Supporting information for

# Reduction of $\Delta^2$-Isoxazolines to *β*-Hydroxy Ketones with Iron and Ammonium Chloride as Reducing Agent

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## Contents

**1. General Procedure** S2
**2. Characterization Data of new compounds** S2
**3. $^1$H and $^{13}$C Spectra of Compound 1d** S4
**4. $^1$H and $^{13}$C Spectra of Compound 1j** S6
**5. $^1$H and $^{13}$C Spectra of Compound 1k** S8
**6. $^1$H and $^{13}$C Spectra of Compound 1l** S10
**7. $^1$H and $^{13}$C Spectra of Compound 2b** S12
**8. $^1$H and $^{13}$C Spectra of Compound 2c** S14
**9. $^1$H and $^{13}$C Spectra of Compound 2d** S16
**10. $^1$H and $^{13}$C Spectra of Compound 2e** S18
**11. $^1$H and $^{13}$C Spectra of Compound 2f** S20
**12. $^1$H and $^{13}$C Spectra of Compound 2g** S22
**13. $^1$H and $^{13}$C Spectra of Compound 2h** S24
**14. $^1$H and $^{13}$C Spectra of Compound 2i** S26
**15. $^1$H and $^{13}$C Spectra of Compound 2j** S28
**16. $^1$H and $^{13}$C Spectra of Compound 2k** S30
**17. $^1$H NMR Spectra of Compound 2l** S32

**1. General procedure for the reduction of $\Delta^2$-isoxazolines to *β*-hydroxy ketones with Fe/$NH_4Cl$ as reducing agent.** To a stirred solution of $\Delta^2$-isoxazoline **1a** (**1b-1l**, 0.3 mmol) and $NH_4Cl$ (161 mg, 3 mmol) in ethanol and water (1:1, 15 mL) was added Fe powder (168 mg, 3 mmol). The mixture was heated to 80 °C and was allowed to stir at this temperature for 6 hours. The reaction mixture was cooled to room temperature, diluted with ethyl acetate and filtered through a silica pad. The filtrate was washed with brine and the organic layer was separated, dried over $MgSO_4$, and evaporated in vacuo. The residue was then purified by flash chromatography on silica gel to give the desired product **2a** (**2b-2l**).

**2. Characterization data of new compounds.** Compounds **1a-1c** and **1e-1i** have been previously reported by our group and their characterizations were confirmed by comparison with those spectral data.

**5-(2-chlorobenzyl)-3-(4-methoxyphenyl)-2-isoxazoline (1d).** Light yellow oil. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 7.59-7.62 (2H, m), 7.33-7.37 (2H, m), 7.20-7.24 (2H, m), 6.90-6.93 (2H, m), 5.03-5.08 (1H, m), 3.84 (3H, s), 3.33 (1H, dd, *J* = 16.5, 10.1 Hz), 3.21 (1H, dd, *J* = 13.9, 6.9 Hz), 3.04-3.12 (2H, m); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 161.0, 156.1, 134.8, 134.2, 131.8, 129.5, 128.2, 128.2, 127.0, 122.2, 114.1, 79.8, 55.3, 39.7, 38.5; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{17}H_{16}ClNNaO_2$ 324.0762, found 324.0758.

**(E)-4-((3-styryl-2-isoxazoline-5-yl)methyl)benzaldehyde (1j).** This starting material contained a chromatography inseparable impurity of *p*-bromobenzyl alcohol and the mixture was used for the reaction(**1j**, 53%). **$^1$H NMR** (300 MHz, $CDCl_3$) δ 10.00 (1H, s), 7.85 (2H, d, *J* = 8.0 Hz), 7.44-7.47 (4H, m), 7.31-7.39 (3H, m), 7.04 (1H, d, *J* = 16.4 Hz), 6.69 (1H, d, *J* = 16.4 Hz), 4.95-5.01 (1H, m), 3.14-3.29 (2H, m), 2.87-3.04 (2H, m); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 191.8, 157.6, 144.1, 136.6, 135.7, 135.3, 130.1, 130.1, 129.0, 128.6, 127.0, 117.9, 81.2, 41.2, 38.2.

**5-(methoxymethyl)-3-p-tolyl-2-isoxazoline (1k).** White solid, mp 109-110 °C. **$^1$H NMR** (300 MHz, $CDCl_3$) δ7.55 (2H, dd, *J* = 6.6, 1.6 Hz), 7.18 (2H, dd, *J* = 6.6, 0.5 Hz), 4.83-4.89 (1H, m), 3.49-3.60 (2H, m), 3.41 (3H, s), 3.35 (1H, dd, *J* = 16.6, 10.7 Hz), 3.20 (1H, dd, *J* = 16.6, 7.6 Hz), 2.36 (3H, s); . **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 156.2, 140.1, 129.2, 126.6, 126.5, 79.4, 73.4, 59.3, 37.2, 21.3; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{12}H_{15}NNaO_2$ 228.0995, found 228.0991.

**5-hydroxymethyl-3-p-tolyl-2-isoxazoline (1l).** Colorless oil. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 7.55 (2H, d, *J* = 8.2 Hz), 7.20 (2H, d, *J* = 8.2 Hz), 4.83-4.87 (1H, m), 3.83-3.86 (1H, m), 3.68-3.72 (1H, m), 3.37 (1H, dd, *J* = 16.6, 10.6 Hz), 3.25 (1H, dd, *J* = 16.6, 7.9 Hz), 2.38 (3H, s), 2.07 (1H, t, *J* = 7.0 Hz); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 157.0, 140.5, 129.4, 126.6, 126.5, 81.0, 63.7, 36.4, 21.4; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{11}H_{13}NNaO_2$ 214.0838, found 214.0843.

**4-(2-chlorophenyl)-3-hydroxy-1-(4-methoxyphenyl) butan-1-one (2d).** Yellowish oil. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 7.91 (2H, d, *J* = 8.9 Hz), 7.33-7.39 (2H, m), 7.17-7.22 (2H, m), 6.92 (2H, d, *J* = 8.9 Hz), 4.51-4.56 (1H, m), 3.86 (3H, s), 3.51 (1H, br), 3.18 (1H, dd, *J* = 17.6, 3.0 Hz), 3.02-3.11 (3H, m); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 199.1, 163.8, 135.9, 134.3, 131.9, 130.4, 129.7, 129.6, 128.0, 126.7, 113.8, 67.7, 55.5, 43.6, 40.1; **IR** (film, $cm^{-1}$) 3481, 3058, 3006, 2958, 2931, 2838, 1665, 1597, 1572, 1508, 1472, 1418, 1259, 1169, 1030, 830, 750; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{17}H_{17}ClNaO_3$ 327.0758, found 327.0755.

**1-(2-(benzyloxy)phenyl)-3-hydroxy-4-(pyridin-3-yl) butan-1-one (2e).** Yellow oil. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 8.42-8.44 (1H, m), 8.32-8.33 (1H, m), 7.75-7.78 (1H, m), 7.41-7.47 (7H, m), 7.15 (1H, m), 7.02-7.04 (2H, m), 5.13 (2H, s), 4.31-4.34 (1H, m), 3.22 (1H, dd, *J* = 17.9, 2.6 Hz), 3.01 (1H, dd, *J* = 17.9, 9.2 Hz), 2.71 (1H, dd, *J* = 13.9, 7.2 Hz), 2.59 (1H, dd, *J* = 13.9, 5.6 Hz);

**$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 202.2, 158.2, 158.5, 147.6, 137.1, 135.8, 134.3, 133.8, 130.5, 128.8, 128.6, 127.9, 127.6, 123.2, 121.1, 112.8, 70.8, 68.6, 49.8, 39.7; **IR** (film, $cm^{-1}$) 3408, 3063, 3031, 2920, 2758, 1668, 1593,1575, 1495, 1446, 1289, 1234, 1077, 1002, 754, 733; **HRMS-ESI** (*m/z*): $[M+H]^+$ calcd for $C_{22}H_{22}NO_3$ 348.1594, found 348.1586.

***tert*-butyl 4-(4-(2,4-dimethoxyphenyl)-2-hydroxy-4-oxobutyl)benzoate (2f).** Yellow oil. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 7.92 (2H, d, *J* = 8.1 Hz), 7.81 (1H, d, *J* = 8.8 Hz), 7.30 (2H, d, *J* = 8.1 Hz), 6.52 (1H, dd, *J* = 8.8, 2.1 Hz), 6.42 (1H, d, *J* = 2.1 Hz), 4.37-4.44 (1H, m), 3.85 (3H, s), 3.81 (3H, s), 3.51 (1H, br), 3.20 (1H, dd, *J* = 18.0, 2.5 Hz), 2.93-3.02 (2H, m), 2.84 (1H, dd, *J* = 13.5, 6.2 Hz), 1.58 (9H, s); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 200.1, 165.8, 165.0, 161.2, 143.5, 132.7, 130.3, 129.5, 129.4, 120.5, 105.4, 98.4, 80.8, 69.0, 55.5, 55.4, 49.2, 42.9, 28.2; **IR** (film, $cm^{-1}$) 3503, 3089, 2973, 2934, 2838, 1707, 1656, 1596, 1571, 1497, 1461, 1414, 1365, 1292, 1252, 1210, 1161, 1113, 1026, 845; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{23}H_{28}NaO_6$ 423.1778, found 423.1779.

**2-hydroxy-1-(p-biphenyl)heptan-4-one (2g).** White solid, mp 54-55 °C. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 7.53-7.60 (4H, m), 7.41-7.46 (2H, m), 7.28-7.36 (3H, m), 4.30-4.39 (1H, m), 3.09 (1H, br), 2.89 (1H, dd, *J* = 13.7, 7.2 Hz), 2.77 (1H, dd, *J* = 13.7, 6.1 Hz), 2.57-2.62 (2H, m), 2.40 (2H, t, *J* = 7.4 Hz), 1.57-1.67 (2H, m), 0.91 (3H, t, *J* = 7.4 Hz); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 211.9, 140.9, 139.5, 137.0, 129.8, 128.7, 127.2, 127.2, 127.0, 68.7, 48.1, 45.5, 42.5, 17.0, 13.6; **IR** (film, $cm^{-1}$) 3358, 3052, 3025, 2956, 2927, 2870, 1706, 1595, 1517, 1483, 1404, 1381, 1095, 760, 695; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{19}H_{22}NaO_2$ 305.1512, found 305.1500.

***tert*-butyl 4-(2-hydroxy-4-oxoheptyl)benzoate (2h).** Colorless oil. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 7.92 (2H, d, *J* = 8.2 Hz), 7.25 (2H, d, *J* = 8.2 Hz), 4.26-4.32 (1H, m), 3.10 (1H, br), 2.88 (1H, dd, *J* = 13.6, 7.2 Hz), 2.76 (1H, dd, *J* = 13.6, 6.1 Hz), 2.51-2.55 (2H, m), 2.36 (2H, t, *J* = 7.3 Hz), 1.58 (9H, s), 1.52-1.61 (2H, m), 0.89 (3H, t, *J* = 7.4 Hz); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 211.8, 165.7, 142.8, 130.4, 129.6, 129.2, 80.9, 68.4, 48.0, 45.5, 42.7, 28.2, 17.0, 13.6; **IR** (film, $cm^{-1}$) 3469, 2963, 2930, 1707, 1608, 1455, 1365, 1308, 1290, 1164, 1114, 1018, 763; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{18}H_{26}NaO_4$ 329.1723, found 329.1726.

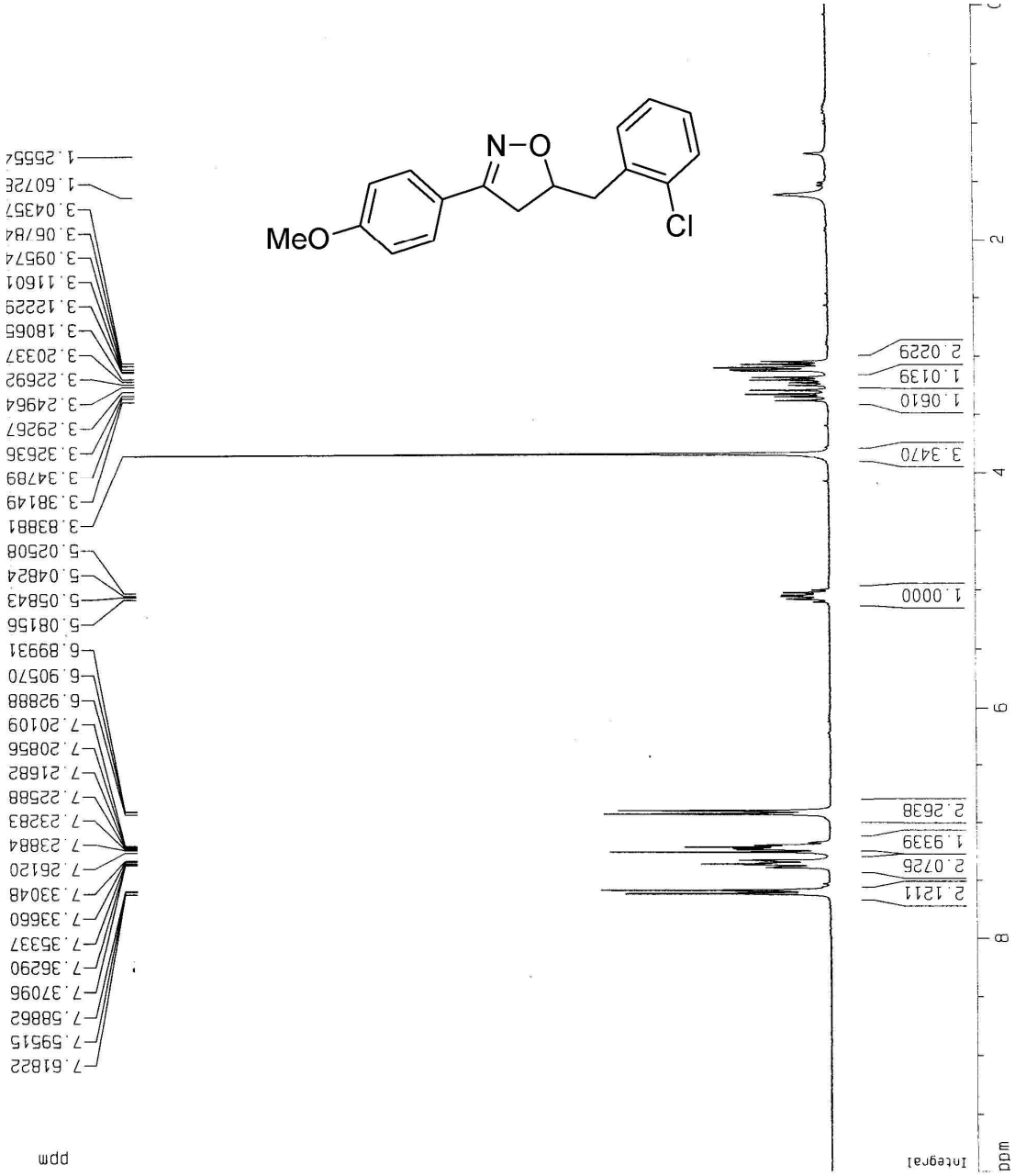
**(E)-6-(3,4-dichlorophenyl)-5-hydroxy-1-phenylhex-1-en-3-one (2i).** White solid, mp 83-86 °C. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 7.53-7.55 (2H, m), 7.54 (1H, d, *J* = 16.2 Hz), 7.36-7.41 (5H, m), 7.09-7.12 (1H, m), 6.70 (1H, d, *J* = 16.2 Hz), 4.36-4.38 (1H, m), 3.35 (1H, br), 2.72-2.90 (4H, m); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 200.1, 143.8, 138.4, 134.0, 132.3, 131.3, 130.9, 130.5, 130.3, 129.0, 128.9, 128.4, 126.0, 68.4, 46.0, 41.8; **IR** (KBr, $cm^{-1}$) 3501, 3060, 3024, 2924, 2882, 1678, 1604, 1572, 1491, 1470, 1375, 1105, 1071, 813, 753; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{18}H_{16}Cl_2NaO_2$ 357.0420, found 357.0416.

