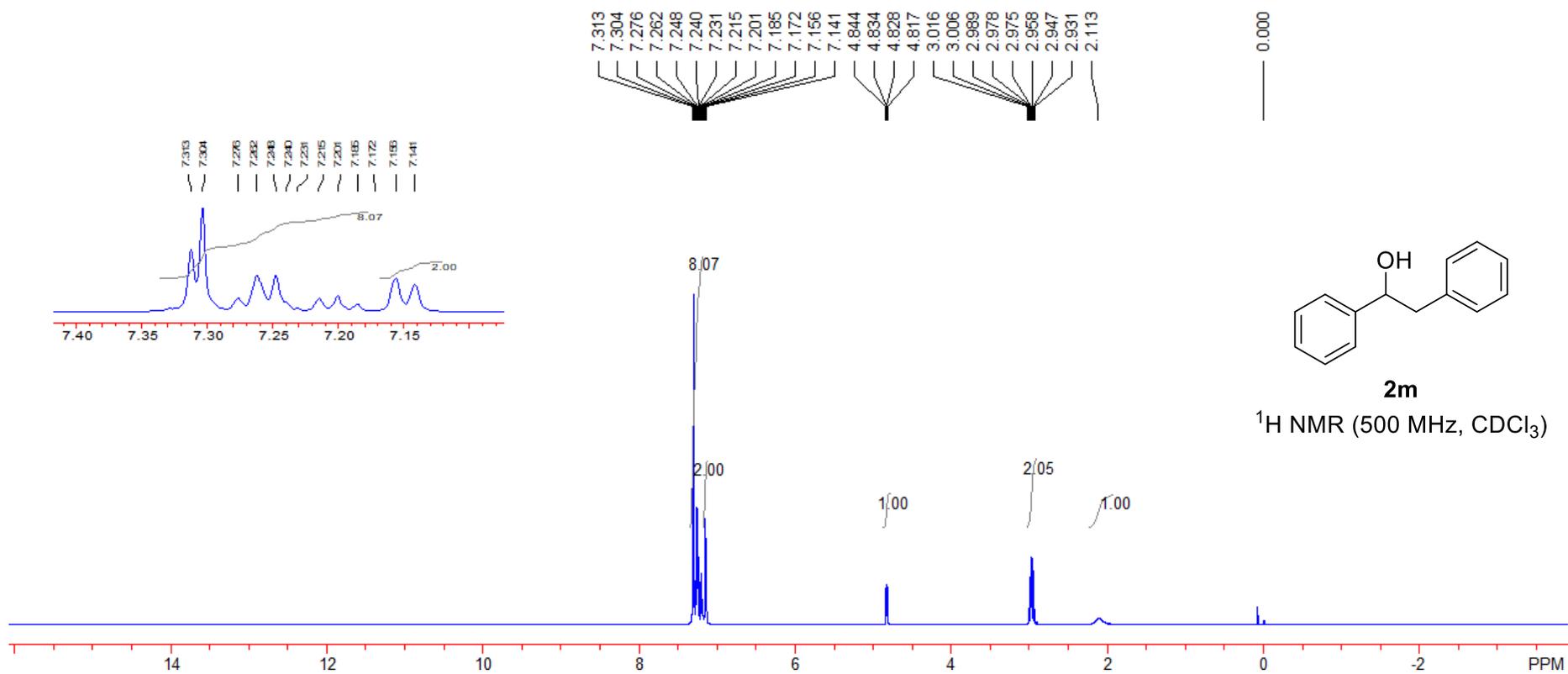


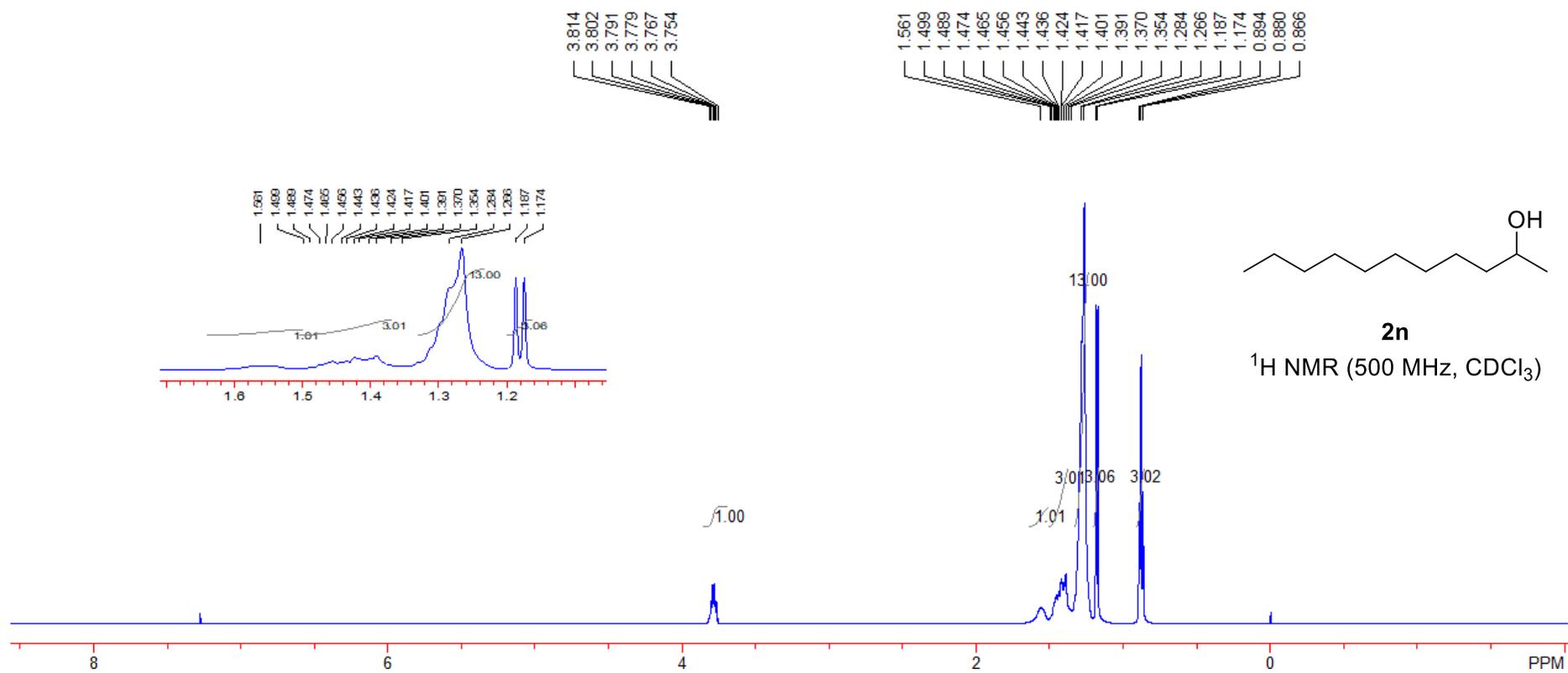


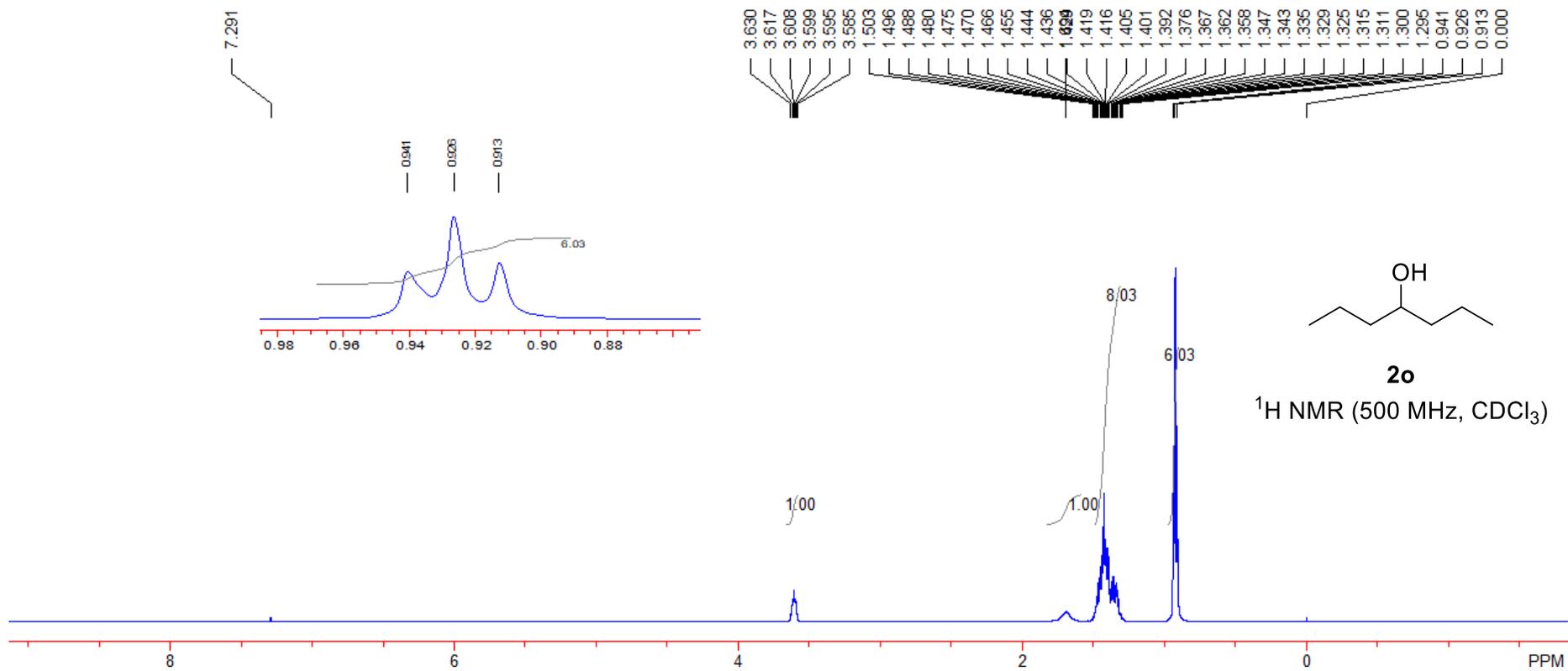


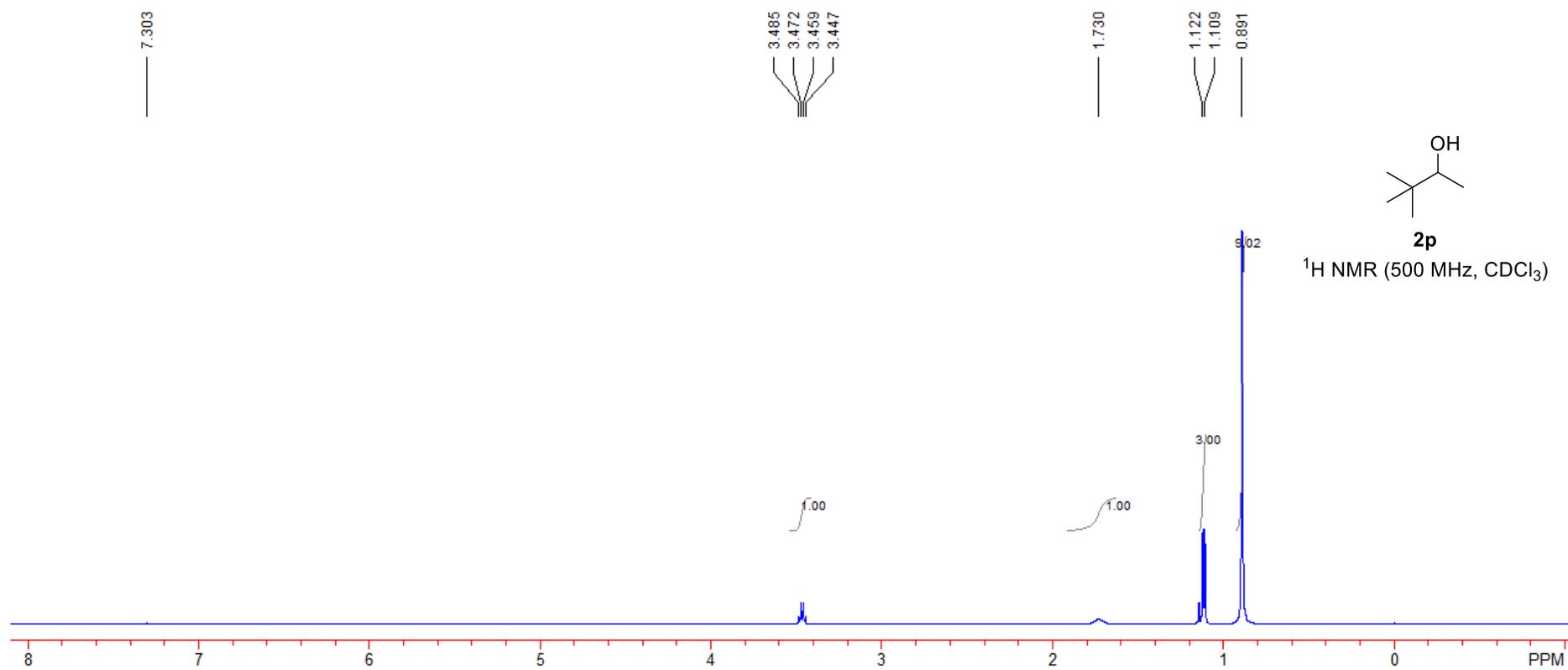


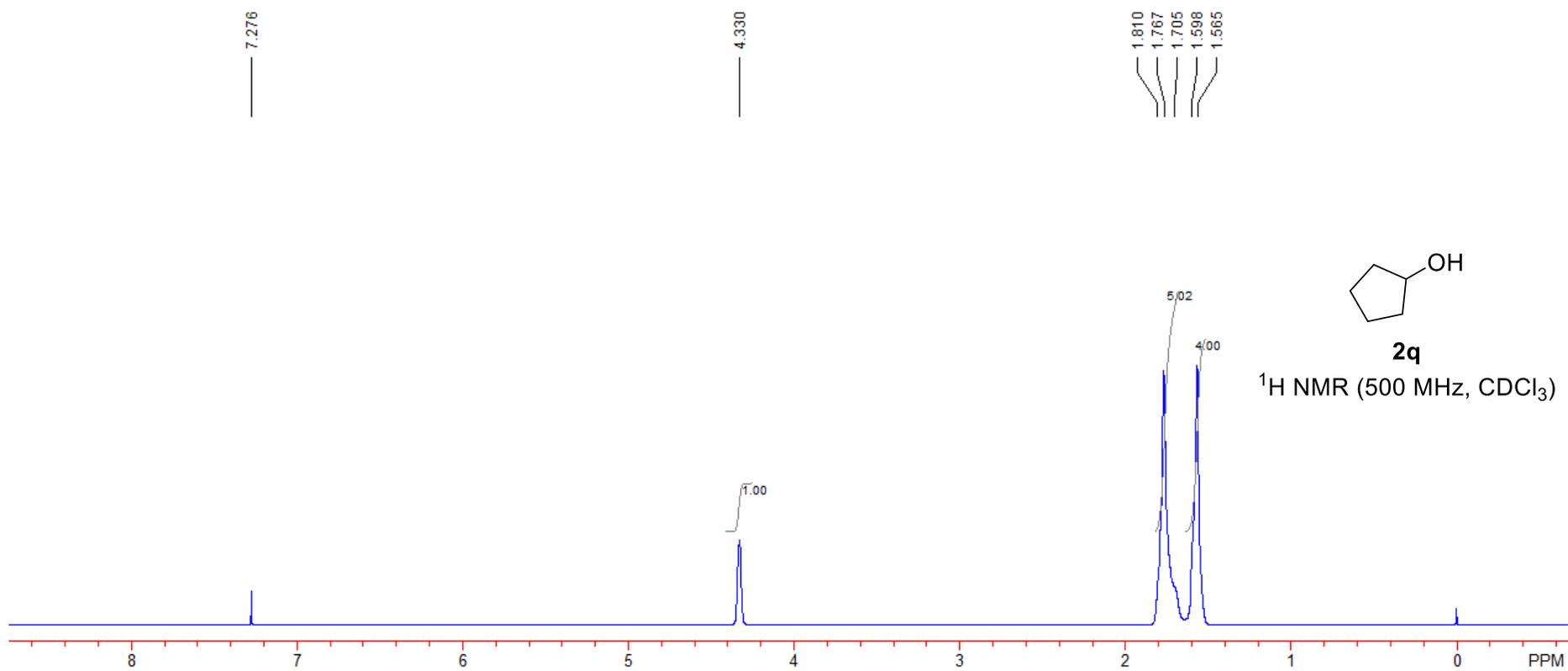


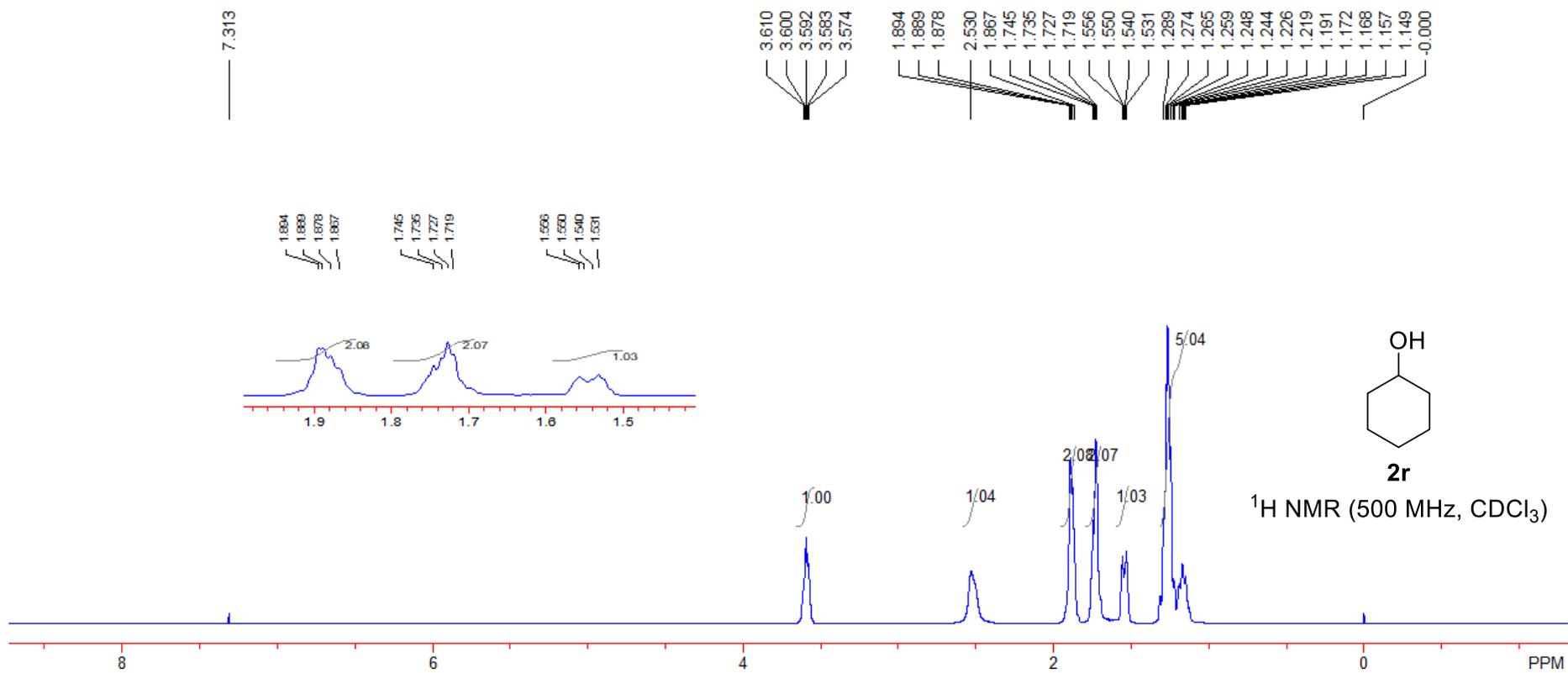


