Cationic Palladium(II) Catalysis : C-H Activation/Suzuki-Miyaura Couplings at Room Temperature

*Takashi Nishikata, Alexander R. Abela, Shenlin Huang and Bruce H. Lipshutz**

Department of Chemistry & Biochemistry

University of California

Santa Barbara, CA 93106

Supporting Information

General Information

For TLC analyses precoated Kieselgel 60 F_{254} plates (Merck, 0.25 mm thick) were used; for column chromatography Silica *Flash*® P60 (SiliCycle, 40-63 μm) was used. Reactions were monitored using an Hewlett-Packard HP6890 gas chromatograph. ^{1}H and ^{13}C NMR spectra were obtained using a Varian UNITY INOVA 400 MHz NMR spectrometer. High resolution mass spectral analyses were obtained using a VG70 double-focusing magnetic sector instrument (VG Analytical) for EI and a PE Sciex QStar Pulsar quadrupole/TOF instrument (API) for ESI.

General procedure

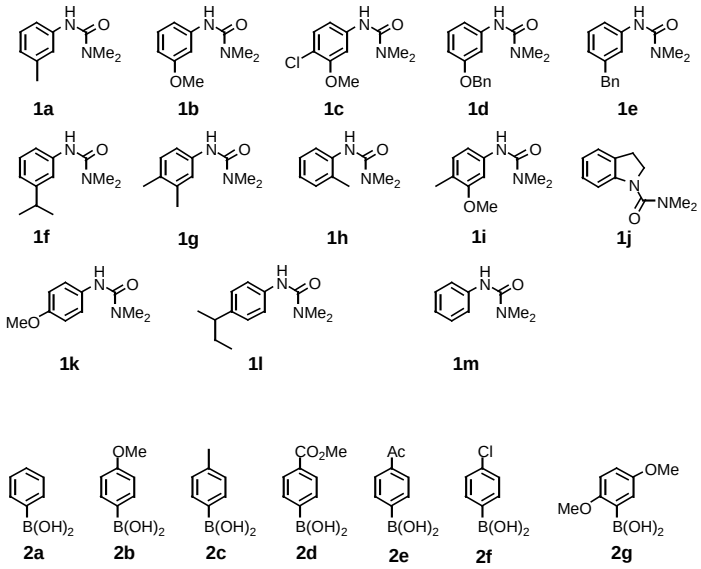
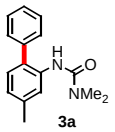


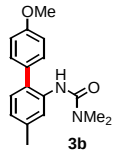
Figure 1. Starting materials.

Aryl urea **1** (0.25 mmol), arylboronic acid **2** (1.5-3 equiv), 1,4-benzoquinone (2-5 equiv), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg) were sequentially added in air to a reaction tube equipped with a stir bar and a septum. EtOAc was added by syringe and the resulting mixture vigorously stirred for 20 h at amibient temperature. After this time, the contents of the flask were quenched with aqueous NaOH (to remove 1,4-hydroxybenzene) and extracted with EtOAc. The solution obtained was filtered through the plug of silica gel and anhydrous $MgSO_4$, and then concentrated by rotary evaporation. The residue was purified by flash chromatography, eluting with hexane/EtOAc to afford the product.



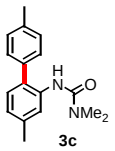
3a

Following the general procedure above, using **1a** (44 mg, 0.25 mmol), **2a** (96 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3a** (61 mg, 96%); ^{1}H NMR ($CDCl_3$) δ: 2.38 (s, 3H), 2.79 (s, 6H), 6.48 (brs, 1H), 6.90 (dd, J = 1.0 and 7.7 Hz, 1H), 7.08 (d, J = 7.7 Hz, 1H), 7.35-7.38 (m, 3H), 7.43-7.47 (m, 2H), 8.02 (brs, 1H). ^{13}C NMR ($CDCl_3$) δ: 21.72, 36.31, 121.07, 123.56, 127.84, 128.79, 129.20, 129.53, 129.57, 136.18, 138.59, 138.86, 155.67; HRESIMS calcd. for $C_{16}H_{18}N_2ONa$ ($M+Na^+$): 277.1317; found 277.1311.



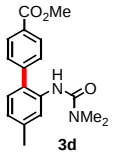
3b

Following the general procedure above, using **1a** (44 mg, 0.25 mmol), **2b** (114 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3b** (53 mg, 75%); ^{1}H NMR ($CDCl_3$) δ: 2.37 (s, 3H), 2.81 (s, 6H), 3.84 (s, 3H), 6.50 (brs, 1H), 6.87 (dd, J = 1.0 and 7.7 Hz, 1H), 6.98 (d, J = 8.7 Hz, 2H), 7.05 (d, J = 7.7 Hz, 1H), 7.28 (d, J = 8.7 Hz, 2H), 8.01 (brs, 1H). ^{13}C NMR ($CDCl_3$) δ: 21.52, 36.17, 55.30, 114.37, 120.78, 123.30, 128.23, 129.54, 130.47, 130.74, 136.16, 138.05, 155.51, 159.04; HRESIMS calcd. for $C_{17}H_{20}N_2O_2Na$ ($M+Na^+$): 307.1422; found 307.1418.



3c

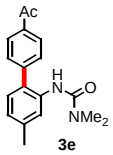
Following the general procedure above, using **1a** (44 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3c** (60 mg, 90%); ^{1}H NMR ($CDCl_3$) δ: 2.38 (s, 3H), 2.40 (s, 3H), 2.81 (s, 6H), 6.52 (brs, 1H), 6.88 (dd, J = 1.0 and 7.7 Hz, 1H), 7.07 (d, J = 7.7 Hz, 1H), 7.26 (brs, 4H), 8.02 (s, 1H).[1]



3d

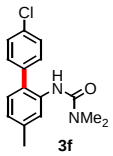
Following the general procedure above, using **1a** (44 mg, 0.25 mmol), **2d** (135 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3d** (73 mg, 94%); ^{1}H NMR ($CDCl_3$) δ: 2.38 (s, 3H), 2.82 (s, 6H), 3.94 (s, 3H), 6.37 (brs, 1H), 6.92 (dd, J = 1.2 and 8.4 Hz, 1H), 7.09 (d, J = 7.8 Hz, 1H), 7.46 (d, J = 7.8 Hz, 2H), 7.95

(brs, 1H), 8.12 (d, J = 8.4 Hz, 2H). ^{13}C NMR ($CDCl_3$) δ: 21.10, 35.81, 51.83, 121.52, 123.51, 127.64, 128.85, 128.96, 129.79, 135.35, 138.68, 143.31, 155.05, 166.29; HRESIMS calcd. for $C_{18}H_{20}N_2O_3Na$ ($M+Na^+$): 335.1372; found 335.1377.



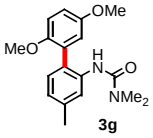
3e

Following the general procedure above, using **1a** (44 mg, 0.25 mmol), **2e** (123 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3e** (63 mg, 86%); ^{1}H NMR ($CDCl_3$) δ: 2.38 (s, 3H), 2.64 (s, 3H), 2.83 (s, 6H), 6.38 (brs, 1H), 6.93 (d, J = 7.7 Hz, 1H), 7.09 (d, J = 7.7 Hz, 1H), 7.49 (d, J = 8.3 Hz, 2H), 7.93 (brs, 1H), 8.04 (d, J = 8.3 Hz, 2H). ^{13}C NMR ($CDCl_3$) δ: 21.50, 26.64, 36.22, 122.12, 124.00, 128.06, 128.92, 129.36, 129.51, 135.71, 136.04, 139.12, 143.91, 155.46, 197.53; HRESIMS calcd. for $C_{18}H_{20}N_2O_2Na$ ($M+Na^+$): 319.1422; found 319.1417.



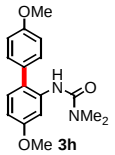
3f

Following the general procedure above, using **1a** (44 mg, 0.25 mmol), **2f** (117 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3f** (67 mg, 94%); ^{1}H NMR ($CDCl_3$) δ: 2.37 (s, 3H), 2.83 (s, 6H), 6.33 (brs, 1H), 6.91 (d, J = 7.7 Hz, 1H), 7.05 (d, J = 7.7 Hz, 1H), 7.30 (d, J = 8.4 Hz, 2H), 7.42 (d, J = 8.4 Hz, 2H), 7.93 (brs, 1H). ^{13}C NMR ($CDCl_3$) δ: 21.69, 36.44, 121.95, 124.06, 127.98, 129.35, 129.64, 130.89, 133.84, 135.94, 137.37, 138.99, 155.72; HRESIMS calcd. for $C_{16}H_{17}N_2ONaCl$ ($M+Na^+$): 311.0927; found 311.0928.



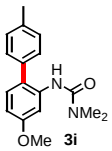
3g

Following the general procedure above, using **1a** (44 mg, 0.25 mmol), **2g** (136 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3g** (77 mg, 98%); ^{1}H NMR ($CDCl_3$) δ: 2.38 (s, 3H), 2.83 (s, 6H), 3.74 (s, 3H), 3.77 (s, 3H), 6.79 (d, J = 3.0 Hz, 1H), 6.89 (dd, J = 3.0 and 8.9 Hz, 1H), 6.89-6.90 (m, 2H), 7.03 (brs, 1H), 7.10 (d, J = 7.8 Hz, 1H), 7.78 (brs, 1H). ^{13}C NMR ($CDCl_3$) δ: 21.53, 36.13, 55.83, 56.97, 112.99, 114.35, 117.75, 123.17, 124.25, 127.19, 129.51, 130.33, 136.75, 138.39, 150.21, 154.42, 156.00; HRESIMS calcd. for $C_{18}H_{22}N_2O_3Na$ ($M+Na^+$): 337.1528; found 337.1535.



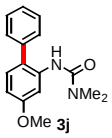
3h

Following the general procedure above, using **1b** (48 mg, 0.25 mmol), **2b** (114 mg, 0.75 mmol), BQ (0.5 mmol, 54 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3h** (60 mg, 81%); ^{1}H NMR ($CDCl_3$) δ: 2.81 (s, 6H), 3.84 (s, 6H), 6.60 (brs, 1H), 6.61 (dd, J = 2.6 and 8.3 Hz, 1H), 6.98 (d, J = 8.6 Hz, 2H), 7.05 (d, J = 8.3 Hz, 1H), 7.27 (d, J = 8.6 Hz, 2H), 7.92 (d, J = 2.6 Hz, 1H).[1]



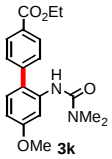
3i

Following the general procedure above, using **1b** (48 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (0.5 mmol, 54 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3i** (61 mg, 87%); ^{1}H NMR ($CDCl_3$) δ: 2.40 (s, 3H), 2.81 (s, 6H), 3.85 (s, 3H), 6.63 (dd, J = 2.7 and 8.5 Hz, 1H), 6.64 (brs, 1H), 7.02 (d, J = 8.5 Hz, 1H), 7.25 (brs, 4H), 7.94 (d, J = 2.7 Hz, 1H).[1]



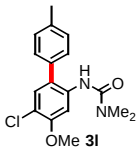
3j

Following the general procedure above, using **1b** (48 mg, 0.25 mmol), **2a** (96 mg, 0.75 mmol), BQ (0.5 mmol, 54 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3j** (62 mg, 70%); ^{1}H NMR ($CDCl_3$) δ: 2.96 (s, 6H), 3.85 (s, 3H), 6.60 (brs, 1H), 6.63 (dd, J = 2.6 and 8.4 Hz, 1H), 7.08 (d, J = 8.6 Hz, 1H), 7.34-7.38 (m, 3H), 7.43-7.47 (m, 2H), 7.94 (d, J = 2.6 Hz, 1H).[1]



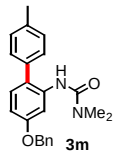
3k

Following the general procedure above, using **1b** (48 mg, 0.25 mmol), **2d** (135 mg, 0.75 mmol), BQ (0.5 mmol, 54 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3k** (60 mg, 70%), ^{1}H NMR ($CDCl_3$) δ: 1.41 (t, J = 7.1 Hz, 3H), 2.82 (s, 6H), 3.84 (s, 3H), 4.39 (q, J = 7.1 Hz, 2H), 6.49 (brs, 1H), 6.65 (dd, J = 2.6 and 8.4 Hz, 1H), 7.08 (d, J = 8.4 Hz, 1H), 7.44 (d, J = 8.4 Hz, 2H), 7.88 (d, J = 2.6 Hz, 1H), 8.11 (d, J = 8.4 Hz, 2H).[1]



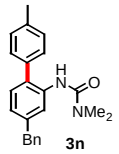
3l

Following the general procedure above, using **1c** (57 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3l** (77 mg, 97%); ^{1}H NMR ($CDCl_3$) δ: 2.40 (s, 3H), 2.81 (s, 6H), 3.95 (s, 3H), 6.65 (brs, 1H), 7.22 (d, J = 8.0 Hz, 2H), 7.27 (d, J = 8.0 Hz, 2H), 8.09 (brs, 1H). ^{13}C NMR ($CDCl_3$) δ: 21.20, 36.15, 56.20, 103.76, 115.12, 123.79, 129.14, 129.90, 130.42, 134.04, 136.13, 137.85, 154.42, 155.18;



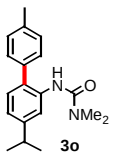
3m

Following the general procedure above, using **1d** (67 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3m** (72 mg, 80%);^{1}H NMR ($CDCl_3$) δ: 2.40 (s, 3H), 2.82 (s, 6H), 5.11 (s, 2H), 6.65 (brs, 1H), 6.70 (dd, J = 2.6 and 8.4 Hz, 1H), 7.08 (d, J = 8.4 Hz, 1H), 7.26 (brs, 4H), 7.32 (d, J = 7.2 Hz, 1H), 7.39 (t, J = 7.2 Hz, 2H), 7.47 (d, J = 7.2 Hz, 2H), 8.05 (d, J = 2.6 Hz, 1H).[1]



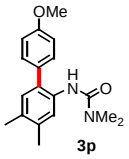
3n

Following the general procedure above, using **1e** (63 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3n** (69 mg, 80%);^{1}H NMR ($CDCl_3$) δ: 2.38 (s, 3H), 2.79 (s, 6H), 3.98 (s, 2H), 6.54 (brs, 1H), 6.85 (dd, J = 1.7 and 7.8 Hz, 1H), 7.07 (d, J = 7.8 Hz, 1H), 7.14-7.19 (m, 1H), 7.24-7.27 (m, 8H), 8.11 (d, J = 1.7 Hz, 1H). ^{13}C NMR ($CDCl_3$) δ: 21.20, 36.20, 42.09, 116.00, 120.73, 123.06, 125.97, 128.43, 128.95, 129.15, 129.73, 129.79, 135.51, 136.24, 137.47, 141.21, 141.44, 155.55; HRESIMS calcd. for $C_{23}H_{24}N_2ONa$ ($M+Na^+$): 367.1786; found 367.1793.



