Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

An Effective Approach to Aryl-substituted Propanoic Acids *via* Pd-catalyzed Hydrocarboxylation of Stilbenes

Jie Dai,[a] Wenlong Ren,[a] Jingfu Li,[a] and Yian Shi*[,a,b]

[a]*State Key Laboratory of Coordination Chemistry, Center for Multimolecular Organic Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, China;*
[b]*Department of Chemistry, Colorado State University, Fort Collins, Colorado 80523, United States.*

Supporting Information

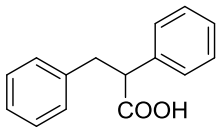
Table of Contents

General Methods. All commercially available reagents were used without further purification. All solvents used for the reaction were purified with solvent purification system. Column chromatography was performed on silica gel (200-300 mesh). ^{1}H NMR spectra were recorded on a 400 MHz NMR spectrometer and ^{13}C NMR spectra were recorded on a 100 MHz NMR spectrometer. IR spectra were recorded on a FT-IR spectrometer. Melting points were uncorrected. The **1a**, **1b**, and **1s** were purchased from commercial suppliers and the other substrates were synthesized *via* Heck reaction according to the reported procedure.[1]

1) Nanteuil, de F.; Waser, J. *Angew. Chem. Int. Ed.* **2013**, *52*, 9009

Representative procedure for hydrocarboxylation (Table 2, 2a). To a mixture of $Pd(OAc)_2$ (0.0056 g, 0.025 mmol), PPh_3 (0.02623 g, 0.10 mmol), and toluene (0.250 mL) in a vial (1.5 mL) were added alkene **1a** (0.0901 g, 0.50 mmol), HCOOPh (0.0733 g, 0.60 mmol), and HCOOH (0.046 g, 1.00 mmol) successively via syringe. The vial was purged with Ar to remove the air and tightly sealed with a septum cap. The reaction mixture was stirred at 90 ^{o}C for 48 h, cooled to rt, and purified by flash chromatography (silica gel, eluent: PE/EA = 4/1) to give compound **2a** as a white solid (0.1042 g, 92% yield).

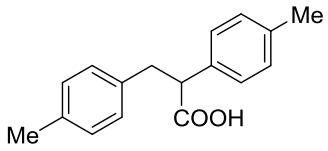
Table 2, 2a



White solid; mp. 75-77 ^{o}C; IR (film) 3434, 1701 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.14 (m, 8H), 7.13-7.08 (m, 2H), 3.86 (dd, *J* = 8.3, 7.1 Hz, 1H), 3.41 (dd, *J* = 13.8, 8.4 Hz, 1H), 3.04 (dd, *J* = 13.8, 7.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.0, 138.9, 138.1, 129.1, 128.9, 128.6, 128.3, 127.9, 126.7, 53.6, 39.5; HRMS (ESI) Calcd for $C_{15}H_{13}O_2$ (M-H): 225.0921; Found: 225.0916.

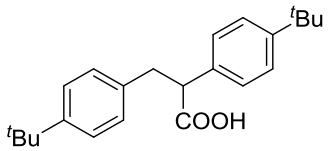
Stivala, C. E.; Zakarian, A. *J. Am. Chem. Soc.* **2011**, *133*, 11936

Table 2, 2b



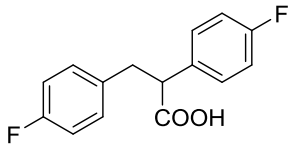
White solid; mp. 138-140 ^{o}C; IR (film) 3428, 1704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, *J* = 8.1 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.3 Hz, 1H), 7.00 (d, *J* = 8.3 Hz, 1H), 3.81 (dd, *J* = 8.4, 6.9 Hz, 1H), 3.35 (dd, *J* = 13.9, 8.6 Hz, 1H), 2.98 (dd, *J* = 13.8, 6.8 Hz, 1H), 2.32 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.7, 137.5, 136.1, 135.9, 135.3, 129.6, 129.3, 129.0, 128.2, 53.3, 39.0, 21.3, 21.2; HRMS (ESI) Calcd for $C_{17}H_{17}O_2$ (M-H): 253.1234; Found: 253.1229.

Table 2, 2c



White solid; mp. 118-120 ^{o}C; IR (film) 3446, 1707 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.32 (m, 2H), 7.31-7.23 (m, 4H), 7.12-7.06 (m, 2H), 3.85 (dd, *J* = 9.6, 5.6 Hz, 1H), 3.39 (dd, *J* = 14.0, 9.6 Hz, 1H), 2.98 (dd, *J* = 14.0, 5.6 Hz, 1H), 1.31 (s, 9H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.3, 150.7, 149.4, 136.2, 135.5, 128.7, 127.8, 125.9, 125.5, 53.0, 38.8, 34.7, 34.6, 31.6, 31.5; HRMS (ESI) Calcd for $C_{23}H_{29}O_2$ (M-H): 337.2173; Found: 337.2168.

Table 2, 2d

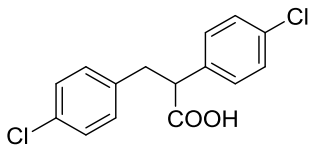


White solid; mp. 88-90 ^{o}C; IR (film) 3408, 1709 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.20 (m, 2H), 7.07-6.96 (m, 4H), 6.94-6.86 (m, 2H), 3.79 (t, *J* = 7.7 Hz,

1H), 3.34 (dd, *J* = 13.9, 8.0 Hz, 1H), 2.98 (dd, *J* = 13.9, 7.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.4, 162.5 (d, *J* = 245 Hz), 161.8 (d, *J* = 243 Hz), 134.1 (d, *J* = 3 Hz), 133.4 (d, *J* = 3 Hz), 130.6 (d, *J* = 8 Hz), 129.9 (d, *J* = 8 Hz), 115.9 (d, *J* = 22 Hz), 115.5 (d, *J* = 21 Hz), 53.0, 38.8; HRMS (ESI) Calcd for $C_{15}H_{11}F_2O_2$ (M-H): 261.0733; Found: 261.0727.

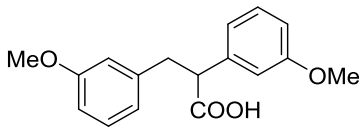
Szollosi, G.; Herman, B.; Felfoldi, K.; Fulop, F.; Bartok, M. *Adv. Synth. Catal.* **2008**, *350*, 2804

Table 2, 2e



White solid; mp. 126-127 ^{o}C; IR (film) 3414, 1709 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.24 (m, 2H), 7.23-7.16 (m, 4H), 7.03-6.97 (m, 2H), 3.79 (t, *J* = 7.8 Hz, 1H), 3.34 (dd, *J* = 13.9, 7.9 Hz, 1H), 2.98 (dd, *J* = 13.9, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.0, 136.8, 136.0, 134.0, 132.7, 130.5, 129.7, 129.2, 128.8, 52.9, 38.7; HRMS (ESI) Calcd for $C_{15}H_{11}Cl_2O_2$ (M-H): 293.0142; Found: 293.0138.

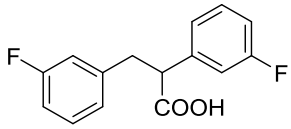
Table 2, 2f



Yellow solid; mp. 92-94 ^{o}C; IR (film) 3443, 1706 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (t, *J* = 7.9 Hz, 1H), 7.15 (t, *J* = 8.0 Hz, 1H), 6.90 (d, *J* = 7.7 Hz, 1H), 6.86 (t, *J* = 2.3 Hz, 1H), 6.84-6.79 (m, 1H), 6.75-6.70 (m, 2H), 6.65 (t, *J* = 2.0 Hz, 1H), 3.84 (dd, *J* = 8.5, 6.8 Hz, 1H), 3.78 (s, 3H), 3.72 (s, 3H), 3.37 (dd, *J* = 13.8, 8.6 Hz, 1H), 3.01 (dd, *J* = 13.8, 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.4, 159.9, 159.7, 140.4, 139.6, 129.9, 129.6, 121.4, 120.7, 114.6, 114.0, 113.3, 112.3, 55.4, 55.3, 53.5, 39.4; HRMS (ESI) Calcd for $C_{17}H_{17}O_4$ (M-H): 285.1132; Found:

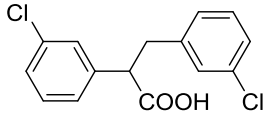
285.1127.

Table 2, 2g



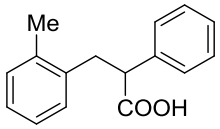
Yellow oil; IR (film) 3402, 1709 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.24 (m, 1H), 7.22-7.14 (m, 1H), 7.07-6.94 (m, 3H), 6.91-6.84 (m, 2H), 6.83-6.77 (m, 1H), 3.84 (t, *J* = 7.8 Hz, 1H), 3.38 (dd, *J* = 13.9, 8.2 Hz, 1H), 3.01 (dd, *J* = 13.9, 7.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.8, 163.1 (d, *J* = 245 Hz), 163.0 (d, *J* = 245 Hz), 140.8 (d, *J* = 7 Hz), 139.9 (d, *J* = 8 Hz), 130.5 (d, *J* = 8 Hz), 130.1 (d, *J* = 8 Hz), 124.8 (d, *J* = 3 Hz), 124.1 (d, *J* = 3 Hz), 116.0 (d, *J* = 21 Hz), 115.3 (d, *J* = 22 Hz), 115.0 (d, *J* = 21 Hz), 113.9 (d, *J* = 21 Hz), 53.0, 39.0 (d, *J* = 1 Hz); HRMS (ESI) Calcd for $C_{15}H_{11}F_2O_2$ (M-H): 261.0733; Found: 261.0727.

Table 2, 2h



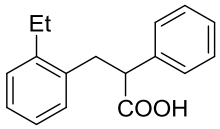
White solid; mp. 89-91 ^{o}C; IR (film) 3419, 1706 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.09 (m, 7H), 7.00-6.93 (m, 1H), 3.81 (t, *J* = 7.7 Hz, 1H), 3.36 (dd, *J* = 13.9, 8.4 Hz, 1H), 2.98 (dd, *J* = 13.9, 7.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.5, 140.3, 139.5, 134.8, 134.4, 130.2, 129.9, 129.2, 128.4, 128.3, 127.3, 127.2, 126.6, 53.0, 38.9; HRMS (ESI) Calcd for $C_{15}H_{11}Cl_2O_2$ (M-H): 293.0142; Found: 293.0139.

Table 3, 2i



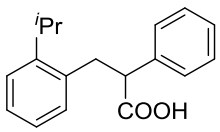
Yellow oil; IR (film) 1704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.29 (m, 4H), 7.29-7.20 (m, 1H), 7.18-7.05 (m, 4H), 3.91 (t, *J* = 6.9 Hz, 1H), 3.48 (dd, *J* = 14.1, 8.4 Hz, 1H), 3.07 (dd, *J* = 14.2, 6.8 Hz, 1H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.8, 138.4, 137.0, 136.4, 130.5, 129.6, 128.9, 128.2, 127.8, 126.8, 126.1, 52.3, 36.8, 19.6; HRMS (ESI) Calcd for $C_{16}H_{15}O_2$ (M-H): 239.1078; Found: 239.1075.

Table 3, 2j



Yellow oil; IR (film) 3420, 1707 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.29 (m, 4H), 7.25-7.09 (m, 3H), 7.07-7.01 (m, 2H), 3.87 (t, *J* = 7.2 Hz, 1H), 3.46 (dd, *J* = 14.1, 8.4 Hz, 1H), 3.05 (dd, *J* = 14.2, 6.7 Hz, 1H), 2.62 (q, *J* = 7.6 Hz, 2H), 1.20 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.6, 142.4, 138.4, 136.3, 129.7, 128.9, 128.7, 128.3, 127.8, 127.0, 126.0, 52.8, 36.2, 25.6, 15.5; HRMS (ESI) Calcd for $C_{17}H_{17}O_2$ (M-H): 253.1234; Found: 253.1231.

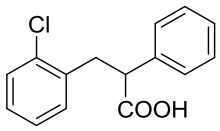
Table 3, 2k



Yellow oil; IR (film) 3431, 1704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.27 (m, 4H), 7.25-7.13 (m, 3H), 7.02-6.97 (m, 2H), 3.80 (t, *J* = 7.2 Hz, 1H), 3.45 (dd, *J* = 14.1, 8.2 Hz, 1H), 3.16-3.00 (m, 2H), 1.19 (d, *J* = 6.8 Hz, 3H), 1.14 (d, *J* = 6.8 Hz,

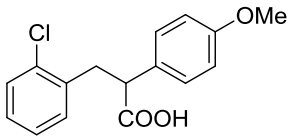
3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.9, 147.2, 138.3, 135.4, 130.0, 128.9, 128.3, 127.8, 127.2, 125.7, 125.6, 53.5, 36.3, 28.9, 24.4, 24.1; HRMS (ESI) Calcd for $C_{18}H_{19}O_2$ (M-H): 267.1391; Found: 267.1387.

Table 3, 2l



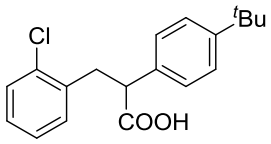
White solid; mp. 105-107 ^{o}C; IR (film) 3419, 1706 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.24 (m, 6H), 7.16-7.09 (m, 1H), 7.04 (d, *J* = 4.2 Hz, 2H), 4.03 (dd, *J* = 8.4, 6.9 Hz, 1H), 3.49 (dd, *J* = 13.8, 8.5 Hz, 1H), 3.14 (dd, *J* = 13.8, 6.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.0, 138.1, 136.4, 134.4, 131.7, 129.7, 129.0, 128.3, 128.2, 127.9, 126.9, 51.1, 37.5; HRMS (ESI) Calcd for $C_{15}H_{12}ClO_2$ (M-H): 259.0531; Found: 259.0527.

Table 3, 2m



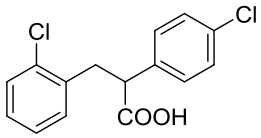
White solid; mp. 124-126 ^{o}C; IR (film) 1695 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.31 (m, 1H), 7.25-7.20 (m, 2H), 7.16-7.10 (m, 1H), 7.09-7.03 (m, 2H), 6.87-6.82 (m, 2H), 3.98 (dd, *J* = 8.0, 7.1 Hz, 1H), 3.79 (s, 3H), 3.46 (dd, *J* = 13.7, 8.3 Hz, 1H), 3.11 (dd, *J* = 13.8, 6.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.3, 159.3, 136.5, 134.4, 131.6, 130.1, 129.7, 129.3, 128.2, 126.9, 114.3, 55.5, 50.2, 37.5; HRMS (ESI) Calcd for $C_{16}H_{14}ClO_3$ (M-H): 289.0637; Found: 289.0635.

Table 3, 2n



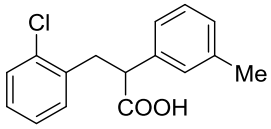
White solid; mp. 130-132 ^{o}C; IR (film) 3416, 1709 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.31 (m, 3H), 7.30-7.23 (m, 2H), 7.17-7.09 (m, 2H), 7.09-7.03 (m, 1H), 4.00 (dd, *J* = 9.2, 5.8 Hz, 1H), 3.46 (dd, *J* = 13.8, 9.2 Hz, 1H), 3.14 (dd, *J* = 13.8, 5.9 Hz, 1H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.7, 150.8, 136.6, 135.1, 134.4, 131.6, 129.7, 128.2, 127.7, 126.9, 125.9, 50.6, 37.5, 34.7, 31.5; HRMS (ESI) Calcd for $C_{19}H_{21}ClNaO_2$ (M+Na): 339.1122; Found: 339.1120.

Table 3, 2o



White solid; mp. 123-125 ^{o}C; IR (film) 3408, 1704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.31-7.21 (m, 4H), 7.14 (td, *J* = 7.4, 1.8 Hz, 1H), 7.06 (td, *J* = 7.5, 1.3 Hz, 1H), 7.01 (dd, *J* = 7.6, 1.8 Hz, 1H), 4.01 (t, *J* = 7.6 Hz, 1H), 3.48 (dd, *J* = 13.8, 8.1 Hz, 1H), 3.11 (dd, *J* = 13.8, 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.9, 136.4, 135.9, 134.3, 133.9, 131.6, 129.8, 129.6, 129.1, 128.5, 127.0, 50.5, 37.4; HRMS (ESI) Calcd for $C_{15}H_{11}Cl_2O_2$ (M-H): 293.0142; Found: 293.0138.

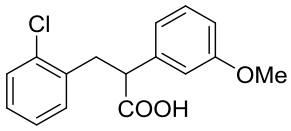
Table 3, 2p



White solid; mp. 81-83 ^{o}C; IR (film) 3414, 1701 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.2 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.17-7.04 (m, 6H), 4.00

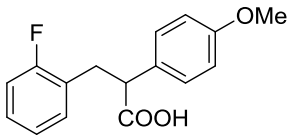
(dd, *J* = 8.7, 6.4 Hz, 1H), 3.48 (dd, *J* = 13.8, 8.8 Hz, 1H), 3.14 (dd, *J* = 13.8, 6.4 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.3, 138.7, 138.0, 136.5, 134.4, 131.6, 129.7, 128.8, 128.7, 128.2, 126.8, 125.2, 51.1, 37.4, 21.6; HRMS (ESI) Calcd for $C_{16}H_{14}ClO_2$ (M-H): 273.0688; Found: 273.0681.

Table 3, 2q



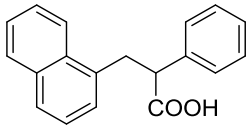
White solid; mp. 69-71 ^{o}C; IR (film) 3425, 1704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 7.7 Hz, 1H), 7.22 (t, *J* = 7.9 Hz, 1H), 7.16-7.09 (m, 1H), 7.09-7.03 (m, 2H), 6.90 (d, *J* = 7.7 Hz, 1H), 6.85 (t, *J* = 2.3 Hz, 1H), 6.84-6.79 (m, 1H), 4.00 (dd, *J* = 8.4, 6.7 Hz, 1H), 3.78 (s, 3H), 3.47 (dd, *J* = 13.8, 8.5 Hz, 1H), 3.14 (dd, *J* = 13.8, 6.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.9, 160.0, 139.5, 136.3, 134.4, 131.7, 129.9, 129.7, 128.3, 126.9, 120.5, 113.9, 113.3, 55.5, 51.1, 37.4; HRMS (ESI) Calcd for $C_{16}H_{14}ClO_3$ (M-H): 289.0637; Found: 289.0630.

Table 3, 2r



White solid; mp. 136-138 ^{o}C; IR (film) 3442, 1698 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.19 (m, 2H), 7.19-7.12 (m, 1H), 7.04 (td, *J* = 7.6, 1.6 Hz, 1H), 7.01-6.92 (m, 2H), 6.86-6.81 (m, 2H), 3.88 (t, *J* = 7.8 Hz, 1H), 3.78 (s, 3H), 3.37 (dd, *J* = 13.9, 8.2 Hz, 1H), 3.06 (dd, *J* = 13.9, 7.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.9, 161.5 (d, *J* = 244 Hz), 159.3, 131.6 (d, *J* = 5 Hz), 130.0, 129.3, 128.5 (d, *J* = 8 Hz), 125.8 (d, *J* = 15 Hz), 124.1 (d, *J* = 4 Hz), 115.4 (d, *J* = 22 Hz), 114.3, 55.5, 50.9, 33.0; HRMS (ESI) Calcd for $C_{16}H_{14}FO_3$ (M-H): 273.0932; Found: 273.0925.

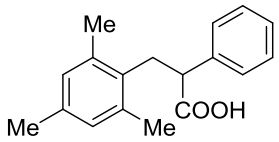
Table 3, 2s



Yellow oil; IR (film) 3417, 1704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.2 Hz, 1H), 7.82 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 8.2 Hz, 1H), 7.52-7.41 (m, 2H), 7.34-7.21 (m, 6H), 7.19-7.13 (m, 1H), 4.05 (dd, *J* = 8.0, 6.8 Hz, 1H), 3.89 (dd, *J* = 14.2, 8.3 Hz, 1H), 3.45 (dd, *J* = 14.2, 6.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.8, 138.4, 134.7, 134.1, 131.8, 129.2, 129.0, 128.2, 127.9, 127.6, 127.3, 126.3, 125.7, 125.6, 123.5, 52.5, 36.6; HRMS (ESI) Calcd for $C_{19}H_{15}O_2$ (M-H): 275.1078; Found: 275.1071.

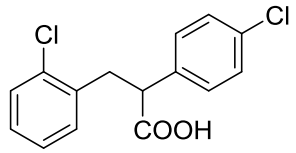
Kubo, T.; Goto, Y.; Uruichi, M.; Yakushi, K.; Nakano, M.; Fuyuhiro, A.; Morita, Y.; Nakasuji, K. *Chem. Asian J.* **2007**, *2*, 1370

Table 3, 2t

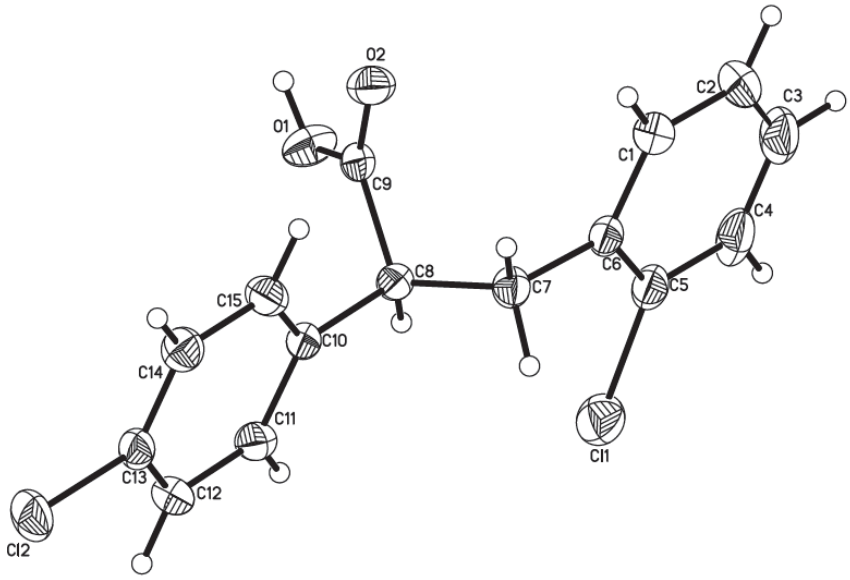


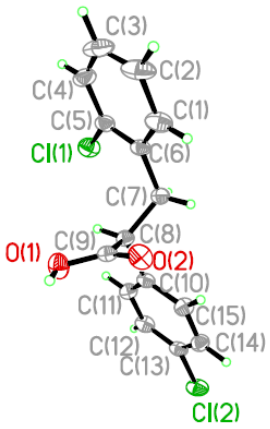
White solid; mp. 151-153 ^{o}C; IR (film) 3420, 1704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.20 (m, 5H), 6.76 (s, 2H), 3.75 (dd, *J* = 7.8, 6.4 Hz, 1H), 3.44 (dd, *J* = 14.1, 6.2 Hz, 1H), 3.02 (dd, *J* = 14.1, 7.9 Hz, 1H), 2.21 (s, 3H), 2.07 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.4, 138.7, 137.0, 135.9, 132.6, 129.3, 128.8, 128.4, 127.7, 51.8, 33.8, 21.0, 20.1; HRMS (ESI) Calcd for $C_{18}H_{19}O_2$ (M-H): 267.1391; Found: 267.1385.

The X-ray structure of compound **2o**



2o





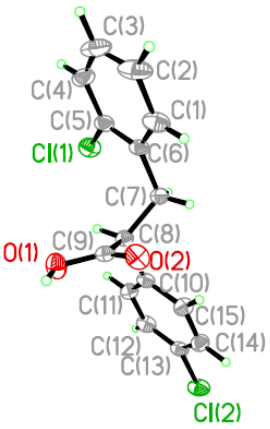


Table 1. Crystal data and structure refinement for **2o**.

Identification code	**2o**	
Empirical formula	C_{15} H_{12} Cl_2 O_2	
Formula weight	295.15	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 6.0590(12) Å	alpha = 91.59(3) deg.
	b = 8.8930(18) Å	beta = 93.75(3) deg.
	c = 13.346(3) Å	gamma = 108.42(3) deg.
Volume	679.9(2) Å ^3	
Z, Calculated density	2, 1.442 Mg/m^3	
Absorption coefficient	0.471 mm^-1	
F(000)	304	
Crystal size	0.30 x 0.20 x 0.10 mm	
Theta range for data collection	1.53 to 25.38 deg.	
Limiting indices	0<=h<=7, -10<=k<=10, -16<=l<=16	
Reflections collected / unique	2759 / 2500 [R(int) = 0.0232]	
Completeness to theta = 25.38	100.0 %	
Absorption correction	Psi-scan	
Max. and min. transmission	0.9544 and 0.8716	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2500 / 0 / 172	
Goodness-of-fit on F^2	1.004	
Final R indices [I>2sigma(I)]	R1 = 0.0514, wR2 = 0.1484	
R indices (all data)	R1 = 0.0734, wR2 = 0.1660	
Largest diff. peak and hole	0.216 and -0.338 e. Å ^-3	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å ^2 x 10^3) for **2o**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cl(1)	10267(2)	806(1)	1363(1)	65(1)
O(1)	11136(4)	4840(3)	3869(2)	55(1)
C(1)	6746(7)	-139(4)	3805(3)	56(1)
Cl(2)	4281(2)	8109(1)	541(1)	64(1)
O(2)	7543(4)	3760(3)	4314(2)	52(1)
C(2)	7629(9)	-1239(4)	4235(3)	74(1)
C(3)	9361(9)	-1659(4)	3808(3)	75(1)
C(4)	10164(7)	-1013(4)	2935(3)	61(1)
C(5)	9262(6)	73(3)	2499(2)	46(1)
C(6)	7535(5)	556(3)	2926(2)	40(1)
C(7)	6563(5)	1774(3)	2480(2)	41(1)
C(8)	8274(5)	3469(3)	2588(2)	36(1)
C(9)	9013(5)	4039(3)	3672(2)	36(1)
C(10)	7248(5)	4635(3)	2070(2)	37(1)
C(11)	8166(6)	5341(4)	1216(2)	43(1)
C(12)	7258(6)	6400(4)	739(2)	49(1)
C(13)	5441(6)	6769(3)	1124(2)	43(1)
C(14)	4496(6)	6089(4)	1972(2)	46(1)
C(15)	5390(6)	5012(4)	2440(2)	45(1)

Table 3. Bond lengths [Å] and angles [deg] for **2o**.

