Cascade Annulation of Malonic Diamides: a Concise Synthesis of Polycyclic Pyrroloindolines

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Support Information

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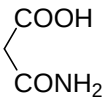
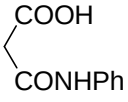
Experimental Section

General Methods. Tetrahydrofuran was freshly redistilled from sodium under Argon when using. Thin layer chromatography was performed on Merck 60 F254 pre-coated silica gel plates. Flash column chromatography was performed using normal phase silica gel (40-60 μm , 230-400 mesh, Silicycle P60). ^{1}H NMR or ^{13}C NMR spectra were recorded on Mercury 300 and MR 400. Proton chemical shifts were given in relative to tetramethylsilane (δ 0.00 ppm) in $CDCl_3$ or the undeuterated solvent residual signals. Carbon chemical shifts were internally referenced to the deuterated solvent signals. Infrared spectra were recorded using Nicolet 380FT-IR. Mass spectra were recorded on a Shimadzu LCMS-2010EV mass spectrometer (ESI). High resolution mass spectra were recorded on IonSpec 4.7 Tesla FTMS mass spectrometer (MALDI) and Bruker APEXIII 7.0 TESLA FTMS (ESI). Preparation of monomalonic amide was following the reported procedure. Relative configuration of **5d** and **5s** was eastablished by analysis of the crystallographic data of **5d** and **5s** and the relative configuration of **5o** was comfirmed by analyzing the NOESY spetrum of **5o**.



Prepared according to literature.[1]



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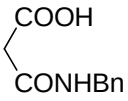
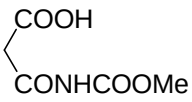
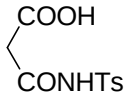


Prepared according to.literature.[3]



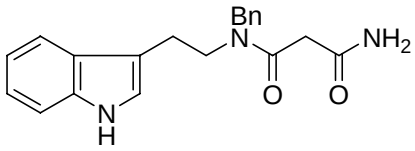
Prepared from benzyl hydrogen malonate according to literature.[4]



Prepared according to literature.[5]

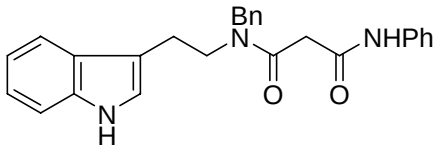
General Procedure for Preparation of malonic diamides of tryptamine derivatives. To a mixture of malonic monoamide (10 mmol, 1 eq)and N_b-benzyl tryptamine (12 mmol, 1.2 eq) in dichloromethane 150 ml was added diisopropylethylamine (4 mmol, 4 eq) and BOP-Cl (15 mmol, 1.5 eq). The solution was stirred at rt overnight, then washed with 0.1 M HCl 100 ml twice and brine, dried over Na_2SO_4, purified through silica gel column to give malonic diamides of tryptamine derivatives.

General procedure of oxidative-coupling reaction using I_2 as oxidant. The malonic diamide (0.2 mmol, 1 eq) was dissolved in anhydrous THF 2.9 ml with stirring under Argon, and cooled to -78^{o}C with dry ice-acetone bath. Then 1 M LiHMDS solution in THF (0.66 ml, 0.66 mmol, 3.3 eq) was added dropwise. After 30 min, the freshly prepared I_2 solution in THF (0.44 ml, 0.22 mmol, 1.1 eq, M = 0.5) was added dropwise. After 10 min, the cold bath was removed. the mixture was stirred at rt for 4 h, then quenched by saturated aqueous NH_4Cl, diluted with ethyl acetate, washed with 10% aqueous $Na_2S_2O_3$ and brine, dried over Na_2SO_4, purified through silica gel column to give product.

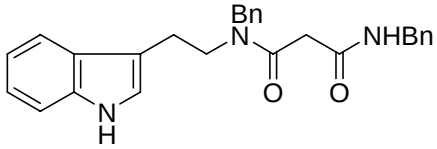


N^1-(2-(1H-indol-3-yl)ethyl)-N^1-benzylmalonamide 1a (mixture of rotamers and tautomers). R_f = 0.6 (dichloromethane:methanol 10:1). Yield 50%, white solid, mp 144-146^{o}C. ^{1}H NMR (300MHz, d_6-acetone) δ 2.99 (t, J = 8.1 Hz, 1H), 3.07 (t, J = 7.8 Hz, 1H), 3.35 (s, 1H), 3.38 (s, 1H), 3.62 (dd, J = 6.6 Hz, 15.3 Hz, 2H), 4.66 (s, 1H), 4.69 (s, 1H), 6.48 (brs, 0.5H), 6.96-7.39 (m, 9H), 7.58 (t, J = 9 Hz, 1H), 10.03 (brs, 0.3H), 10.11 (brs, 0.4H); ^{13}C NMR (100MHz, d_6-acetone) δ 169.17, 169.10, 167.94,

149.15, 138.48, 137.98, 136.64, 136.59, 129.05, 128.76, 127.87, 127.76, 127.52, 127.44, 127.41, 127.30, 123.60, 123.12, 121.45, 121.38, 118.83, 118.70, 118.66, 118.53, 111.88, 111.86, 111.81, 111.30, 98.71, 51.78, 48.79, 47.92, 46.95, 42.23, 41.79, 24.40, 23.48; IR (thin film): 3383, 3192, 2935, 1673, 1613, 1457, 1399, 1250, 1233, 1168, 954, 739, 702, 540, 427 cm^{-1}; ESI-MS m/z 336.3 $(M + H)^+$; HRMS (ESI) calcd. for $C_{20}H_{22}N_3O_2$ 336.1707 $(M + H)^+$, found 336.1698.

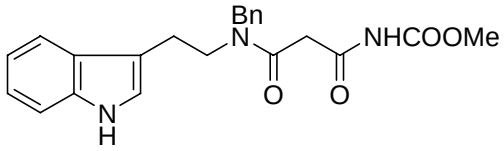


N^1-(2-(1H-indol-3-yl)ethyl)-N^1-benzyl-N^3-phenylmalonamide 1b (mixture of rotamers and tautomers). R_f = 0.3 (ethyl acetate-petroleum 1:1). Yield 84%, light yellow solid, mp 60-62°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.96 (t, *J* = 7.2 Hz, 1H), 3.04 (t, *J* = 7.5 Hz, 1H), 3.33 (s, 1H), 3.43 (s, 1H), 3.54 (t, *J* = 7.2 Hz, 1H), 3.72 (t, *J* = 7.5 Hz, 1H), 4.04 (s, 1H), 4.64 (s, 1H), 6.89 (dd, *J* = 1.5 Hz, 17.1 Hz, 1H), 7.04-7.31 (m, 11H), 7.44-7.60 (m, 3H), 8.27 (brs, 0.4H), 8.42 (brs, 0.5H), 9.98 (brs, 0.5H), 10.21 (brs, 0.4H); ^{13}C NMR (100MHz, $CDCl_3$) δ 169.16, 169.05, 164.38, 137.80, 137.74, 136.72, 136.29, 135.86, 129.07, 128.98, 128.94, 128.81, 127.97, 127.90, 127.72, 127.29, 126.91, 126.29, 124.38, 124.34, 122.47, 122.34, 122.14, 122.11, 120.19, 120.17, 119.70, 119.46, 118.57, 118.21, 112.56, 111.55, 111.52, 111.35, 52.36, 49.14, 48.51, 48.26, 40.50, 40.14, 24.57, 23.45; IR (thin film): 3303, 3058, 2925, 1626, 1548, 1497, 1443, 1338, 1233, 1159, 1080, 955, 743, 695, 504 cm^{-1}; ESI-MS m/z 412.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{26}H_{26}N_3O_2$ 412.2019 $(M + H)^+$, found 412.2014.

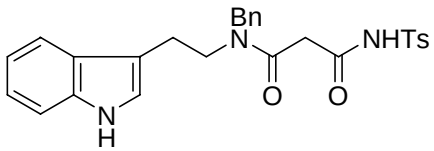


N^1-(2-(1H-indol-3-yl)ethyl)-N^1,N^3-dibenzylmalonamide 1c (mixture of rotamers and tautomers). R_f = 0.4 (ethyl acetate-petroleum 1:1). Yield 73%, white solid, mp 150-152°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.96-3.04 (m, 2H), 3.28 (s, 1H), 3.36 (s,

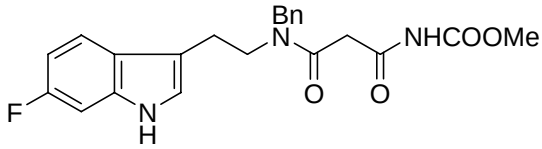
1H), 3.58 (t, *J* = 7.5 Hz, 1H), 3.70 (t, *J* = 7.5 Hz, 1H), 4.43-4.45 (m, 2H), 4.49 (d, *J* = 6 Hz, 1H), 4.61 (s, 1H), 6.94 (s, 1H), 7.06-7.14 (m, 2H), 7.16-7.20 (m, 2H), 7.26-7.35 (m, 9H), 7.51 (dd, *J* = 7.8 Hz, 21.6 Hz, 1H), 7.94 (brs, 0.5H), 8.04 (brs, 0.4H), 8.15 (brs, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 168.94, 168.85, 166.19, 166.16, 138.10, 136.86, 136.21, 136.00, 128.99, 128.72, 128.67, 128.64, 127.93, 127.81, 127.70, 127.67, 127.60, 127.38, 127.34, 127.25, 126.90, 122.35, 122.12, 121.99, 119.73, 118.60, 118.27, 112.75, 111.75, 111.39, 111.21, 52.37, 48.94, 48.01, 43.44, 40.20, 39.84, 24.56, 23.39; IR (thin film): 3305, 3058, 3029, 2922, 1725, 1632, 1496, 1453, 1339, 1233, 1203, 1061, 953, 815, 741, 698, 607, 422 cm^{-1}; ESI-MS m/z 426.3 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{27}H_{28}N_3O_2$ 426.2176 (M + H)$^+$, found 426.2186.



methyl 3-((2-(1H-indol-3-yl)ethyl)(benzyl)amino)-3-oxopropanoylcarbamate 1d (mixture of rotamers and tautomers). R_f = 0.3 (ethyl acetate-petroleum 2:1). Yield 57%, white solid, mp 66-68°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.97-3.06 (m, 2H), 3.50 (t, *J* = 6 Hz), 3.66-3.75 (m, 5H), 4.35 (s, 1H), 4.64 (s, 1H), 6.97 (d, *J* = 18 Hz), 7.10-7.17 (m, 3H), 7.24-7.36 (m, 5H), 7.49 (dd, *J* = 7.8, 27.6 Hz), 8.36 (brs, 0.4H), 8.52 (brs, 0.6H), 9.21 (brs, 0.4H), 9.35 (brs, 0.4H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.54, 167.47, 151.97, 151.88, 151.86, 136.81, 136.34, 135.89, 129.00, 128.68, 128.05, 127.86, 127.57, 127.28, 126.87, 126.50, 122.71, 122.66, 122.45, 122.41, 122.16, 122.13, 121.93, 119.54, 119.51, 119.29, 118.58, 118.07, 112.53, 112.46, 111.67, 111.36, 53.01, 52.93, 52.46, 48.78, 48.28, 47.94, 42.02, 41.54, 24.22, 23.31; IR (thin film): 3346, 2953, 1781, 1708, 1630, 1496, 1452, 1356, 1340, 1216, 1079, 1010, 934, 775, 742, 700, 606, 566, 508, 458, 426 cm^{-1}; ESI-MS m/z 394.3 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{22}H_{24}N_3O_4$ 394.1761 (M + H)$^+$, found 422.1750.

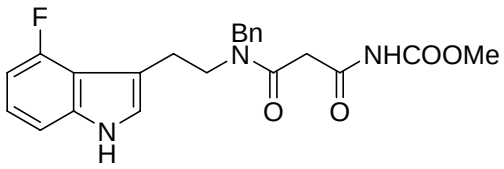


N^1-(2-(1H-indol-3-yl)ethyl)-N^1-benzyl-N^3-tosylmalonamide 1e (mixture of rotamers and tautomers). R_f = 0.3 (ethyl acetate-petroleum 5:1). Yield 48%, white solid, mp 78-80°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.38 (s, 3H), 2.90 (t, *J* = 6.9 Hz, 1H), 2.98-3.00 (m, 2H), 3.03 (s, 1H), 3.43 (t, *J* = 6.6 Hz, 1H), 3.68 (d, *J* = 6.9 Hz, 1H), 4.27 (s, 1H), 4.61 (s, 1H), 6.89-7.53 (m, 12H), 7.94 (dd, *J* = 8.1 Hz, 15.6 Hz, 2H), 8.24 (brs, 0.3H), 8.29 (brs, 0.5H), 11.25 (brs, 0.4H), 11.64 (brs, 0.3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.74, 167.72, 164.81, 164.69, 144.96, 144.90, 136.37, 136.31, 136.28, 135.86, 135.81, 135.38, 129.55, 129.51, 129.10, 128.82, 128.42, 128.38, 128.08, 127.99, 127.81, 127.24, 126.72, 126.27, 122.74, 122.45, 122.38, 122.06, 119.71, 119.42, 118.44, 117.95, 112.16, 111.79, 111.50, 111.12, 52.18, 49.06, 48.38, 48.20, 39.86, 39.44, 24.15, 23.30, 21.67; IR (thin film): 3403, 3059, 2922, 1717, 1624, 1425, 1347, 1169, 1088, 944, 865, 814, 743, 703, 665, 547, 426 cm^{-1}; ESI-MS m/z 490.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{27}H_{28}N_3O_4S$ 490.1795 $(M + H)^+$, found 490.1799.

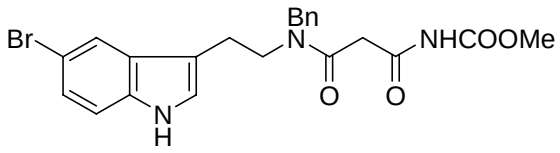


methyl 3-(benzyl(2-(6-fluoro-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1f (mixture of rotamers and tautomers). R_f = 0.7 (ethyl acetate-petroleum 3:1). Yield 85%, brown solid, mp 62-64°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.95-3.03 (q, *J* = 6.9 Hz, 2H), 3.44 (s, 1H), 3.51 (t, *J* = 6.6 Hz, 1H), 3.64-3.77 (m, 5H), 4.37 (s, 1H), 4.65 (s, 1H), 6.85 (q, *J* = 8.4 Hz, 1H), 6.97 (d, *J* = 8.7 Hz, 1H), 7.04 (t, *J* = 9 Hz, 1H), 7.11 (d, *J* = 6.9 Hz, 1H), 7.23-7.46 (m, 5H), 8.46 (brs, 0.4H), 8.77 (brs, 0.5H), 9.24 (brs, 0.6H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.58, 167.45, 161.17, 158.81, 151.76, 136.67, 136.39, 136.27, 135.78, 129.02, 128.90, 128.70, 128.23, 128.01, 127.91, 127.63, 126.48, 126.24, 123.89, 123.52, 122.60, 119.37, 119.26, 118.81, 118.71,

112.65, 111.54, 108.43, 108.18, 108.15, 107.90, 98.09, 97.84, 97.69, 97.43, 53.03, 52.93, 52.45, 48.75, 48.29, 47.78, 42.04, 41.34, 24.11, 23.27; IR (thin film): 3334, 2955, 1771, 1708, 1627, 1496, 1453, 1284, 1221, 1081, 1062, 951, 803, 766, 624, 597, 563, 520 cm^{-1}; ESI-MS m/z 412.3 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{22}H_{23}N_3O_4F$ 412.1667 $(M + H)^+$, found 412.1665.

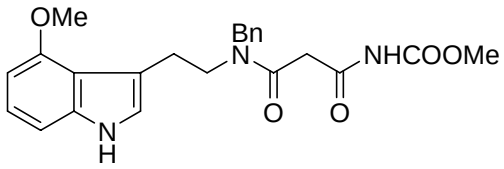


methyl 3-(benzyl(2-(4-fluoro-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1g (mixture of rotamers and tautomers). R_f = 0.2 (ethyl acetate-petroleum 2:1). Yield 60%, white solid, mp 73-75°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 3.03-3.14 (m, 2H), 3.52 (t, J = 6.9 Hz), 3.62-3.72 (m, 6H), 4.37, 4.62 (2s, 2H), 6.65-6.75 (m, 1H), 6.90 (s, 0.6H), 7.02-7.13 (m, 3H), 7.24-7.29 (m, 4H), 8.85 (brs, 0.3H), 9.01 (brs, 0.6H), 9.39 (brs, 0.7H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.60, 158.12, 155.69, 151.96, 139.40, 139.34, 139.28, 139.22, 136.92, 135.96, 129.14, 128.95, 128.80, 128.61, 128.24, 128.01, 127.91, 127.79, 127.48, 126.44, 126.24, 123.31, 123.13, 122.54, 122.46, 122.28, 122.21, 116.02, 115.82, 115.77, 115.57, 110.89, 109.80, 108.02, 107.95, 107.93, 107.68, 107.66, 104.49, 104.30, 104.12, 53.01, 52.95, 52.38, 52.33, 49.33, 48.95, 48.69, 41.99, 41.51, 25.54, 24.41; IR (thin film): 3328, 2954, 1780, 1708, 1629, 1581, 1505, 1451, 1349, 1224, 1079, 1034, 935, 838, 778, 735, 699, 589, 482 cm^{-1}; ESI-MS m/z 412.3 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{22}H_{23}N_3O_4F$ 412.1667 $(M + H)^+$, found 412.1672.

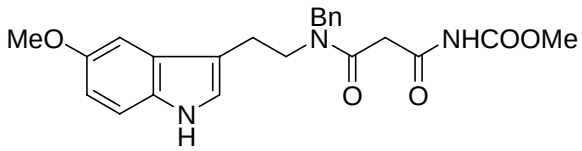


methyl 3-(benzyl(2-(5-bromo-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1h (mixture of rotamers and tautomers). R_f = 0.4 (ethyl acetate-petroleum 3:1). Yield 53%, white solid, mp 72-74°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.96 (dd, J =

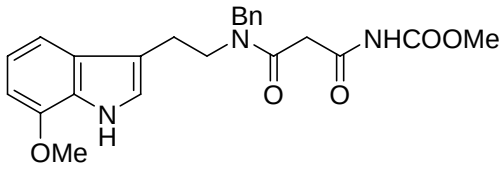
8.4 Hz, 15.3 Hz, 2H), 3.46-3.51 (m, 2H), 3.65 (t, J = 7.2 Hz, 1H), 3.72-3.78 (m, 4H), 4.37 (s, 1H), 4.64 (s, 1H), 7.00 (d, J = 17.4 Hz), 7.13 (d, J = 6.9 Hz, 1H), 7.23-7.35 (m, 6H), 7.58 (d, J = 17.7 Hz, 1H), 8.41 (brs, 0.5H), 8.63 (brs, 0.5H), 9.15 (brs, 0.7H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.62, 167.52, 152.12, 152.02, 136.64, 135.71, 135.03, 134.97, 129.07, 129.03, 128.77, 128.65, 128.03, 127.68, 126.69, 124.78, 124.57, 124.27, 123.97, 121.00, 120.62, 113.35, 113.01, 112.62, 112.41, 111.95, 110.86, 53.07, 53.01, 52.54, 48.81, 47.59, 42.24, 41.74, 24.01, 23.07; IR (thin film): 3330, 2952, 1779, 1707, 1629, 1451, 1215, 1079, 883, 798, 775, 737, 701, 605, 423 cm^{-1}; ESI-MS m/z 472.4 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{22}H_{23}N_3O_4Br$ 472.0867 (M + H)$^+$, found 472.0878.



methyl 3-(benzyl(2-(4-methoxy-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1i (mixture of rotamers and tautomers). R_f = 0.7 (ethyl acetate-petroleum 2:1). Yield 49%, pink solid, mp 72-74^{o}C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.88, 2.95 (2s, 1H), 3.08 (t, J = 7.2 Hz, 1.4H), 3.15 (t, J = 7.2 Hz, 0.5H), 3.56 (t, J = 7.5 Hz, 2H), 3.64 (s, 1H), 3.76, 3.78(2s, 4H), 3.92 (s, 2H), 4.41 (s, 0.5H), 4.63 (s, 1.5H), 6.42-6.51 (m, 1H), 6.82 (s, 0.7H), 6.91-6.97 (m, 1H), 7.08 (t, J = 7.8 Hz, 1.4H), 7.22-7.31 (m, 5H), 8.20 (brs, 0.2H), 8.27 (brs, 0.6H), 9.67 (brs, 0.6H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.64, 167.50, 166.88, 162.67, 154.40, 154.20, 151.84, 151.76, 138.23, 138.19, 137.02, 136.21, 128.91, 128.58, 127.91, 127.65, 127.42, 126.22, 122.90, 122.63, 121.73, 117.11, 116.84, 112.73, 111.65, 104.99, 104.82, 99.39, 99.18, 55.13, 54.92, 52.93, 52.87, 52.11, 49.95, 48.98, 41.68, 40.95, 36.53, 31.49, 26.10, 24.98; IR (thin film): 3342, 2953, 2837, 1782, 1708, 1631, 1508, 1451, 1361, 1255, 1217, 1081, 734 cm^{-1}; ESI-MS m/z 424.2 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{23}H_{26}N_3O_5$ 424.1867 (M + H)$^+$, found 424.1861.

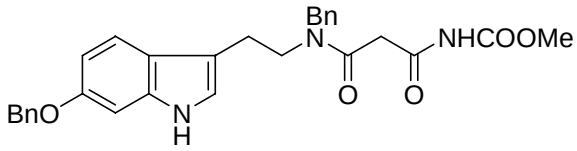


methyl 3-(benzyl(2-(5-methoxy-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1j (mixture of rotamers and tautomers). R_f = 0.6 (ethyl acetate-petroleum 3:1). Yield 70%, white solid, mp 68-70°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.95-3.04 (m, 2H), 3.50-3.54 (m, 2H), 3.67-3.84 (m, 8H), 4.38 (s, 1H), 4.66 (s, 1H), 6.83-6.95 (m, 2H), 7.01 (s, 1H), 7.12 (d, J = 7.2 Hz, 1H), 7.23-7.33 (m, 5H), 8.08 (brs, 0.4H), 8.22 (brs, 0.4H), 9.14 (brs, 0.5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 154.10, 153.92, 151.90, 151.80, 136.79, 135.88, 131.47, 128.98, 128.87, 128.67, 128.03, 127.87, 127.64, 127.58, 127.22, 126.50, 123.36, 123.13, 112.41, 112.30, 112.16, 112.04, 111.19, 100.44, 99.94, 55.93, 53.01, 52.92, 52.45, 48.70, 48.07, 47.62, 41.95, 41.94, 41.46, 24.25, 23.35; IR (thin film): 3346, 2951, 1781, 1761, 1708, 1628, 1486, 1452, 1215, 1070, 924, 799, 775, 738, 702, 633, 569 cm^{-1}; ESI-MS m/z 424.3 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{23}H_{26}N_3O_5$ 424.1867 (M + H)$^+$, found 424.1883.

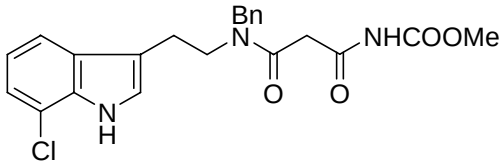


methyl 3-(benzyl(2-(7-methoxy-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1k (mixture of rotamers and tautomers). R_f = 0.5 (ethyl acetate-petroleum 2:1). Yield 68%, pink solid, mp 70-72°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.97-3.06 (m, 2H), 3.49-3.54 (m, 2H), 3.66-3.72 (m, 1.6H), 3.76, 3.79 (2s, 3H), 3.94 (s, 3H), 4.35 (s, 1H), 4.63 (s, 1H), 6.63-6.66 (m, 1H), 6.94-7.33 (m, 8H), 8.29 (brs, 0.4H), 8.37 (brs, 0.5H), 9.16 (brs, 0.6H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.40, 151.87, 146.32, 146.19, 136.89, 135.97, 128.96, 128.69, 128.64, 128.27, 128.09, 127.81, 127.52, 126.81, 126.75, 126.49, 122.07, 121.92, 120.14, 119.82, 113.15, 112.06, 111.46, 110.93, 102.12, 101.91, 55.31, 52.97, 52.91, 52.47, 48.80, 48.26, 48.03, 41.62, 24.44, 23.45; IR (thin film): 3307, 2953, 1785, 1760, 1708, 1628, 1578, 1497, 1450, 1261, 1215, 1080, 1055, 781, 732, 701, 600 cm^{-1}; ESI-MS m/z 424.3 (M + H)$^+$; HRMS

(MALDI) calcd. for $C_{23}H_{26}N_3O_5$ 424.1867 $(M + H)^+$, found 424.1874.

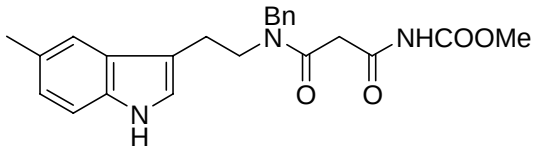


methyl 3-(benzyl(2-(6-(benzyloxy)-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1l (mixture of rotamers and tautomers). R_f = 0.7 (ethyl acetate-petroleum 2:1). Yield 58%, light yellow solid, mp 66-68°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.94-3.03 (m, 2H), 3.46-3.53 (m, 2H), 3.67-3.71 (m, 2H), 3.74 (s, 2H), 3.71 (s, 1H), 4.37 (s, 1H), 4.64 (s, 1H), 5.09 (d, J = 2.4 Hz, 2H), 6.86-6.91 (m, 3H), 7.11 (d, J = 6.6 Hz, 1H), 7.23-7.47 (m, 10H), 7.98 (brs, 0.3H), 8.13 (brs, 0.5H), 9.15 (brs, 0.5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.61, 167.45, 155.68, 155.55, 151.86, 137.47, 137.39, 137.09, 137.01, 136.81, 135.92, 128.99, 128.68, 128.53, 128.05, 127.84, 127.53, 126.53, 122.02, 121.63, 121.57, 121.36, 119.91, 118.69, 112.44, 111.39, 110.32, 109.99, 96.45, 70.60, 53.01, 52.92, 52.45, 48.74, 48.31, 47.92, 42.04, 41.47, 24.25, 23.41; IR (thin film): 3346, 2952, 1781, 1760, 1708, 1628, 1497, 1452, 1215, 1164, 1080, 802, 737, 698 cm^{-1}; ESI-MS m/z 500.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{29}H_{30}N_3O_5$ 500.2180 $(M + H)^+$, found 500.2170.

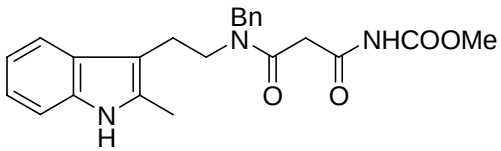


methyl 3-(benzyl(2-(7-chloro-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1m (mixture of rotamers and tautomers). R_f = 0.6 (ethyl acetate-petroleum 2:1). Yield 86%, grey solid, mp 60-62°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 3.02 (dd, J = 8.4 Hz, 15.3 Hz, 2H), 3.52 (t, J = 7.2 Hz, 1H), 3.60, 3.66 (2s, 1H), 3.69 (s, 1H), 3.77, 3.79 (2s, 3H), 7.00-7.25 (m, 5H), 7.30-7.48 (m, 4H), 8.32-8.51 (m, 1H), 9.08, (brs, 0.6H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.45, 167.41, 152.10, 152.01, 136.77, 135.86, 133.52, 133.48, 128.98, 128.86, 128.66, 128.46, 128.04, 127.87, 127.57, 126.53, 123.42, 123,18, 121.59, 121.33, 120.35, 120.11, 117.37, 116.94, 116.86, 116.65,

113.81, 112.71, 53.04, 52.98, 52.53, 48.86, 48.14, 47.76, 42.26, 41.91, 24.33, 23.36; IR (thin film): 3285, 2954, 1782, 1708, 1635, 1494, 1215, 1079, 894, 778, 735, 700, 621, 597, 560 cm^{-1}; ESI-MS m/z 428.3 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{22}H_{23}N_3O_4Cl$ 428.1372 $(M + H)^+$, found 428.1387.

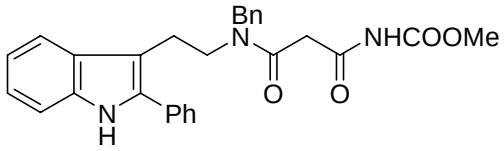


methyl 3-(benzyl(2-(5-methyl-1H-indol-3-yl)ethyl)amino)-3-oxopropanoyl carbamate 1n (mixture of rotamers and tautomers). R_f = 0.2 (ethyl acetate-petroleum 1:1). Yield 60%, white foam. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.43, 2.44 (2s, 3H), 2.95-3.05 (m, 2H), 3.48-3.53 (m, 2H), 3.66-3.71 (m, 1.6H), 3.76, 3.79 (2s, 3H), 4.36 (s, 0.7H), 4.66 (s, 1H), 6.93 (s, 0.5H), 6.99, 7.02 (2s, 1.4H), 7.12 (d, J = 6.6 Hz, 1H), 7.21-7.35 (m, 6H), 8.07 (brs, 0.3H), 8.21 (brs, 0.5H), 9.25 (brs, 0.5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.48, 167.39, 151.69, 136.77, 135.85, 134.59, 134.57, 128.99, 129.89, 128.86, 128.67, 128.58, 128.31, 128.11, 127.87, 127.59, 127.47, 127.05, 126.50, 123.91, 123.64, 122.56, 122.38, 118.22, 117.79, 112.20, 111.21, 111.07, 110.89, 53.00, 52.90, 52.47, 48.73, 48.14, 47.93, 24.23, 23.27, 21.50, 21.47; IR (thin film): 3350, 2952, 1781, 1708, 1632, 1496, 1451, 1363, 1318, 1215, 1080, 797, 775, 739, 702, 618, 567, 427 cm^{-1}; ESI-MS m/z 408.2 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{23}H_{26}N_3O_4$ 408.1918 $(M + H)^+$, found 408.1906.