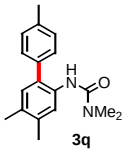
3o

Following the general procedure above, using **1f** (51 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3o** (66 mg, 89%); ^{1}H NMR ($CDCl_3$) δ: 1.29 (t, J = 7.0 Hz, 6H), 2.40 (s, 3H), 2.82 (s, 6H), 2.95 (sept, J = 7.0 Hz, 1H), 6.57 (brs, 1H), 6.93 (dd, J = 1.7 and 8.7 Hz, 1H), 7.11 (d, J = 8.7 Hz, 1H), 7.26 (brs, 4H), 8.10 (d, J = 1.7 Hz, 1H).[1]



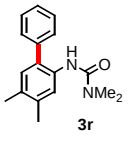
3p

Following the general procedure above, using **1g** (48 mg, 0.25 mmol), **2b** (114 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3p** (64 mg, 86%), ^{1}H NMR ($CDCl_3$) δ: 2.22 (s, 3H), 2.28 (s, 3H), 2.82 (s, 6H), 3.85 (s, 3H), 6.38 (brs, 1H), 6.95 (brs, 1H), 6.98 (dd, J= 8.8 Hz, 2H), 7.28 (d, J= 8.8 Hz, 2H), 7.91 (s, 1H).[1]



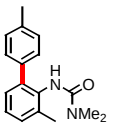
3q

Following the general procedure above, using **1g** (48 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3q** (67 mg, 96%), ^{1}H NMR ($CDCl_3$) δ: 2.22 (s, 3H), 2.29 (s, 3H), 2.39 (s, 3H), 2.81 (s, 6H), 6.42 (brs, 1H), 6.96 (s, 1H), 7.25 (brs, 4H), 7.92 (s, 1H).[1]



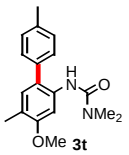
3r

Following the general procedure above, using **1g** (48 mg, 0.25 mmol), **2a** (96 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3r** (64 mg, 95%), ^{1}H NMR ($CDCl_3$) δ: 2.23 (s, 3H), 2.30 (s, 3H), 2.80 (s, 6H), 6.37 (brs, 1H), 6.98 (s, 1H), 7.34-7.38(m, 3H), 7.43-7.47 (m, 2H), 7.93 (s, 1H).[1]



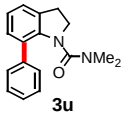
3s

Following the general procedure above, using **1h** (44 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3s** (40 mg, 60%); ^{1}H NMR ($CDCl_3$) δ: 2.32 (s, 3H), 2.39 (s, 3H), 2.85 (s, 6H), 5.71 (brs, 1H), 7.12 (dd, J= 1.8 and 7.5 Hz, 1H), 7.17 (t, J= 7.5 Hz, 1H), 7.20-7.26 (m, 5H).[1]



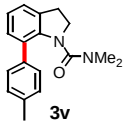
3t

Following the general procedure above, using **1i** (52 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3t** (60 mg, 80%); ^{1}H NMR ($CDCl_3$) δ: 2.18 (s, 3H), 2.39 (s, 3H), 2.82 (s, 6H), 3.89 (s, 3H), 6.59 (brs, 1H), 6.94 (s, 1H), 7.24 (brs, 4H), 7.87 (s, 1H).[1]



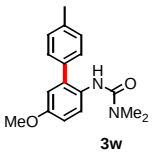
3u

Following the general procedure above, using **1j** (47 mg, 0.25 mmol), **2a** (96 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3u** (60 mg, 90%); ^{1}H NMR ($CDCl_3$) δ: 2.51 (s, 6H), 3.07 (t, J = 8.0 Hz, 2H), 3.94 (t, J = 8.0 Hz, 2H), 6.99 (t, J = 7.8 Hz, 1H), 7.12 (t, J = 8.1 Hz, 2H), 7.20-7.24 (m, 2H), 7.32 (d, J = 7.8 Hz, 2H), 7.39 (d, J = 8.1 Hz, 2H). ^{13}C NMR ($CDCl_3$) δ: 29.85, 36.95, 52.00, 123.10, 126.85, 127.16, 128.34, 129.13, 129.23, 133.81, 140.46, 142.49, 159.79; HRESIMS calcd. for $C_{17}H_{18}N_2ONa$ ($M+Na^+$): 289.1317; found 289.1314.



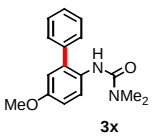
3v

Following the general procedure above, using **1j** (47 mg, 0.25 mmol), **2c** (102 mg, 0.75 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3v** (62 mg, 89%); ^{1}H NMR ($CDCl_3$) δ: 2.34 (s, 3H), 2.57 (s, 6H), 3.09 (t, J = 7.9 Hz, 2H), 3.96 (t, J = 7.9 Hz, 2H), 6.99 (t, J = 7.8 Hz, 1H), 6.98-7.02 (m, 4H), 7.26 (brs, 1H), 7.32 (d, J = 8.0 Hz, 2H). ^{13}C NMR ($CDCl_3$) δ: 21.24, 29.80, 36.90, 51.92, 123.03, 123.45, 126.86, 128.95, 129.22, 133.70, 136.29, 137.49, 142.38, 159.80; HRESIMS calcd. for $C_{18}H_{20}N_2ONa$ ($M+Na^+$): 303.1473; found 303.1473.



3w

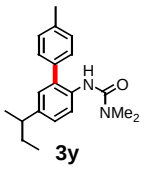
Following the general procedure above, using **1k** (48 mg, 0.25 mmol), **2c** (51 mg, 0.375 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3w** (58 mg, 82%), ^{1}H NMR ($CDCl_3$) δ: 2.39 (s, 3H), 2.80 (s, 6H), 3.78 (s, 3H), 6.29 (brs, 1H), 6.75 (d, J = 3.0 Hz, 1H), 6.87 (dd, J = 3.0 and 9.0 Hz, 1H), 7.28 (brs, 4H), 7.93 (d, J = 9.0 Hz, 1H).[1]



3x

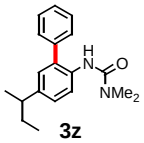
Following the general procedure above, using **1k** (48 mg, 0.25 mmol), **2a** (46 mg, 0.375 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded

the product **3x** (60 mg, 89%), ^{1}H NMR ($CDCl_3$) δ: 2.79 (s, 6H), 3.79 (s, 3H), 6.24 (brs, 1H), 6.77 (d, *J* = 3.0 Hz, 1H), 6.89 (dd, *J* = 3.0 and 9.0 Hz, 1H), 7.26-7.40 (m, 3H), 7.44-7.48 (m, 2H), 7.93 (d, *J* = 9.0 Hz, 1H). ^{13}C NMR ($CDCl_3$) δ: 35.75, 55.12, 113.10, 114.67, 122.69, 127.44, 128.54, 128.73, 129.03, 133.16, 138.28, 154.90, 155.54; HRESIMS calcd. for $C_{16}H_{19}N_2O_2Na$ ($M+Na^+$): 271.1447; found 271.1450.



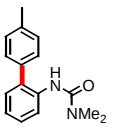
3y

Following the general procedure above, using **1l** (55 mg, 0.25 mmol), **2c** (51 mg, 0.375 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3y** (68 mg, 88%); ^{1}H NMR ($CDCl_3$) δ: 0.83 (t, *J* = 7.3 Hz, 3H), 1.22 (t, *J* = 7.0 Hz, 3H), 1.52-1.61 (m, 2H), 2.40 (s, 3H), 2.56 (sext, *J* = 7.0 Hz, 1H), 2.81 (s, 6H), 6.47 (brs, 1H), 6.99 (d, *J* = 2.2 Hz, 1H), 7.13 (dd, *J* = 2.2 and 8.4 Hz, 1H), 7.25-7.30 (m, 4H), 8.01 (d, *J* = 8.4 Hz, 1H).[1]



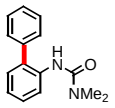
3z

Following the general procedure above, using **1l** (55 mg, 0.25 mmol), **2a** (46 mg, 0.375 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3z** (59 mg, 80%); ^{1}H NMR ($CDCl_3$) δ: 0.83 (t, *J* = 7.5 Hz, 3H), 1.22 (t, *J* = 6.9 Hz, 3H), 1.56 (sept, *J* = 7.5 Hz, 2H), 2.54-2.62 (m, 1H), 2.79 (s, 6H), 6.42 (brs, 1H), 7.00 (d, *J* = 1.9 Hz, 1H), 7.15 (dd, *J* = 1.9 and 8.5 Hz, 1H), 7.35-7.40 (m, 3H), 7.44-7.48 (m, 2H), 8.02 (t, *J* = 8.5 Hz, 1H).[1]



3aa

Following the general procedure above, using **1m** (41 mg, 0.25 mmol), **2c** (51 mg, 0.375 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3aa** (50 mg, 78%); ^{1}H NMR ($CDCl_3$) δ: 2.40 (s, 3H), 2.80 (s, 6H), 6.55 (brs, 1H), 7.05 (t, *J* = 7.4 Hz, 1H), 7.17 (d, *J* = 7.4 Hz, 1H), 7.27 (brs, 4H), 7.31 (t, *J* = 8.3 Hz, 1H), 8.17 (d, *J* = 8.3 Hz, 1H).[1]

