Electronic Supplementary Material (ESI) for RSC Advances

$Fe_2(SO_4)_3 \cdot xH_2O$ on silica: an efficient and low-cost reaction system for the direct nucleophilic substitution of alcohols at solventless conditions

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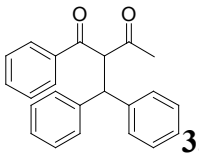
1 Characterization of compounds 3a-n

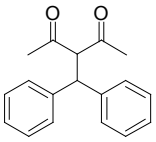
2-Benzhydryl-1-phenyl-butane-1,3-dione (3a).



3a

The physical data shown below were comparable to those reported in literature.[1] White solid, m.p. 155-156 °C, reported m.p. 156-157 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96 (d, J = 8 Hz, 2 H), 7.77-7.03 (m, 13 H), 5.62 (d, J = 12 Hz, 1H), 5.10 (d, J = 12 Hz, 1H), 2.05 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 202.3, 193.1, 140.6, 140.2, 136.1, 132.5, 128.2, 128.0, 127.9, 127.3, 126.9, 126.6, 125.9, 68.2, 50.1, 27.1.

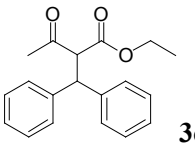
3-Benzhydryl-pentane-2,4-dione (3b).



3b

The physical data shown below were comparable to those reported in literature.[2] White solid, m.p. 114-115 °C, reported m.p.112-114°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.48-7.03 (m, 10 H), 4.80 (d, J = 12 Hz, 1H), 7.73 (d, J = 12 Hz, 1H), 2.05 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 203.0, 141.3, 129.1, 127.9, 126.9, 74.1, 51.5, 30.1.

2-Benzhydryl-3-oxo-butyric acid ethyl ester (3c)

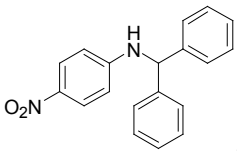


3c

The physical data shown below were comparable to those reported in literature.[3] White solid, m.p. 86-87°C, reported m.p. 84-86°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.30-7.15 (m, 10 H), 4.76 (d, J = 12 Hz, 1 H), 4.56 (d, J = 12 Hz, 1 H), 4.01 (q, J = 7 Hz, 2 H), 2. 13 (s, 3 H), 1.05 (t, J = 7 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 202.1,

167.7, 141.3, 141.0, 129.0, 128.8, 128.0, 127.8, 127.1, 126.8, 65.3, 61.6, 51.1, 32.8, 13.6.

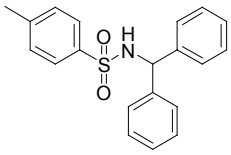
_N_-(4-nitrophenyl)-1,1'-diphenyl)-methyamine (3d)



3d

The physical data shown below were comparable to those reported in literature.[4] Yellow solid, m.p. 194-195°C, reported m.p. 195°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.06-8.02 (m, 2H), 7.41-7.30 (m, 10H), 6.53-6.50 (m, 2H), 5.68 (d, J = 4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 153.1, 141.2, 139.3, 129.5, 128.7, 127.6, 126.7, 113.1, 63.0.

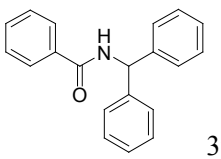
_N_-benzhydryl-_p_-toluenesulfonamide (3e)



3e

The physical data shown below were comparable to those reported in literature. [4] White solid, m.p. 156-158 °C, reported m.p.157°C; ^{1}H NMR (400 MHz, CDCl3) δ: 7.55 (d, J = 8.2 Hz, 2H), 7.23-7.05 (m, 12H), 5.59 (d, J = 7.2 Hz, 1H), 5.18 (d, J = 7.2 Hz, 1H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, CDCl3) δ: 143.0, 142.8, 139.9, 133.1, 131.1, 130.6, 130.2, 130.0, 59.8, 23.3.

_N_-benzhydrylbenzamide (3f)

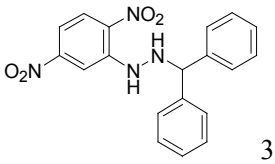


3f

The physical data shown below were comparable to those reported in literature. [5]

White solid, m.p. 168-170 °C, reported m.p. 170°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88-7.42 (m, 5H), 7.41-7.33 (m, 10H), 6.71 (d, J = 7.8 Hz, 1H), 6.47 (d, J = 7.8 Hz, 1H), ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 166.5, 141.1, 133.6, 131.2, 128.0, 127.7, 127.5, 127.0, 126.8, 56.1.

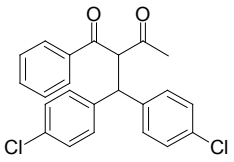
N-Benzhydryl-N'-(2,4-dinitro-phenyl)-hydrazine (3g)



3g

The physical data shown below were comparable to those reported in literature. [4] White solid, m.p. 164-165 °C, reported m.p. 165°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.25 (s, 1H), 9.01 (d, J = 2 Hz, 1H), 8.31 (dd, J_1 = 9 Hz, J_2 = 1 Hz), 7.97 (d, J = 9 Hz, 1H), 7.46-7.32 (m, 10 H), 5.23 (d, J = 5.2 Hz, 1H), 4.50 (d, J = 6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.9, 140.2, 138.5, 134.1, 131.9, 130.2, 129.1, 128.6, 126.5, 121.2, 66.3.

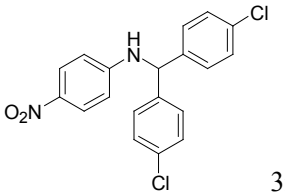
2-[Bis-(4-chloro-phenyl)-methyl]-1-phenyl-butane-1,3-dione (3h).