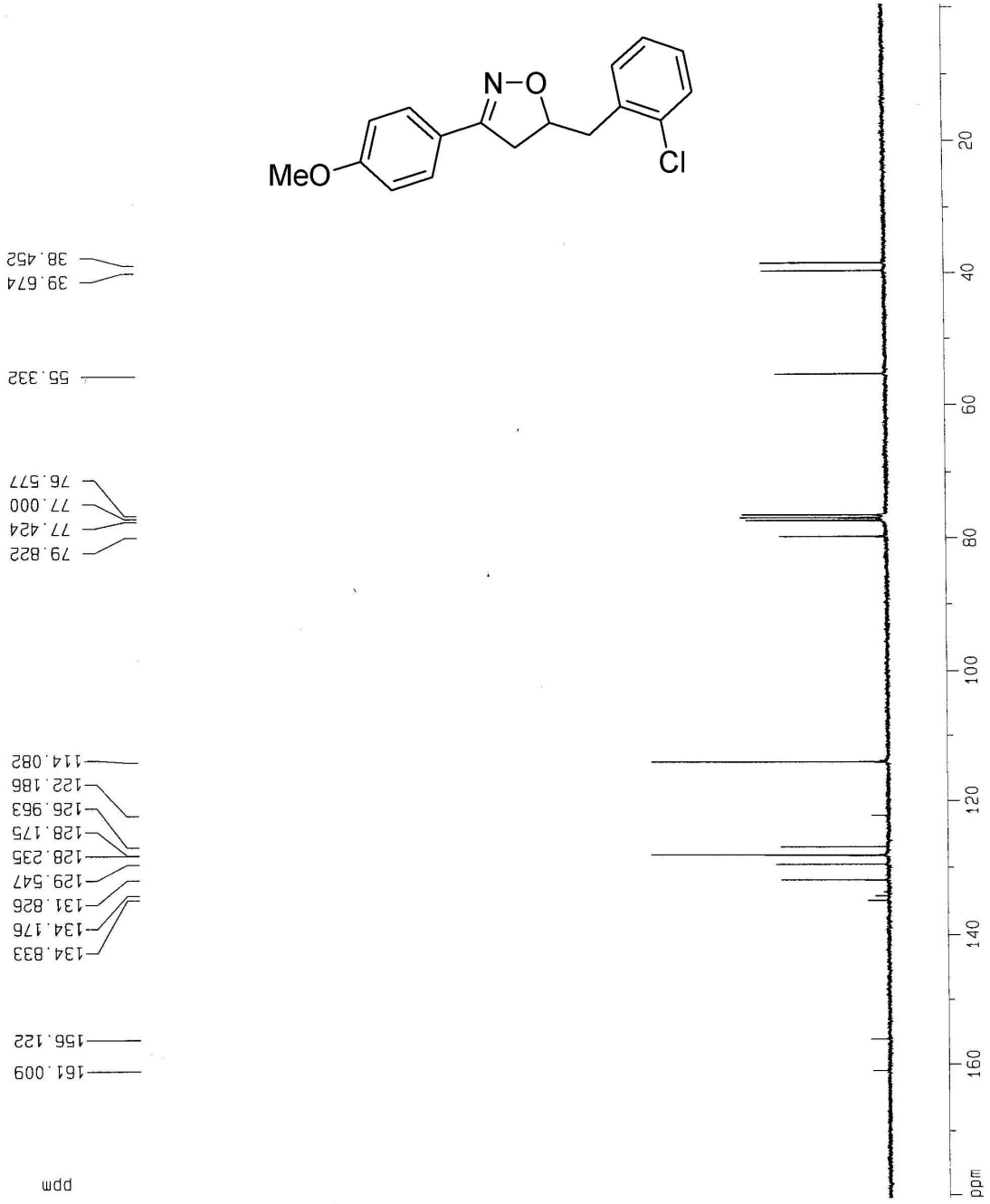
**(E)-4-(2-hydroxy-4-oxo-6-phenylhex-5-enyl) benzaldehyde (2j).** Yellow solid, mp 118-120 °C. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 9.99 (1H, s), 7.84 (2H, d, *J* = 8.1 Hz), 7.52-7.57 (3H, m), 7.38-7.45 (5H, m), 6.70 (1H, d, *J* = 16.2 Hz), 4.43-4.46 (1H, m), 3.35 (1H, br), 2.76-3.03 (4H, m); **$^{13}$C NMR** (75 MHz, $CDCl_3$) δ 200.1, 191.8, 145.5, 143.8, 135.1, 134.1, 130.9, 130.2, 129.9, 129.0, 128.4, 126.1, 68.5, 46.2, 43.0; **IR** (KBr, $cm^{-1}$) 3352, 2934, 2916, 2819, 2730, 1692, 1660, 1603, 1574, 1491, 1447, 1377, 1186, 1092, 810, 745; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{19}H_{18}NaO_3$ 317.1148, found 317.1136.

**3-hydroxy-4-methoxy-1-p-tolylbutan-1-one (2k).** White solid, mp 54-55 °C. **$^1$H NMR** (300 MHz, $CDCl_3$) δ 7.86 (2H, d, *J* = 8.1 Hz), 7.25 (2H, d, *J* = 8.1 Hz), 4.38-4.42 (1H, m), 3.43-3.52 (2H, m), 3.40 (3H, s), 3.30 (1H, br), 3.16 (2H, d, *J* = 6.1 Hz), 2.40 (3H, s); **$^{13}$C NMR** (75 MHz,

$CDCl_3$) δ 199.5, 144.4, 134.3, 129.3, 128.2, 75.8, 66.9, 59.1, 41.5, 21.6; **IR** (KBr, $cm^{-1}$) 3457, 3410, 2915, 2884, 2846, 1679, 1602, 1570, 1451, 1404, 1373, 1271, 1179, 1119, 1072, 812; **HRMS-ESI** (*m/z*): $[M+Na]^+$ calcd for $C_{12}H_{16}NaO_3$ 231.0992, found 231.1001.

## 3. $^1$H and $^{13}$C Spectra of Compound 1d

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128.235
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140
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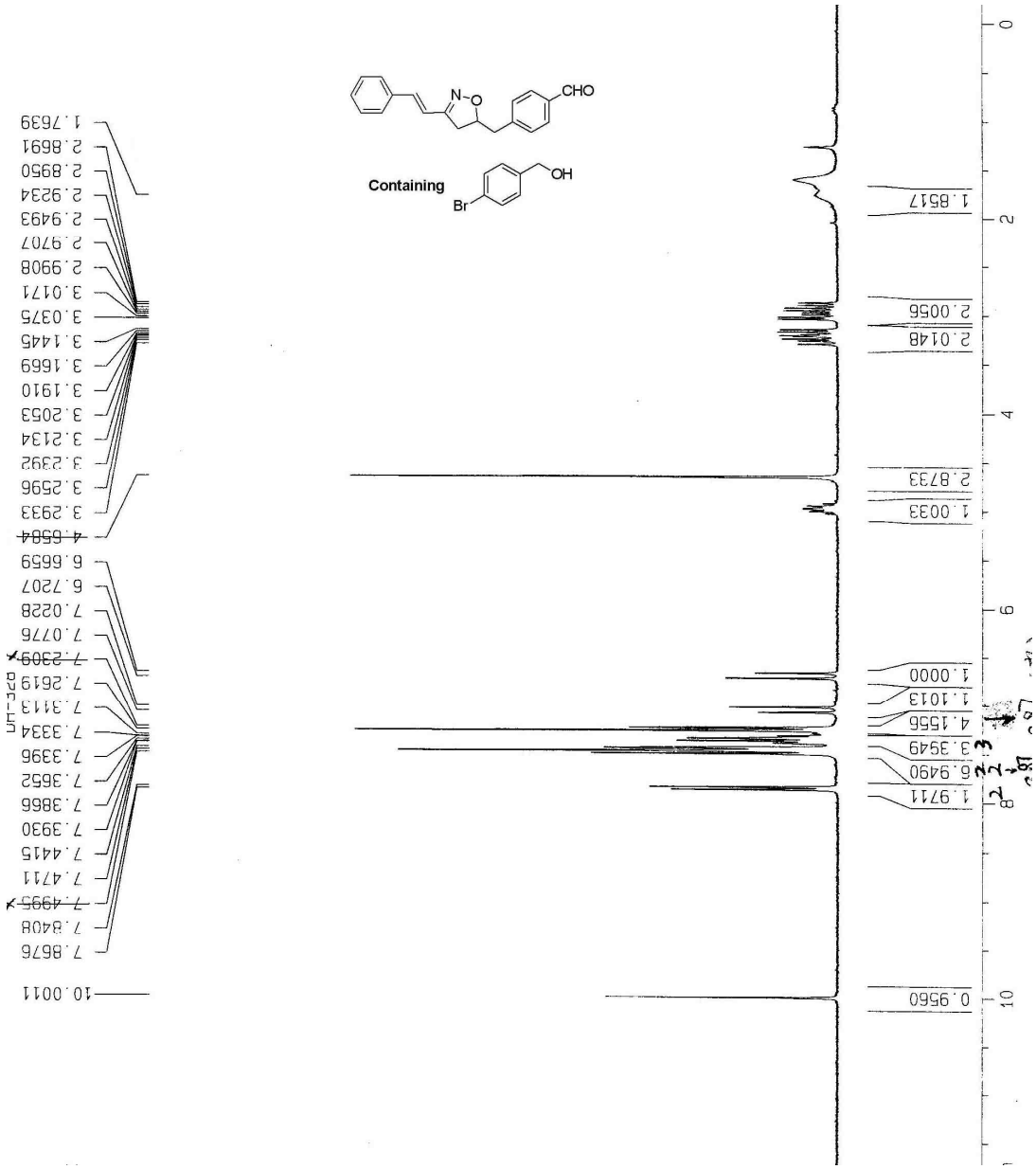
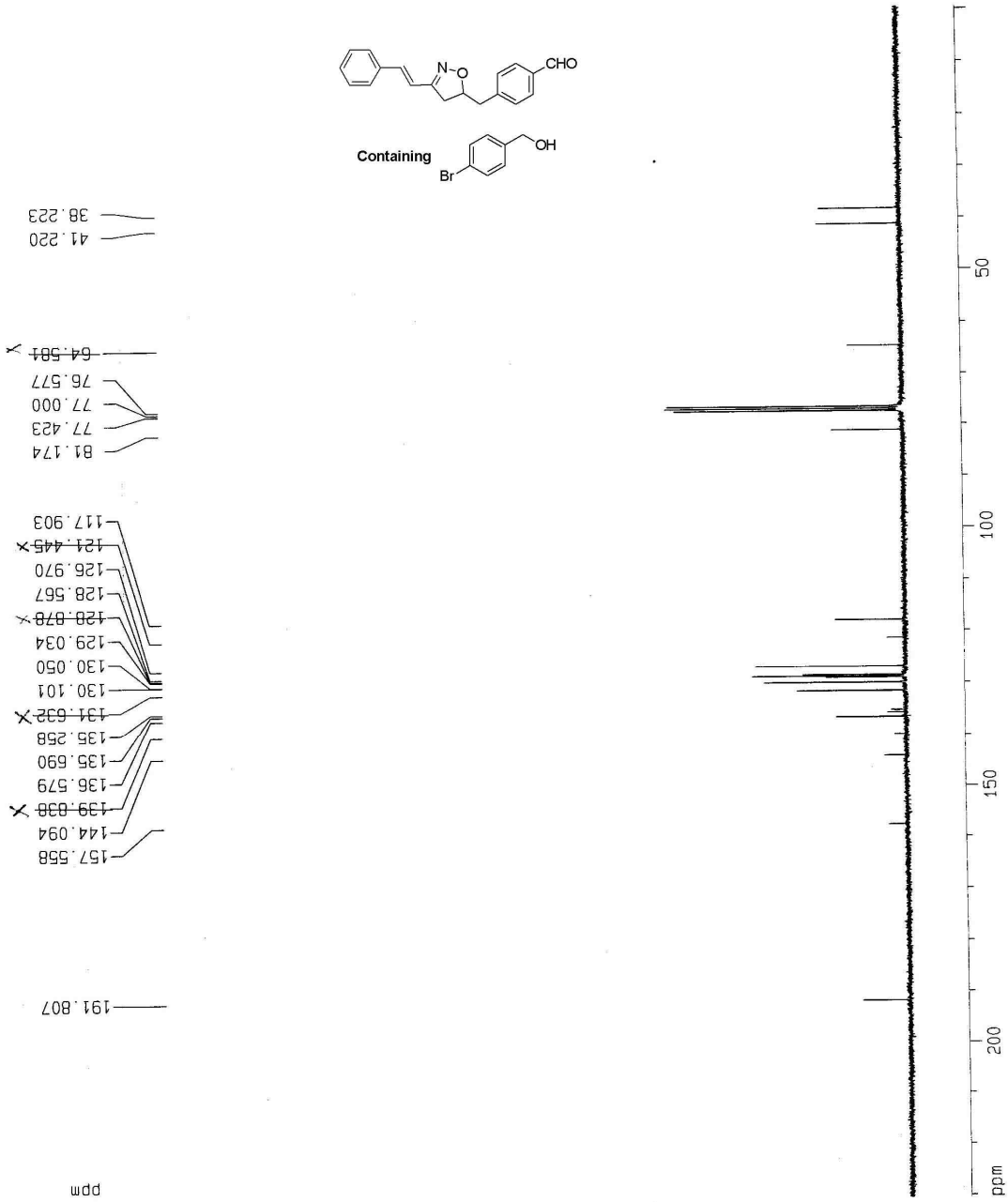
ppm