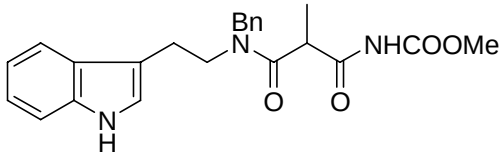


methyl 3-(benzyl(2-(2-methyl-1H-indol-3-yl)ethyl)amino)-3-oxopropanoylcarbamate 1o (mixture of rotamers and tautomers). R_f = 0.2 (ethyl acetate-petroleum 1:1). Yield 58%, white foam. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.30, 2.33 (2s, 3H), 2.91-2.99 (m, 2H), 3.43 (t, J = 6.6 Hz, 2H), 3.57 (t, J = 7.2 Hz, 1H), 3.67 (s, 1H), 3.73, 3.78 (2s, 3H), 4.35 (s, 0.7H), 4.66 (s, 1H), 7.04-7.13 (m, 3H), 7.26-7.43 (m, 6H), 7.97 (brs,

0.3H), 8.17 (brs, 0.5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.32, 167.26, 151.69, 136.77, 135.84, 135.26, 135.20, 132.04, 131.84, 128.99, 128.67, 128.42, 128.08, 128.00, 127.91, 127.59, 126.61, 121.31, 121.04, 119.52, 119.28, 117.66, 117.13, 110.67, 110.30, 108.24, 107.31, 53.01, 52.92, 52.64, 48.70, 47.91, 47.86, 41.72, 41.29, 23.16, 22.18, 11.52, 11.45; IR (thin film): 3350, 2953, 1782, 1761, 1708, 1632, 1496, 1463, 1363, 1303, 1215, 1080, 935, 742, 702 cm^{-1}; ESI-MS m/z (M + H)$^+$; HRMS (MALDI) calcd. for $C_{23}H_{26}N_3O_4$ 408.1918 $(M + H)^+$, found 408.1906.

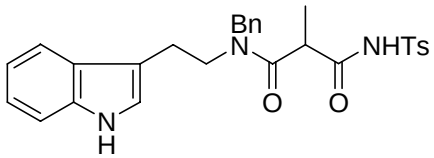


methyl 3-(benzyl(2-(2-phenyl-1H-indol-3-yl)ethyl)amino)-3-oxopropanoylcarbamate 1p (mixture of rotamers and tautomers). R_f = 0.5 (ethyl acetate-petroleum 1:1). Yield 58%, white foam. ^{1}H NMR (400MHz, $CDCl_3$) δ 3.17 (t, J = 7.2 Hz, 1H), 3.23 (t, J = 7.4 Hz, 1H), 3.37-3.41 (m, 3H), 3.67 (t, J = 7.4 Hz, 1H), 3.76, 3.79 (2s, 3H), 4.27 (s, 1H), 4.46 (s, 1H), 7.05 (d, J = 6.8 Hz, 1H), 7.11-7.17 (m, 2H), 7.19-7.32 (m, 3H), 7.37-7.42 (m, 2H), 7.45-7.51 (m, 4H), 7.55-7.62 (m, 2H), 8.10 (brs, 0.5 H), 8.18 (brs, 0.6H); ^{13}C NMR (100MHz, $CDCl_3$) δ167.55, 167.19, 151.73, 136.78, 135.93, 135.81, 135.07, 132.93, 132.73, 129.11, 129.04, 128.64, 128.49, 128.21, 128.13, 127.97, 127.87, 127.82, 127.56, 126.59, 122.61, 122.53, 120.11, 119.92, 118.94, 118.17, 111.36, 111.00, 109.63, 108.46, 52.96, 52.92, 52.50, 48.46, 47.82, 47.71, 41.14, 41.09, 23.23, 22.51; IR (thin film): 3293, 3057, 3027, 2953, 1760, 1708, 1632, 1496, 1451, 1213, 1077, 742, 699 cm^{-1}; ESI-MS m/z 470.3 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{28}H_{28}N_3O_4$ 470.2074 $(M + H)^+$, found 470.2064.



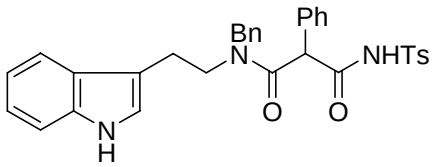
methyl 3-((2-(1H-indol-3-yl)ethyl)(benzyl)amino)-2-methyl-3-oxopropanoyl carbamate. **1q** (mixture of rotamers and tautomers). R_f = 0.6 (ethyl acetate-petroleum

3:1). Yield 86%, white foam. ^{1}H NMR (300MHz, $CDCl_3$) δ 1.35 (d, *J* = 7.2 Hz, 2H), 1.41 (d, *J* = 6.9 Hz, 1H), 2.97-3.07 (m, 2H), 3.52-3.70 (m, 2H), 3.76 (s, 2H), 3.78 (s, 1H), 3.80-3.90 (m, 1H), 4.43-4.54 (m, 1.4H), 4.84, 4.89 (2s, 0.5H), 6.99 (d, *J* = 3.6 Hz, 1H), 7.09-7.22 (m, 4H), 7.28-7.37 (m, 4H), 7.45 (d, *J* = 7.5 Hz, 0.6H), 7.55 (d, *J* = 7.8 Hz, 0.4H), 8.32 (brs, 0.3H), 8.50 (brs, 0.5H), 9.44 (brs, 0.5H), 9.48 (brs, 0.3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 172.19, 171.96, 169.87, 169.45, 169.43, 151.76, 151.53, 136.90, 136.44, 136.38, 136.05, 129.04, 128.96, 128.93, 128.90, 128.82, 128.10, 128.08, 127.96, 127.72, 127.31, 126.95, 126.70, 126.63, 122.75, 122.68, 122.42, 122.35, 122.18, 122.01, 119.65, 119.36, 118.63, 118.62, 118.60, 118.15, 112.42, 111.75, 111.38, 53.01, 52.89, 52.80, 52.29, 52.24, 48.83, 48.38, 48.33, 47.92, 45.30, 45.21, 44.85, 44.77, 24.57, 23.16, 17.19, 17.15, 16.97, 16.89; IR (thin film): 3351, 2987, 2952, 1780, 1715, 1624, 1496, 1457, 1356, 1220, 1098, 1066, 1030, 743, 699, 600, 560, 427 cm^{-1}; ESI-MS m/z 408.2 (M + H)$^{+}$; HRMS (MALDI) calcd. for $C_{23}H_{26}N_3O_4$ 408.1918 $(M + H)^{+}$, found 408.1918.

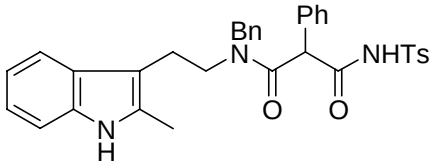


N^1-(2-(1H-indol-3-yl)ethyl)-N^1-benzyl-2-methyl-N^3-tosylmalonamide 1r (mixture of rotamers and tautomers). R_f = 0.6 (ethyl acetate-petroleum 1:1). Yield 50%, white solid, mp 78-80°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 1.18 (d, *J* = 7.2 Hz, 1.9H), 1.29 (d, *J* = 6.9 Hz, 1.2H), 2.41 (s, 3H), 2.93 (dd, *J* = 6.6 Hz, 10.2 Hz, 1H), 3.01 (t, *J* = 7.2 Hz, 1H), 3.15 (q, *J* = 6.9 Hz, 0.6H), 3.41-3.85 (m, 2H), 4.21-4.89 (m, 2H), 6.88 (d, *J* = 2.1 Hz, 1H), 6.96 (d, *J* = 2.4 Hz, 1H), 7.09-7.21 (m, 3H), 7.26-7.55 (m, 7H), 7.89-7.96 (m, 2H), 8.17 (brs, 1H), 10.36 (s, 0.6H), 10.73 (s, 0.3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 172.19, 171.92, 168.53, 168.09, 144.86, 144.78, 136.60, 136.28, 135.80, 135.64, 129.51, 129.45, 129.05, 128.83, 128.33, 127.99, 127.92, 127.79, 127.22, 126.58, 126.21, 122.56, 122.47, 122.23, 122.15, 119.89, 119.51, 118.47, 117.83, 112.27, 111.83, 111.39, 111.16, 52.16, 48.91, 48.29, 47.86, 44.85, 44.26, 24.44, 23.23, 21.66, 17.93, 17.51; IR (thin film): 3403, 3059, 2923, 1716, 1624, 1495, 1421, 1347, 1174,

1081, 857, 814, 743, 701, 663, 548 cm^{-1}; ESI-MS m/z 504.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{28}H_{29}N_3O_4SNa$ 526.1771 $(M + Na)^+$, found 526.1782.

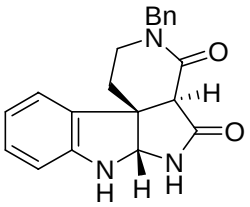


N^1-(2-(1H-indol-3-yl)ethyl)-N^1-benzyl-2-phenyl-N^3-tosylmalonamide 1s (mixture of rotamers and tautomers). R_f = 0.6 (ethyl acetate-petroleum 1:1). Yield 46%, pink solid, mp 70-72°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.38 (s, 3H), 2.85-3.01 (m, 2H), 3.21-3.30 (m, 0.6H), 3.40-3.47 (m, 1H), 3.92 (brs, 0.4H), 4.05, 4.10 (2s, 1H), 4.23-5.04 (m, 2H), 6.84 (s, 2H), 7.11-7.49 (m, 17H), 7.84 (d, J = 8.4 Hz, 1H), 7.88 (d, J = 8.1 Hz, 1H), 8.03 (brs, 0.3H), 8.13 (brs, 0.5H), 11.13 (brs, 0.4H), 11.84 (brs, 0.2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 169.67, 169.54, 166.27, 165.73, 144.57, 136.43, 136.38, 135.73, 132.73, 129.51, 129.47, 129.33, 129.21, 129.01, 128.72, 128.63, 128.55, 128.47, 128.28, 128.03, 127.88, 127.80, 127.73, 127.64, 127.11, 126.98, 126.49, 126.45, 122.69, 122.32, 122.14, 119.87, 119.50, 118.52, 117.80, 112.25, 111.97, 111.37, 111.27, 65.36, 55.52, 55.07, 51.82, 49.01, 48.02, 47.73, 24.18, 23.00, 21.62; IR (thin film): 3400, 3059, 2923, 1717, 1620, 1455, 1417, 1348, 1173, 1087, 891, 814, 743, 701, 658, 583, 545, 425 cm^{-1}; ESI-MS m/z 566.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{33}H_{31}N_3O_4SNa$ 588.1927 $(M + Na)^+$, found 588.1943.

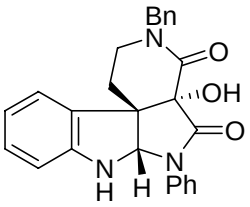


N^1-benzyl-N^1-(2-(2-methyl-1H-indol-3-yl)ethyl)-2-phenyl-N^3-tosylmalonamide 1t (mixture of rotamers and tautomers). R_f = 0.4 (ethyl acetate-petroleum 2:1). Yield 48%, white foam. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.19 (s, 3H), 2.36 (s, 3H), 2.80-3.39 (m, 3.6H), 3.67-3.75 (m, 0.3H), 3.95 (s, 0.5H), 4.14, 4.19 (2s, 1H), 4.52 (s, 0.3H), 5.10, 5.15, 5.26 (3s, 0.7H), 7.01-7.28 (m, 16H), 7.82-7.91 (m, 2.7H), 11.09 (brs, 0.5H),

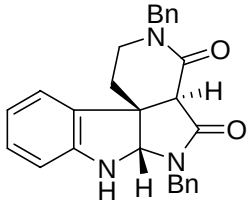
11.94 (brs, 0.2H); ^{13}C NMR (100MHz, $CDCl_3$) δ169.55, 169.45, 166.34, 165.52, 144.65, 144.60, 136.33, 135.76, 135.72, 135.55, 135.20, 135.05, 132.93, 132.63, 131.94, 131.92, 129.49, 129.34, 129.24, 129.14, 128.98, 128.92, 128.84, 128.78, 128.70, 128.63, 128.56, 128.47, 128.42, 128.30, 128.25, 128.14, 128.05, 127.99, 127.98, 127.93, 127.90, 127.86, 127.78, 127.73, 127.70, 127.61, 126.69, 121.86, 121.06, 119.75, 119.30, 117.53, 116.77, 111.27, 110.41, 107.74, 107.13, 55.48, 54.75, 51.98, 48.87, 47.88, 47.28, 23.03, 21.93, 21.61, 11.37, 11.33; IR (thin film): 3394, 3059, 3029, 2920, 1718, 1621, 1541, 1419, 1350, 1173, 1139, 1087, 963, 814, 742, 695, 581, 545 cm^{-1}; ESI-MS m/z 580.4 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{34}H_{34}N_3O_4S$ 580.2265 (M + H)$^+$, found 580.2269.



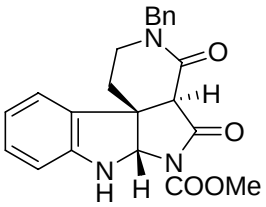
5a R_f = 0.4 (dichloromethane-methane 10:1). Yield 21%, white foam. ^{1}H NMR (300MHz, d_6-DMSO) δ 1.87 (d, J = 13.5 Hz, 1H), 1.94-2.04 (m, 1H), 3.19 (d, J = 13.2 Hz, 1H), 3.25 (s, 1H), 3.48 (td, J = 1.2 Hz, 3.43-3.53 (m, 1H), 4.58 (ABq, J = 15 Hz, 2H), 5.19 (d, J = 1.8 Hz, 1H), 6.51 (s, 1H), 6.54 (d, J = 7.8 Hz, 1H), 6.66 (t, J = 6.9 Hz, 1H), 7.02 (t, J = 7.2 Hz, 1H), 7.19-7.38 (m, 6H), 8.49 (s, 1H); ^{13}C NMR (100MHz, d_6-DMSO) δ 170.34, 164.27, 149.16, 149.01, 137.82, 133.67, 129.15, 128.99, 127.85, 127.61, 123.80, 118.78, 109.83, 75.17, 55.76, 53.53, 50.18, 43.62, 32.17; IR (thin film): 3421, 2254, 2128, 1659, 1051, 1027, 1005, 826, 764, 630 cm^{-1}; ESI-MS m/z 334.2 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{20}H_{20}N_3O_2$ 334.1550 (M + H)$^+$, found 334.1545.



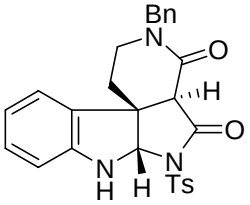
6 R_f = 0.3 (ethyl acetate-petroleum 1:1). Yield 43%, white solid, mp 120-122°C. ^{1}H NMR (300MHz, d_6-acetone) δ 2.20 (dt, *J* = 2.7 Hz, 13.5 Hz, 1H), 2.33 (td, *J* = 4.5 Hz, 13.8 Hz, 1H), 3.36 (ddd, *J* = 2.7 Hz, 4.5 Hz, 13.5 Hz, 1H), 3.69 (td, *J* = 3.3 Hz, 13.5 Hz, 1H), 4.60 (d, *J* = 14.7 Hz, 1H), 4.79 (d, *J* = 14.7 Hz, 1H), 5.19 (s, 1H, disappeared after 1 drop D_2O was added), 6.07 (d, *J* = 3.9 Hz, 1H), 6.21 (d, *J* = 2.7 Hz, 1H, disappeared after 1 drop D_2O was added), 6.58 (d, *J* = 7.5 Hz, 1H), 6.72 (t, *J* = 7.5 Hz, 1H), 7.04 (td, *J* = 0.6 Hz, 7.8 Hz, 1H), 7.20-7.32 (m, 7H), 7.41 (d, *J* = 7.5 Hz, 2H), 7.66 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (100MHz, d_6-acetone) δ 169.11, 167.86, 147.72, 136.47, 135.96, 129.46, 129.37, 128.92, 128.12, 127.95, 127.43, 126.83, 126.79, 123.48, 119.94, 110.19, 79.49, 54.05, 51.56, 42.71, 32.04; IR (thin film): 3352, 2925, 1704, 1651, 1489, 1403, 1298, 1155, 1062, 997, 801, 750, 696 cm^{-1}; ESI-MS m/z 426.4 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{26}H_{23}N_3O_3Na$ 488.1632 (M + Na)$^+$, found 448.1639.



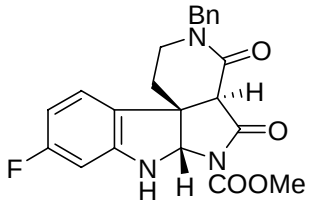
5c R_f = 0.3 (ethyl acetate-petroleum 1:1). Yield 59%, white foam. ^{1}H NMR (300MHz, $CDCl_3$) δ 1.86 (d, *J* = 14.1 Hz, 1H), 2.00-2.05 (m, 1H), 3.09-3.13 (m, 2H), 3.75 (s, 1H), 4.11 (d, *J* = 15 Hz, 1H), 4.31 (d, *J* = 14.7 Hz, 1H), 4.70 (brs, 1H), 4.87 (s, 1H), 5.02 (d, *J* = 3 Hz, 1H), 5.07 (d, *J* = 2.4 Hz, 1H), 5.29 (s, 1H), 6.64 (d, *J* = 7.8 Hz, 1H), 6.82 (t, *J* = 7.2 Hz, 1H), 7.07-7.36 (m, 12H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.90, 164.56, 147.37, 136.68, 135.89, 133.32, 129.38, 128.87, 128.83, 128.17, 128.01, 127.73, 123.23, 120.69, 111.29, 78.22, 55.77, 51.41, 51.04, 44.17, 43.26, 32.73; IR (thin film): 3305, 3058, 3029, 2922, 1725, 1632, 1496, 1453, 1339, 1233, 1203, 1061, 953, 741, 698, 607, 422 cm^{-1}; ESI-MS m/z 424.2 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{27}H_{26}N_3O_2$ 424.2019 (M + H)$^+$, found 424.2029.



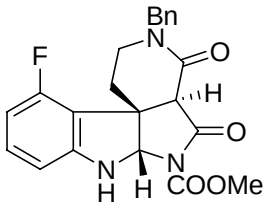
5d R_f = 0.3 (ethyl acetate-petroleum 4:1). Yield 80%, white solid, mp 200-202°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.03, 2.05 (2s, 2H), 3.30-3.36 (m, 1H), 3.42-3.49 (m, 1H), 3.83 (s, 1H), 3.91 (s, 3H), 4.44 (d, *J* = 14.7 Hz, 1H), 4.89 (d, *J* = 14.7 Hz, 1H), 5.61 (s, 1H), 6.64 (d, *J* = 7.8 Hz, 1H), 6.81 (d, *J* = 7.5 Hz, 1H), 7.12 (q, *J* = 7.8 Hz, 2H), 7.26-7.38 (m, 5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.18, 162.64, 152.64, 146.79, 136.26, 130.99, 129.67, 128.83, 128.27, 127.85, 123.08, 120.10, 109.80, 78.81, 56.08, 53.99, 50.89, 49.06, 43.12, 31.02; IR (thin film): 3369, 2954, 1760, 1723, 1640, 1608, 1486, 1436, 1286, 1156, 955, 754 cm^{-1}; ESI-MS m/z 393.3 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{22}H_{22}N_3O_4$ 392.1605 $(M + H)^+$, found 392.1616.



5e R_f = 0.6 (ethyl acetate-petroleum 1:1). Yield 80%, white solid, mp 114-116°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.02-2.05 (m, 2H), 2.42 (s, 3H), 3.21-3.28 (m, 1H), 3.32-3.40 (m, 1H), 3.71 (s, 1H), 4.62 (s, 2H), 5.79 (s, 1H), 6.69 (d, *J* = 7.8 Hz, 1H), 6.80 (t, *J* = 7.8 Hz, 1H), 7.03 (d, *J* = 7.8 Hz, 1H), 7.11-7.20 (m, 3H), 7.26-7.33 (m, 6H), 7.92 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.46, 162.10, 146.52, 145.53, 136.13, 134.73, 130.80, 129.79, 129.64, 128.82, 128.59, 128.05, 127.76, 123.09, 120.40, 109.91, 98.72, 80.15, 55.57, 50.90, 43.04, 31.76, 21.73; IR (thin film): 3374, 2924, 1740, 1648, 1487, 1356, 1171, 1088, 958, 814, 749, 664, 583, 545 cm^{-1}; ESI-MS m/z 488.4 $(M + H)^+$; HRMS (ESI) calcd. for $C_{27}H_{26}N_3O_4S$ 488.1639 $(M + H)^+$, found 488.1643.

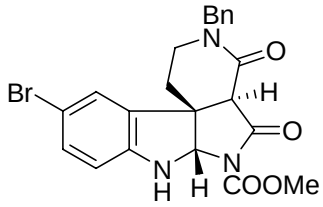


5f R_f = 0.8 (dichloromethane-methane 10:1). Yield 83%, pink solid, mp 98-100°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.04 (d, *J* = 4.5 Hz, 2H), 3.31 (dt, *J* = 3.9 Hz, 13.5 Hz, 1H), 3.41-3.51 (m, 1H), 3.79 (s, 1H), 3.89 (s, 3H), 4.41 (d, *J* = 14.4 Hz, 1H), 4.88 (d, *J* = 14.4 Hz, 1H), 5.40 (s, 1H), 5.64 (s, 1H), 6.33 (dd, *J* = 2.1 Hz, 9.6 Hz, 1H), 6.43-6.50 (m, 1H), 7.00 (dd, *J* = 2.1 Hz, 8.1 Hz, 1H), 7.27-7.35 (m, 5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.01, 164.18 (d, *J* = 243.5 Hz), 162.50, 152.55, 148.46 (d, *J* = 12.2 Hz), 136.15, 128.84, 128.24, 127.88, 126.62, 123.95 (d, *J* = 10.8 Hz), 106.33 (d, *J* = 23.3 Hz), 97.59 (d, *J* = 26.7 Hz), 79.16, 56.12, 54.04, 50.89, 48.38, 43.00, 31.16; IR (thin film): 3363, 2954, 1793, 1759, 1728, 1647, 1621, 1497, 1437, 1268, 1141, 1095, 958, 840, 783, 733, 698, 565, 457 cm^{-1}; ESI-MS m/z 410.2 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{22}H_{21}N_3O_4F$ 410.1511 (M + H)$^+$, found 410.1512.

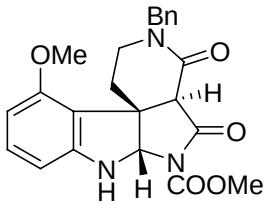


5g R_f = 0.7 (dichloromethane-methane 10:1). Yield 82%, white solid, mp 90-92°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 1.99-2.05 (m, 1H), 2.27-2.37 (m, 1H), 3.32 (d, *J* = 12.6 Hz, 1H), 3.45 (t, *J* = 11.7 Hz, 1H), 3.91 (s, 3H), 4.04 (s, 1H), 4.42 (d, *J* = 14.7 Hz, 1H), 4.91 (d, *J* = 14.7 Hz, 1H), 5.60 (s, 1H), 6.41 (d, *J* = 7.8 Hz, 1H), 6.47 (t, *J* = 9 Hz, 1H), 7.09 (dd, *J* = 7.8 Hz, 13.8 Hz, 1H), 7.27-7.36 (m, 5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.02, 162.29, 159.72 (d, *J* = 245.2 Hz), 152.68, 149.98 (d, *J* = 8.1 Hz), 136.17, 131.71 (d, *J* = 8.9 Hz), 128.82, 128.21, 127.83, 116.23 (d, *J* = 17.2 Hz), 106.96 (d, *J* = 20.1 Hz), 105.65 (d, *J* = 2.3 Hz), 78.72, 54.53, 54.08, 50.91, 48.40, 42.90, 28.98; IR (thin film): 3354, 2954, 1792, 1759, 1731, 1628, 1479, 1437, 1293, 1039, 780, 727, 698 cm^{-1}; ESI-MS m/z 410.3 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{22}H_{21}N_3O_4F$

410.1511 $(M + H)^+$, found 410.1512.

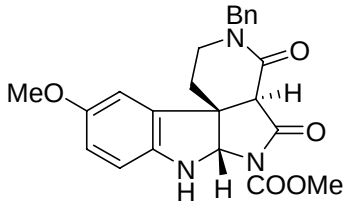


5h R_f = 0.8 (dichloromethane-methane 10:1). Yield 70%, white solid, mp 214-216°C. ^{1}H NMR (300MHz, d_6-DMSO) δ 1.90 (d, J = 13.5 Hz, 1H), 1.99-2.08 (m, 1H), 3.20 (d, J = 12.6 Hz, 1H), 3.60 (td, J = 0.6 Hz, 11.4 Hz, 1H), 3.77, 3.79 (2s, 4H), 4.27 (d, J = 15 Hz, 1H), 4.84 (d, J = 15 Hz, 1H), 5.72 (s, 1H), 6.56 (d, J = 8.1 Hz, 1H), 7.08 (s, 1H), 7.19 (dd, J = 1.2 Hz, 8.1 Hz, 1H), 7.28-7.36 (m, 5H), 7.61, (s, 1H); ^{13}C NMR (100MHz, d_6-DMSO) δ 166.75, 162.48, 151.58, 149.16, 147.54, 137.45, 134.85, 132.03, 128.98, 127.92, 127.66, 127.05, 111.33, 109.47, 98.71, 78.48, 55.53, 53.77, 50.25, 49.42, 43.44, 30.22; IR (thin film): 3349, 2952, 1791, 1727, 1647, 1482, 1437, 1271, 1074, 1042, 814, 759, 733, 697, 519 cm^{-1}; ESI-MS m/z 470.2 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{22}H_{21}N_3O_4Br$ 470.0710 $(M + H)^+$, found 470.0719.

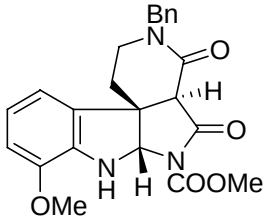


5i R_f = 0.7 (dichloromethane-methane 10:1). Yield 87%, yellow solid, mp 102-104°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 1.87 (dt, J = 3 Hz, 14.1 Hz, 1H), 2.38-2.49 (m, 1H), 3.26-3.33 (m, 1H), 3.39-3.48 (m, 1H), 3.81 (s, 3H), 3.90 (s, 3H), 4.18 (s, 1H), 4.45 (d, J = 14.4 Hz, 1H), 4.88 (d, J = 14.7 Hz, 1H), 5.55 (s, 1H), 6.30 (dd, J = 8.1 Hz, 18.9 Hz, 2H), 7.09 (t, J = 8.1 Hz, 1H), 7.26-7.33 (m, 5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.74, 163.07, 156.46, 152.88, 148.09, 136.48, 131.14, 128.73, 128.21, 127.71, 116.02, 103.02, 102.53, 78.63, 55.27, 53.99, 53.93, 50.94, 48.76, 43.21, 27.78; IR (thin film): 3365, 2953, 1790, 1758, 1648, 1608, 1494, 1474, 1438, 1292, 1086, 779, 728 cm^{-1}; ESI-MS m/z 422.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{23}H_{24}N_3O_5$

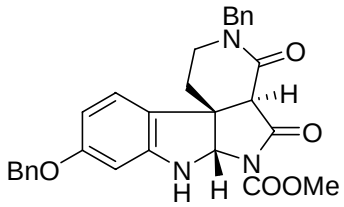
422.1710 $(M + H)^+$, found 422.1724.



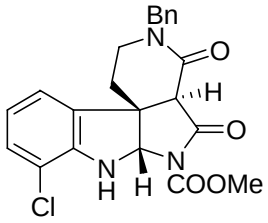
5j R_f = 0.8 (dichloromethane-methane 10:1). Yield 83%, pink solid, mp 98-100°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.03-2.06 (m, 2H), 3.31-3.37 (m, 1H), 3.41-3.48 (m, 1H), 3.73 (s, 3H), 3.80 (s, 1H), 3.91 (s, 3H), 4.42 (d, *J* = 14.7 Hz, 1H), 4.90 (d, *J* = 14.7 Hz, 1H), 5.61 (s, 1H), 6.57 (d, *J* = 8.4 Hz, 1H), 6.68-6.72 (m, 2H), 7.27-7.38 (m, 5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 165.99, 162.57, 154.43, 152.68, 140.44, 136.24, 132.24, 128.83, 128.29, 127.86, 115.05, 110.68, 109.56, 79.51, 56.07, 55.95, 53.98, 50.89, 49.44, 43.16, 30.84; IR (thin film): 3364, 2952, 1791, 1759, 1728, 1647, 1496, 1437, 1272, 1210, 1036, 812, 760, 699, 519 cm^{-1}; ESI-MS m/z 422.3 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{23}H_{24}N_3O_5$ 422.1710 $(M + H)^+$, found 422.1701.



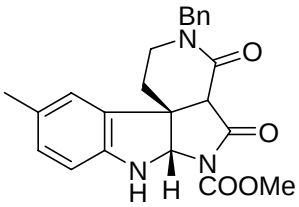
5k R_f = 0.8 (dichloromethane-methane 10:1). Yield 80%, white solid, mp 97-99°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.04-2.07 (m, 2H), 3.33 (dt, *J* = 4.2 Hz, 12.9 Hz, 1H), 3.40-3.50 (m, 1H), 3.82 (s, 4H), 3.92 (s, 3H), 4.45 (d, *J* = 14.7 Hz, 1H), 4.89 (d, *J* = 15 Hz, 1H), 5.65 (s, 1H), 6.71-6.83 (m, 3H), 7.27-7.34 (m, 5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.12, 162.60, 152.56, 145.06, 136.26, 135.90, 131.55, 128.83, 128.29, 127.84, 121.05, 114.99, 111.17, 79.27, 56.06, 55.46, 54.01, 50.86, 49.81, 43.10, 31.01; IR (thin film): 3373, 2953, 1793, 1724, 1651, 1495, 1439, 1294, 1067, 957, 759, 731, 698 cm^{-1}; ESI-MS m/z 422.3 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{23}H_{24}N_3O_5$ 422.1711 $(M + H)^+$, found 422.1725.