3h

The physical data shown below were comparable to those reported in literature. [1] White solid, m.p. 166-168 °C, reported m.p. 167-168 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02 - 7.11 (m, 13 H), 5.55 (d, J = 12 Hz, 1H), 5.11 (d, J = 12 Hz, 1H), 2.08 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 201.1, 192.9, 139.1, 138.5, 135.4, 133.5, 132.5, 131.8, 128.2, 128.4, 128.3, 128.1, 127.9, 127.6, 67.8, 49.3, 26.8.

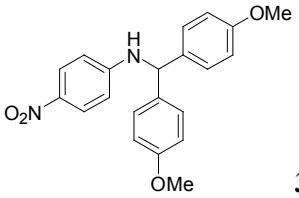
[Bis-(4-chloro-phenyl)-methyl]-(4-nitro-phenyl)-amine (3i).



3i

The physical data shown below were comparable to those reported in literature. [1] Yellow solid, m.p. 194-195 °C, reported m.p. 193-195 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (m, 2H), 7.75-7.22 (m, 8H), 6.49 (d, J = 8.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.8, 137.8, 133.3, 128.7, 127.6, 125.0, 111.3, 60.0.

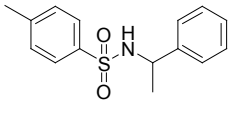
N-(bis(4-methoxyphenyl)methyl)-4-nitrobenzenamine(3j)



3j

Yellow solid, m.p. 148-149 °C,^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.04-6.48 (m, 12H), 5.54 (d, J = 4.8 Hz, 1H), 6.48 (d, J = 4.4 Hz, 1H), ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 159.1, 152.1, 133.3, 128.4, 126.2, 112.1, 114.3, 112.1, 61.1, 55.3. Element analysis C21H20N2O4 ($C_{21}H_{20}N_2O_4$): calculated C, 69.22; H, 5.53; N, 7.69; found C, 69.08; H, 5.66; N, 7.57. HRMS (ESI): calculated 387.1321 for $C_{21}H_{20}N_2O_4$ Na^+, found 387.1311 ($M+Na^+$).

4-methyl-N-(1-phenylethyl)benzenesulfonamide(3k)

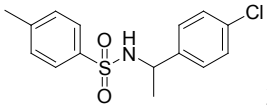


3k

White solid, m.p. 81-82 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.63-7.09 (m, 9H), 4.77 (d, J = 6.8 Hz, 1H), 4.46 (m, 1H), 2.39 (s, 3H), 1.43 (d, J = 6.8 Hz, 3H), ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 143.1, 142.0, 137.5, 129.4, 128.5, 127.1, 126.1, 53.6, 23.6, 21.5.

Element analysis ($C_{15}H_{17}NO_2S$): calculated C, 65.43; H, 6.22; N, 5.09; found C, 65..39; H, 6.24; N, 5.06. HRMS (ESI): calculated 298.0878 for $C_{15}H_{17}NO_2S\ Na^+$, found 298.0871 ($M+Na^+$).

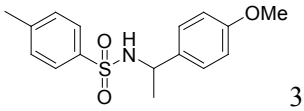
N-(1-(4-chlorophenyl)ethyl)-4-methylbenzenesulfonamide(3l)



3l

White solid, m.p. 138-139 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.59-7.029 (m, 8H), 4.84 (d, J = 6.8 Hz, 1H), 4.45 (m, 1H), 2.40 (s, 3H), 1.39 (d, J = 6.8 Hz, 3H), ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 143.4, 142.0, 137.4, 133.2, 129.5, 128.6, 127.6, 127.0, 53.0, 23.5, 21.5. Element analysis ($C_{15}H_{16}ClNO_2S$): calculated C, 58.15; H, 5.21; N, 4.52; found C, 58.11; H, 5.25; N, 4.50. HRMS (ESI): calculated 332.0488 for $C_{15}H_{16}ClNO_2S\ Na^+$, found 332.0490 ($M+Na^+$)

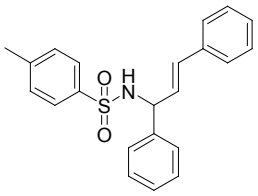
N-(1-(4-methoxyphenyl)ethyl)-4-methylbenzenesulfonamide(3m)



3m

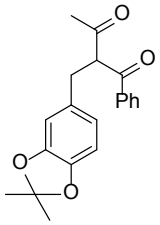
White solid, m.p. 96-98 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.63-6.72 (m, 8H), 4.69 (d, J = 6.4 Hz, 1H), 4.41 (m, 1H), 3.76 (s, 3H), 2.40 (s, 3H), 3.76 (s, 3H), 1.41 (d, J = 6.8 Hz, 3H), ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 158.9, 143.1, 137.6, 134.1, 129.4, 127.3, 127.1, 113.8, 55.3, 53.1, 23.4, 21.5. Element analysis ($C_{16}H_{19}NO_3S$): calculated C, 62.93; H, 6.27; N, 4.59; found C, 62.86; H, 6.30; N, 4.56. HRMS (ESI): calculated 328.0983 for $C_{16}H_{19}NO_3S\ Na^+$, found 328.0991 ($M+Na^+$)

N-(1,3-Diphenyl-allyl)-4-methyl-benzenesulfonamide (3n).



The physical data shown below were comparable to those reported in literature. [1] White solid, m.p. 166-167 °C, reported m.p. 167-168 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69-7.66 (m, 2 H), 7.30-7.07 (m, 16 H (including the H of chloroform)), 6.39 (d, J = 16 Hz), 6.12 (dd, J_1 = 16 Hz, J_2 = 6.8 Hz), 5.15-5.12 (m, 1 H), 4.83 (d, J = 7.2 Hz), 2.41 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.5, 133.9, 131.8, 130.2, 126.7, 124.0, 123.3, 122.9, 122.6, 122.1, 122.0, 121.8, 121.3, 120.7, 53.8, 16.1.