## 4. $^{1}H$ and $^{13}C$ Spectra of Compound 1j

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126.970
121.445
117.903
81.174
77.423
77.000
76.577
64.581
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OH
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O
CHO
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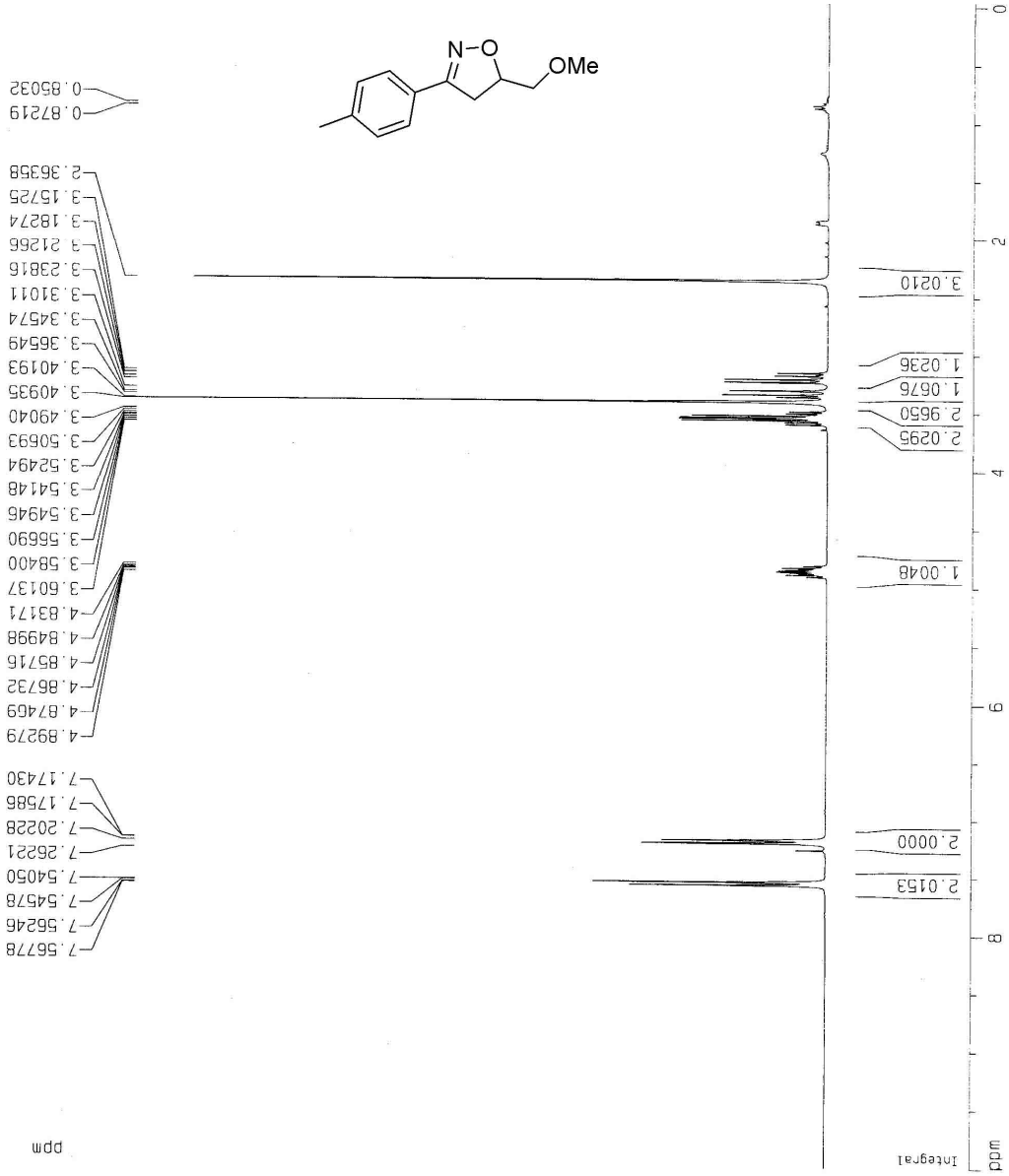
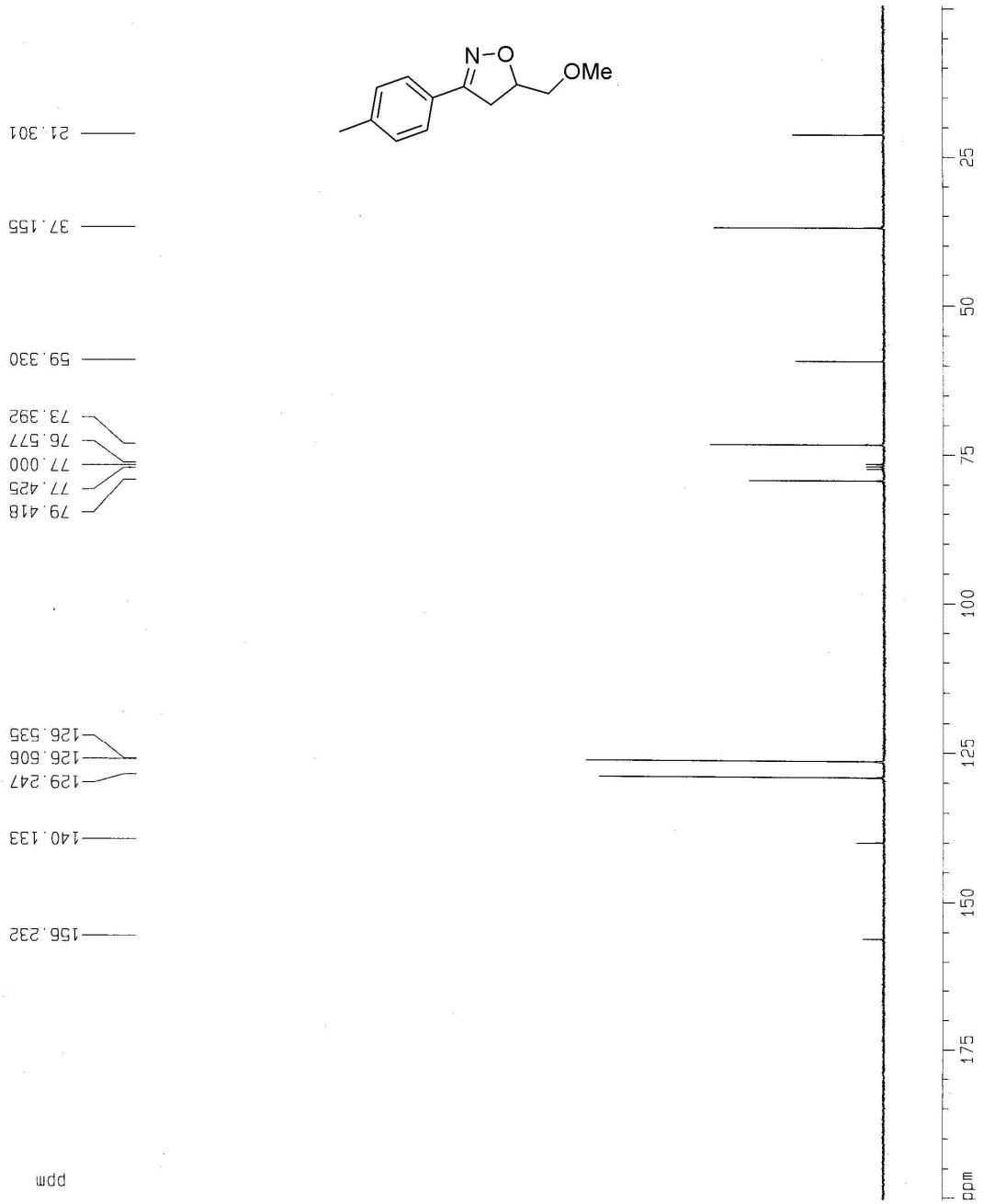
100
50

## 5. $^{1}$H and $^{13}$C Spectra of Compound 1k

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77.000
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25
50
75
100
125
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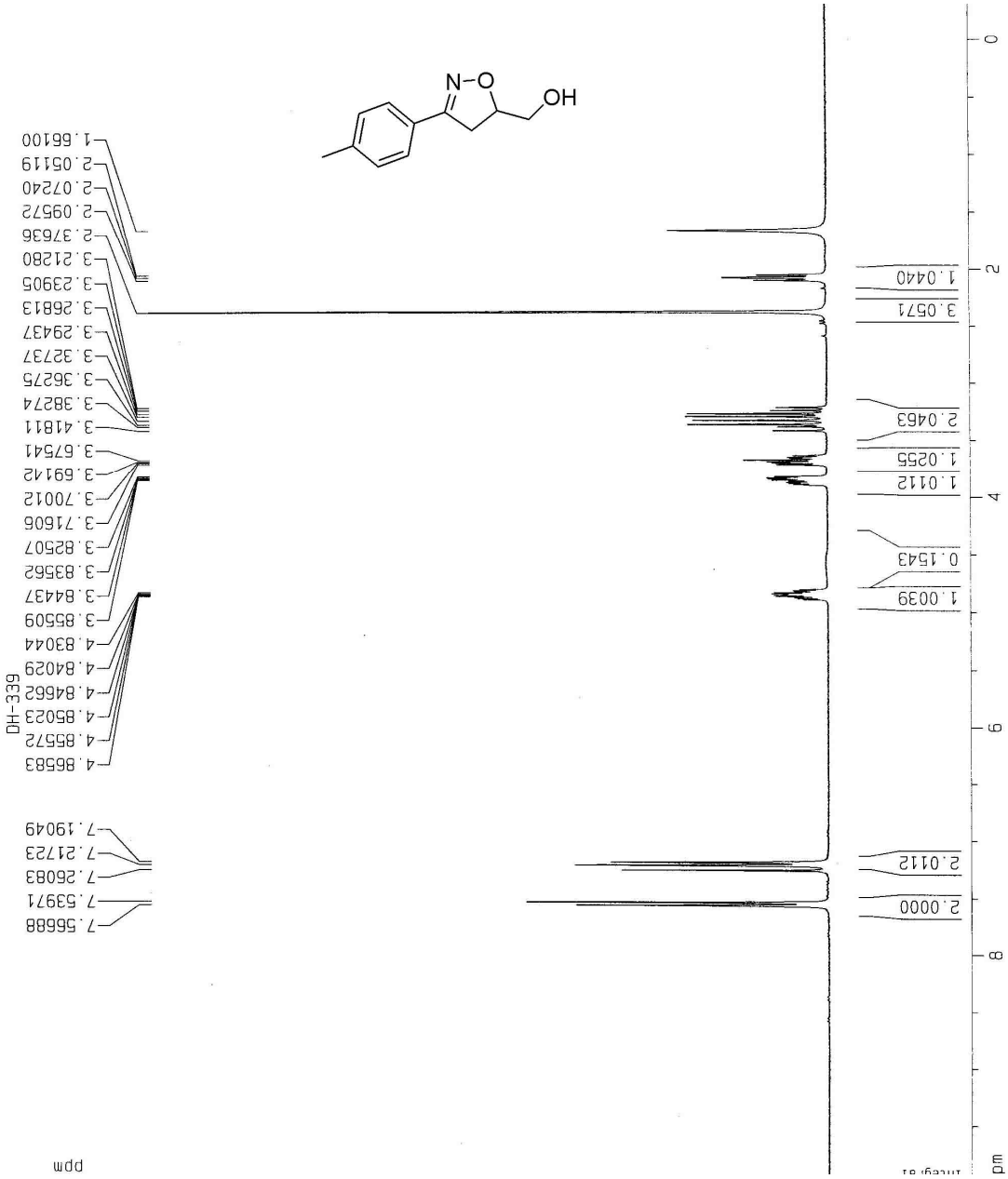
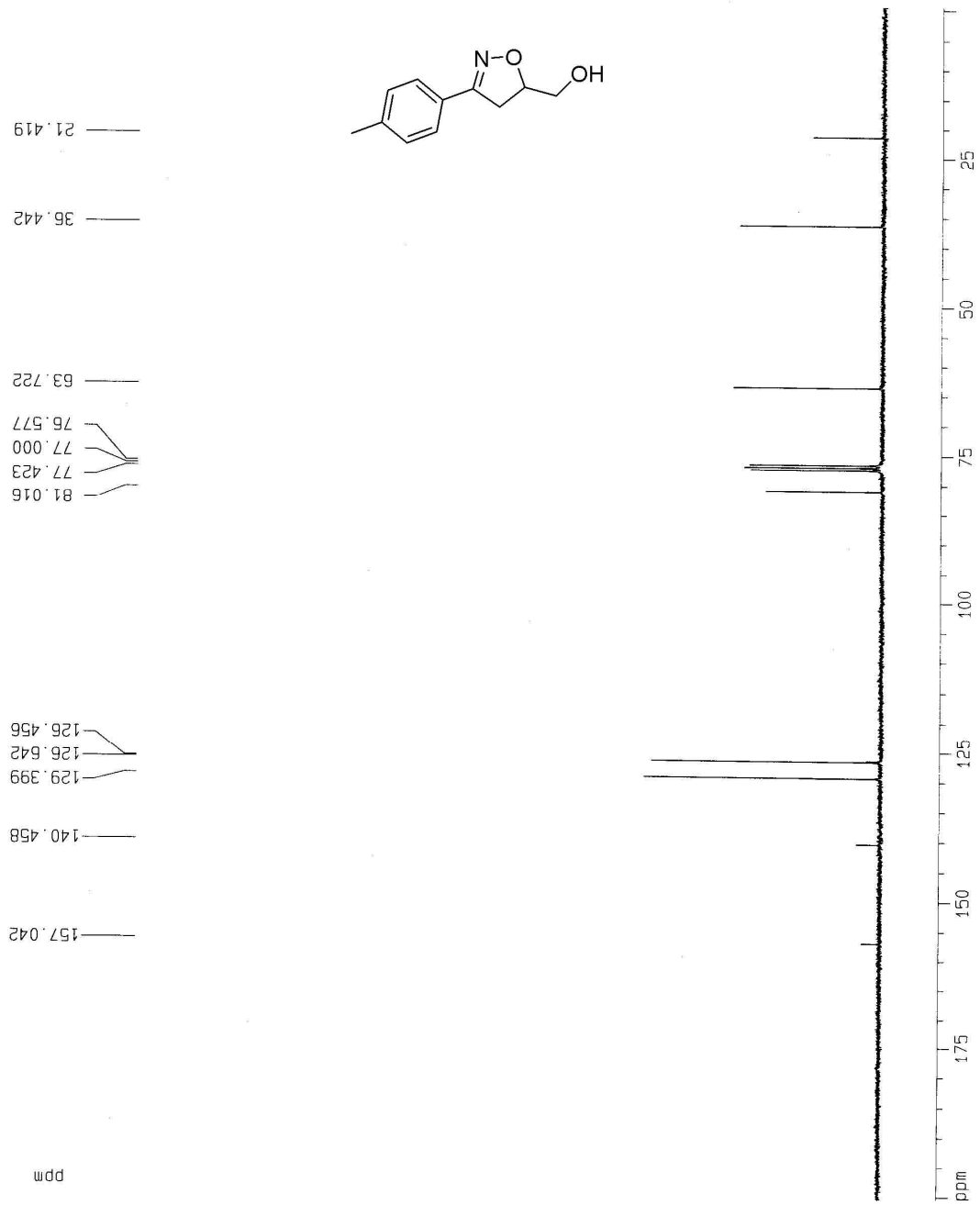
175
ppm