Cl(1)-C(5)	1.738(4)
O(1)-C(9)	1.264(4)
O(1)-H(1B)	0.8200
C(1)-C(2)	1.376(5)
C(1)-C(6)	1.383(5)
C(1)-H(1A)	0.9300
Cl(2)-C(13)	1.740(3)
O(2)-C(9)	1.249(4)
C(2)-C(3)	1.371(6)
C(2)-H(2A)	0.9300
C(3)-C(4)	1.363(6)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.376(5)
C(4)-H(4A)	0.9300
C(5)-C(6)	1.397(4)
C(6)-C(7)	1.506(4)
C(7)-C(8)	1.532(4)
C(7)-H(7A)	0.9700
C(7)-H(7B)	0.9700
C(8)-C(9)	1.510(4)
C(8)-C(10)	1.527(4)
C(8)-H(8A)	0.9800
C(10)-C(11)	1.380(4)
C(10)-C(15)	1.388(4)
C(11)-C(12)	1.382(4)
C(11)-H(11A)	0.9300
C(12)-C(13)	1.370(5)
C(12)-H(12A)	0.9300

C(13)-C(14)	1.370(5)
C(14)-C(15)	1.385(4)
C(14)-H(14A)	0.9300
C(15)-H(15A)	0.9300

C(9)-O(1)-H(1B)	109.5
C(2)-C(1)-C(6)	121.6(4)
C(2)-C(1)-H(1A)	119.2
C(6)-C(1)-H(1A)	119.2
C(3)-C(2)-C(1)	120.5(4)
C(3)-C(2)-H(2A)	119.7
C(1)-C(2)-H(2A)	119.7
C(4)-C(3)-C(2)	119.5(3)
C(4)-C(3)-H(3A)	120.2
C(2)-C(3)-H(3A)	120.2
C(3)-C(4)-C(5)	119.9(4)
C(3)-C(4)-H(4A)	120.0
C(5)-C(4)-H(4A)	120.0
C(4)-C(5)-C(6)	122.0(3)
C(4)-C(5)-Cl(1)	118.1(3)
C(6)-C(5)-Cl(1)	119.8(2)
C(1)-C(6)-C(5)	116.4(3)
C(1)-C(6)-C(7)	120.5(3)
C(5)-C(6)-C(7)	123.2(3)
C(6)-C(7)-C(8)	113.7(2)
C(6)-C(7)-H(7A)	108.8
C(8)-C(7)-H(7A)	108.8
C(6)-C(7)-H(7B)	108.8
C(8)-C(7)-H(7B)	108.8
H(7A)-C(7)-H(7B)	107.7

C(9)-C(8)-C(10)	109.0(2)
C(9)-C(8)-C(7)	112.7(2)
C(10)-C(8)-C(7)	111.4(2)
C(9)-C(8)-H(8A)	107.9
C(10)-C(8)-H(8A)	107.9
C(7)-C(8)-H(8A)	107.9
O(2)-C(9)-O(1)	123.3(3)
O(2)-C(9)-C(8)	120.2(3)
O(1)-C(9)-C(8)	116.4(3)
C(11)-C(10)-C(15)	118.5(3)
C(11)-C(10)-C(8)	120.2(3)
C(15)-C(10)-C(8)	121.4(3)
C(10)-C(11)-C(12)	120.9(3)
C(10)-C(11)-H(11A)	119.6
C(12)-C(11)-H(11A)	119.6
C(13)-C(12)-C(11)	119.7(3)
C(13)-C(12)-H(12A)	120.2
C(11)-C(12)-H(12A)	120.2
C(14)-C(13)-C(12)	120.8(3)
C(14)-C(13)-Cl(2)	118.7(3)
C(12)-C(13)-Cl(2)	120.5(2)
C(13)-C(14)-C(15)	119.4(3)
C(13)-C(14)-H(14A)	120.3
C(15)-C(14)-H(14A)	120.3
C(14)-C(15)-C(10)	120.8(3)
C(14)-C(15)-H(15A)	119.6
C(10)-C(15)-H(15A)	119.6

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Å ^2 x 10^3) for **2o**. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12].

	U11	U22	U33	U23	U13	U12
Cl(1)	76(1)	58(1)	65(1)	-1(1)	21(1)	24(1)
O(1)	48(1)	70(2)	41(1)	-12(1)	-3(1)	13(1)
C(1)	81(2)	42(2)	43(2)	2(1)	6(2)	18(2)
Cl(2)	86(1)	46(1)	64(1)	12(1)	-14(1)	30(1)
O(2)	59(1)	55(1)	36(1)	1(1)	7(1)	10(1)
C(2)	122(4)	46(2)	52(2)	13(2)	-4(2)	27(2)
C(3)	112(3)	41(2)	75(3)	-2(2)	-32(3)	35(2)
C(4)	70(2)	39(2)	78(3)	-13(2)	-17(2)	27(2)
C(5)	54(2)	33(2)	48(2)	-6(1)	-6(2)	13(1)
C(6)	51(2)	29(1)	39(2)	-3(1)	-5(1)	12(1)
C(7)	45(2)	35(2)	41(2)	1(1)	0(1)	15(1)
C(8)	43(2)	35(2)	33(2)	0(1)	0(1)	15(1)
C(9)	44(2)	30(1)	36(2)	2(1)	2(1)	16(1)
C(10)	46(2)	30(1)	33(1)	0(1)	-2(1)	12(1)
C(11)	52(2)	44(2)	36(2)	1(1)	4(1)	20(1)
C(12)	66(2)	44(2)	36(2)	9(1)	4(2)	14(2)
C(13)	54(2)	32(2)	40(2)	1(1)	-12(1)	13(1)
C(14)	49(2)	46(2)	48(2)	2(1)	-1(1)	21(2)
C(15)	52(2)	46(2)	40(2)	9(1)	9(1)	19(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å ^2 x 10^3) for **2o**.

	x	y	z	U(eq)
H(1B)	11331	5214	4449	82
H(1A)	5590	143	4112	67
H(2A)	7048	-1700	4821	89
H(3A)	9983	-2381	4112	90
H(4A)	11320	-1305	2635	74
H(7A)	5166	1748	2805	49
H(7B)	6116	1490	1772	49
H(8A)	9674	3476	2258	44
H(11A)	9412	5102	959	52
H(12A)	7877	6860	159	59
H(14A)	3266	6348	2230	55
H(15A)	4736	4535	3010	54

Table 6. Torsion angles [deg] for **2o**.

C(6)-C(1)-C(2)-C(3)	1.0(6)
C(1)-C(2)-C(3)-C(4)	-1.8(6)
C(2)-C(3)-C(4)-C(5)	1.0(6)
C(3)-C(4)-C(5)-C(6)	0.5(5)
C(3)-C(4)-C(5)-Cl(1)	-177.7(3)
C(2)-C(1)-C(6)-C(5)	0.4(5)
C(2)-C(1)-C(6)-C(7)	-179.1(3)
C(4)-C(5)-C(6)-C(1)	-1.2(4)
Cl(1)-C(5)-C(6)-C(1)	176.9(2)
C(4)-C(5)-C(6)-C(7)	178.3(3)
Cl(1)-C(5)-C(6)-C(7)	-3.6(4)
C(1)-C(6)-C(7)-C(8)	108.0(3)
C(5)-C(6)-C(7)-C(8)	-71.4(4)
C(6)-C(7)-C(8)-C(9)	-60.4(3)
C(6)-C(7)-C(8)-C(10)	176.7(2)
C(10)-C(8)-C(9)-O(2)	81.7(3)
C(7)-C(8)-C(9)-O(2)	-42.5(4)
C(10)-C(8)-C(9)-O(1)	-96.0(3)
C(7)-C(8)-C(9)-O(1)	139.8(3)
C(9)-C(8)-C(10)-C(11)	123.7(3)
C(7)-C(8)-C(10)-C(11)	-111.3(3)
C(9)-C(8)-C(10)-C(15)	-56.2(3)
C(7)-C(8)-C(10)-C(15)	68.8(3)
C(15)-C(10)-C(11)-C(12)	0.0(4)
C(8)-C(10)-C(11)-C(12)	-180.0(3)
C(10)-C(11)-C(12)-C(13)	0.8(5)
C(11)-C(12)-C(13)-C(14)	-0.7(5)
C(11)-C(12)-C(13)-Cl(2)	179.6(2)

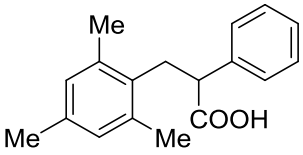
C(12)-C(13)-C(14)-C(15)	-0.2(5)
Cl(2)-C(13)-C(14)-C(15)	179.5(2)
C(13)-C(14)-C(15)-C(10)	1.0(5)
C(11)-C(10)-C(15)-C(14)	-0.9(4)
C(8)-C(10)-C(15)-C(14)	179.1(3)

Symmetry transformations used to generate equivalent atoms:

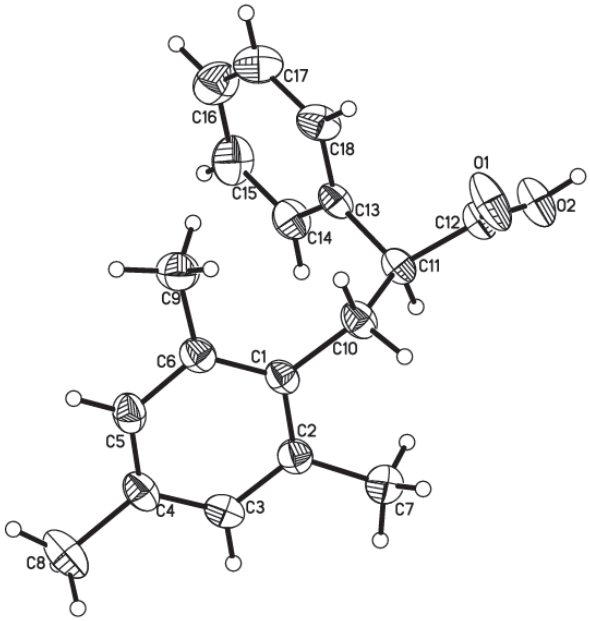
Table 7. Hydrogen bonds for **2o** [Å and deg.].

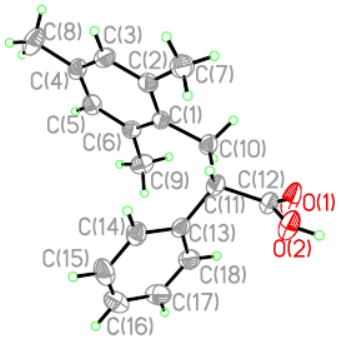
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

The X-ray structure of compound **2t**



2t





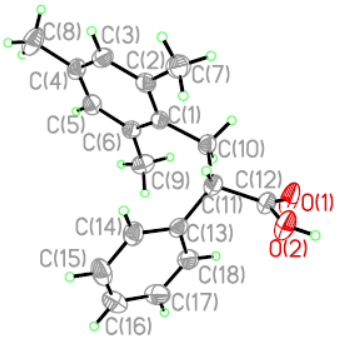


Table 1. Crystal data and structure refinement for **2t**.

Identification code	**2t**	
Empirical formula	$C_{18}H_{20}O_2$	
Formula weight	268.34	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 8.2600(17) Å	alpha = 102.50(3) deg.
	b = 10.221(2) Å	beta = 107.52(3) deg.
	c = 10.602(2) Å	gamma = 109.03(3) deg.
Volume	756.2(3) Å ^3	
Z, Calculated density	2, 1.179 Mg/m^3	
Absorption coefficient	0.075 mm^-1	
F(000)	288	
Crystal size	0.30 x 0.20 x 0.10 mm	
Theta range for data collection	2.15 to 25.38 deg.	
Limiting indices	0<=h<=9, -12<=k<=11, -12<=l<=12	
Reflections collected / unique	2984 / 2776 [R(int) = 0.0150]	
Completeness to theta = 25.38	100.0 %	
Absorption correction	None	
Max. and min. transmission	0.9925 and 0.9778	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2776 / 0 / 182	
Goodness-of-fit on F^2	1.001	
Final R indices [I>2sigma(I)]	R1 = 0.0617, wR2 = 0.1680	
R indices (all data)	R1 = 0.0901, wR2 = 0.1912	
Extinction coefficient	0.119(14)	
Largest diff. peak and hole	0.251 and -0.200 e. Å ^-3	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å ^2 x 10^3) for **2t**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	4944(3)	6652(2)	6498(2)	45(1)
O(1)	8011(3)	4842(2)	9025(3)	88(1)
O(2)	10578(3)	6919(2)	10010(2)	71(1)
C(2)	5619(4)	7387(3)	5661(3)	49(1)
C(3)	4522(4)	7920(3)	4840(3)	55(1)
C(4)	2773(4)	7744(3)	4799(3)	55(1)
C(5)	2144(4)	7041(3)	5638(3)	56(1)
C(6)	3195(4)	6496(3)	6496(3)	50(1)
C(7)	7481(4)	7577(4)	5601(3)	68(1)
C(8)	1581(5)	8299(4)	3857(4)	85(1)
C(9)	2373(4)	5743(4)	7367(3)	70(1)
C(10)	6036(4)	5955(3)	7306(3)	53(1)
C(11)	7785(3)	7016(3)	8664(3)	48(1)
C(12)	8796(4)	6144(3)	9243(3)	50(1)
C(13)	7336(3)	7843(3)	9778(2)	47(1)
C(14)	7575(4)	9292(3)	9958(3)	58(1)
C(15)	7127(5)	10031(3)	10952(4)	74(1)
C(16)	6421(5)	9358(4)	11771(3)	78(1)
C(17)	6150(5)	7918(4)	11595(3)	76(1)
C(18)	6640(4)	7185(3)	10617(3)	62(1)

Table 3. Bond lengths [Å] and angles [deg] for **2t.**

C(1)-C(6)	1.400(4)
C(1)-C(2)	1.406(3)
C(1)-C(10)	1.508(3)
O(1)-C(12)	1.208(3)
O(2)-C(12)	1.302(3)
O(2)-H(2A)	0.8200
C(2)-C(3)	1.388(4)
C(2)-C(7)	1.509(4)
C(3)-C(4)	1.382(4)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.375(4)
C(4)-C(8)	1.518(4)
C(5)-C(6)	1.397(4)
C(5)-H(5A)	0.9300
C(6)-C(9)	1.500(4)
C(7)-H(7A)	0.9600
C(7)-H(7B)	0.9600
C(7)-H(7C)	0.9600
C(8)-H(8A)	0.9600
C(8)-H(8B)	0.9600
C(8)-H(8C)	0.9600
C(9)-H(9A)	0.9600
C(9)-H(9B)	0.9600
C(9)-H(9C)	0.9600
C(10)-C(11)	1.545(4)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(11)-C(12)	1.510(3)

C(11)-C(13)	1.511(4)
C(11)-H(11A)	0.9800
C(13)-C(18)	1.375(4)
C(13)-C(14)	1.390(4)
C(14)-C(15)	1.375(4)
C(14)-H(14A)	0.9300
C(15)-C(16)	1.367(4)
C(15)-H(15A)	0.9300
C(16)-C(17)	1.376(5)
C(16)-H(16A)	0.9300
C(17)-C(18)	1.383(4)
C(17)-H(17A)	0.9300
C(18)-H(18A)	0.9300

C(6)-C(1)-C(2)	119.0(2)
C(6)-C(1)-C(10)	120.7(2)
C(2)-C(1)-C(10)	120.1(2)
C(12)-O(2)-H(2A)	109.5
C(3)-C(2)-C(1)	119.2(2)
C(3)-C(2)-C(7)	119.1(2)
C(1)-C(2)-C(7)	121.7(2)
C(4)-C(3)-C(2)	122.5(3)
C(4)-C(3)-H(3A)	118.7
C(2)-C(3)-H(3A)	118.7
C(5)-C(4)-C(3)	117.6(2)
C(5)-C(4)-C(8)	121.0(3)
C(3)-C(4)-C(8)	121.4(3)
C(4)-C(5)-C(6)	122.3(2)
C(4)-C(5)-H(5A)	118.9
C(6)-C(5)-H(5A)	118.9

C(5)-C(6)-C(1)	119.3(2)
C(5)-C(6)-C(9)	117.9(2)
C(1)-C(6)-C(9)	122.8(2)
C(2)-C(7)-H(7A)	109.5
C(2)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(2)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(4)-C(8)-H(8A)	109.5
C(4)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(4)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(6)-C(9)-H(9A)	109.5
C(6)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(6)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(1)-C(10)-C(11)	116.5(2)
C(1)-C(10)-H(10A)	108.2
C(11)-C(10)-H(10A)	108.2
C(1)-C(10)-H(10B)	108.2
C(11)-C(10)-H(10B)	108.2
H(10A)-C(10)-H(10B)	107.3
C(12)-C(11)-C(13)	110.2(2)
C(12)-C(11)-C(10)	108.9(2)
C(13)-C(11)-C(10)	113.2(2)

C(12)-C(11)-H(11A)	108.1
C(13)-C(11)-H(11A)	108.1
C(10)-C(11)-H(11A)	108.1
O(1)-C(12)-O(2)	122.9(2)
O(1)-C(12)-C(11)	122.7(2)
O(2)-C(12)-C(11)	114.3(2)
C(18)-C(13)-C(14)	118.0(3)
C(18)-C(13)-C(11)	121.5(2)
C(14)-C(13)-C(11)	120.5(2)
C(15)-C(14)-C(13)	120.4(3)
C(15)-C(14)-H(14A)	119.8
C(13)-C(14)-H(14A)	119.8
C(16)-C(15)-C(14)	121.0(3)
C(16)-C(15)-H(15A)	119.5
C(14)-C(15)-H(15A)	119.5
C(15)-C(16)-C(17)	119.5(3)
C(15)-C(16)-H(16A)	120.3
C(17)-C(16)-H(16A)	120.3
C(16)-C(17)-C(18)	119.5(3)
C(16)-C(17)-H(17A)	120.2
C(18)-C(17)-H(17A)	120.2
C(13)-C(18)-C(17)	121.6(3)
C(13)-C(18)-H(18A)	119.2
C(17)-C(18)-H(18A)	119.2

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Å ^2 x 10^3) for **2t**. The anisotropic isplacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	49(1)	40(1)	38(1)	7(1)	8(1)	21(1)
O(1)	59(1)	53(1)	123(2)	39(1)	-4(1)	22(1)
O(2)	52(1)	58(1)	89(2)	32(1)	6(1)	24(1)
C(2)	50(2)	47(1)	41(1)	8(1)	12(1)	21(1)
C(3)	60(2)	52(2)	45(1)	19(1)	15(1)	21(1)
C(4)	53(2)	48(1)	50(2)	12(1)	4(1)	22(1)
C(5)	43(1)	57(2)	54(2)	10(1)	9(1)	22(1)
C(6)	50(2)	45(1)	41(1)	8(1)	10(1)	18(1)
C(7)	65(2)	85(2)	64(2)	29(2)	28(2)	37(2)
C(8)	71(2)	79(2)	87(2)	40(2)	1(2)	31(2)
C(9)	62(2)	78(2)	62(2)	29(2)	23(2)	22(2)
C(10)	58(2)	44(1)	49(1)	12(1)	10(1)	24(1)
C(11)	47(1)	42(1)	50(1)	19(1)	11(1)	19(1)
C(12)	45(1)	45(1)	54(2)	19(1)	11(1)	20(1)
C(13)	43(1)	41(1)	42(1)	12(1)	3(1)	16(1)
C(14)	60(2)	45(1)	66(2)	22(1)	20(1)	22(1)
C(15)	79(2)	46(2)	89(2)	10(2)	30(2)	28(2)
C(16)	87(2)	71(2)	63(2)	6(2)	30(2)	31(2)
C(17)	89(2)	69(2)	56(2)	19(2)	27(2)	23(2)
C(18)	77(2)	48(2)	51(2)	18(1)	18(1)	23(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å ^2 x 10^3) for **2t**.

	x	y	z	U(eq)
H(2A)	11035	6390	10311	107
H(3A)	4983	8415	4296	65
H(5A)	977	6924	5634	67
H(7A)	7692	8097	4972	102
H(7B)	7477	6624	5266	102
H(7C)	8459	8130	6528	102
H(8A)	426	8086	3975	128
H(8B)	1323	7817	2890	128
H(8C)	2241	9347	4112	128
H(9A)	1172	5754	7213	104
H(9B)	3189	6254	8348	104
H(9C)	2232	4740	7099	104
H(10A)	6427	5410	6680	63
H(10B)	5201	5245	7557	63
H(11A)	8620	7741	8412	58
H(14A)	8040	9764	9403	70
H(15A)	7307	11004	11070	89
H(16A)	6126	9869	12442	93
H(17A)	5642	7440	12130	91
H(18A)	6494	6223	10525	74

Table 6. Torsion angles [deg] for **2t**.

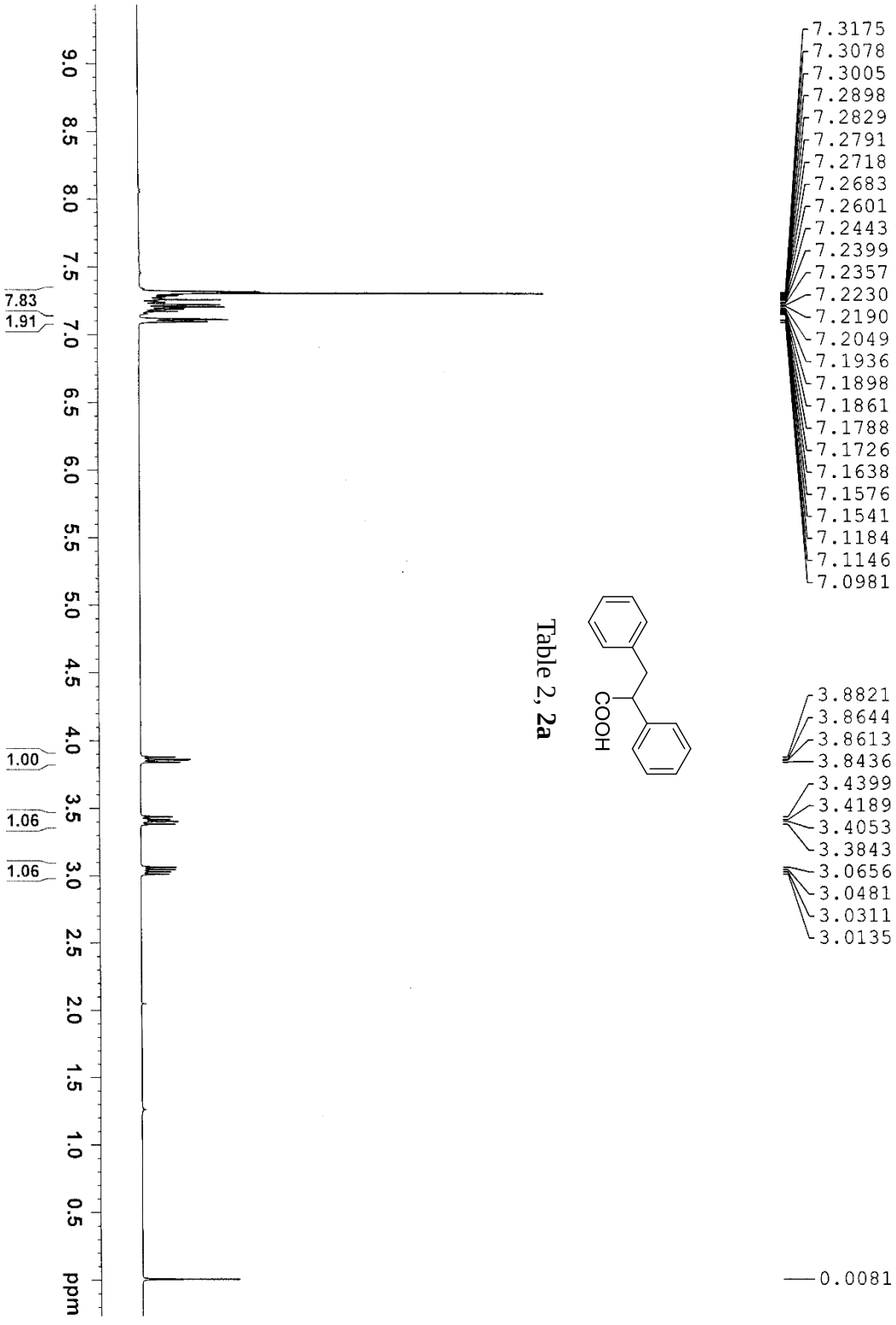
C(6)-C(1)-C(2)-C(3)	-0.9(3)
C(10)-C(1)-C(2)-C(3)	175.0(2)
C(6)-C(1)-C(2)-C(7)	-179.2(2)
C(10)-C(1)-C(2)-C(7)	-3.4(4)
C(1)-C(2)-C(3)-C(4)	-0.7(4)
C(7)-C(2)-C(3)-C(4)	177.7(2)
C(2)-C(3)-C(4)-C(5)	1.6(4)
C(2)-C(3)-C(4)-C(8)	-178.2(2)
C(3)-C(4)-C(5)-C(6)	-0.9(4)
C(8)-C(4)-C(5)-C(6)	178.9(2)
C(4)-C(5)-C(6)-C(1)	-0.7(4)
C(4)-C(5)-C(6)-C(9)	-179.9(2)
C(2)-C(1)-C(6)-C(5)	1.5(3)
C(10)-C(1)-C(6)-C(5)	-174.3(2)
C(2)-C(1)-C(6)-C(9)	-179.3(2)
C(10)-C(1)-C(6)-C(9)	4.8(4)
C(6)-C(1)-C(10)-C(11)	-107.8(3)
C(2)-C(1)-C(10)-C(11)	76.5(3)
C(1)-C(10)-C(11)-C(12)	-174.4(2)
C(1)-C(10)-C(11)-C(13)	62.6(3)
C(13)-C(11)-C(12)-O(1)	98.5(3)
C(10)-C(11)-C(12)-O(1)	-26.3(4)
C(13)-C(11)-C(12)-O(2)	-80.8(3)
C(10)-C(11)-C(12)-O(2)	154.5(2)
C(12)-C(11)-C(13)-C(18)	-43.7(3)
C(10)-C(11)-C(13)-C(18)	78.5(3)
C(12)-C(11)-C(13)-C(14)	137.9(2)
C(10)-C(11)-C(13)-C(14)	-99.9(3)

C(18)-C(13)-C(14)-C(15)	0.2(4)
C(11)-C(13)-C(14)-C(15)	178.6(2)
C(13)-C(14)-C(15)-C(16)	-0.7(5)
C(14)-C(15)-C(16)-C(17)	-0.2(5)
C(15)-C(16)-C(17)-C(18)	1.6(5)
C(14)-C(13)-C(18)-C(17)	1.3(4)
C(11)-C(13)-C(18)-C(17)	-177.1(3)
C(16)-C(17)-C(18)-C(13)	-2.2(5)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **2t** [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)