2-((2,2-dimethylbenzo[d][1,3]dioxol-5-yl)methyl)-1-phenylbutane-1,3-dione (3o)



White oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 7.8 Hz, 2H), 7.58 (d, J = 7.3 Hz, 1H), 7.47 (t, J = 7.4 Hz, 2H), 6.67 (dd, J = 16.0, 7.1 Hz, 3H), 5.90 (s, 2H), 4.75 (t, J = 7.1 Hz, 1H), 3.31 – 3.17 (m, 2H), 2.14 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.28, 195.72, 147.73, 146.28, 136.41, 133.78, 132.04, 128.89, 128.73, 121.83, 109.24, 108.37, 100.93, 65.11, 34.53, 28.62. Element analysis ($C_{20}H_{20}O_4$): calculated C, 74.06; H, 6.21; found C, 74.03; H, 6.22. HRMS (ESI): calculated 347.1254 for $C_{20}H_{20}O_4Na^+$, found 347.1257 ($M+Na^+$)

References

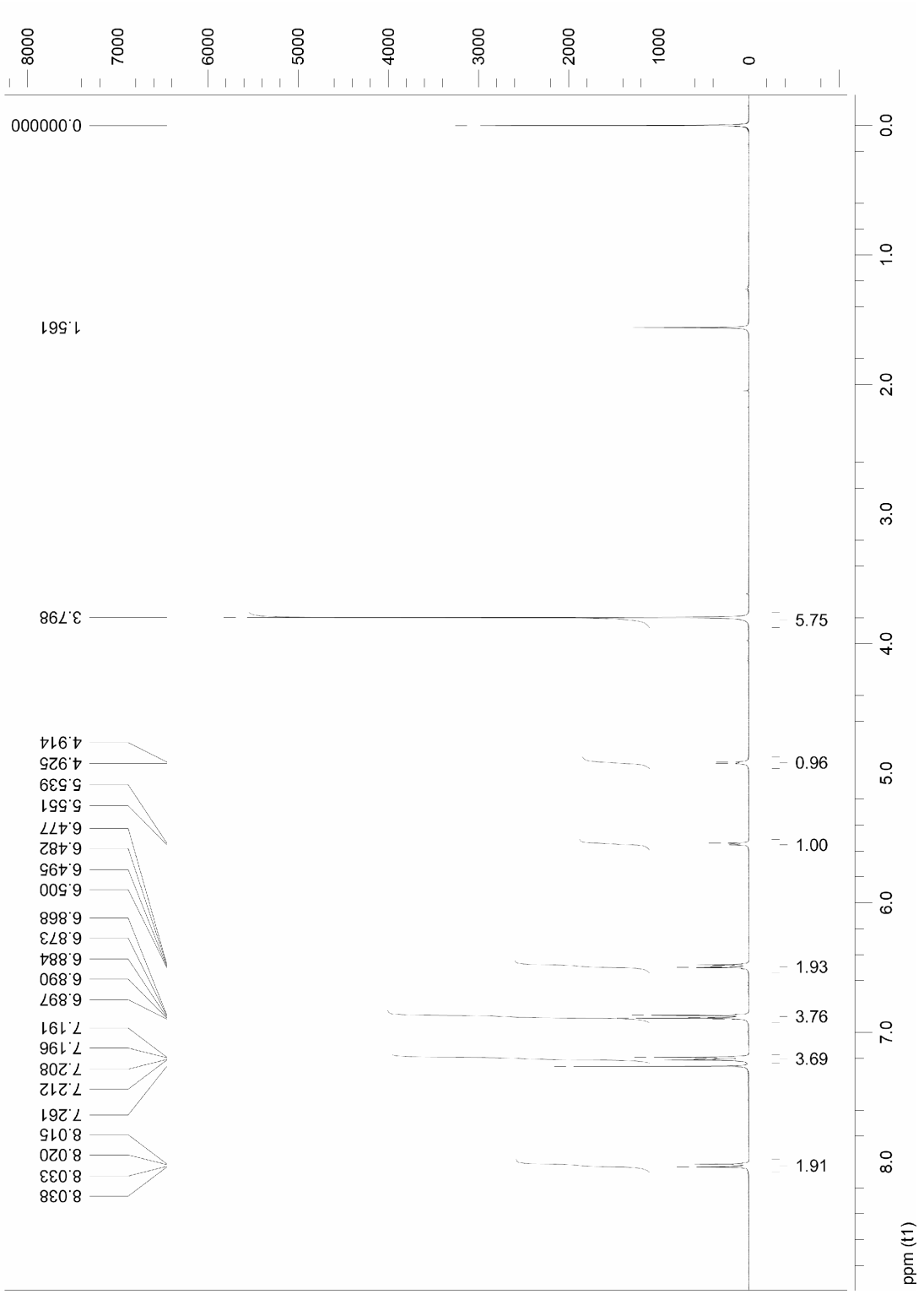
[1] A. L. Zhu, L. J. Li, J. J. Wang, K. L. Zhuo, *Green Chem.*, 2011, **13**, 1244.

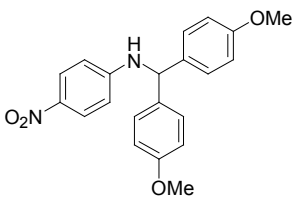
[2] K. Motokura, N. Nakagiri, T. Mizugaki, K. Ebitani and K. Kaneda, *J. Org. Chem.*, 2007, **72**, 6006.