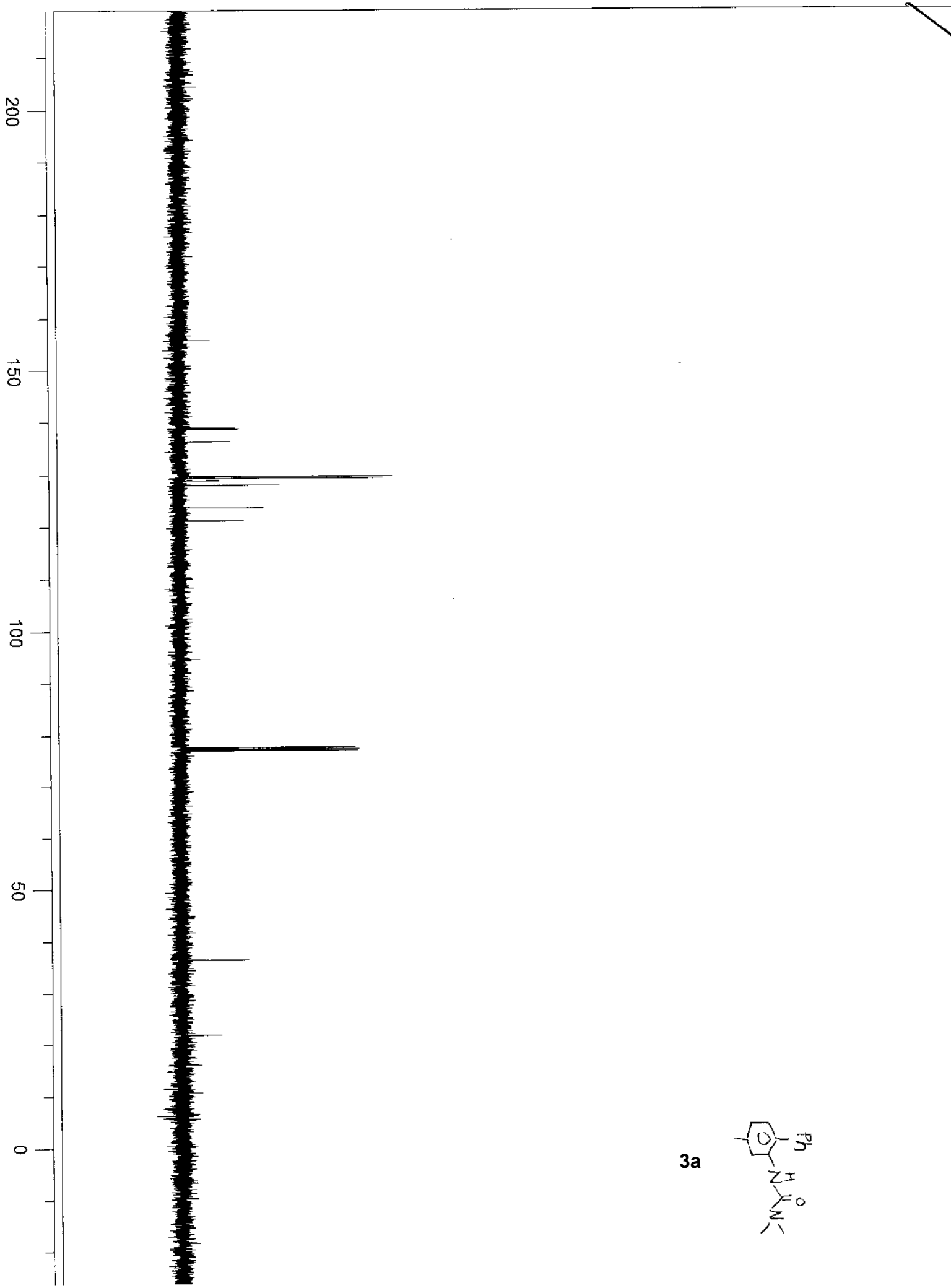


3bb

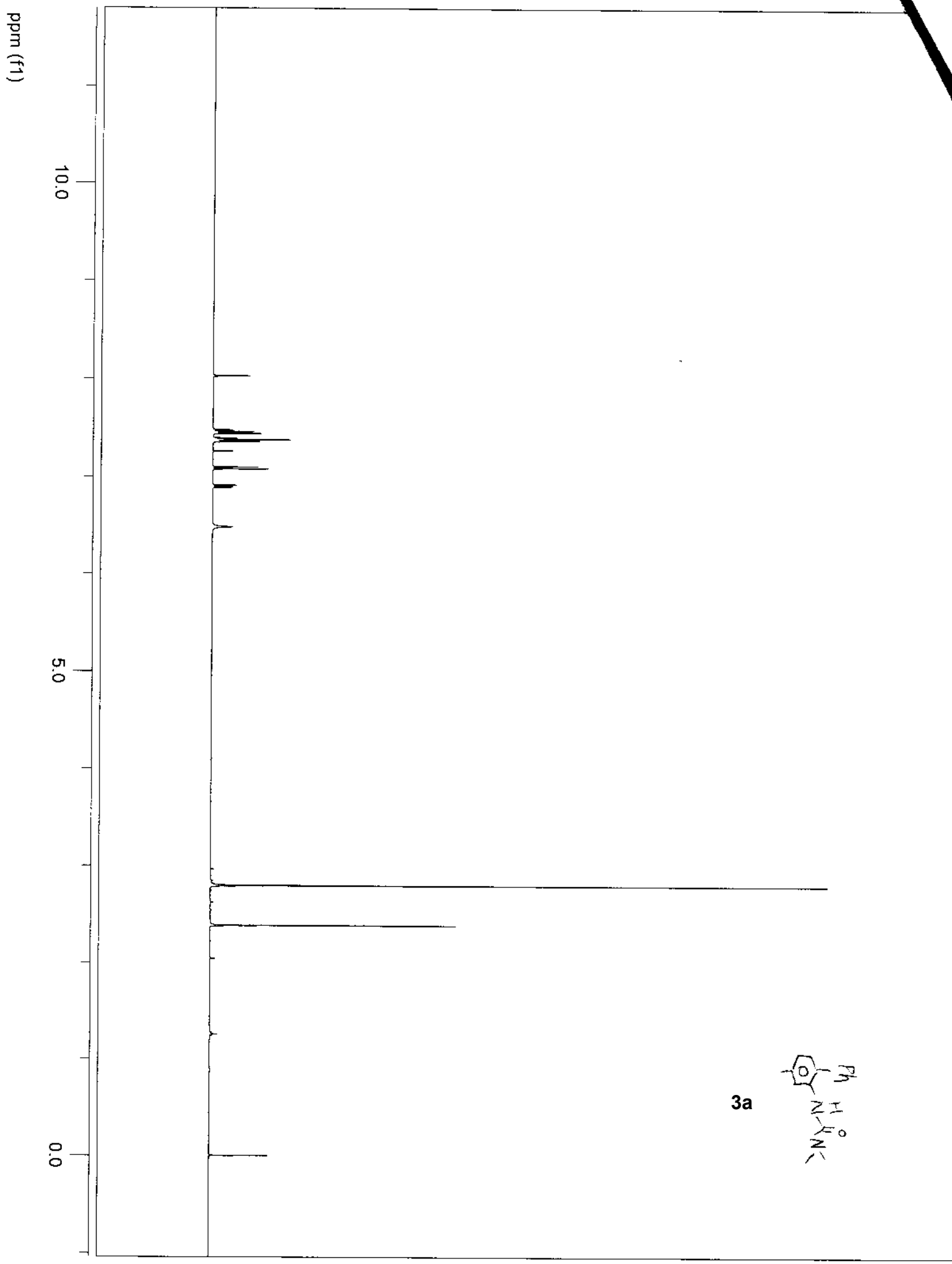
Following the general procedure above, using **1m** (41 mg, 0.25 mmol), **2a** (46 mg, 0.375 mmol), BQ (1.25 mmol, 135 mg), and $[Pd(MeCN)_4](BF_4)_2$ (0.025 mmol, 11 mg), EtOAc (1.0 mL), yielded the product **3bb** (50 mg, 83%); ^{1}H NMR ($CDCl_3$) δ: 2.78 (s, 6H), 6.51 (brs, 1H), 7.06 (dt, J = 1.2 and 7.5 Hz, 1H), 7.19 (dd, J = 1.6 and 7.5 Hz, 1H), 7.31-7.40 (m, 4H), 7.45-7.49 (m, 2H), 8.16 (dd, J = 1.2 and 7.4 Hz, 1H). ^{13}C NMR ($CDCl_3$) δ: 36.31, 120.54, 122.74, 128.01, 128.60, 129.23, 129.41, 129.74, 131.55, 136.38, 138.76, 155.64; HRESIMS calcd. for $C_{15}H_{16}N_2ONa$ ($M+Na^+$): 263.1160; found 263.1151.

Reference

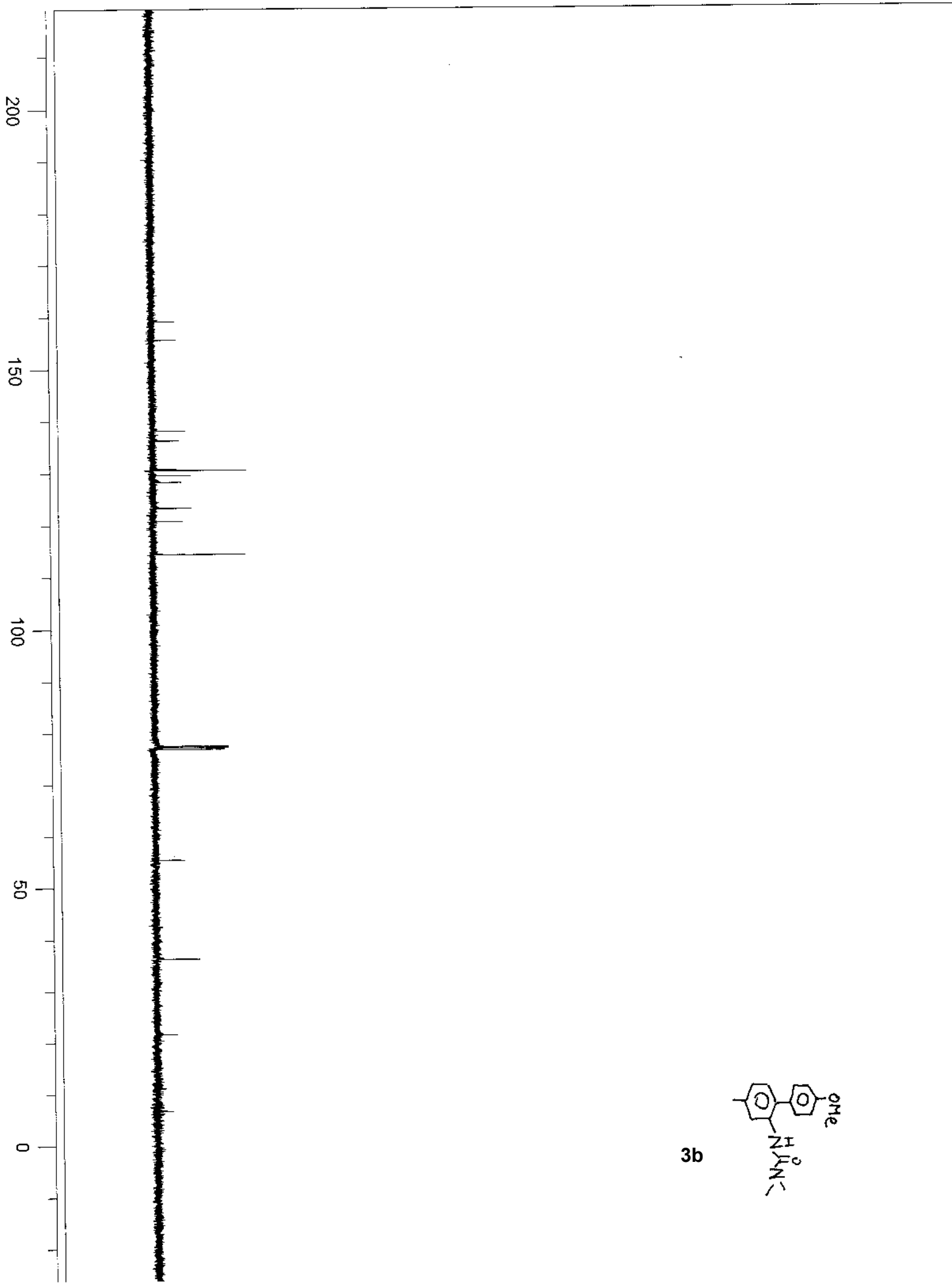
1) Nishikata, T.; Abela, A. R.; Lipshutz, B. H. *Angew. Chem., Int. Ed*. DOI: 10.1002/anie.200905967.