## 6. $^{1}$H and $^{13}$C Spectra of Compound 1l

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77.423
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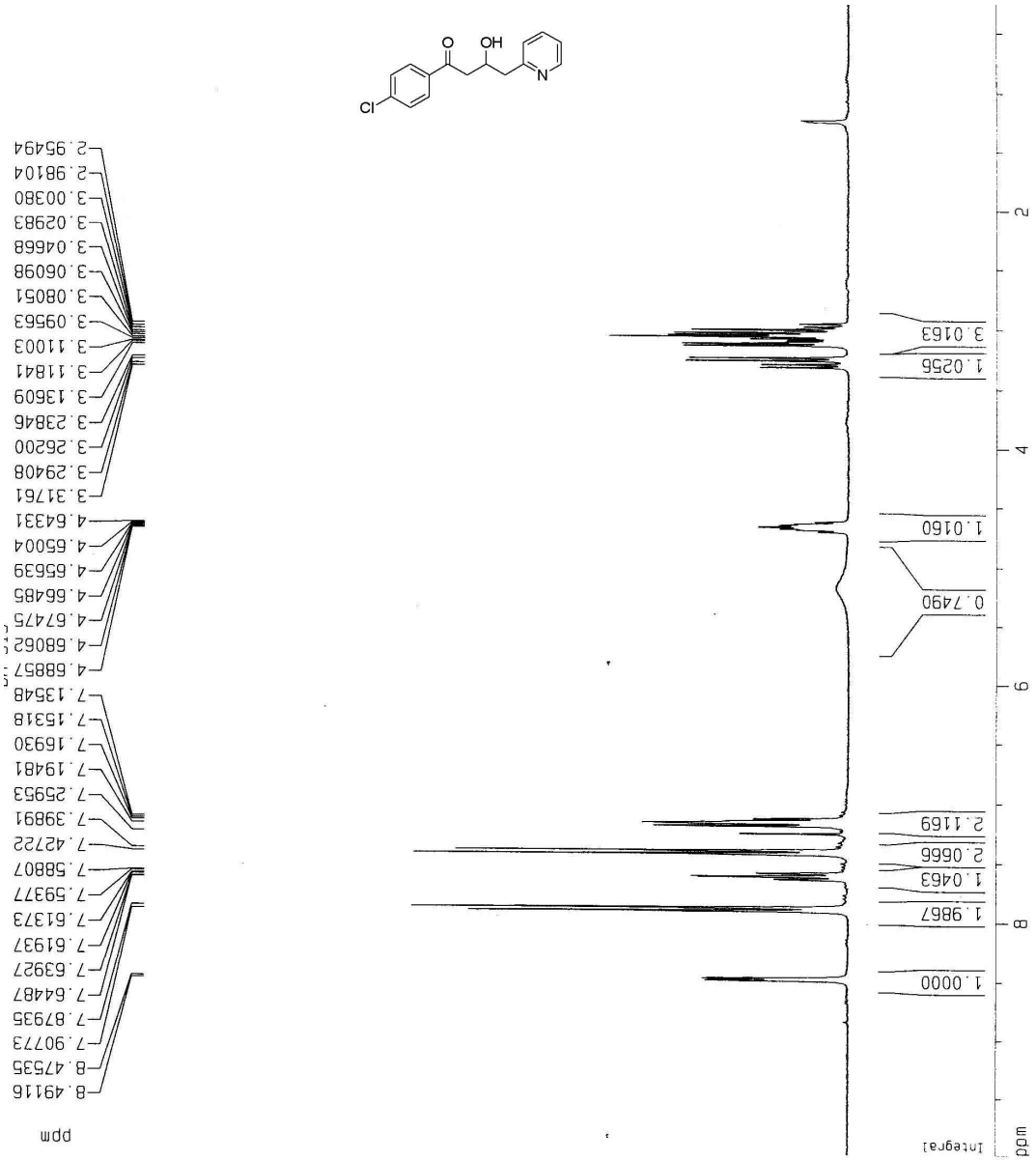
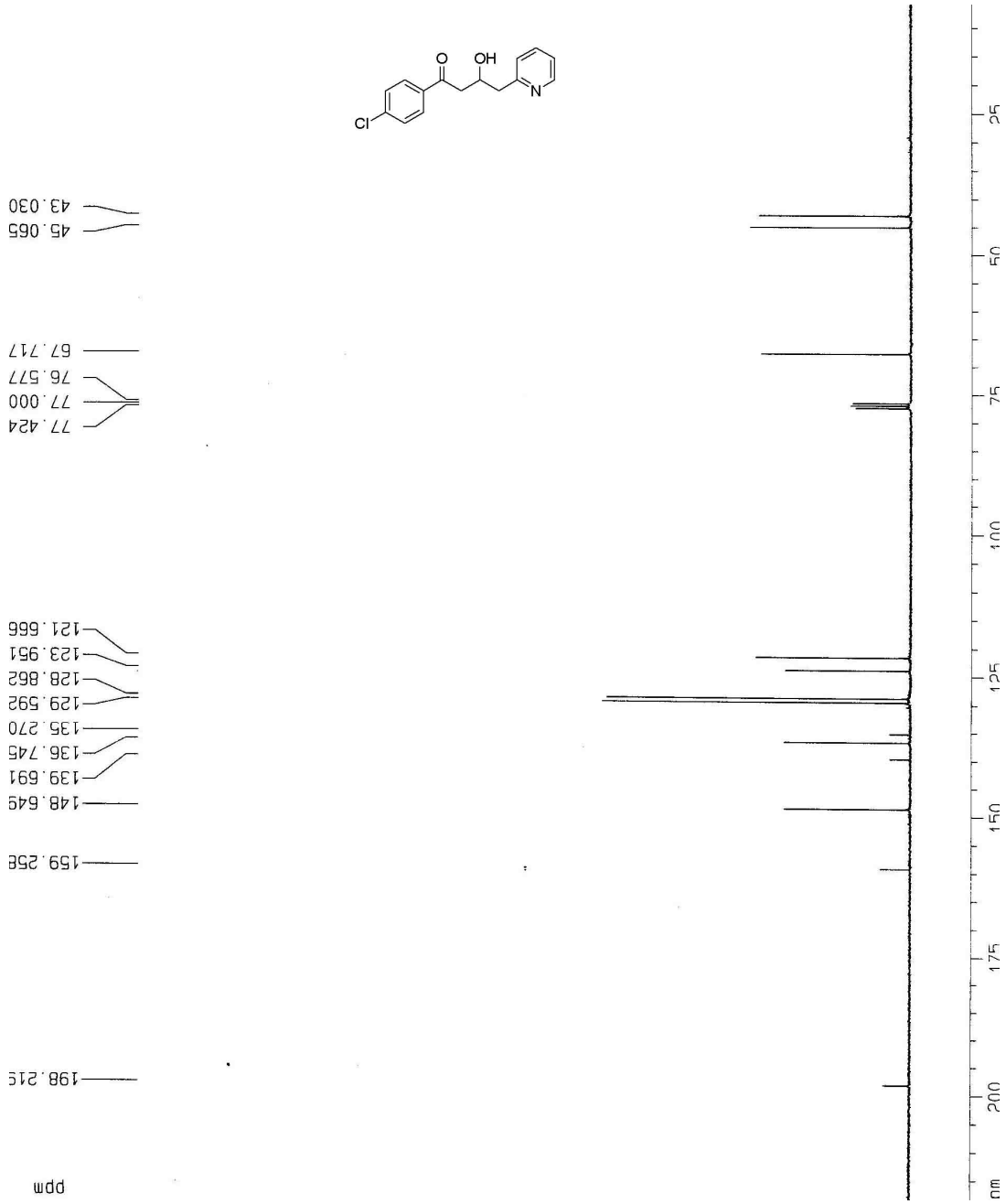
ppm

## 7. $^{1}$H and $^{13}$C Spectra of Compound 2b

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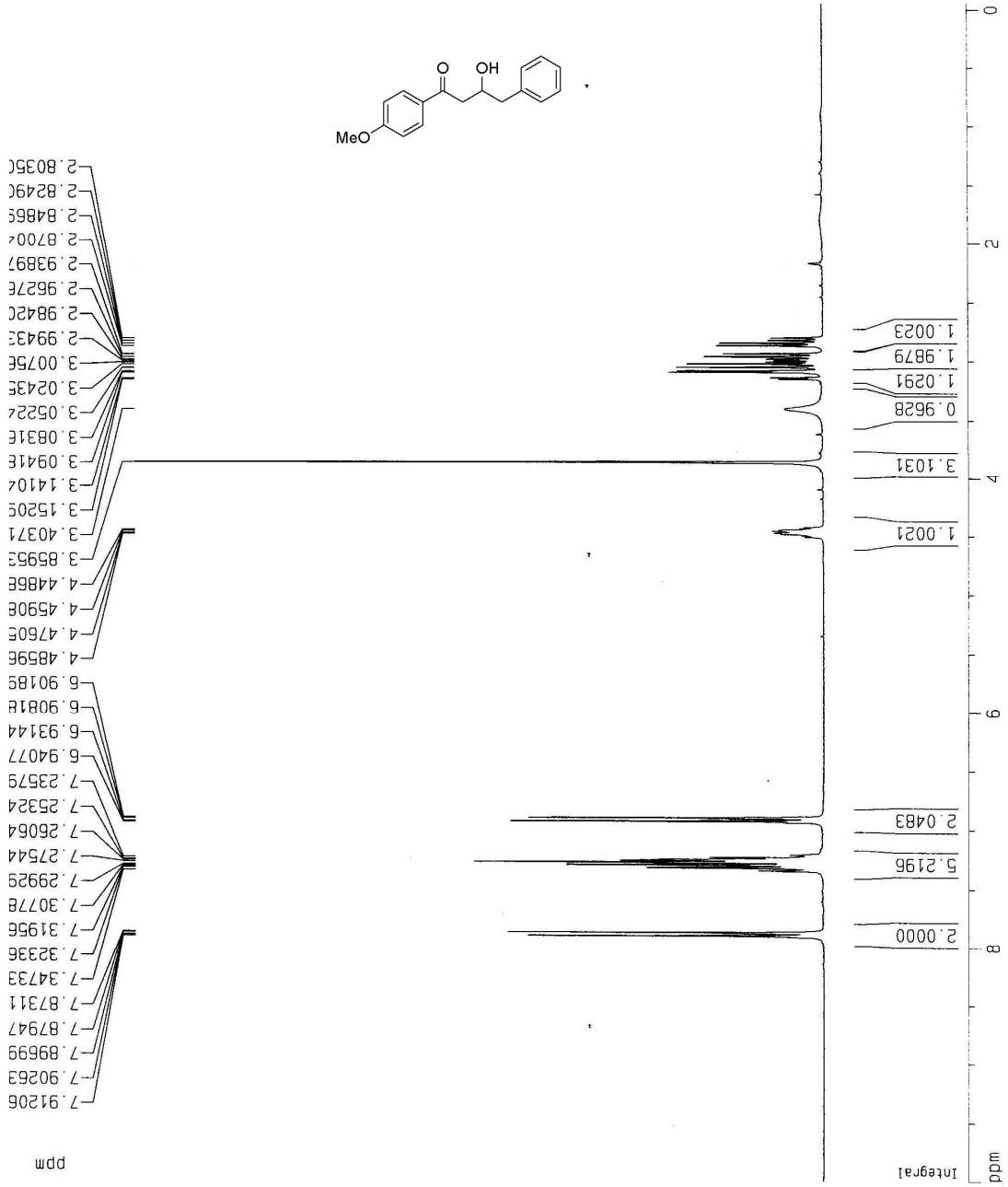
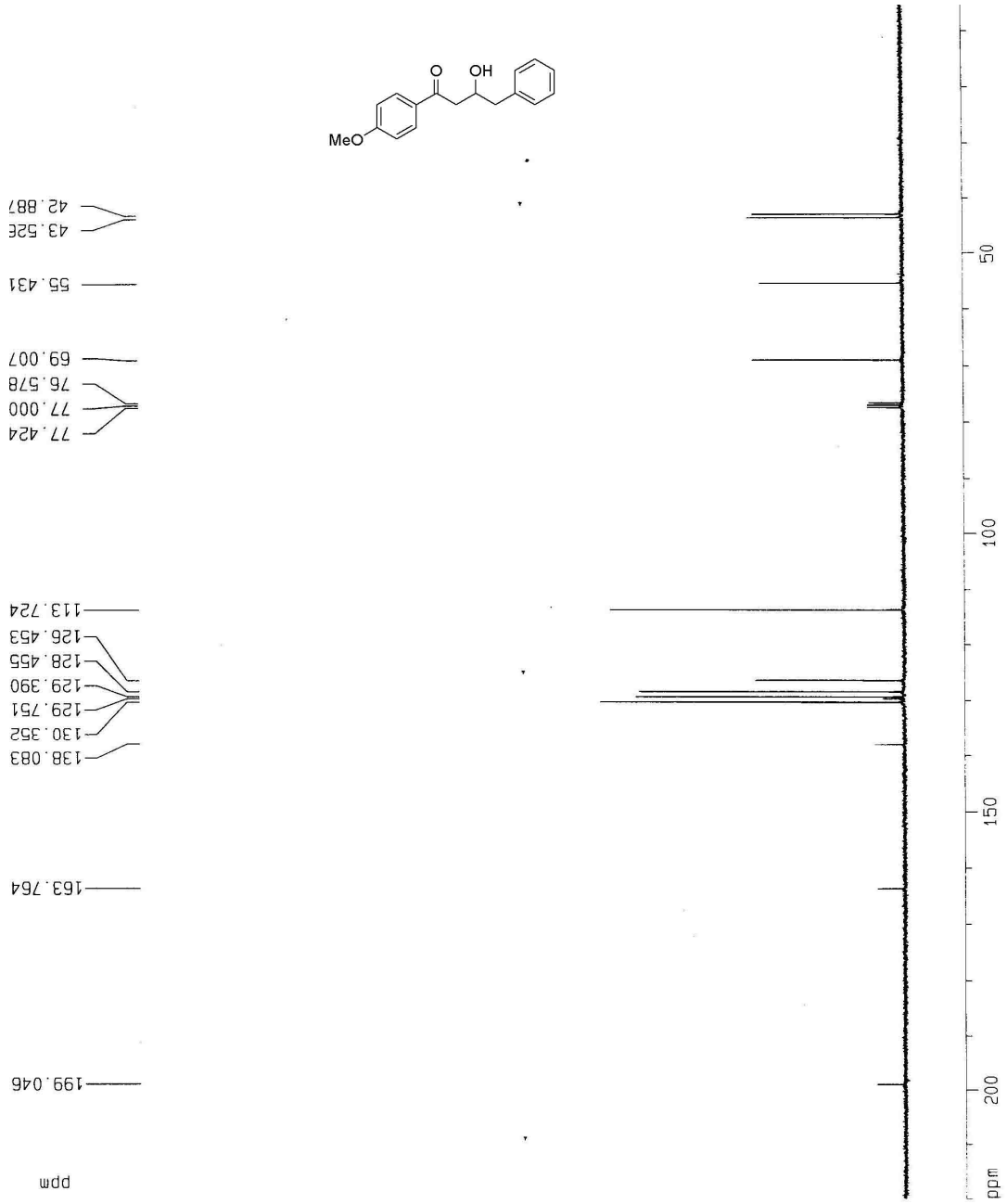
Cl
O
OH
N