7.3175
7.3078
7.3005
7.2898
7.2829
7.2791
7.2718
7.2683
7.2601
7.2443
7.2399
7.2357
7.2230
7.2190
7.2049
7.1936
7.1898
7.1861
7.1788
7.1726
7.1638
7.1576
7.1541
7.1184
7.1146
7.0981
3.8821
3.8644
3.8613
3.8436
3.4399
3.4189
3.4053
3.3843
3.0656
3.0481
3.0311
3.0135
0.0081
7.83
1.91
1.00
1.06
1.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
COOH
Table 2, **2a**

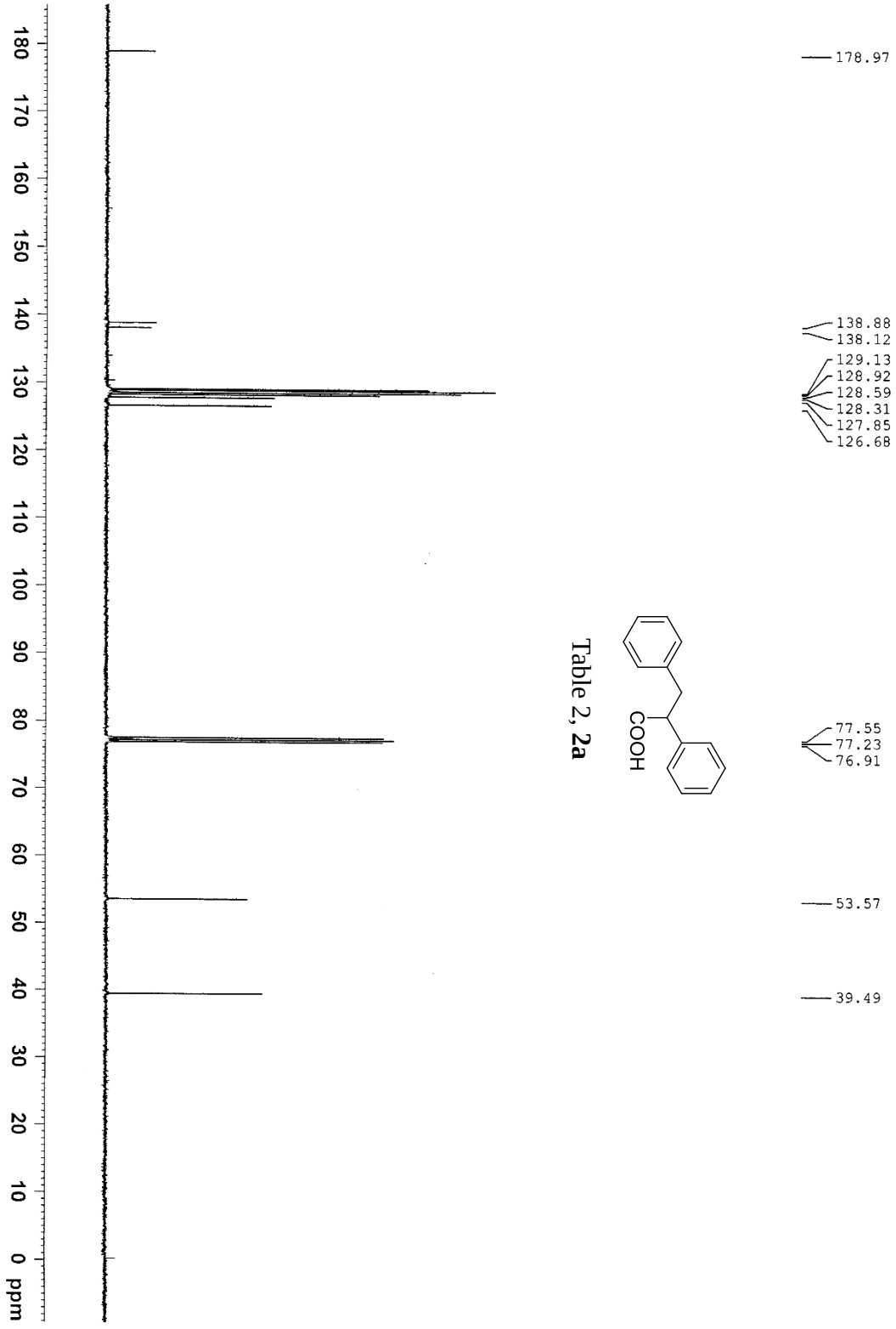

178.97
138.88
138.12
129.13
128.92
128.59
128.31
127.85
126.68
77.55
77.23
76.91
53.57
39.49
COOH
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Table 2, **2a**

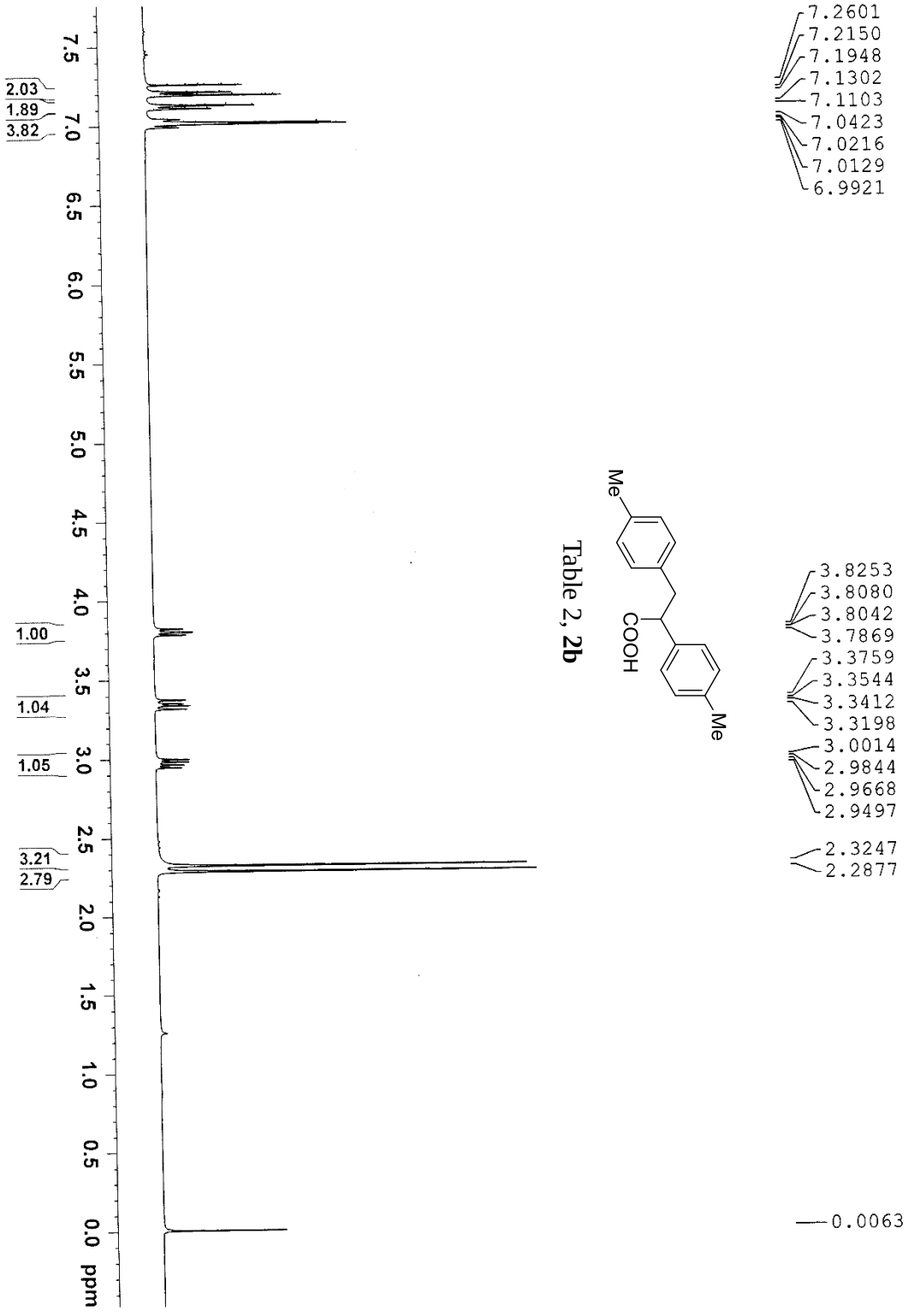

7.2601
7.2150
7.1948
7.1302
7.1103
7.0423
7.0216
7.0129
6.9921
3.8253
3.8080
3.8042
3.7869
3.3759
3.3544
3.3412
3.3198
3.0014
2.9844
2.9668
2.9497
2.3247
2.2877
0.0063
2.03
1.89
3.82
1.00
1.04
1.05
3.21
2.79
Me
COOH
Me
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

Table 2, **2b**

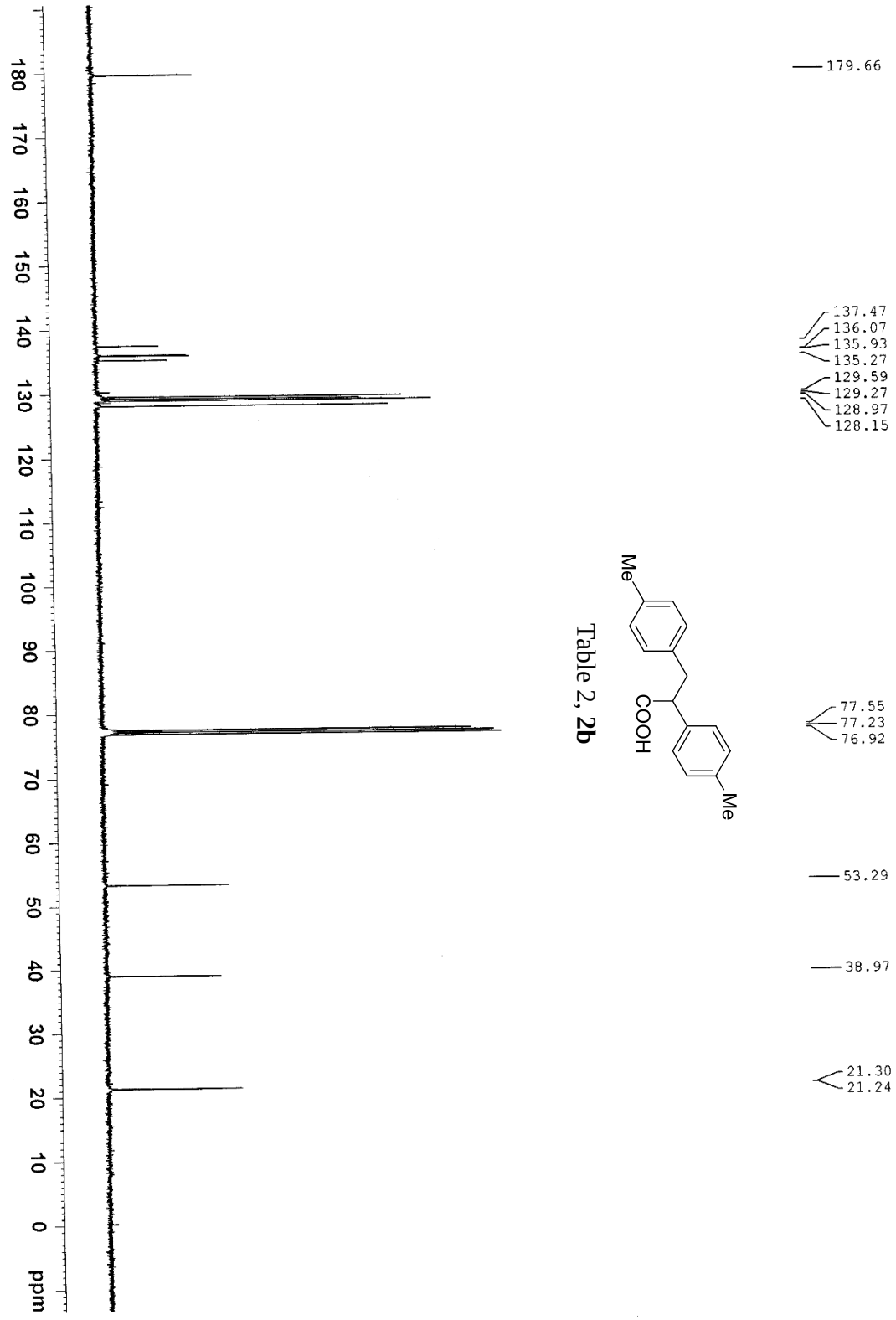

179.66
137.47
136.07
135.93
135.27
129.59
129.27
128.97
128.15
77.55
77.23
76.92
53.29
38.97
21.30
21.24
Me
COOH
Me
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Table 2, **2b**

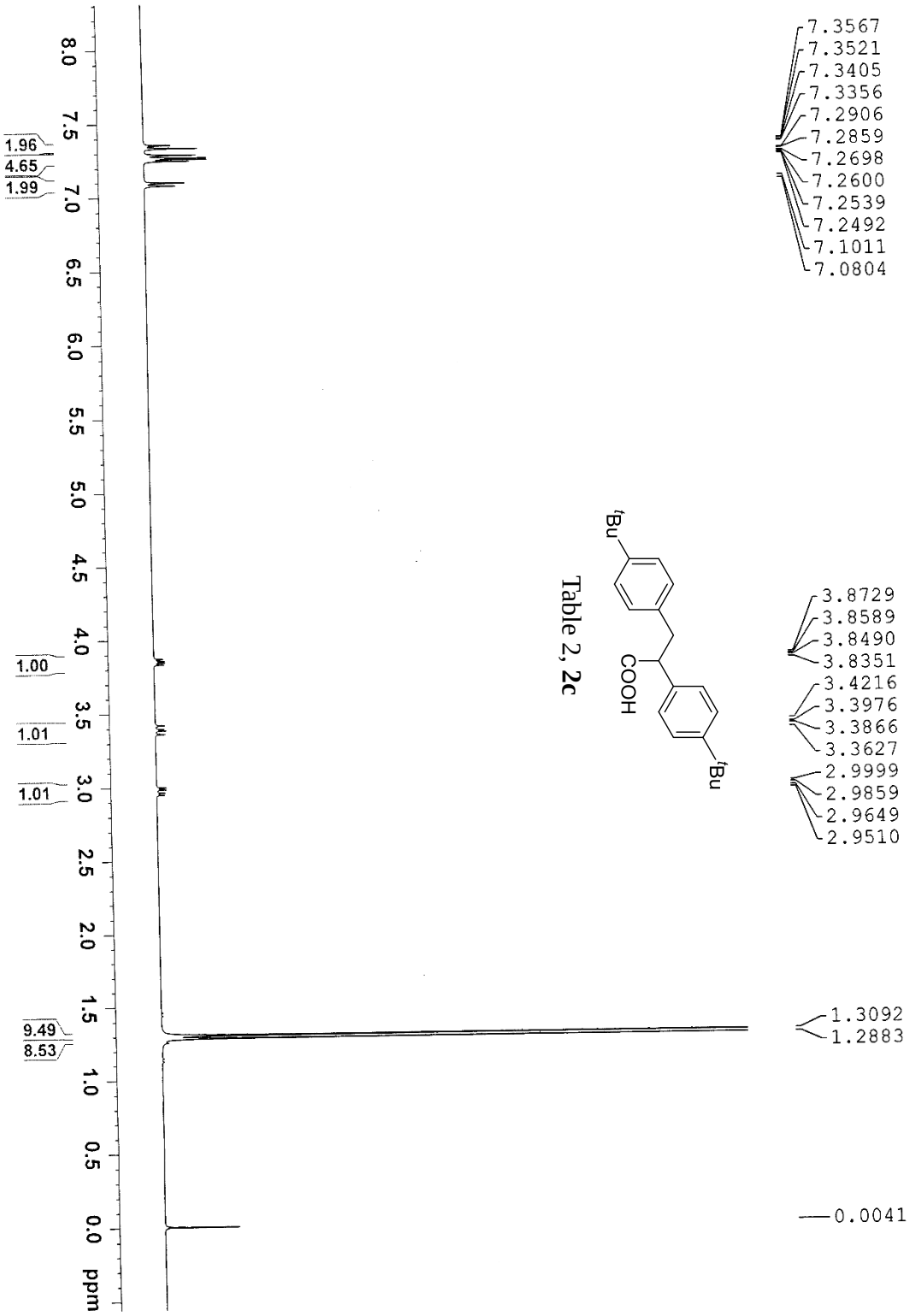

7.3567
7.3521
7.3405
7.3356
7.2906
7.2859
7.2698
7.2600
7.2539
7.2492
7.1011
7.0804
3.8729
3.8589
3.8490
3.8351
3.4216
3.3976
3.3866
3.3627
2.9999
2.9859
2.9649
2.9510
1.3092
1.2883
0.0041
1.96
4.65
1.99
1.00
1.01
1.01
9.49
8.53
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
tBu
COOH
tBu

Table 2, **2c**

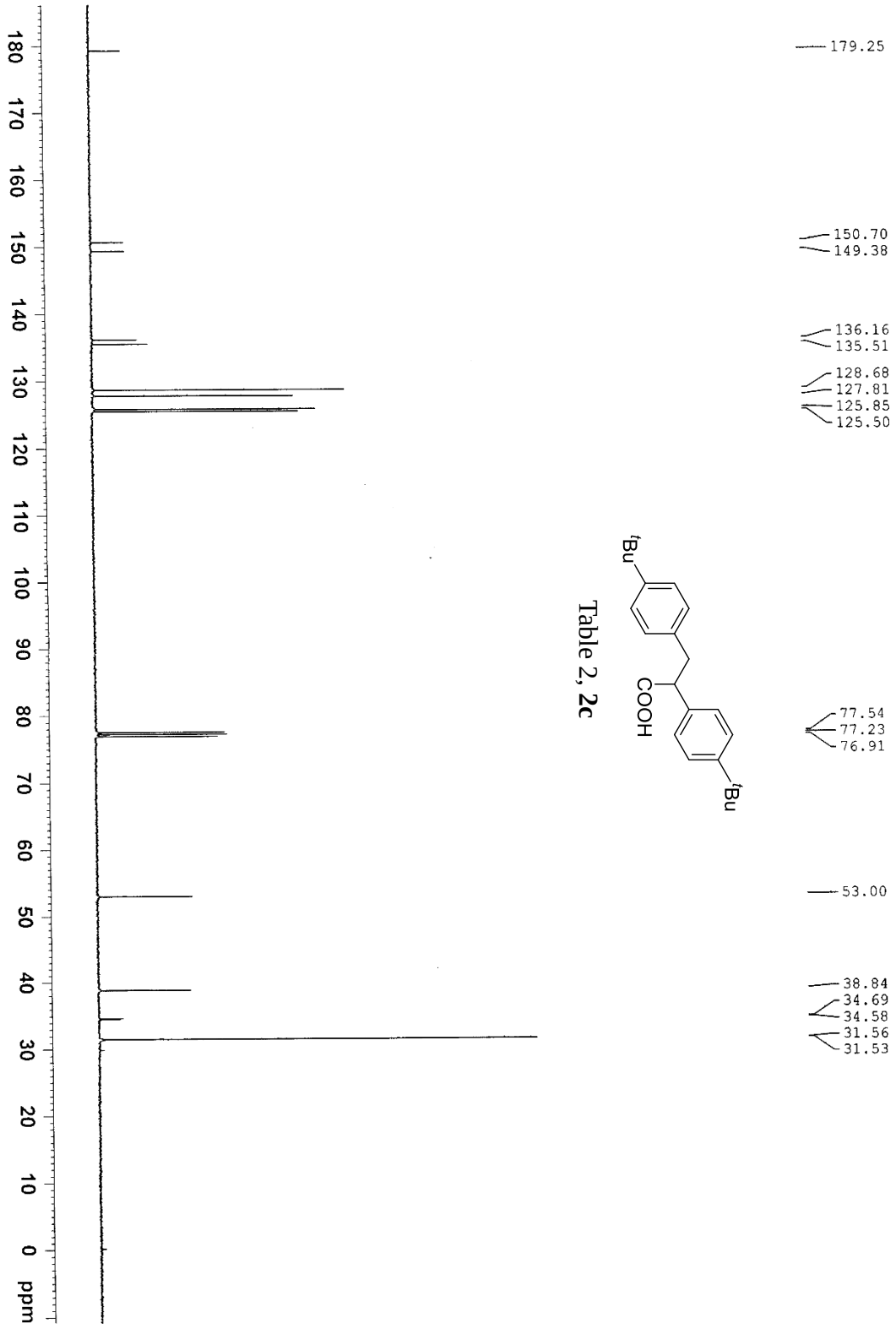

179.25
150.70
149.38
136.16
135.51
128.68
127.81
125.85
125.50
77.54
77.23
76.91
53.00
38.84
34.69
34.58
31.56
31.53
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Table 2, **2c**

tBu
COOH
tBu

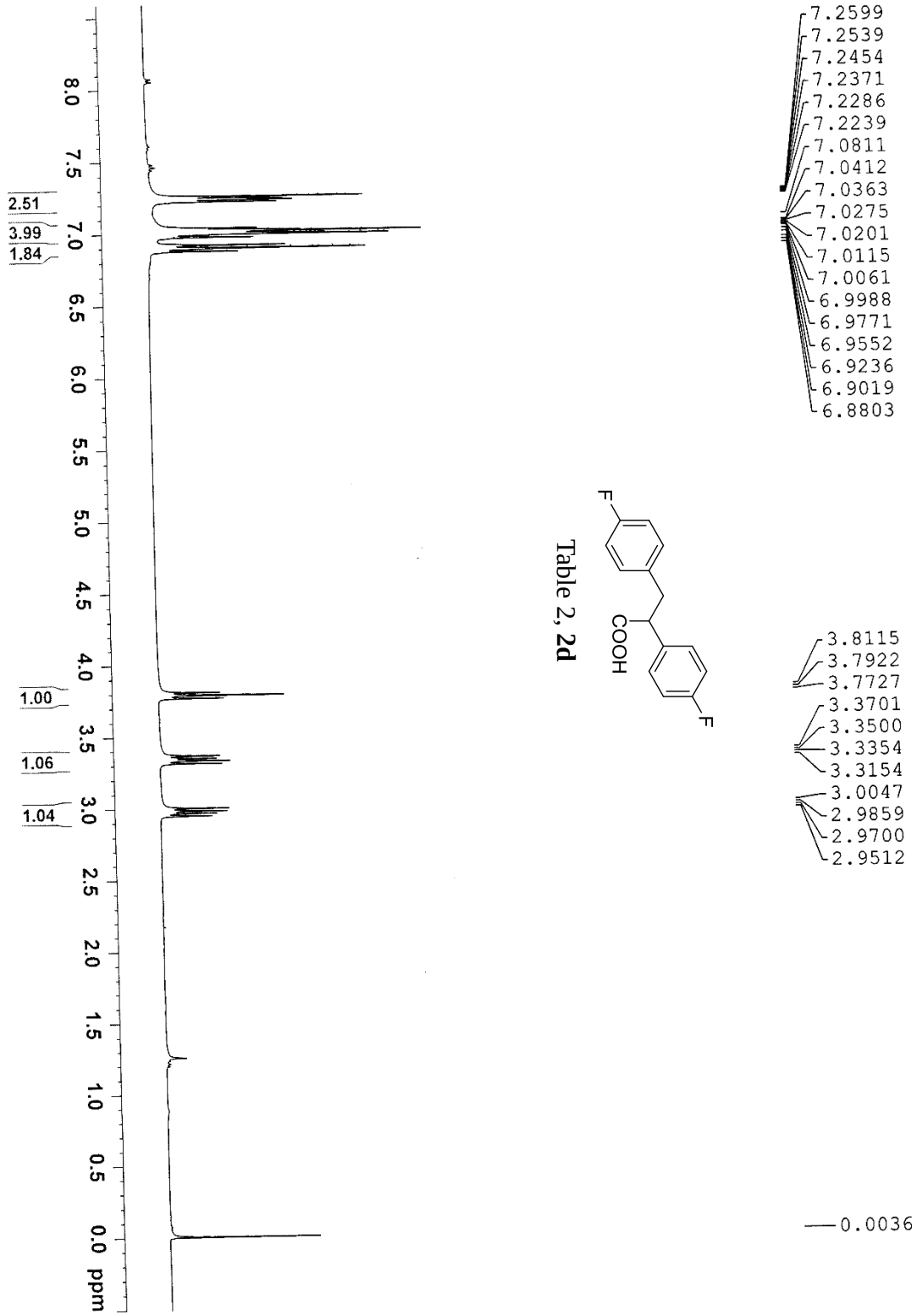

7.2599
7.2539
7.2454
7.2371
7.2286
7.2239
7.0811
7.0412
7.0363
7.0275
7.0201
7.0115
7.0061
6.9988
6.9771
6.9552
6.9236
6.9019
6.8803
3.8115
3.7922
3.7727
3.3701
3.3500
3.3354
3.3154
3.0047
2.9859
2.9700
2.9512
0.0036
2.51
3.99
1.84
1.00
1.06
1.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
F
COOH
F