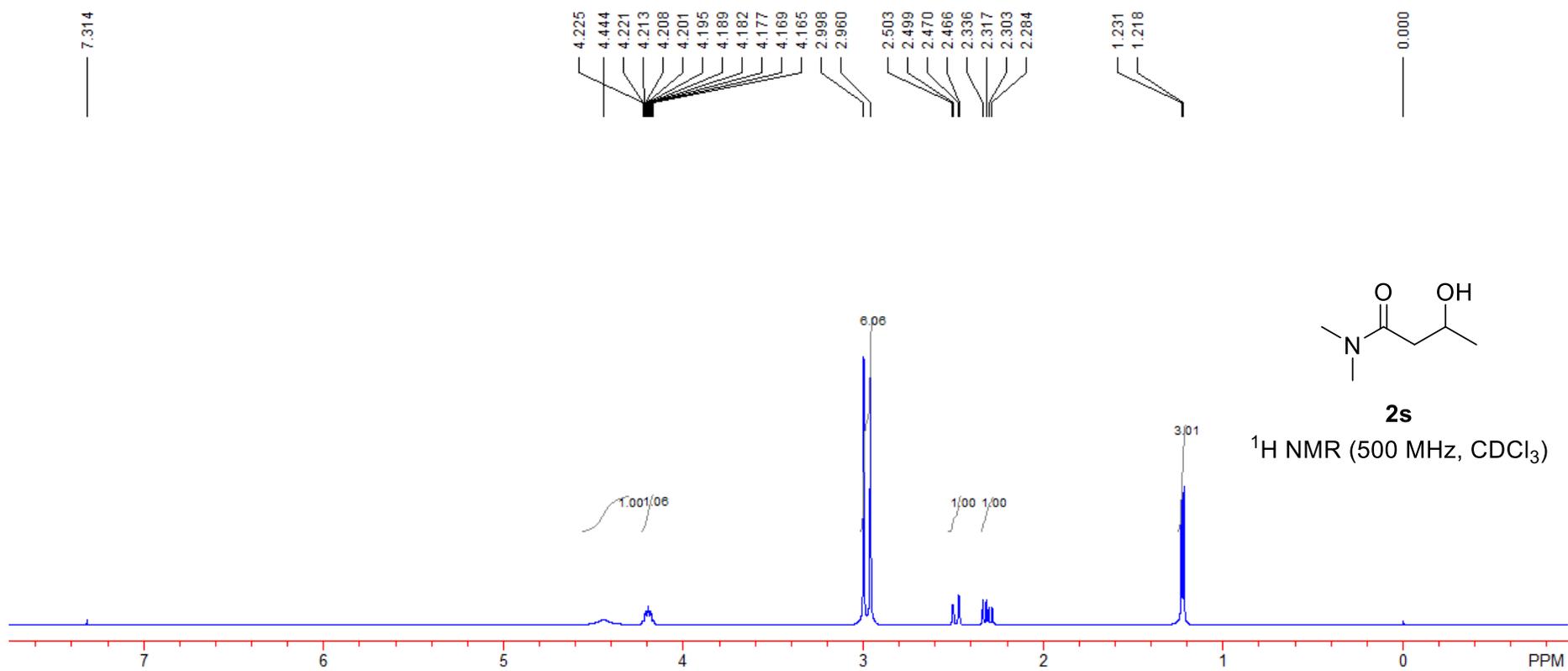


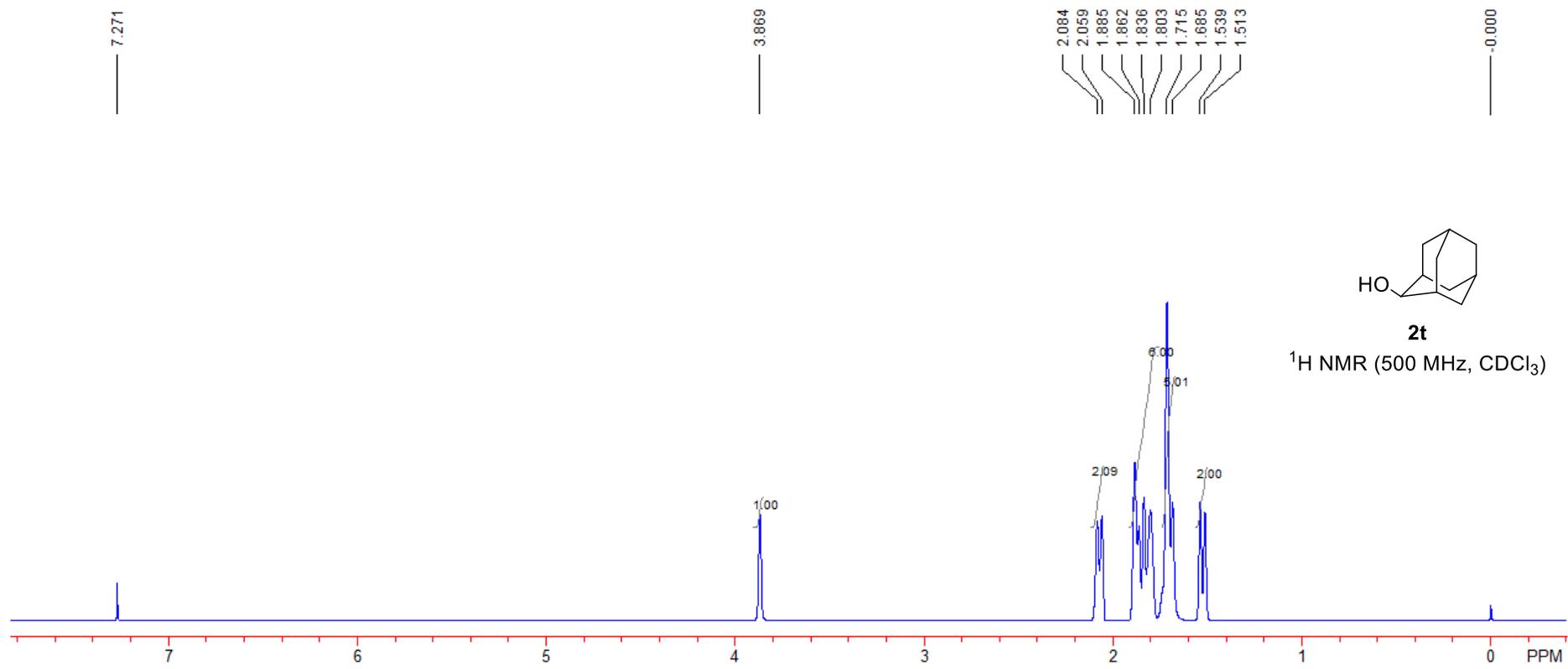


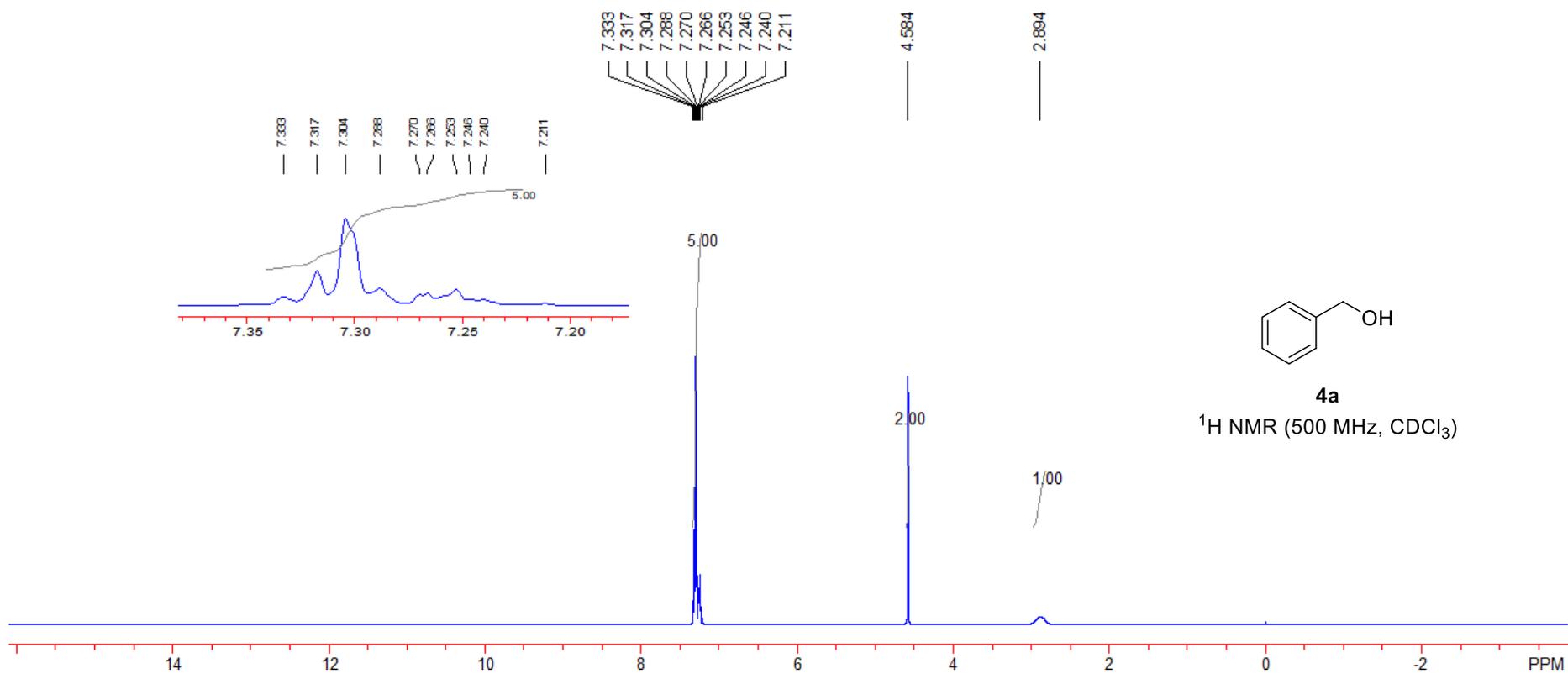


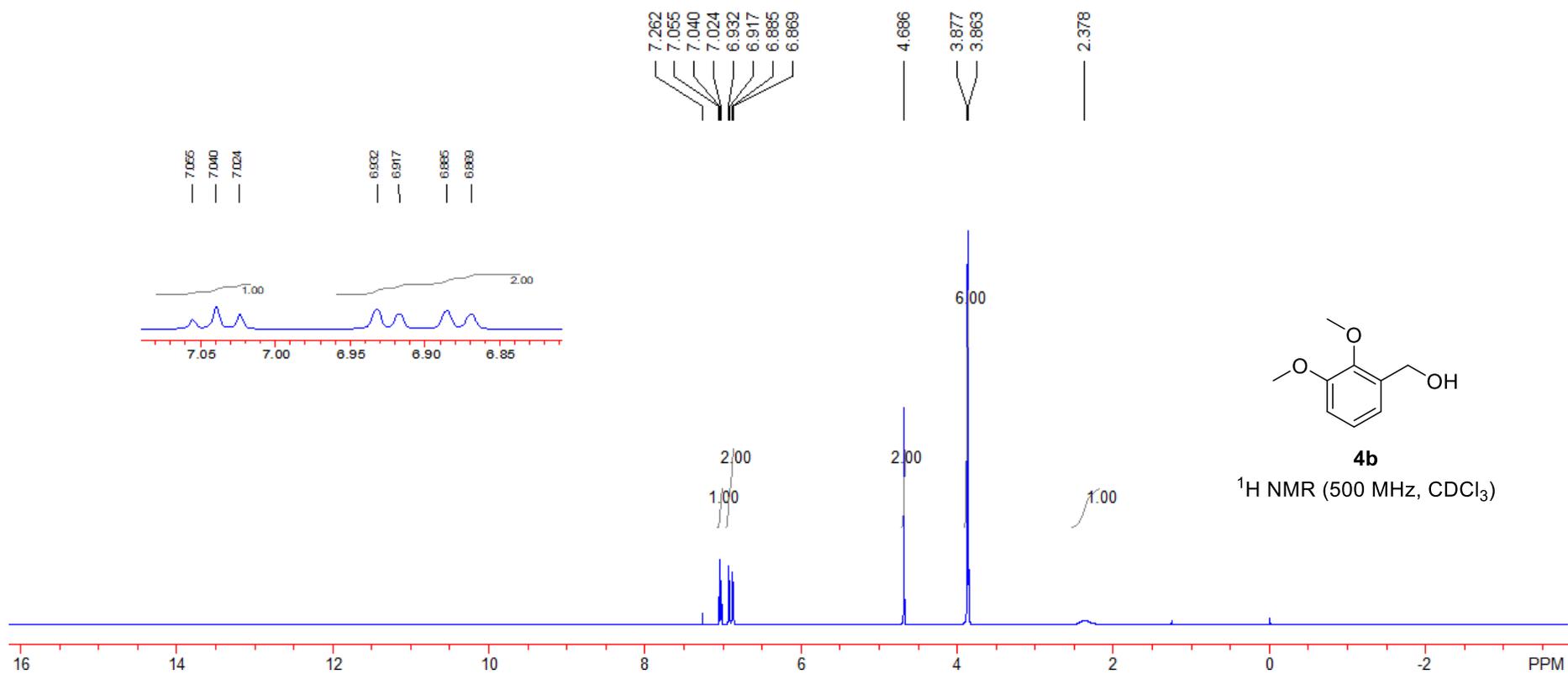


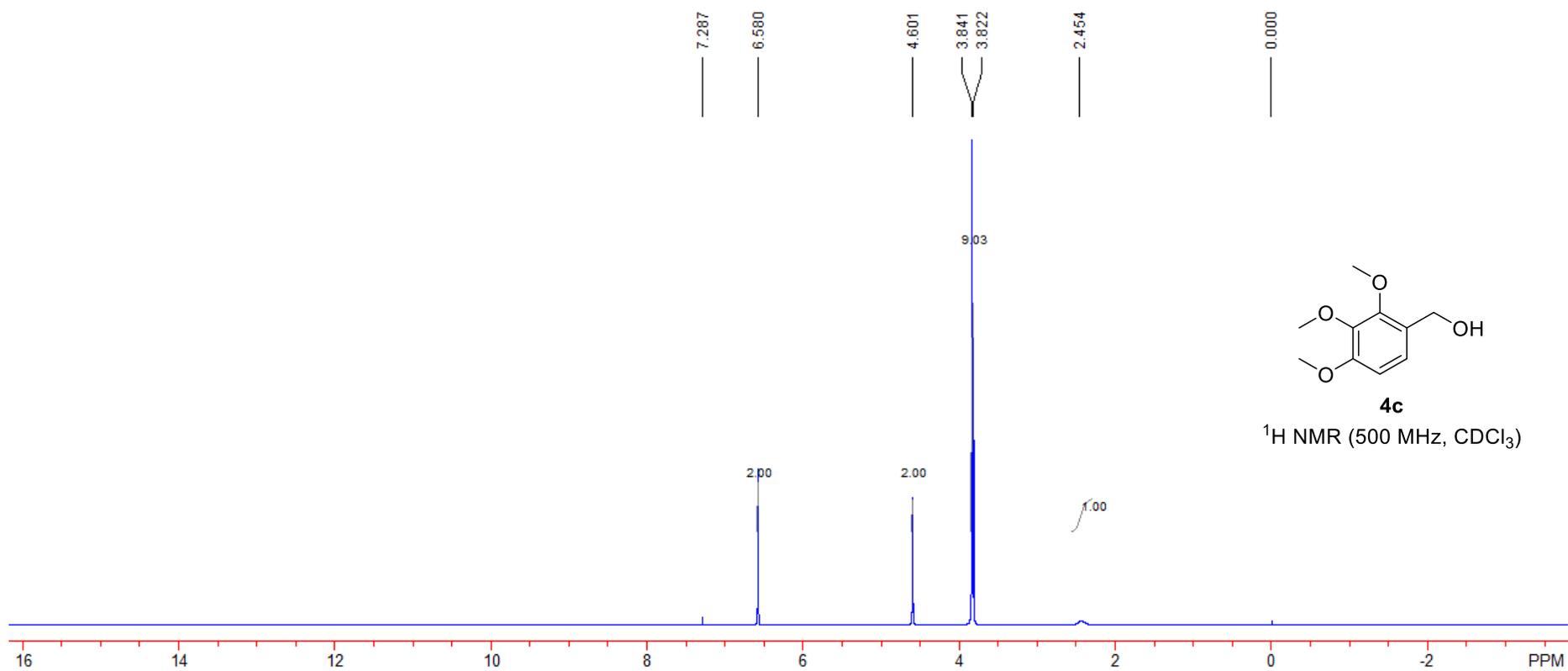