## 8. $^{1}$H and $^{13}$C Spectra of Compound 2c

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76.578
69.007
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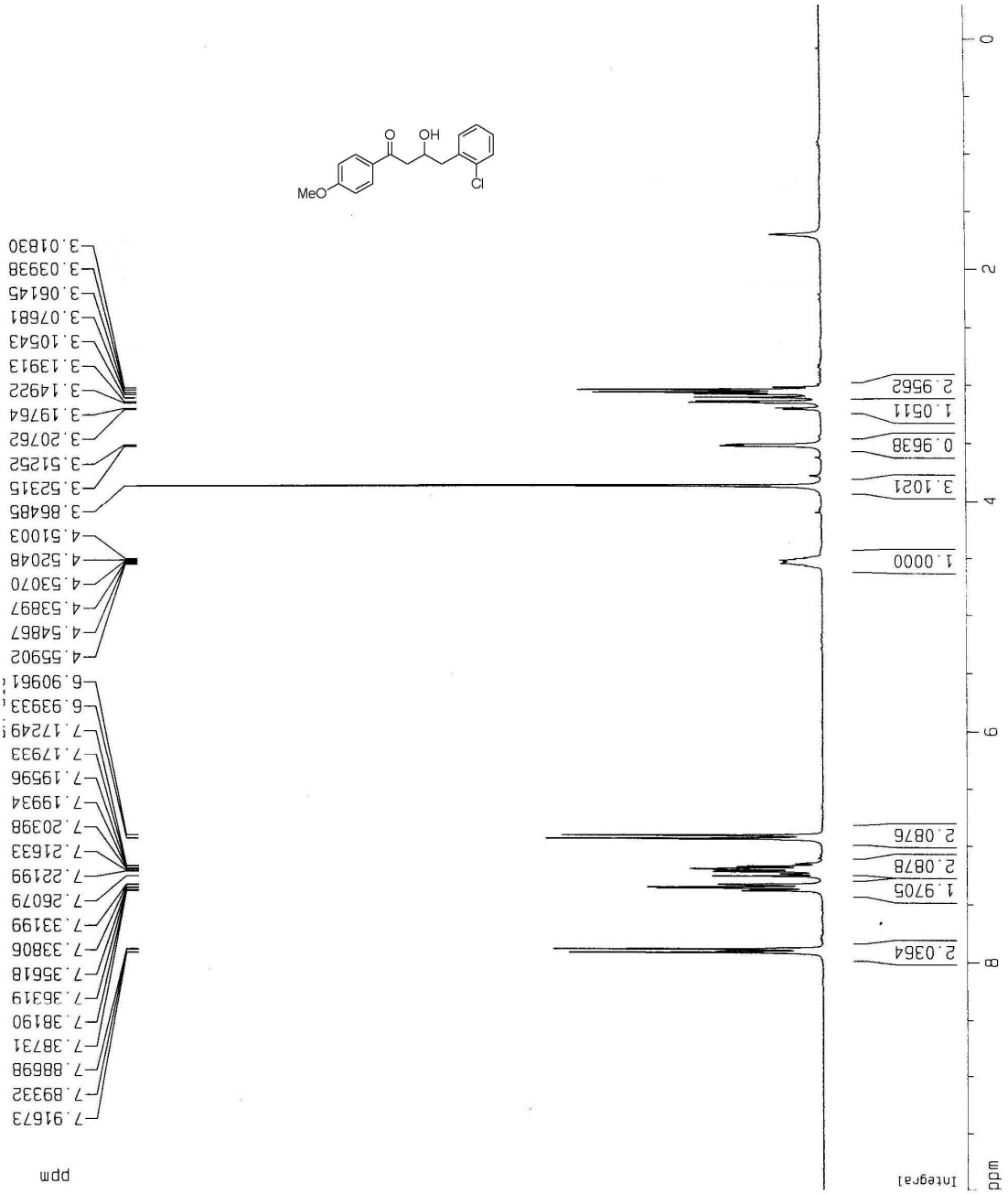
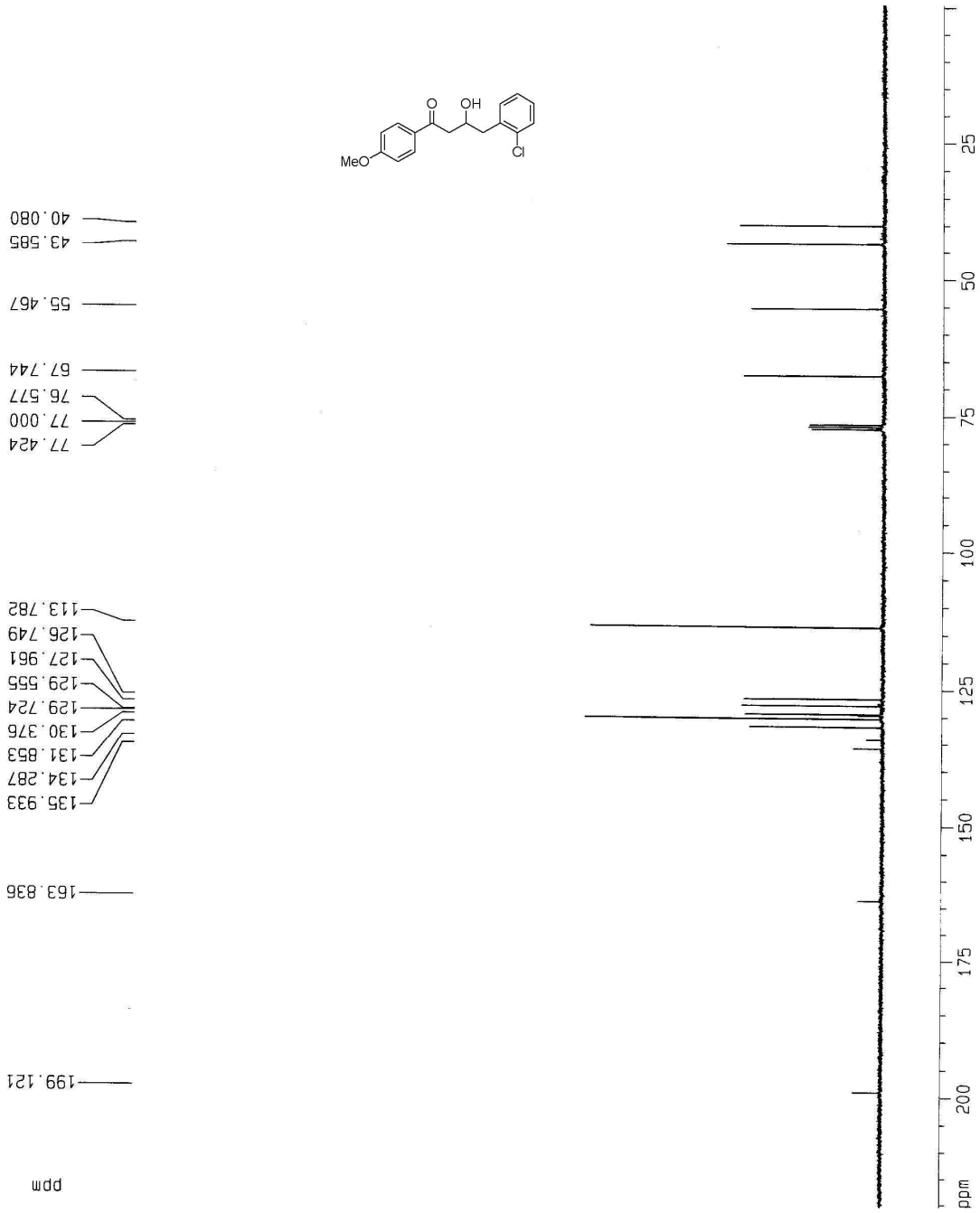
MeO
O
OH
ppm
200
150
100
50

## 9. $^{1}$H and $^{13}$C Spectra of Compound 2d

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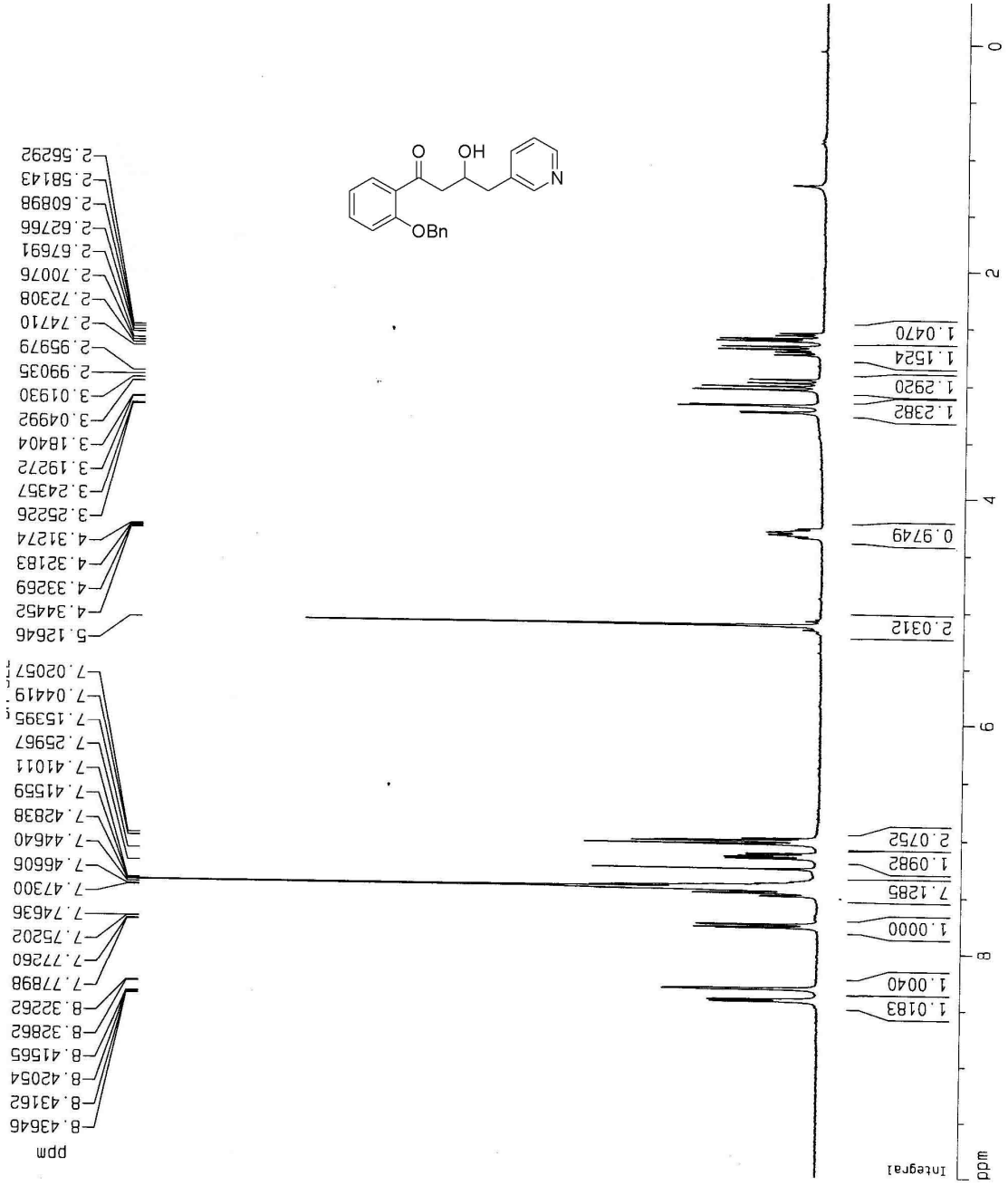
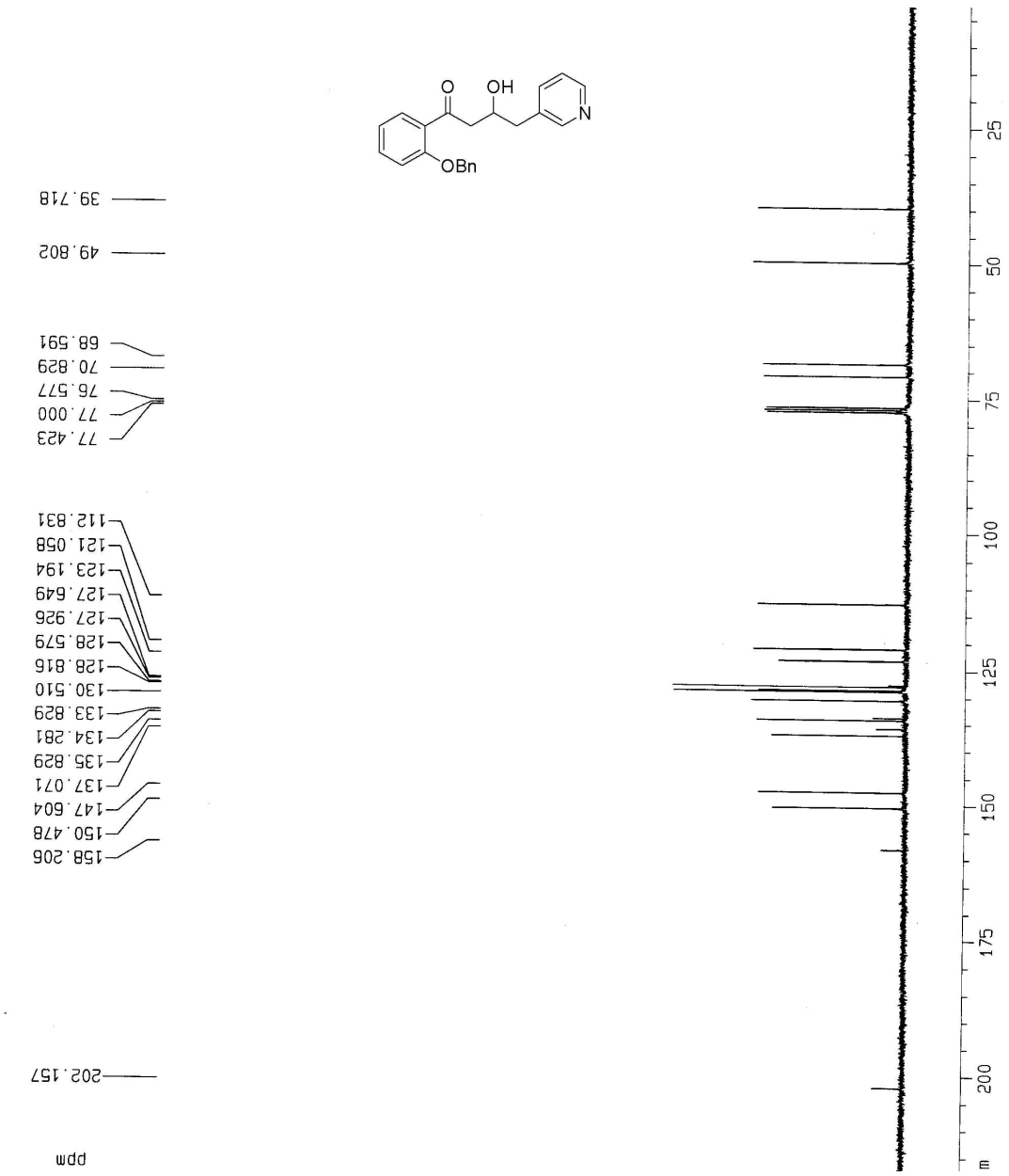
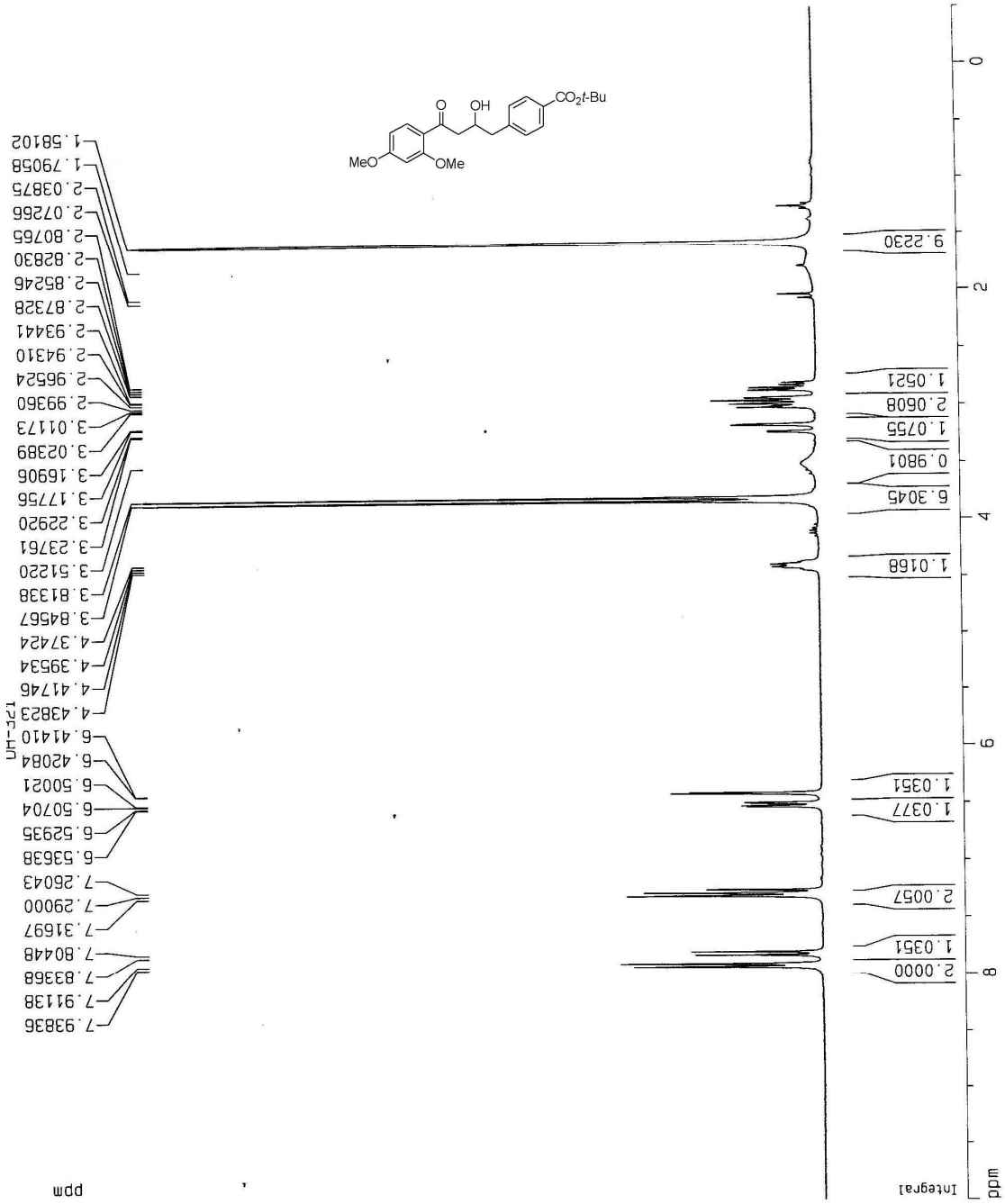
130.376
129.724
129.555
127.961
126.749
113.782
77.424
77.000
76.577
67.744
55.467
43.585
40.080
MeO
O
OH
Cl
ppm
200
175
150
125
100
75
50
25