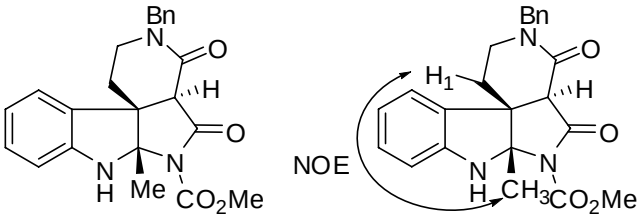
5l R_f = 0.8 (dichloromethane-methane 10:1). Yield 87%, yellow solid, mp 93-95°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 1.99-2.03 (m, 2H), 3.30 (dt, *J* = 4.2 Hz, 13.5 Hz, 1H), 3.38-3.48 (m, 1H), 3.78 (s, 1H), 3.89 (s, 3H), 4.42 (d, *J* = 14.7 Hz, 1H), 4.88 (d, *J* = 14.7 Hz, 1H), 4.99 (s, 2H), 5.17 (brs, 1H), 5.59 (s, 1H), 6.26 (d, *J* = 2.4 Hz, 1H), 6.41 (dd, *J* = 2.4 Hz, 8.4 Hz, 1H), 6.96 (d, *J* = 8.4 Hz, 1H), 7.26-7.39 (m, 10H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.17, 162.69, 160.65, 152.73, 148.19, 136.85, 136.28, 128.83, 128.58, 128.28, 127.97, 127.85, 127.37, 123.62, 106.10, 97.19, 79.21, 70.17, 56.32, 54.01, 50.87, 48.41, 43.13, 31.19; IR (thin film): 3364, 2952, 1791, 1727, 1647, 1621, 1498, 1282, 1157, 1100, 1027, 956, 825, 782, 735, 697 cm^{-1}; ESI-MS m/z 498.4 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{29}H_{28}N_3O_5$ 498.2023 $(M + H)^+$, found 498.2023.



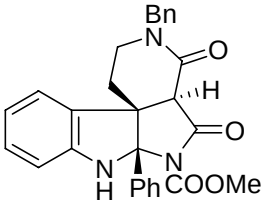
5m R_f = 0.8 (dichloromethane-methane 10:1). Yield 87%, white solid, mp 100-102°C. ^{1}H NMR (300MHz, $CDCl_3$) δ 2.05-2.08 (m, 2H), 3.33 (dt, *J* = 3.9 Hz, 13.5 Hz, 1H), 3.42-3.51 (m, 1H), 3.81 (s, 1H), 3.93 (s, 3H), 4.44 (d, *J* = 14.7 Hz, 1H), 4.89 (d, *J* = 14.7 Hz, 1H), 5.37 (brs, 0.5H), 5.67 (s, 1H), 6.76 (t, *J* = 7.5 Hz, 1H), 6.99 (d, *J* = 7.5 Hz, 1H), 7.14 (d, *J* = 7.2 Hz, 1H), 7.27-7.36 (m, 5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 165.78, 162.27, 152.50, 144.10, 136.14, 132.37, 129.41, 128.85, 128.28, 127.90, 121.37, 121.00, 115.08, 78.42, 56.01, 54.16, 50.89, 50.01, 42.90, 31.23; IR (thin film): 3342, 2953, 1793, 1760, 1724, 1652, 1478, 1437, 1300, 1044, 757, 731, 698 cm^{-1}; ESI-MS m/z 426.2 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{22}H_{21}N_3O_4Cl$ 426.1215 $(M + H)^+$, found 426.1227.



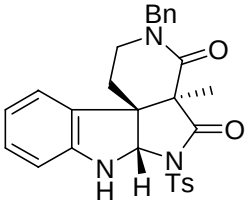
5n R_f = 0.4 (ethyl acetate-petroleum 1:1). Yield 66%, white foam. ^{1}H NMR (400MHz, $CDCl_3$) δ 2.06 (s, 2H), 2.26 (s, 3H), 3.35 (s, 1H), 3.46 (s, 1H), 3.82 (s, 1H), 3.91 (s, 3H), 4.44 (d, *J* = 13.6 Hz, 1H), 4.91 (d, *J* = 13.2 Hz, 1H), 5.08 (brs, 1H), 5.62 (s, 1H), 6.55 (d, *J* = 8 Hz, 1H), 6.93 (t, *J* = 8.4 Hz, 2H), 7.30-7.37 (m, 8H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.16, 162.68, 152.71, 144.39, 136.27, 131.15, 130.08, 129.68, 128.82, 128.29, 127.83, 123.70, 109.74, 79.24, 56.13, 54.03, 50.89, 49.04, 43.20, 31.01, 20.79; IR (thin film): 3367, 2953, 2862, 1791, 1729, 1648, 1498, 1437, 1293, 1042, 957, 815, 761, 700, 520 cm^{-1};ESI-MS m/z 406.2 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{23}H_{24}N_3O_4$ 406.1761 $(M + H)^+$, found 406.1746.



5o R_f = 0.4 (ethyl acetate-petroleum 1:1). Yield 80%, white foam. The relative configuration of **5o** was established by NOE experiment. The existence of NOE between H1 and methyl proton indicated that methyl group was *syn* to the piperidinone ring. ^{1}H NMR (400MHz, $CDCl_3$) δ 1.73 (s, 3H), 1.87-1.96 (m, 1H), 2.11-2.20 (m, 1H), 3.35-3.54 (m, 2H), 3.71 (s, 1H), 3.87 (s, 3H), 4.69 (dd, *J* = 14.4 Hz, 36.3 Hz, 2H), 5.52 (brs, 1H), 6.62 (d, *J* = 10 Hz, 1H), 6.80 (t, *J* = 10 Hz, 1H), 7.10 (d, *J* = 10 Hz, 2H), 7.31-7.38 (m, 5H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.20, 163.53, 152.79, 146.28, 136.36, 132.66, 129.40, 128.78, 128.51, 127.91, 122.74, 120.20, 109.89, 88.97, 54.18, 53.64, 51.59, 50.58, 44.07, 28.94, 20.97; NOE ($CDCl_3$) H_1 (H_2); IR (thin film): 3382, 2952, 1789, 1759, 1717, 1652, 1610, 1487, 1470, 1436, 1305, 1160, 1100, 1068, 938, 750, 705 cm^{-1}; ESI-MS m/z 406.2 (M + H)$^+$; HRMS (MALDI) calcd. for $C_{23}H_{24}N_3O_4$ 406.1761 $(M + H)^+$, found 406.1757.

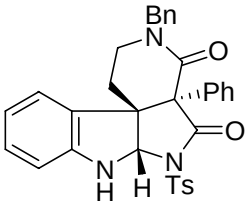


5p R_f = 0.2 (ethyl acetate-petroleum 1:1). Yield 77%, yellow foam. ^{1}H NMR (400MHz, $CDCl_3$) δ 1.69-1.76 (m, 3H), 2.88-2.92 (m, 1H), 3.77 (s, 4H), 4.18 (d, *J* = 14 Hz, 1H), 4.52 (d, *J* = 14.4 Hz, 1H), 5.79 (s, 1H), 6.74 (d, *J* = 7.6 Hz, 1H), 6.85 (t, *J* = 7.6 Hz, 1H), 7.07-7.10 (m, 4H), 7.15-7.19 (m, 2H), 7.24-7.26 (m, 3H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.41 (t, *J* = 7.2 Hz, 1H), 7.88 (d, *J* = 7.6 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 166.54, 162.72, 152.56, 146.33, 137.67, 136.08, 132.41, 129.99, 129.25, 129.03, 128.98, 128.74, 128.61, 127.72, 127.45, 124.99, 122.95, 120.55, 110.13, 95.52, 53.83, 53.61, 53.29, 50.68, 43.14, 29.13; IR (thin film): 3392, 2953, 1791, 1763, 1720, 1647, 1609, 1488, 1436, 1292, 1157, 1094, 1072, 1042, 958, 747, 703, 604, 534 cm^{-1}; ESI-MS m/z 468.2 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{28}H_{26}N_3O_4$ 468.1918 $(M + H)^+$, found 468.1921.

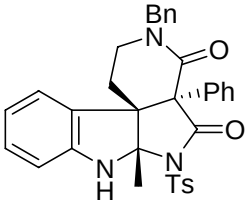


5r R_f = 0.7 (ethyl acetate-petroleum 1:1). Yield 25%, white foam. ^{1}H NMR (300MHz, $CDCl_3$) δ 1.29 (s, 3H), 2.03 (d, *J* = 13.8 Hz, 1H), 2.13-2.22 (m, 1H), 2.41 (s, 3H), 3.14-3.16 (m, 2H), 4.46 (d, *J* = 14.4 Hz, 1H), 4.71 (d, *J* = 14.4 Hz, 1H), 5.79 (s, 1H), 6.70 (d, *J* = 7.8 Hz, 1H), 6.79 (t, *J* = 7.5 Hz, 1H), 6.96 (d, *J* = 7.5 Hz, 1H), 7.12-7.19 (m, 3H), 7.26-7.33 (m, 5H), 7.95 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 171.12, 166.37, 148.16, 145.50, 136.47, 134.75, 129.66, 129.58, 128.76, 128.41, 128.05, 127.84, 127.65, 124.93, 119.90, 110.11, 81.21, 56.14, 54.46, 51.28, 42.30, 33.85, 21.73, 19.97; IR (thin film): 3371, 3027, 2935, 1735, 1652, 1488, 1471, 1451, 1432, 1358, 1310, 1171, 1086, 885, 814, 753, 702, 666, 580, 548 cm^{-1}; ESI-MS m/z 502.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{28}H_{28}N_3O_4S$ 502.1795 $(M + H)^+$, found

502.1779.



5s R_f = 0.6 (ethyl acetate-petroleum 1:1). Yield 70%, white foam. ^{1}H NMR (300MHz, $CDCl_3$) δ 1.85 (dd, *J* = 4.2 Hz, 15 Hz, 1H), 2.21 (ddd, *J* = 6.9 Hz, 12.6 Hz, 14.4 Hz, 1H), 2.43 (s, 3H), 3.42-3.49 (m, 1H), 3.57 (td, *J* = 5.4 Hz, 12.6 Hz, 1H), 4.49 (d, *J* = 14.1 Hz, 1H), 4.66 (d, *J* = 14.4 Hz, 1H), 5.22 (d, *J* = 7.5 Hz, 1H), 5.40 (s, 1H), 5.82 (s, 1H), 6.35 (t, *J* = 7.5 Hz, 1H), 6.63 (d, *J* = 7.8 Hz, 1H), 7.05 (d, *J* = 8.7 Hz, 2H), 7.23-7.41 (m, 11H), 7.95 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.50, 164.13, 145.98, 145.46, 136.49, 135.87, 134.80, 129.64, 129.43, 128.92, 128.63, 128.31, 128.03, 127.86, 127.38, 126.48, 119.06, 109.70, 76.09, 65.03, 55.81, 51.51, 43.39, 26.41, 21.75; IR (thin film): 3416, 3058, 3030, 2986, 2929, 2864, 1752, 1706, 1651, 1473, 1352, 1242, 1166, 1090, 1053, 812, 746, 705, 587, 547 cm^{-1}; ESI-MS m/z 564.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{33}H_{30}N_3O_4S$ 564.1951 $(M + H)^+$, found 564.1950.



5t R_f = 0.4 (ethyl acetate-petroleum 1:1). Yield 21%, white foam. ^{1}H NMR (400MHz, $CDCl_3$) δ 1.88 (s, 3H), 1.98-2.02 (m, 1H), 2.07-2.13 (m, 1H), 2.43 (s, 3H), 3.57 (s, 2H), 4.68 (dd, *J* = 14 Hz, 22.8 Hz, 2H), 5.11 (d, *J* = 7.6 Hz, 1H), 5.67 (brs, 1H), 6.33 (t, *J* = 7.6 Hz, 1H), 6.63 (d, *J* = 7.6 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 3H), 7.23 (t, *J* = 6.8 Hz, 2H), 7.28-7.34 (m, 8H), 7.89 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 167.61, 163.83, 145.85, 145.10, 136.00, 135.60, 129.60, 129.42, 129.33, 128.73, 128.60, 128.55, 128.23, 127.91, 127.14, 118.93, 109.20, 89.45, 62.54, 59.00, 52.69,

43.59, 29.73, 25.05, 22.44, 21.79; IR (thin film): 3407, 3059, 2924, 2853, 1741, 1636, 1496, 1486, 1473, 1454, 1356, 1238, 1174, 1152, 1085, 750, 704, 664, 579 cm^{-1}; ESI-MS m/z 578.4 $(M + H)^+$; HRMS (MALDI) calcd. for $C_{34}H_{32}N_3O_4S$ 578.2108 $(M + H)^+$, found 578.2100.

Ref:

1) P. Arrizabalaga, P. Castan, J. P. Laurent, *J. Am. Chem. Soc.,* **1984**, *106*, 4814.

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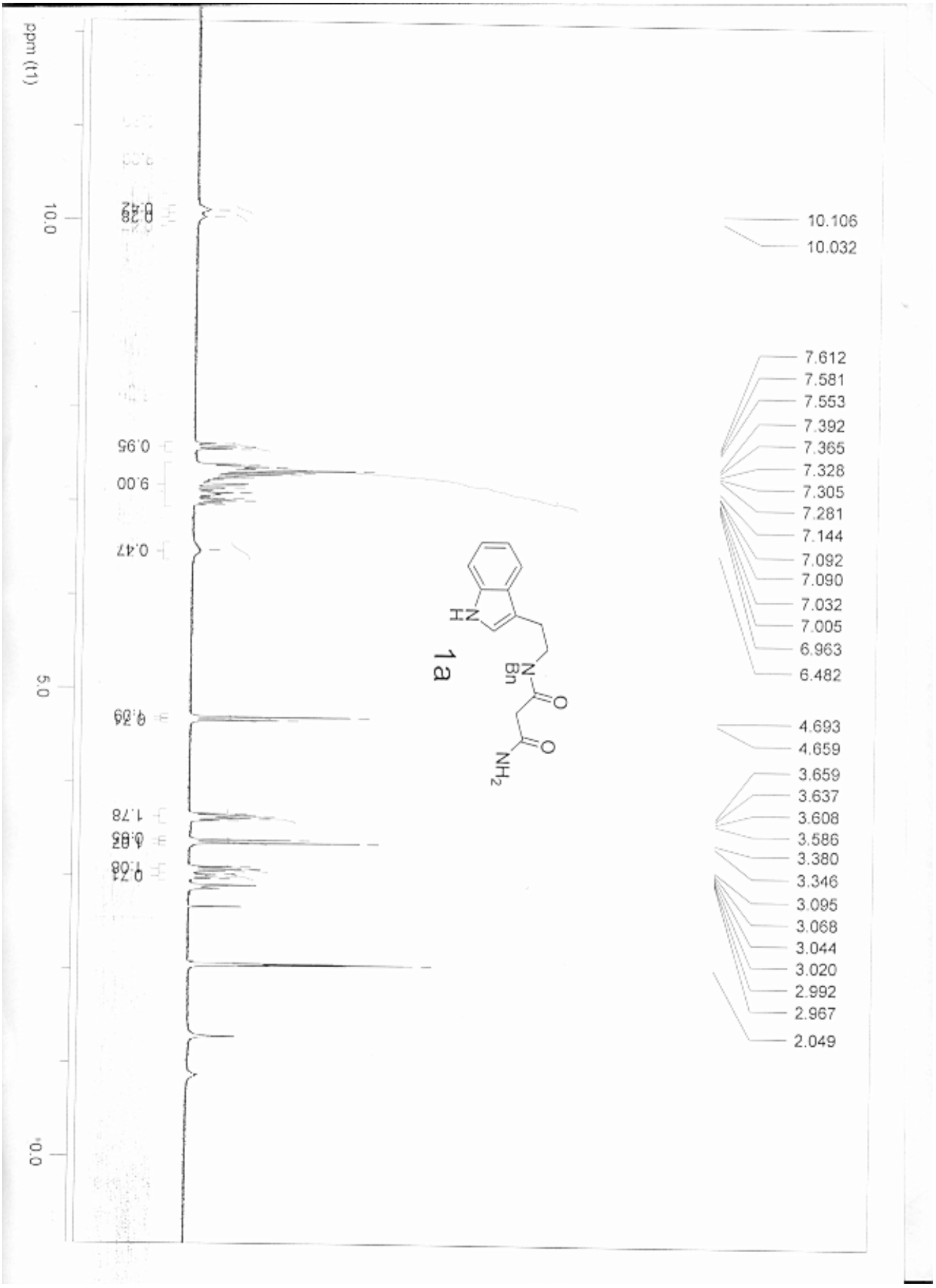
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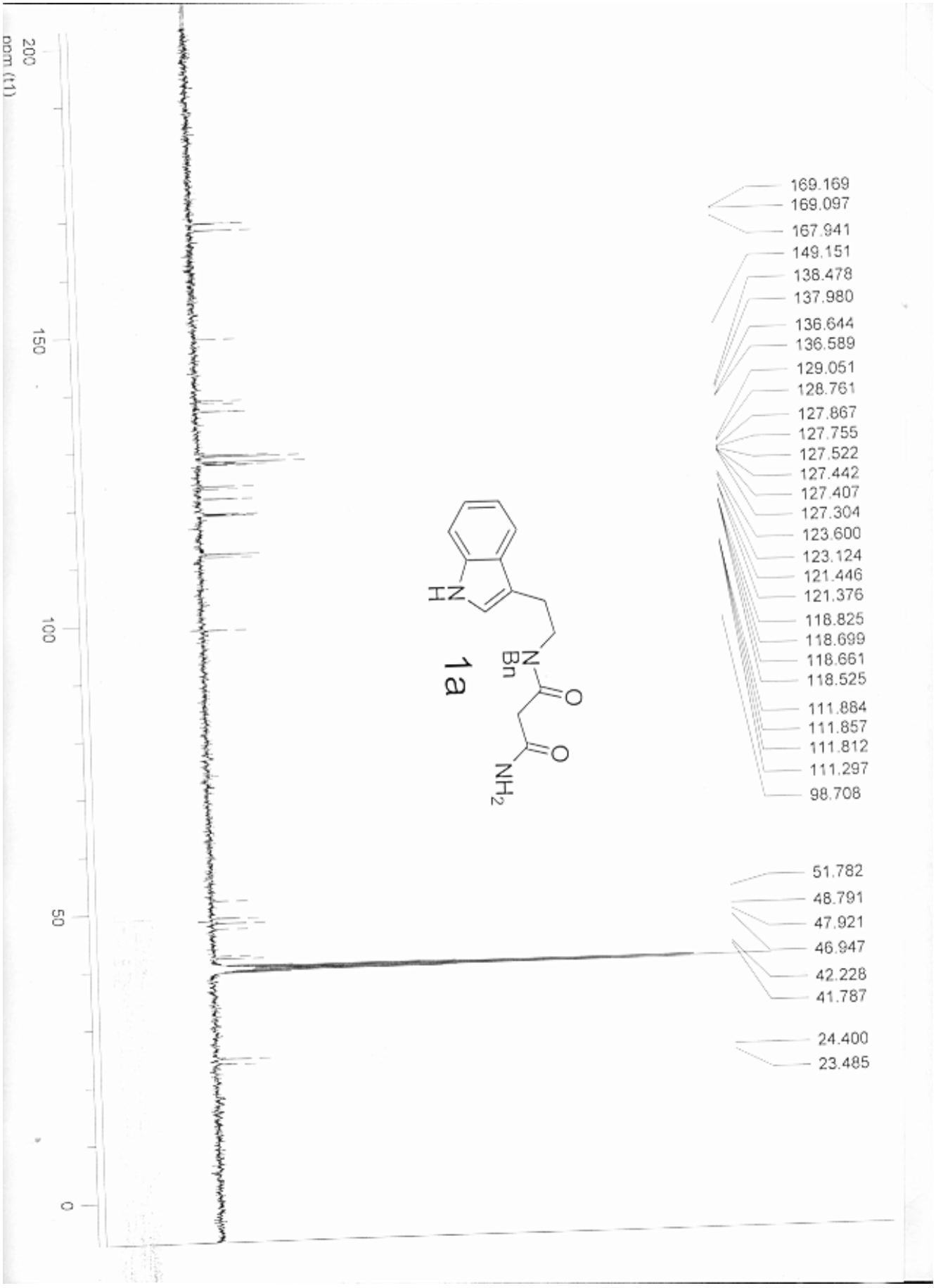
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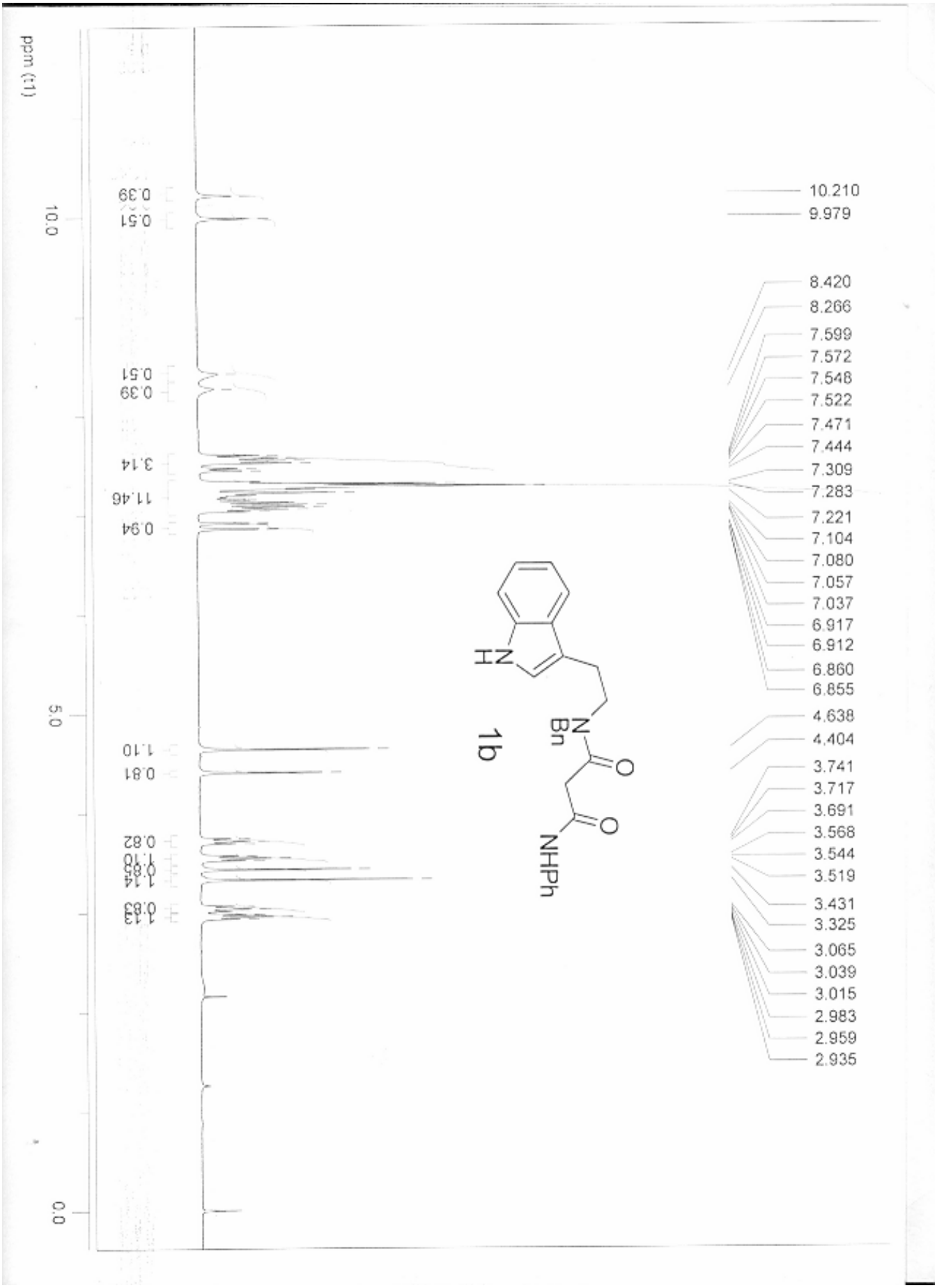
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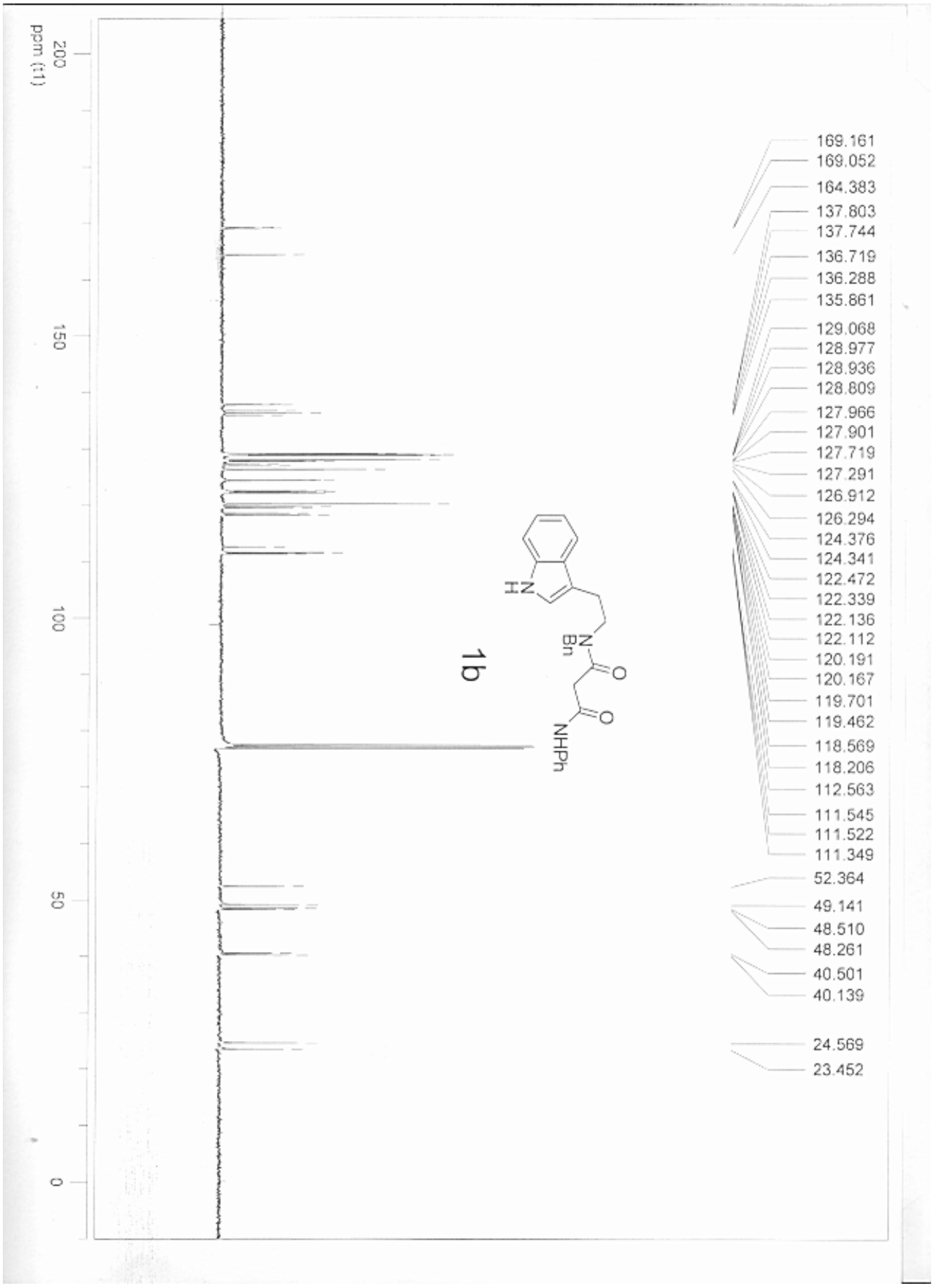
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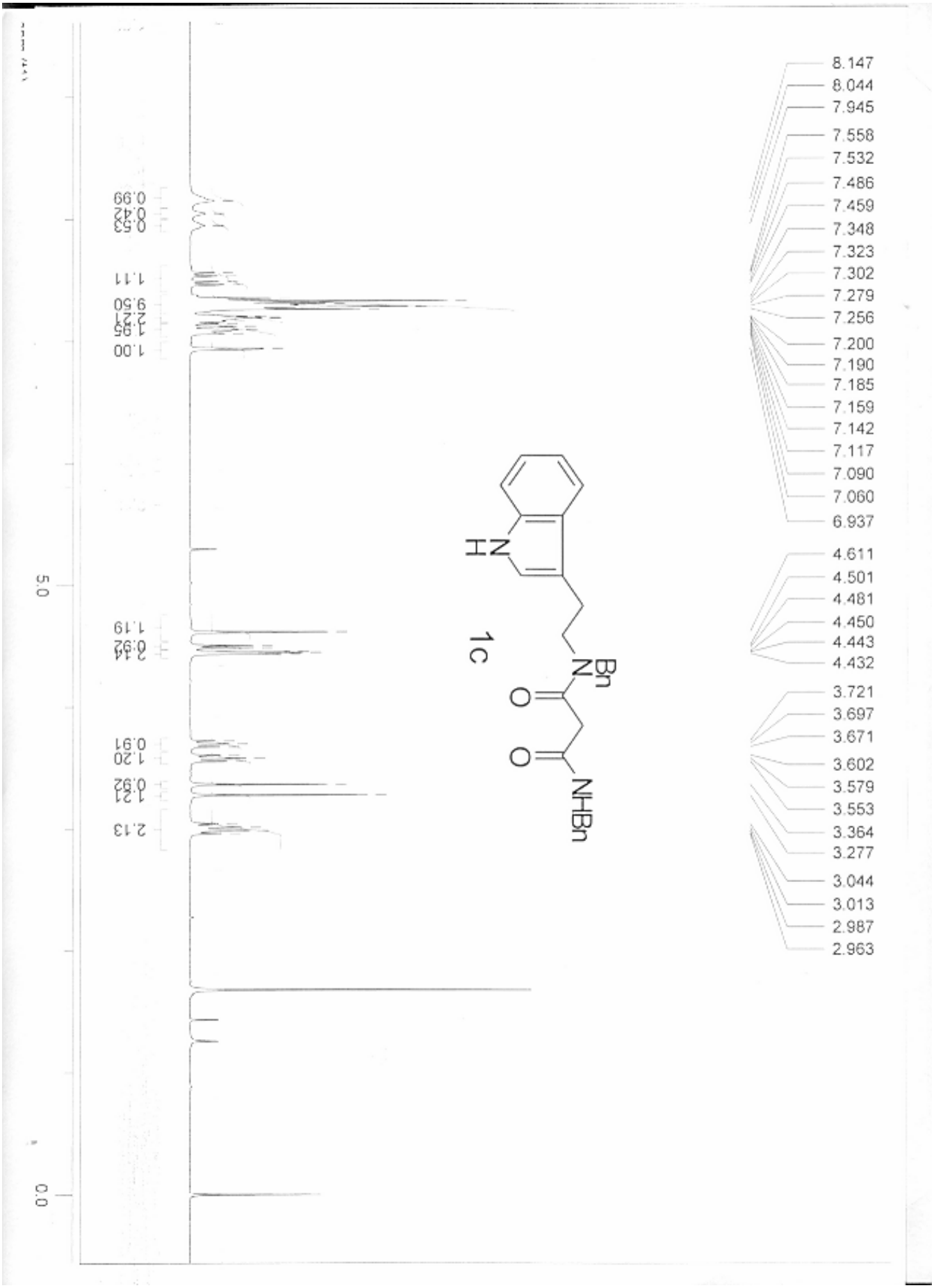
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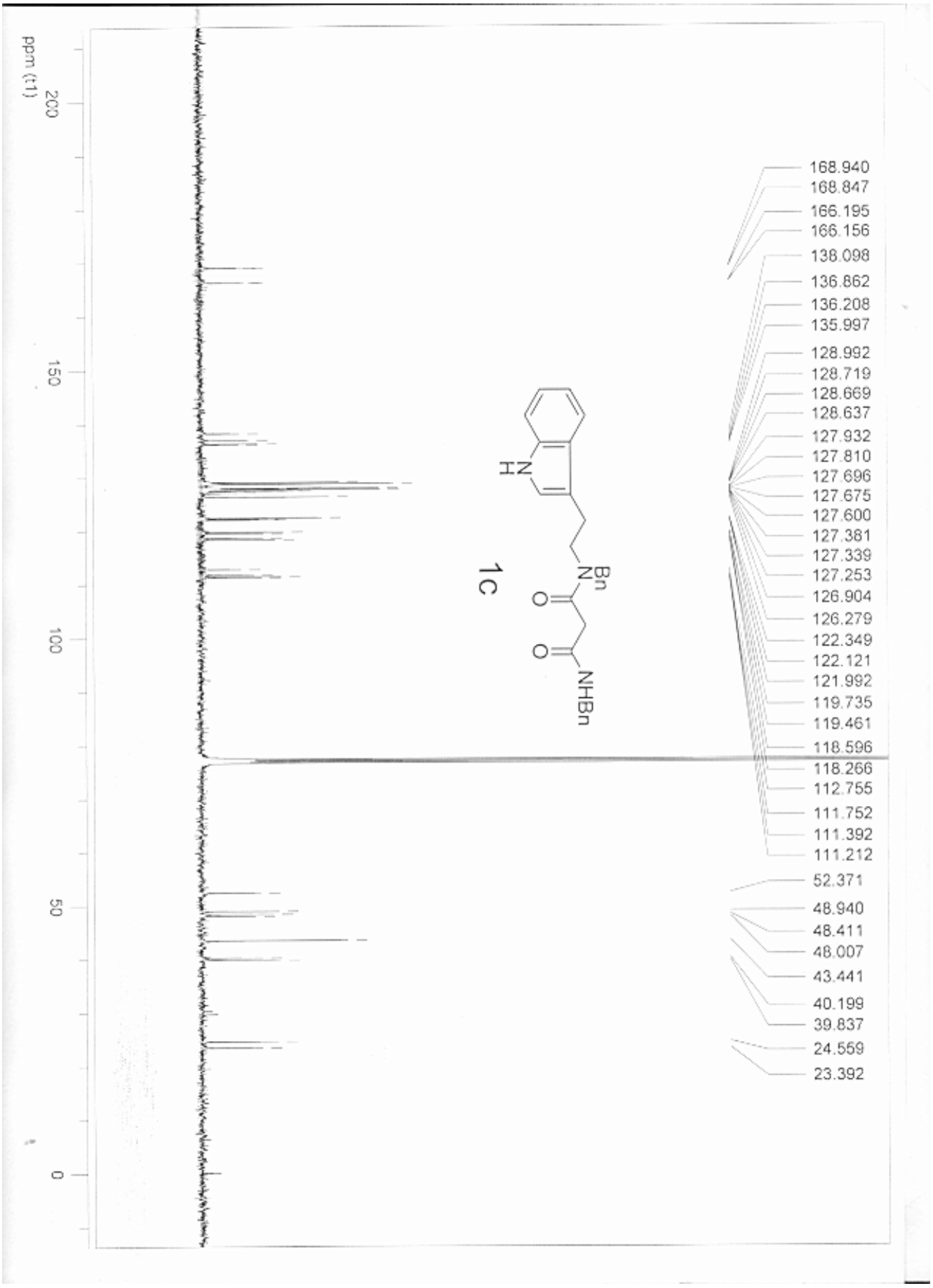
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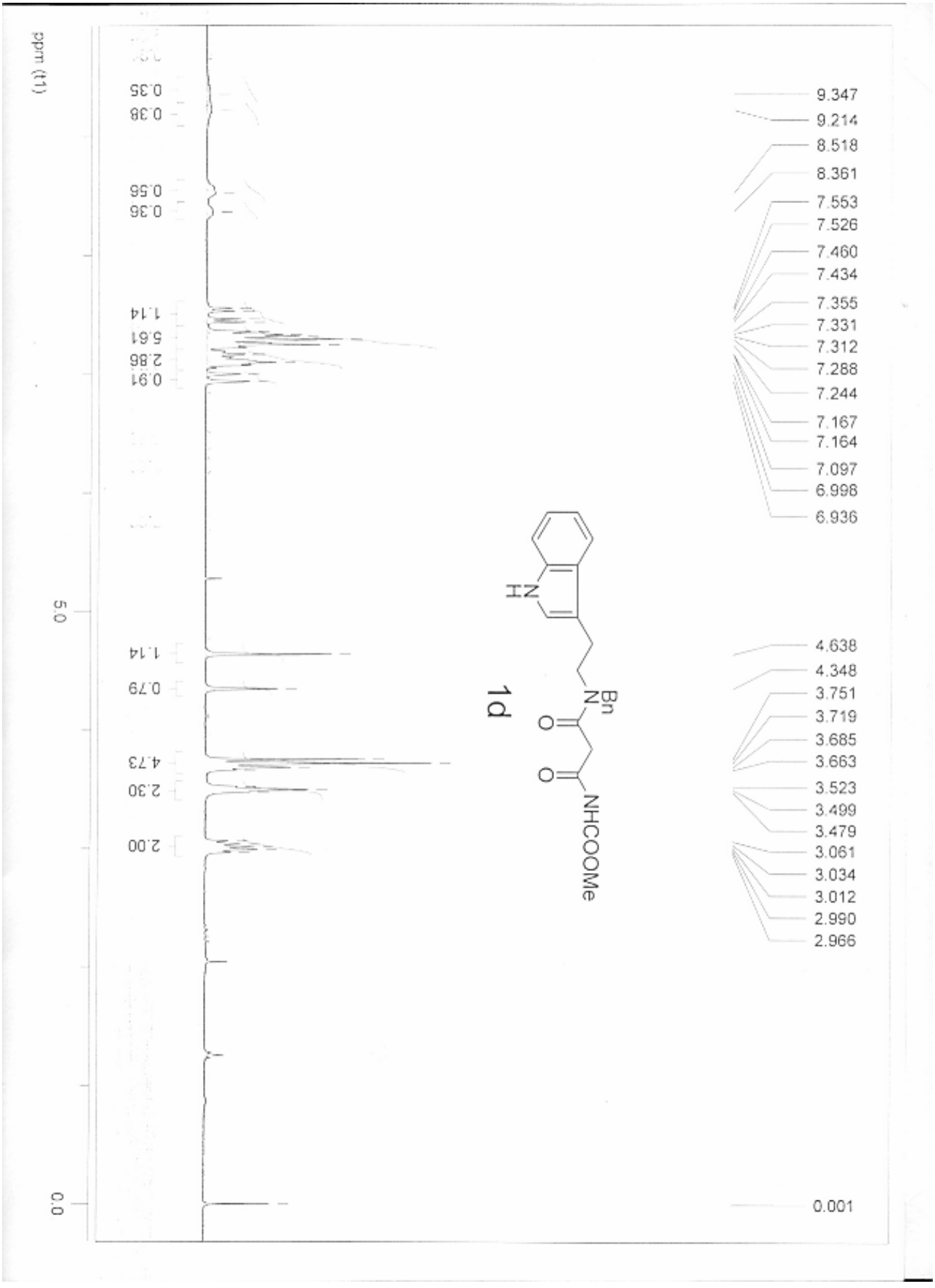
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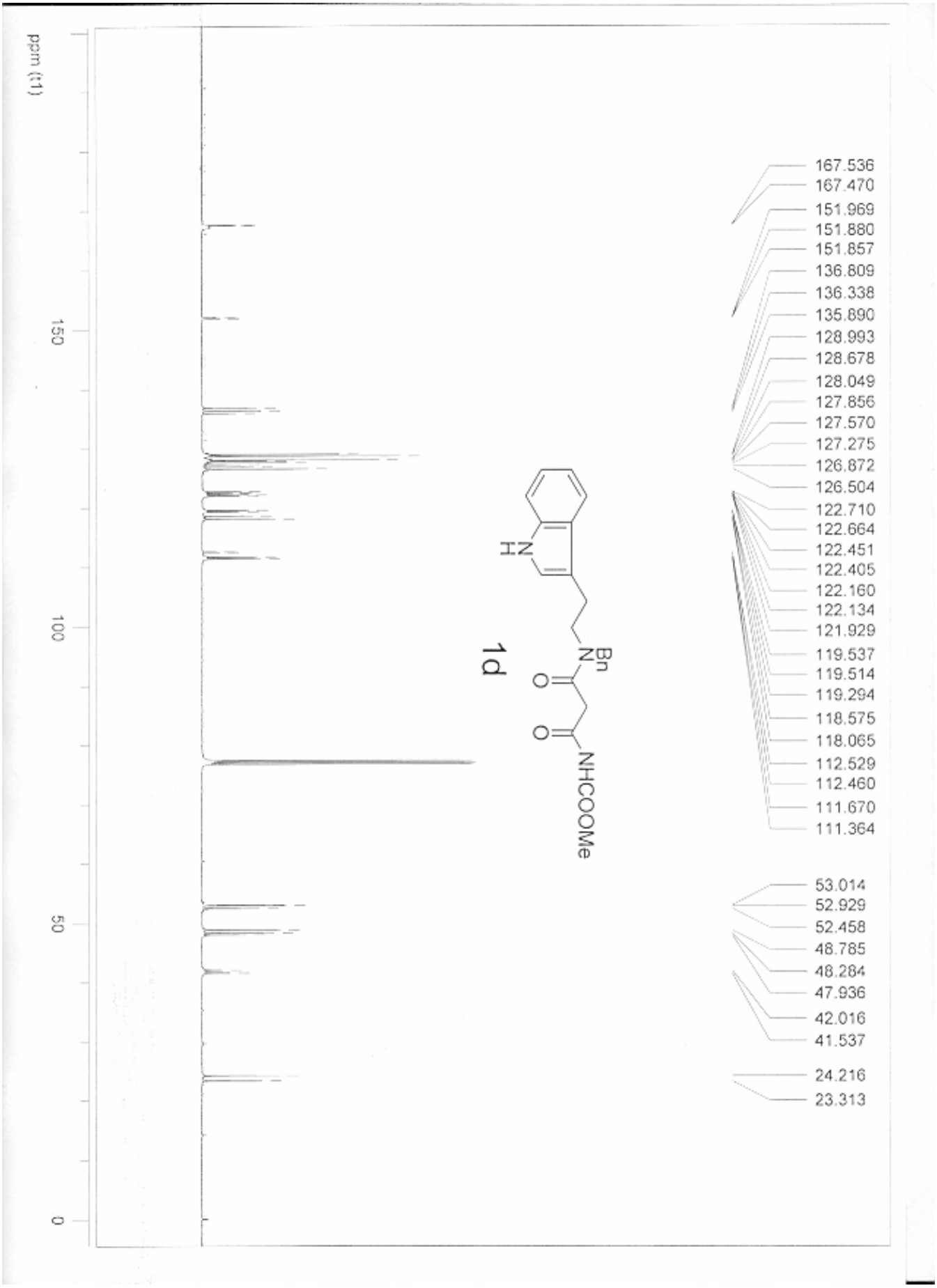
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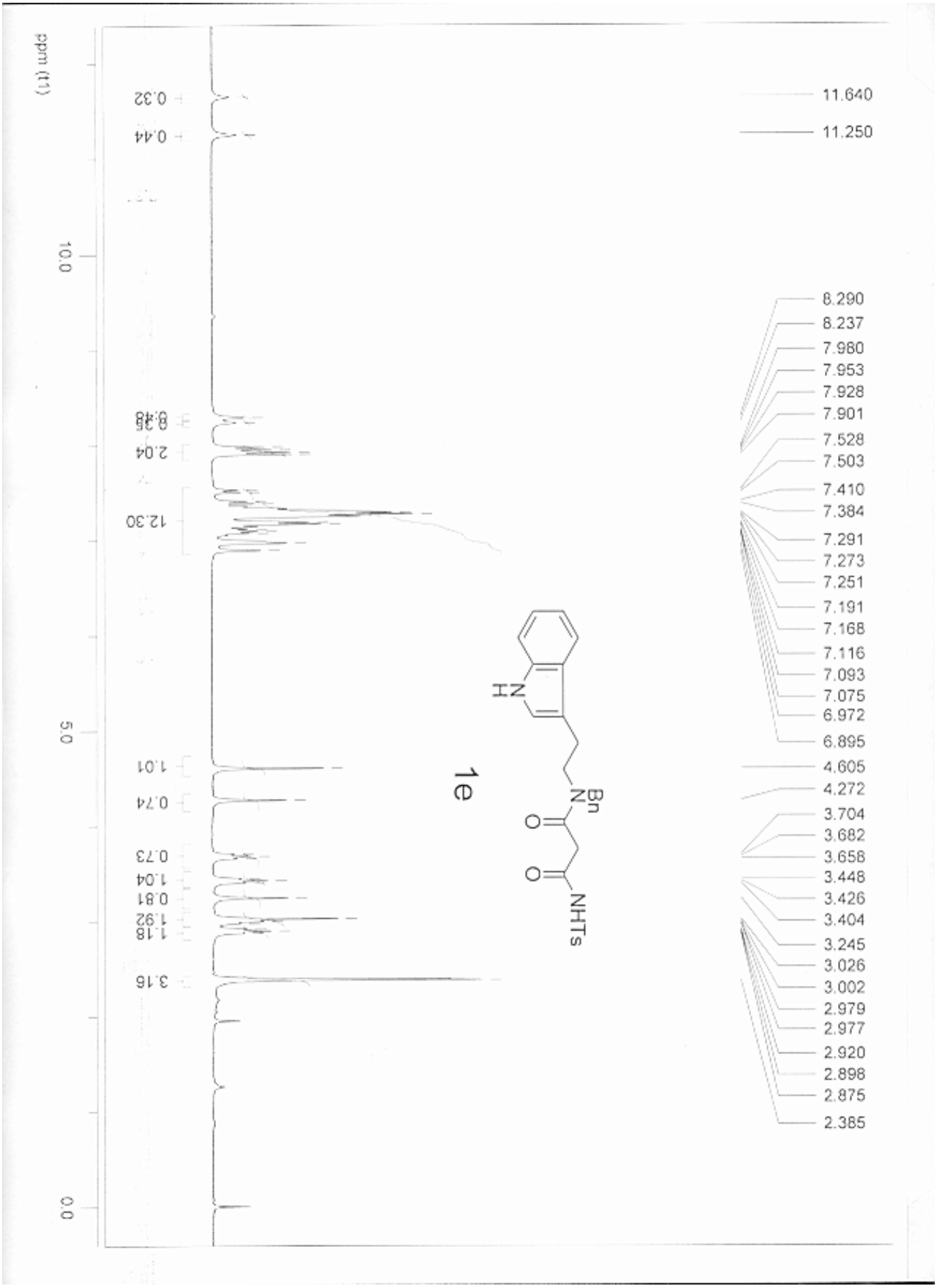
122.134
121.929
119.537
119.514
119.294
118.575
118.065
112.529
112.460
111.670
111.364
53.014
52.929
52.458
48.785
48.284
47.936
42.016
41.537
24.216
23.313
1d
Bn
N
O
O
NHCOOMe
N
H