[3] R. Sanz, D. Miguel, A. Martı´nez, J. M. Álvarez-Gutiérrez, F. Rodríguez. *Org. Lett.*, 2007, **9**, 2027.

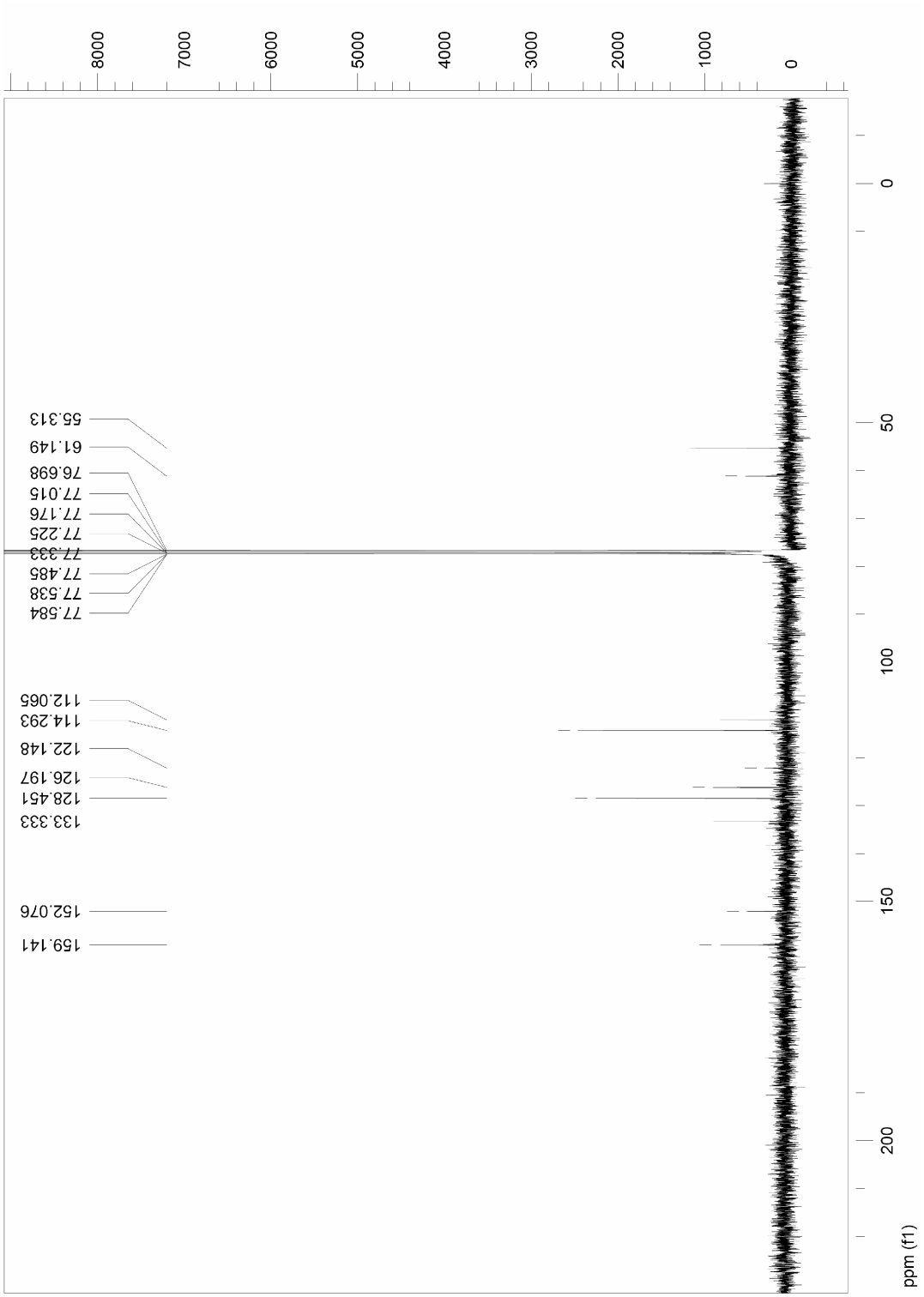
[4] V. Terrasson, S. Marque, M. Georgy, J. M. Campagne, D. Prim, *Adv. Synth. Catal.* 2006, **348**, 2063.

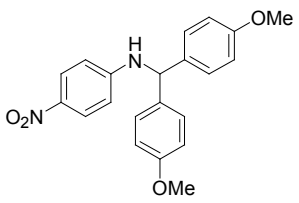
[5] T. Maki, K. Ishihara, H. Yamamoto, *Org. Lett.*, 2006, **8**, 1431.