Table 2, **2d**

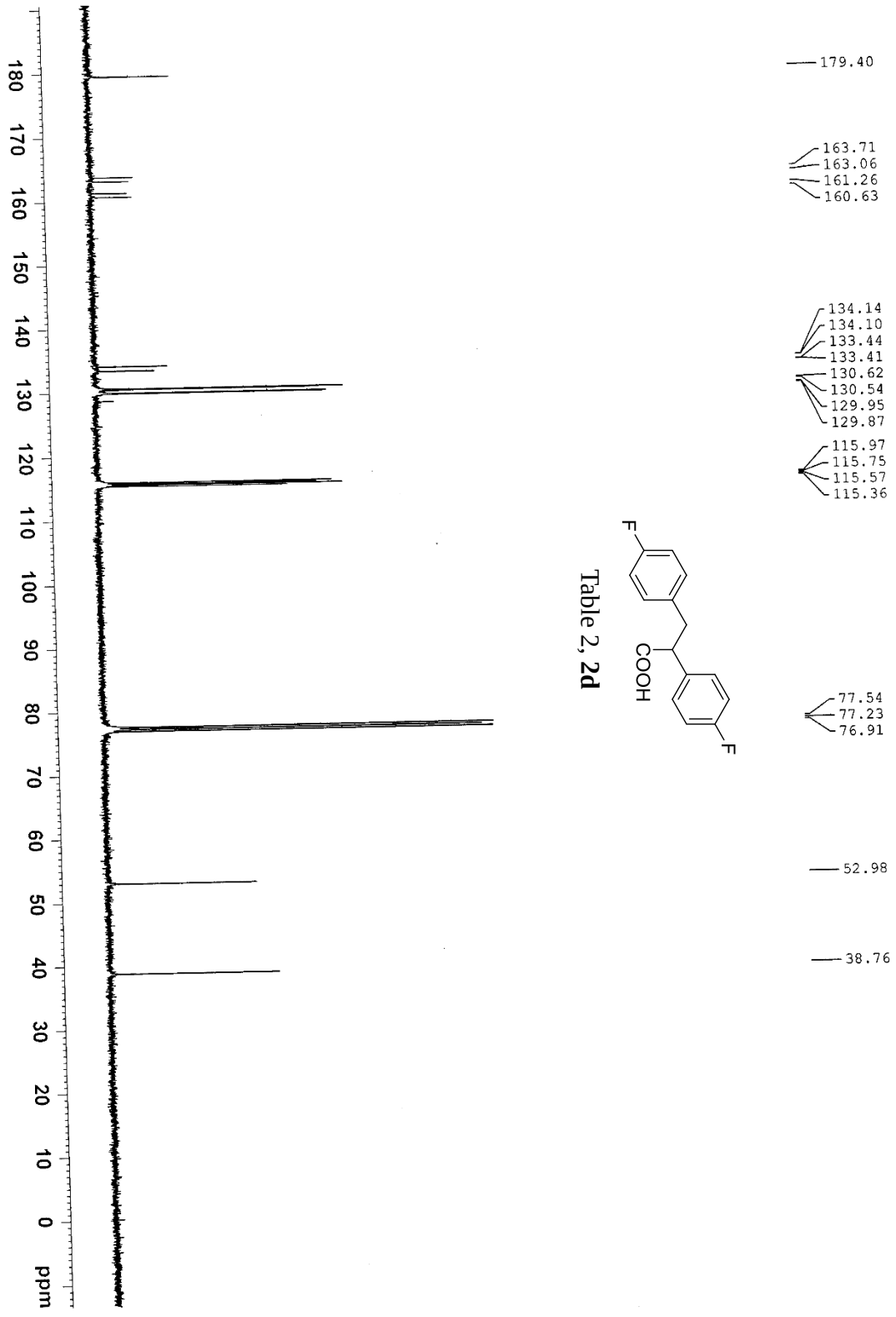

179.40
163.71
163.06
161.26
160.63
134.14
134.10
133.44
133.41
130.62
130.54
129.95
129.87
115.97
115.75
115.57
115.36
77.54
77.23
76.91
52.98
38.76
F
COOH
F
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Table 2, **2d**


7.2963
7.2919
7.2797
7.2752
7.2574
7.2129
7.2079
7.2009
7.1963
7.1917
7.1846
7.1801
7.0086
6.9878
Cl
COOH
Cl
Table 2, **2e**
3.8070
3.7876
3.7682
3.3666
3.3468
3.3319
3.3121
3.0022
2.9833
2.9675
2.9486
0.0000
2.31
3.81
2.03
1.00
1.04
1.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

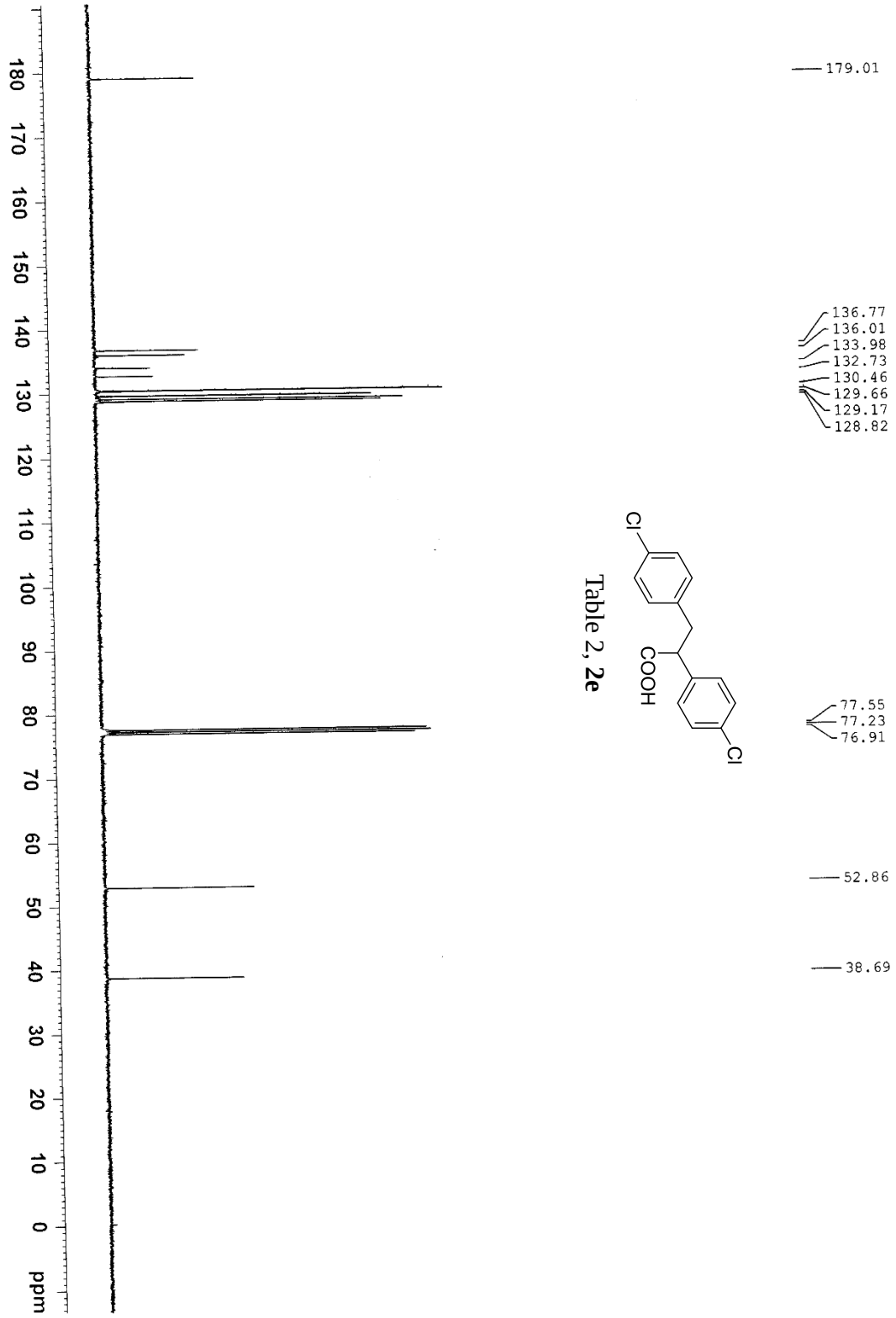

179.01
136.77
136.01
133.98
132.73
130.46
129.66
129.17
128.82
77.55
77.23
76.91
52.86
38.69
Cl
COOH
Cl
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Table 2, **2e**

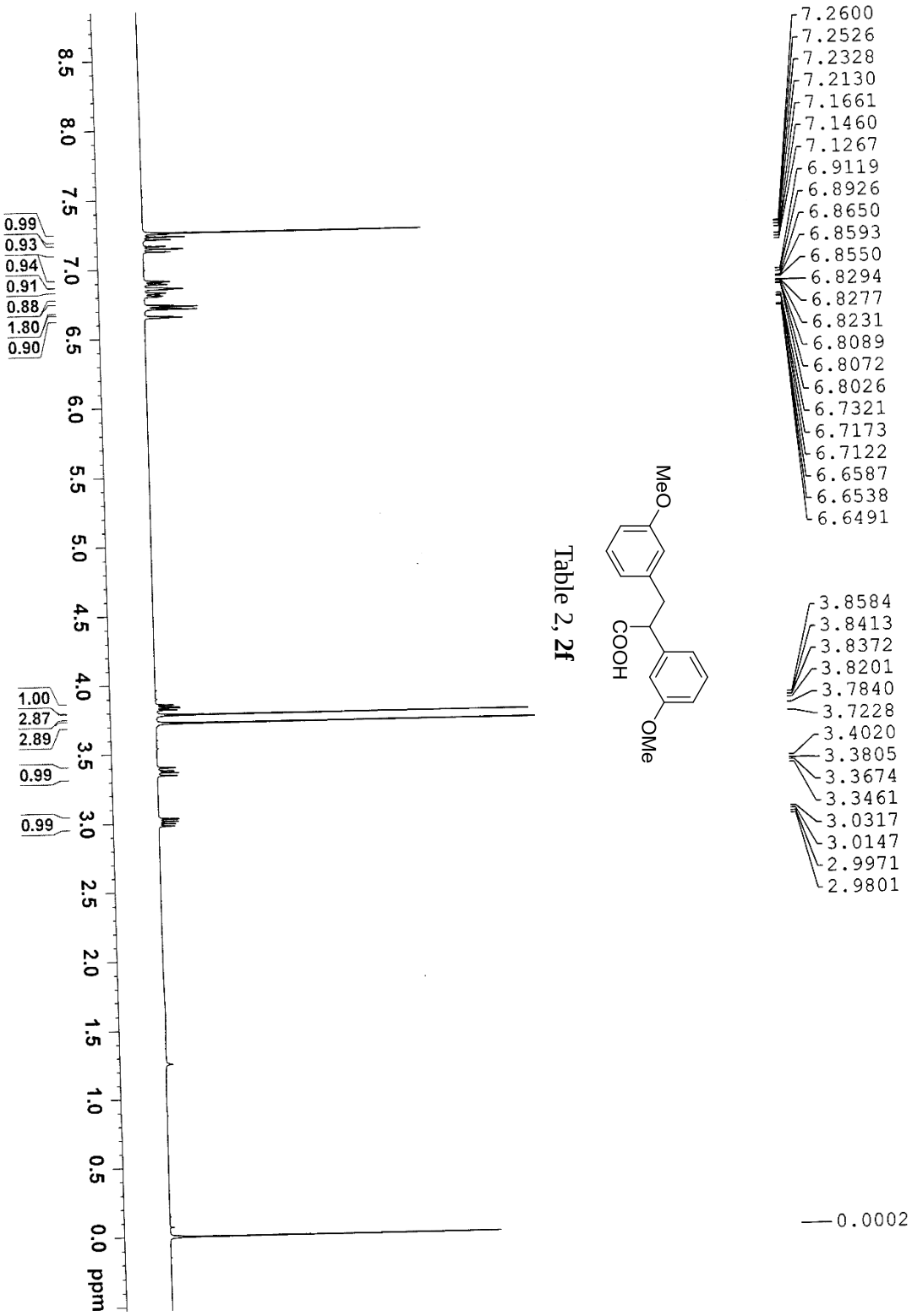

7.2600
7.2526
7.2328
7.2130
7.1661
7.1460
7.1267
6.9119
6.8926
6.8650
6.8593
6.8550
6.8294
6.8277
6.8231
6.8089
6.8072
6.8026
6.7321
6.7173
6.7122
6.6587
6.6538
6.6491
3.8584
3.8413
3.8372
3.8201
3.7840
3.7228
3.4020
3.3805
3.3674
3.3461
3.0317
3.0147
2.9971
2.9801
0.0002
0.99
0.93
0.94
0.91
0.88
1.80
0.90
1.00
2.87
2.89
0.99
0.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
MeO
COOH
OMe

Table 2, **2f**

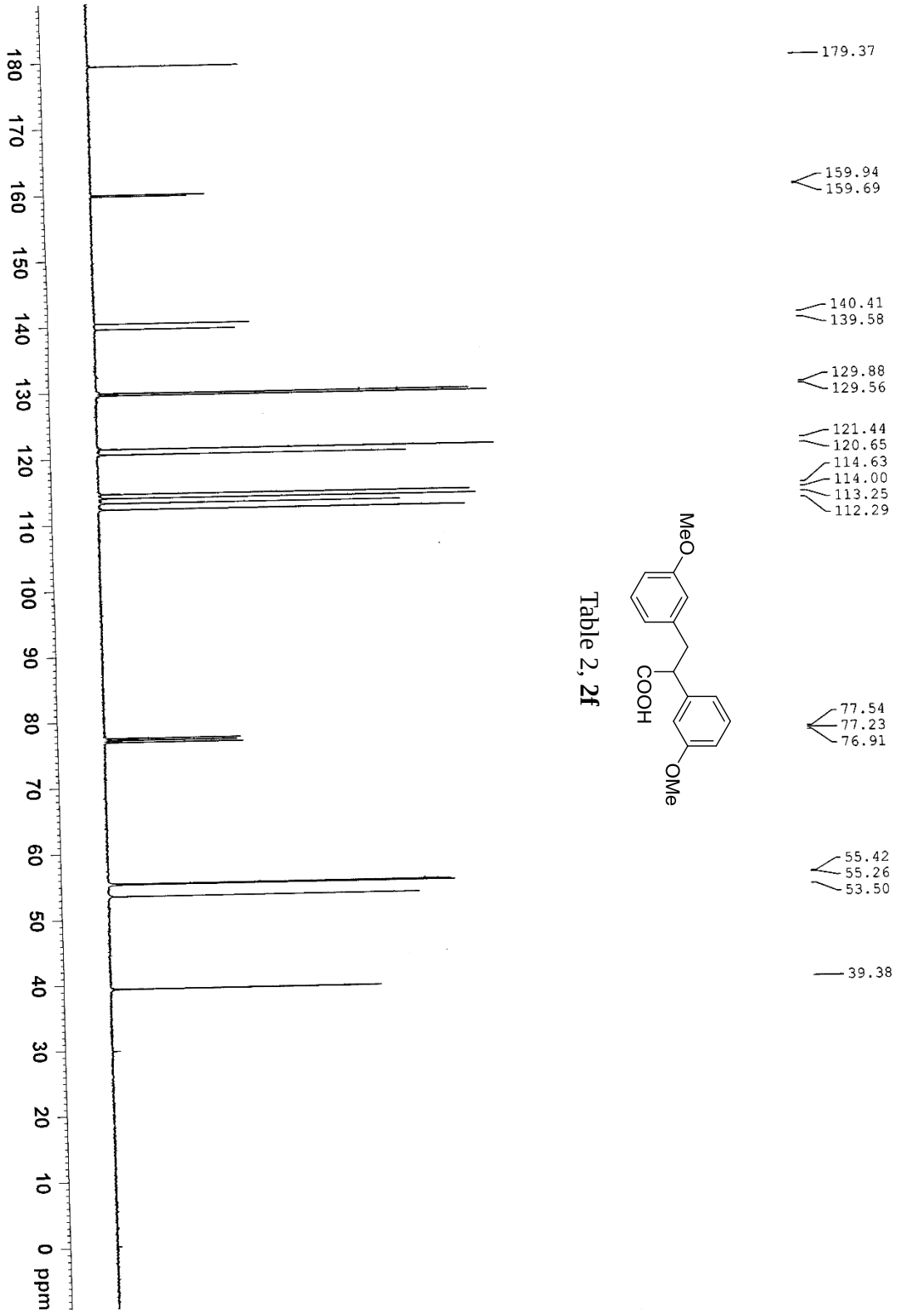
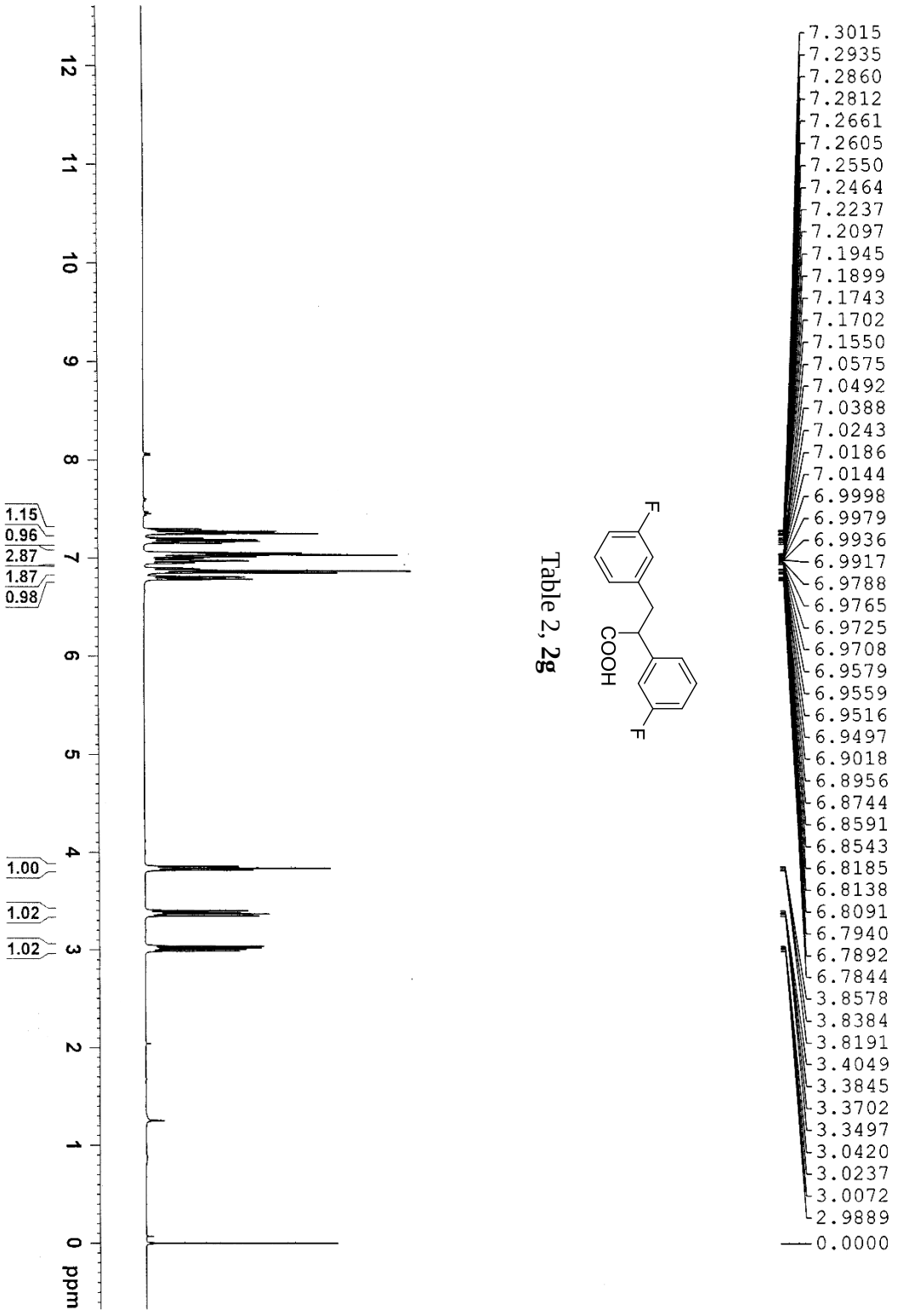

179.37
159.94
159.69
140.41
139.58
129.88
129.56
121.44
120.65
114.63
114.00
113.25
112.29
77.54
77.23
76.91
55.42
55.26
53.50
39.38
MeO
COOH
OMe
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Table 2, **2f**


7.3015
7.2935
7.2860
7.2812
7.2661
7.2605
7.2550
7.2464
7.2237
7.2097
7.1945
7.1899
7.1743
7.1702
7.1550
7.0575
7.0492
7.0388
7.0243
7.0186
7.0144
6.9998
6.9979
6.9936
6.9917
6.9788
6.9765
6.9725
6.9708
6.9579
6.9559
6.9516
6.9497
6.9018
6.8956
6.8744
6.8591
6.8543
6.8185
6.8138
6.8091
6.7940
6.7892
6.7844
3.8578
3.8384
3.8191
3.4049
3.3845
3.3702
3.3497
3.0420
3.0237
3.0072
2.9889
0.0000
F
COOH
F
Table 2, **2g**
1.15
0.96
2.87
1.87
0.98
1.00
1.02
1.02
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm

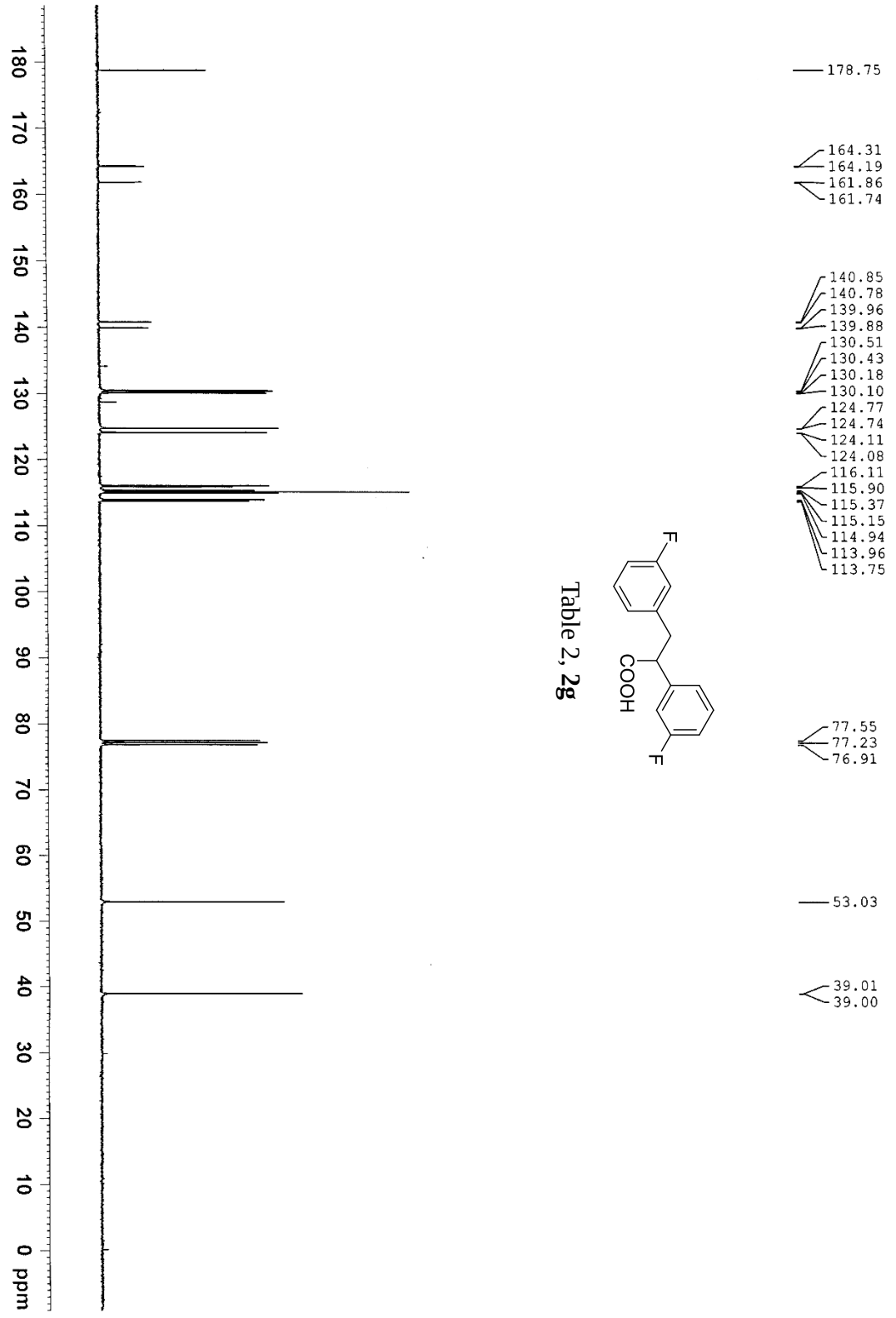

178.75
164.31
164.19
161.86
161.74
140.85
140.78
139.96
139.88
130.51
130.43
130.18
130.10
124.77
124.74
124.11
124.08
116.11
115.90
115.37
115.15
114.94
113.96
113.75
77.55
77.23
76.91
53.03
39.01
39.00
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm
F
COOH
F

Table 2, **2g**

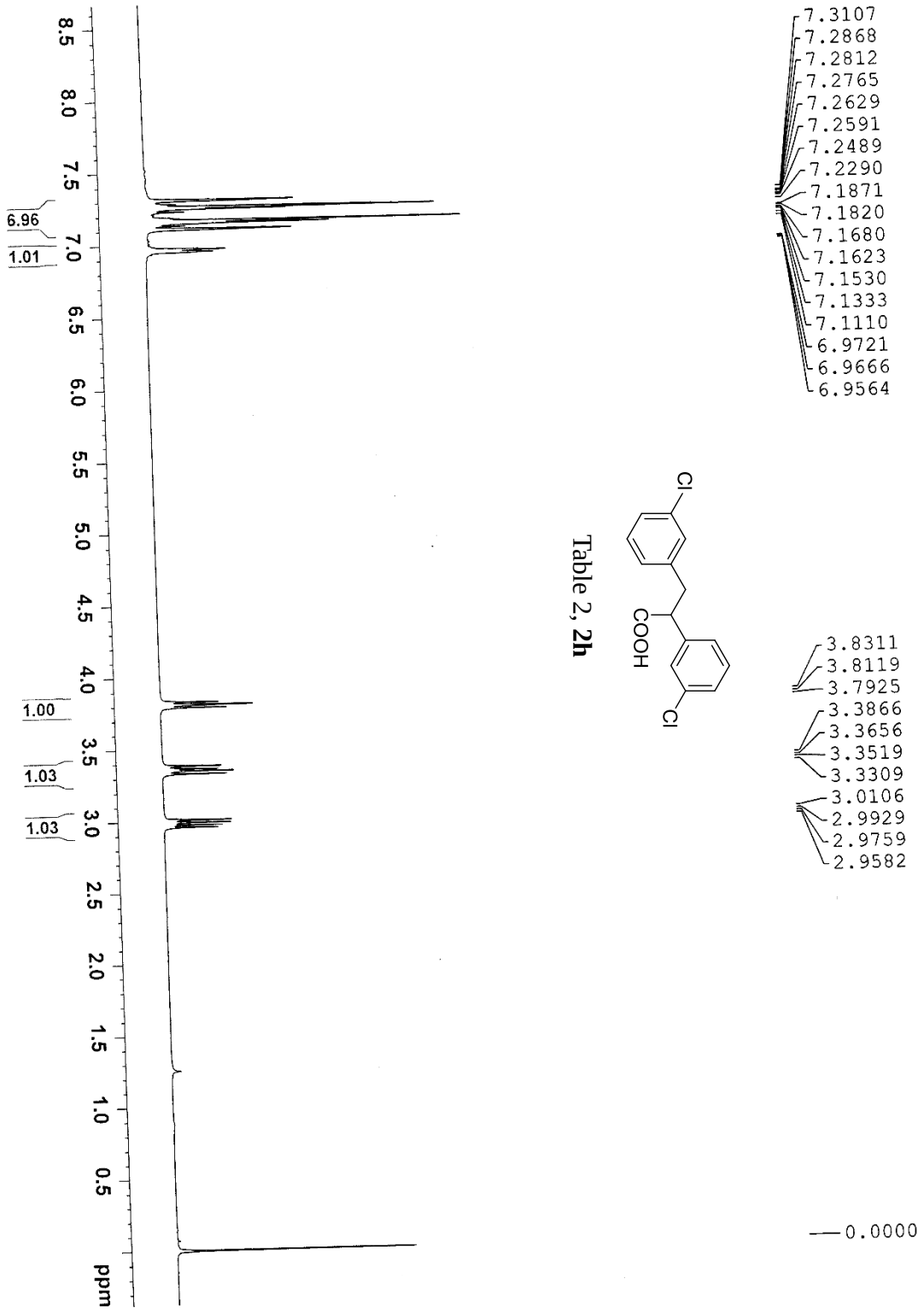

7.3107
7.2868
7.2812
7.2765
7.2629
7.2591
7.2489
7.2290
7.1871
7.1820
7.1680
7.1623
7.1530
7.1333
7.1110
6.9721
6.9666
6.9564
Cl
COOH
Cl
3.8311
3.8119
3.7925
3.3866
3.3656
3.3519
3.3309
3.0106
2.9929
2.9759
2.9582
0.0000
6.96
1.01
1.00
1.03
1.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