ppm (t1)
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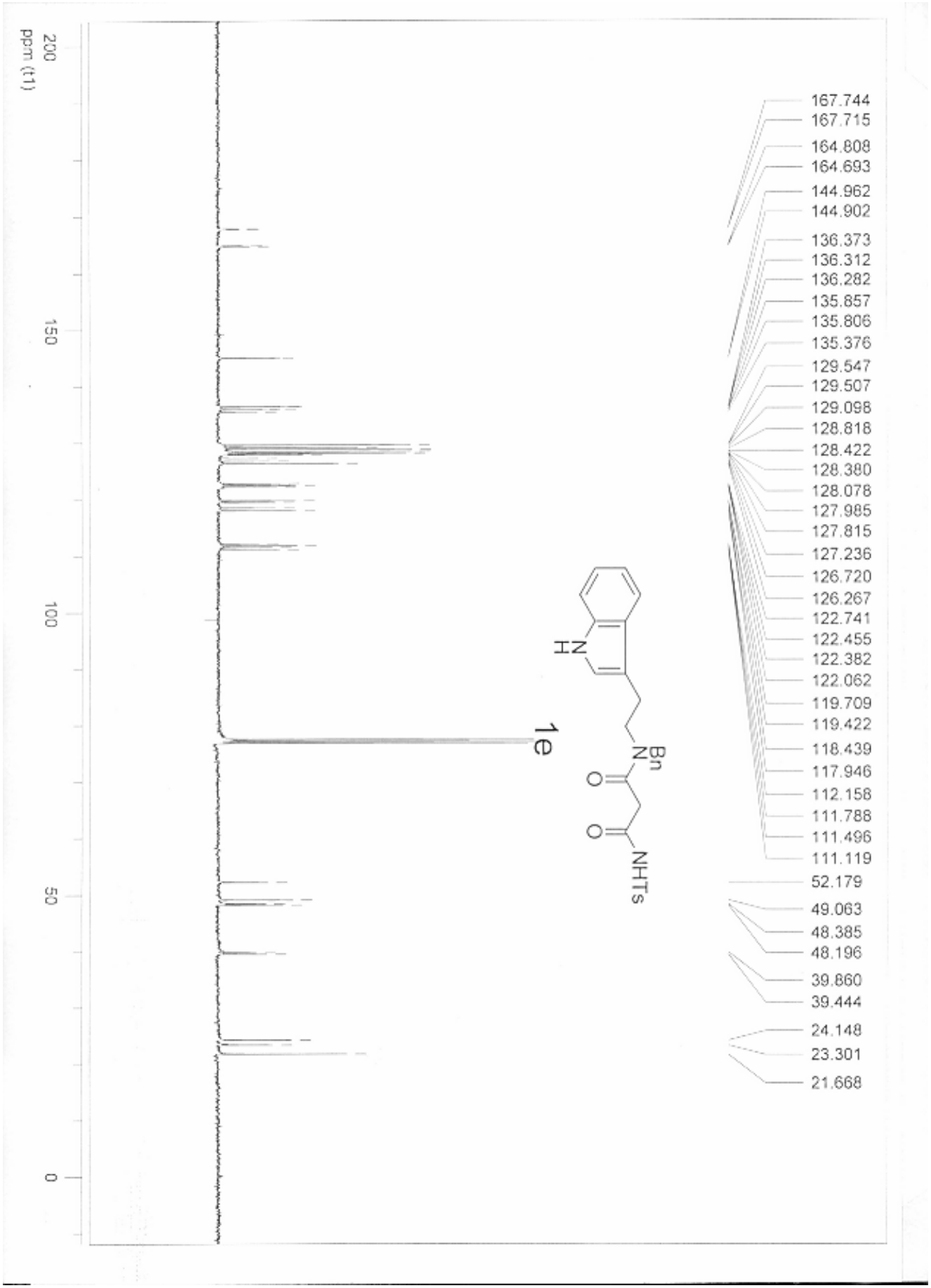
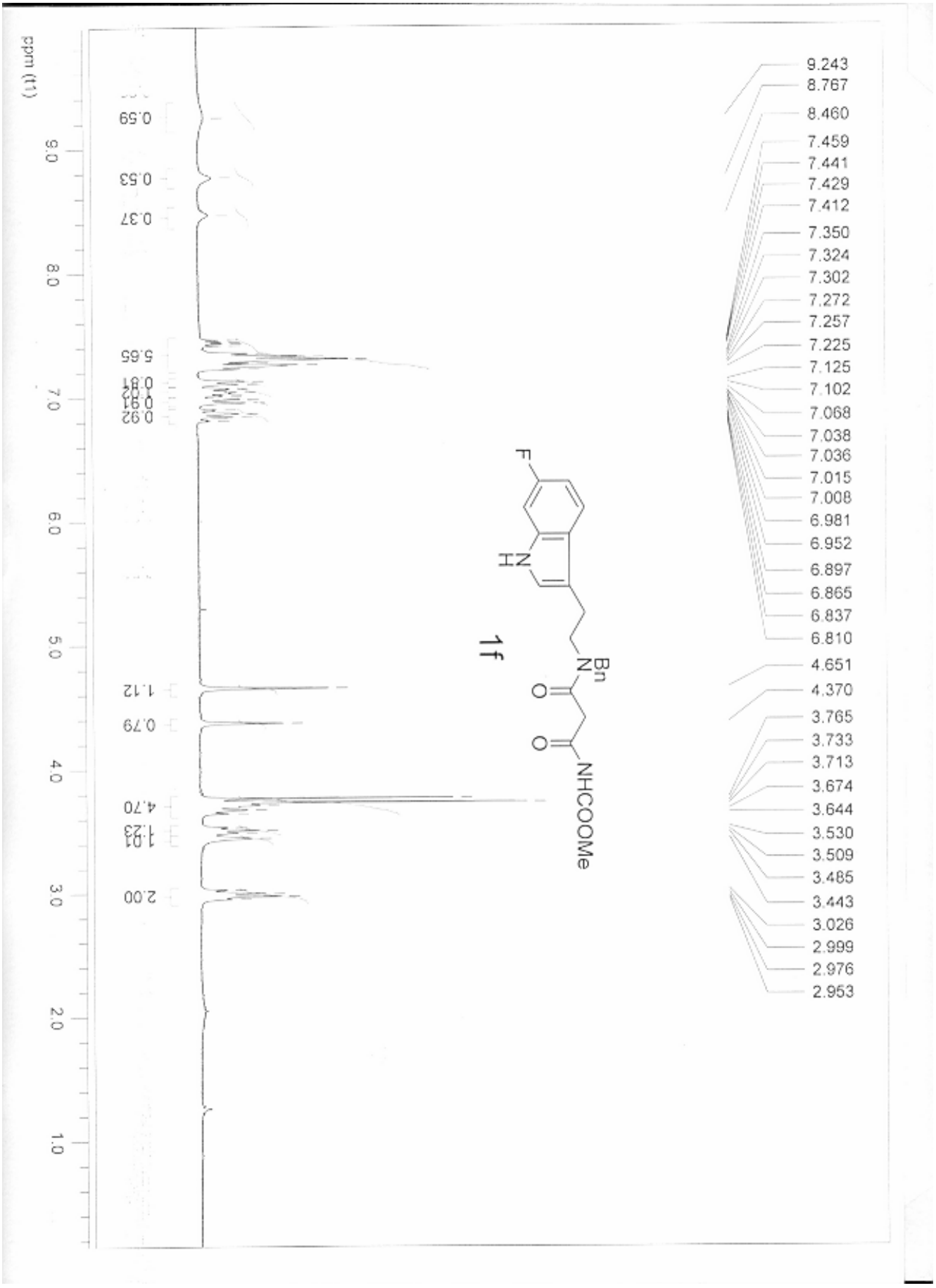
5.0
0.0
0.32
0.44
8.48
8.35
2.04
12.30
1.01
0.74
0.73
1.04
0.81
1.92
1.18
3.16
11.640
11.250
8.290
8.237
7.980
7.953
7.928
7.901
7.528
7.503
7.410
7.384
7.291
7.273
7.251
7.191
7.168
7.116
7.093
7.075
6.972
6.895
4.605
4.272
3.704
3.682
3.658
3.448
3.426
3.404
3.245
3.026
3.002
2.979
2.977
2.920
2.898
2.875
2.385
1e
H
N
Bn
N
O
O
NHTs


200
150
100
50
0
ppm (t1)
167.744
167.715
164.808
164.693
144.962
144.902
136.373
136.312
136.282
135.857
135.806
135.376
129.547
129.507
129.098
128.818
128.422
128.380
128.078
127.985
127.815
127.236
126.720
126.267
122.741
122.455
122.382
122.062
119.709
119.422
118.439
117.946
112.158
111.788
111.496
111.119
52.179
49.063
48.385
48.196
39.860
39.444
24.148
23.301
21.668
1e
H
N
Bn
N
O
O
NHTs