3j





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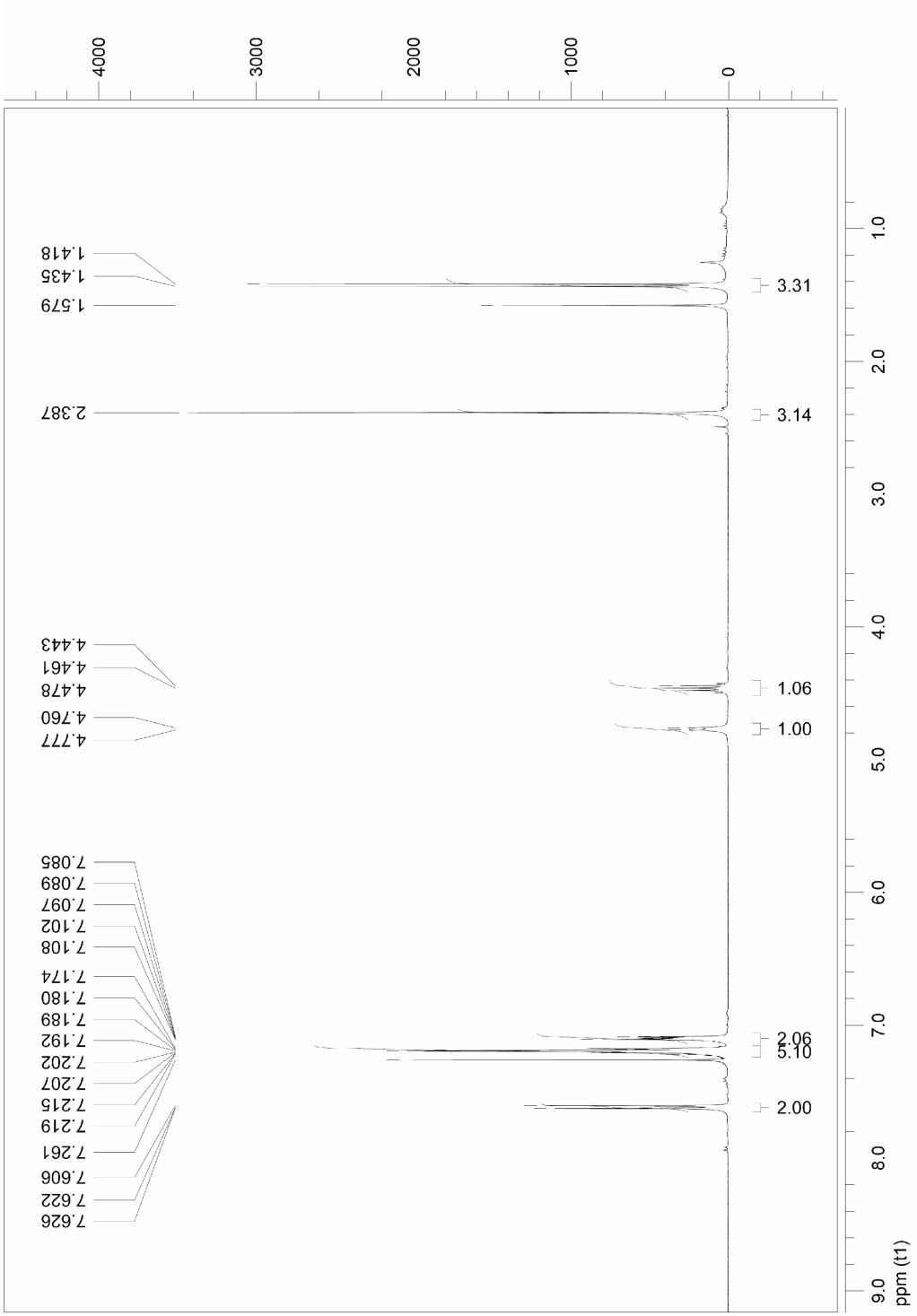
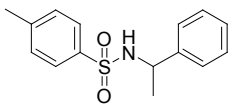
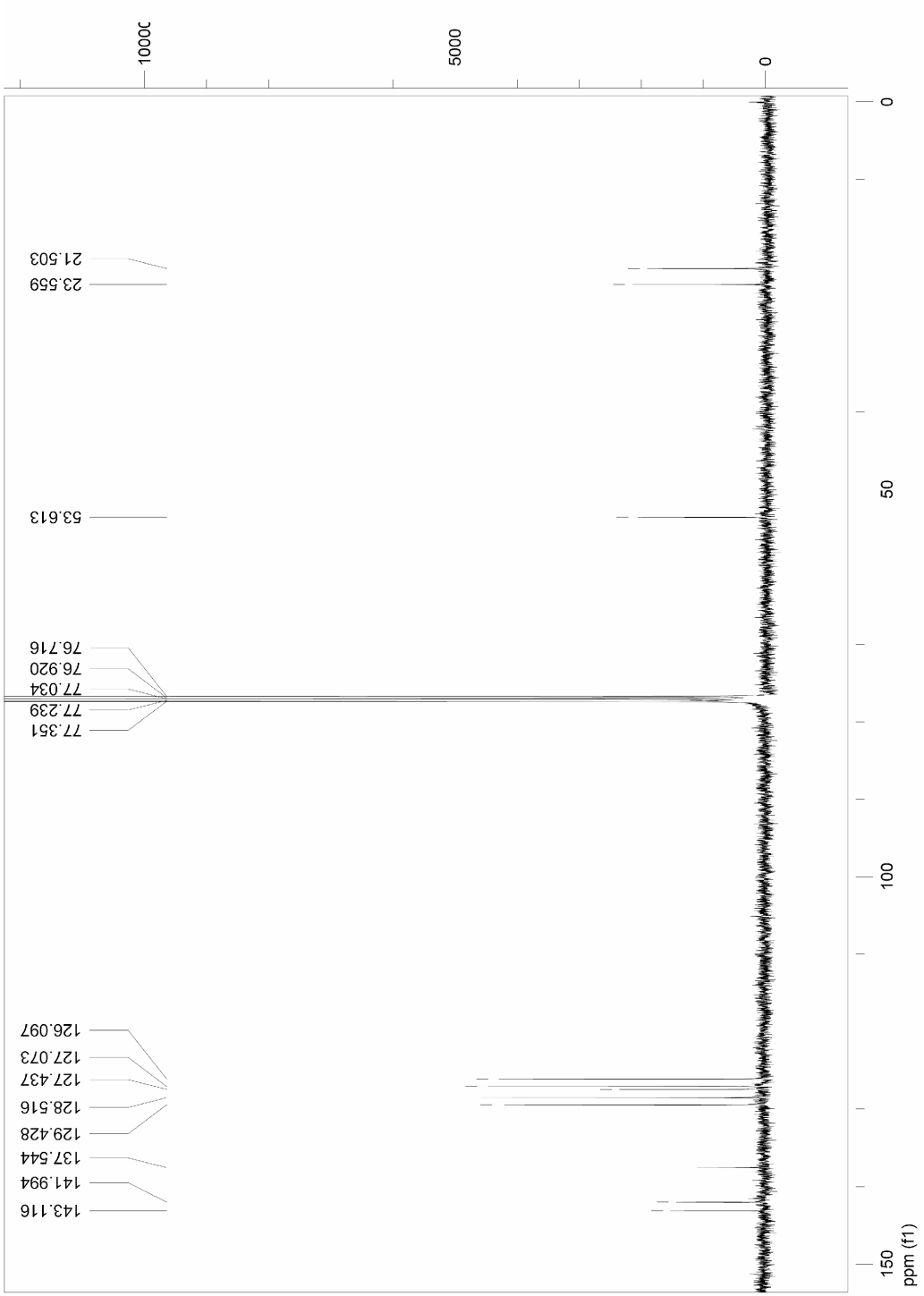
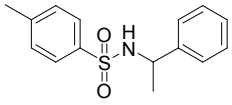