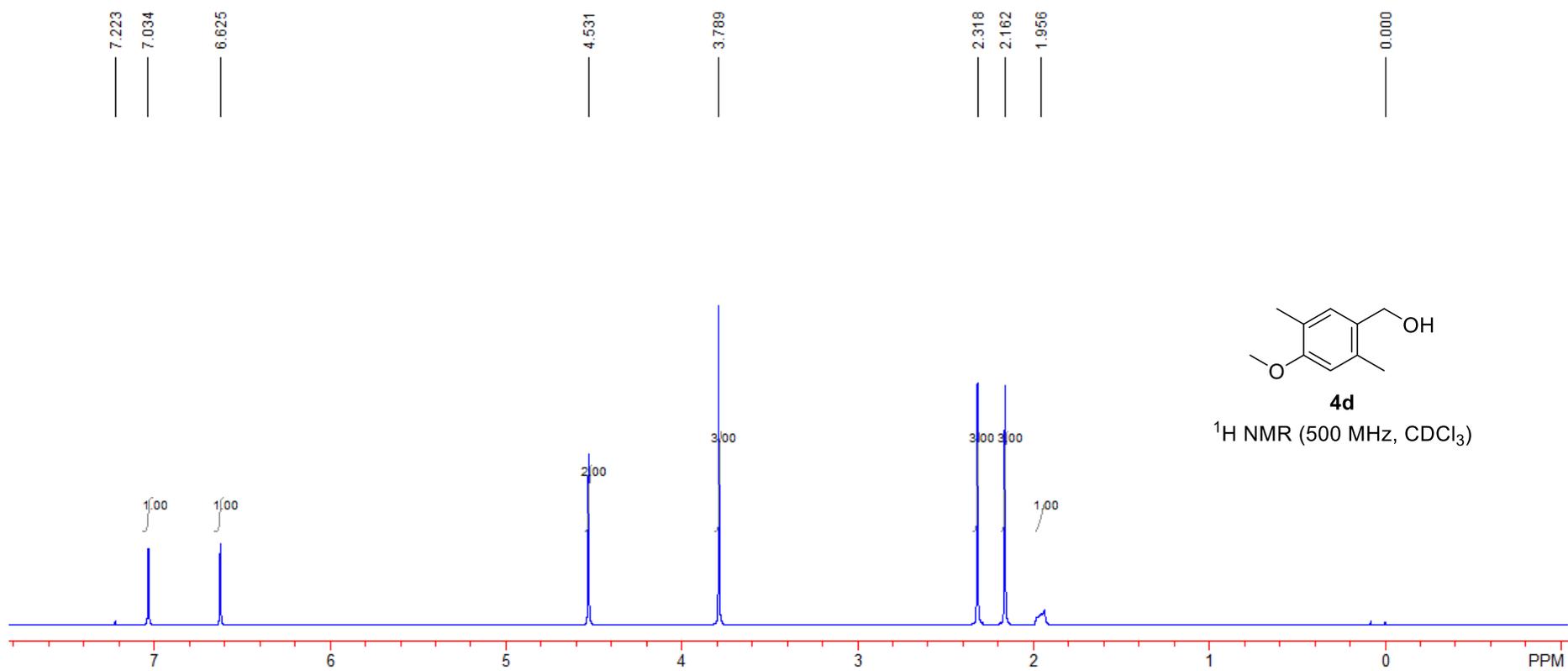


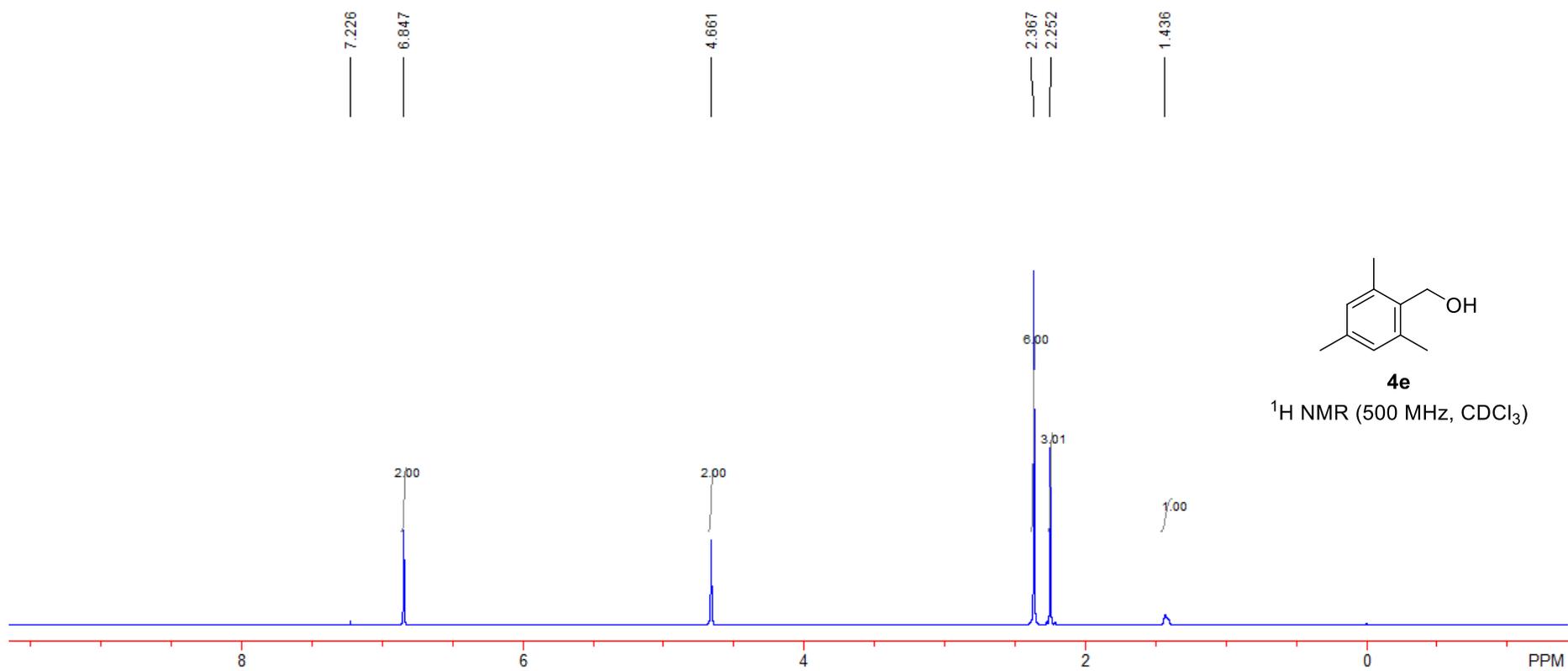


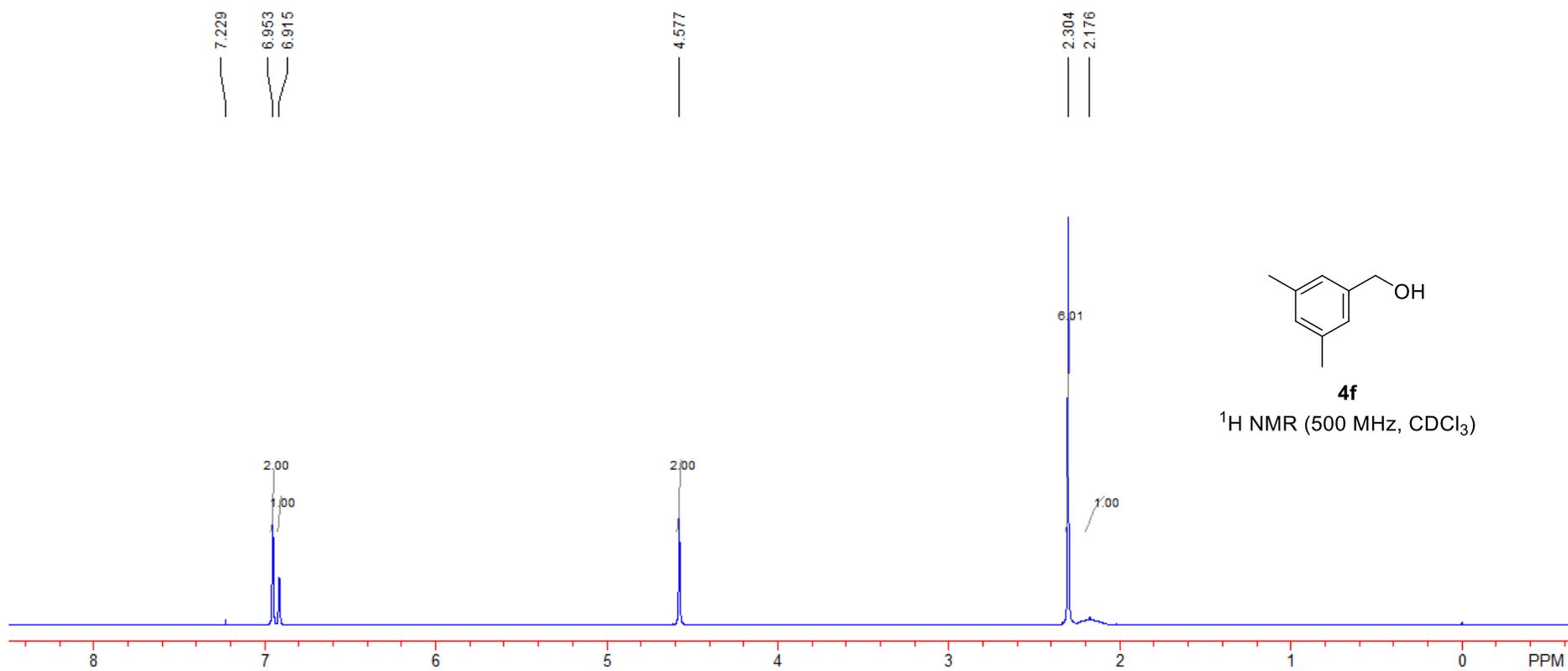








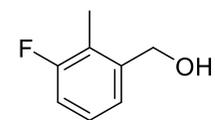




7.247
7.152
7.137
7.127
7.122
7.113
6.954