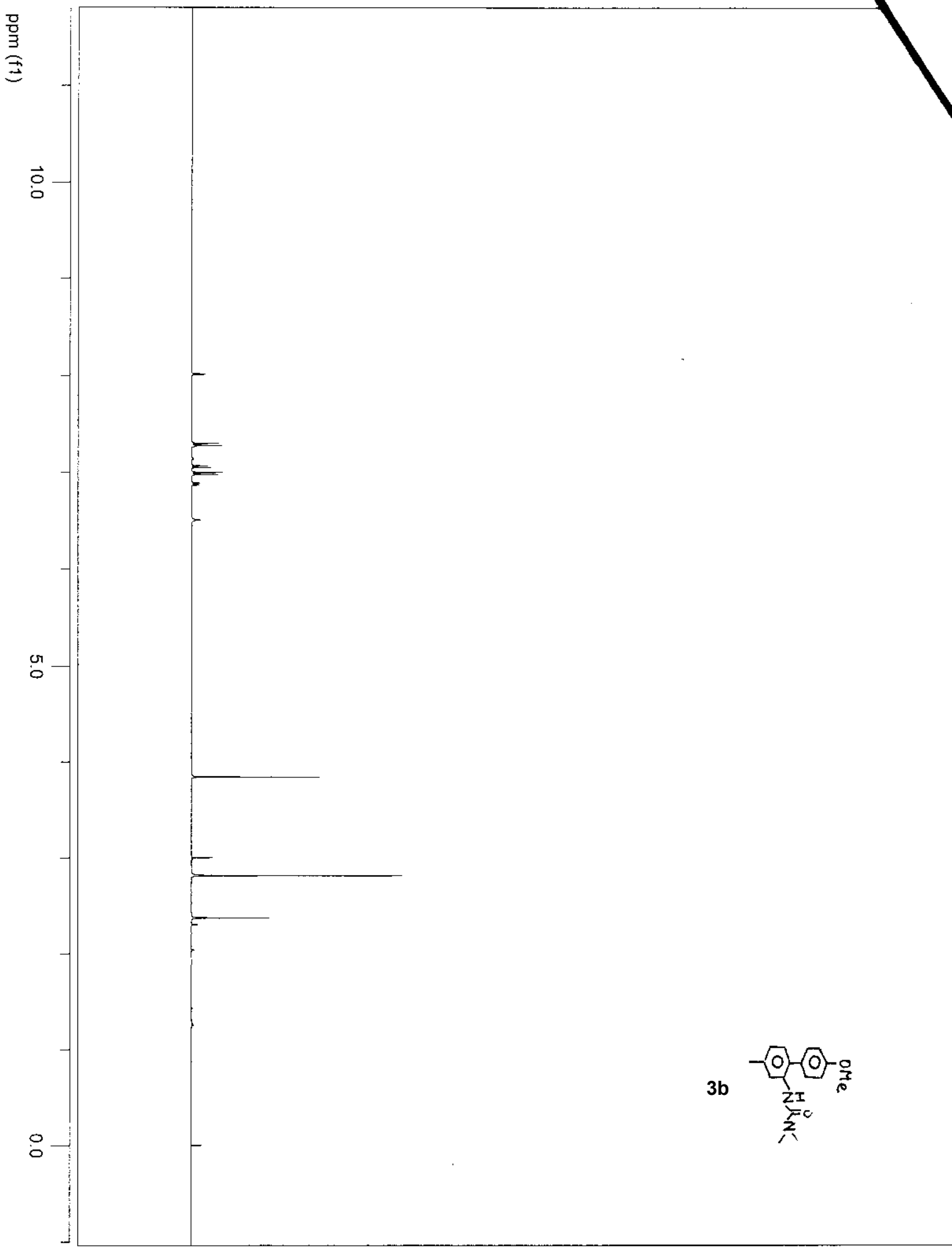
3a

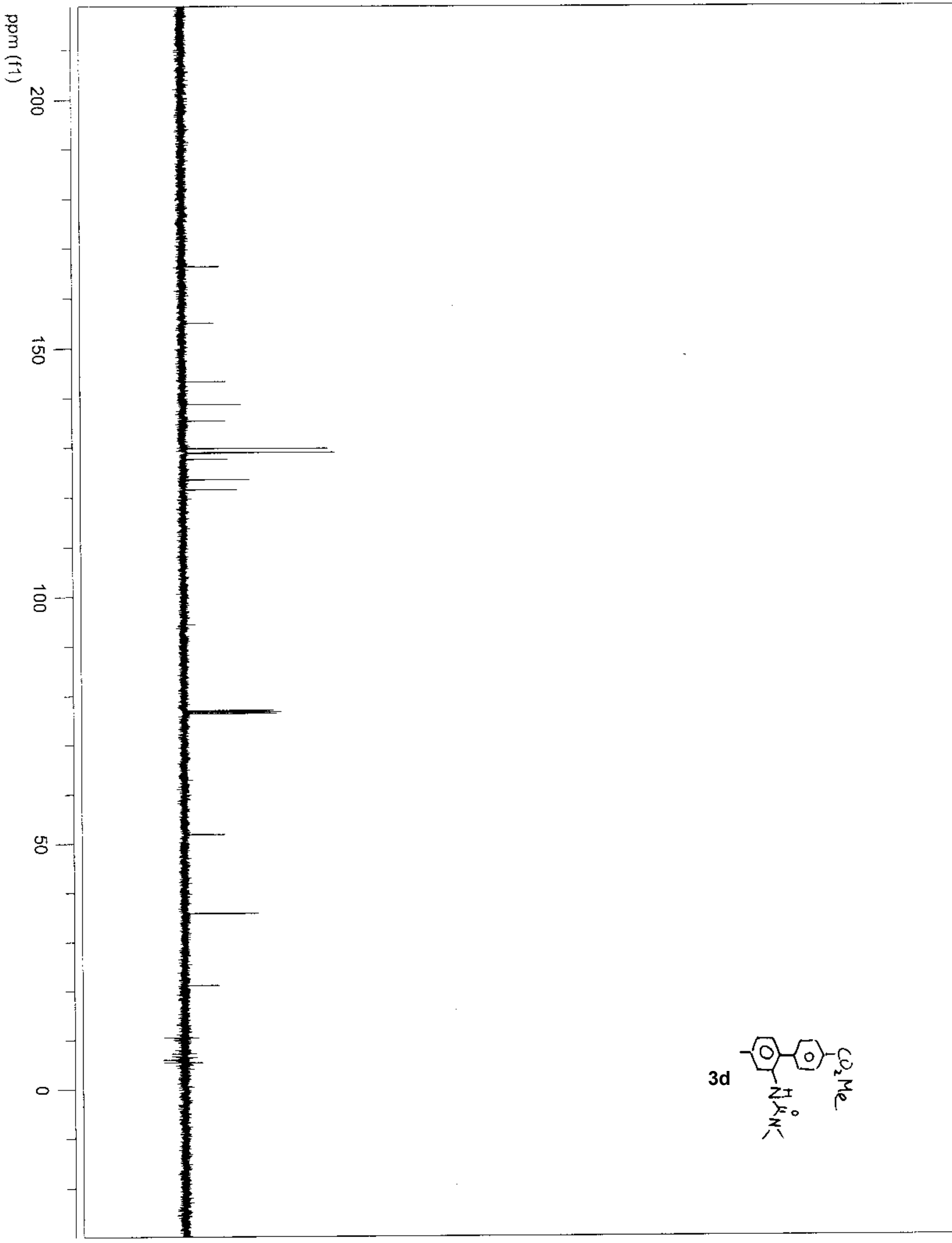


3a

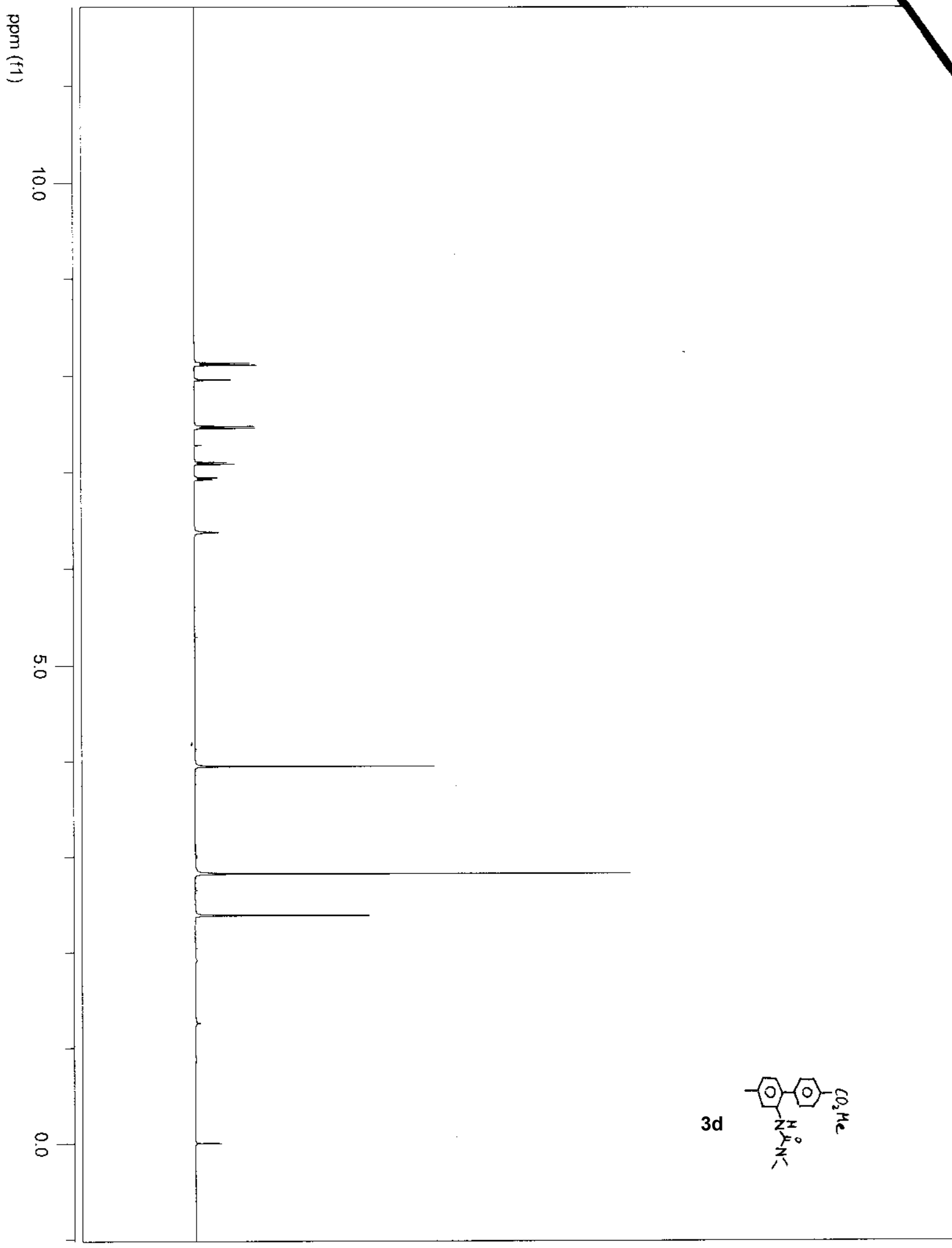


3b

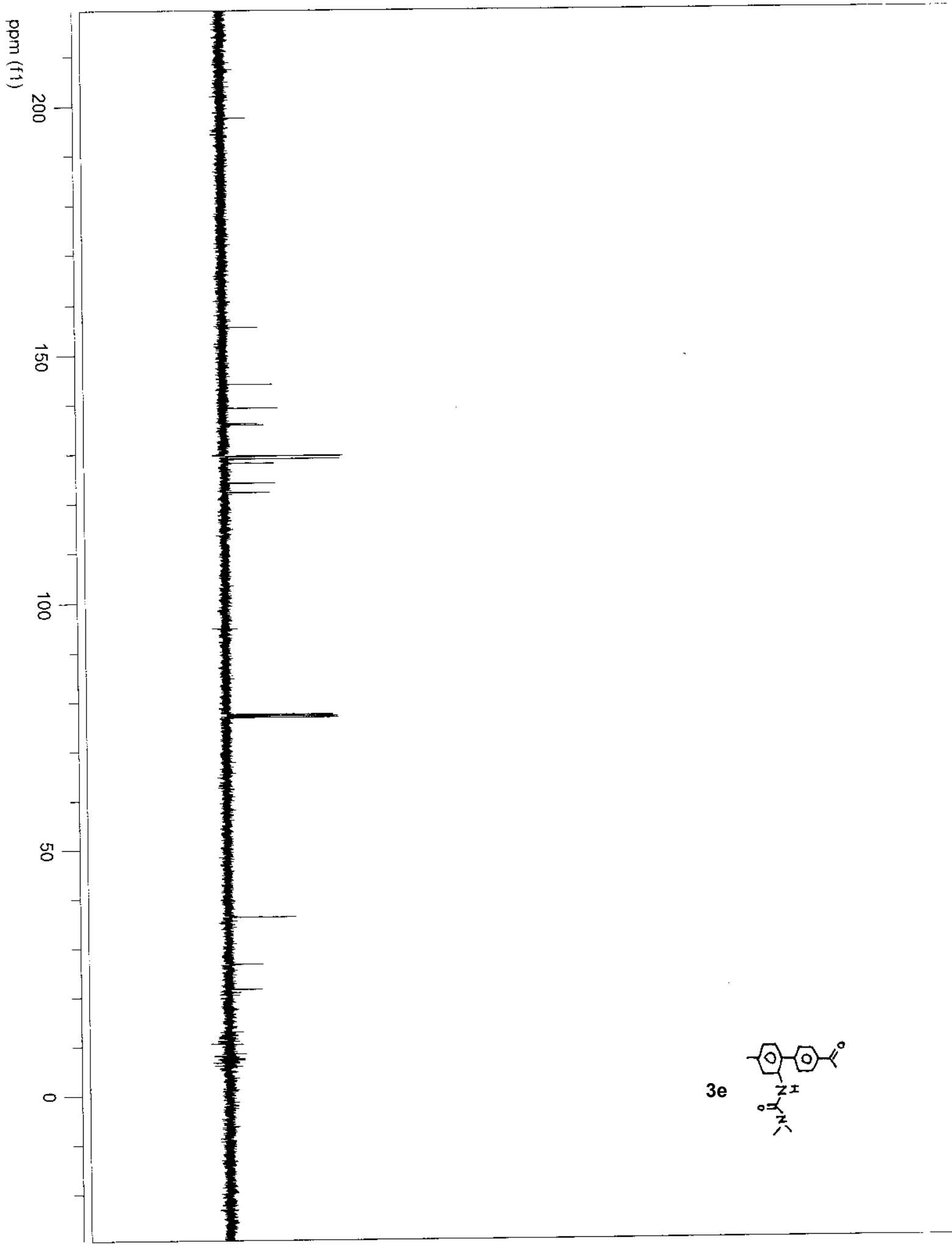

ppm (f1)
10.0
5.0
0.0
3b
OMe
H
N
O