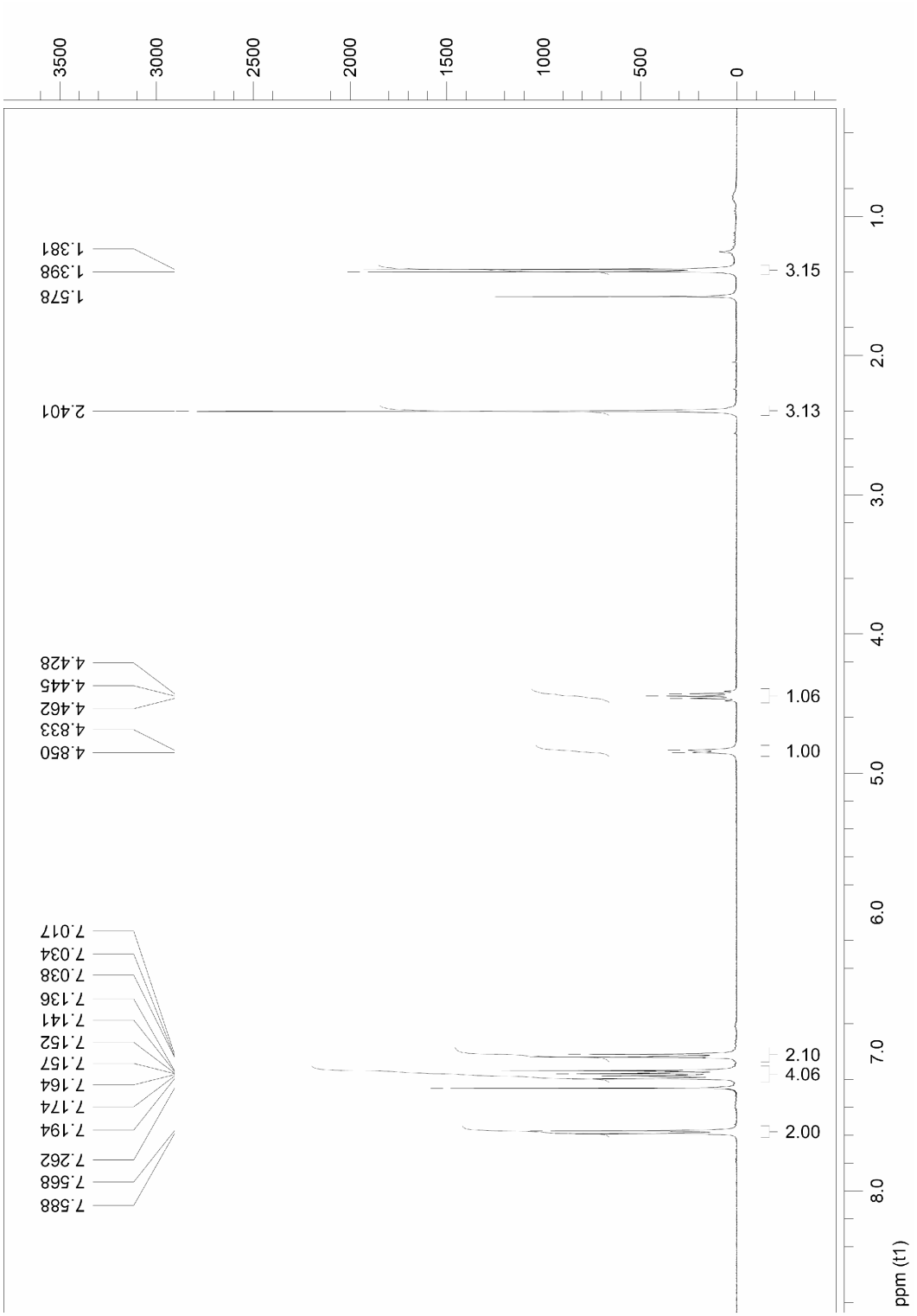


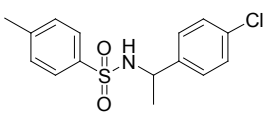
3k



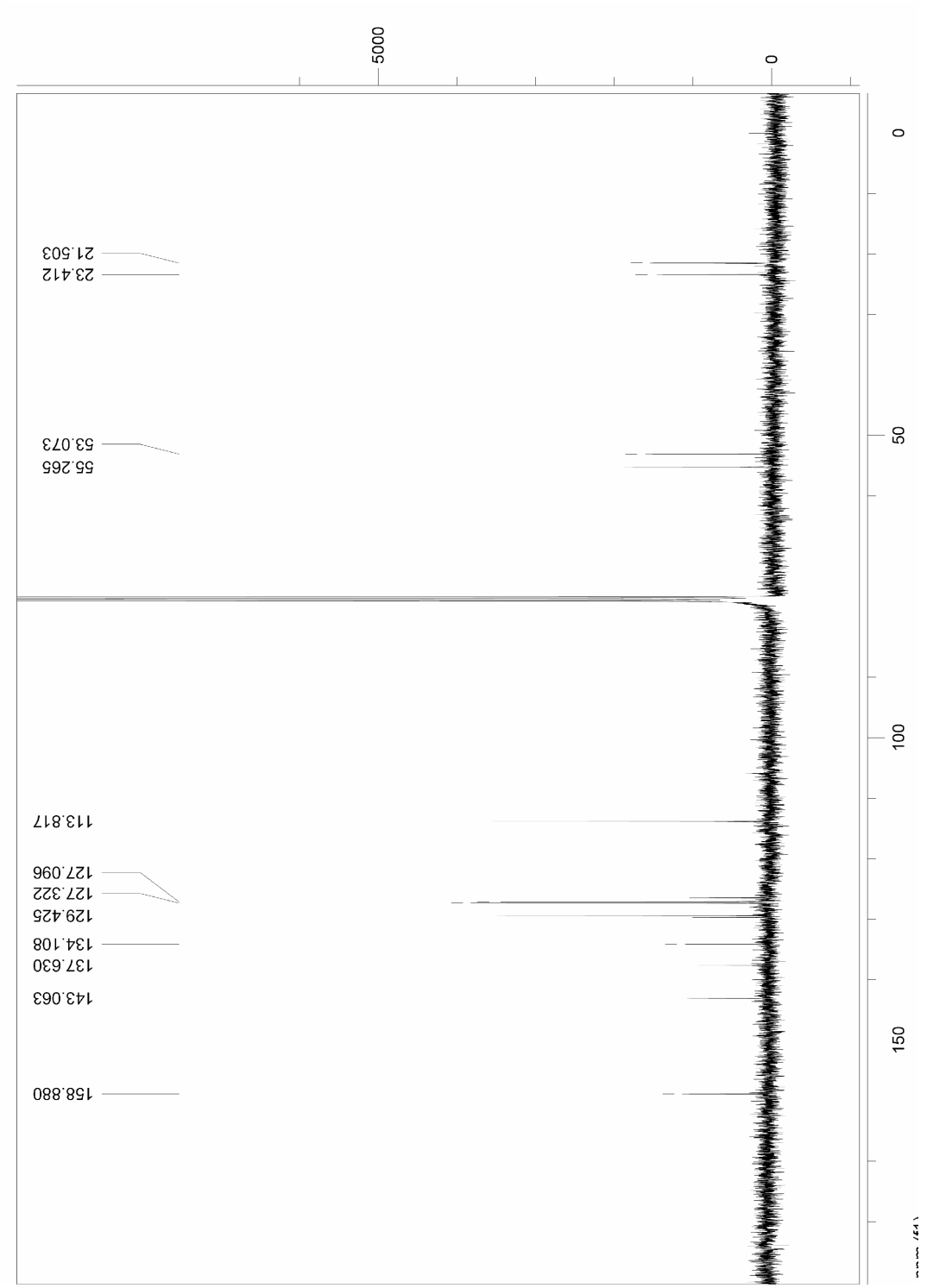