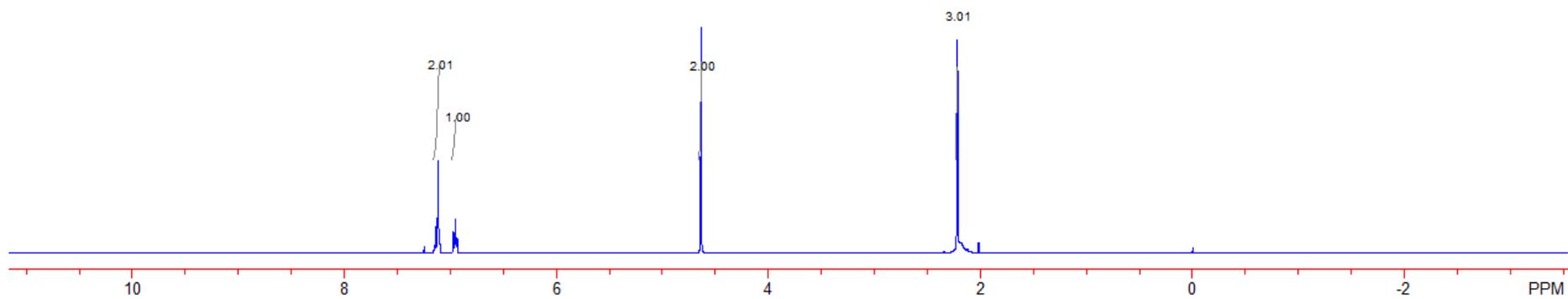
4.636

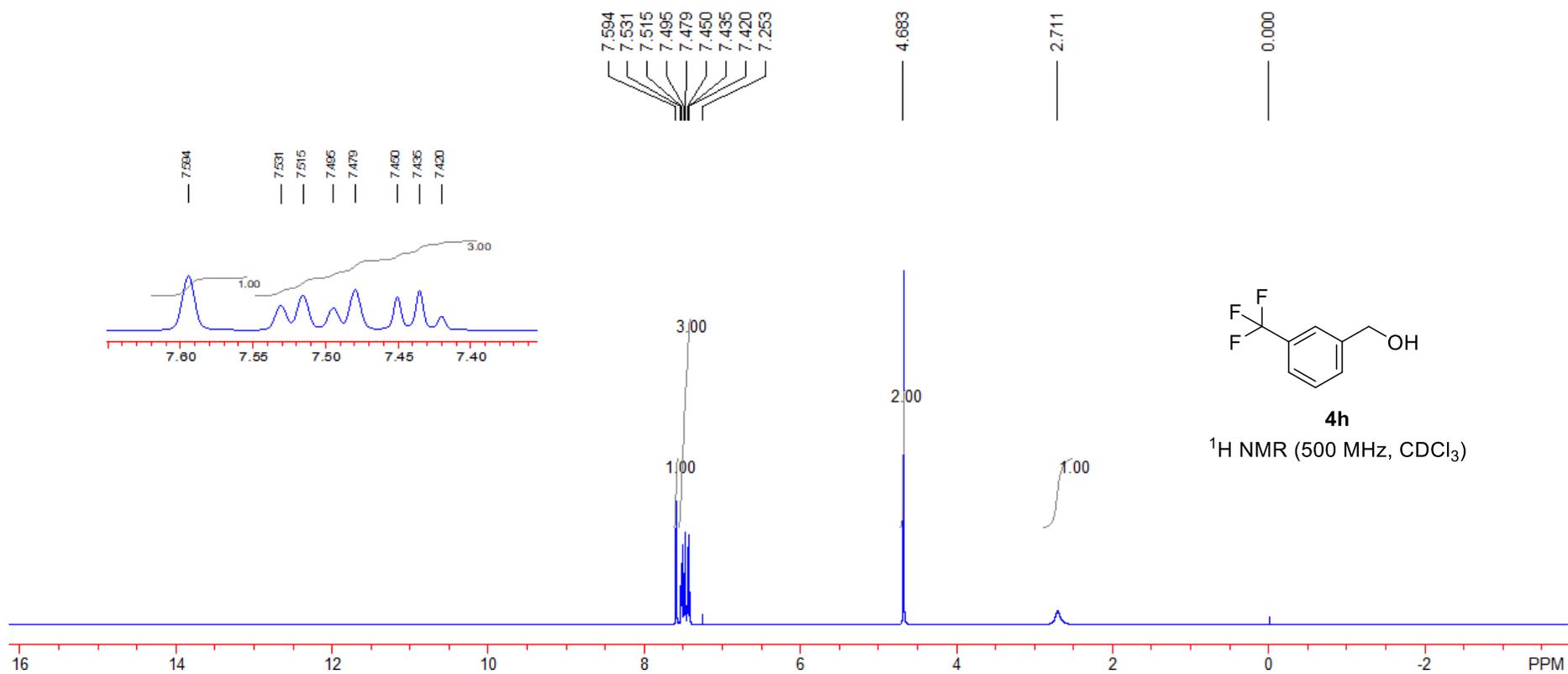
2.221
2.217

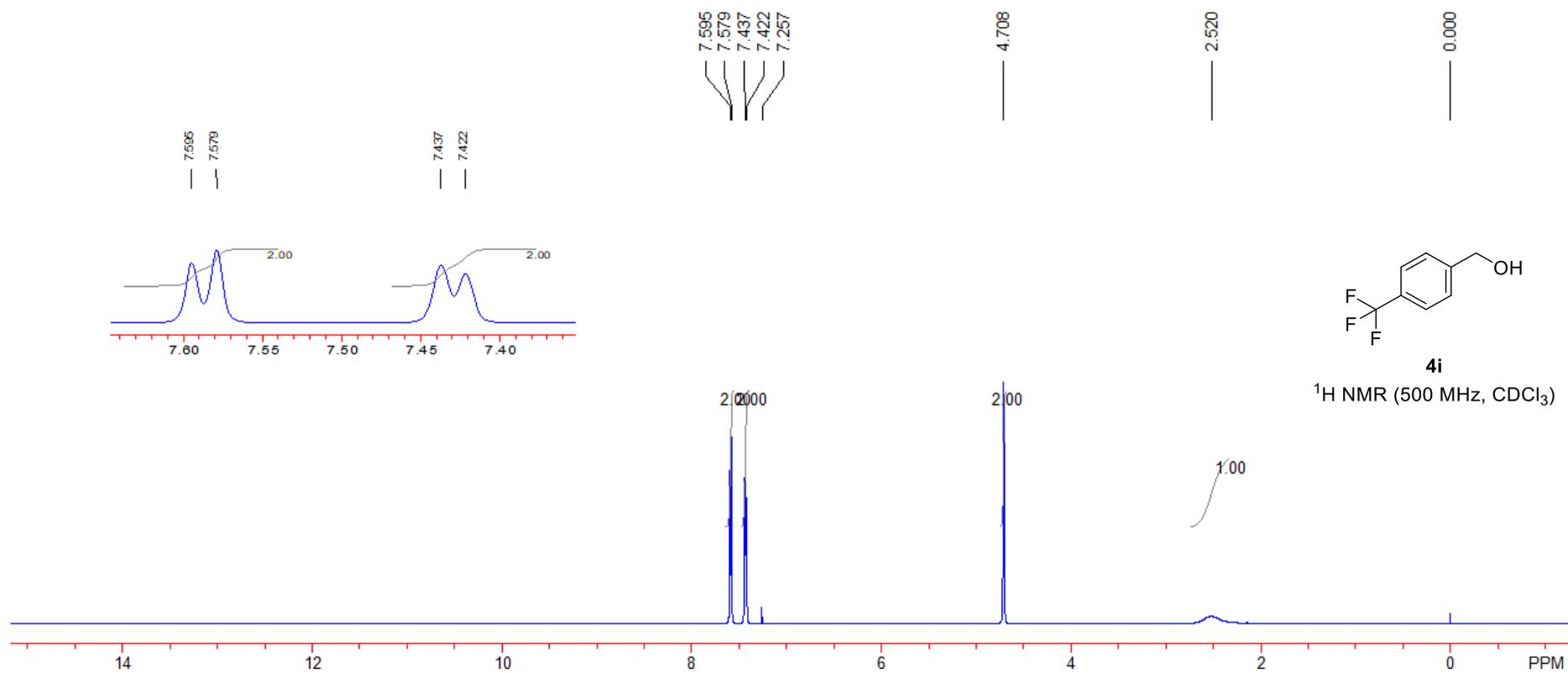


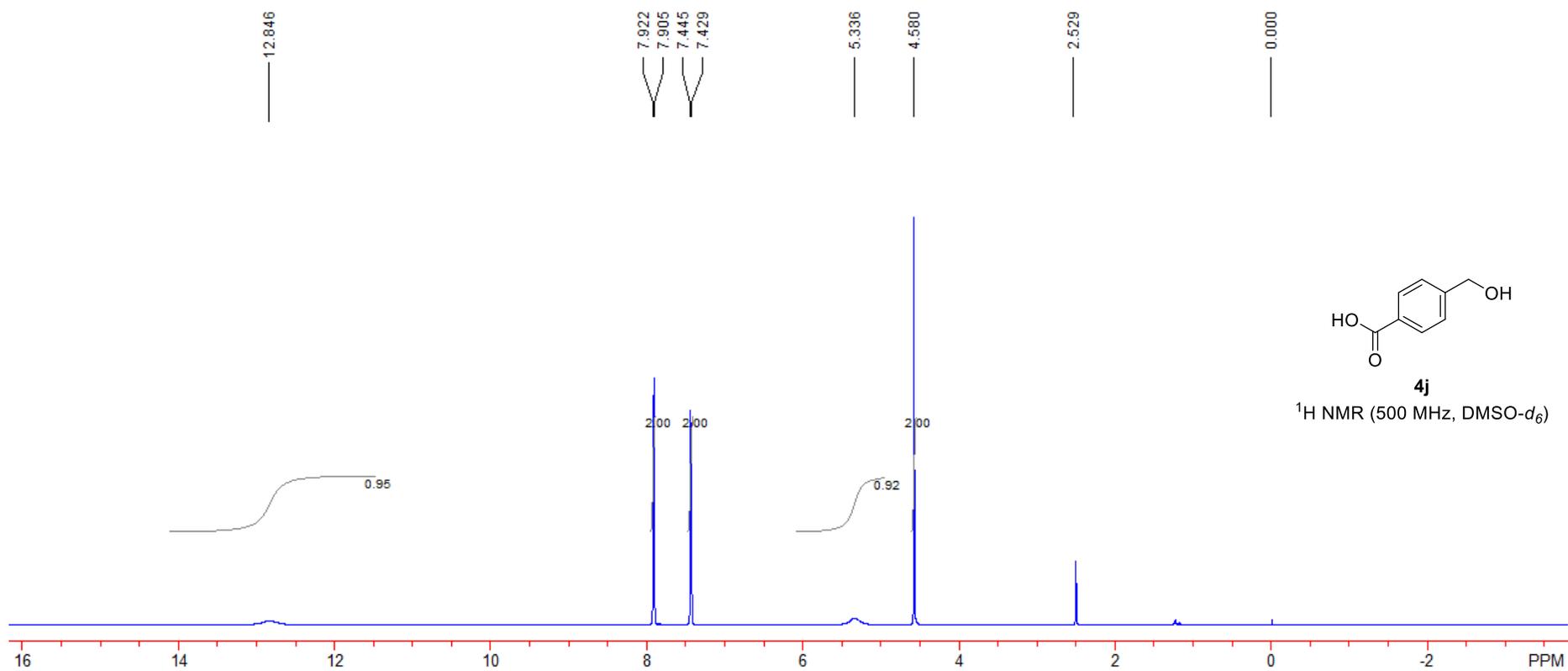
4g

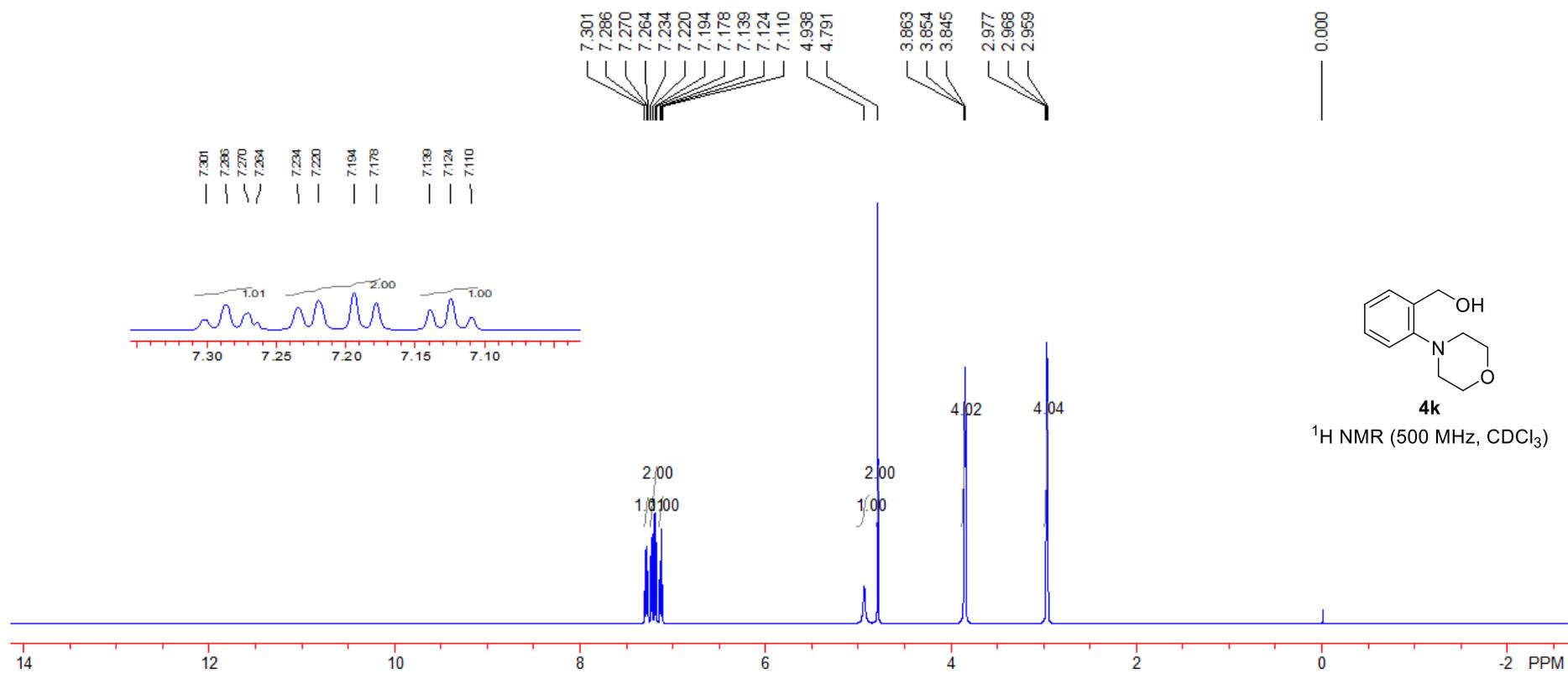
¹H NMR (500 MHz, CDCl₃)