ppm (t1)
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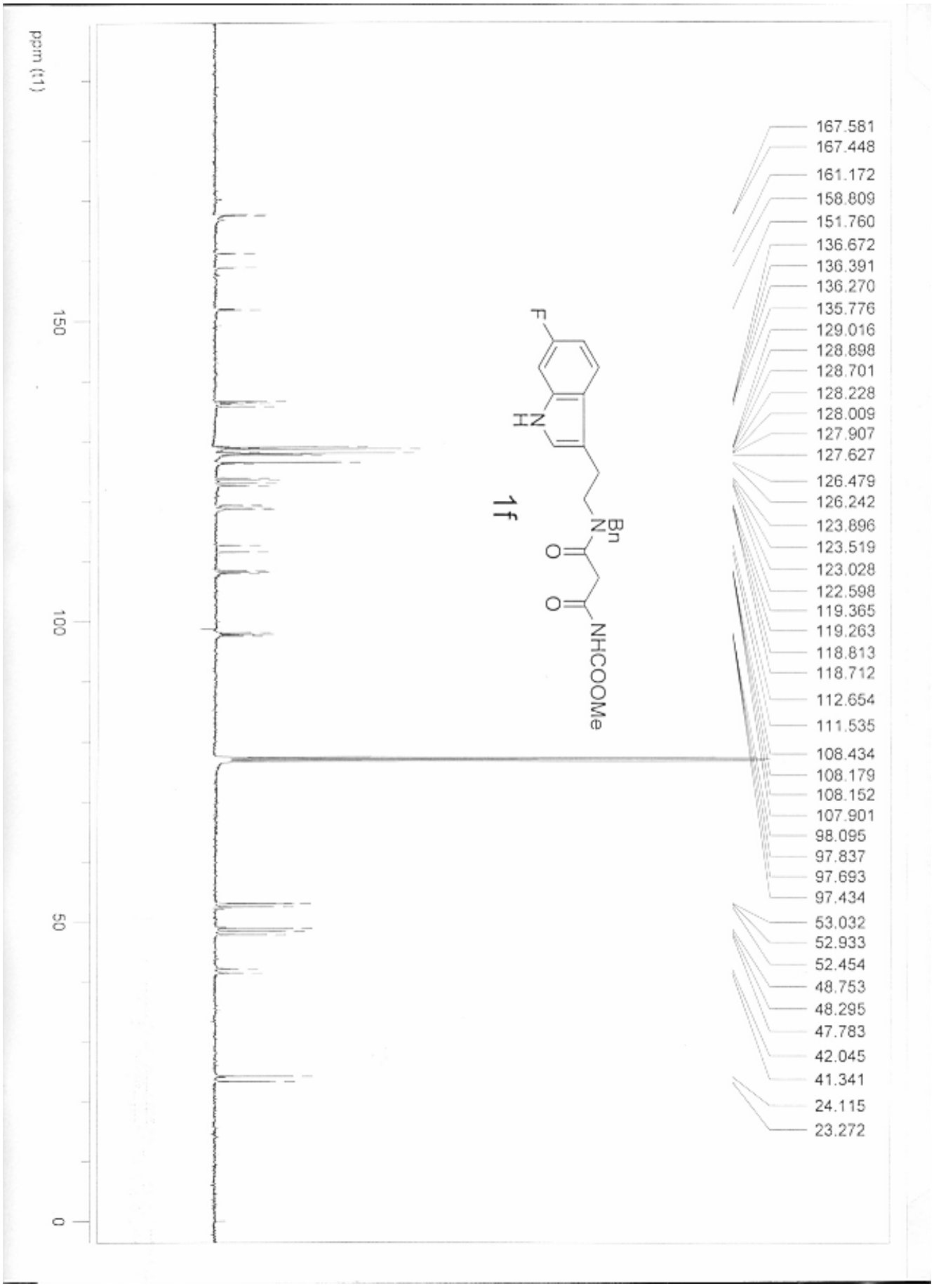
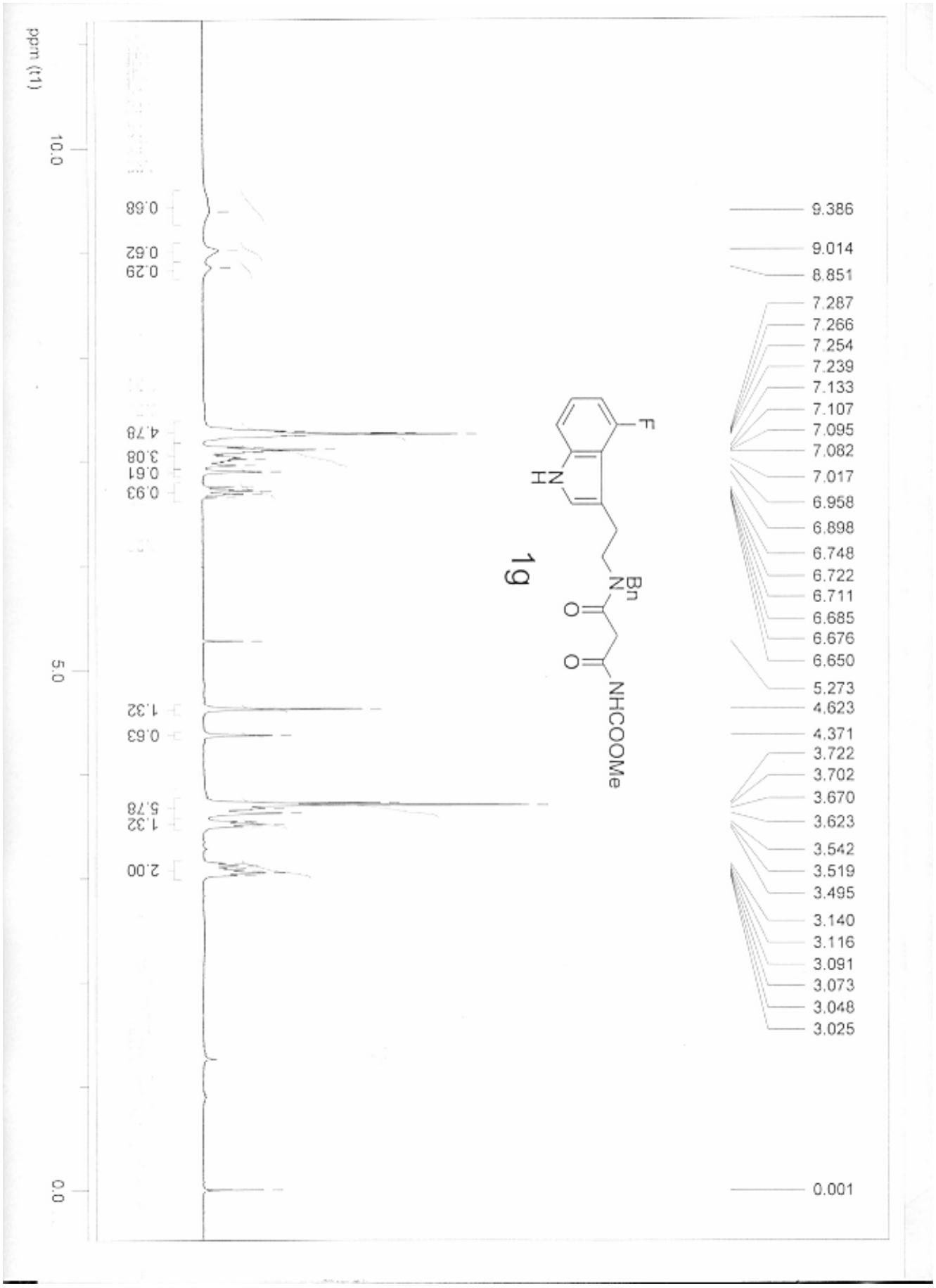
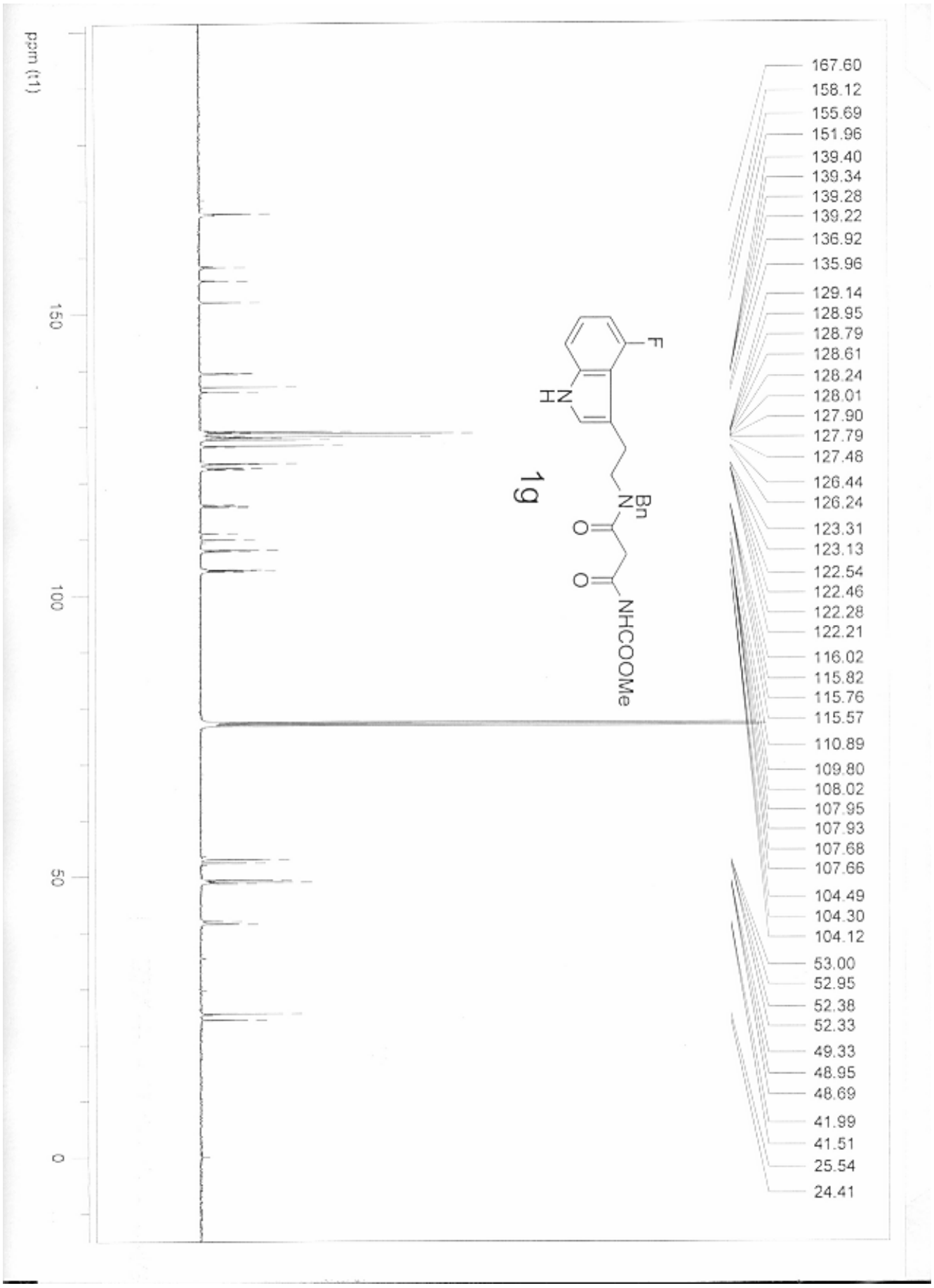
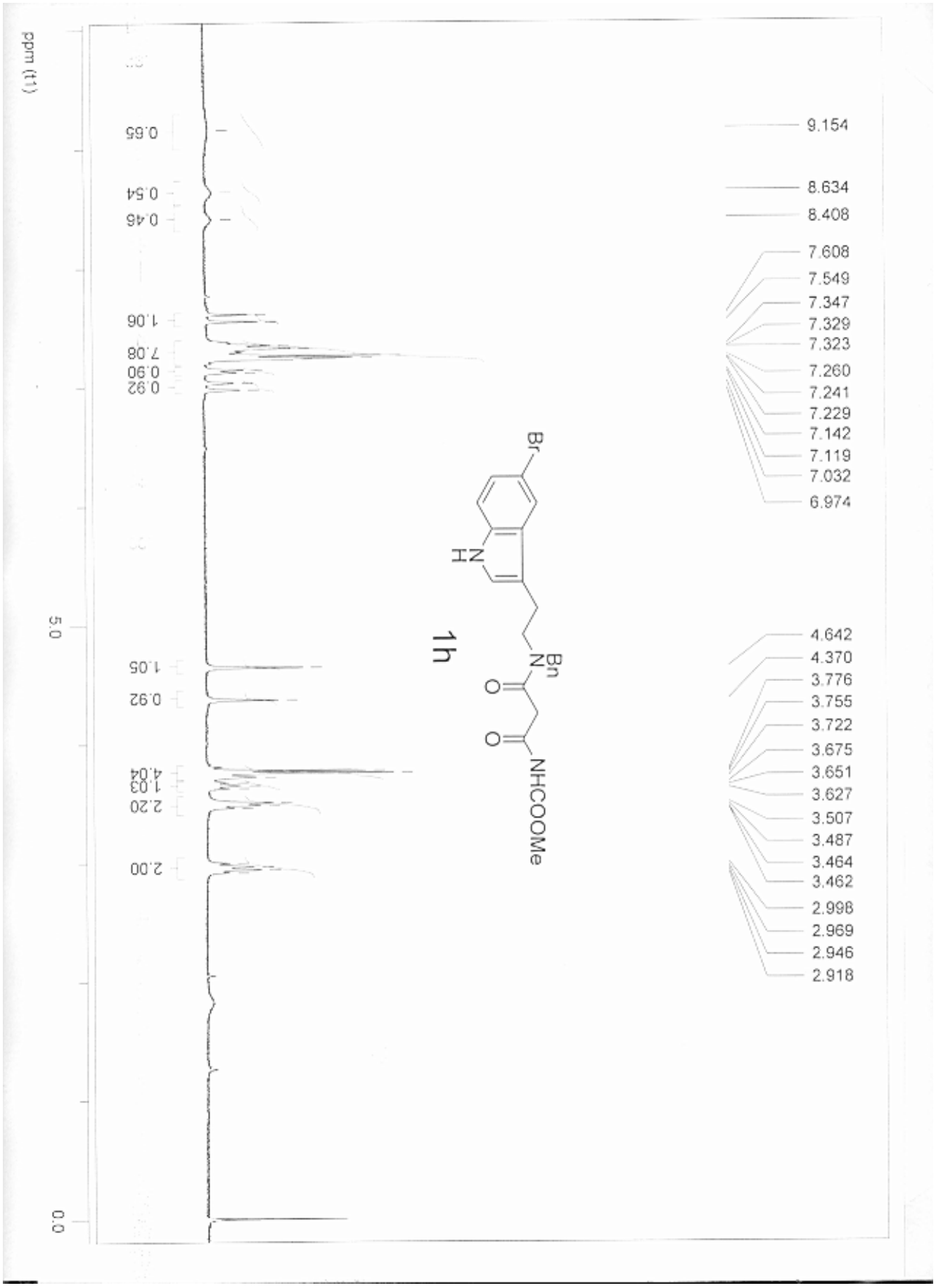
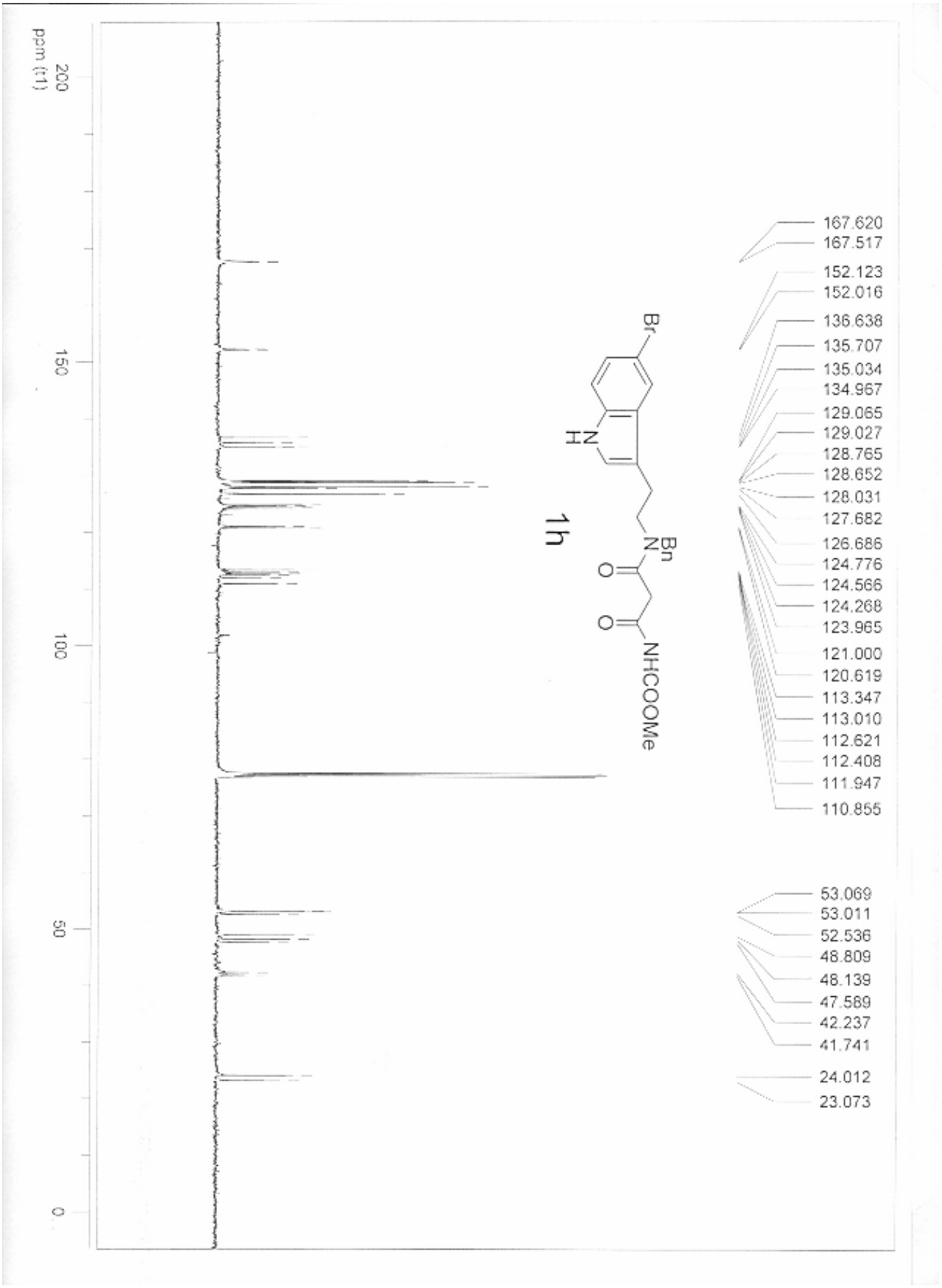
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.59
0.53
0.37
5.65
0.91
0.92
1.12
0.79
4.70
1.01
1.23
2.00
F
N
H
Bn
N
O
O
NHCOOMe
1f
9.243
8.767
8.460
7.459
7.441
7.429
7.412
7.350
7.324
7.302
7.272
7.257
7.225
7.125
7.102
7.068
7.038
7.036
7.015
7.008
6.981
6.952
6.897
6.865
6.837
6.810
4.651
4.370
3.765
3.733
3.713
3.674
3.644
3.530
3.509
3.485
3.443
3.026
2.999
2.976
2.953

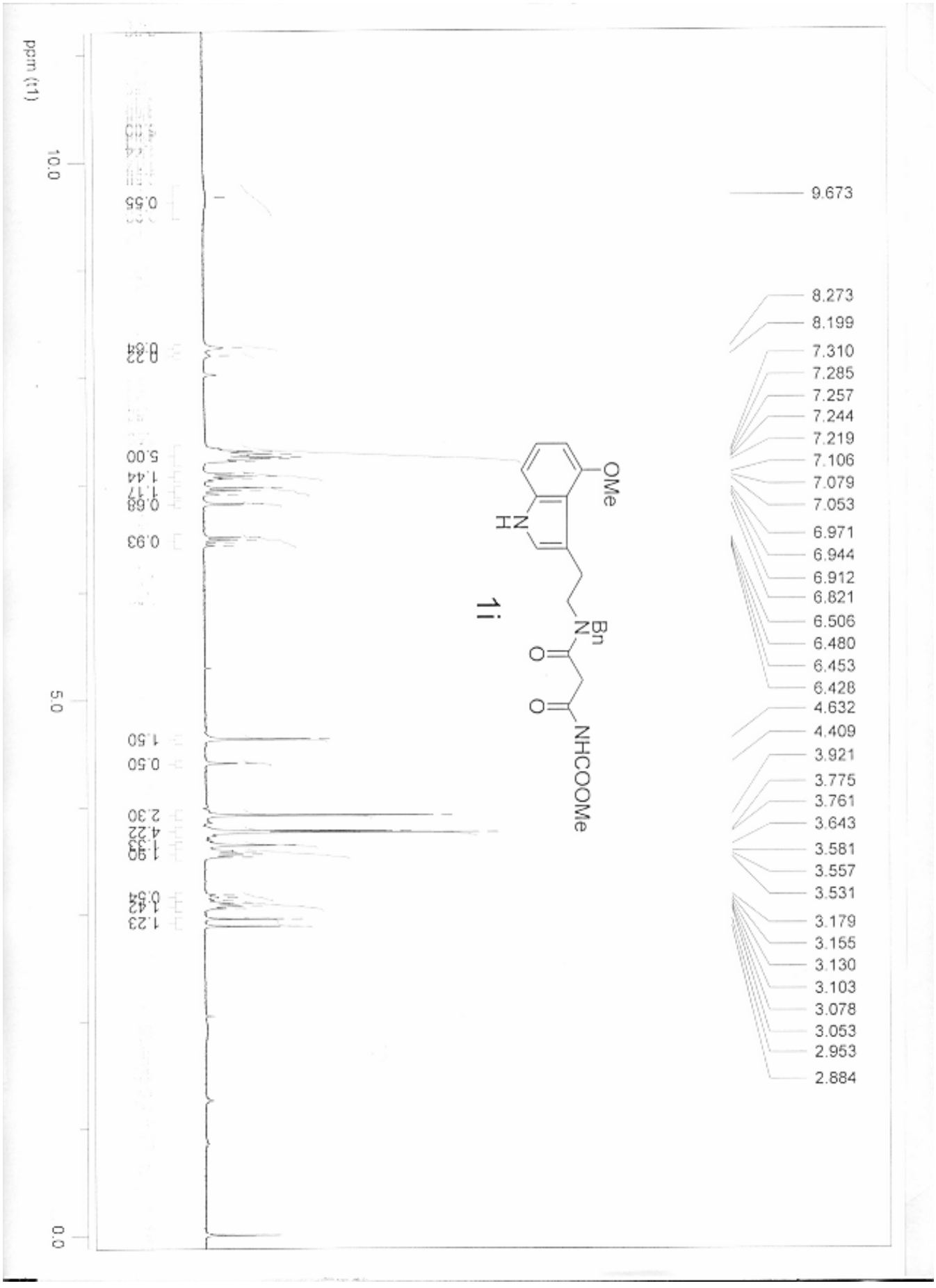

ppm (t1)
150
100
50
0
F
N
H
Bn
N
O
O
NHCOOMe
1f
167.581
167.448
161.172
158.809
151.760
136.672
136.391
136.270
135.776
129.016
128.898
128.701
128.228
128.009
127.907
127.627
126.479
126.242
123.896
123.519
123.028
122.598
119.365
119.263
118.813
118.712
112.654
111.535
108.434
108.179
108.152
107.901
98.095
97.837
97.693
97.434
53.032
52.933
52.454
48.753
48.295
47.783
42.045
41.341
24.115
23.272

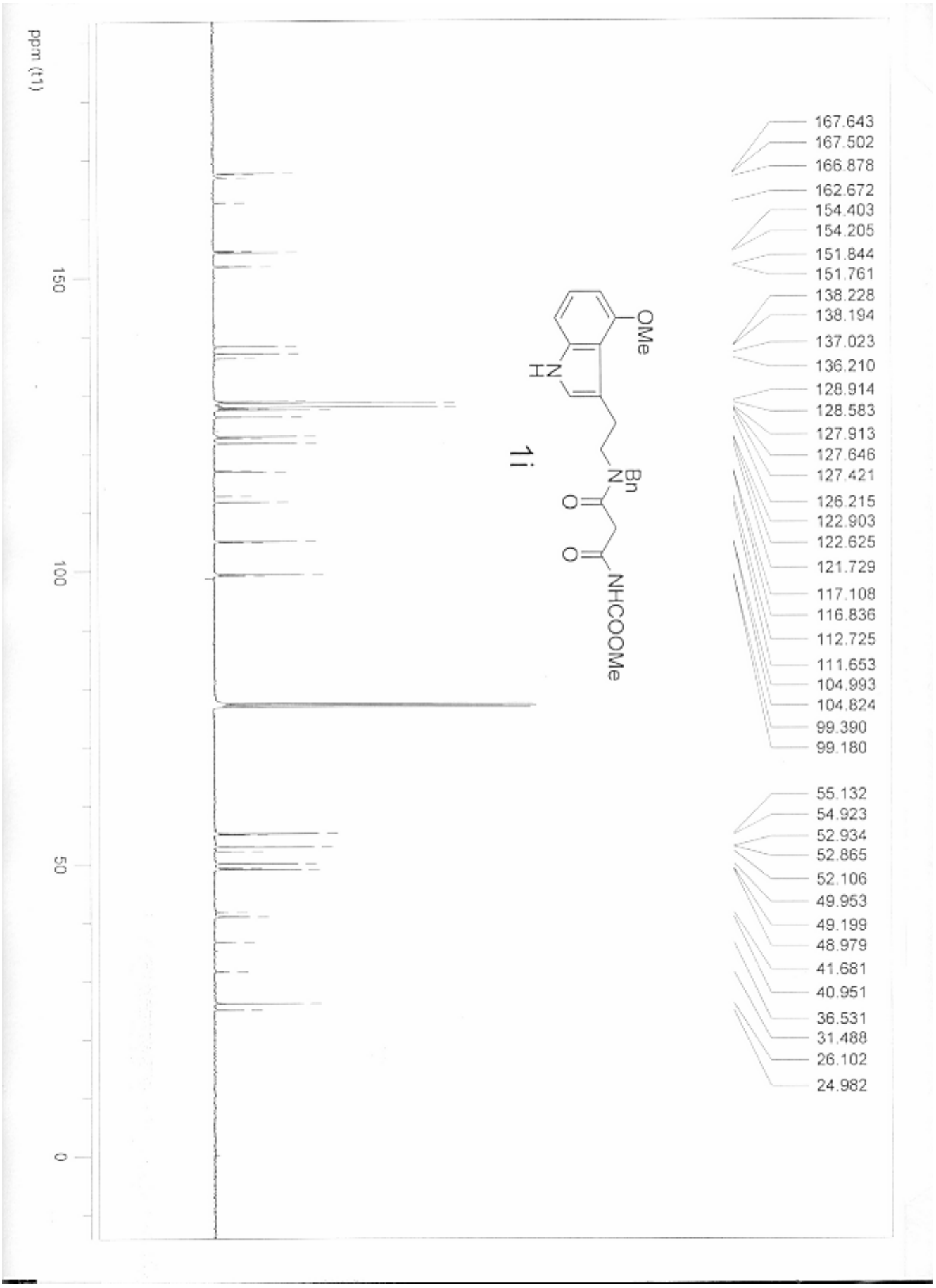

ppm (t1)
10.0
5.0
0.0
0.68
0.62
0.29
4.78
3.08
0.61
0.93
1.32
0.63
5.78
1.32
2.00
F
N
H
Bn
N
O
O
NHCOOMe
1g
9.386
9.014
8.851
7.287
7.266
7.254
7.239
7.133
7.107
7.095
7.082
7.017
6.958
6.898
6.748
6.722
6.711
6.685
6.676
6.650
5.273
4.623
4.371
3.722
3.702
3.670
3.623
3.542
3.519
3.495
3.140
3.116
3.091
3.073
3.048
3.025
0.001

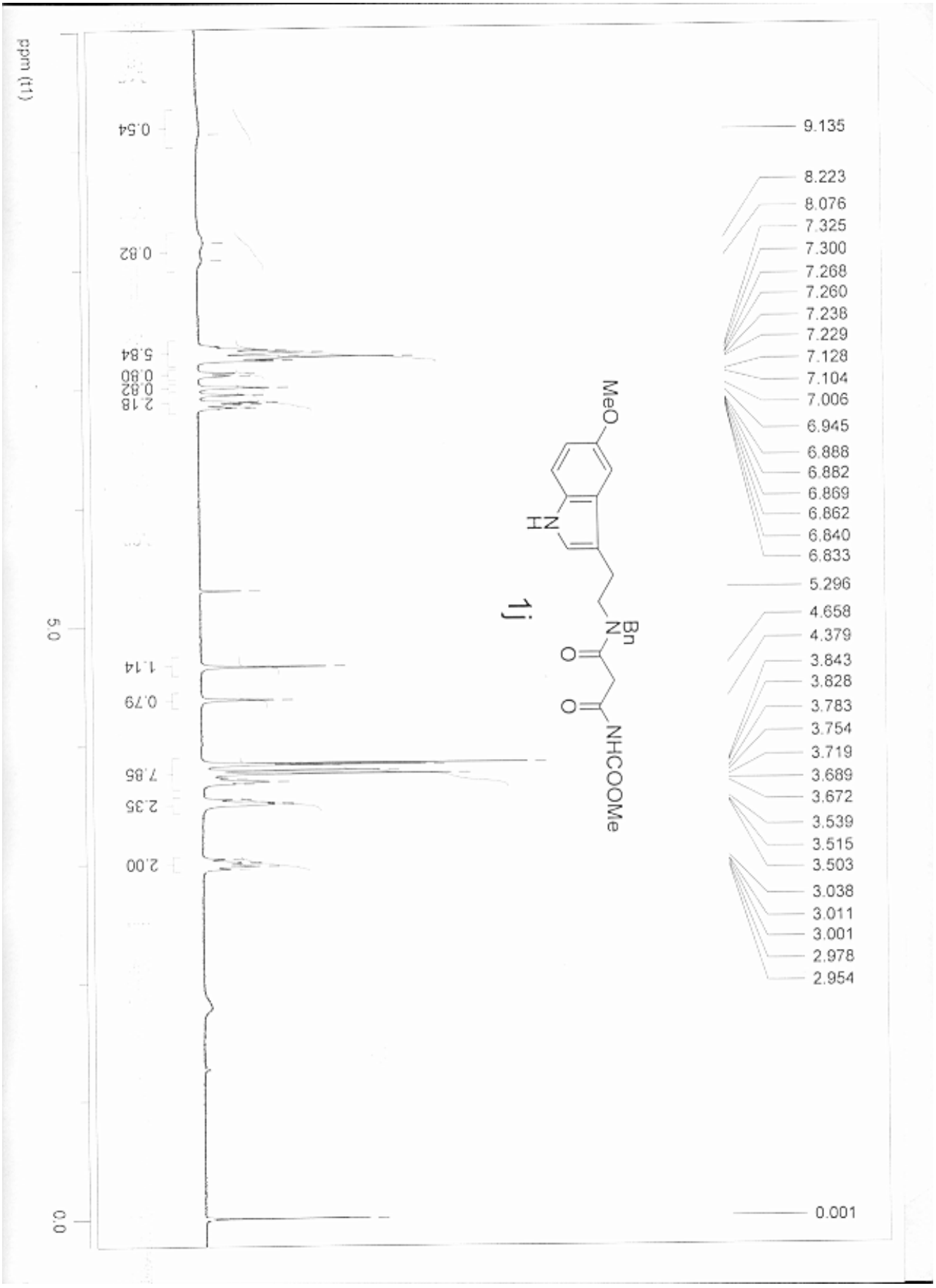

ppm (t1)
150
100
50
0
167.60
158.12
155.69
151.96
139.40
139.34
139.28
139.22
136.92
135.96
129.14
128.95
128.79
128.61
128.24
128.01
127.90
127.79
127.48
126.44
126.24
123.31
123.13
122.54
122.46
122.28
122.21
116.02
115.82
115.76
115.57
110.89
109.80
108.02
107.95
107.93
107.68
107.66
104.49
104.30
104.12
53.00
52.95
52.38
52.33
49.33
48.95
48.69
41.99
41.51
25.54
24.41
F
H
N
Bn
N
O
O
NHCOOMe
1g

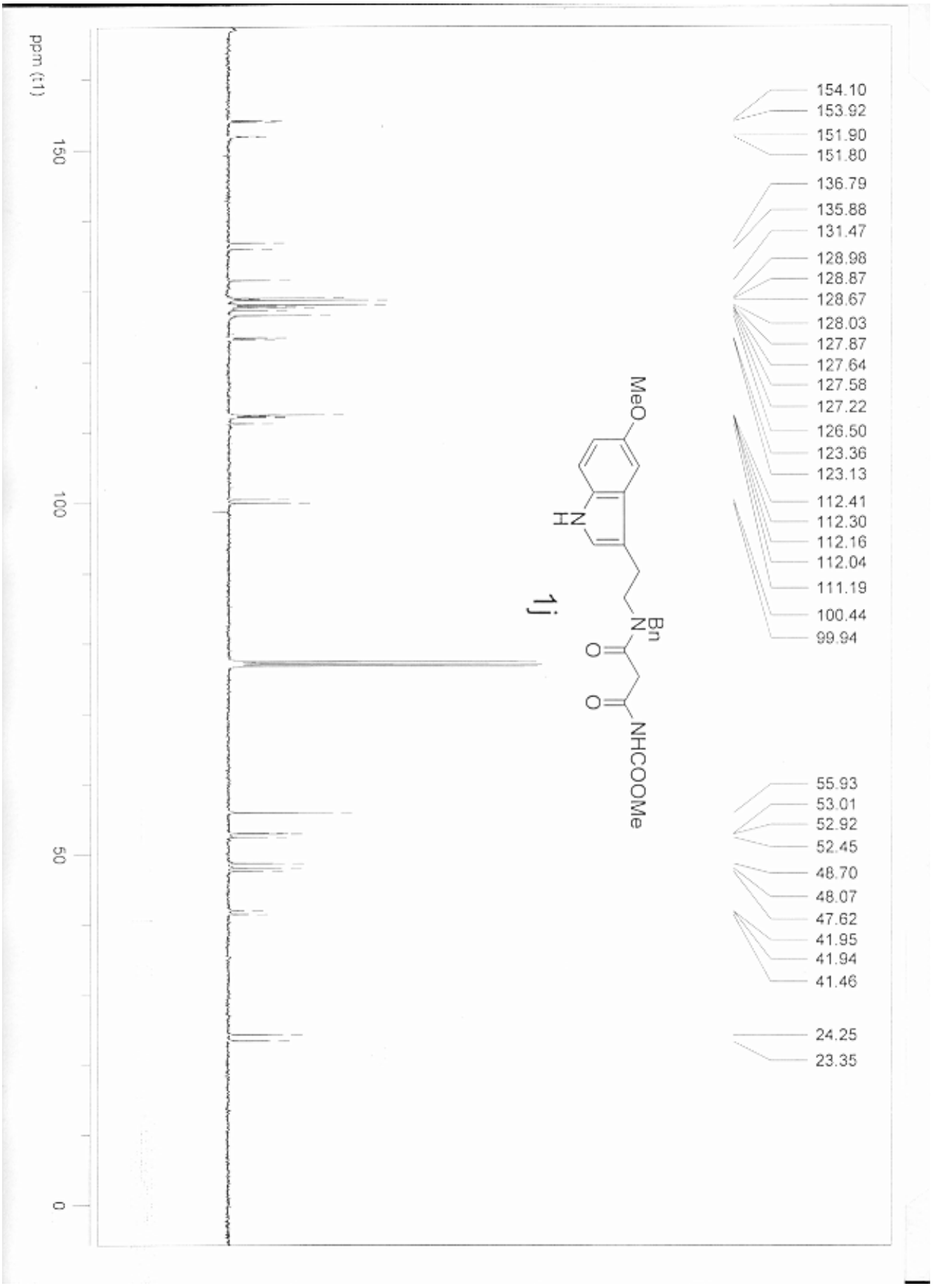

ppm (t1)
9.154
8.634
8.408
7.608
7.549
7.347
7.329
7.323
7.260
7.241
7.229
7.142
7.119
7.032
6.974
4.642
4.370
3.776
3.755
3.722
3.675
3.651
3.627
3.507
3.487
3.464
3.462
2.998
2.969
2.946
2.918
0.65
0.54
0.46
1.06
7.08
0.90
0.92
1.05
0.92
4.04
1.03
2.20
2.00
Br
N
H
Bn
N
O
O
NHCOOMe
1h
5.0
0.0