N



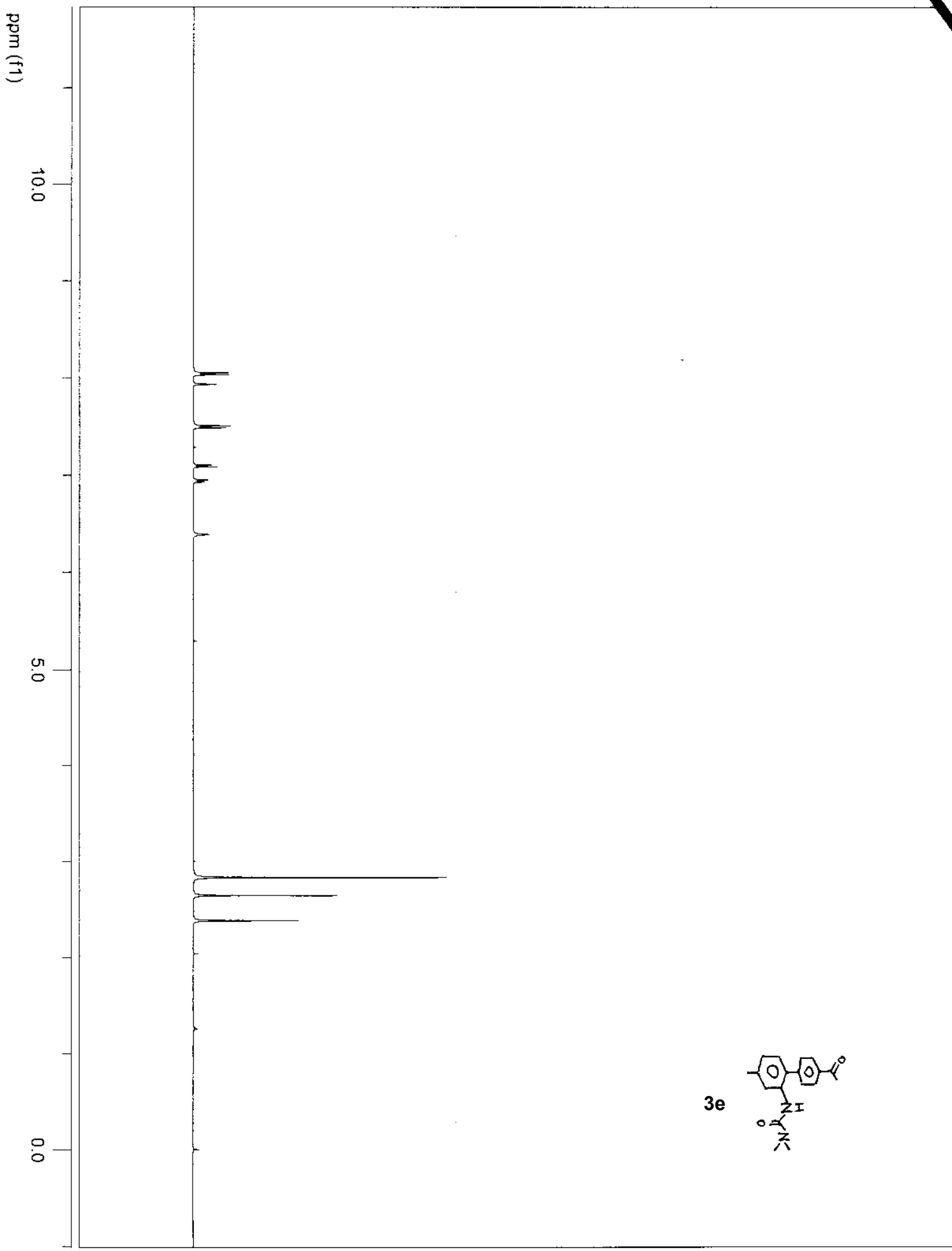
3d



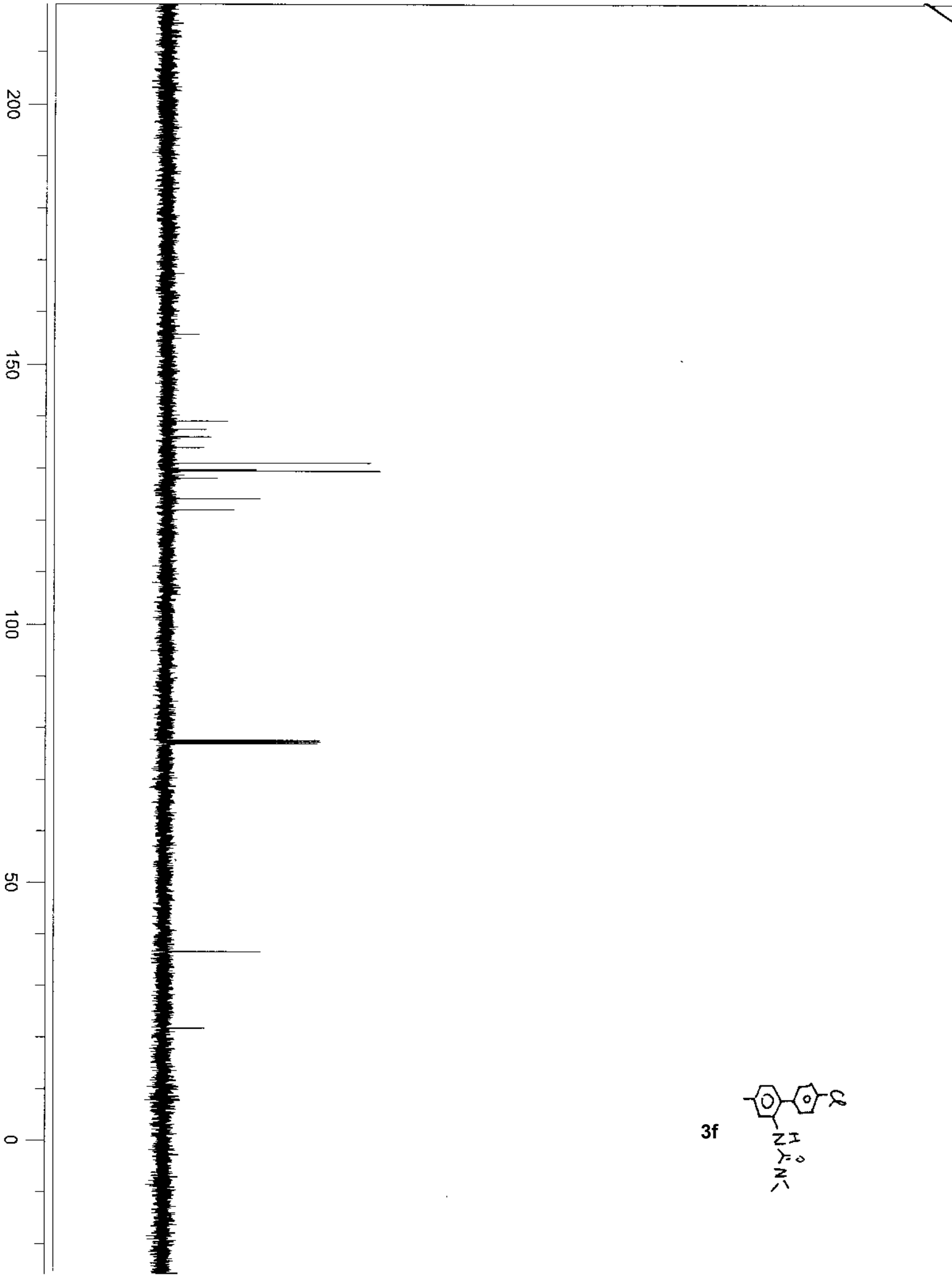
3d



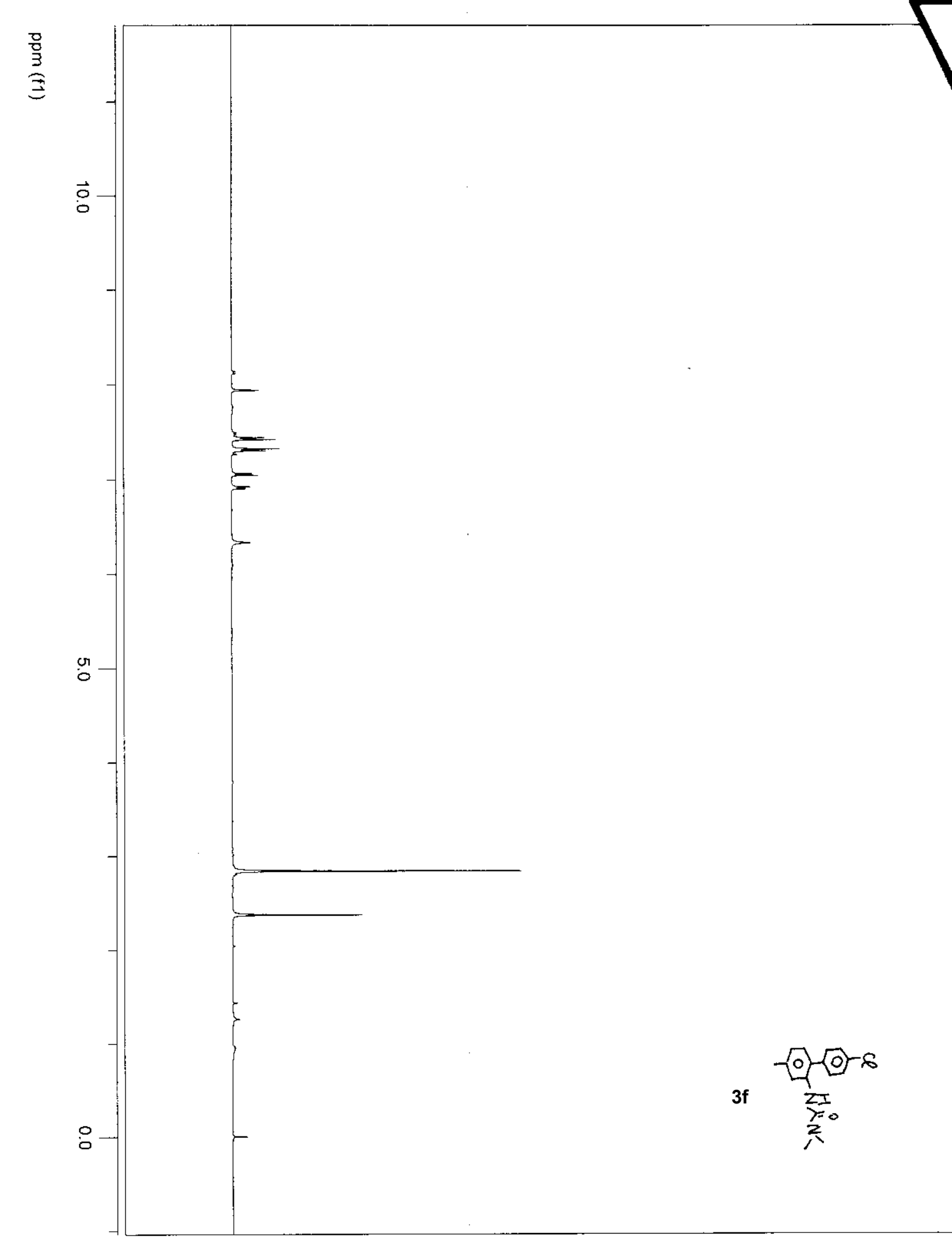
3e



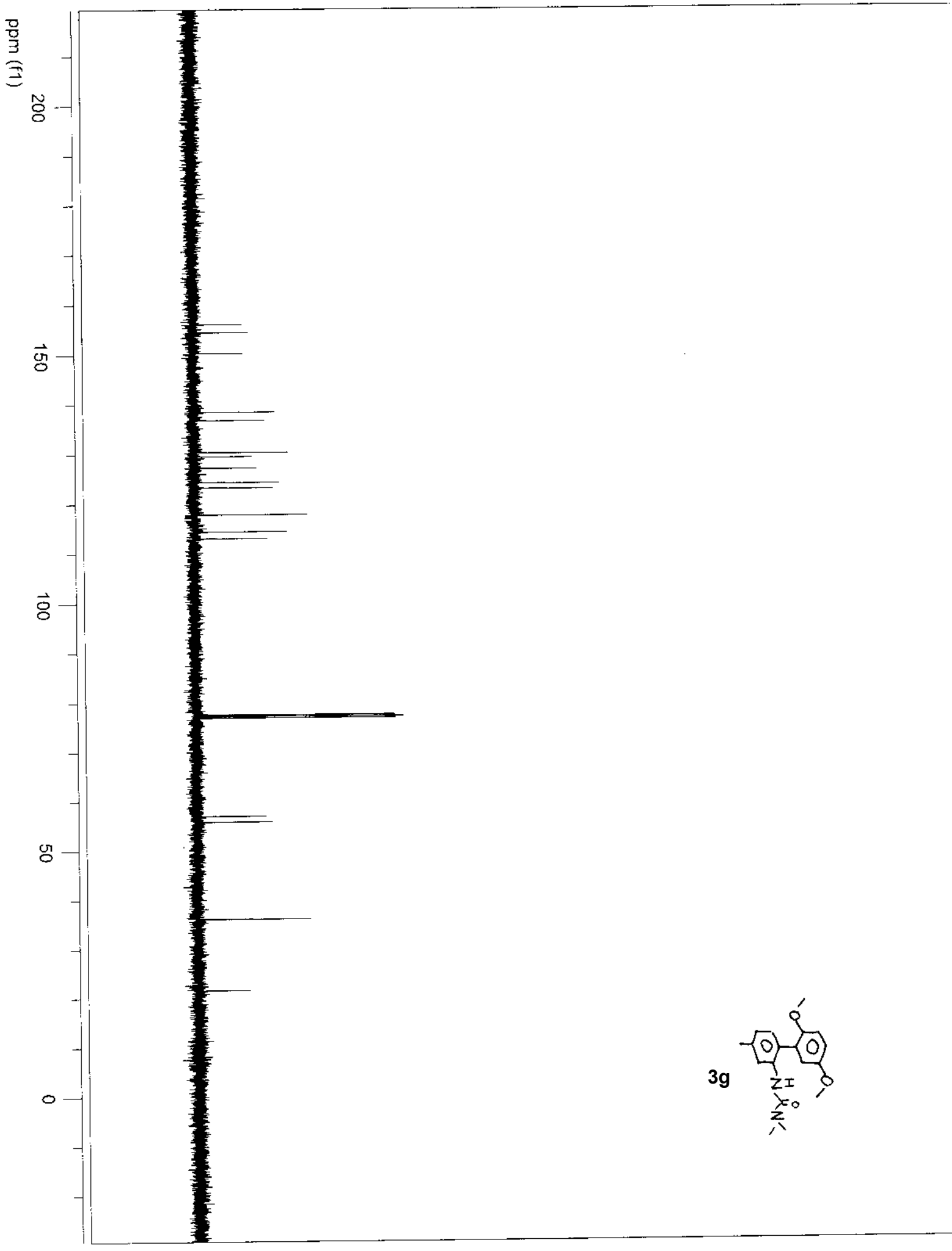
3e



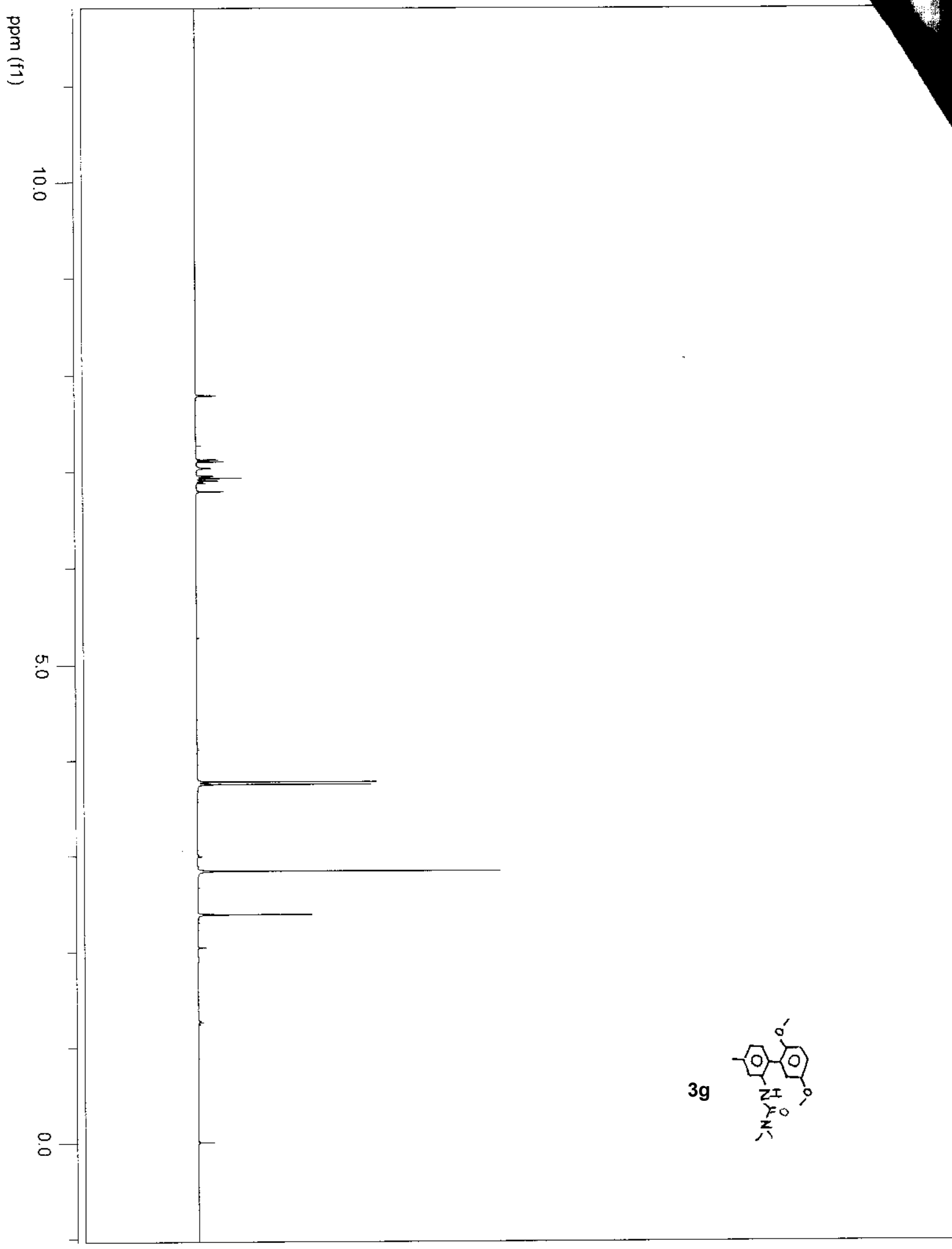