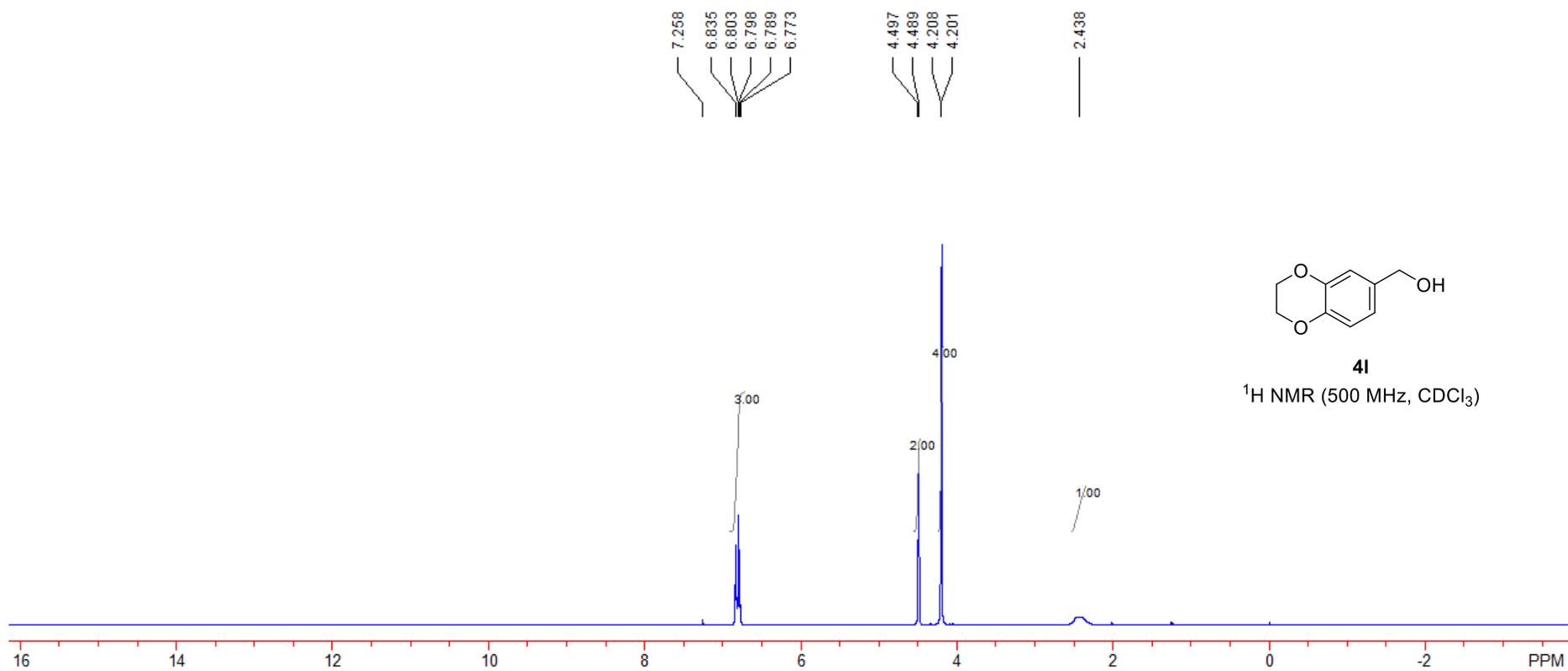


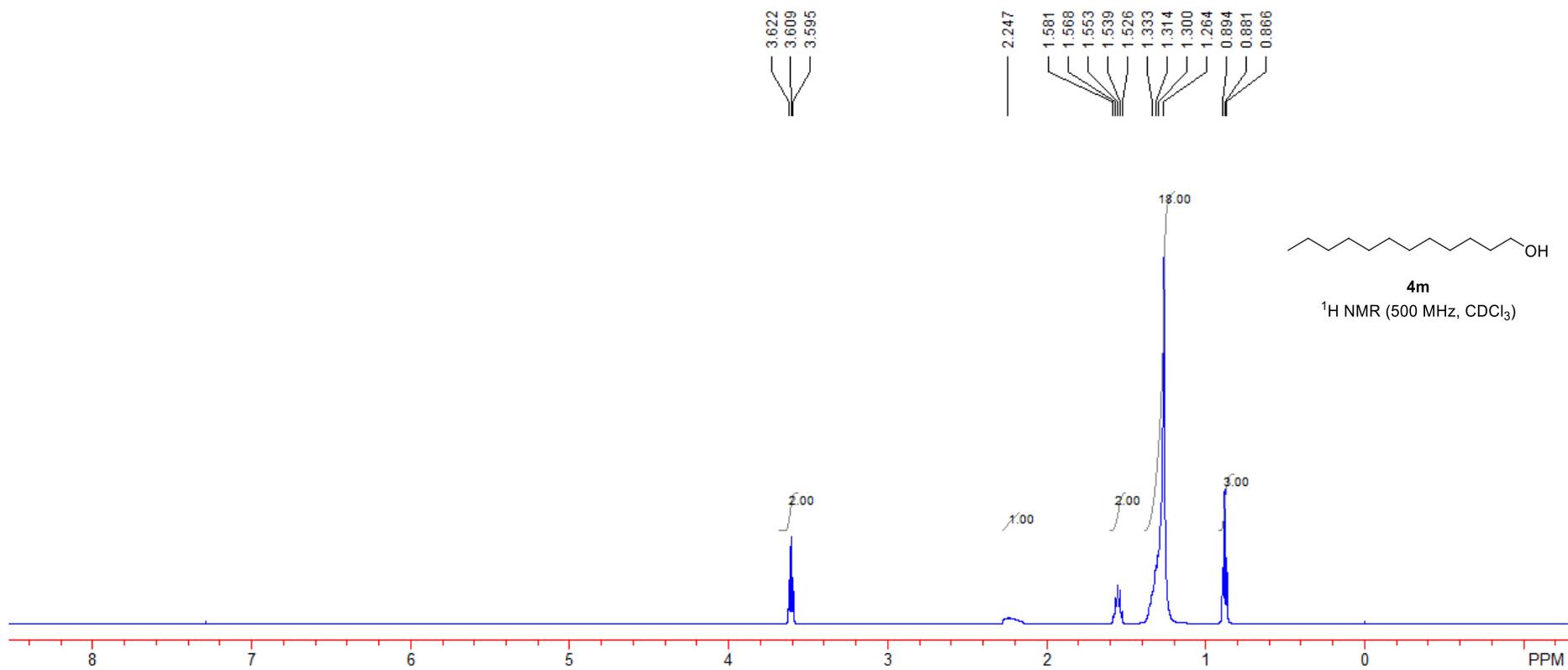


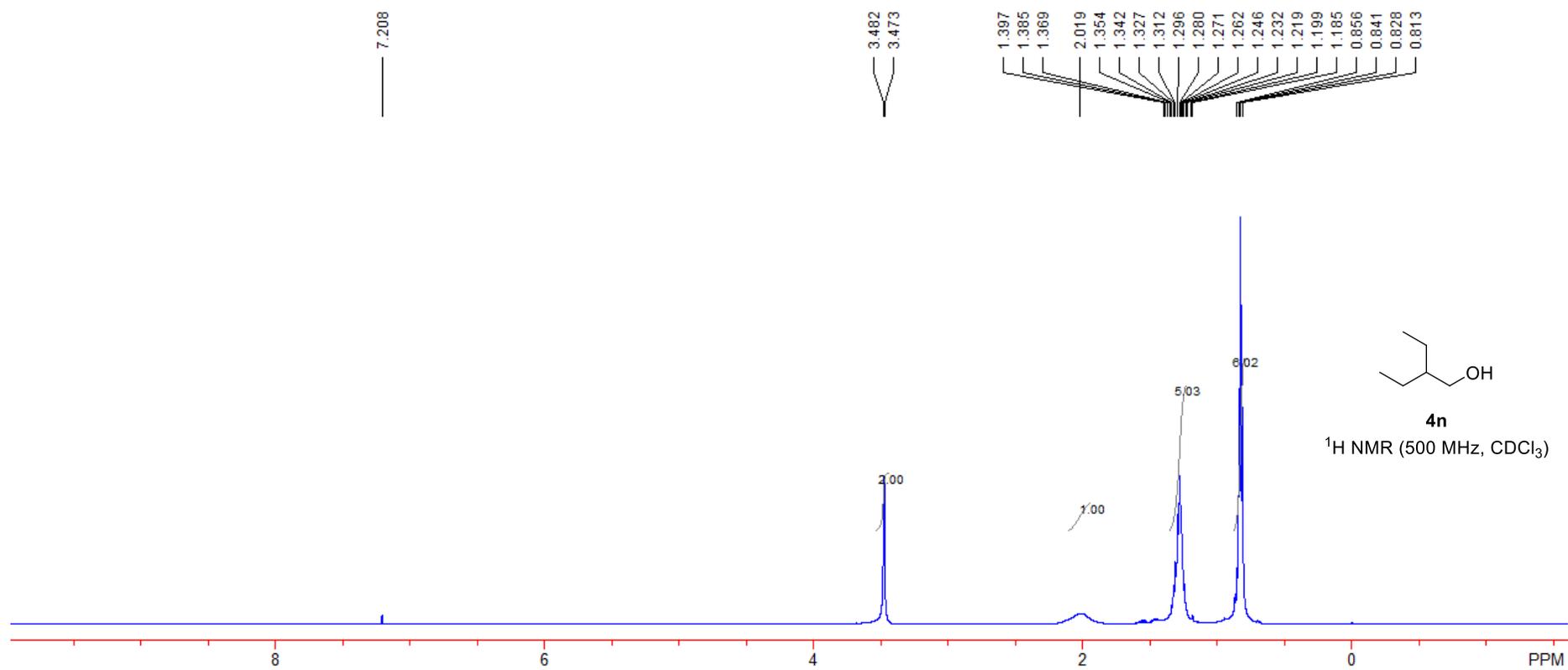


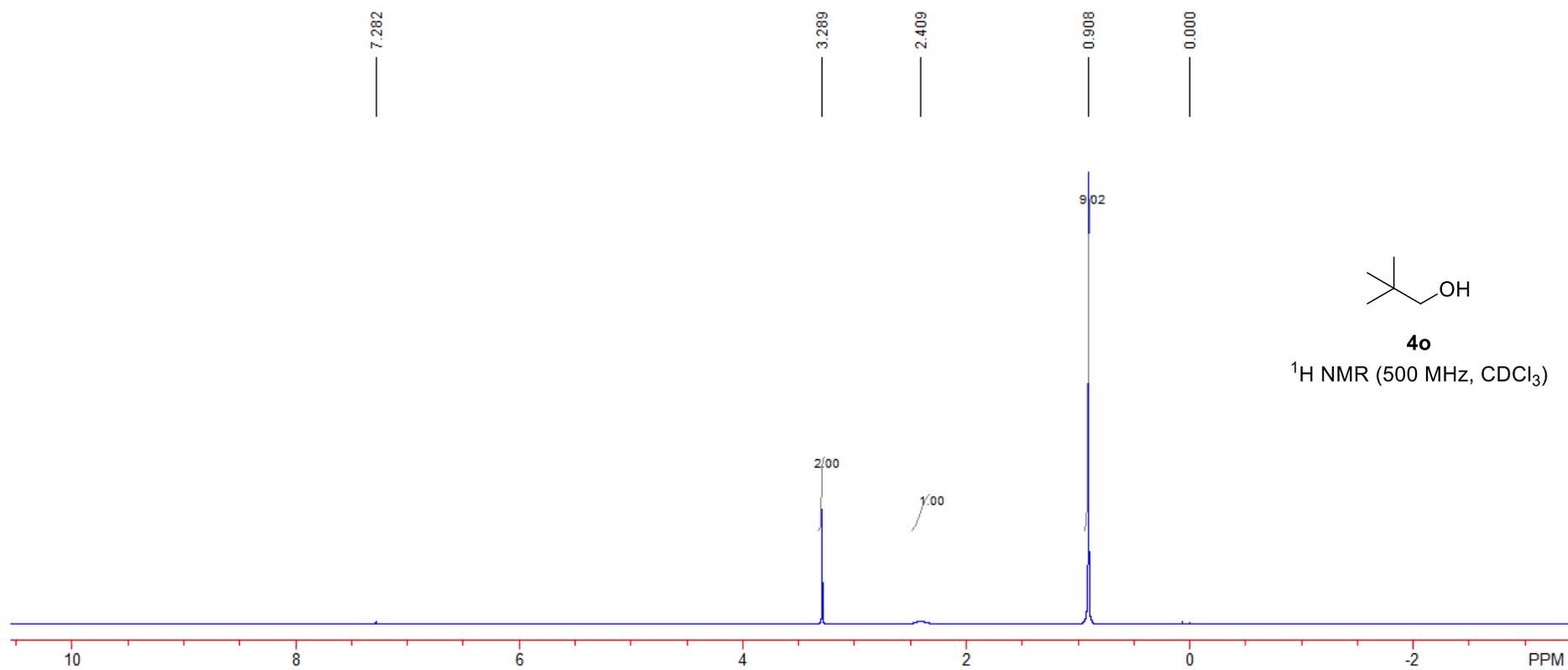


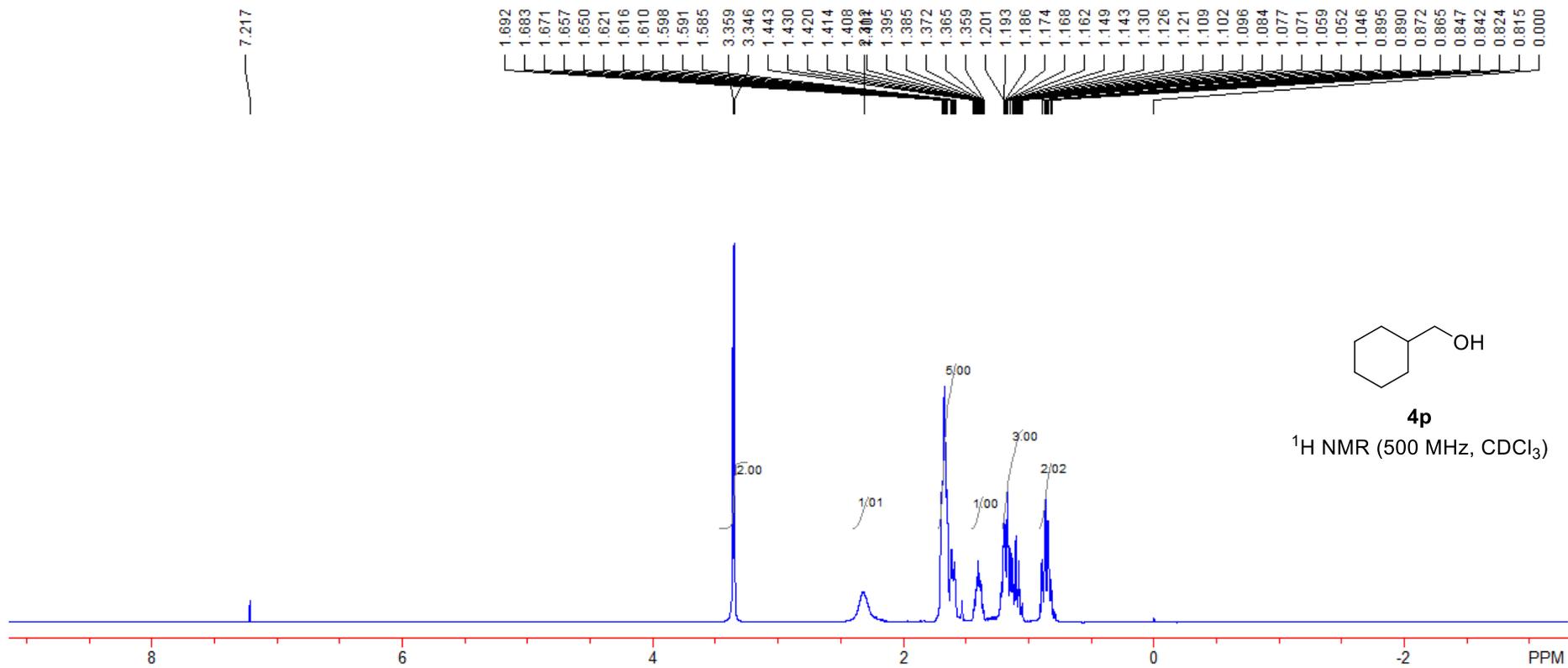