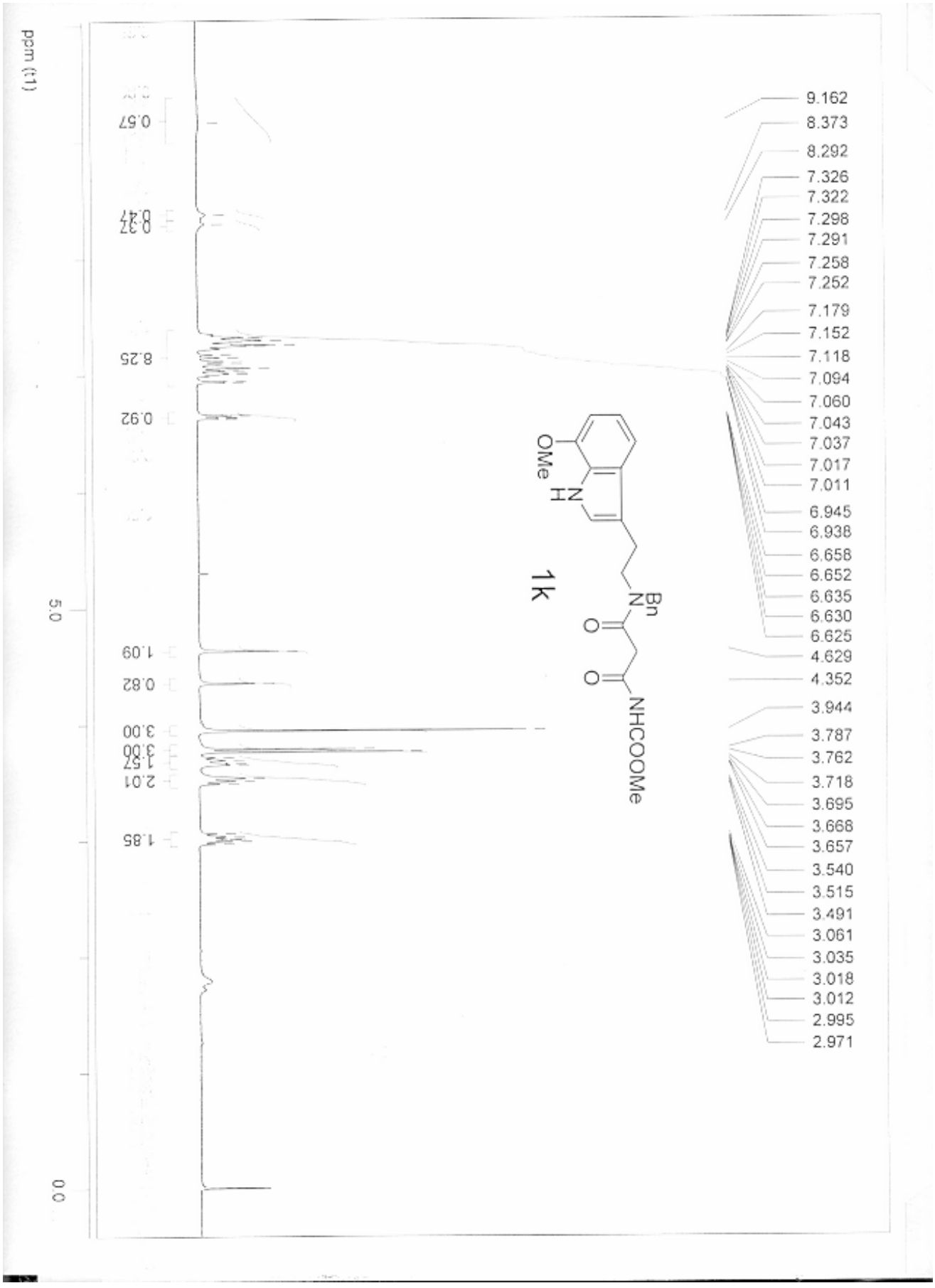

ppm (t1)
200
150
100
50
0
Br
N
H
N
Bn
O
O
O
NHCOOMe
1h
167.620
167.517
152.123
152.016
136.638
135.707
135.034
134.967
129.065
129.027
128.765
128.652
128.031
127.682
126.686
124.776
124.566
124.268
123.965
121.000
120.619
113.347
113.010
112.621
112.408
111.947
110.855
53.069
53.011
52.536
48.809
48.139
47.589
42.237
41.741
24.012
23.073


ppm (t1)
10.0
5.0
0.0
0.55
0.84
0.93
0.69
1.17
1.44
5.00
1.50
0.50
2.30
4.22
1.99
0.54
1.42
1.23
OMe
N
H
Bn
N
O
O
NHCOOMe
1i
9.673
8.273
8.199
7.310
7.285
7.257
7.244
7.219
7.106
7.079
7.053
6.971
6.944
6.912
6.821
6.506
6.480
6.453
6.428
4.632
4.409
3.921
3.775
3.761
3.643
3.581
3.557
3.531
3.179
3.155
3.130
3.103
3.078
3.053
2.953
2.884


ppm (t1)
150
100
50
0
OMe
N
H
Bn
N
O
O
NHCOOMe
1i
167.643
167.502
166.878
162.672
154.403
154.205
151.844
151.761
138.228
138.194
137.023
136.210
128.914
128.583
127.913
127.646
127.421
126.215
122.903
122.625
121.729
117.108
116.836
112.725
111.653
104.993
104.824
99.390
99.180
55.132
54.923
52.934
52.865
52.106
49.953
49.199
48.979
41.681
40.951
36.531
31.488
26.102
24.982


f1 (ppm)
5.0
0.0
0.54
0.82
5.84
0.80
0.82
2.18
1.14
0.79
7.85
2.35
2.00
MeO
H
N
Bn
N
O
O
NHCOOMe
1j
9.135
8.223
8.076
7.325
7.300
7.268
7.260
7.238
7.229
7.128
7.104
7.006
6.945
6.888
6.882
6.869
6.862
6.840
6.833
5.296
4.658
4.379
3.843
3.828
3.783
3.754
3.719
3.689
3.672
3.539
3.515
3.503
3.038
3.011
3.001
2.978
2.954
0.001


ppm (t1)
150
100
50
0
154.10
153.92
151.90
151.80
136.79
135.88
131.47
128.98
128.87
128.67
128.03
127.87
127.64
127.58
127.22
126.50
123.36
123.13
112.41
112.30
112.16
112.04
111.19
100.44
99.94
55.93
53.01
52.92
52.45
48.70
48.07
47.62
41.95
41.94
41.46
24.25
23.35
MeO
N
H
Bn
N
O
O
NHCOOMe
1j


ppm (t1)
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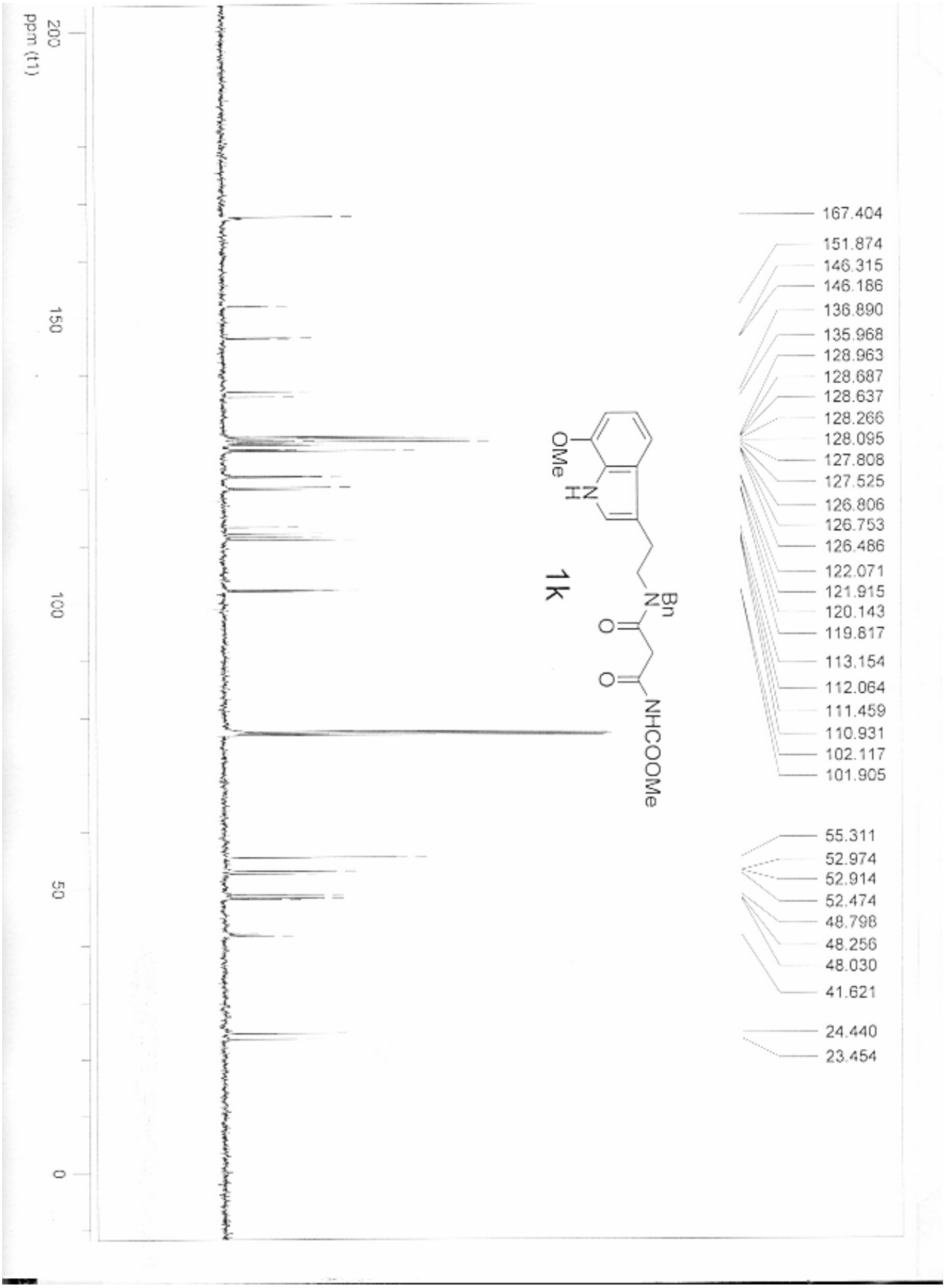
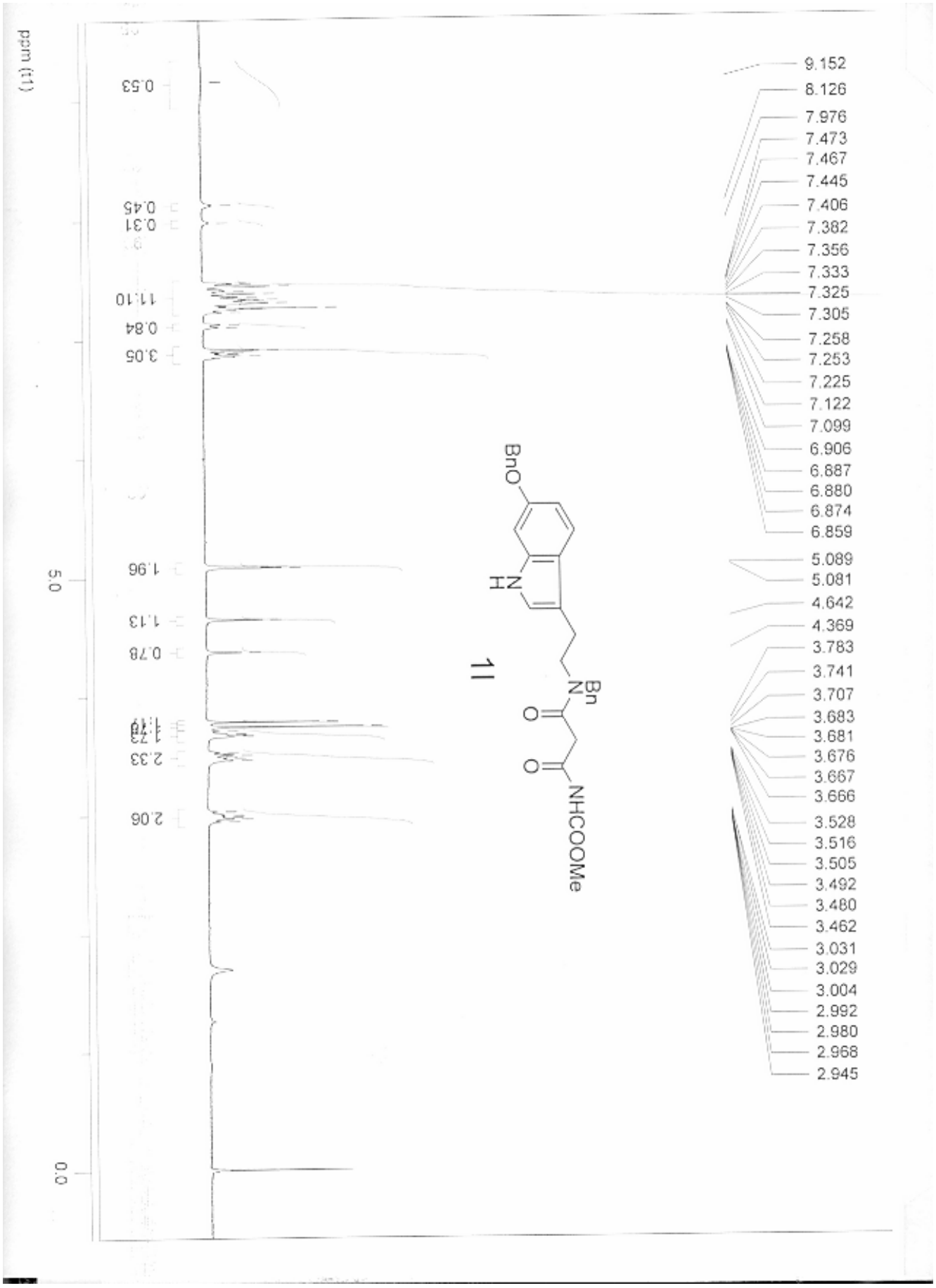
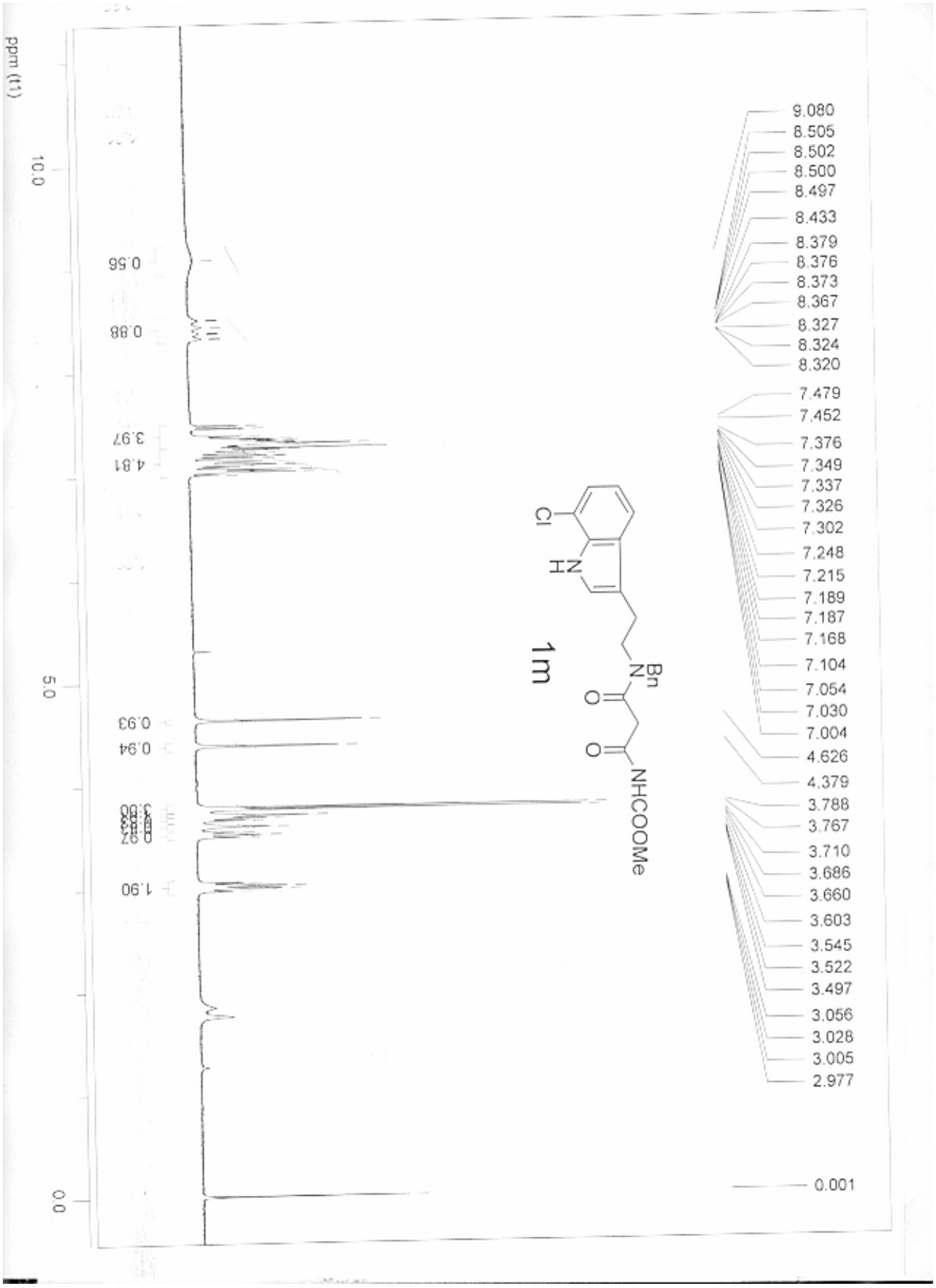
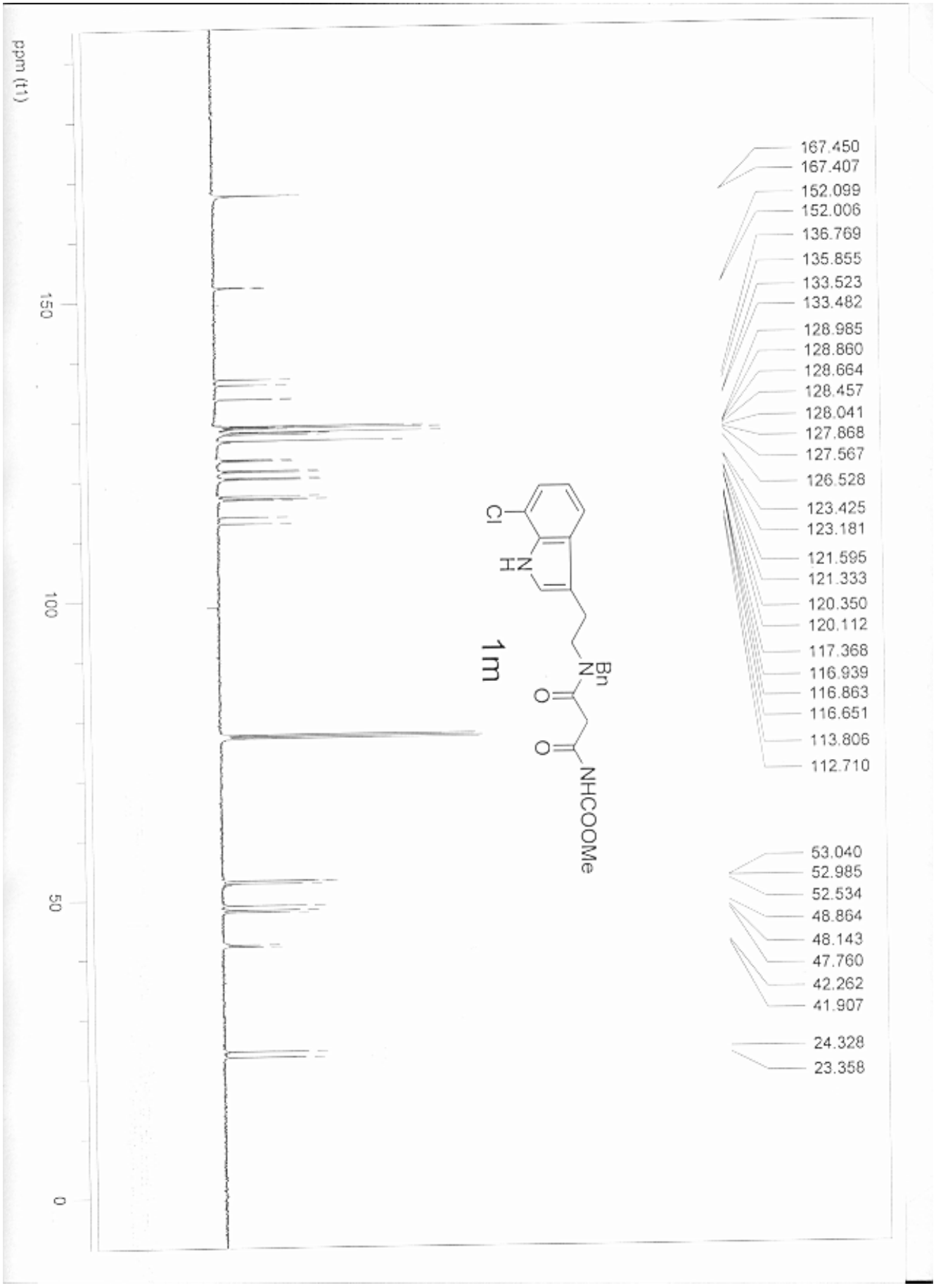
0.0
0.57
0.37
0.47
8.25
0.92
1.09
0.82
3.00
3.00
1.57
2.01
1.85
9.162
8.373
8.292
7.326
7.322
7.298
7.291
7.258
7.252
7.179
7.152
7.118
7.094
7.060
7.043
7.037
7.017
7.011
6.945
6.938
6.658
6.652
6.635
6.630
6.625
4.629
4.352
3.944
3.787
3.762
3.718
3.695
3.668
3.657
3.540
3.515
3.491
3.061
3.035
3.018
3.012
2.995
2.971
OMe
N
H
1k
Bn
N
O
O
NHCOOMe

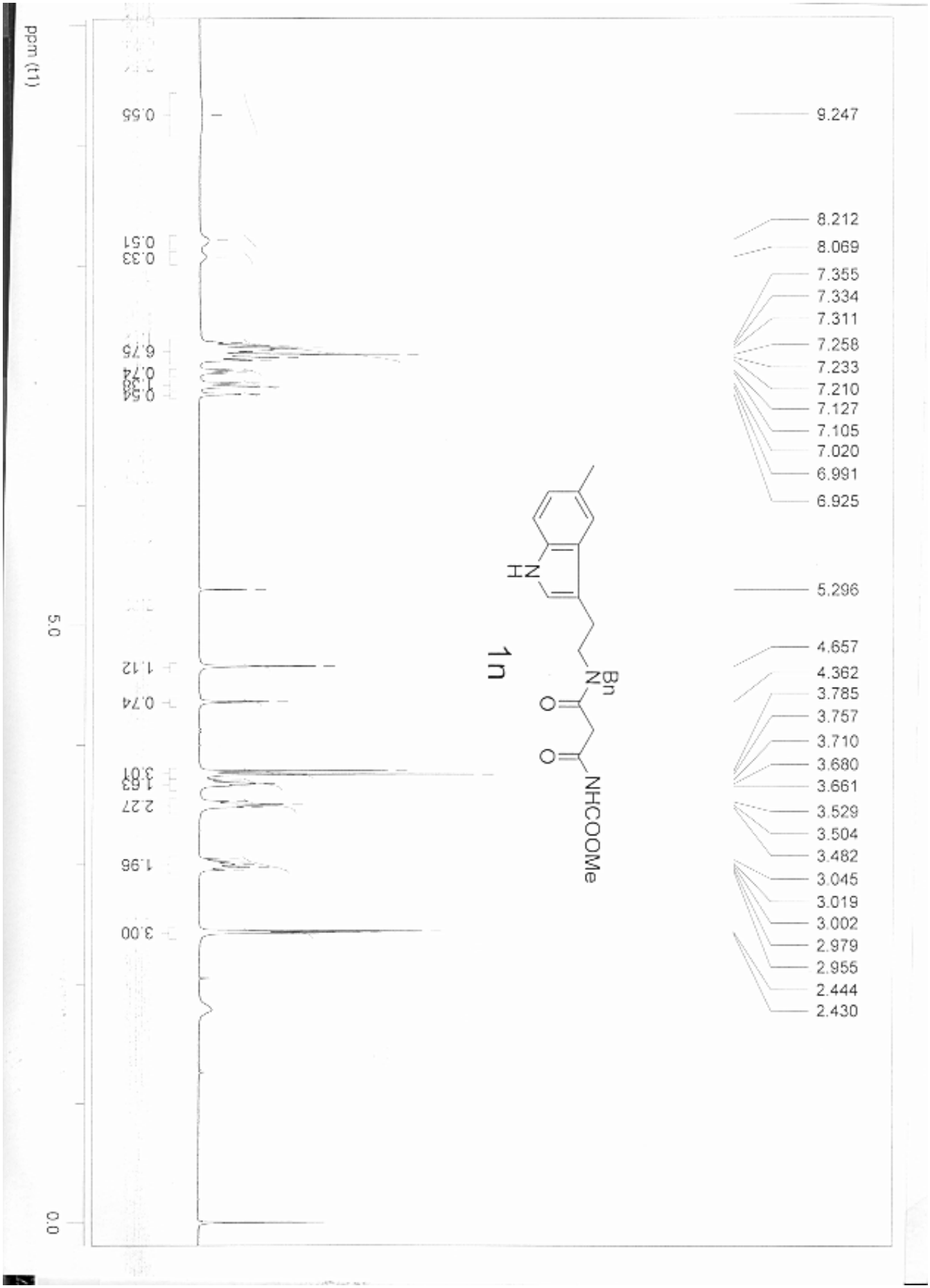

ppm (t1)
200
150
100
50
0
OMe
H
N
1k
Bn
N
O
O
NHCOOMe
167.404
151.874
146.315
146.186
136.890
135.968
128.963
128.687
128.637
128.266
128.095
127.808
127.525
126.806
126.753
126.486
122.071
121.915
120.143
119.817
113.154
112.064
111.459
110.931
102.117
101.905
55.311
52.974
52.914
52.474
48.798
48.256
48.030
41.621
24.440
23.454