Table 2, **2h**

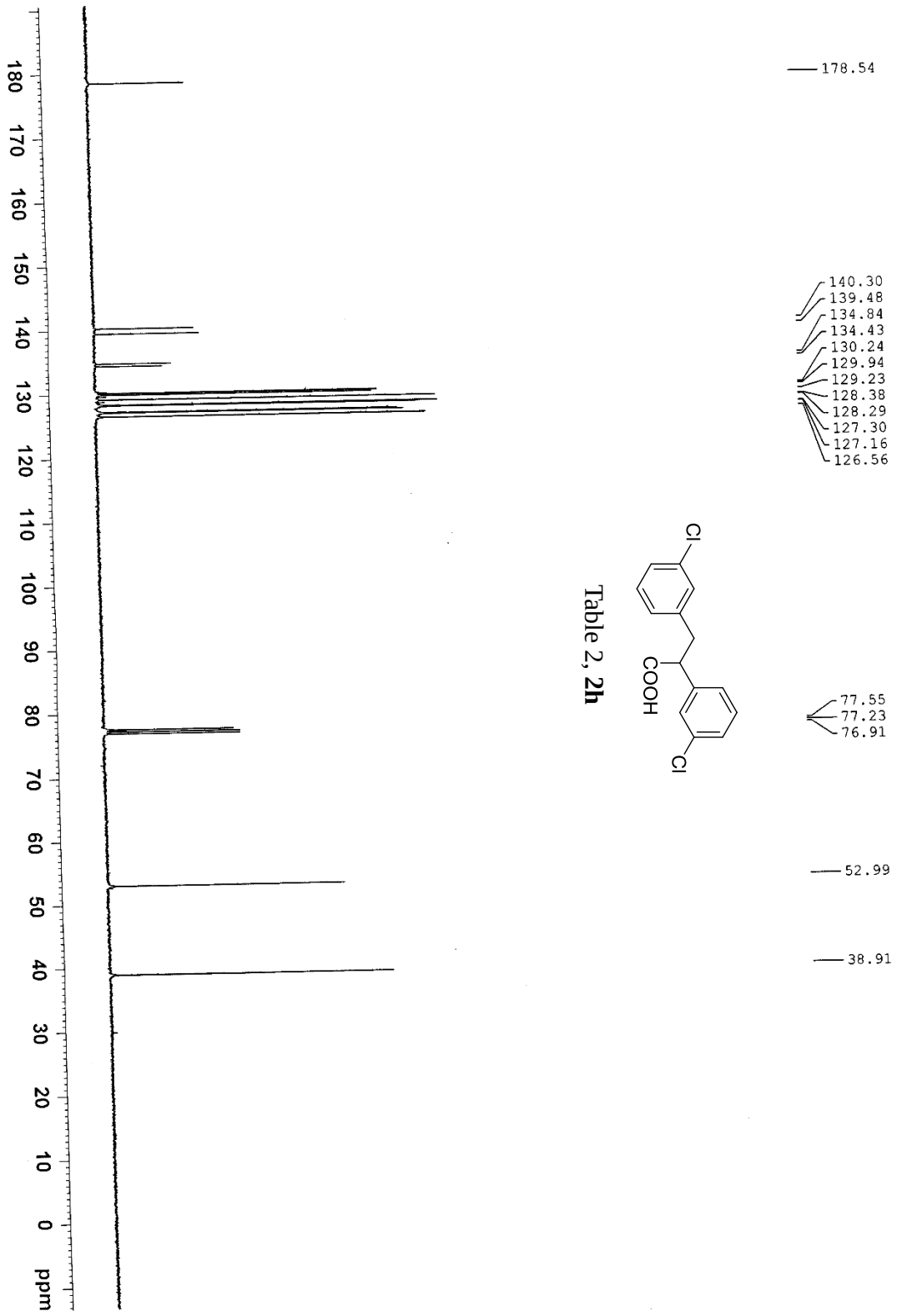

178.54
140.30
139.48
134.84
134.43
130.24
129.94
129.23
128.38
128.29
127.30
127.16
126.56
77.55
77.23
76.91
52.99
38.91
Cl
COOH
Cl
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Table 2, **2h**

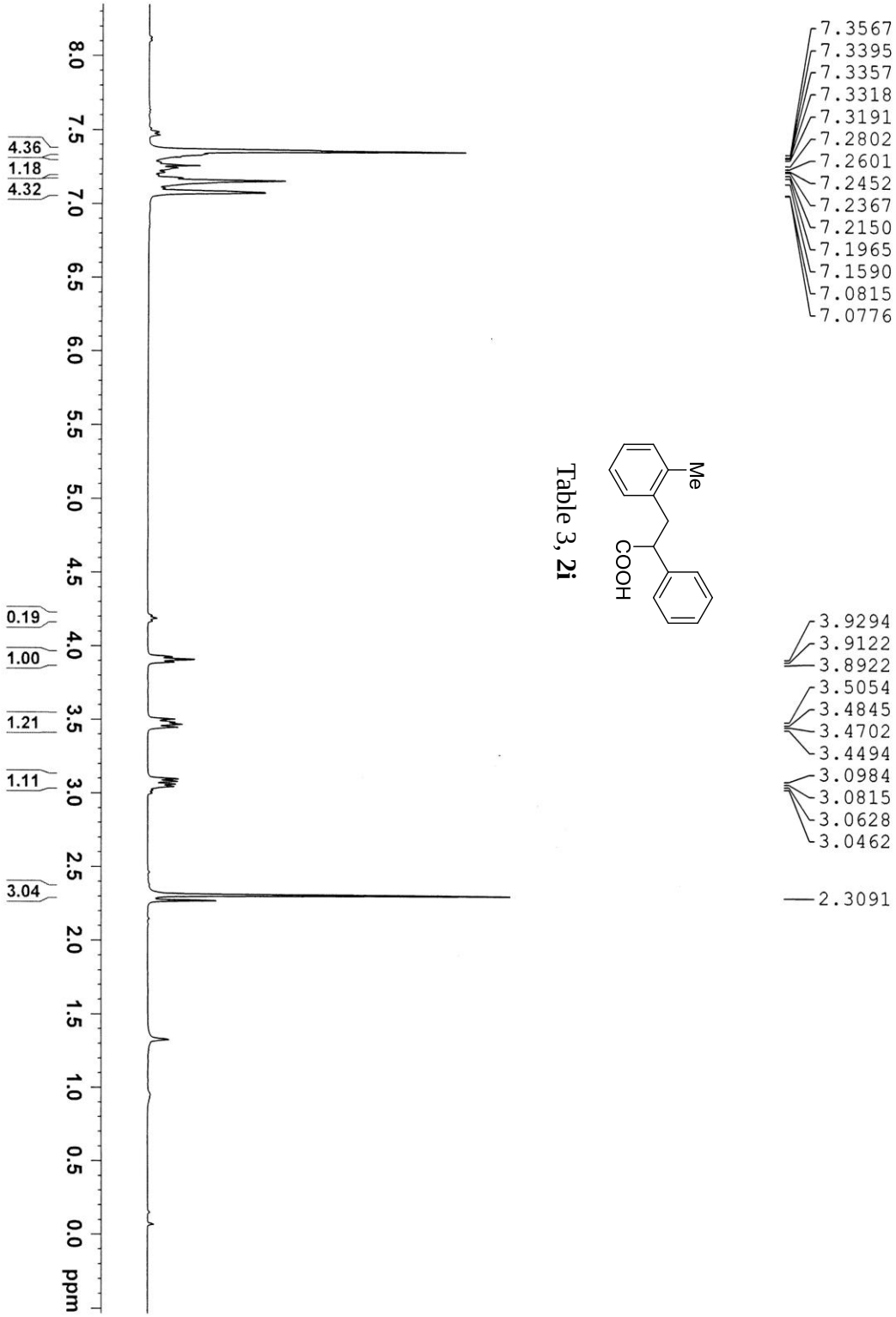

7.3567
7.3395
7.3357
7.3318
7.3191
7.2802
7.2601
7.2452
7.2367
7.2150
7.1965
7.1590
7.0815
7.0776
Me
COOH
3.9294
3.9122
3.8922
3.5054
3.4845
3.4702
3.4494
3.0984
3.0815
3.0628
3.0462
2.3091
4.36
1.18
4.32
0.19
1.00
1.21
1.11
3.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

Table 3, **2i**

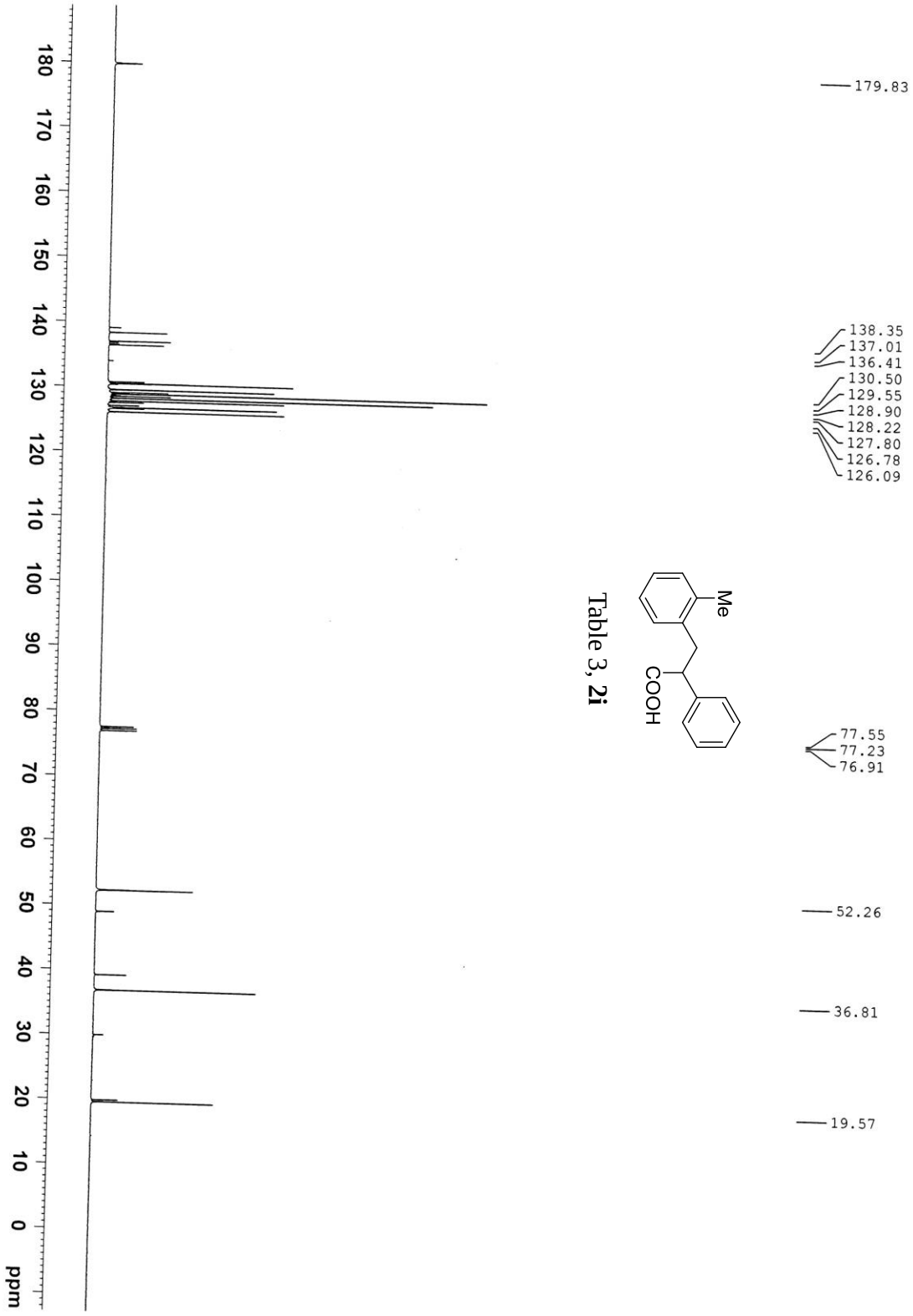
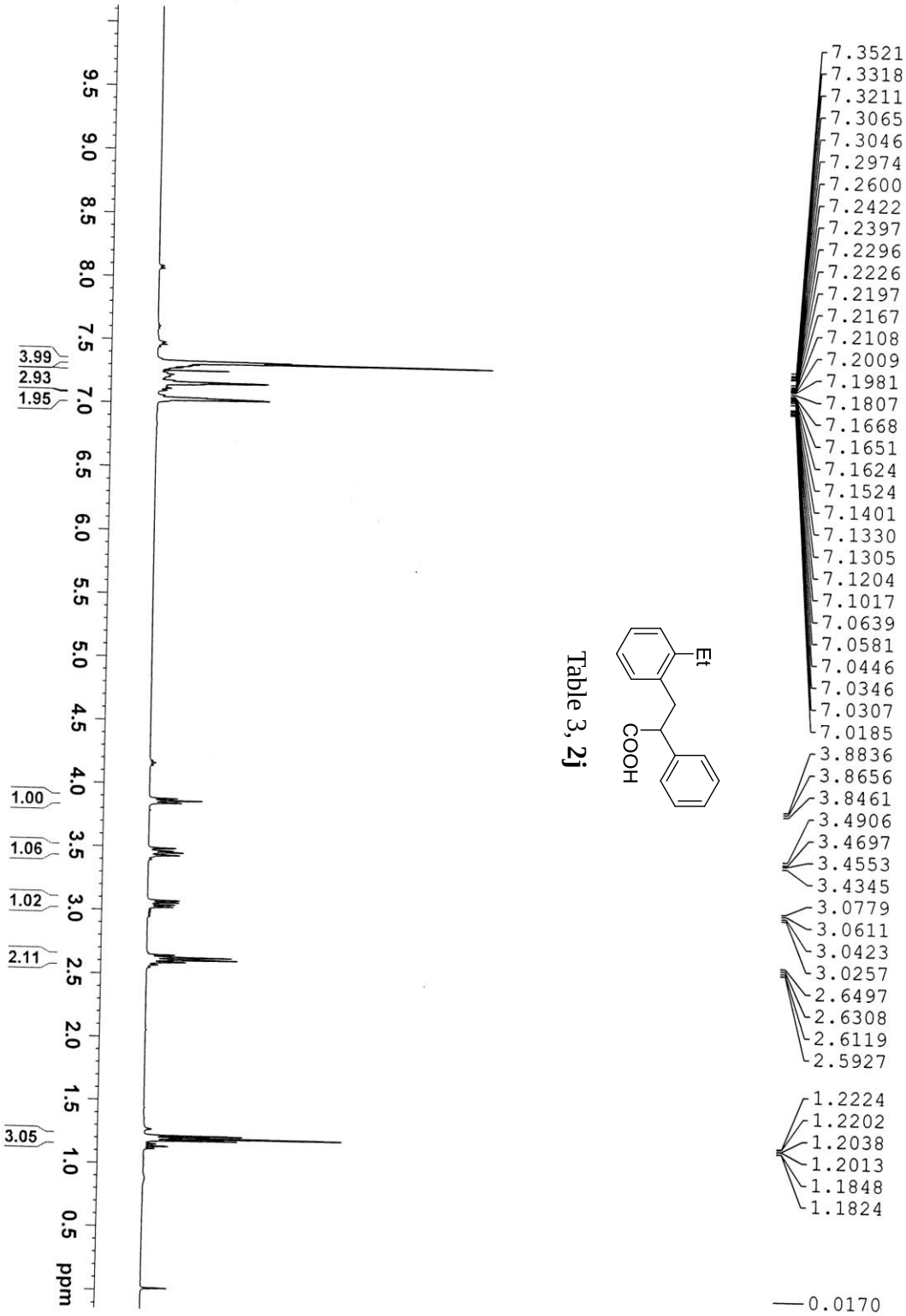

179.83
138.35
137.01
136.41
130.50
129.55
128.90
128.22
127.80
126.78
126.09
77.55
77.23
76.91
52.26
36.81
19.57
Me
COOH
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Table 3, **2i**


7.3521
7.3318
7.3211
7.3065
7.3046
7.2974
7.2600
7.2422
7.2397
7.2296
7.2226
7.2197
7.2167
7.2108
7.2009
7.1981
7.1807
7.1668
7.1651
7.1624
7.1524
7.1401
7.1330
7.1305
7.1204
7.1017
7.0639
7.0581
7.0446
7.0346
7.0307
7.0185
3.8836
3.8656
3.8461
3.4906
3.4697
3.4553
3.4345
3.0779
3.0611
3.0423
3.0257
2.6497
2.6308
2.6119
2.5927
1.2224
1.2202
1.2038
1.2013
1.1848
1.1824
0.0170
Et
COOH
Table 3, 2j
3.99
2.93
1.95
1.00
1.06
1.02
2.11
3.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

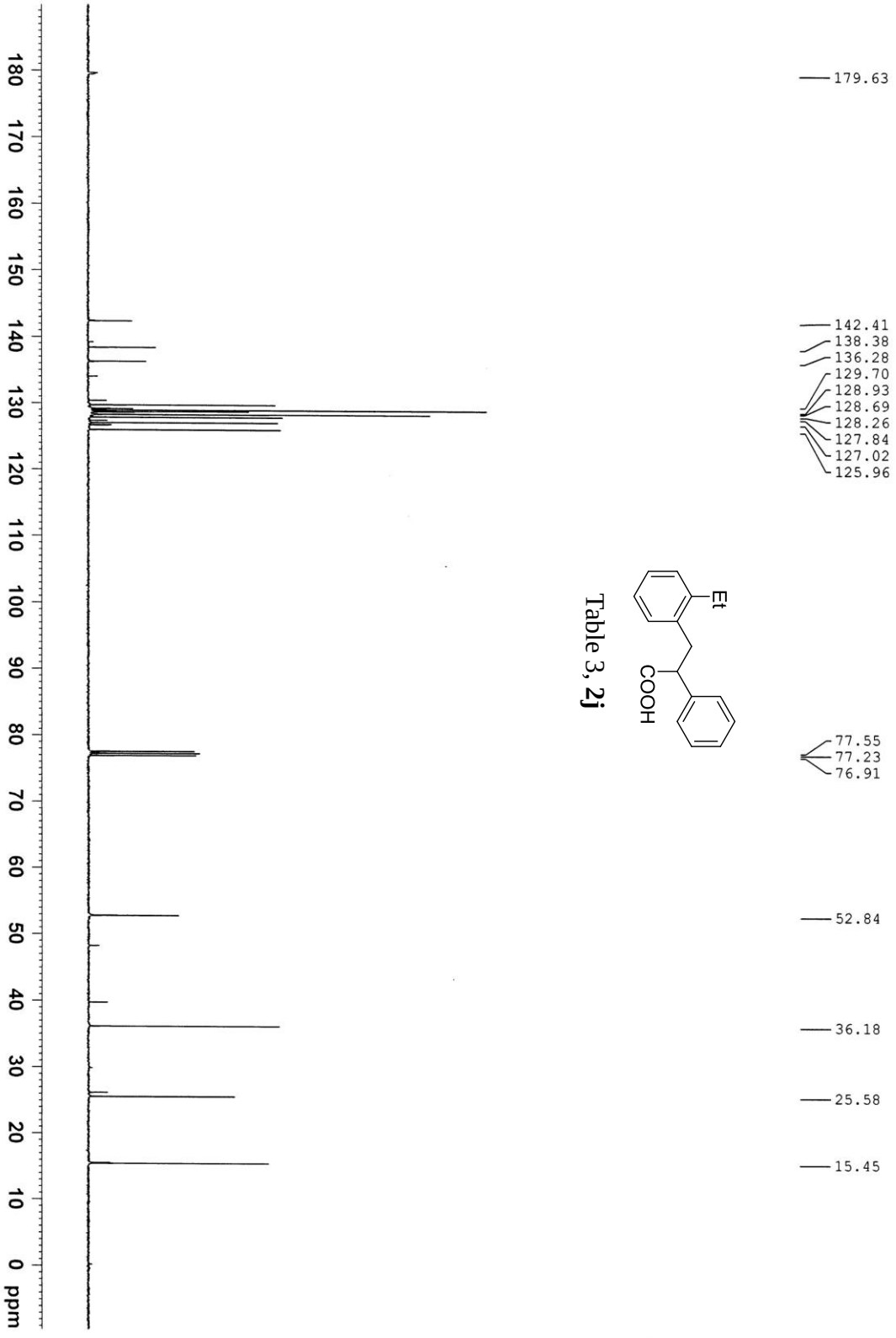

179.63
142.41
138.38
136.28
129.70
128.93
128.69
128.26
127.84
127.02
125.96
77.55
77.23
76.91
52.84
36.18
25.58
15.45
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm
Et
COOH

Table 3, **2j**

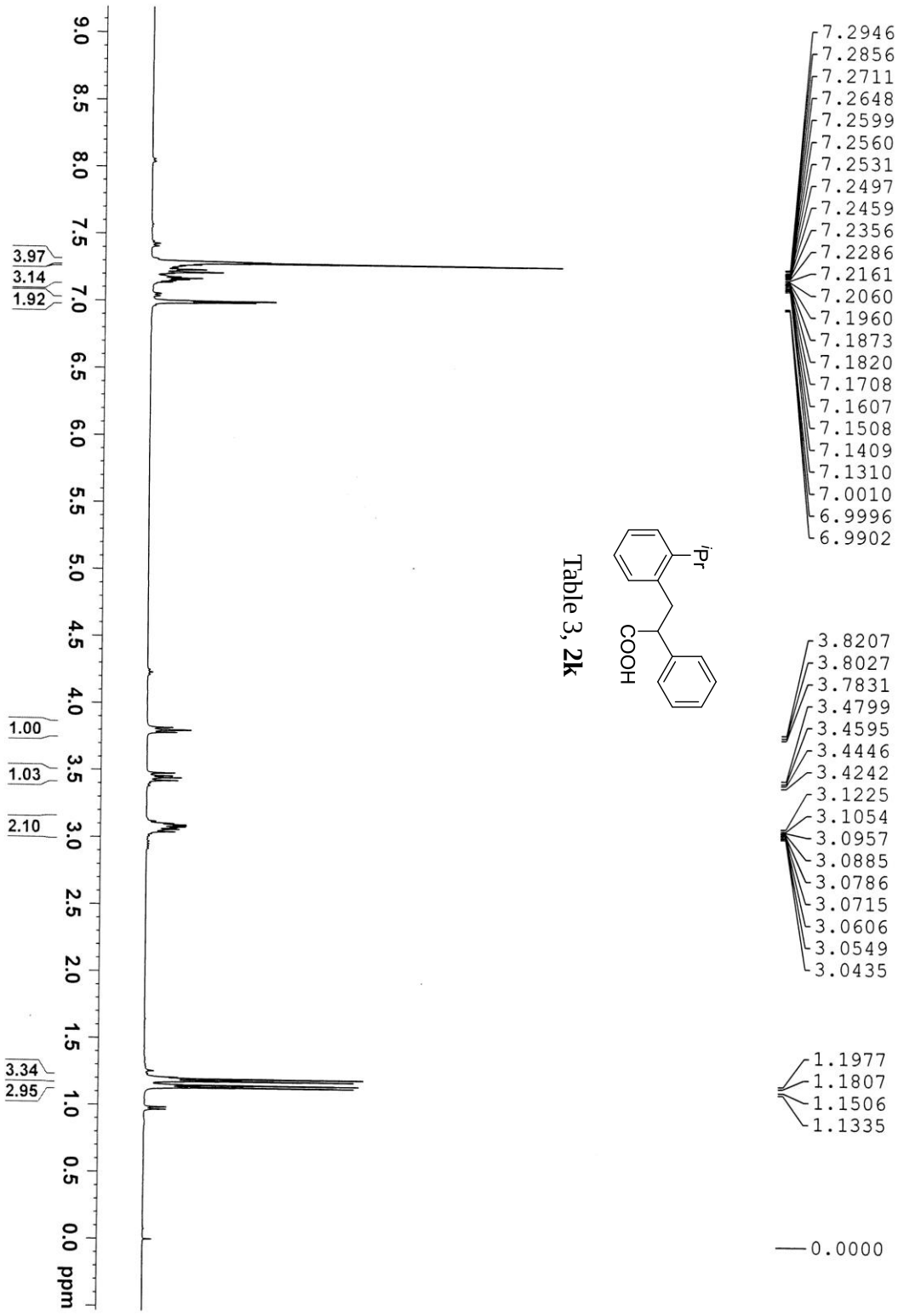
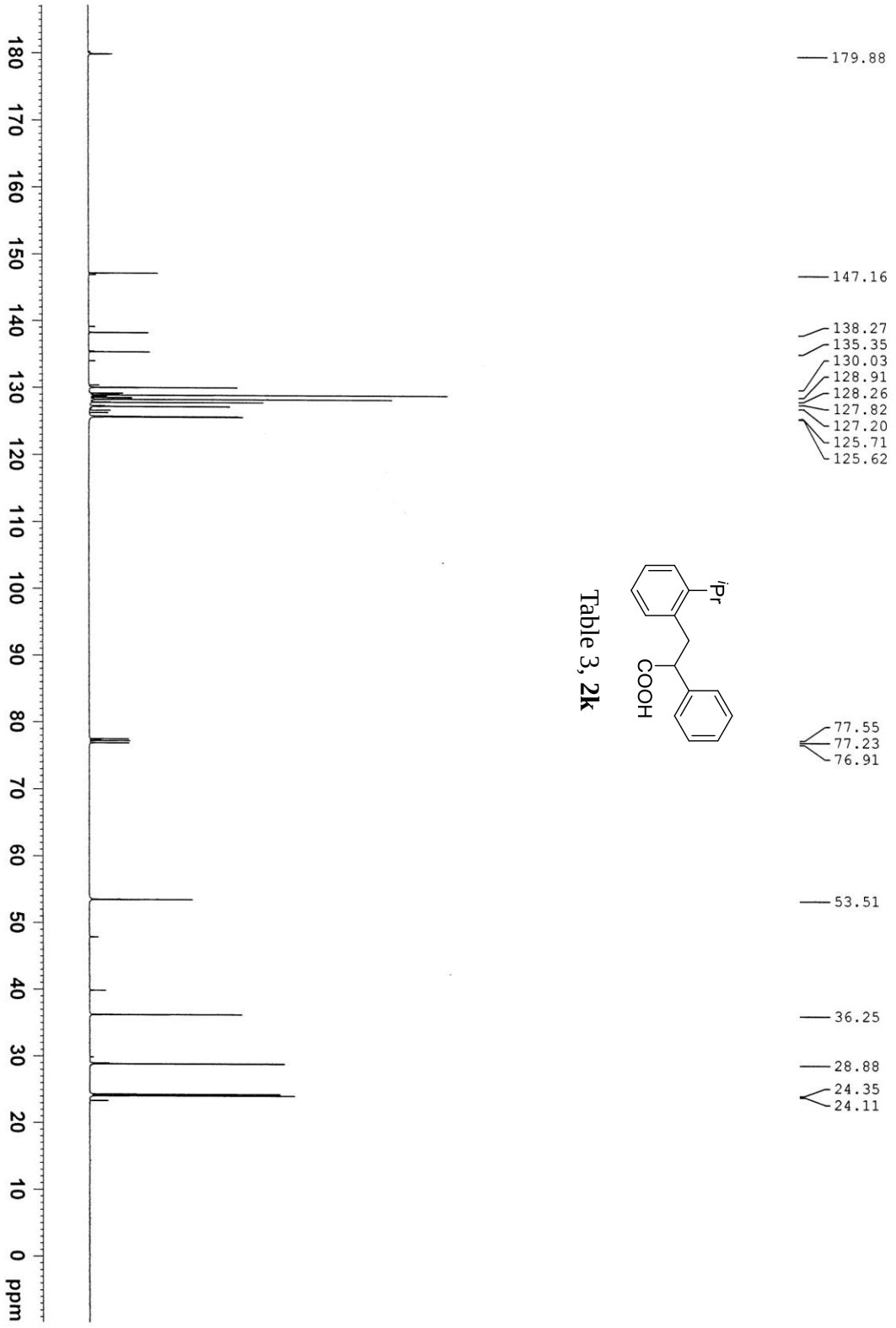