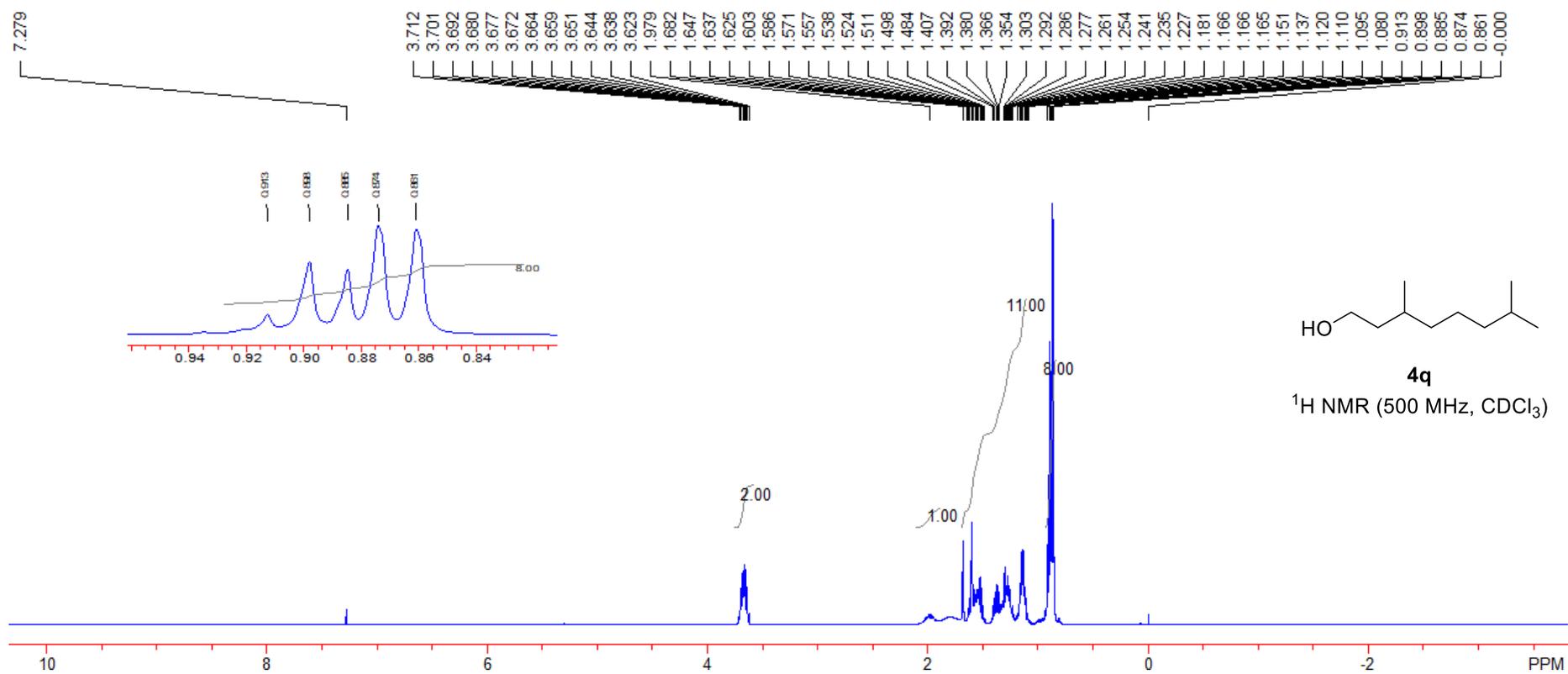


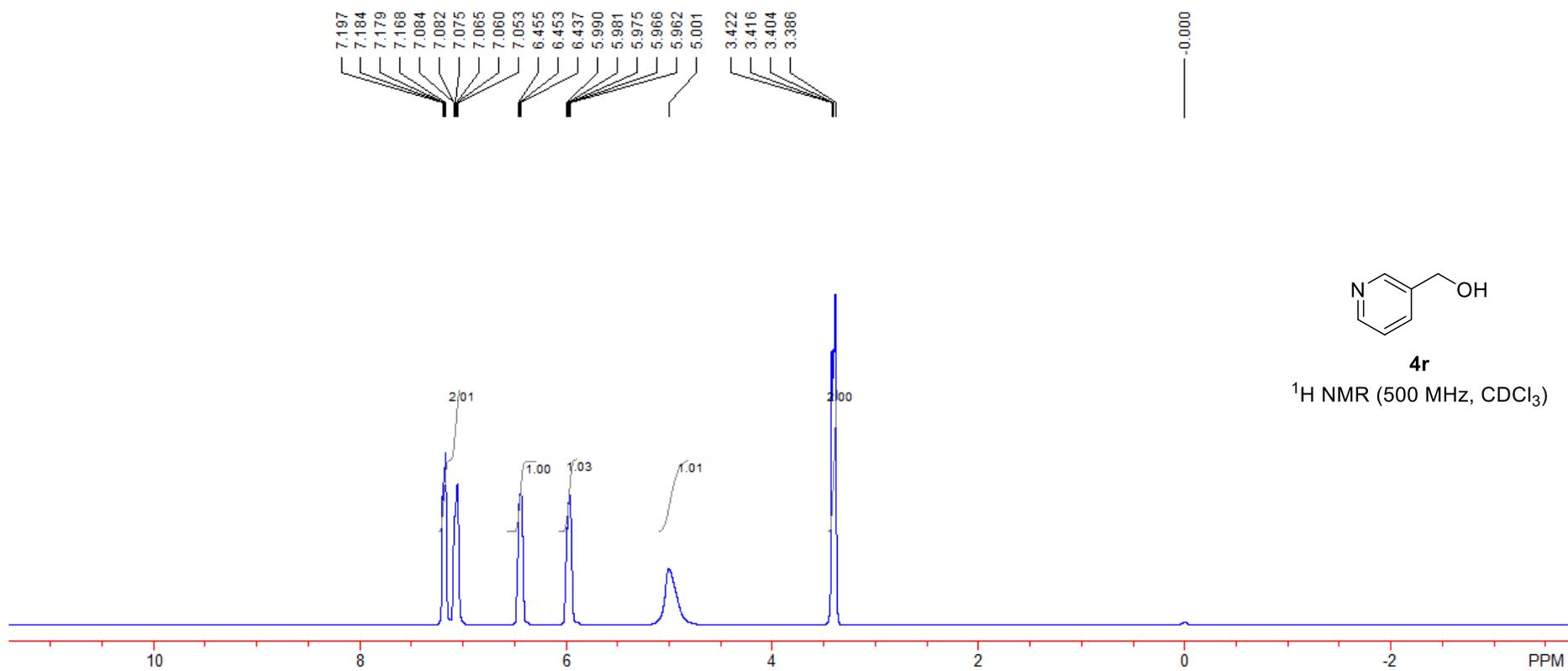


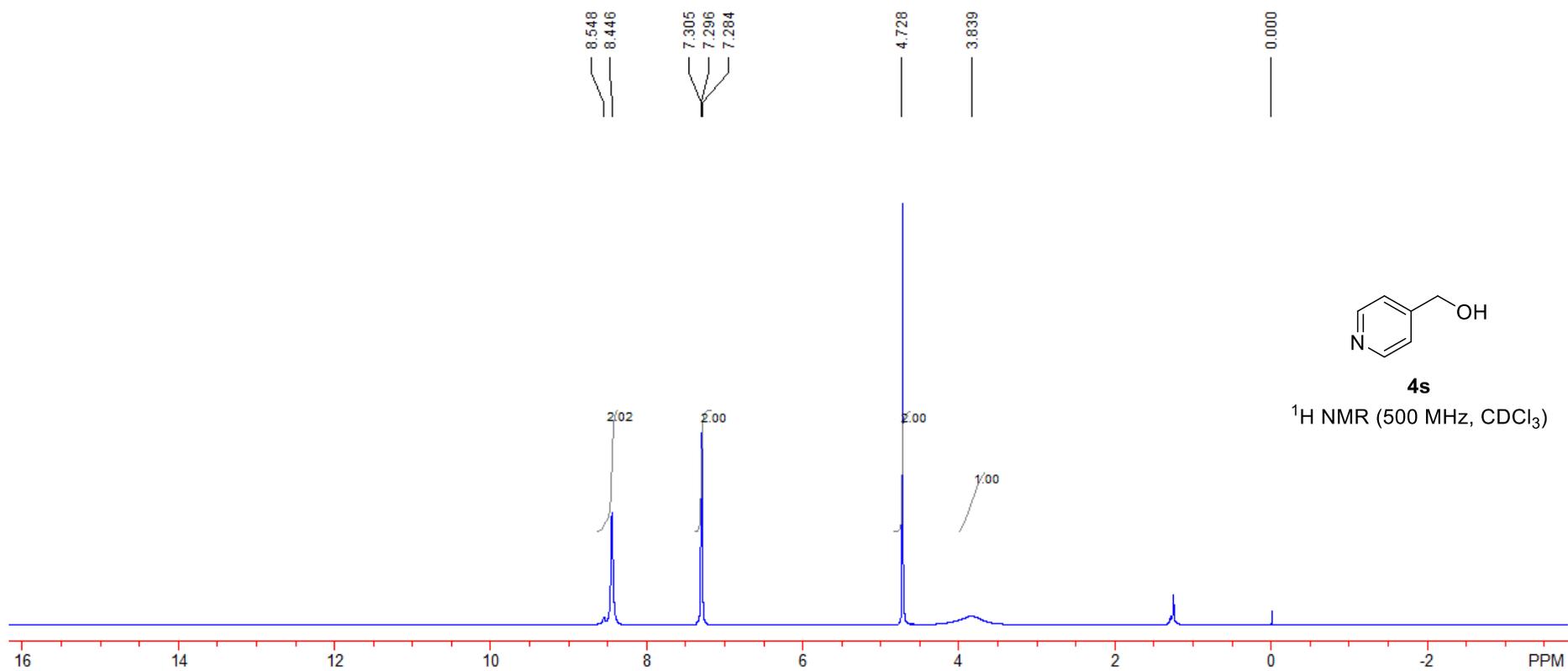


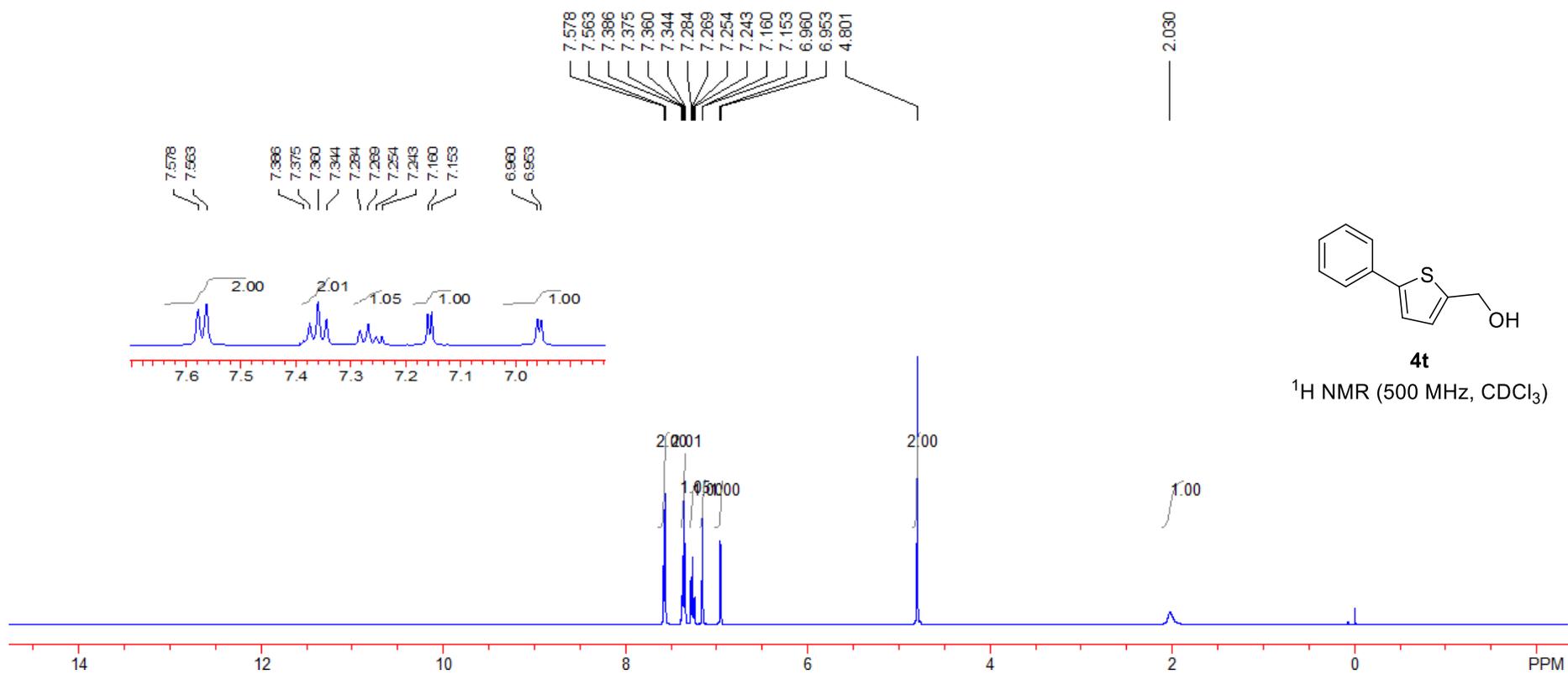