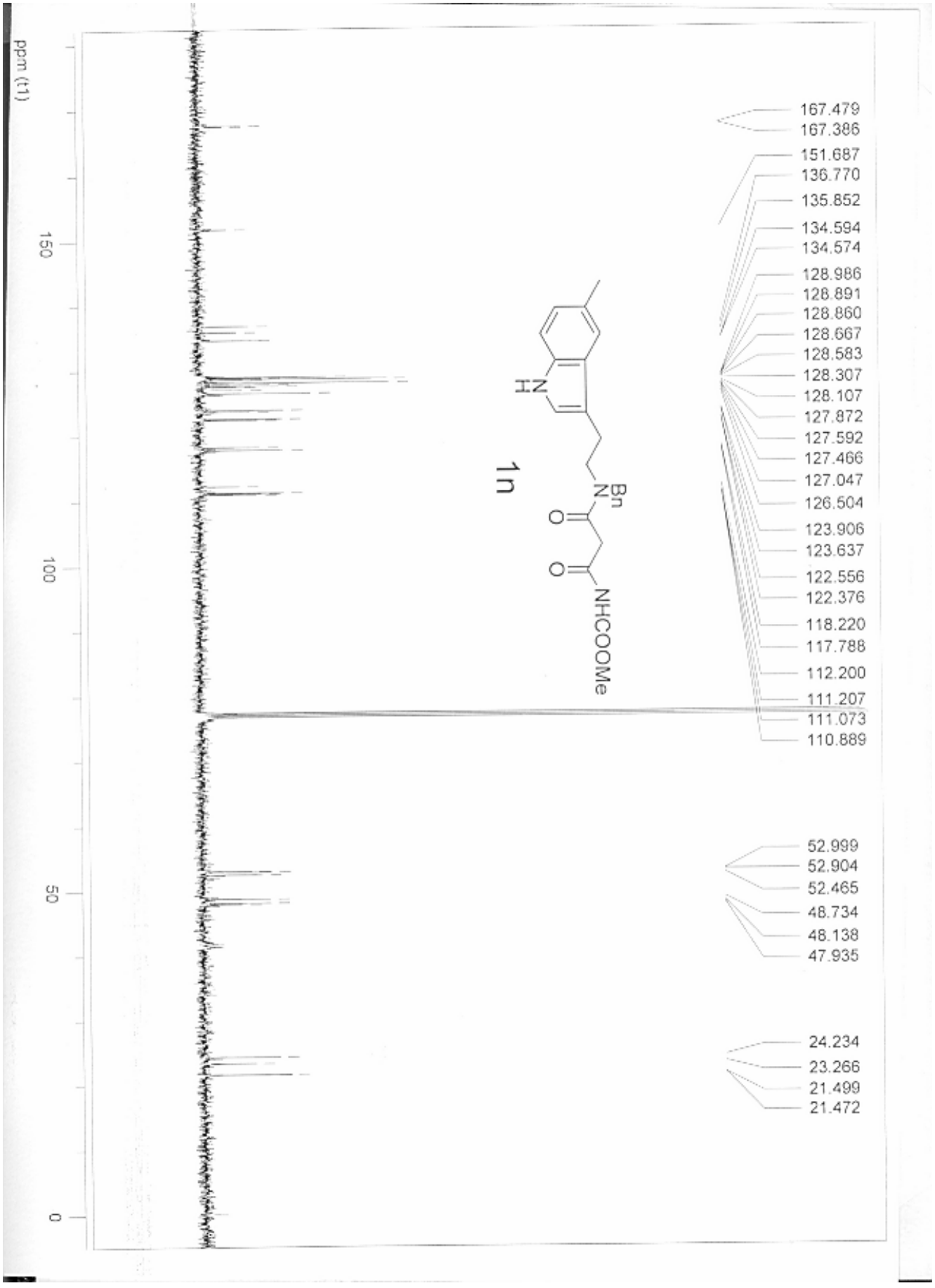

ppm (t1)
5.0
0.0
0.53
0.45
0.31
11.10
0.84
3.05
1.96
1.13
0.78
1.73
2.33
2.06
BnO
N
H
Bn
N
O
O
NHCOOMe
11
9.152
8.126
7.976
7.473
7.467
7.445
7.406
7.382
7.356
7.333
7.325
7.305
7.258
7.253
7.225
7.122
7.099
6.906
6.887
6.880
6.874
6.859
5.089
5.081
4.642
4.369
3.783
3.741
3.707
3.683
3.681
3.676
3.667
3.666
3.528
3.516
3.505
3.492
3.480
3.462
3.031
3.029
3.004
2.992
2.980
2.968
2.945

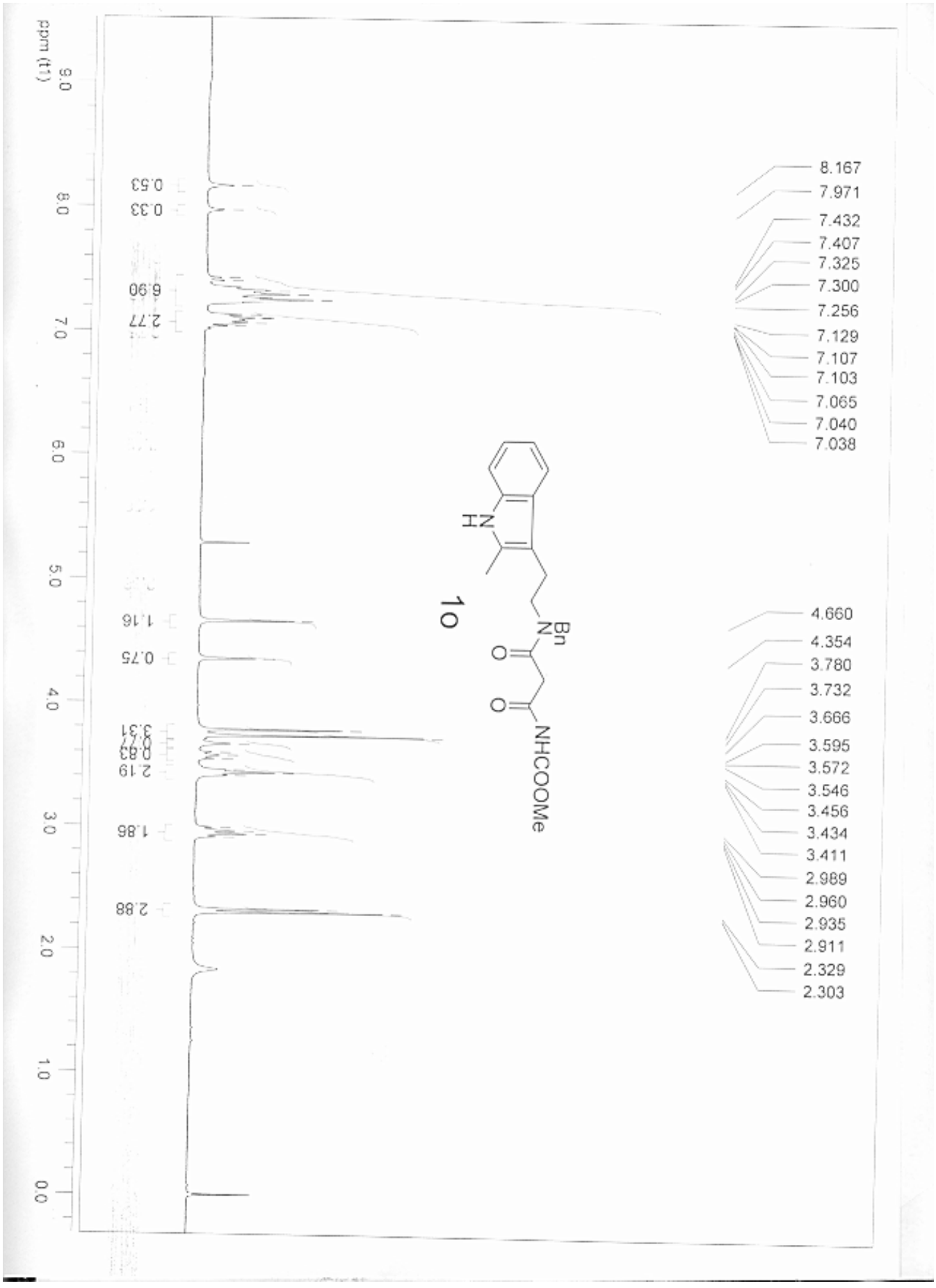

ppm (t1)
200
150
100
50
0
167.612
167.450
155.681
155.547
151.859
137.467
137.392
137.091
137.014
136.813
135.918
128.993
128.683
128.530
128.046
127.843
127.534
126.531
122.021
121.627
121.570
121.355
119.209
118.690
112.438
111.389
110.320
109.988
96.449
70.595
70.509
53.005
52.919
52.447
48.739
48.309
47.917
42.042
41.468
24.246
23.407
BnO
N
H
11
N
Bn
O
O
NHCOOMe

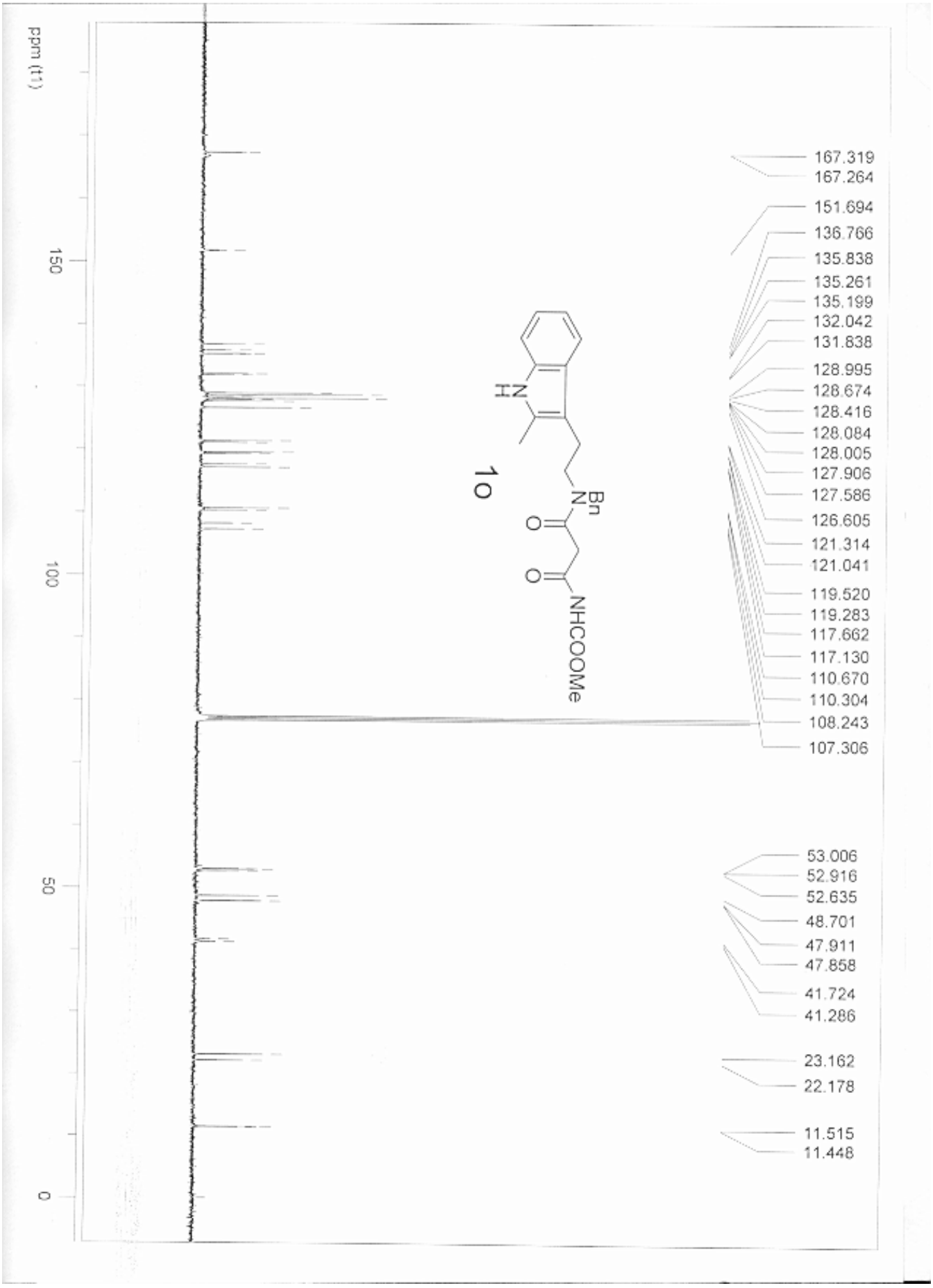

ppm (t1)
10.0
5.0
0.0
0.56
0.88
3.97
4.81
0.93
0.94
1.90
9.080
8.505
8.502
8.500
8.497
8.433
8.379
8.376
8.373
8.367
8.327
8.324
8.320
7.479
7.452
7.376
7.349
7.337
7.326
7.302
7.248
7.215
7.189
7.187
7.168
7.104
7.054
7.030
7.004
4.626
4.379
3.788
3.767
3.710
3.686
3.660
3.603
3.545
3.522
3.497
3.056
3.028
3.005
2.977
0.001
Cl
H
N
Bn
N
O
O
NHCOOMe
1m

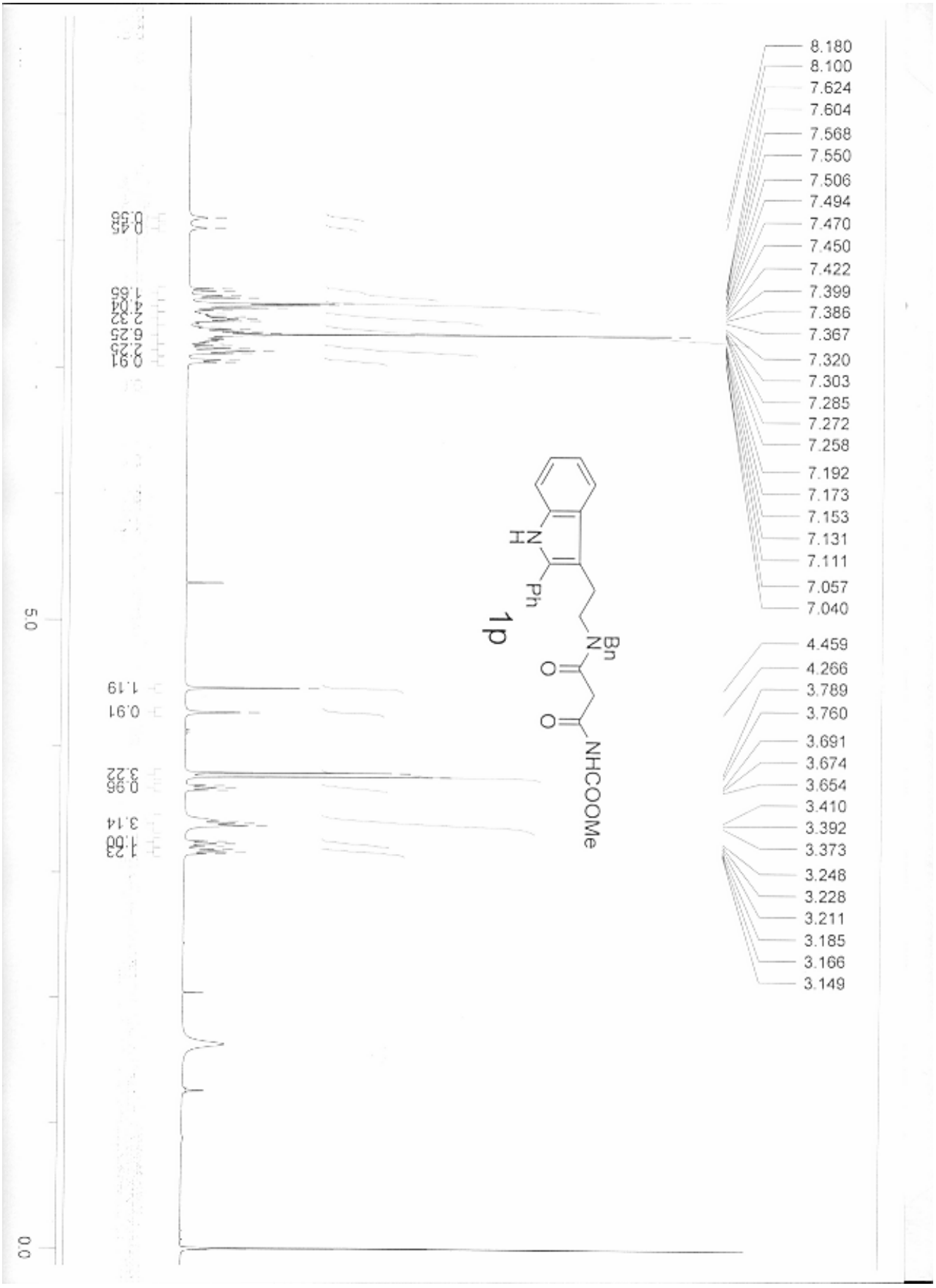

ppm (t1)
150
100
50
0
167.450
167.407
152.099
152.006
136.769
135.855
133.523
133.482
128.985
128.860
128.664
128.457
128.041
127.868
127.567
126.528
123.425
123.181
121.595
121.333
120.350
120.112
117.368
116.939
116.863
116.651
113.806
112.710
53.040
52.985
52.534
48.864
48.143
47.760
42.262
41.907
24.328
23.358
Cl
H
N
Bn
N
O
O
NHCOOMe
1m

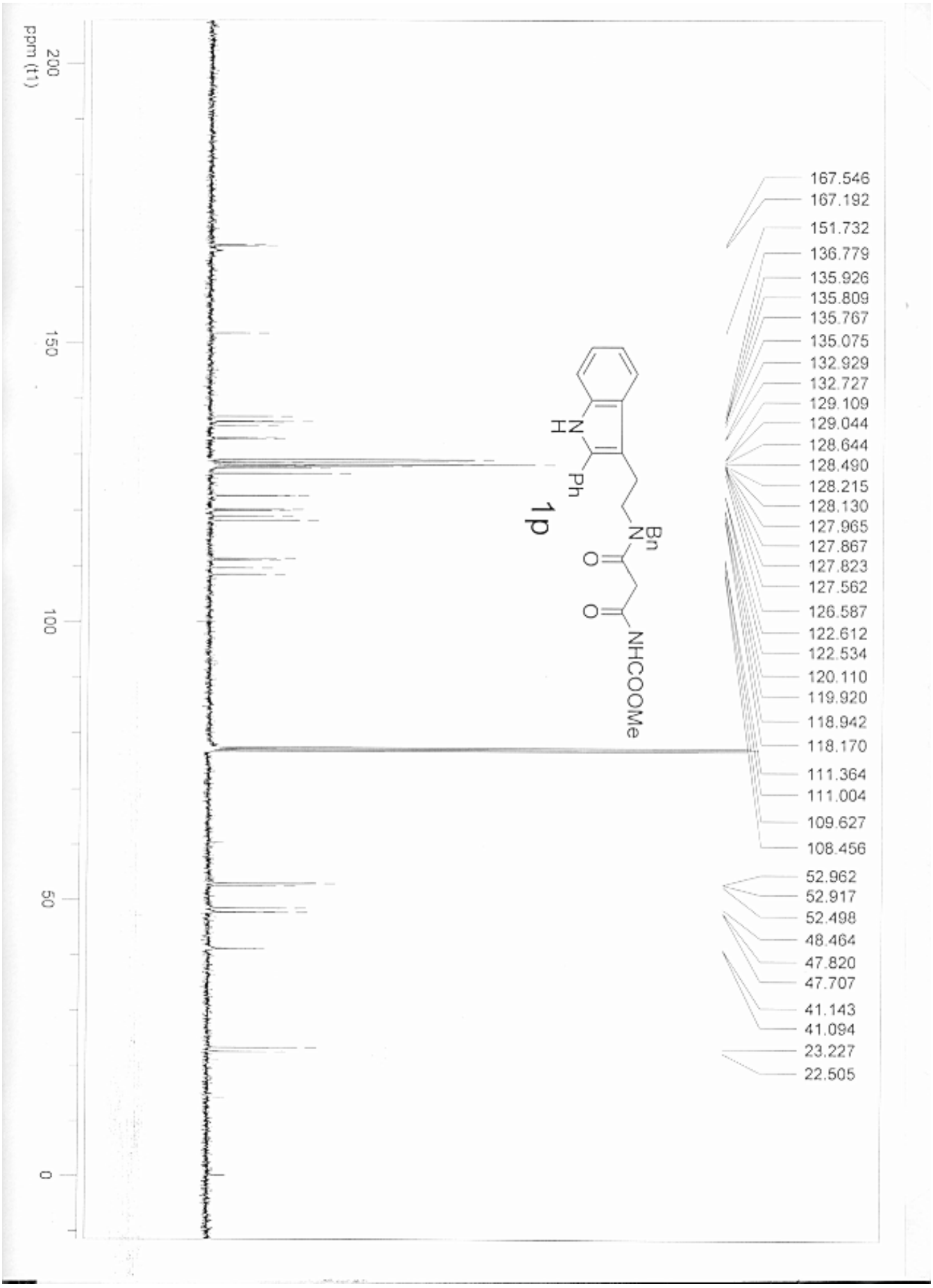

ppm (t1)
5.0
0.0
0.55
0.51
0.33
6.75
0.74
1.12
0.74
3.01
1.63
2.27
1.96
3.00
1n
Bn
N
H
O
O
NHCOOMe
9.247
8.212
8.069
7.355
7.334
7.311
7.258
7.233
7.210
7.127
7.105
7.020
6.991
6.925
5.296
4.657
4.362
3.785
3.757
3.710
3.680
3.661
3.529
3.504
3.482
3.045
3.019
3.002
2.979
2.955
2.444
2.430

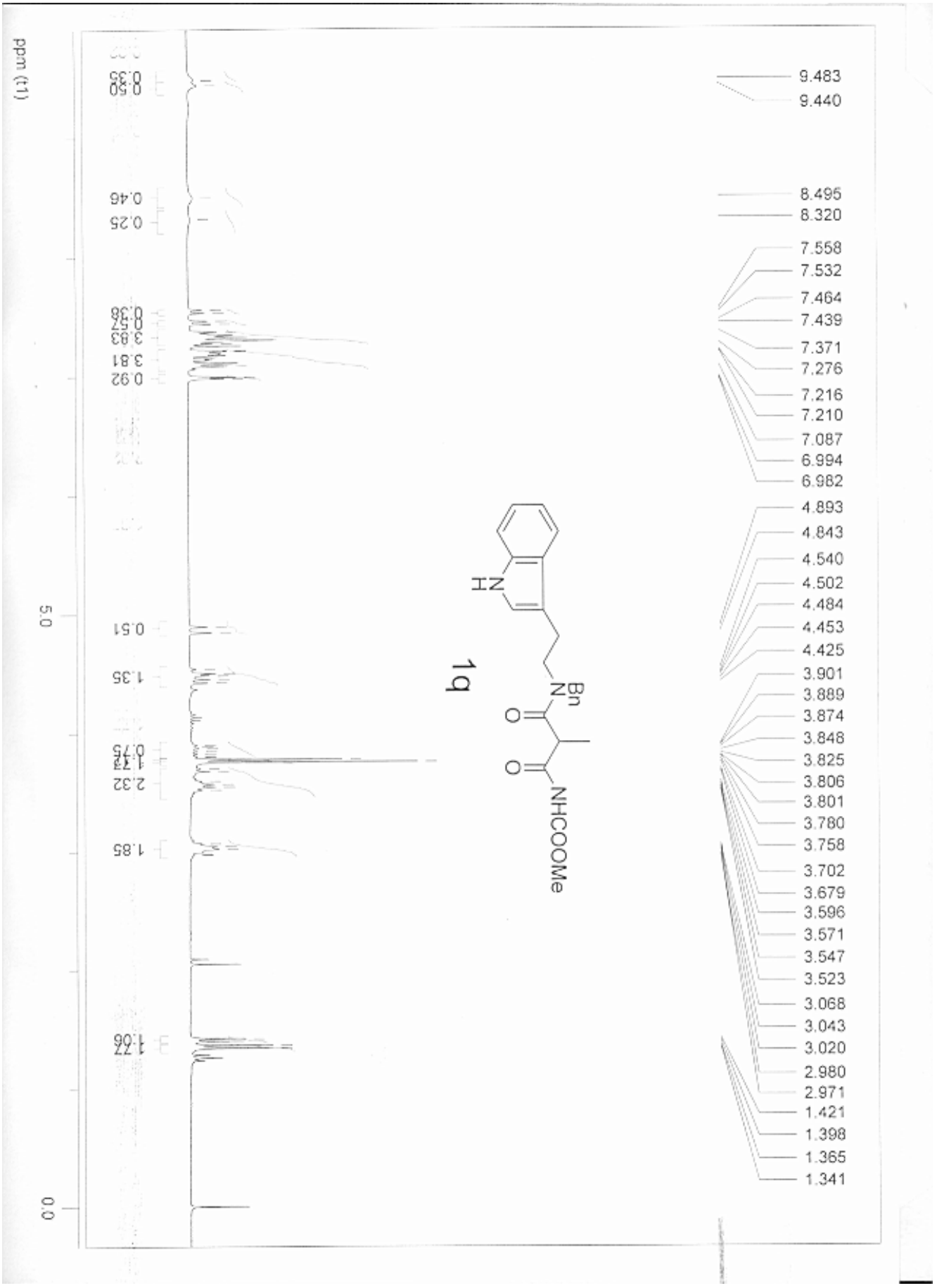

ppm (t1)
150
100
50
0
167.479
167.386
151.687
136.770
135.852
134.594
134.574
128.986
128.891
128.860
128.667
128.583
128.307
128.107
127.872
127.592
127.466
127.047
126.504
123.906
123.637
122.556
122.376
118.220
117.788
112.200
111.207
111.073
110.889
52.999
52.904
52.465
48.734
48.138
47.935
24.234
23.266
21.499
21.472
1n
Bn
N
O
O
NHCOOMe
H
N

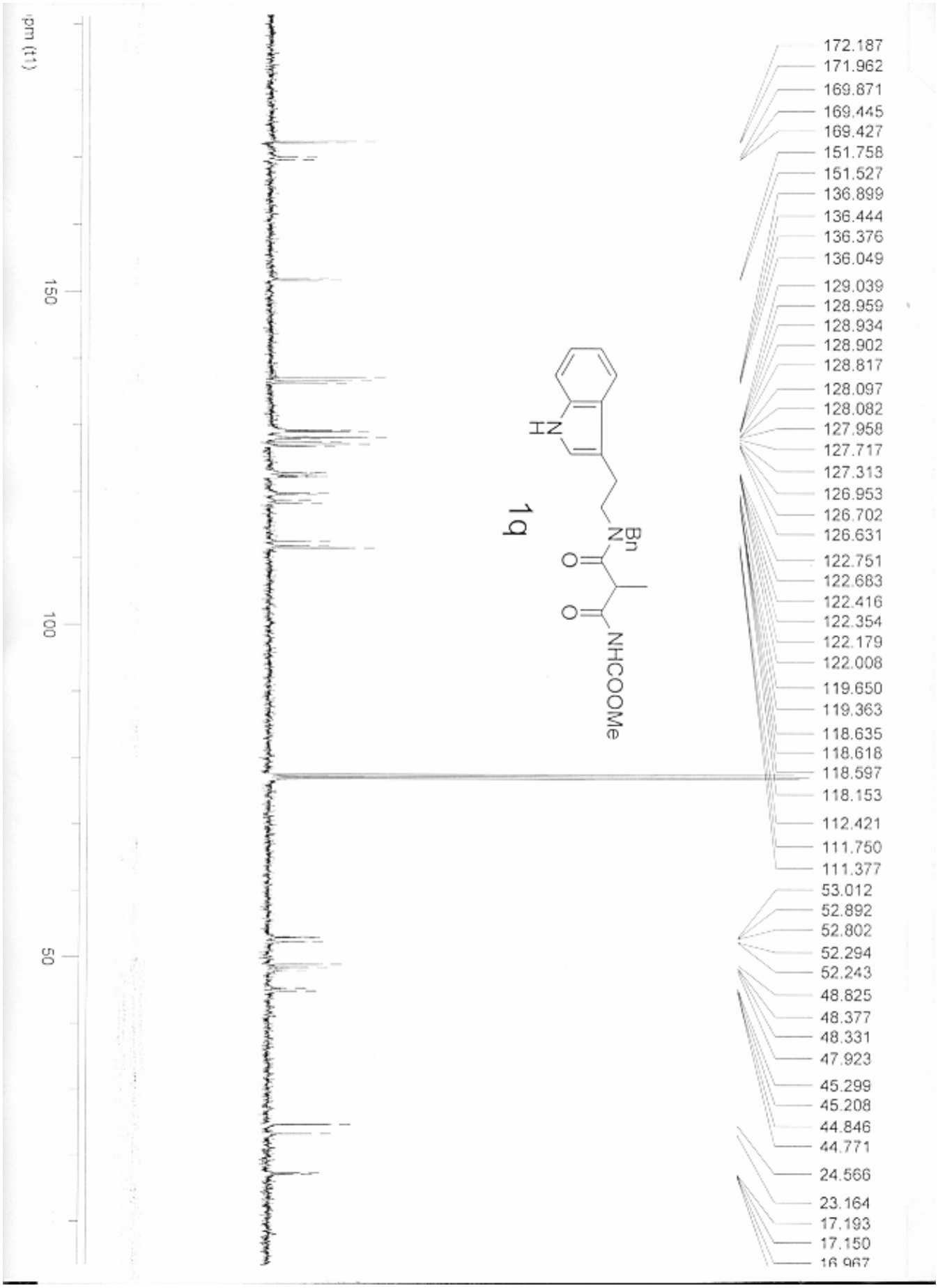

ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0.53
0.33
6.90
2.77
1.16
0.75
3.31
0.83
2.19
1.86
2.88
8.167
7.971
7.432
7.407
7.325
7.300
7.256
7.129
7.107
7.103
7.065
7.040
7.038
4.660
4.354
3.780
3.732
3.666
3.595
3.572
3.546
3.456
3.434
3.411
2.989
2.960
2.935
2.911
2.329
2.303
1o
Bn
N
O
O
NHCOOMe
H
N