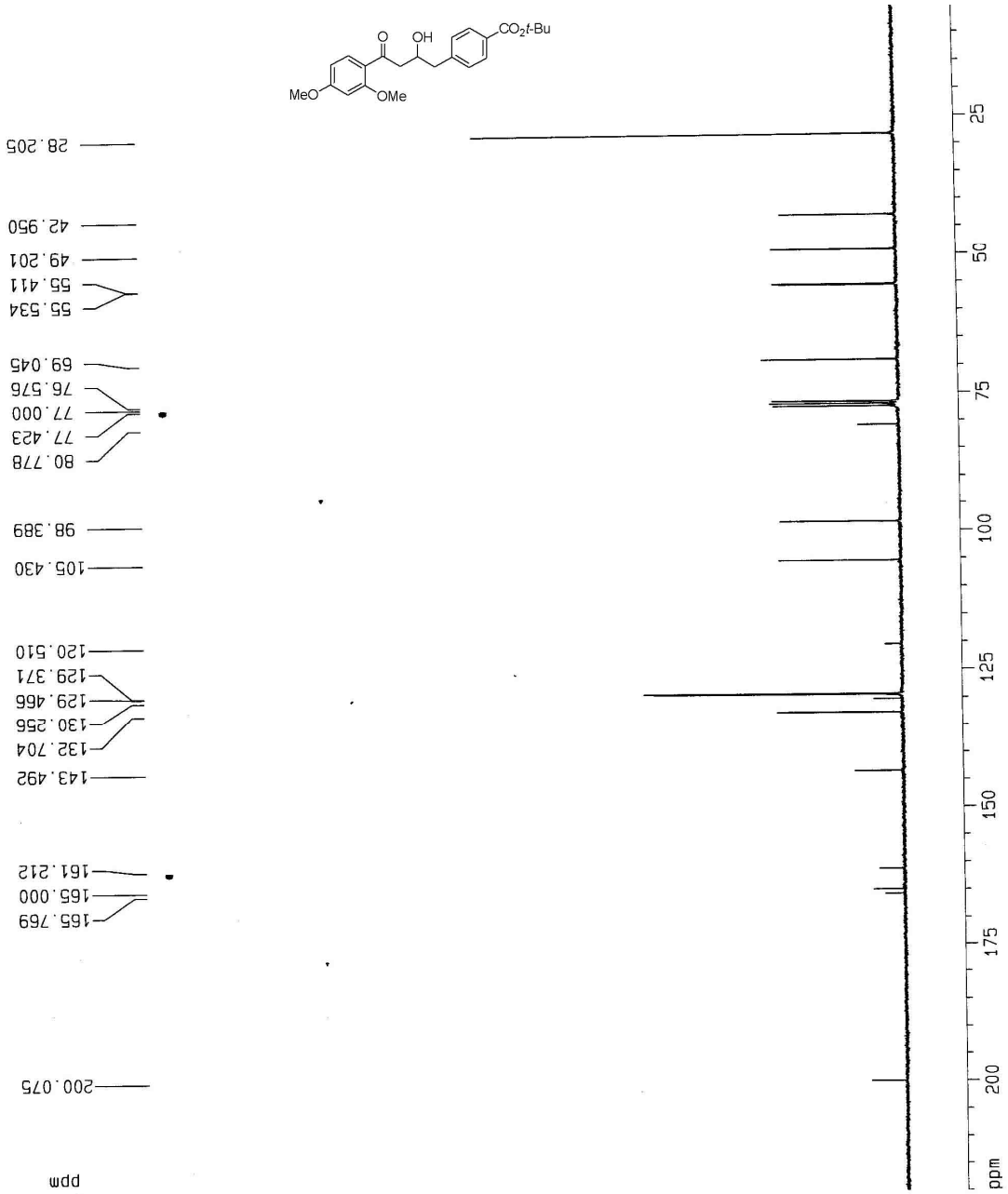
## 10. $^{1}$H and $^{13}$C Spectra of Compound 2e



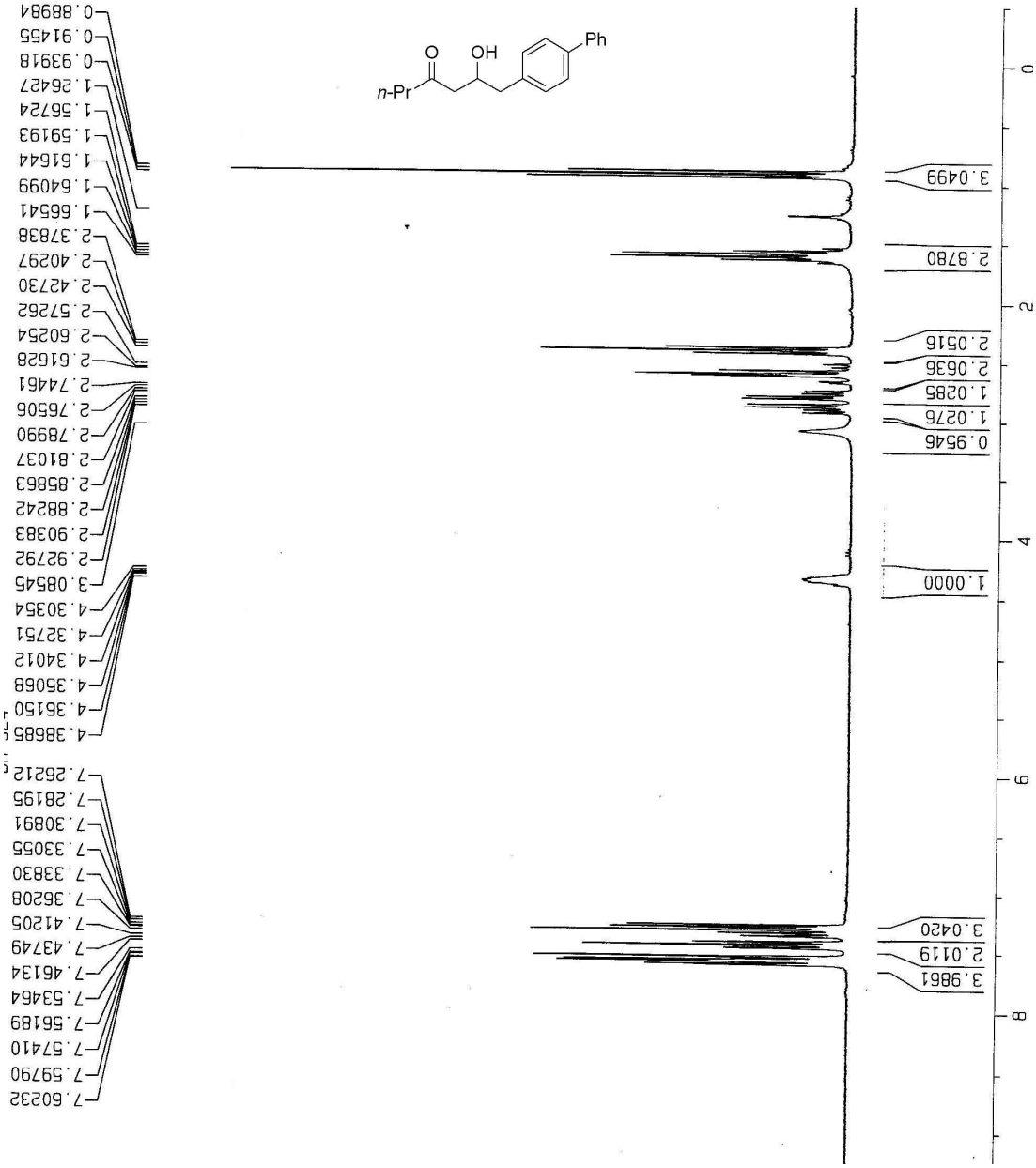

202.157
158.206
150.478
147.604
137.071
135.829
134.281
133.829
130.510
128.816
128.579
127.926
127.649
123.194
121.058
112.831
77.423
77.000
76.577
70.829
68.591
49.802
39.718
OBn
O
OH
N
ppm
m
25
50
75
100
125
150
175
200

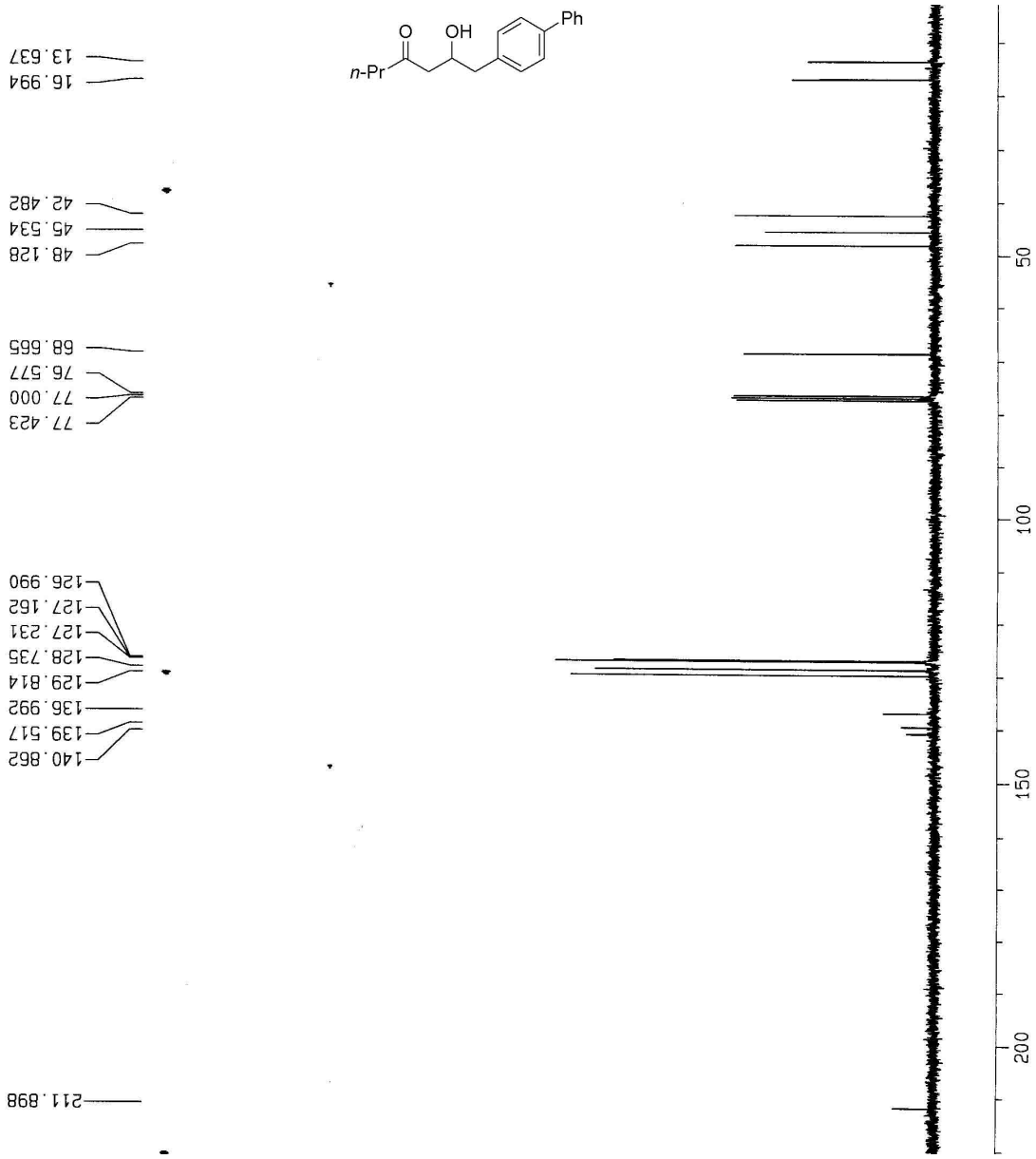
## 11. $^{1}$H and $^{13}$C Spectra of Compound 2f



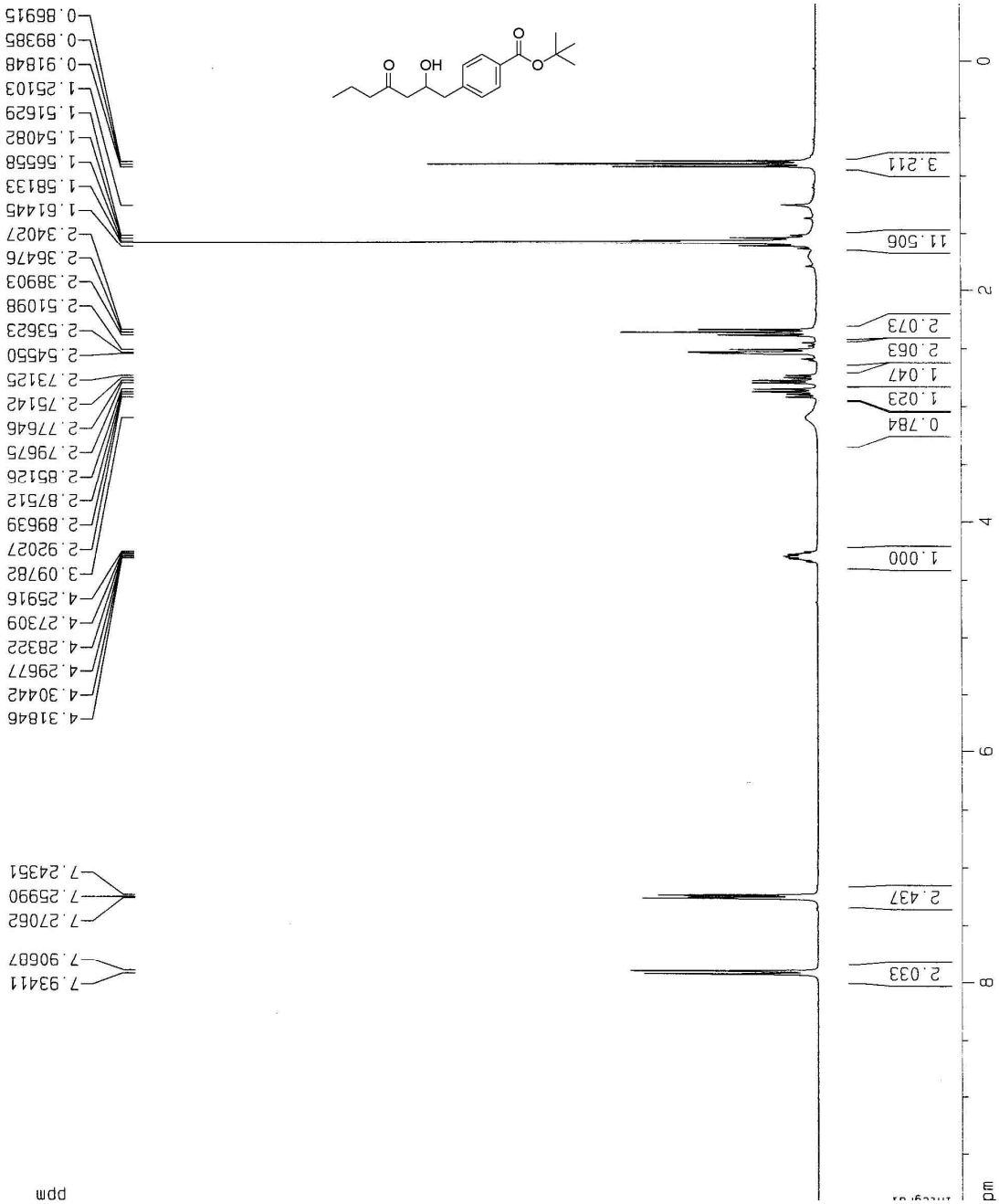

ppm
200.075
161.212
165.000
165.769
143.492
132.704
130.256
129.466
129.371
120.510
105.430
98.389
80.778
77.423
77.000
76.576
69.045
55.534
55.411
49.201
42.950
28.205
MeO
OMe
O
OH
CO2t-Bu