7.2946
7.2856
7.2711
7.2648
7.2599
7.2560
7.2531
7.2497
7.2459
7.2356
7.2286
7.2161
7.2060
7.1960
7.1873
7.1820
7.1708
7.1607
7.1508
7.1409
7.1310
7.0010
6.9996
6.9902
3.8207
3.8027
3.7831
3.4799
3.4595
3.4446
3.4242
3.1225
3.1054
3.0957
3.0885
3.0786
3.0715
3.0606
3.0549
3.0435
1.1977
1.1807
1.1506
1.1335
0.0000
3.97
3.14
1.92
1.00
1.03
2.10
3.34
2.95
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
iPr
COOH

Table 3, **2k**


179.88
147.16
138.27
135.35
130.03
128.91
128.26
127.82
127.20
125.71
125.62
77.55
77.23
76.91
53.51
36.25
28.88
24.35
24.11
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
Table 3, 2k
iPr
COOH

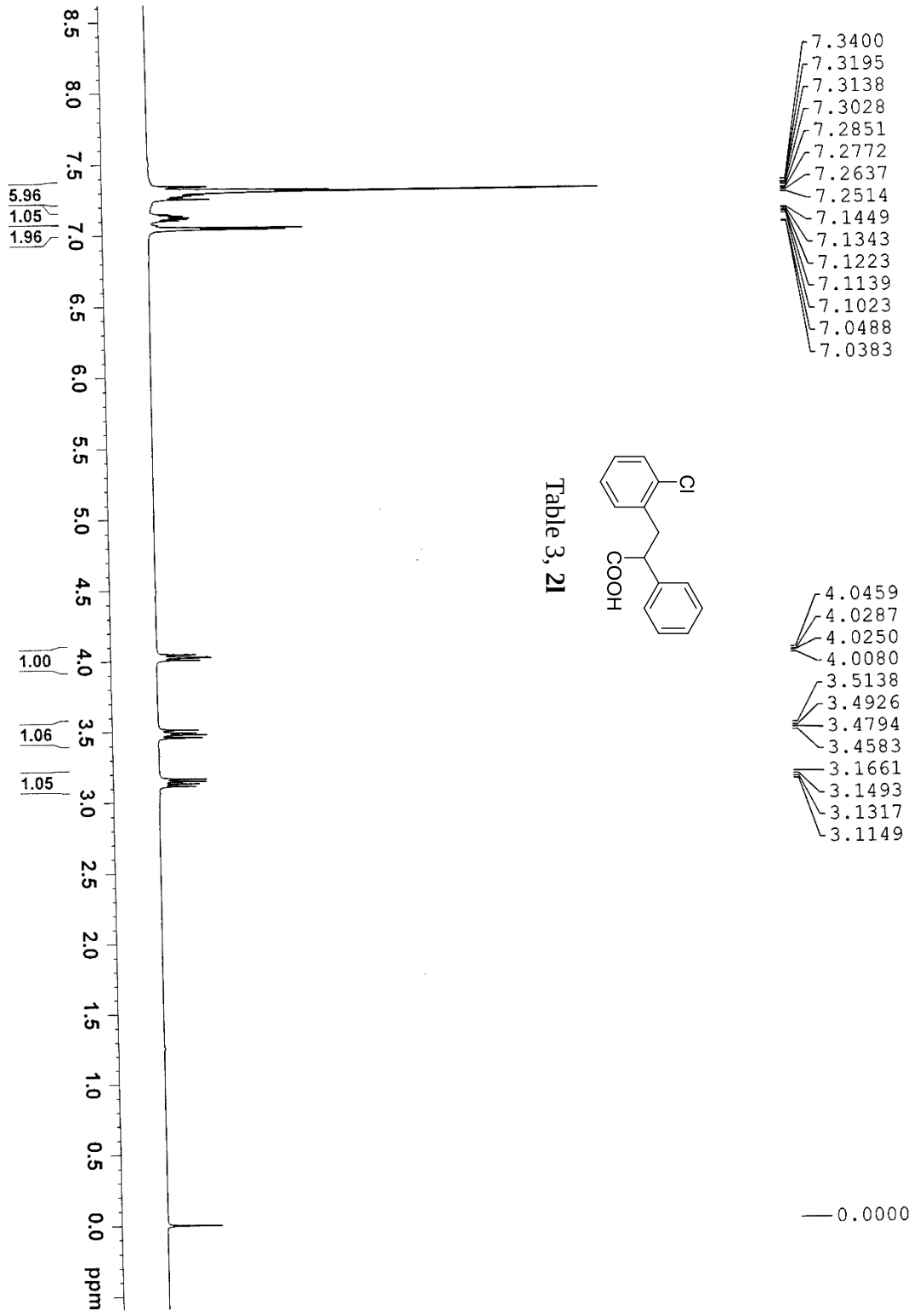

8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
5.96
1.05
1.96
1.00
1.06
1.05
7.3400
7.3195
7.3138
7.3028
7.2851
7.2772
7.2637
7.2514
7.1449
7.1343
7.1223
7.1139
7.1023
7.0488
7.0383
4.0459
4.0287
4.0250
4.0080
3.5138
3.4926
3.4794
3.4583
3.1661
3.1493
3.1317
3.1149
0.0000
Cl
COOH

Table 3, **2l**

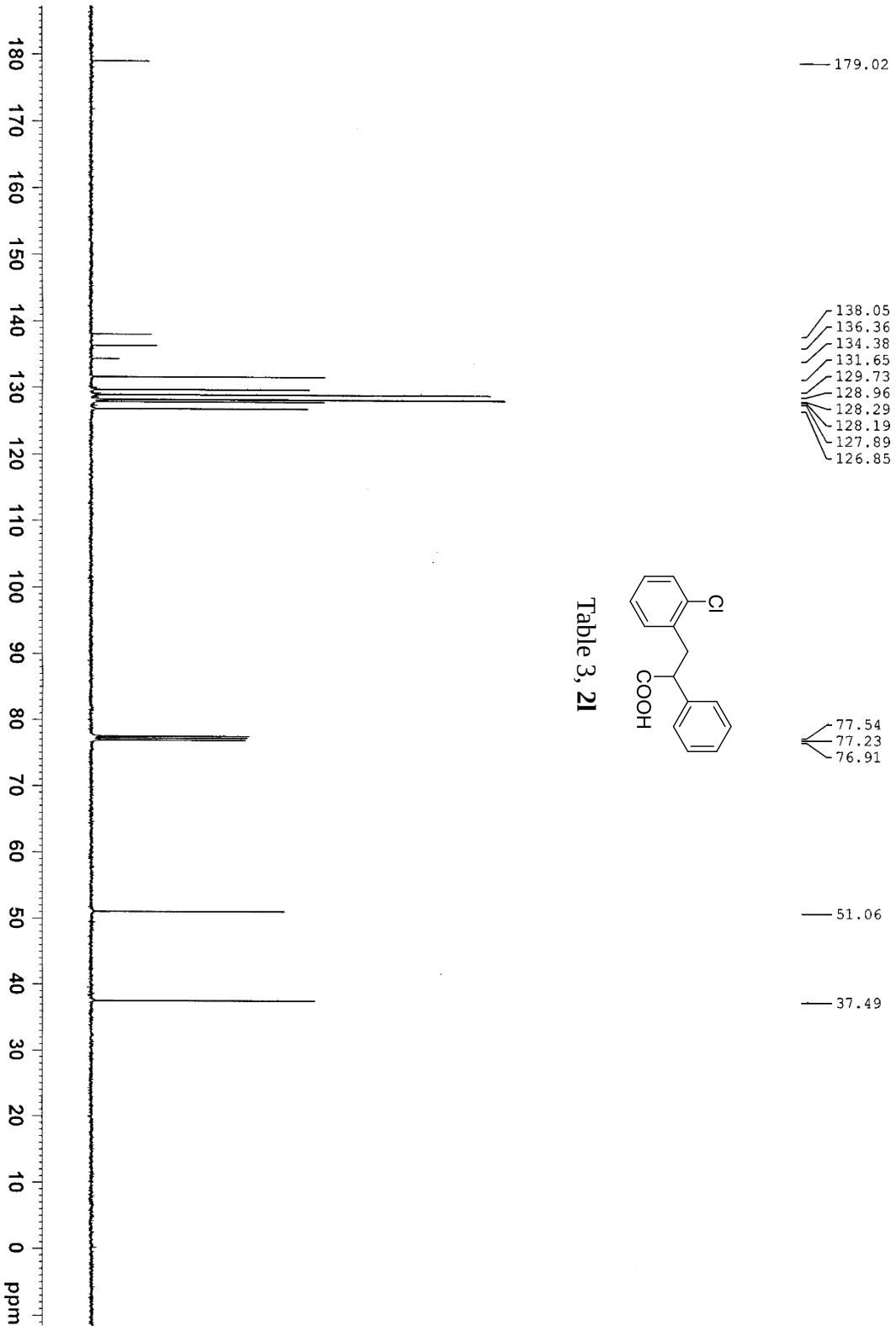

179.02
138.05
136.36
134.38
131.65
129.73
128.96
128.29
128.19
127.89
126.85
77.54
77.23
76.91
51.06
37.49
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
Cl
COOH

Table 3, **2l**

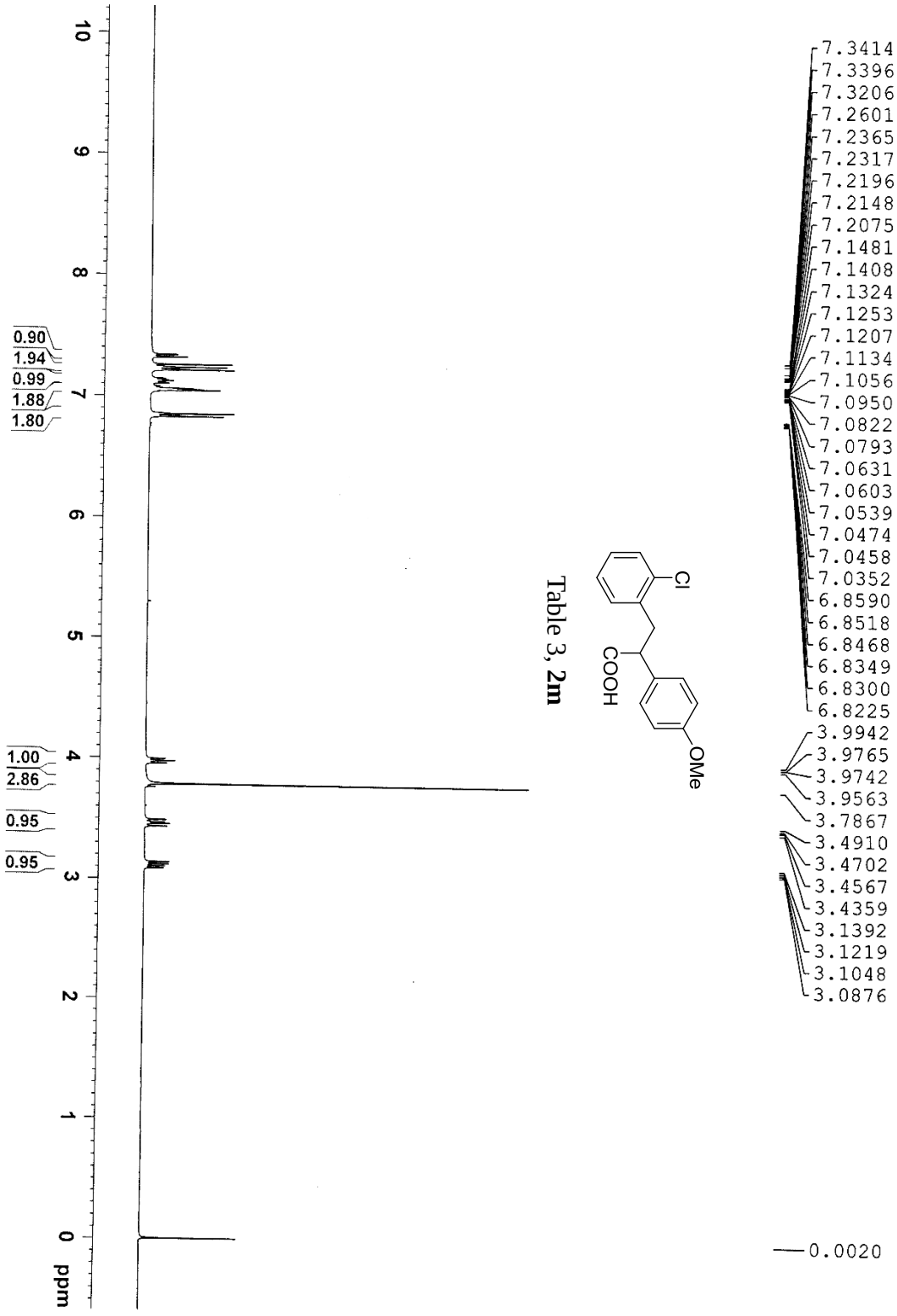

7.3414
7.3396
7.3206
7.2601
7.2365
7.2317
7.2196
7.2148
7.2075
7.1481
7.1408
7.1324
7.1253
7.1207
7.1134
7.1056
7.0950
7.0822
7.0793
7.0631
7.0603
7.0539
7.0474
7.0458
7.0352
6.8590
6.8518
6.8468
6.8349
6.8300
6.8225
3.9942
3.9765
3.9742
3.9563
3.7867
3.4910
3.4702
3.4567
3.4359
3.1392
3.1219
3.1048
3.0876
0.0020
0.90
1.94
0.99
1.88
1.80
1.00
2.86
0.95
0.95
10
9
8
7
6
5
4
3
2
1
0
ppm
Cl
COOH
OMe

Table 3, **2m**

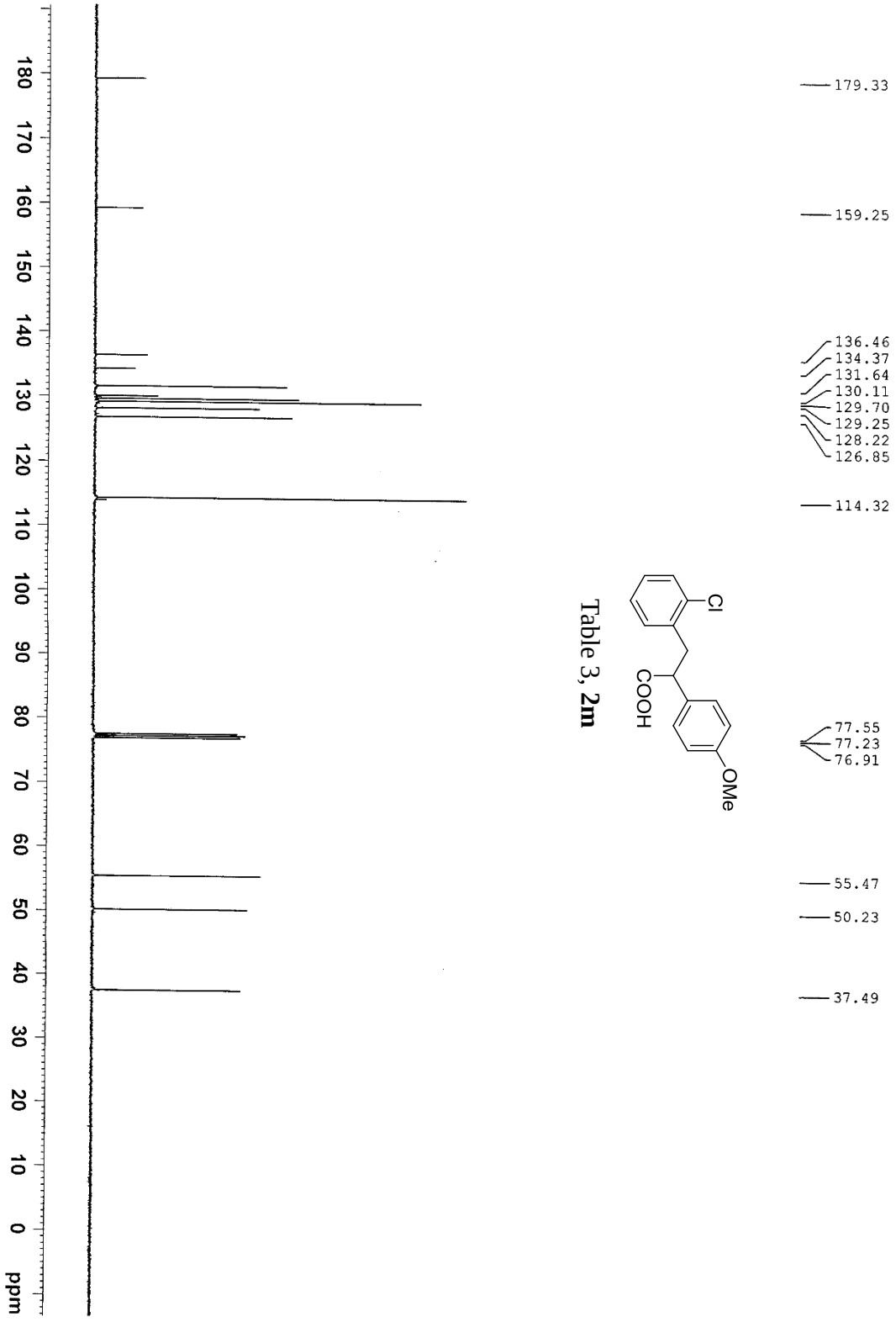
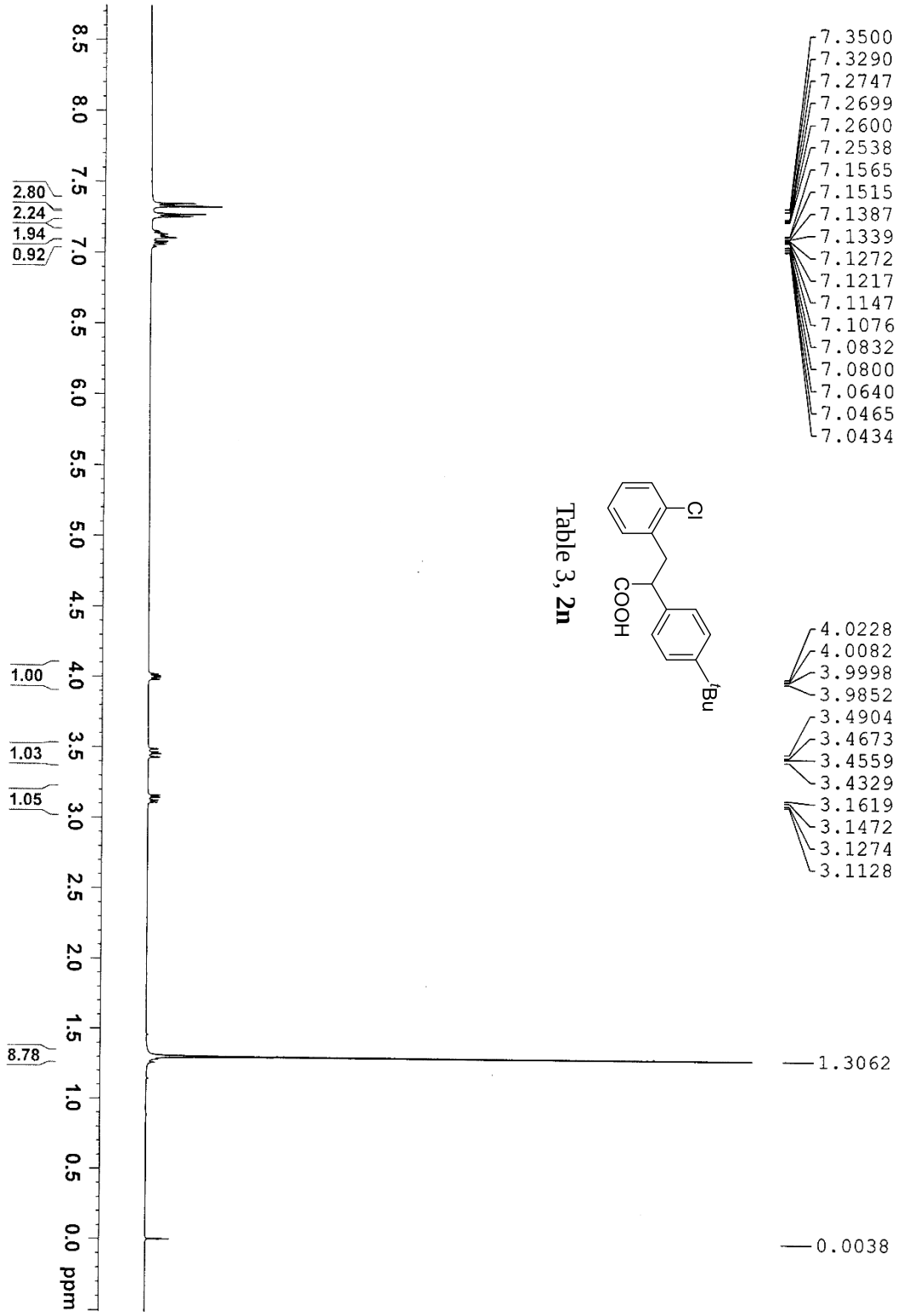
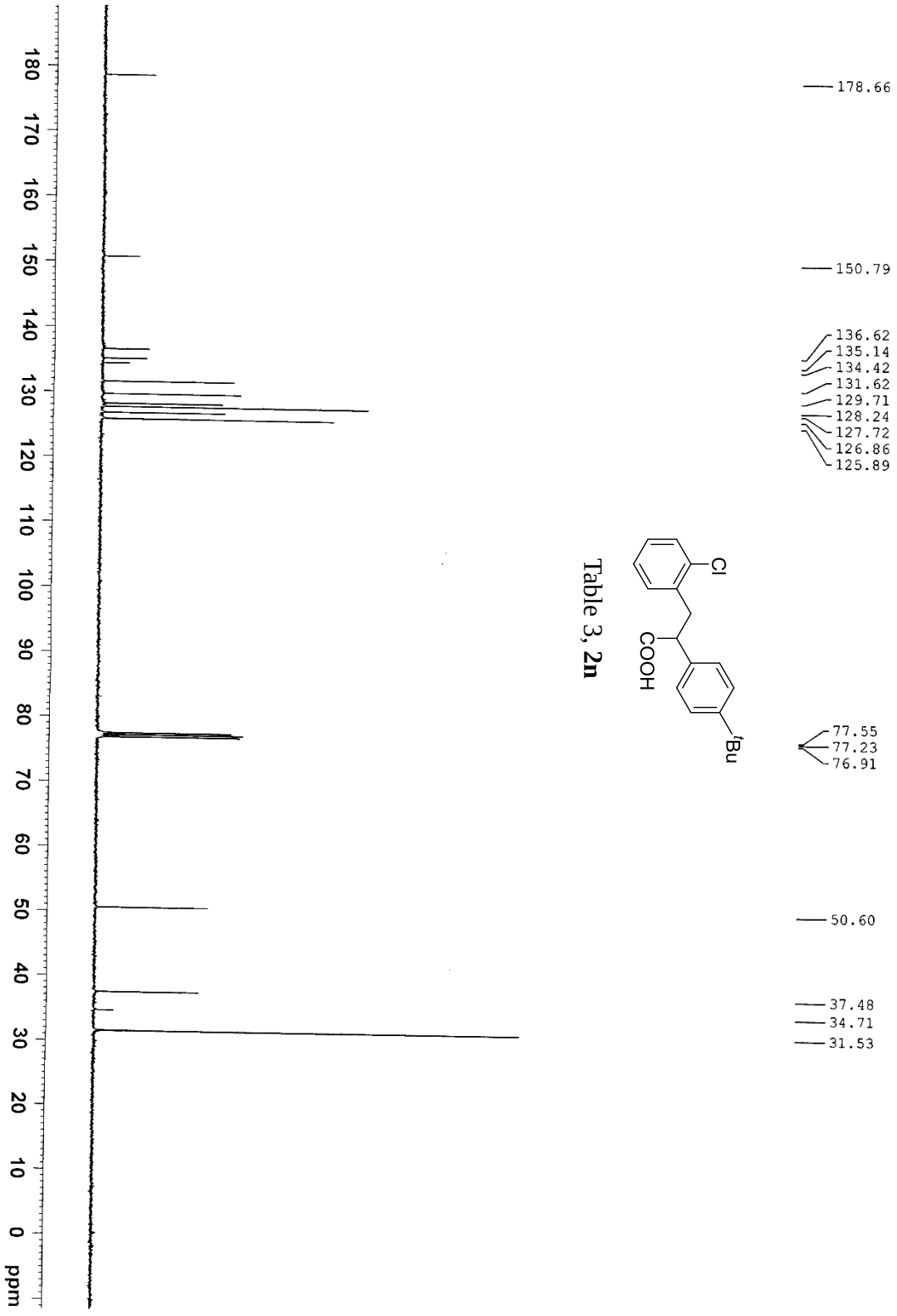