3f



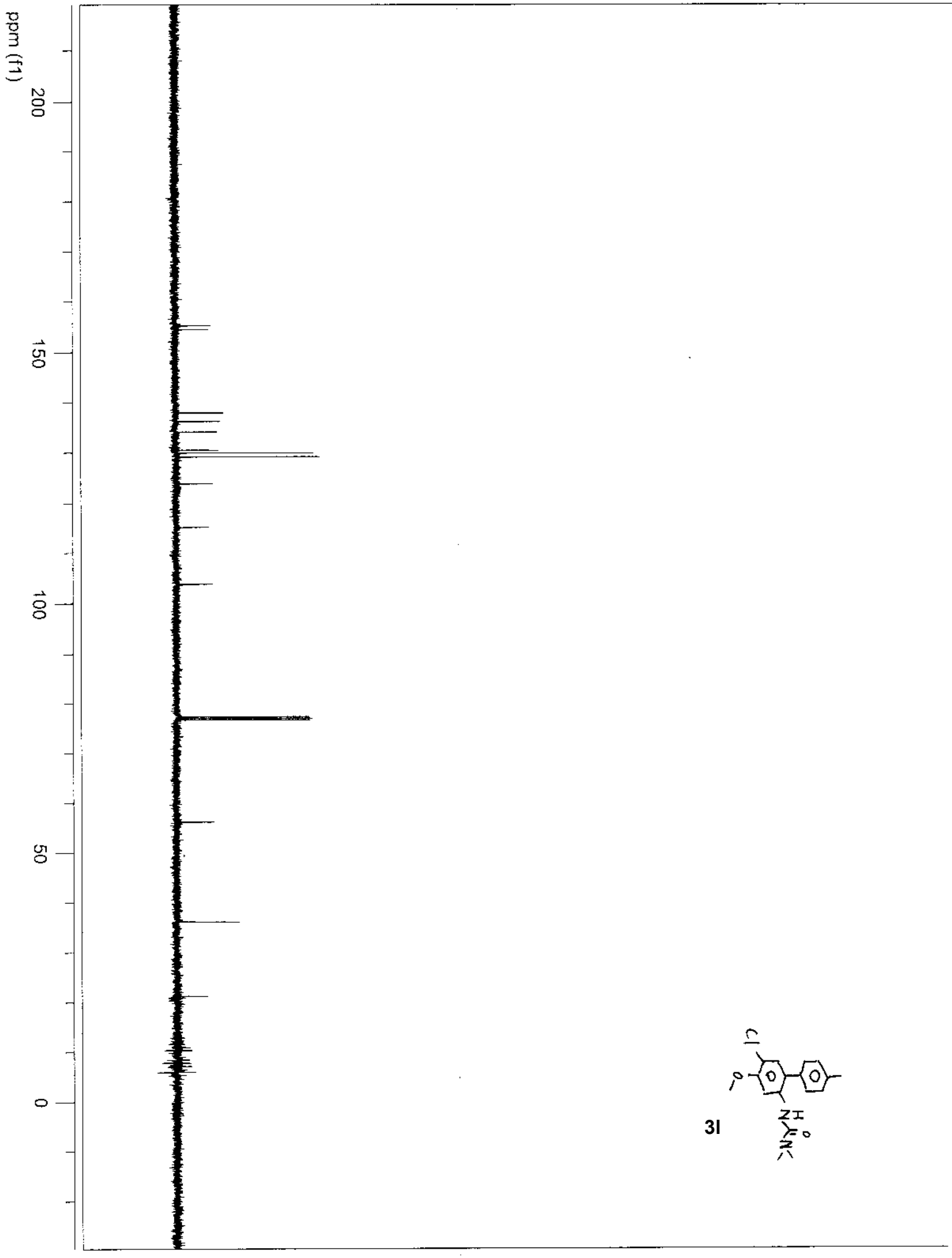
3f



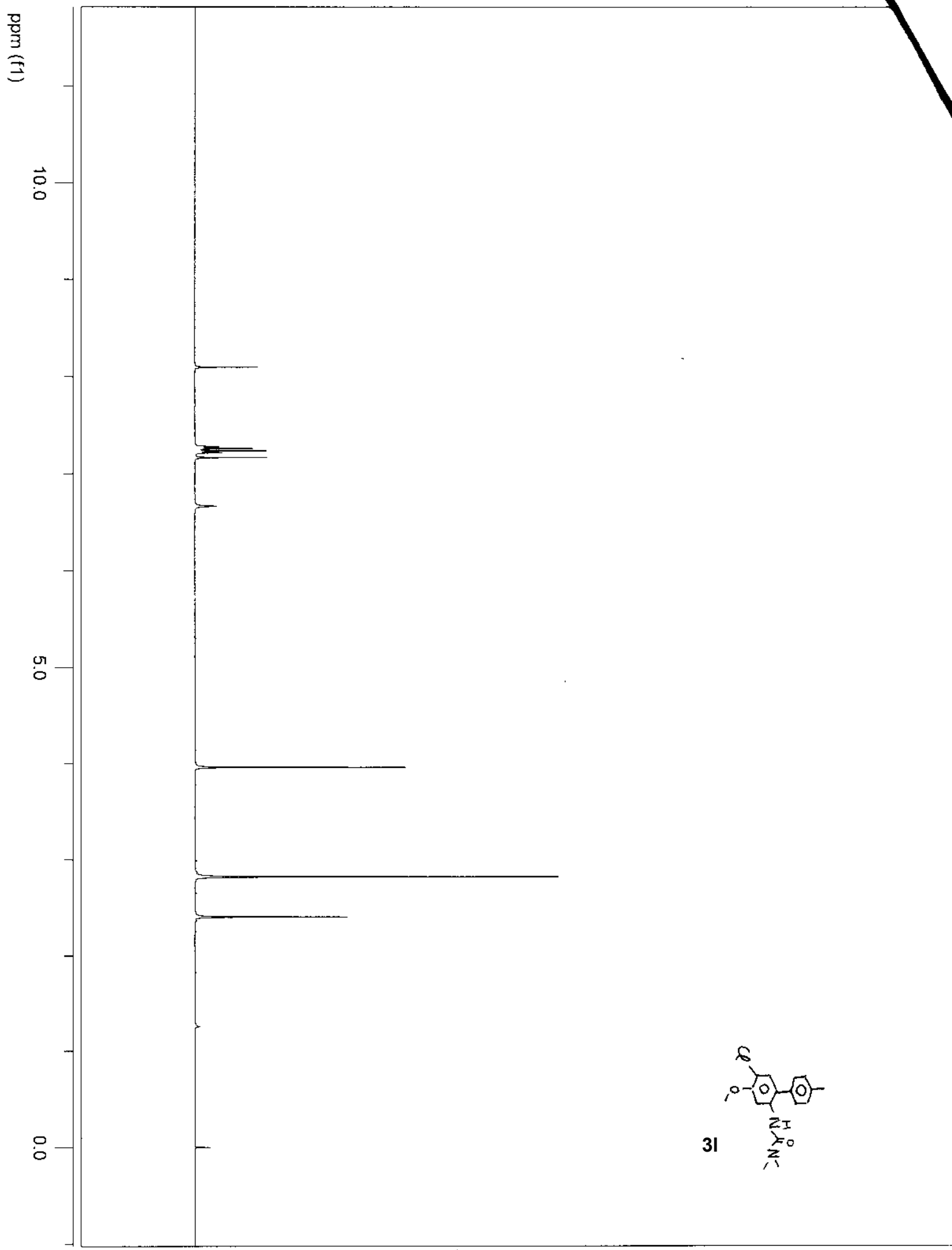
3g



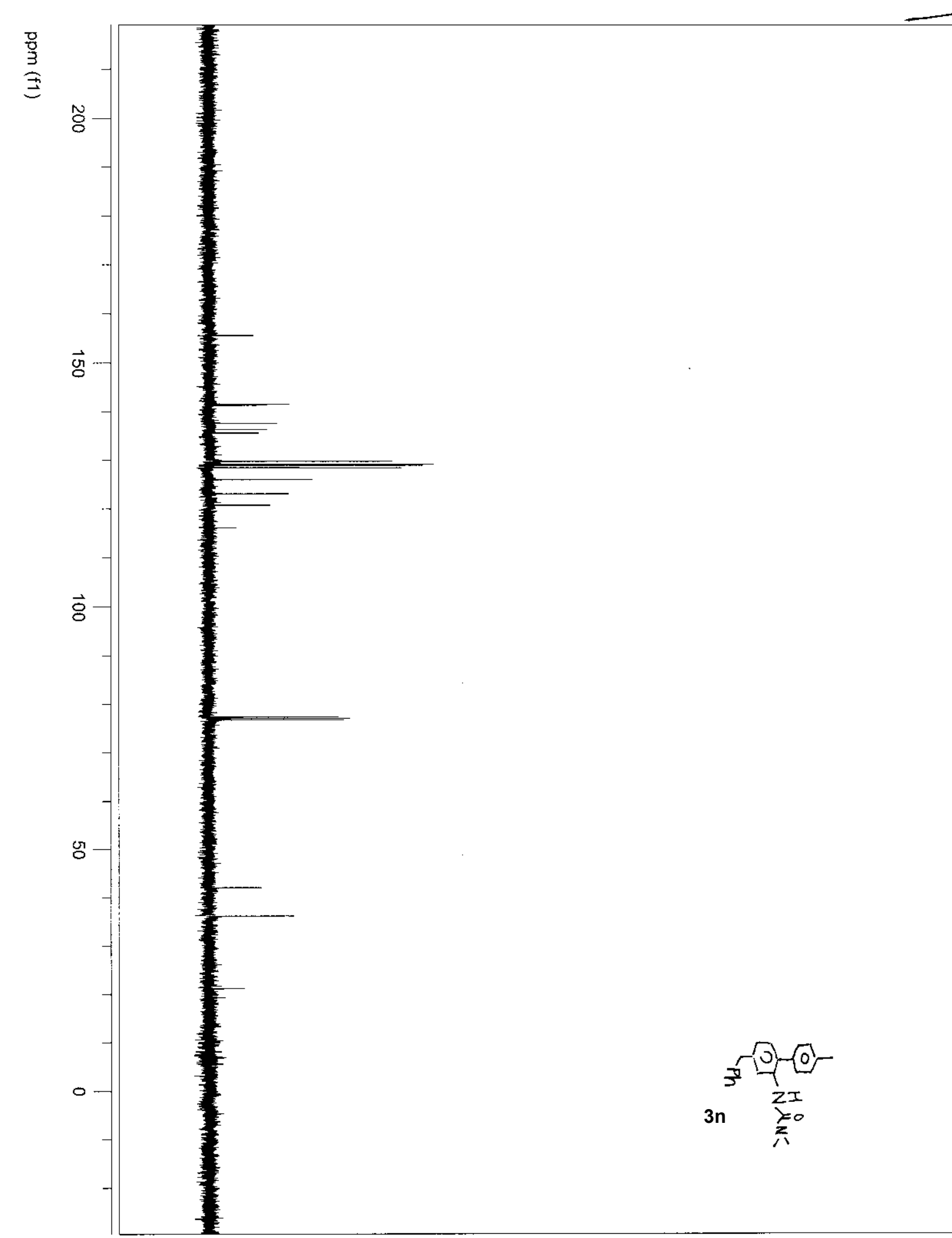
3g



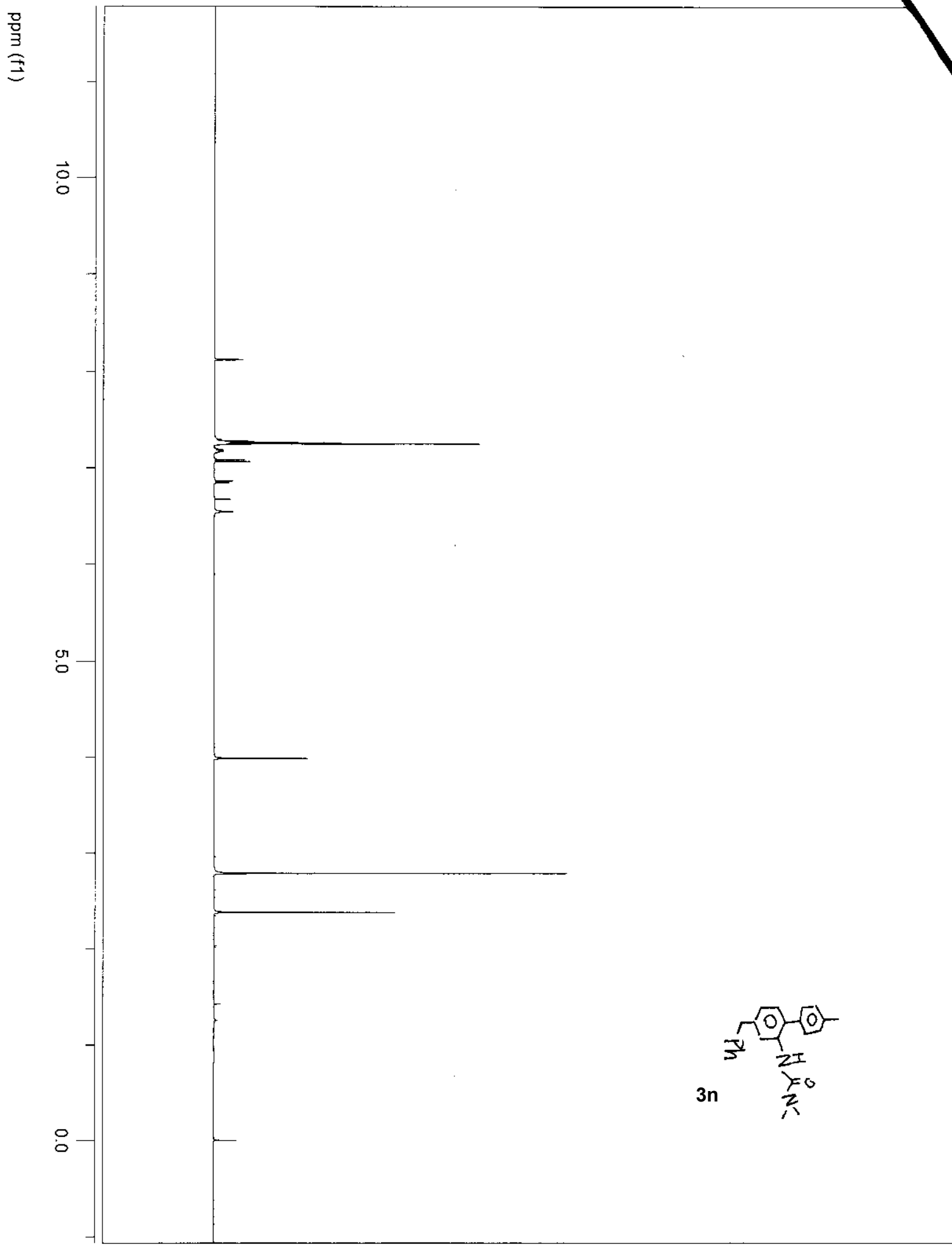
3l