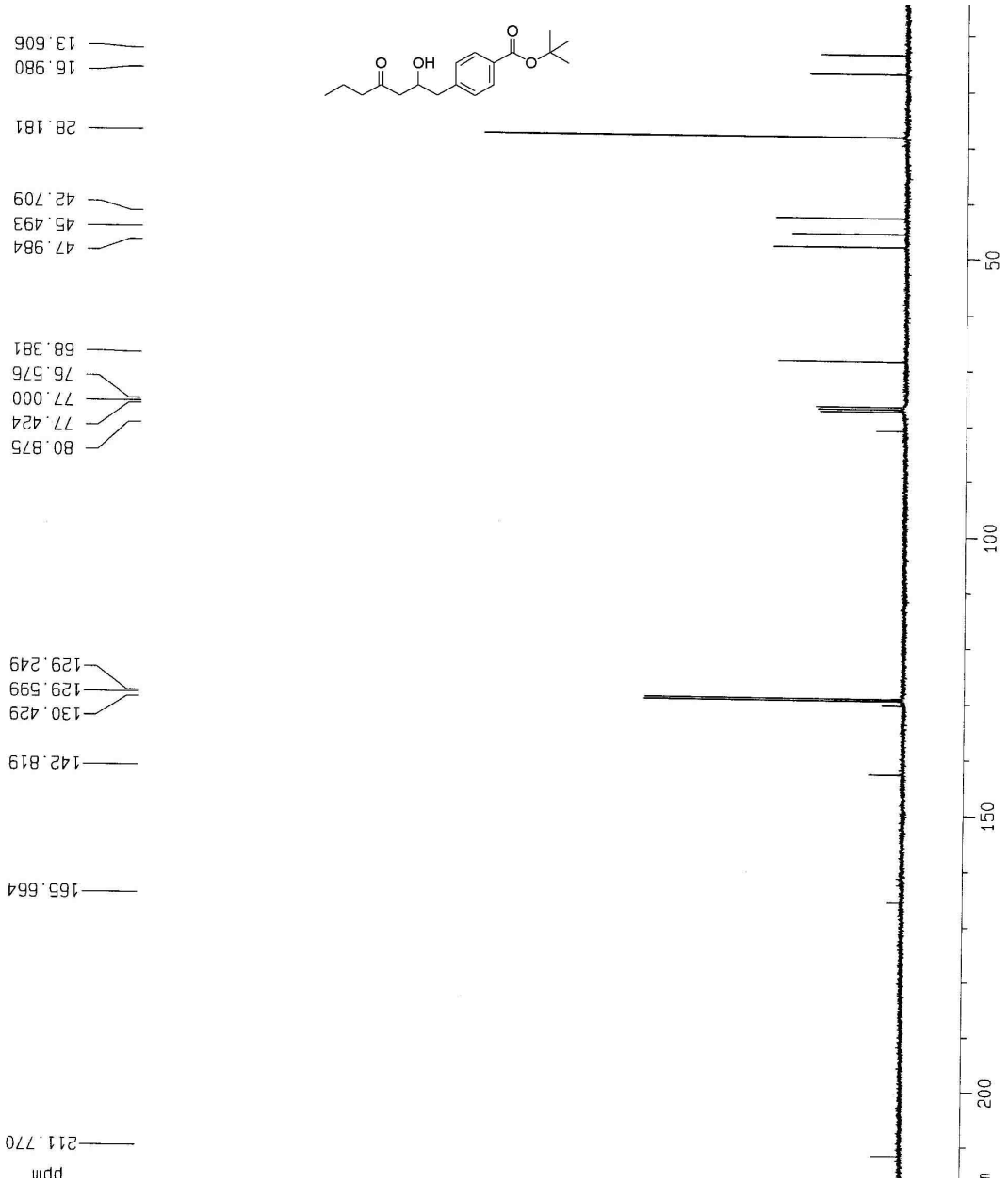
## 12. $^{1}$H and $^{13}$C Spectra of Compound 2g



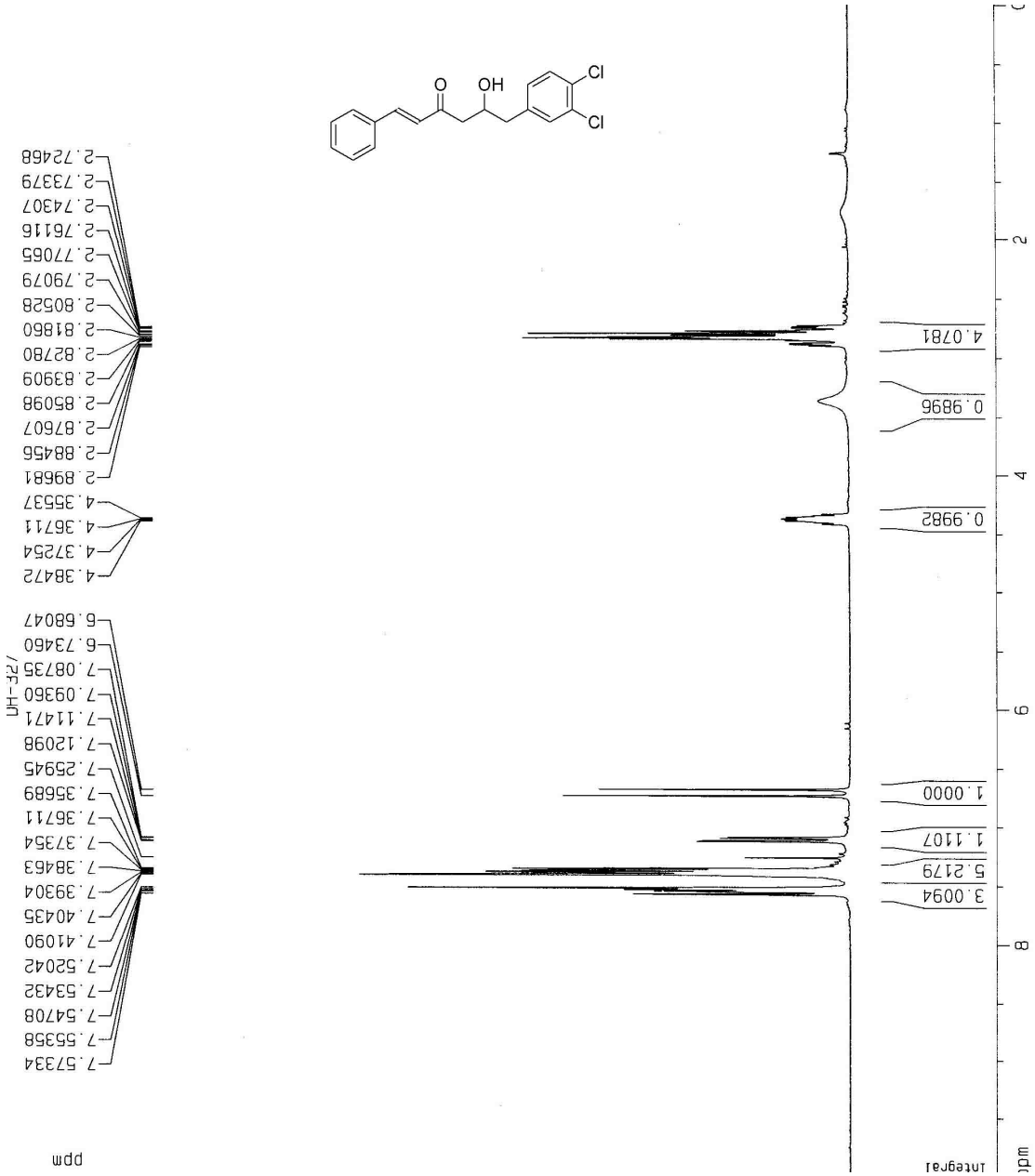

211.898
140.862
139.517
136.992
129.814
128.735
127.231
127.162
126.990
77.423
77.000
76.577
68.665
48.128
45.534
42.482
16.994
13.637
n-Pr
O
OH
Ph
200
150
100
50

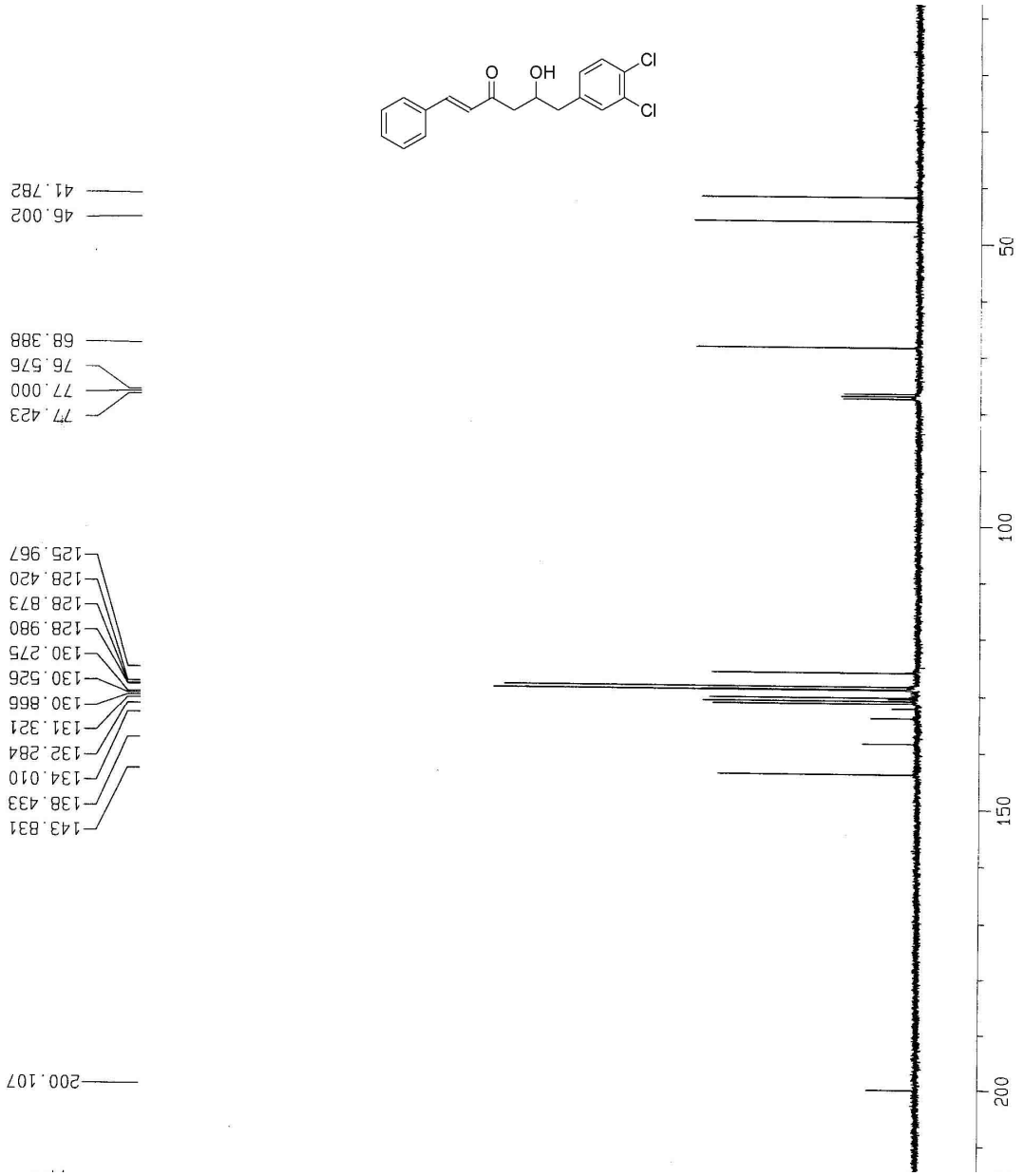
## 13. $^{1}$H and $^{13}$C Spectra of Compound 2h



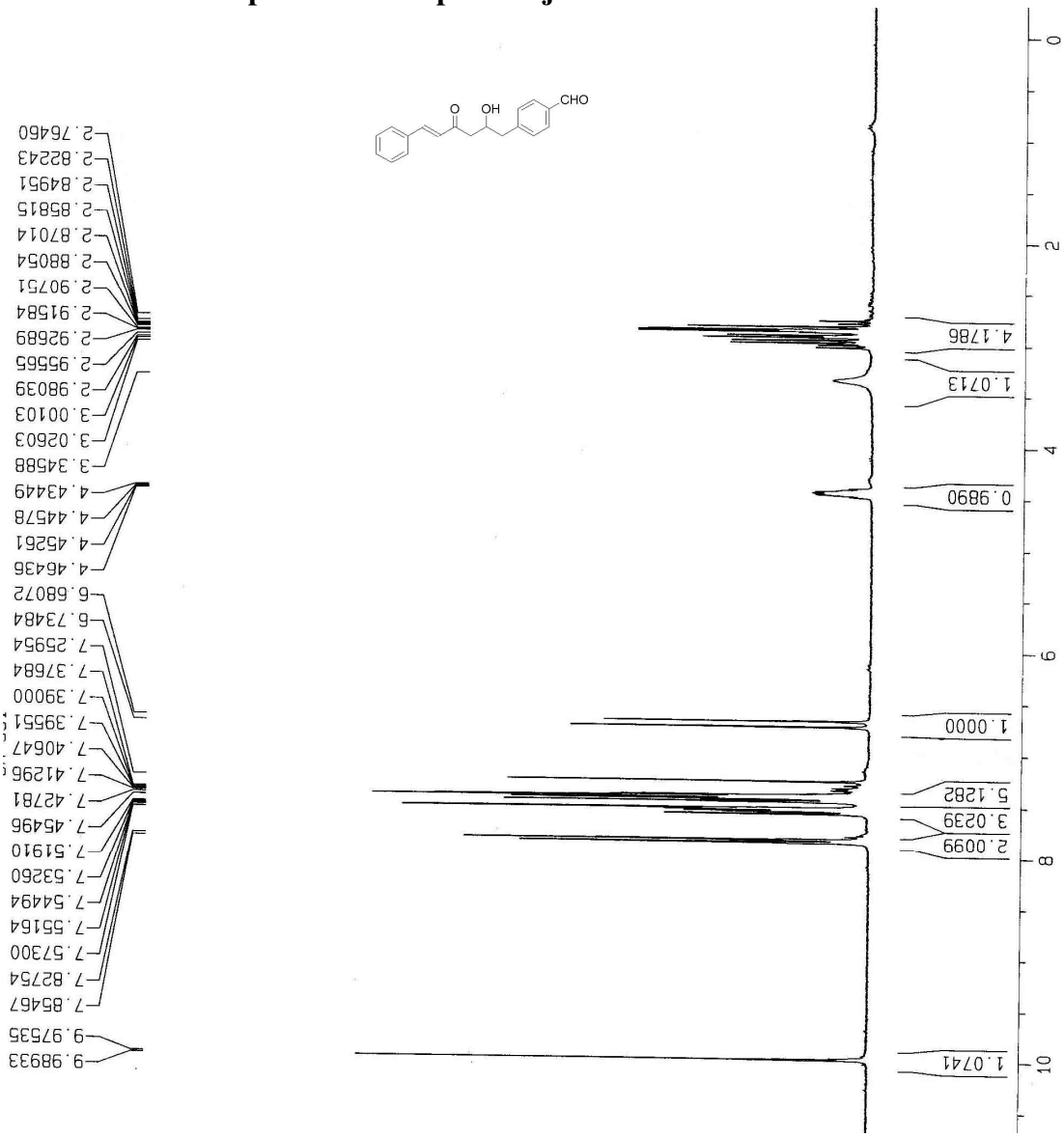

211.770
165.664
142.819
130.429
129.599
129.249
80.875
77.424
77.000
76.576
68.381
47.984
45.493
42.709
28.181
16.980
13.506
ppm
200
150
100
50
0
O
OH
O
O