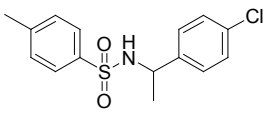
3k



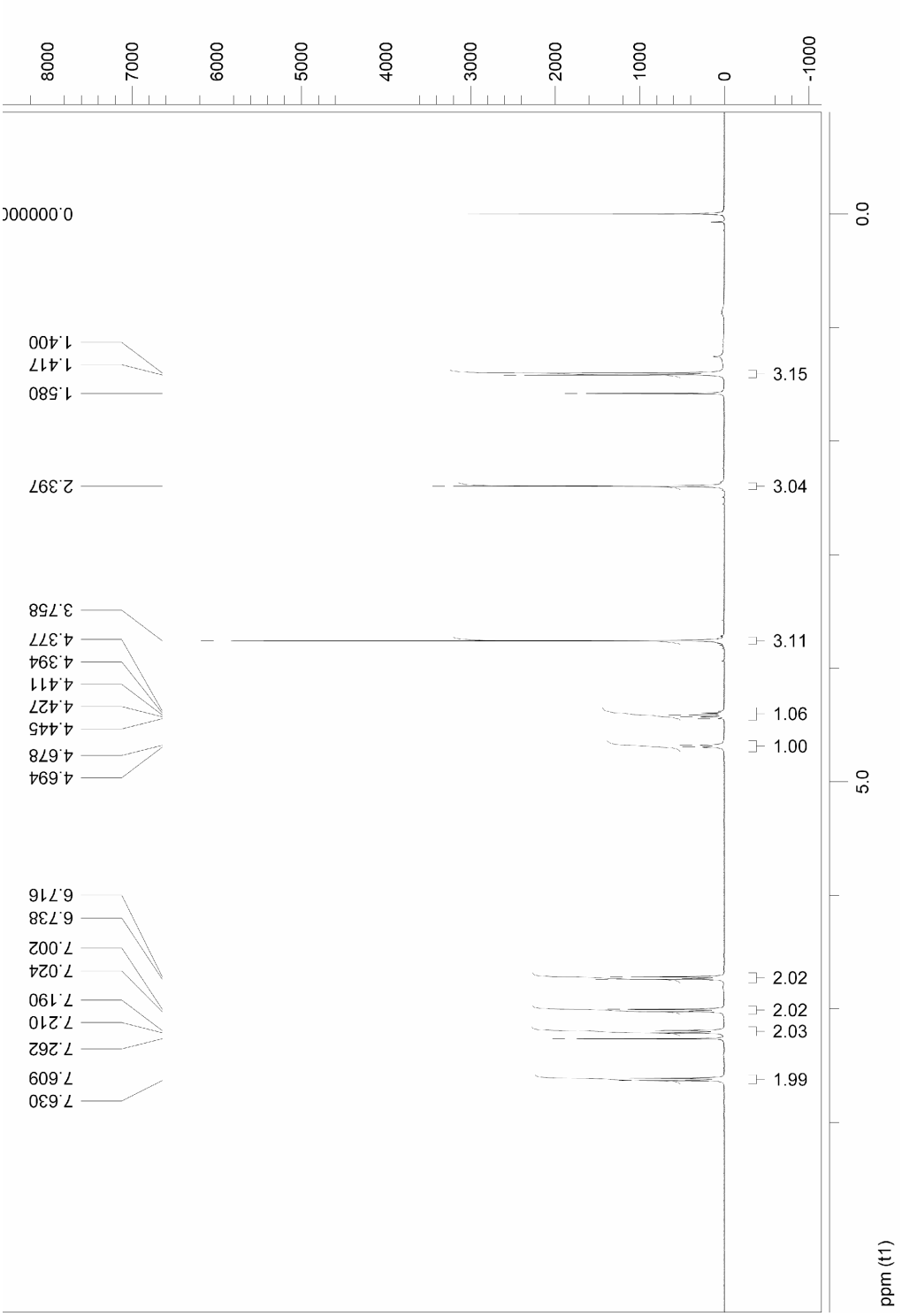


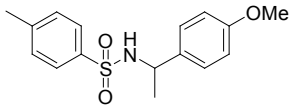
3l