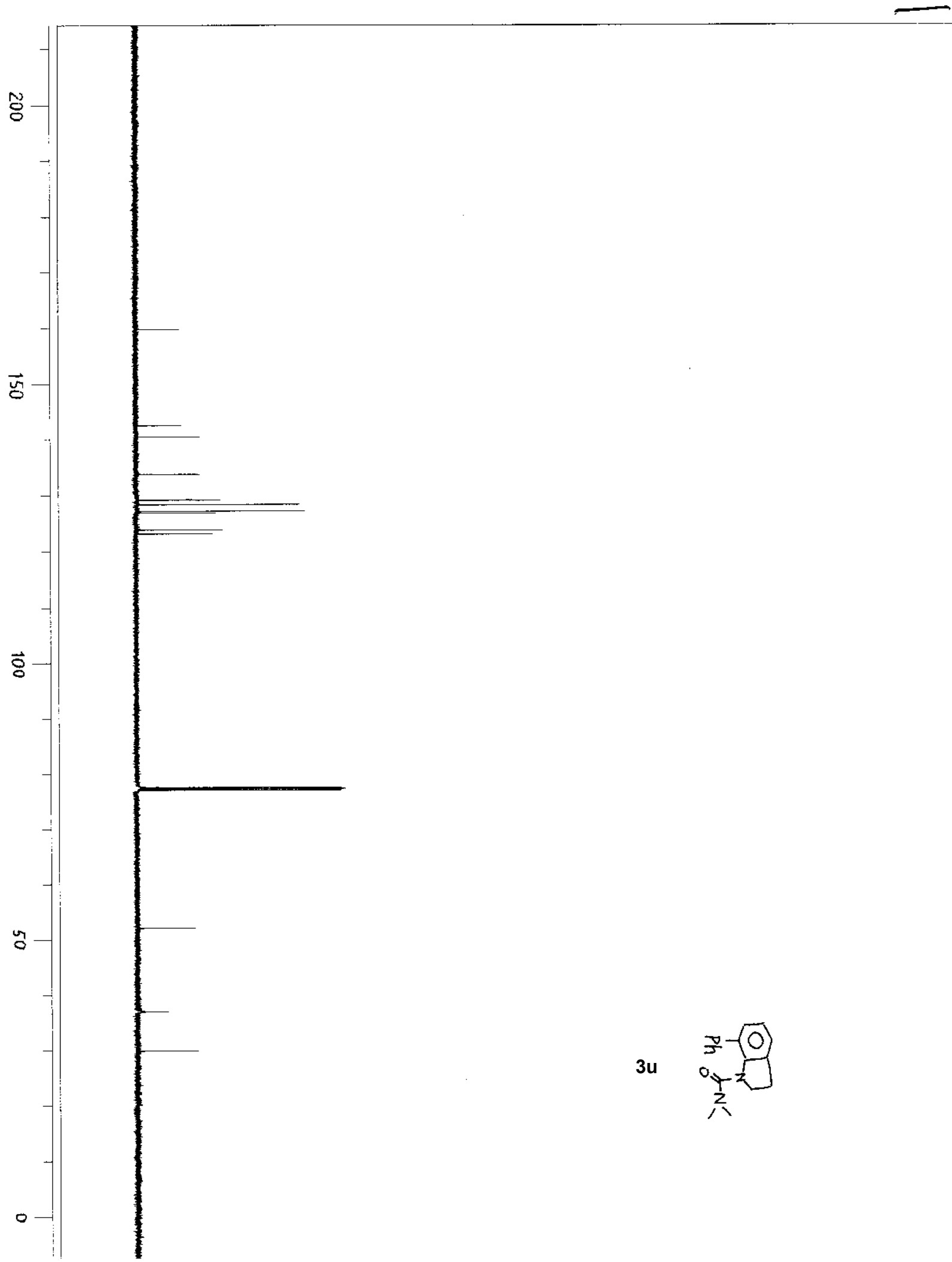
3l



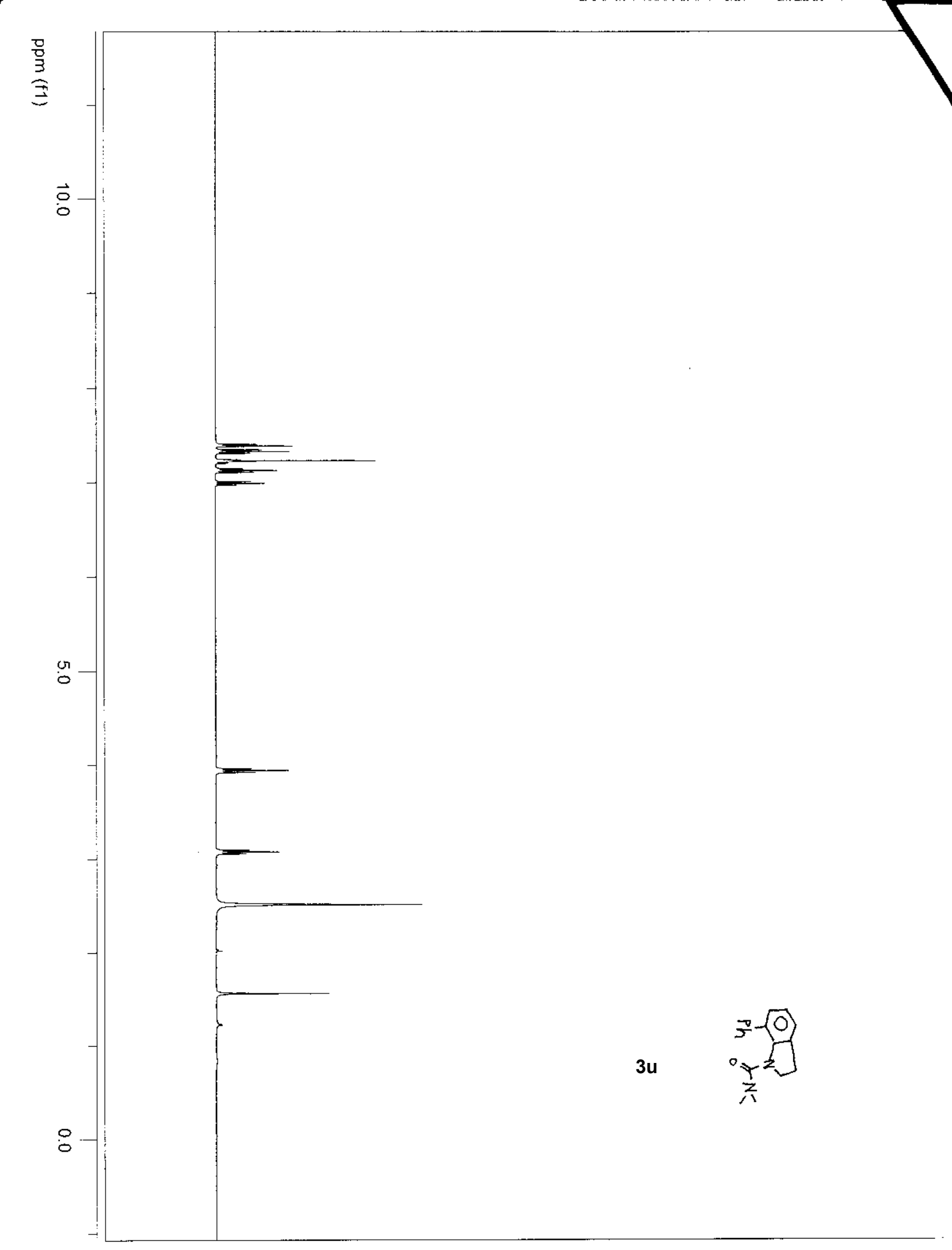
3n



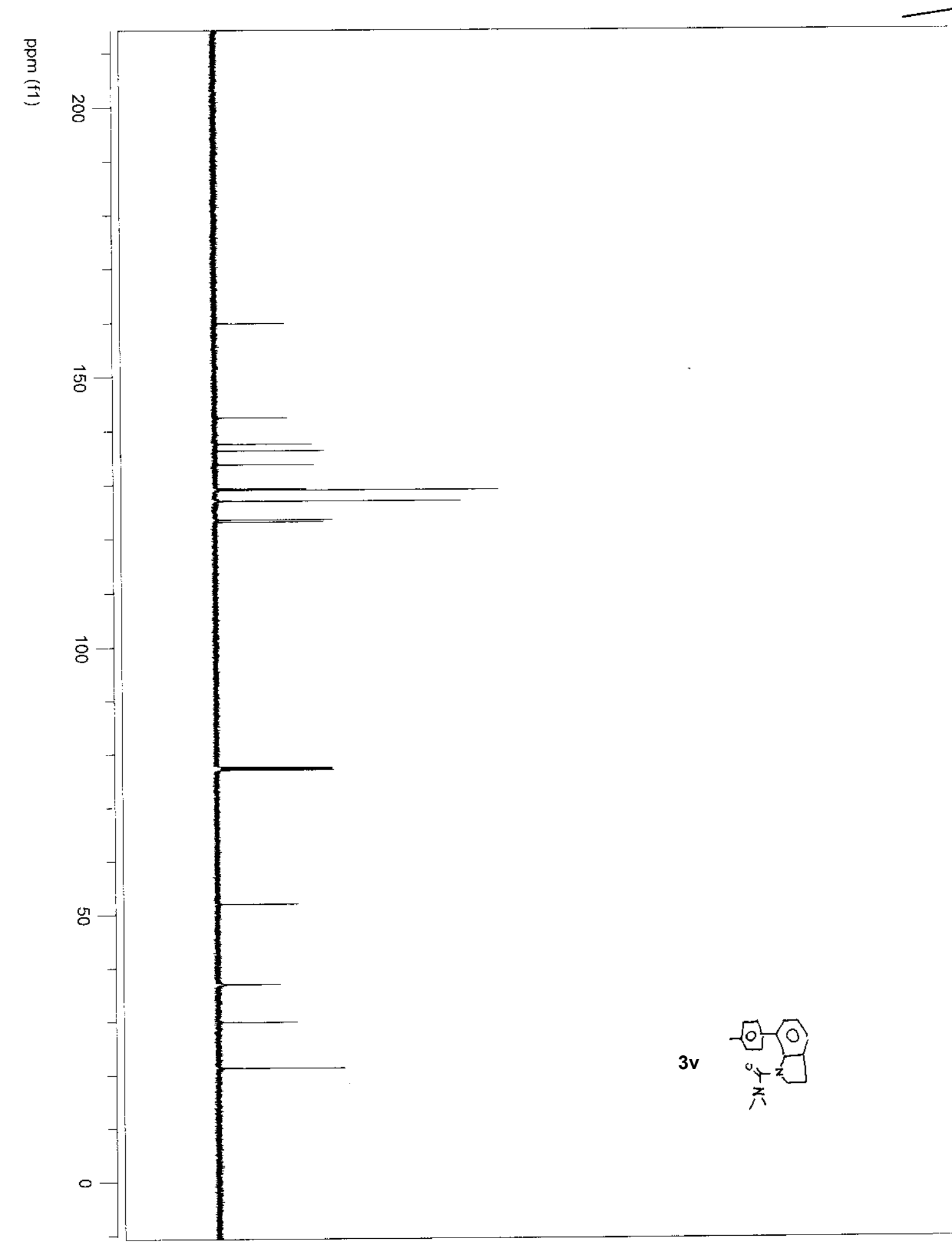
3n



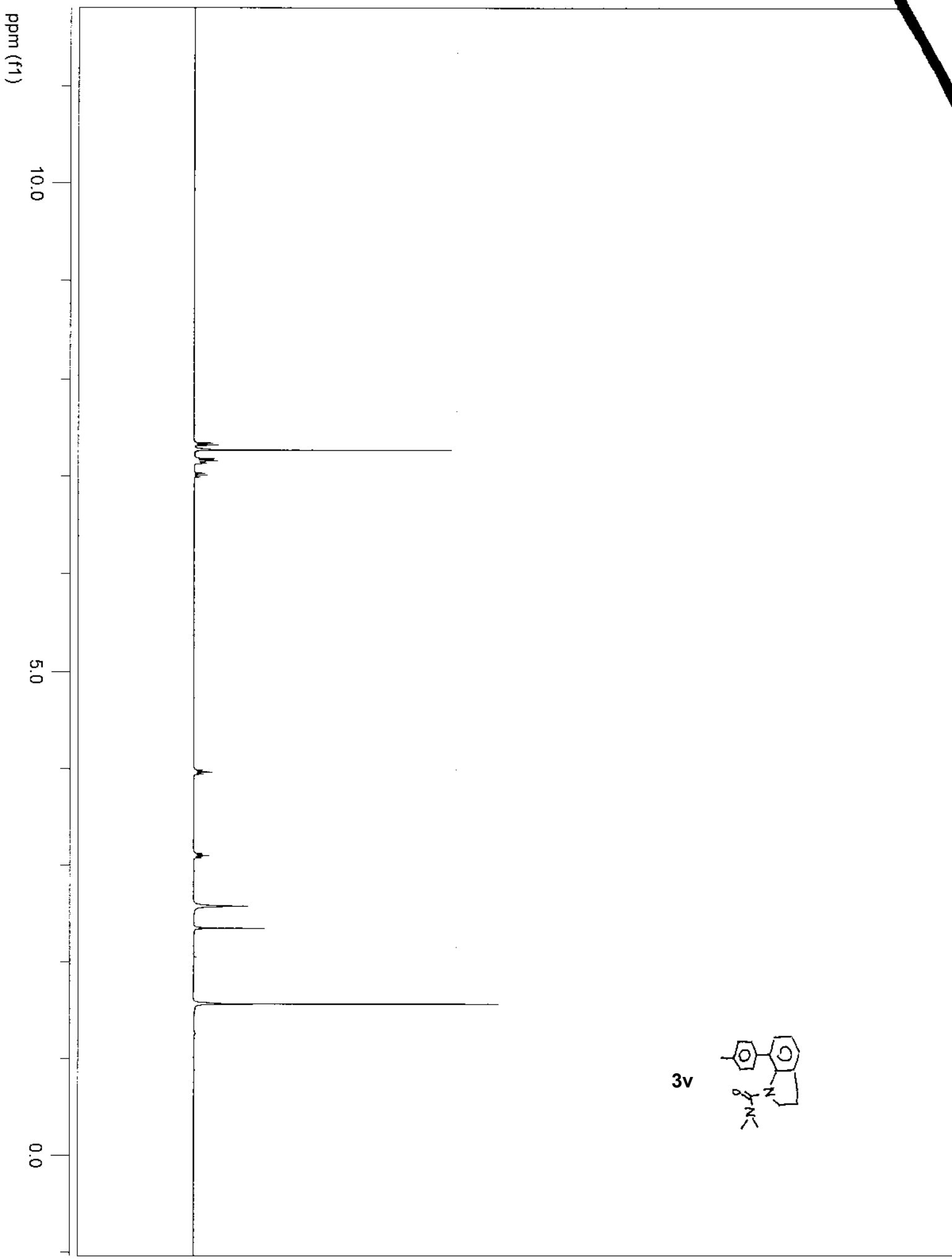
3u



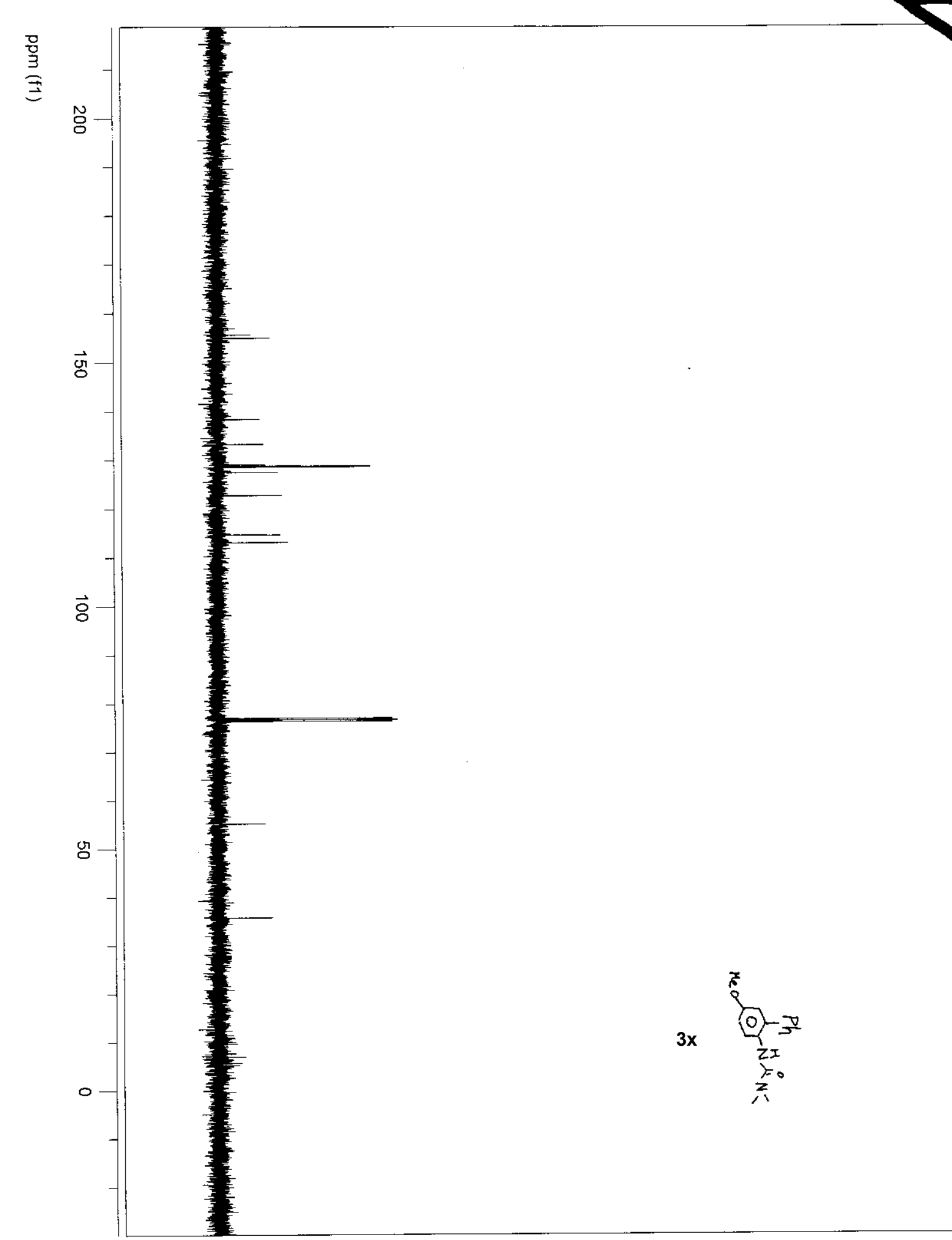