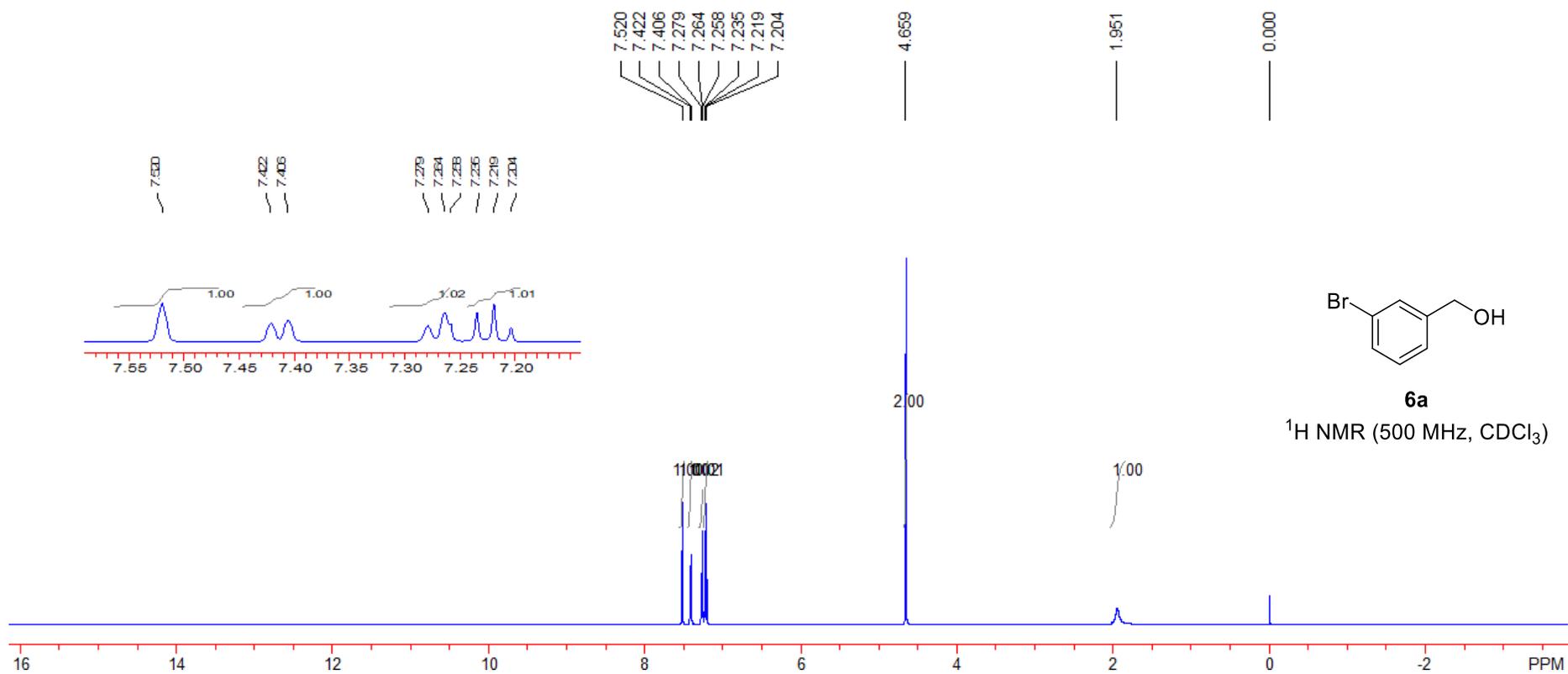


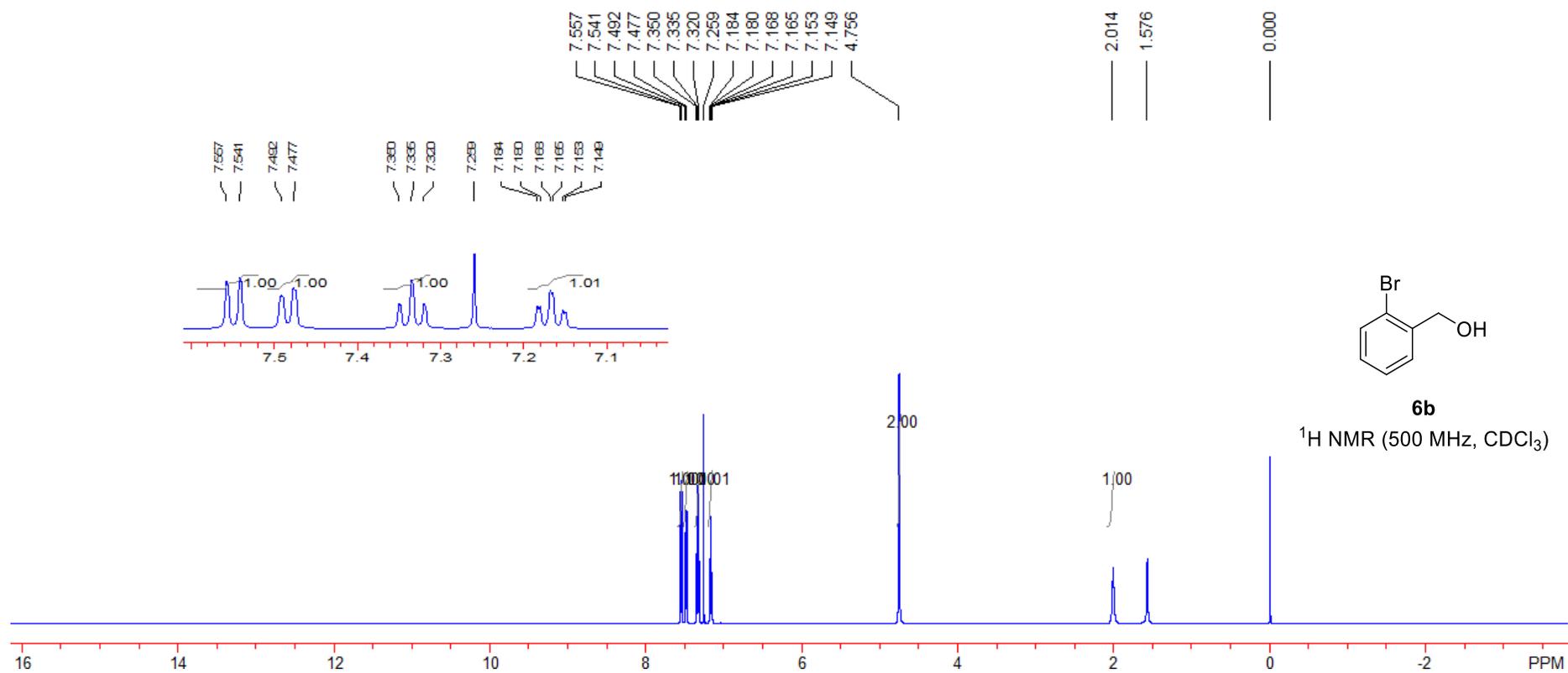


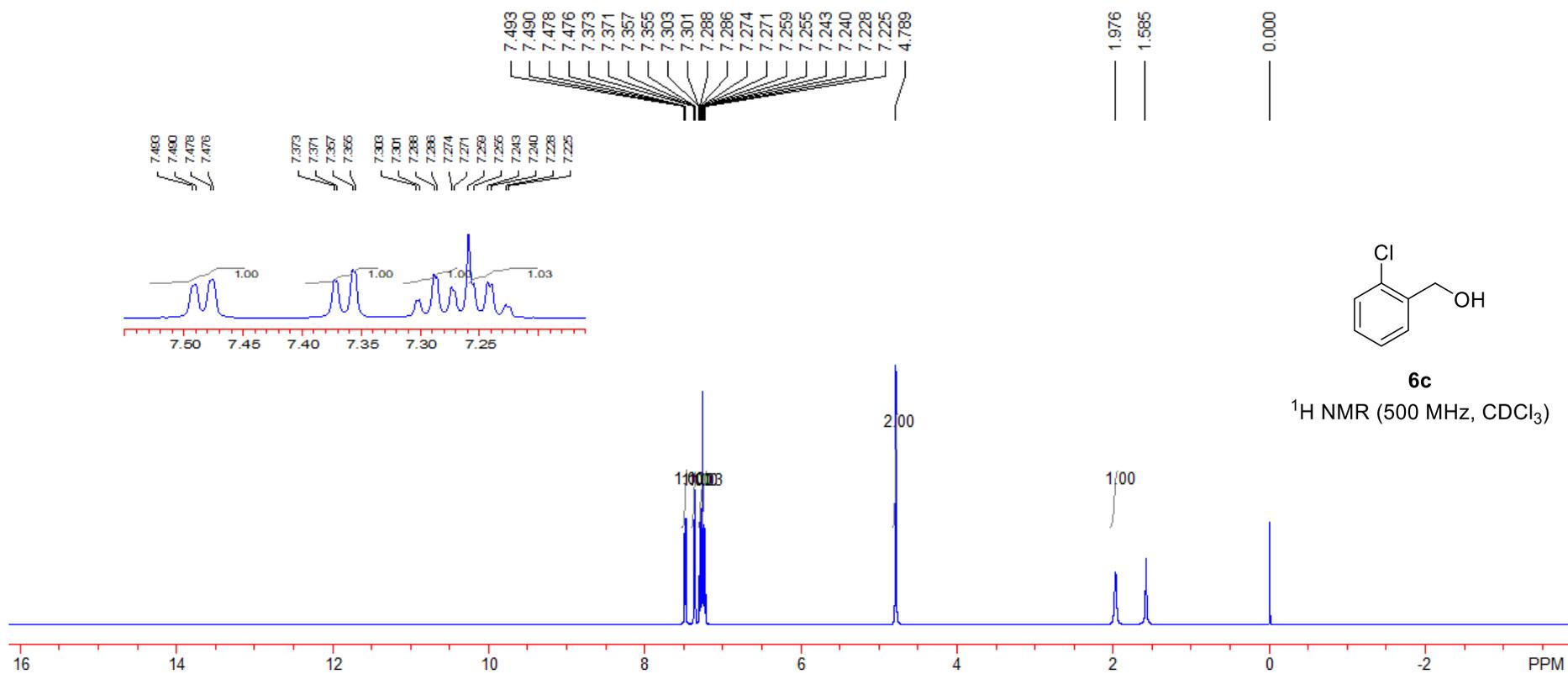


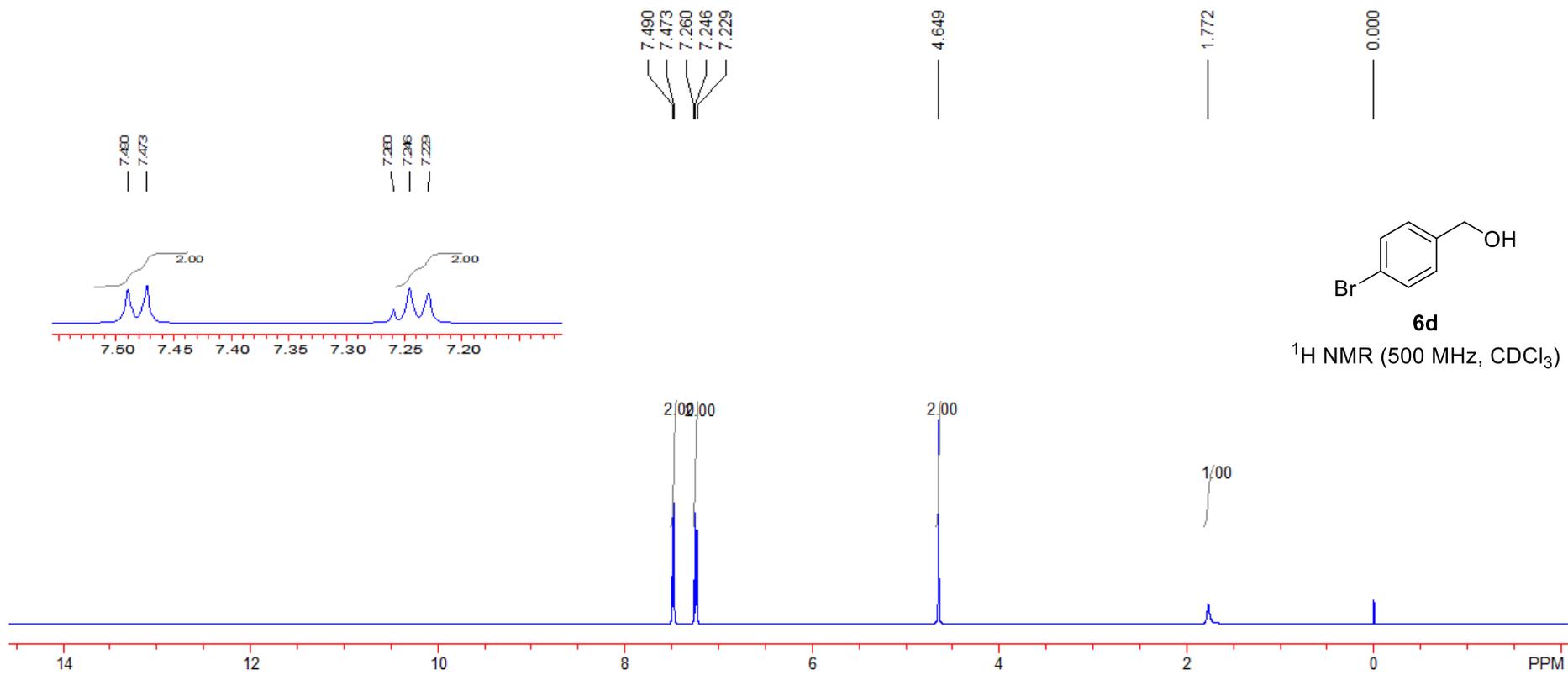


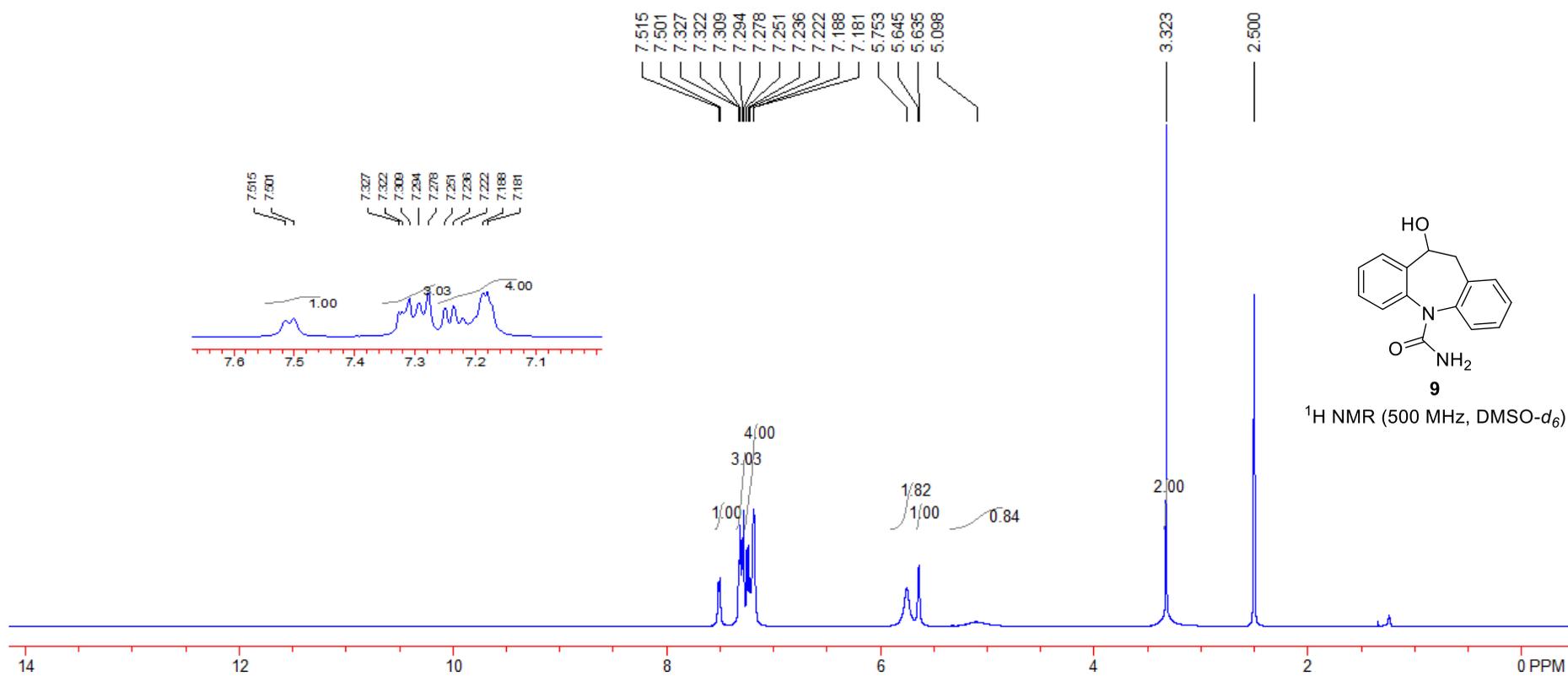