179.33
159.25
136.46
134.37
131.64
130.11
129.70
129.25
128.22
126.85
114.32
77.55
77.23
76.91
55.47
50.23
37.49
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Cl
COOH
OMe

Table 3, **2m**


7.3500
7.3290
7.2747
7.2699
7.2600
7.2538
7.1565
7.1515
7.1387
7.1339
7.1272
7.1217
7.1147
7.1076
7.0832
7.0800
7.0640
7.0465
7.0434
4.0228
4.0082
3.9998
3.9852
3.4904
3.4673
3.4559
3.4329
3.1619
3.1472
3.1274
3.1128
1.3062
0.0038
2.80
2.24
1.94
0.92
1.00
1.03
1.05
8.78
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Cl
COOH
tBu
Table 3, 2n


178.66
150.79
136.62
135.14
134.42
131.62
129.71
128.24
127.72
126.86
125.89
77.55
77.23
76.91
50.60
37.48
34.71
31.53
Cl
COOH
tBu
Table 3, 2n
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

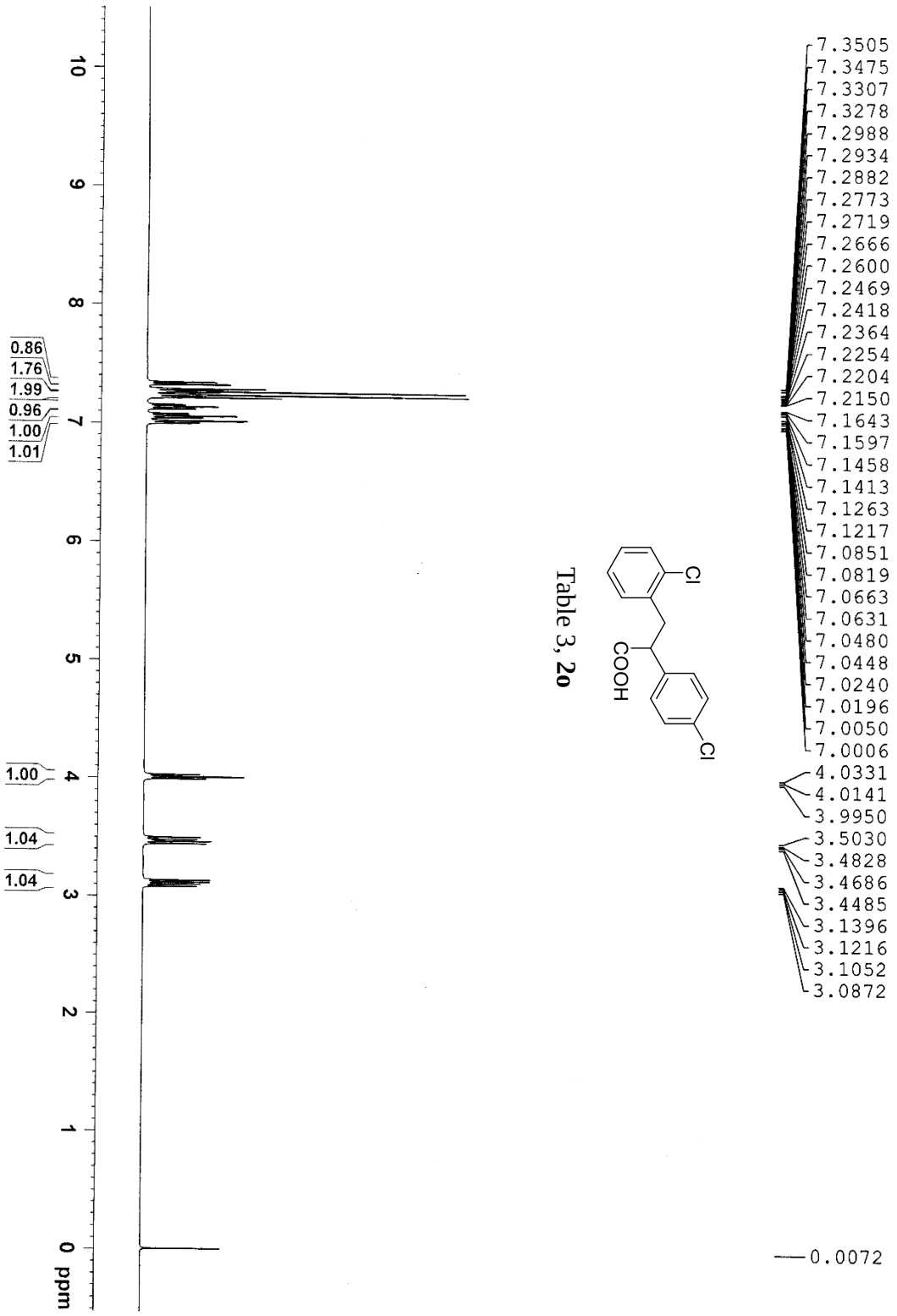

7.3505
7.3475
7.3307
7.3278
7.2988
7.2934
7.2882
7.2773
7.2719
7.2666
7.2600
7.2469
7.2418
7.2364
7.2254
7.2204
7.2150
7.1643
7.1597
7.1458
7.1413
7.1263
7.1217
7.0851
7.0819
7.0663
7.0631
7.0480
7.0448
7.0240
7.0196
7.0050
7.0006
4.0331
4.0141
3.9950
3.5030
3.4828
3.4686
3.4485
3.1396
3.1216
3.1052
3.0872
0.0072
0.86
1.76
1.99
0.96
1.00
1.01
1.00
1.04
1.04
10
9
8
7
6
5
4
3
2
1
0
ppm
Cl
COOH
Cl

Table 3, **2o**

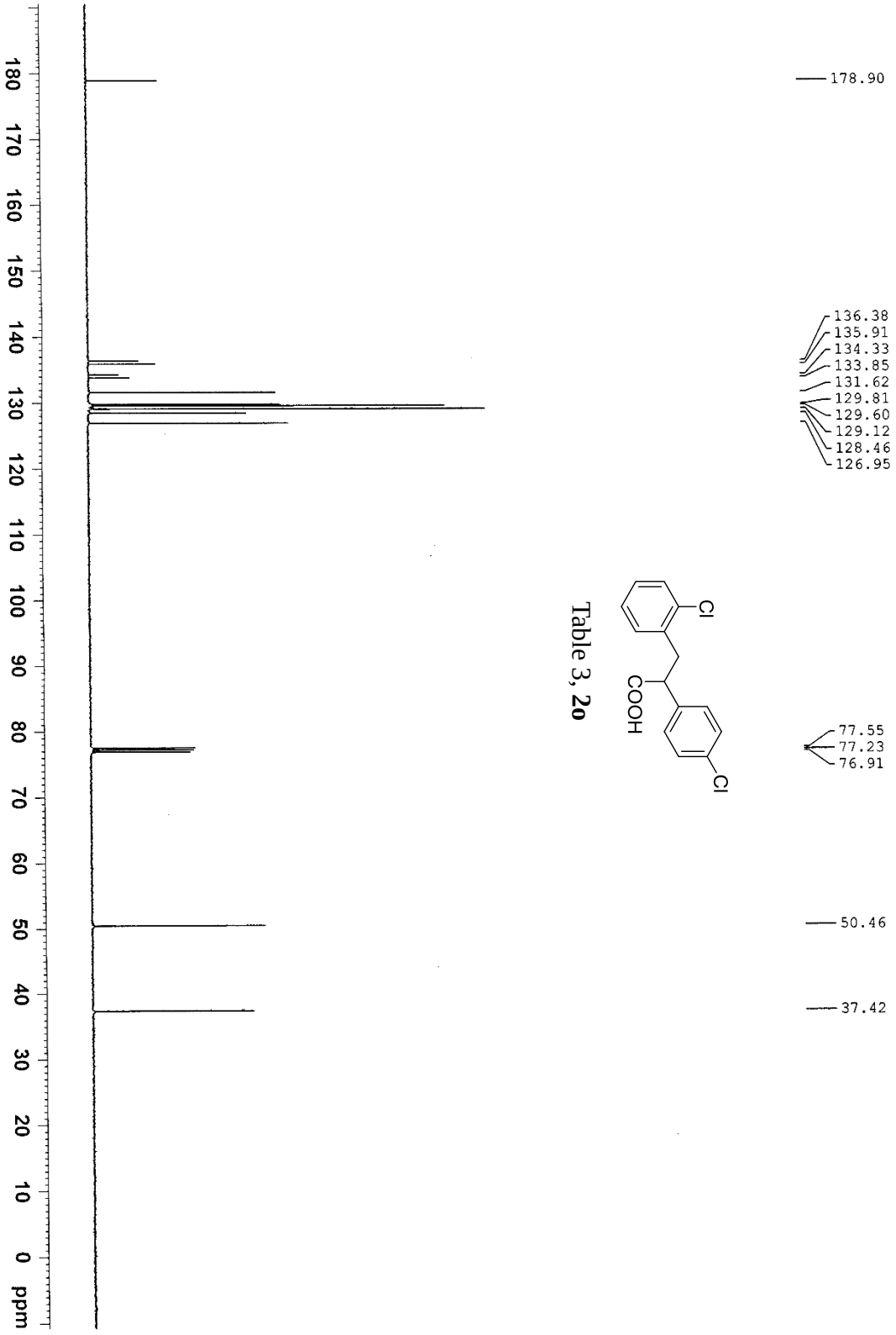
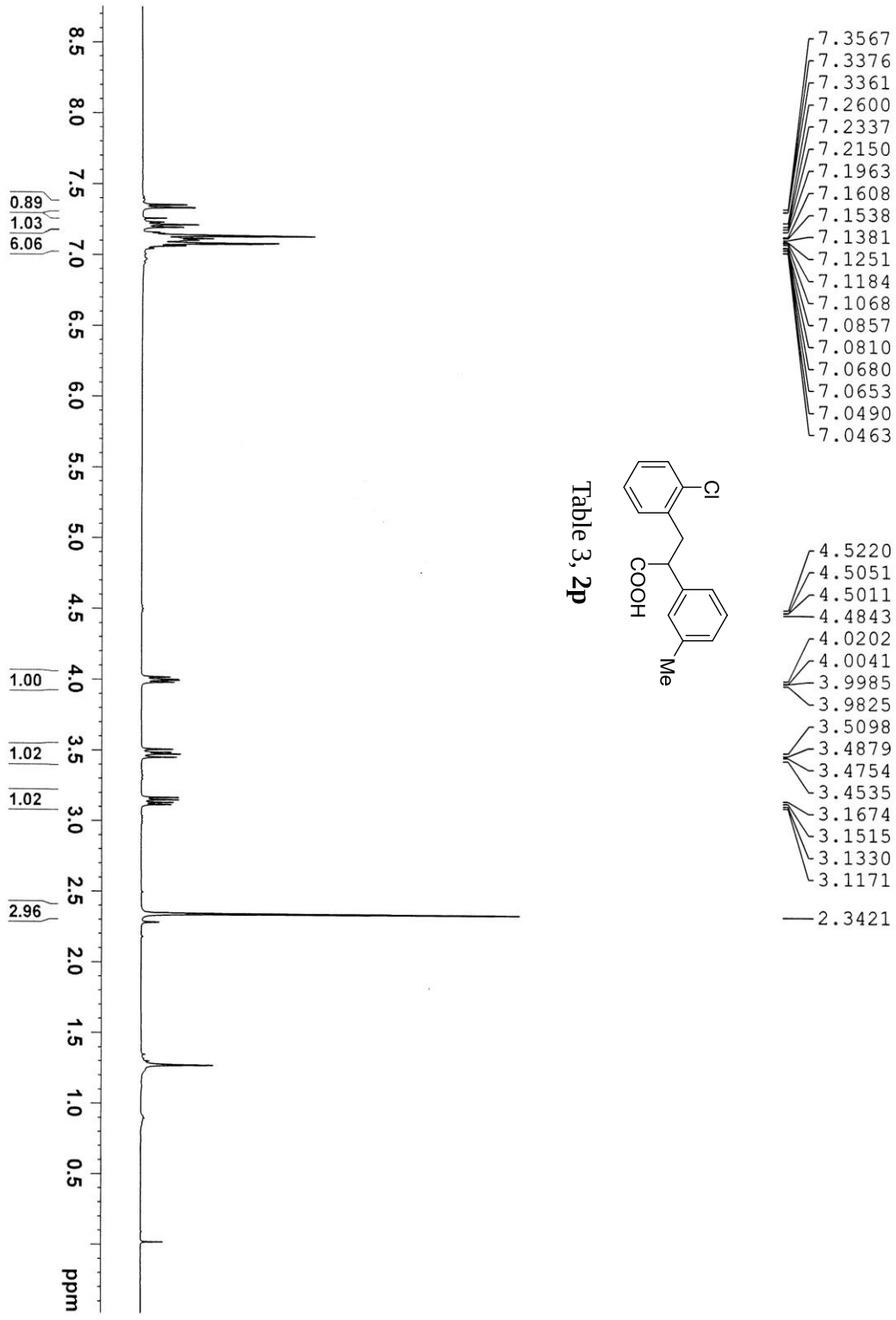

178.90
136.38
135.91
134.33
133.85
131.62
129.81
129.60
129.12
128.46
126.95
77.55
77.23
76.91
50.46
37.42
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Cl
COOH
Cl

Table 3, **2o**


7.3567
7.3376
7.3361
7.2600
7.2337
7.2150
7.1963
7.1608
7.1538
7.1381
7.1251
7.1184
7.1068
7.0857
7.0810
7.0680
7.0653
7.0490
7.0463
4.5220
4.5051
4.5011
4.4843
4.0202
4.0041
3.9985
3.9825
3.5098
3.4879
3.4754
3.4535
3.1674
3.1515
3.1330
3.1171
2.3421
Cl
COOH
Me
Table 3, 2p
0.89
1.03
6.06
1.00
1.02
1.02
2.96
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

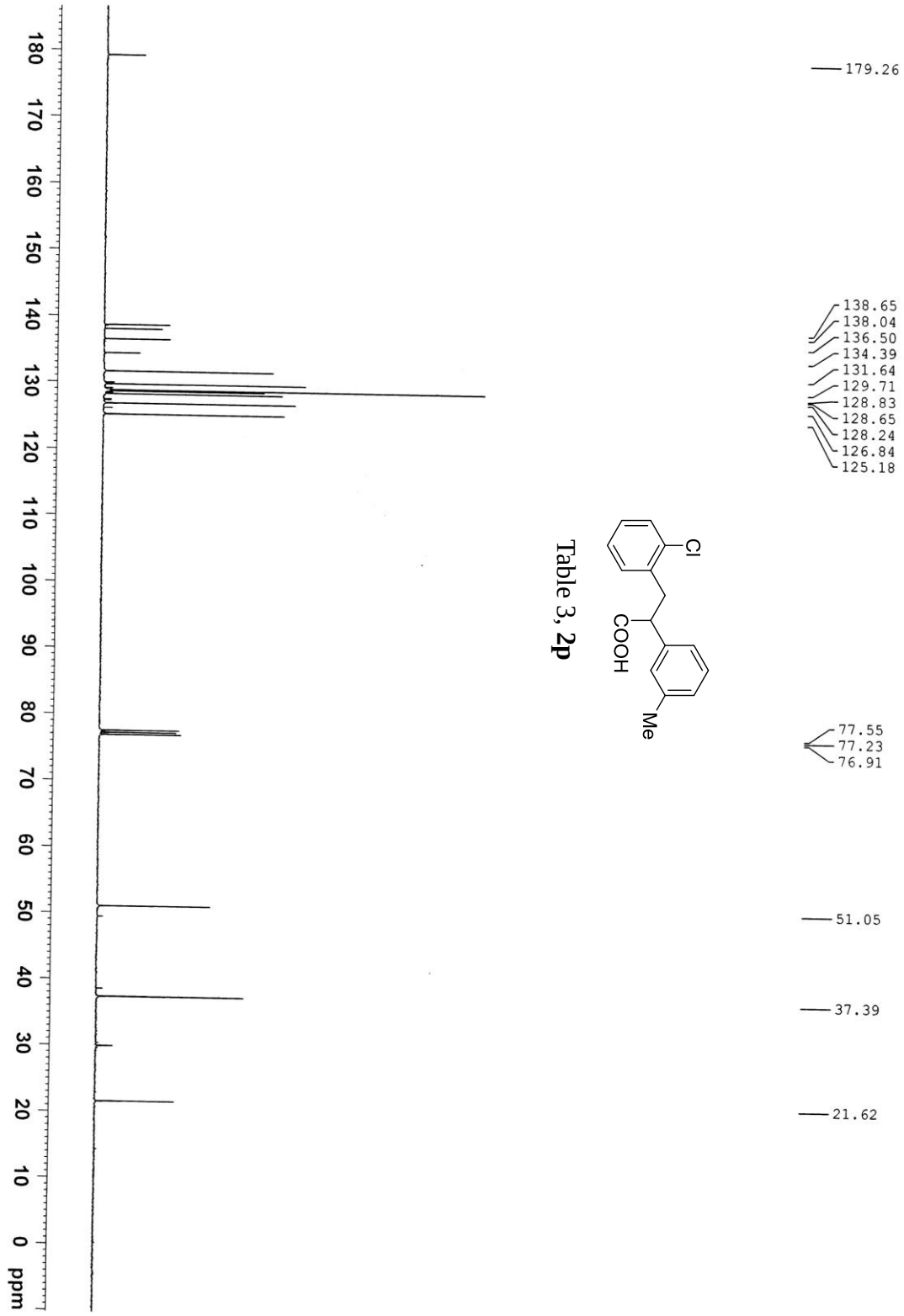
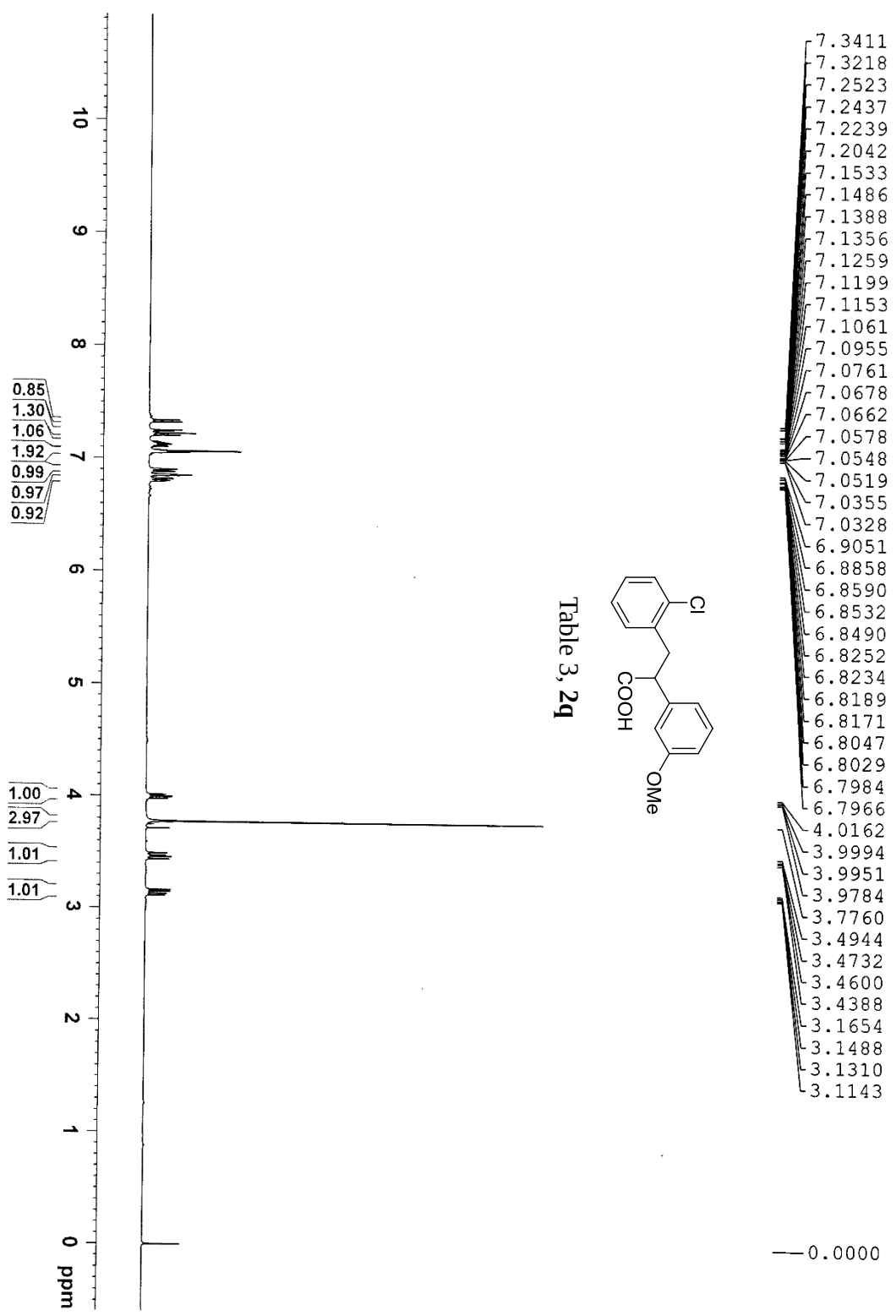

179.26
138.65
138.04
136.50
134.39
131.64
129.71
128.83
128.65
128.24
126.84
125.18
77.55
77.23
76.91
51.05
37.39
21.62
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
Cl
COOH
Me

Table 3, **2p**


7.3411
7.3218
7.2523
7.2437
7.2239
7.2042
7.1533
7.1486
7.1388
7.1356
7.1259
7.1199
7.1153
7.1061
7.0955
7.0761
7.0678
7.0662
7.0578
7.0548
7.0519
7.0355
7.0328
6.9051
6.8858
6.8590
6.8532
6.8490
6.8252
6.8234
6.8189
6.8171
6.8047
6.8029
6.7984
6.7966
4.0162
3.9994
3.9951
3.9784
3.7760
3.4944
3.4732
3.4600
3.4388
3.1654
3.1488
3.1310
3.1143
0.0000
Cl
COOH
OMe
Table 3, 2q
0.85
1.30
1.06
1.92
0.99
0.97
0.92
1.00
2.97
1.01
1.01
10
9
8
7
6
5
4
3
2
1
0
ppm

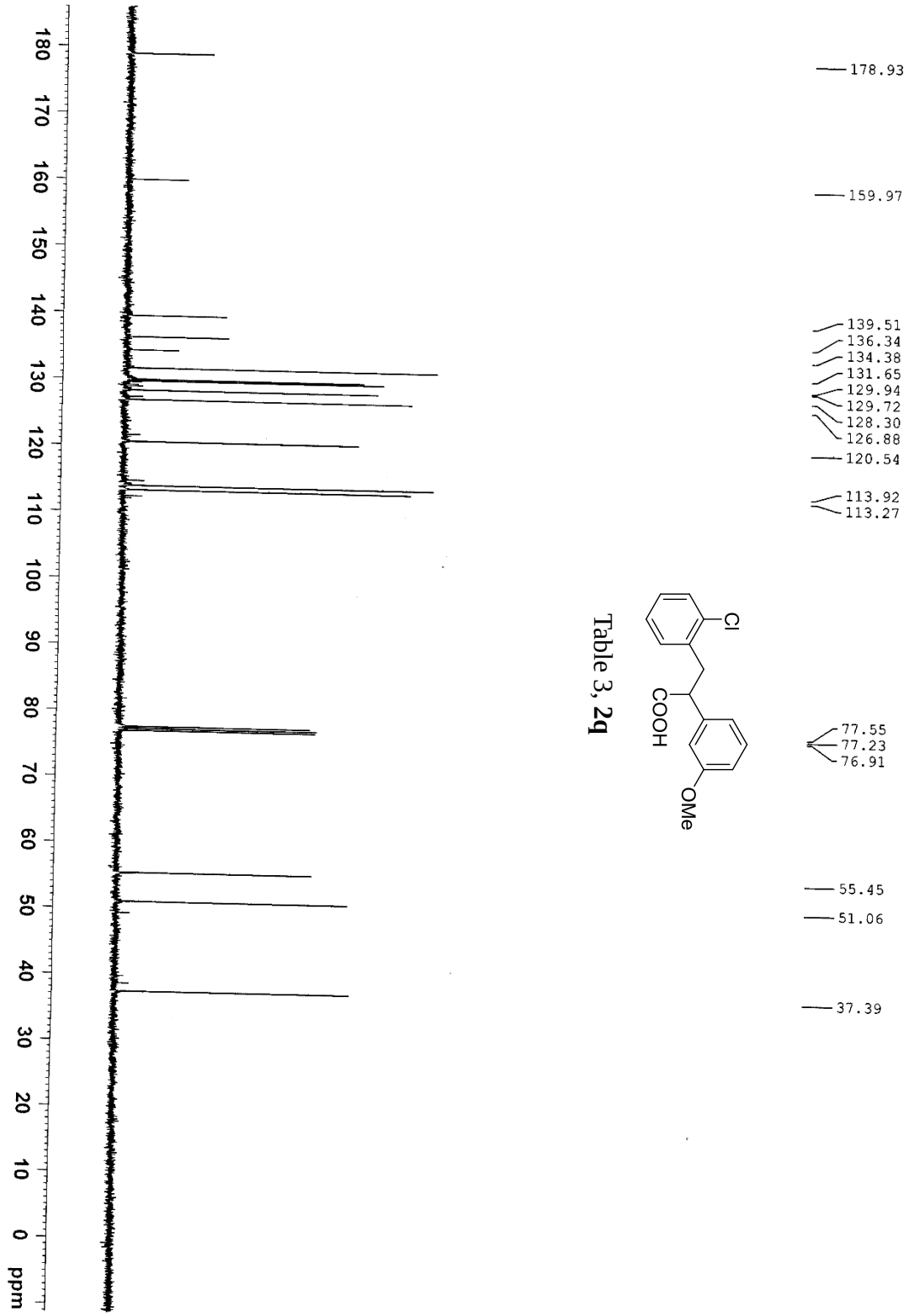

178.93
159.97
139.51
136.34
134.38
131.65
129.94
129.72
128.30
126.88
120.54
113.92
113.27
77.55
77.23
76.91
55.45
51.06
37.39
Cl
COOH
OMe
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Table 3, **2q**