3u

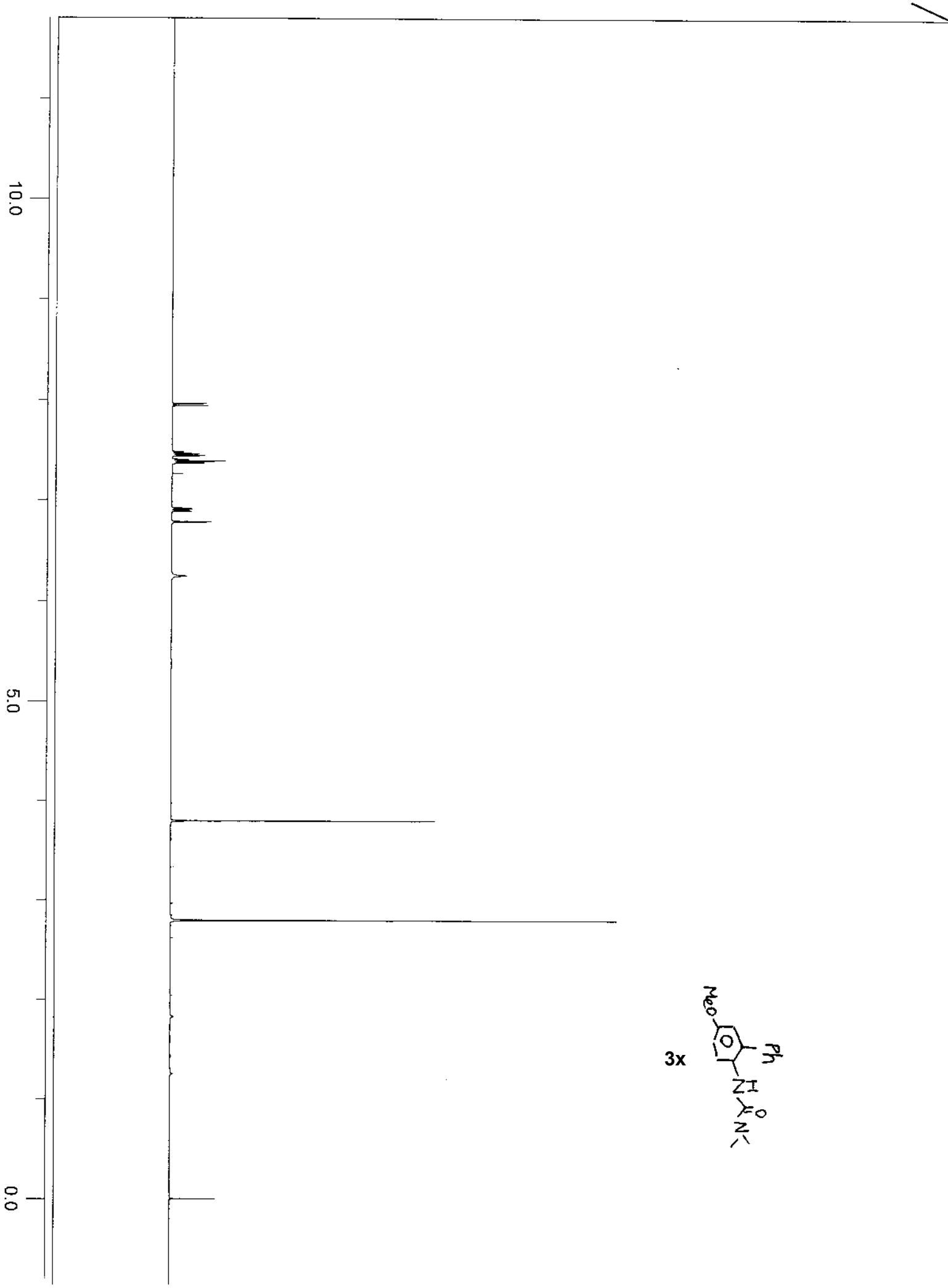


3v

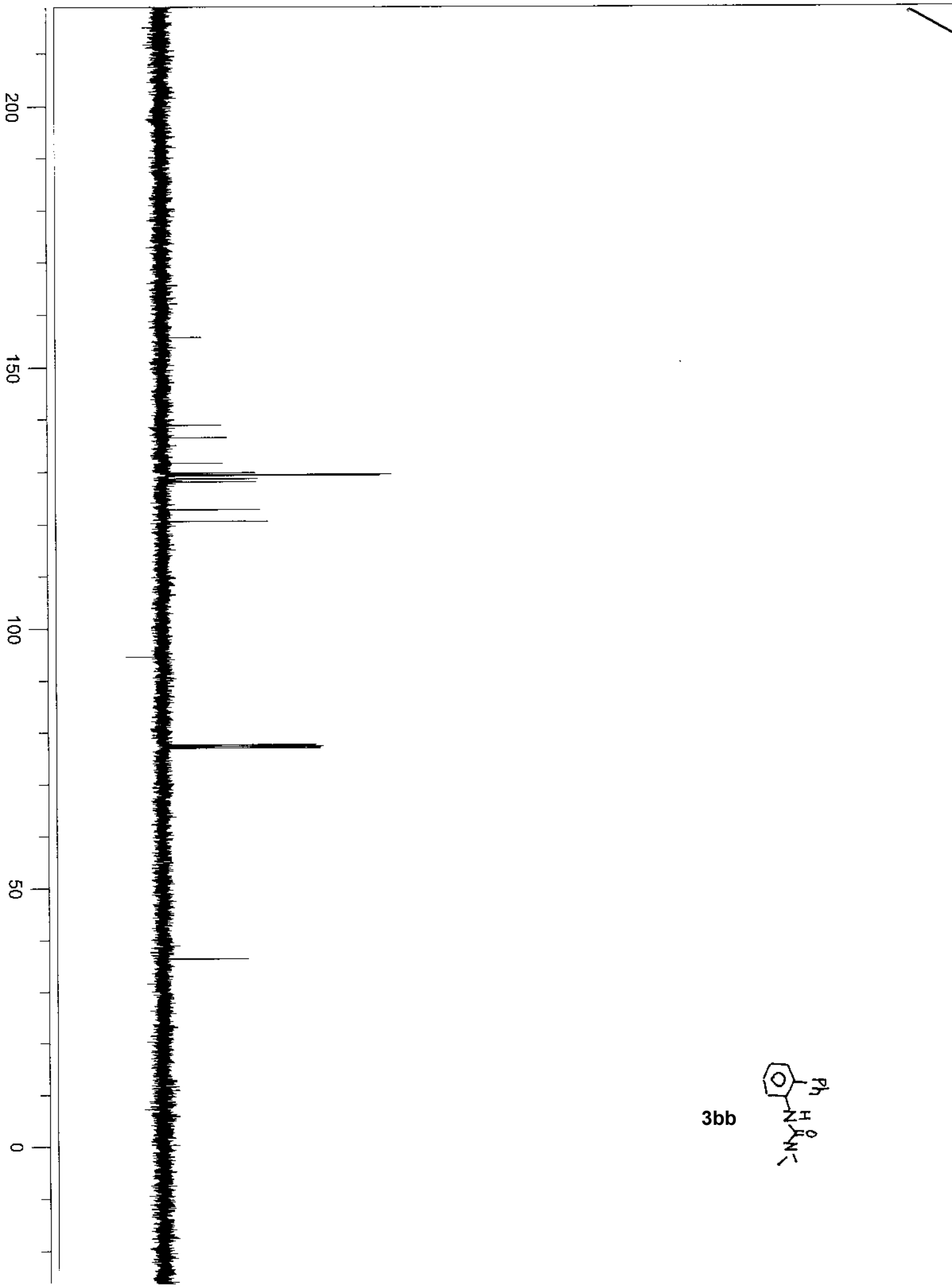


3v

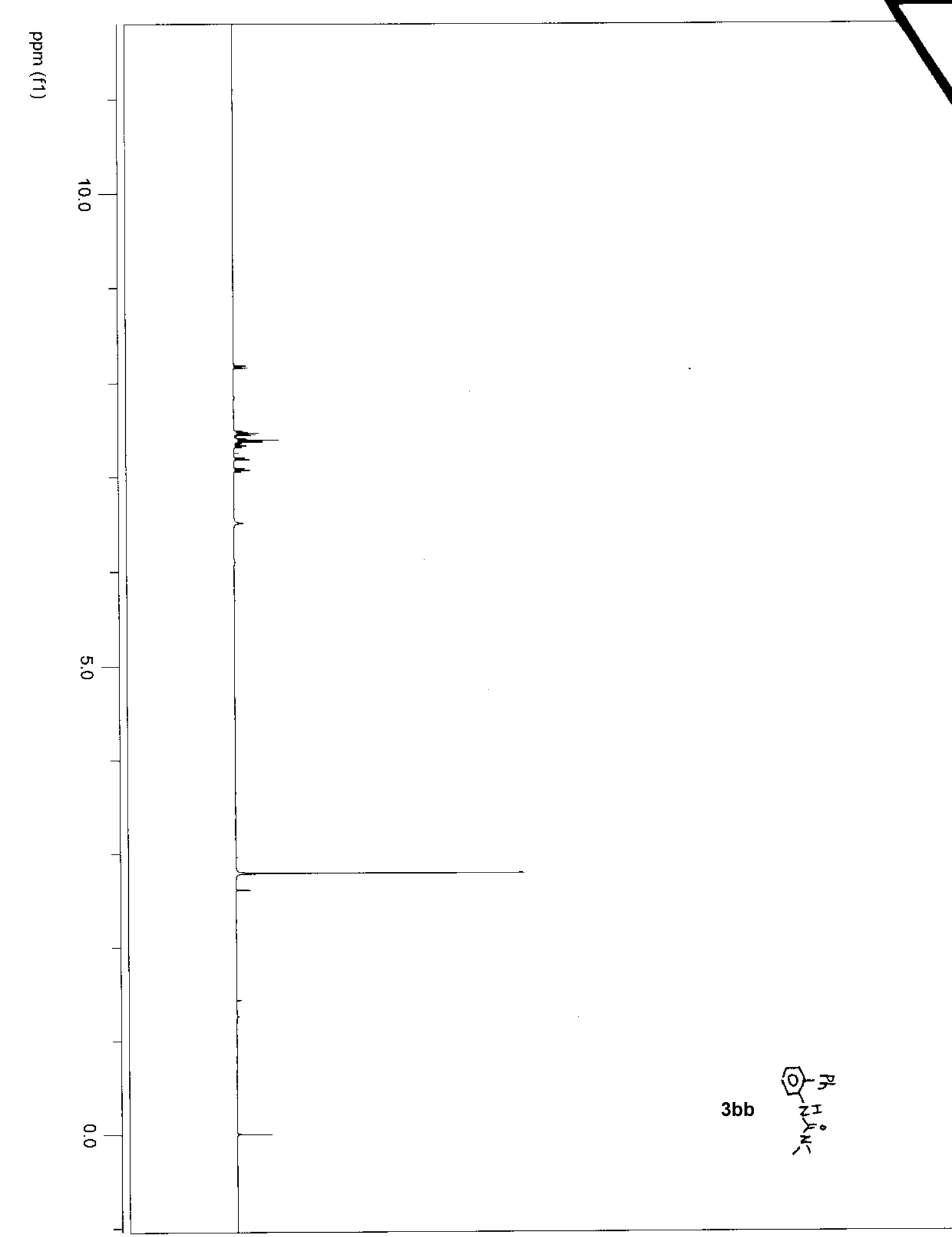

ppm (f1)
200
150
100
50
0
3x
Ph
MeO
H
N
O
N



3x



3bb



3bb