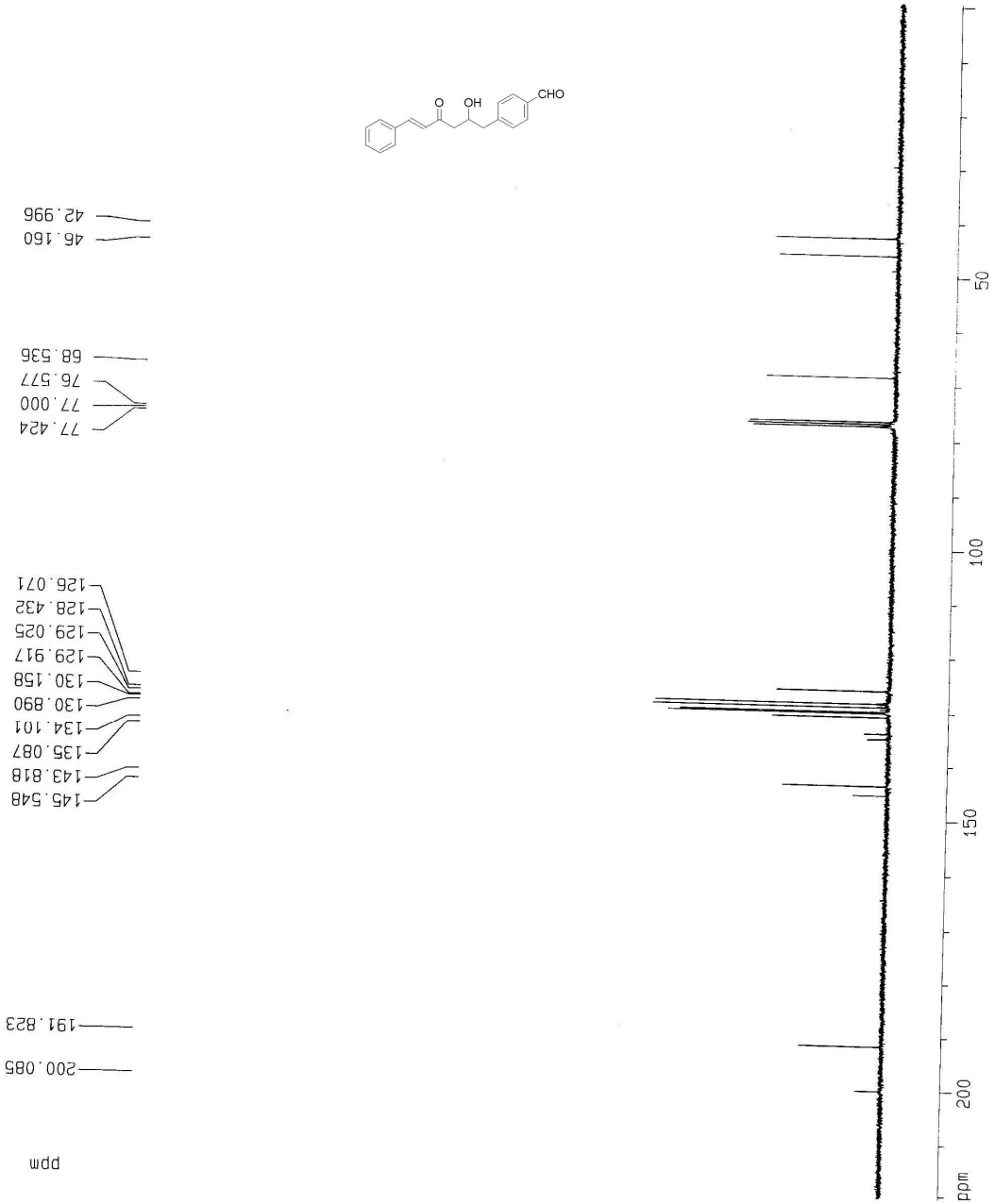
## 14. $^{1}H$ and $^{13}C$ Spectra of Compound 2i



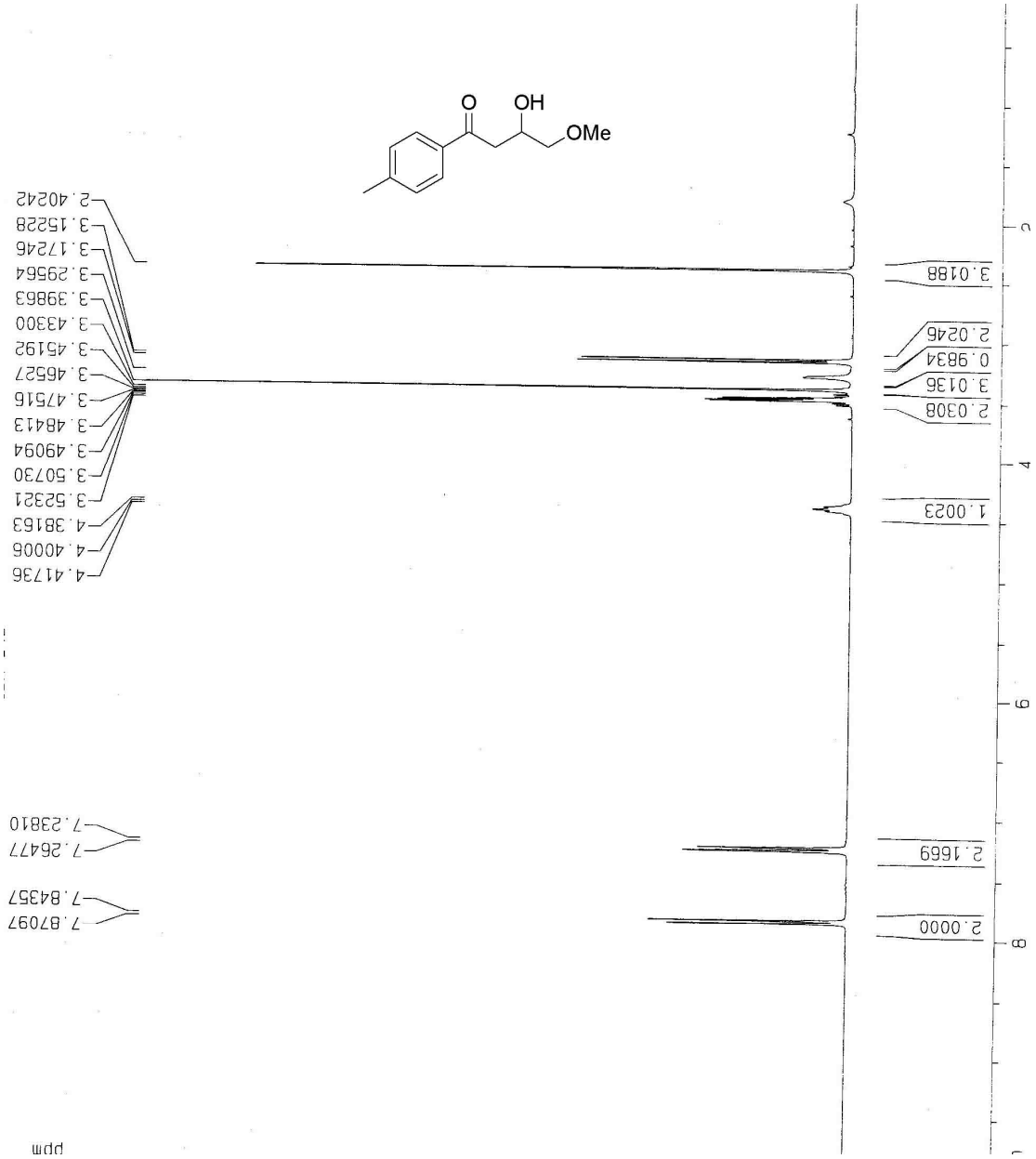

O
OH
Cl
Cl
200.107
143.831
138.433
134.010
132.284
131.321
130.866
130.526
130.275
128.980
128.873
128.420
125.967
77.423
77.000
76.576
68.388
46.002
41.782
50
100
150
200

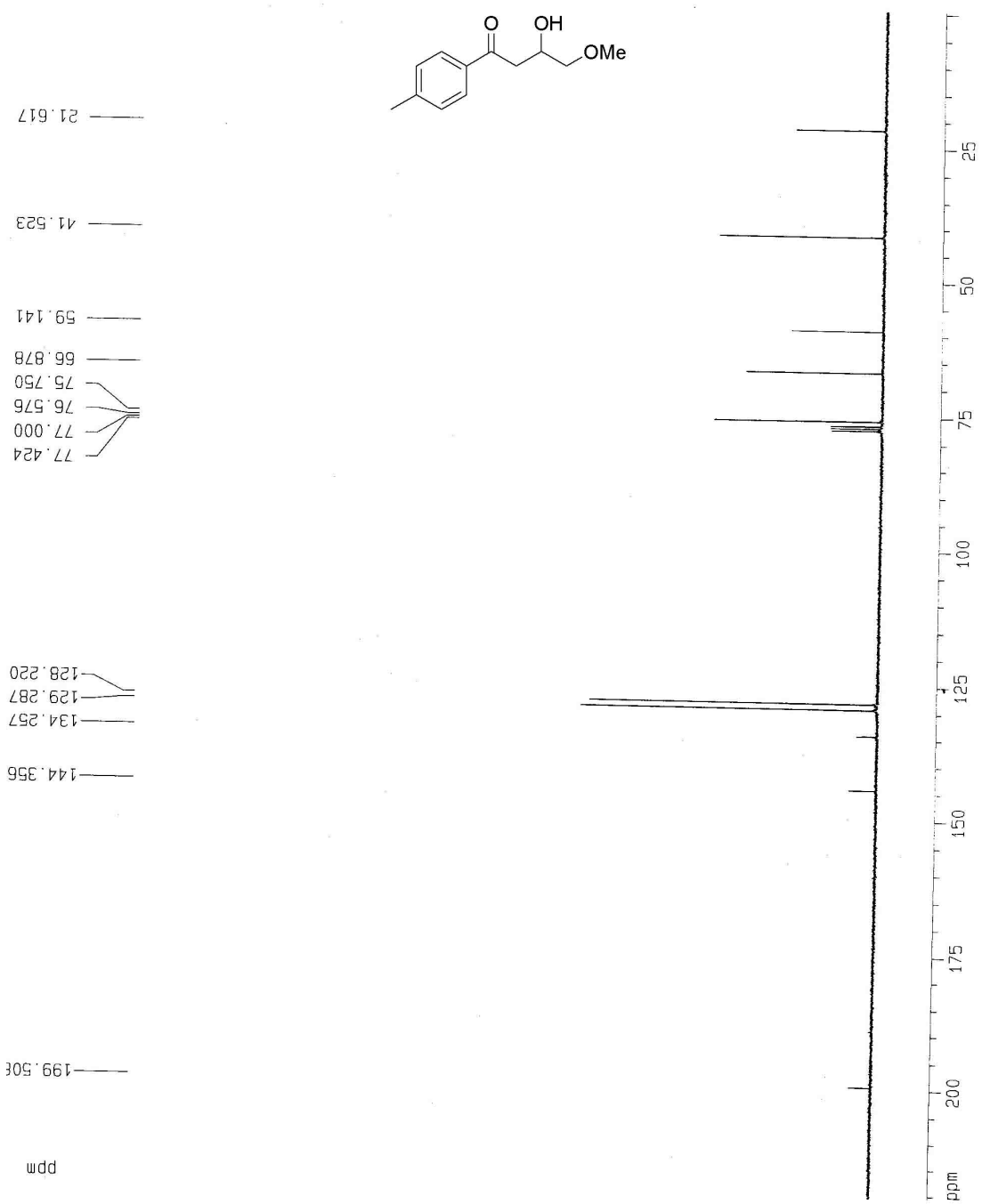
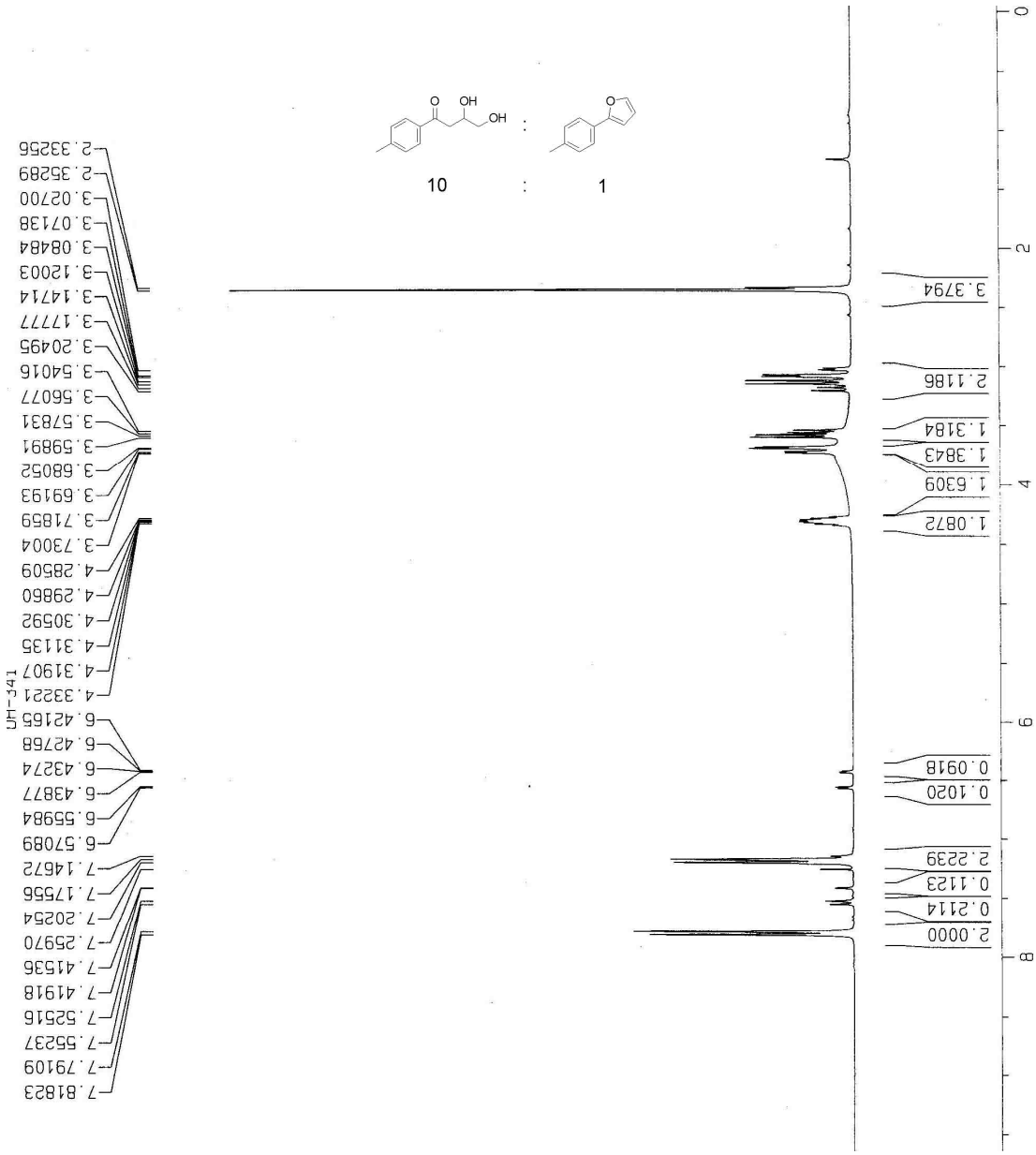
## 15. $^{1}H$ and $^{13}C$ Spectra of Compound 2j




ppm
200.085
191.823
145.548
143.818
135.087
134.101
130.890
130.158
129.917
129.025
128.432
126.071
77.424
77.000
76.577
68.536
46.160
42.996
CHO
OH
O
ppm
200
150
100
50

## 16. $^{1}$H and $^{13}$C Spectra of Compound 2k




O
OH
OMe
21.617
41.523
59.141
66.878
75.750
76.576
77.000
77.424
128.220
129.287
134.257
144.356
199.506
ppm
25
50
75
100
125
150
175
200
ppm

## 17. $^{1}$H Spectra of Compound 2l