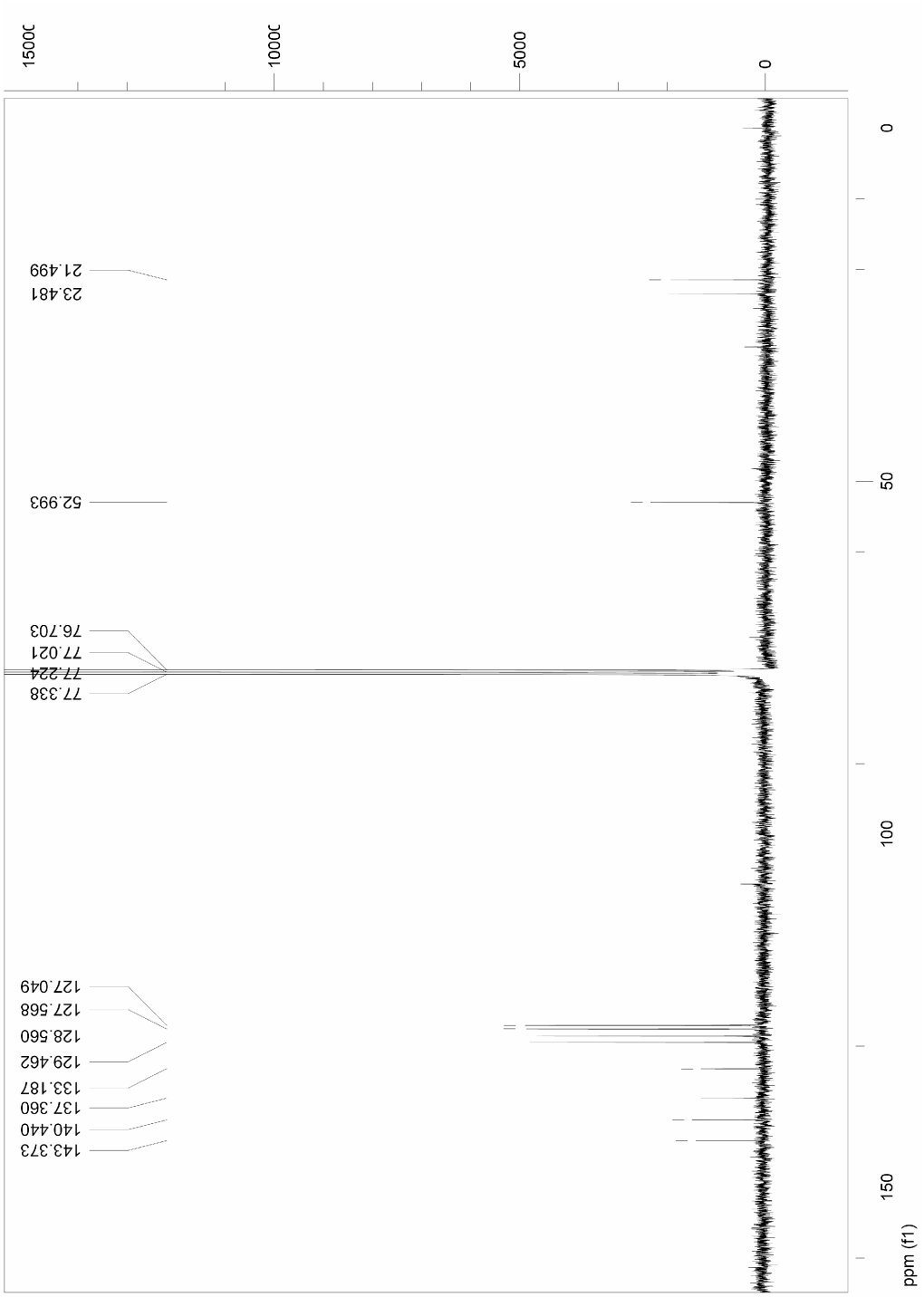


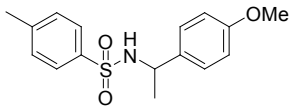
3l





3m





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