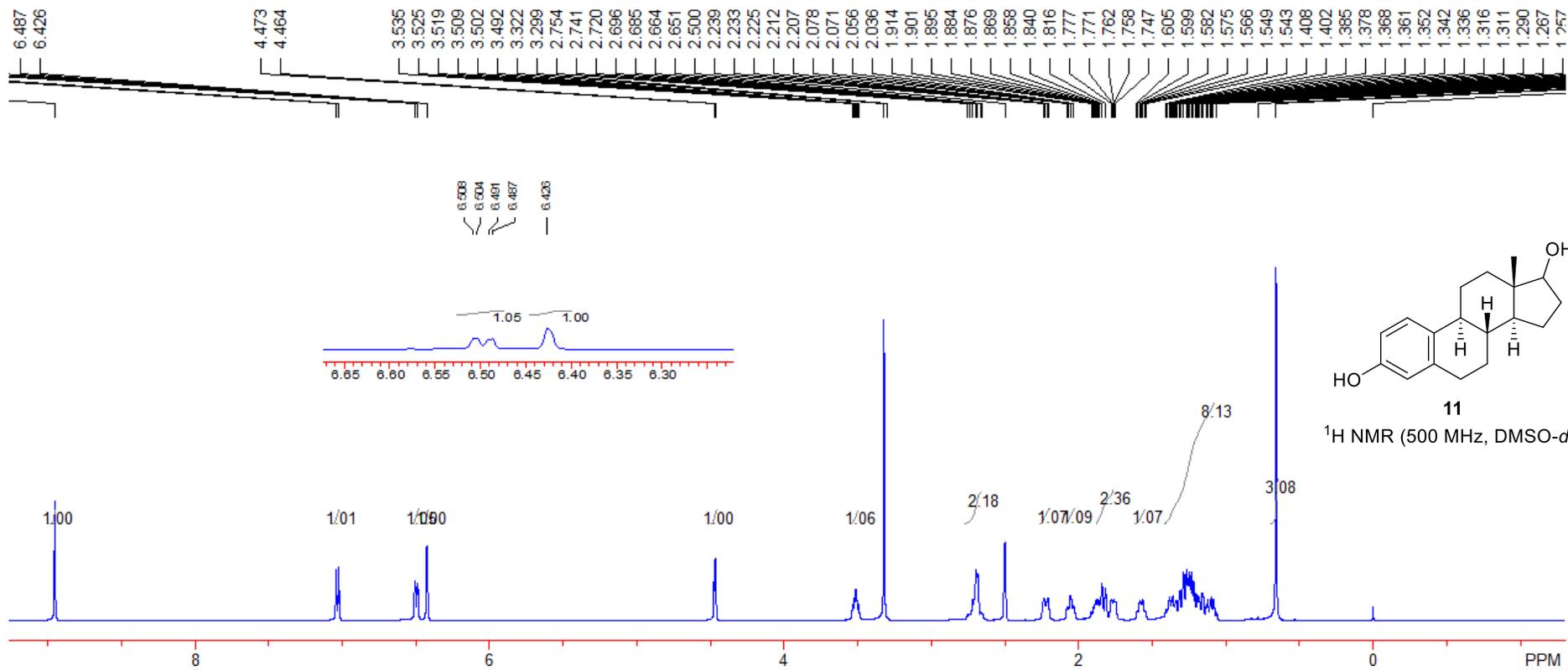


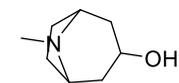












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¹H NMR (500 MHz, DMSO-d₆)