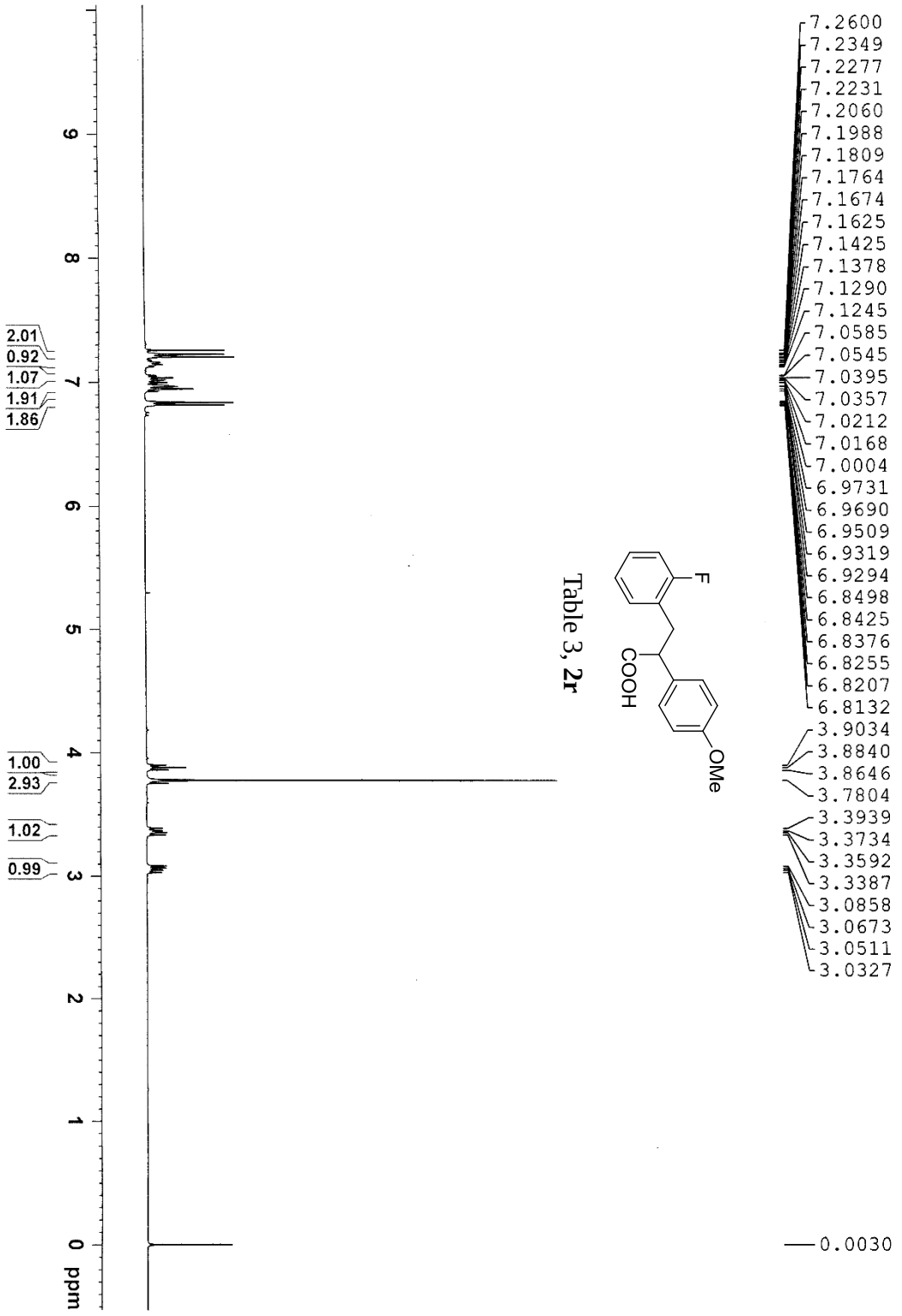

7.2600
7.2349
7.2277
7.2231
7.2060
7.1988
7.1809
7.1764
7.1674
7.1625
7.1425
7.1378
7.1290
7.1245
7.0585
7.0545
7.0395
7.0357
7.0212
7.0168
7.0004
6.9731
6.9690
6.9509
6.9319
6.9294
6.8498
6.8425
6.8376
6.8255
6.8207
6.8132
3.9034
3.8840
3.8646
3.7804
3.3939
3.3734
3.3592
3.3387
3.0858
3.0673
3.0511
3.0327
0.0030
2.01
0.92
1.07
1.91
1.86
1.00
2.93
1.02
0.99
9
8
7
6
5
4
3
2
1
0 ppm
F
COOH
OMe

Table 3, **2r**

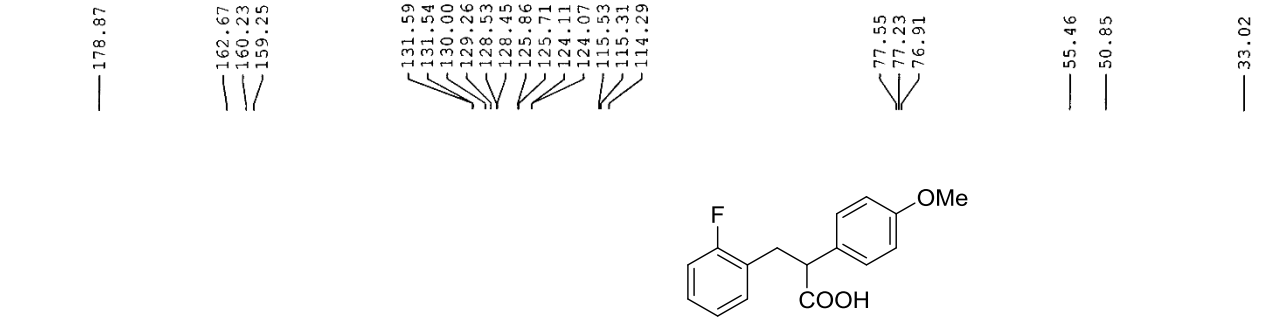
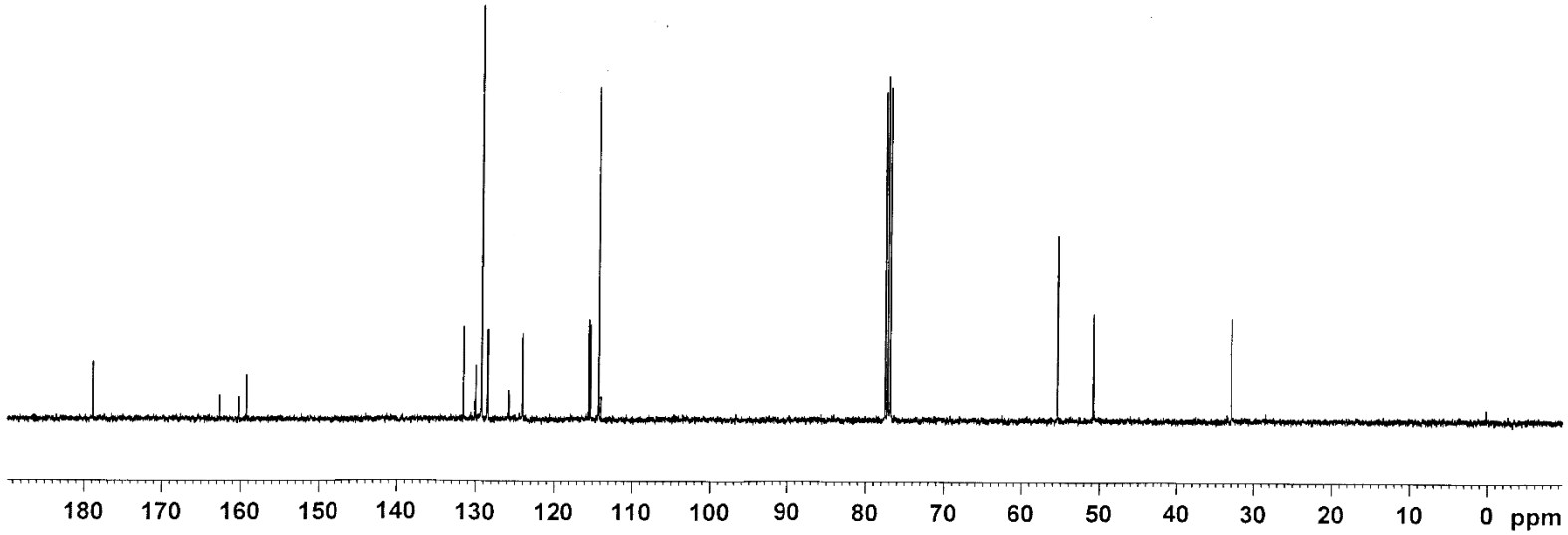

178.87
162.67
160.23
159.25
131.59
131.54
130.00
129.26
128.53
128.45
125.86
125.71
124.11
124.07
115.53
115.31
114.29
77.55
77.23
76.91
55.46
50.85
33.02
F
OMe
COOH

Table 3, **2r**


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

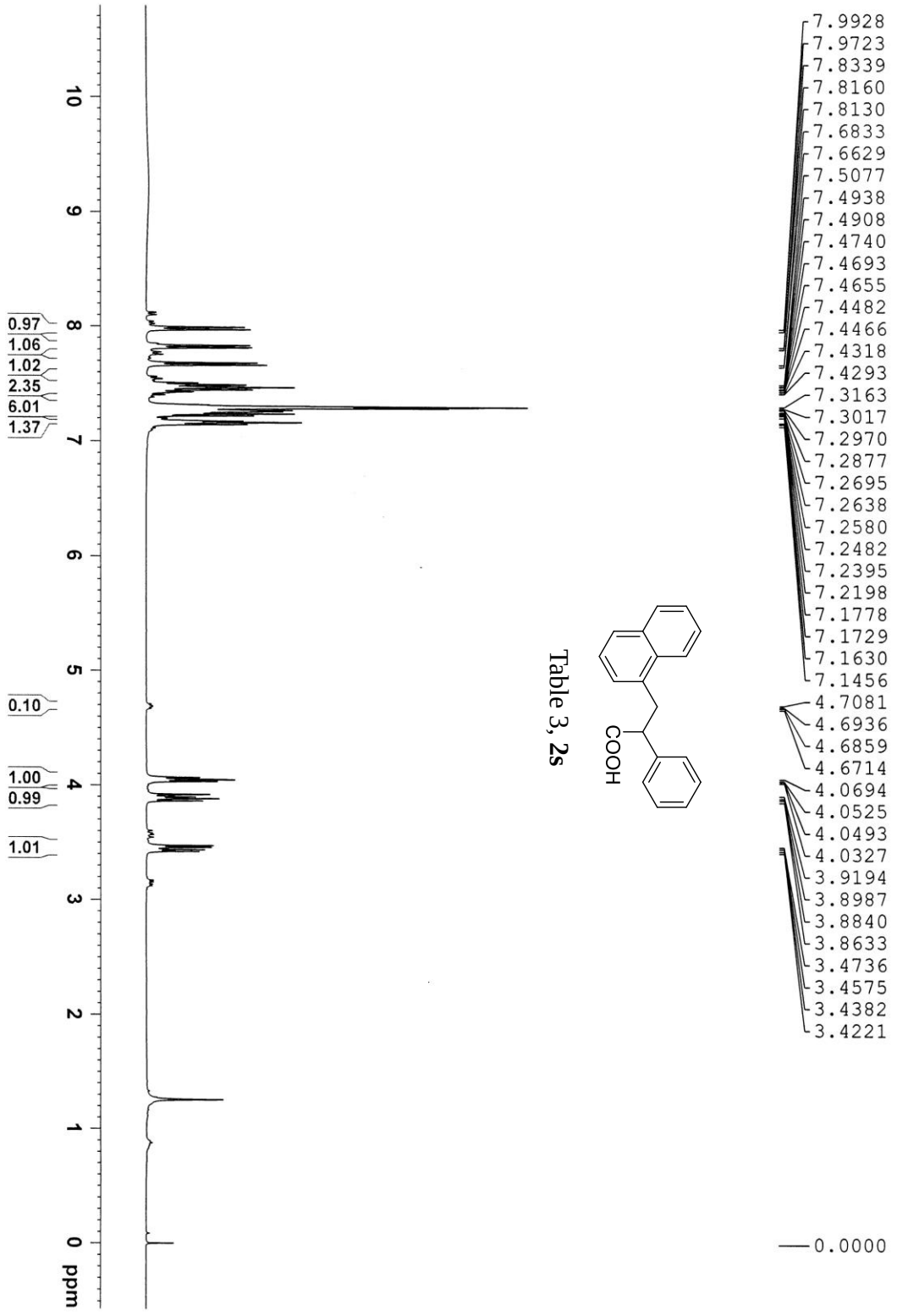

7.9928
7.9723
7.8339
7.8160
7.8130
7.6833
7.6629
7.5077
7.4938
7.4908
7.4740
7.4693
7.4655
7.4482
7.4466
7.4318
7.4293
7.3163
7.3017
7.2970
7.2877
7.2695
7.2638
7.2580
7.2482
7.2395
7.2198
7.1778
7.1729
7.1630
7.1456
4.7081
4.6936
4.6859
4.6714
4.0694
4.0525
4.0493
4.0327
3.9194
3.8987
3.8840
3.8633
3.4736
3.4575
3.4382
3.4221
0.0000
0.97
1.06
1.02
2.35
6.01
1.37
0.10
1.00
0.99
1.01
10
9
8
7
6
5
4
3
2
1
0
ppm

Table 3, **2s**

COOH

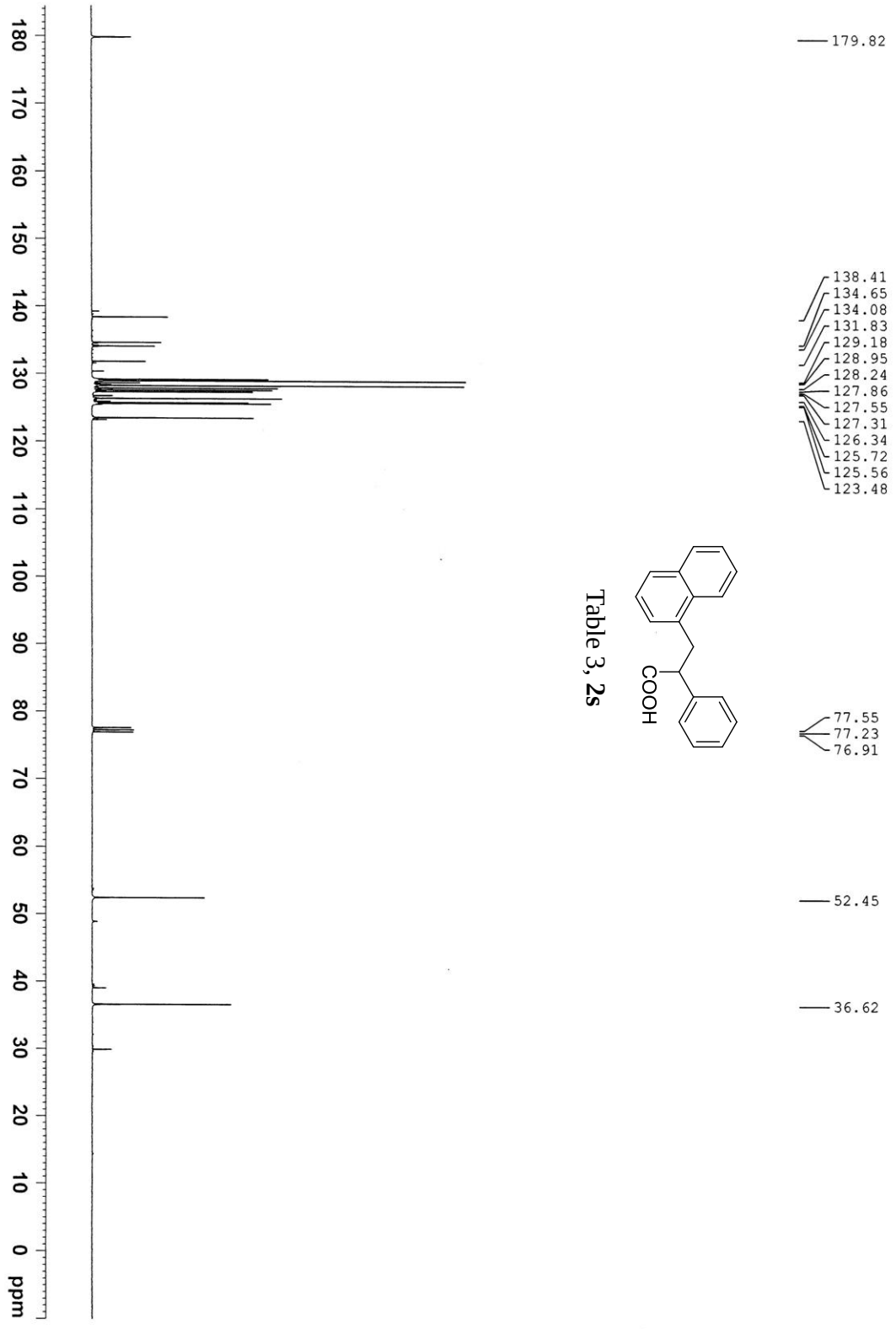

179.82
138.41
134.65
134.08
131.83
129.18
128.95
128.24
127.86
127.55
127.31
126.34
125.72
125.56
123.48
77.55
77.23
76.91
52.45
36.62
COOH
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Table 3, **2s**

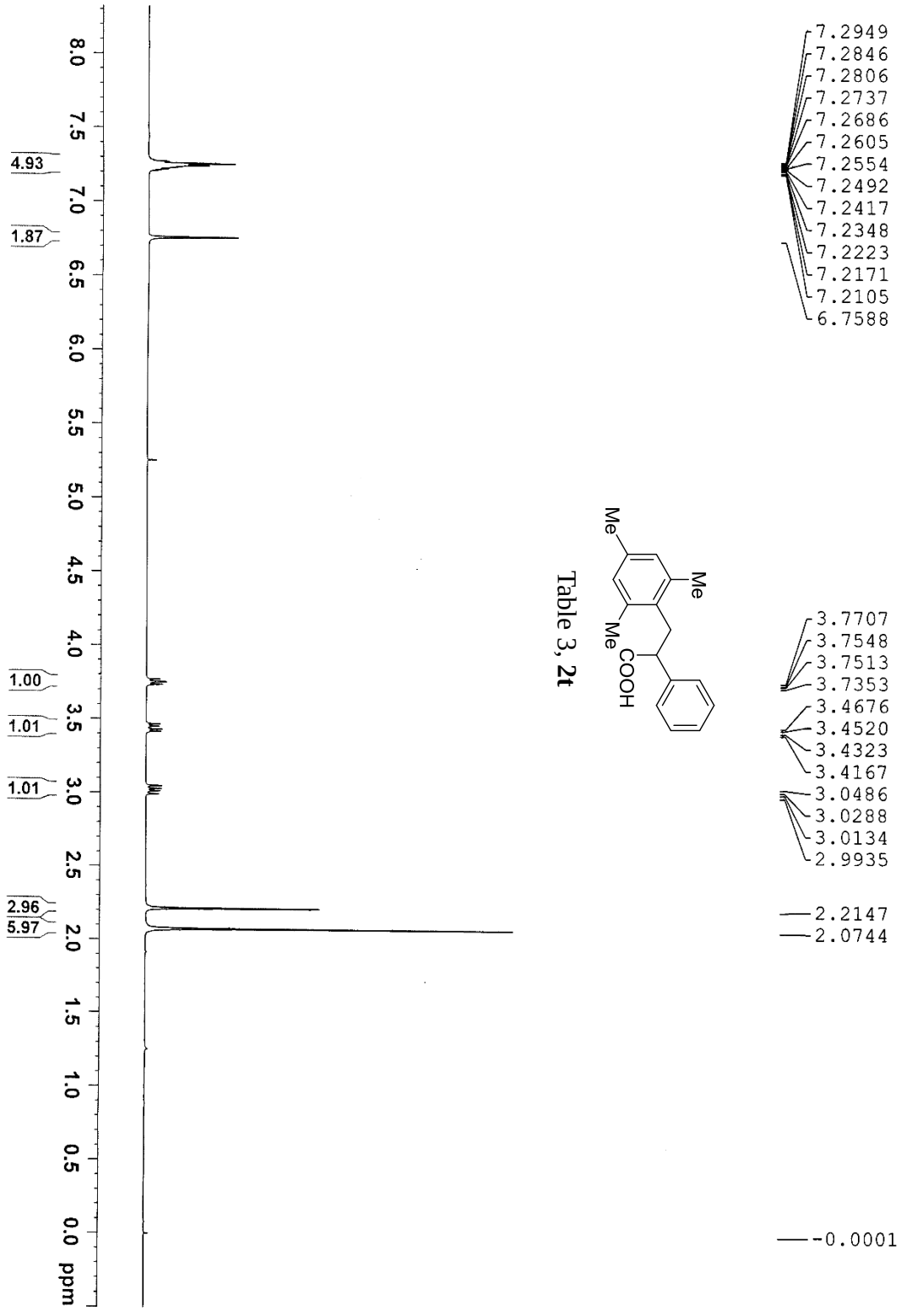

7.2949
7.2846
7.2806
7.2737
7.2686
7.2605
7.2554
7.2492
7.2417
7.2348
7.2223
7.2171
7.2105
6.7588
3.7707
3.7548
3.7513
3.7353
3.4676
3.4520
3.4323
3.4167
3.0486
3.0288
3.0134
2.9935
2.2147
2.0744
-0.0001
4.93
1.87
1.00
1.01
1.01
2.96
5.97
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Me
Me
Me
COOH

Table 3, **2t**

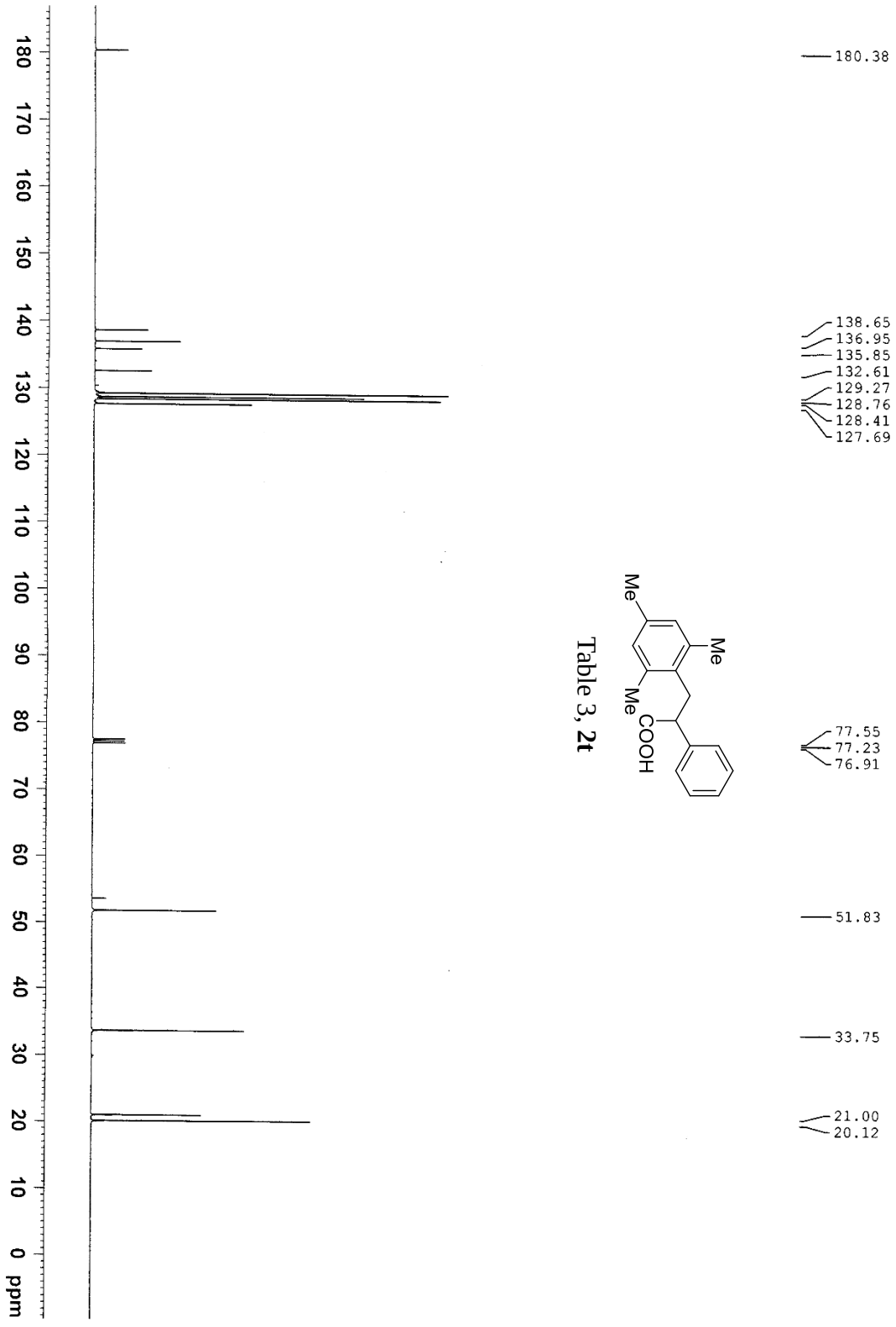

180.38
138.65
136.95
135.85
132.61
129.27
128.76
128.41
127.69
77.55
77.23
76.91
51.83
33.75
21.00
20.12
Me
Me
Me
COOH
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Table 3, **2t**