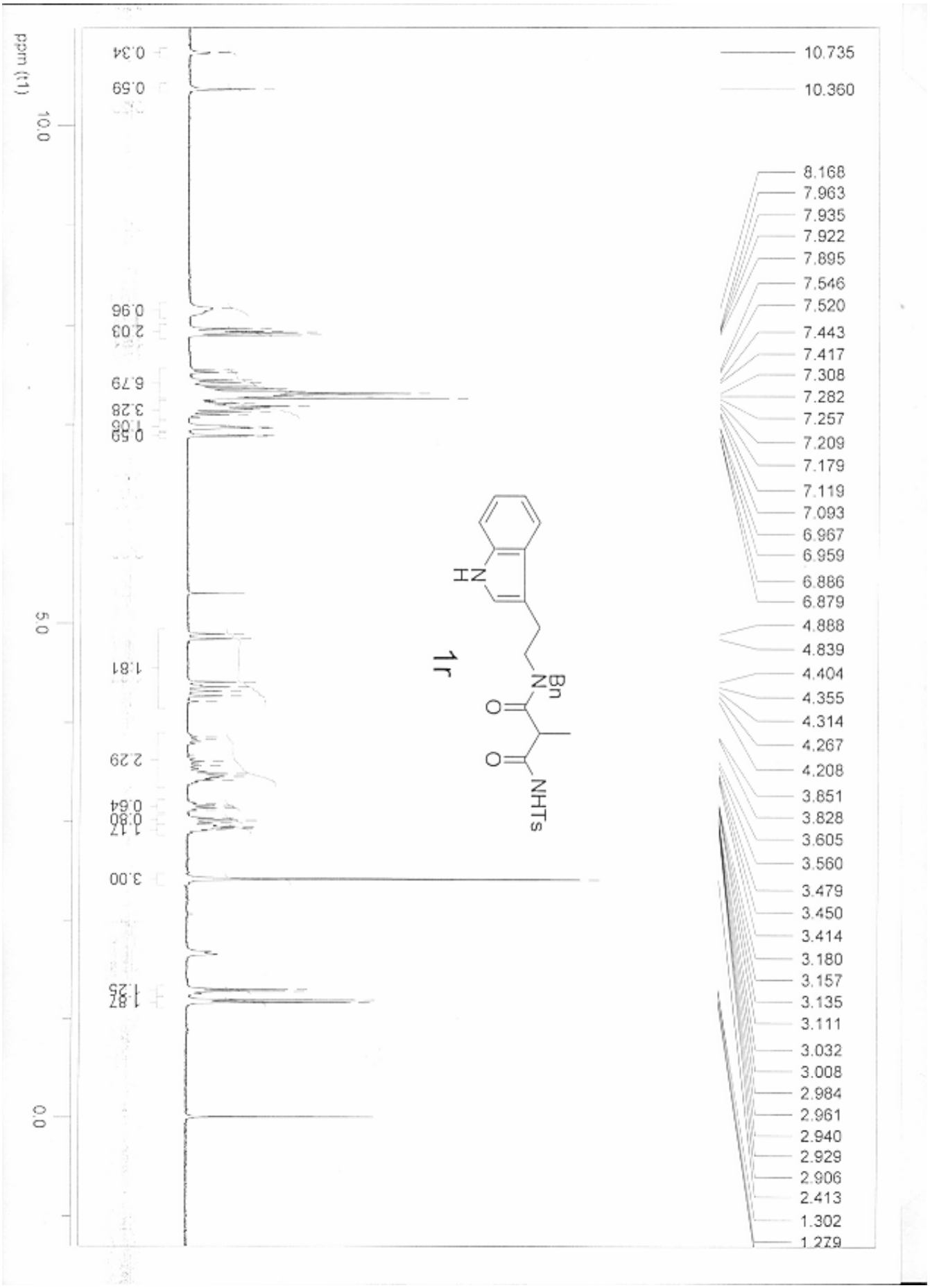

ppm (t1)
150
100
50
0
167.319
167.264
151.694
136.766
135.838
135.261
135.199
132.042
131.838
128.995
128.674
128.416
128.084
128.005
127.906
127.586
126.605
121.314
121.041
119.520
119.283
117.662
117.130
110.670
110.304
108.243
107.306
53.006
52.916
52.635
48.701
47.911
47.858
41.724
41.286
23.162
22.178
11.515
11.448
H
N
Bn
N
O
O
NHCOOMe
1o

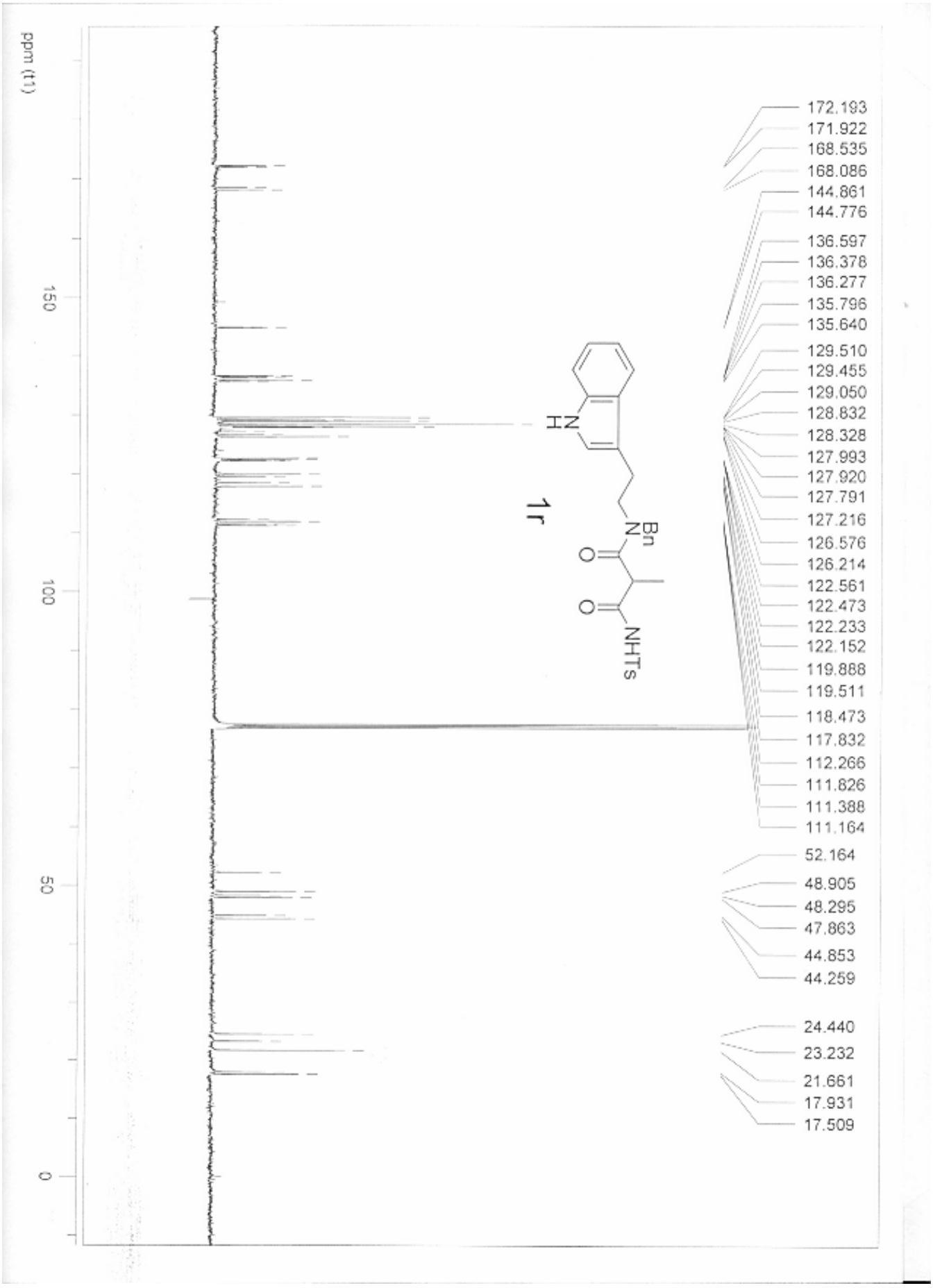

8.180
8.100
7.624
7.604
7.568
7.550
7.506
7.494
7.470
7.450
7.422
7.399
7.386
7.367
7.320
7.303
7.285
7.272
7.258
7.192
7.173
7.153
7.131
7.111
7.057
7.040
4.459
4.266
3.789
3.760
3.691
3.674
3.654
3.410
3.392
3.373
3.248
3.228
3.211
3.185
3.166
3.149
0.45
1.65
2.41
2.32
6.29
2.25
0.91
1.19
0.91
0.95
3.22
3.14
1.00
1.23
5.0
0.0
1p
Ph
N
H
Bn
N
O
O
NHCOOMe

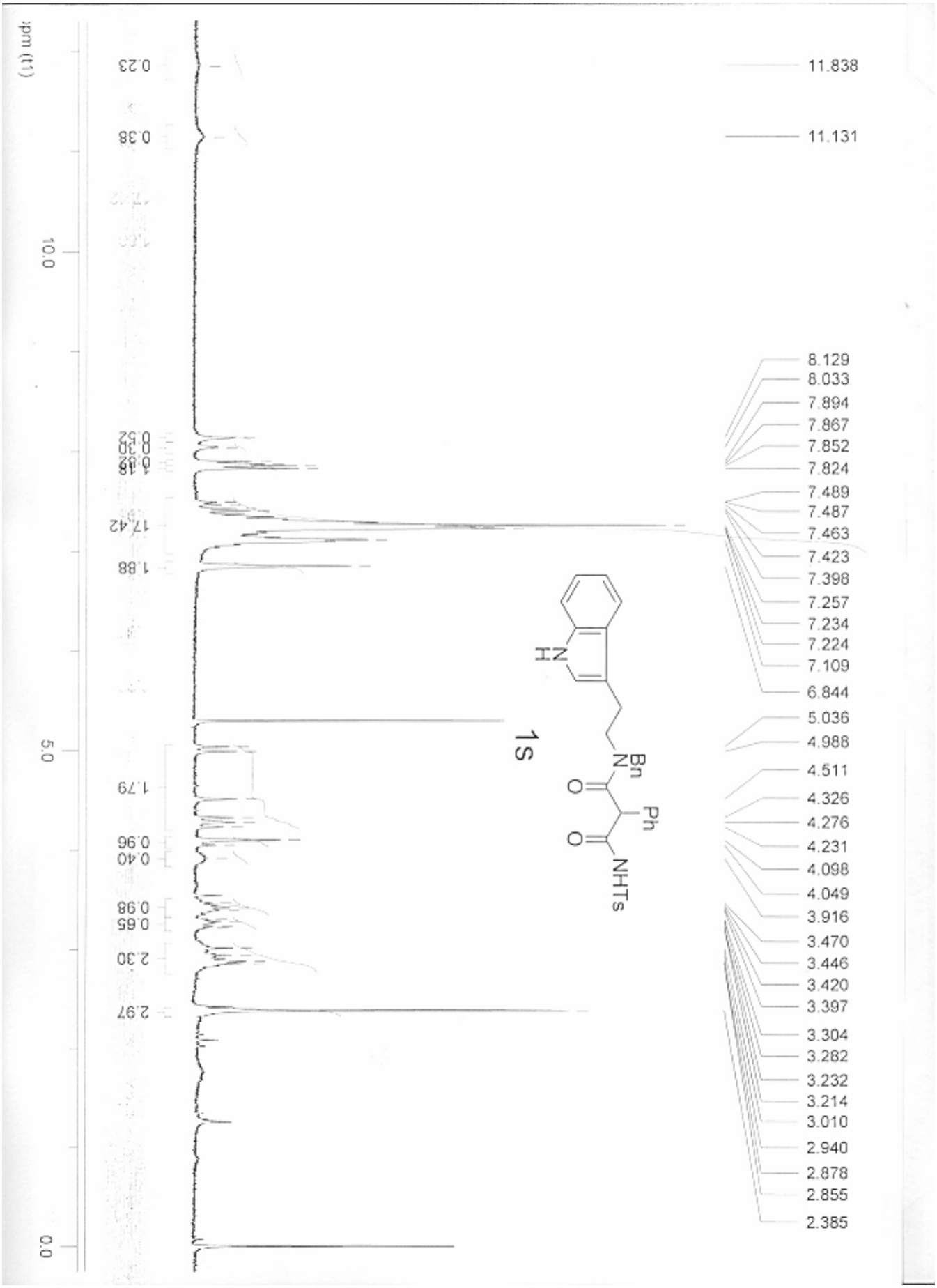

ppm (t1)
200
150
100
50
0
167.546
167.192
151.732
136.779
135.926
135.809
135.767
135.075
132.929
132.727
129.109
129.044
128.644
128.490
128.215
128.130
127.965
127.867
127.823
127.562
126.587
122.612
122.534
120.110
119.920
118.942
118.170
111.364
111.004
109.627
108.456
52.962
52.917
52.498
48.464
47.820
47.707
41.143
41.094
23.227
22.505
N
H
Ph
1p
Bn
N
O
O
NHCOOMe


ppm (t1)
0.35
0.50
0.46
0.25
0.38
0.57
3.83
3.81
0.92
0.51
1.35
0.75
1.73
2.32
1.85
1.06
5.0
0.0
9.483
9.440
8.495
8.320
7.558
7.532
7.464
7.439
7.371
7.276
7.216
7.210
7.087
6.994
6.982
4.893
4.843
4.540
4.502
4.484
4.453
4.425
3.901
3.889
3.874
3.848
3.825
3.806
3.801
3.780
3.758
3.702
3.679
3.596
3.571
3.547
3.523
3.068
3.043
3.020
2.980
2.971
1.421
1.398
1.365
1.341
H
N
Bn
N
O
O
NHCOOMe
1q


ppm (t1)
150
100
50
172.187
171.962
169.871
169.445
169.427
151.758
151.527
136.899
136.444
136.376
136.049
129.039
128.959
128.934
128.902
128.817
128.097
128.082
127.958
127.717
127.313
126.953
126.702
126.631
122.751
122.683
122.416
122.354
122.179
122.008
119.650
119.363
118.635
118.618
118.597
118.153
112.421
111.750
111.377
53.012
52.892
52.802
52.294
52.243
48.825
48.377
48.331
47.923
45.299
45.208
44.846
44.771
24.566
23.164
17.193
17.150
16.967
N
H
Bn
N
O
O
NHCOOMe
1q


ppm (t1)
10.0
5.0
0.0
0.34
0.59
0.96
2.03
6.79
3.28
1.06
0.59
1.81
2.29
0.64
0.80
1.17
3.00
1.82
1.25
10.735
10.360
8.168
7.963
7.935
7.922
7.895
7.546
7.520
7.443
7.417
7.308
7.282
7.257
7.209
7.179
7.119
7.093
6.967
6.959
6.886
6.879
4.868
4.839
4.404
4.355
4.314
4.267
4.208
3.851
3.828
3.605
3.560
3.479
3.450
3.414
3.180
3.157
3.135
3.111
3.032
3.008
2.984
2.961
2.940
2.929
2.906
2.413
1.302
1.279
1r
Bn
N
O
O
NHTs
H
N


ppm (t1)
150
100
50
0
172.193
171.922
168.535
168.086
144.861
144.776
136.597
136.378
136.277
135.796
135.640
129.510
129.455
129.050
128.832
128.328
127.993
127.920
127.791
127.216
126.576
126.214
122.561
122.473
122.233
122.152
119.888
119.511
118.473
117.832
112.266
111.826
111.388
111.164
52.164
48.905
48.295
47.863
44.853
44.259
24.440
23.232
21.661
17.931
17.509
H
N
Bn
N
O
O
NHTs
1r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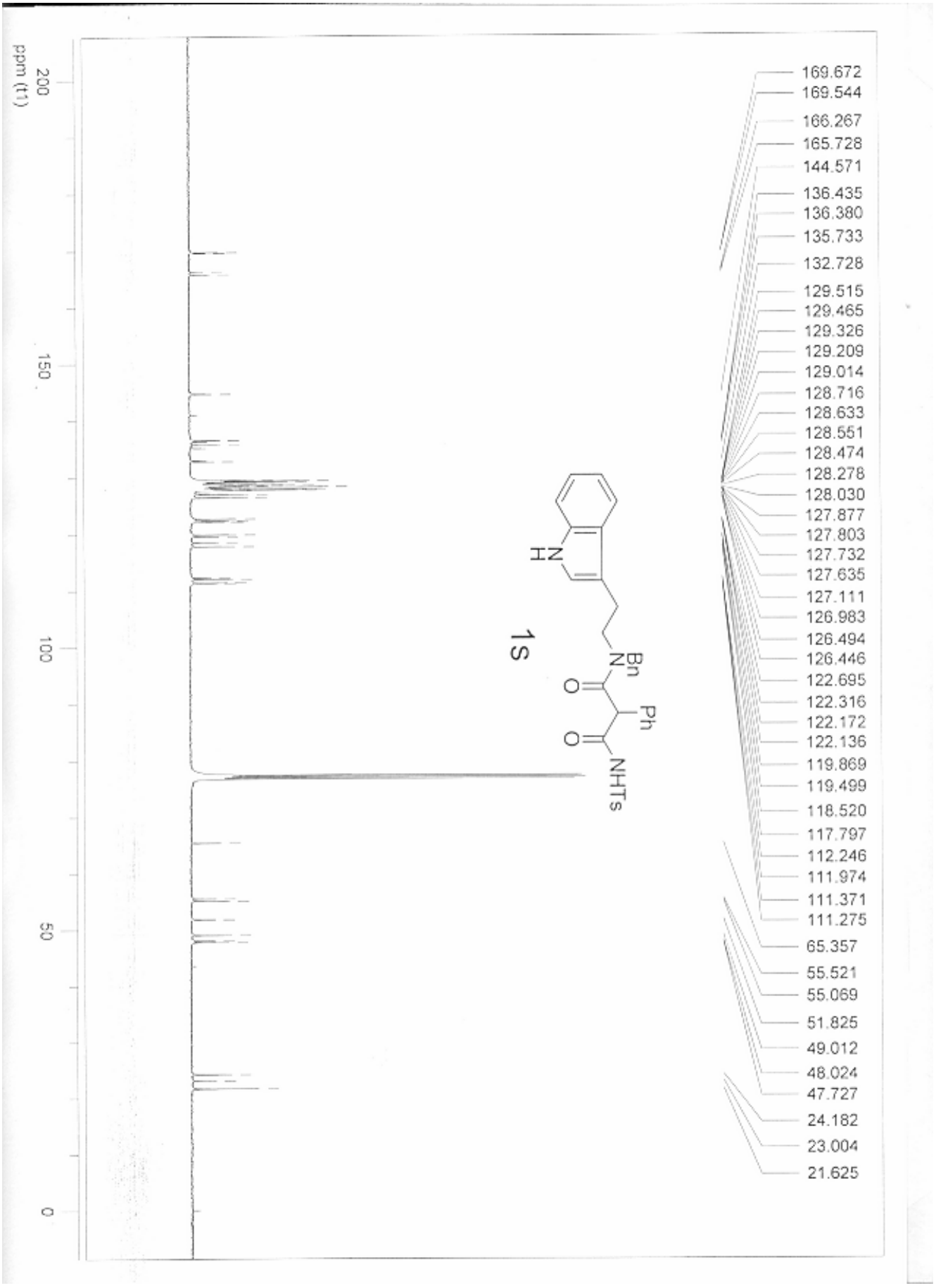
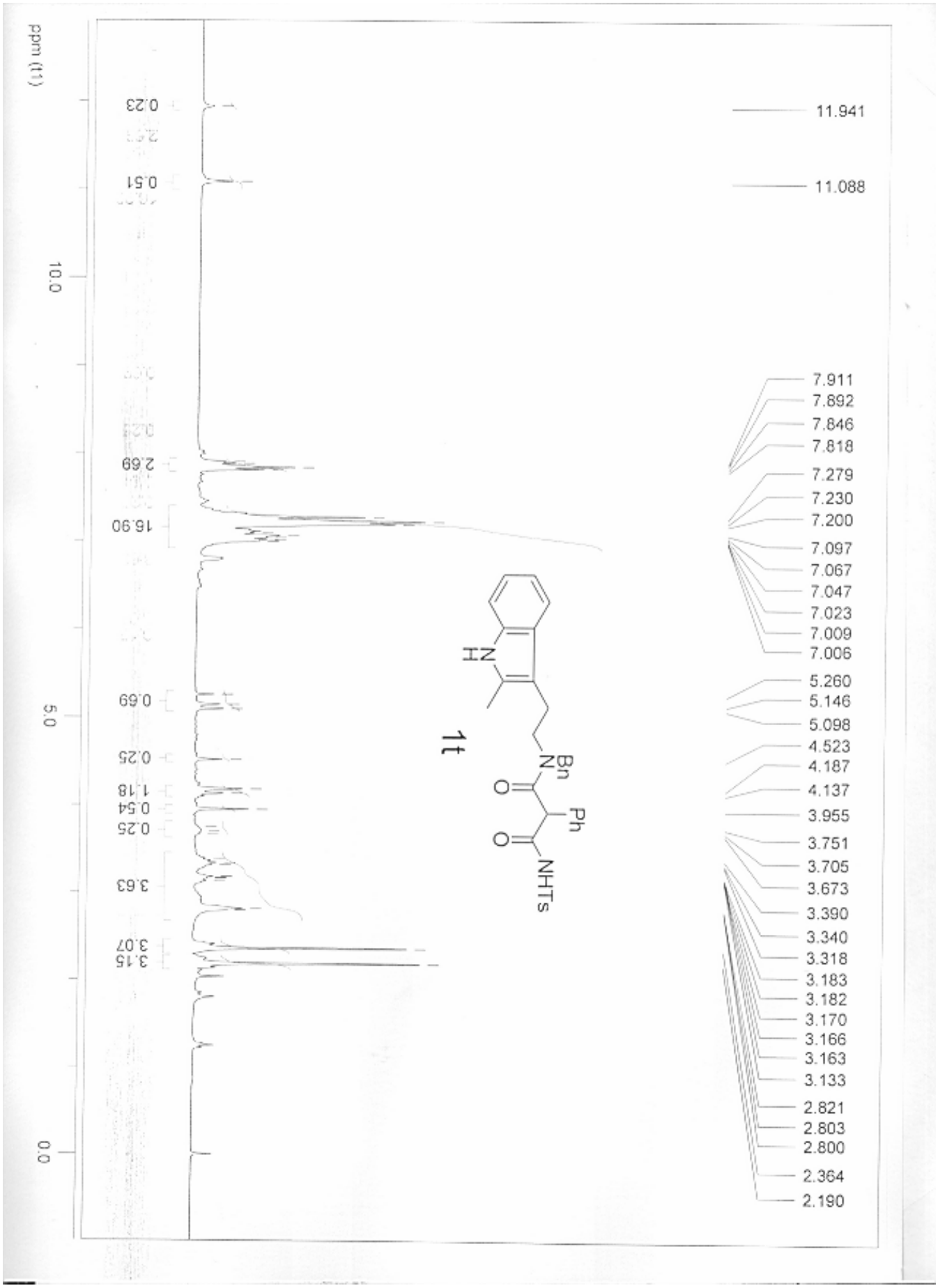
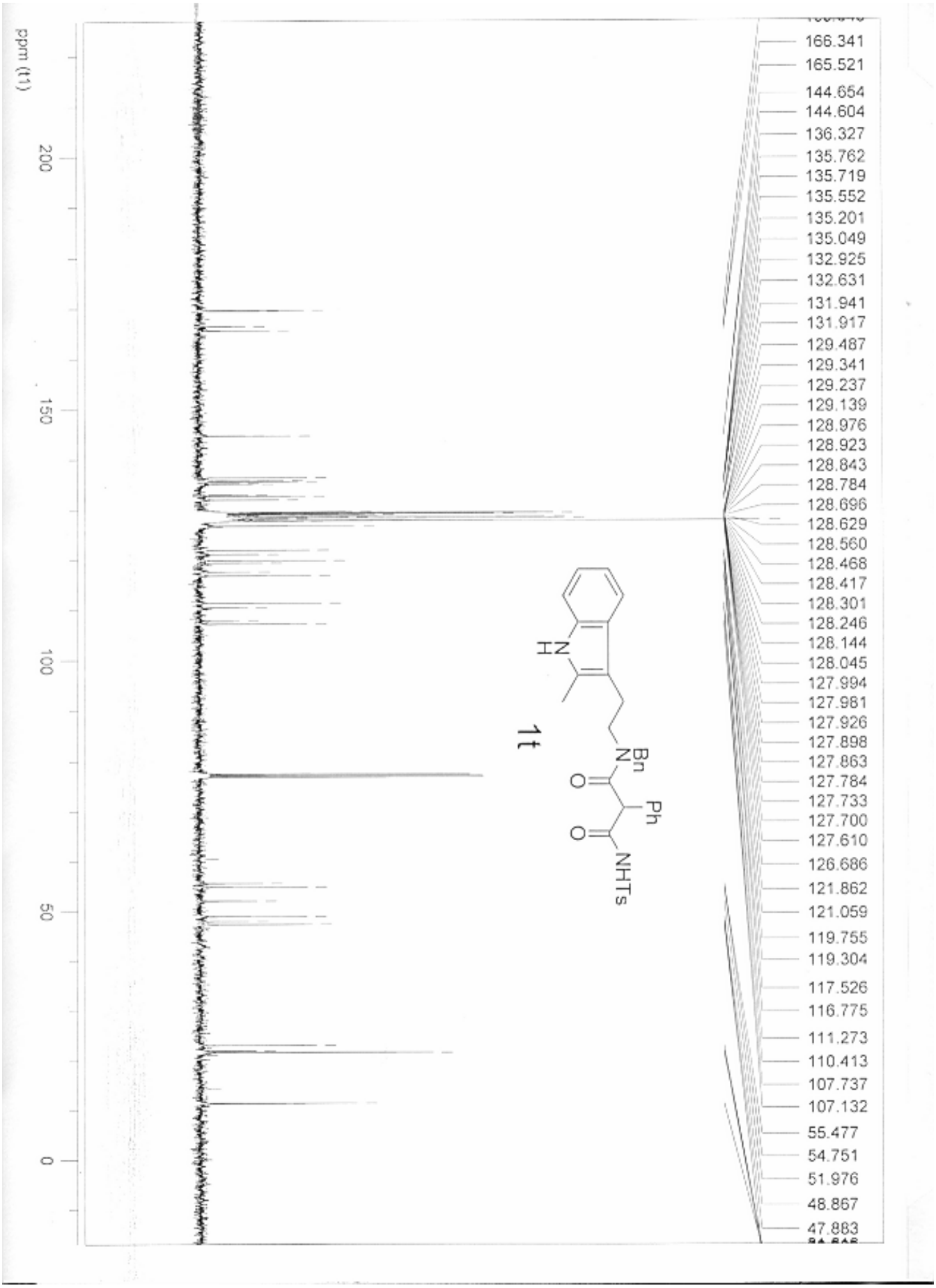
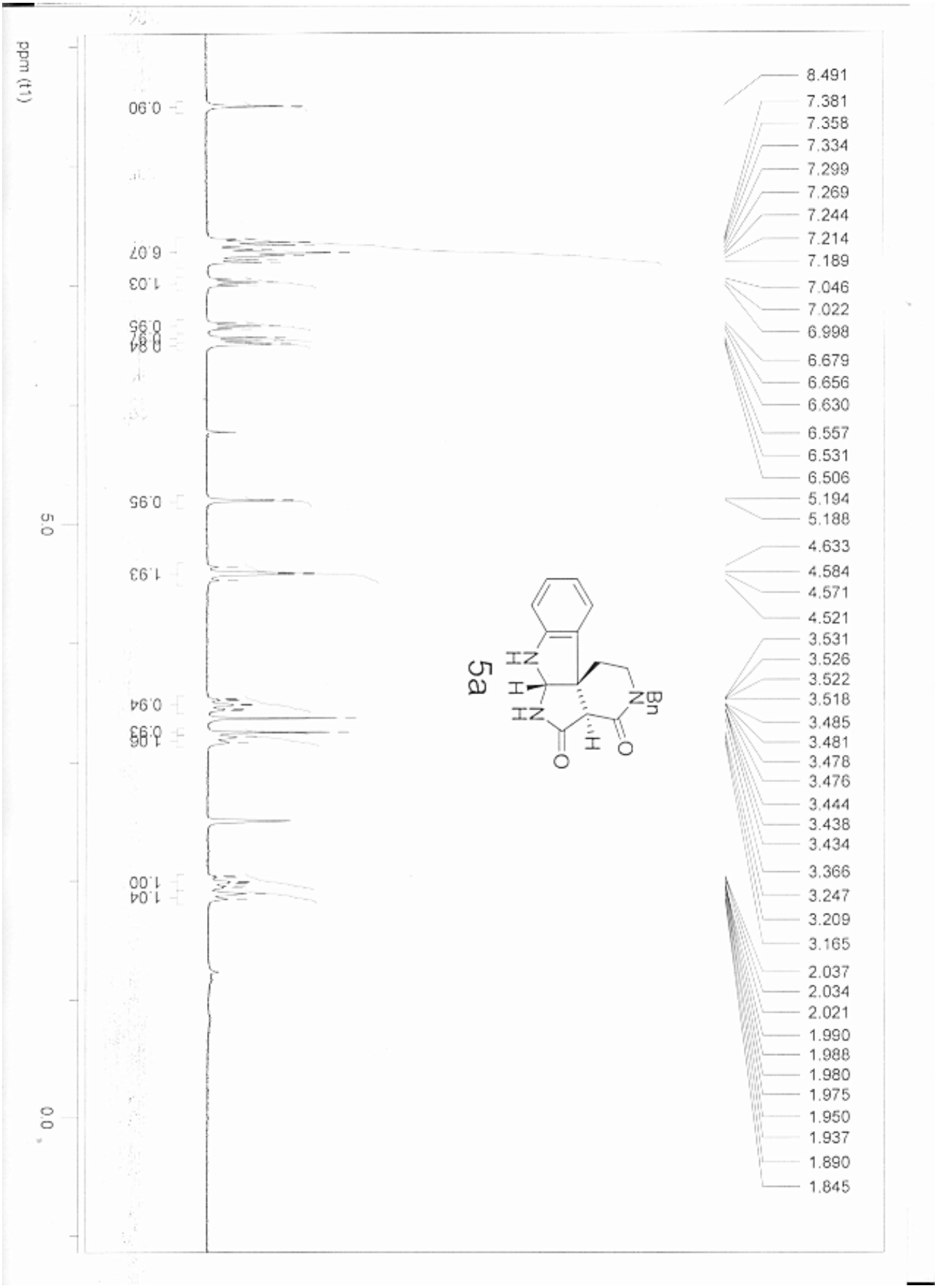
11.131
8.129
8.033
7.894
7.867
7.852
7.824
7.489
7.487
7.463
7.423
7.398
7.257
7.234
7.224
7.109
6.844
5.036
4.988
4.511
4.326
4.276
4.231
4.098
4.049
3.916
3.470
3.446
3.420
3.397
3.304
3.282
3.232
3.214
3.010
2.940
2.878
2.855
2.385
0.23
0.38
17.42
1.88
1.79
0.96
0.40
0.98
0.65
2.30
2.97
10.0
5.0
0.0
1s
Bn
Ph
NHTs


ppm (t1)
200
150
100
50
0
169.672
169.544
166.267
165.728
144.571
136.435
136.380
135.733
132.728
129.515
129.465
129.326
129.209
129.014
128.716
128.633
128.551
128.474
128.278
128.030
127.877
127.803
127.732
127.635
127.111
126.983
126.494
126.446
122.695
122.316
122.172
122.136
119.869
119.499
118.520
117.797
112.246
111.974
111.371
111.275
65.357
55.521
55.069
51.825
49.012
48.024
47.727
24.182
23.004
21.625
N
H
1s
N
Bn
Ph
O
O
NHTs


ppm (t1)
10.0
5.0
0.0
0.23
0.51
2.69
16.90
0.69
0.25
1.18
0.54
0.25
3.63
3.07
3.15
11.941
11.088
7.911
7.892
7.846
7.818
7.279
7.230
7.200
7.097
7.067
7.047
7.023
7.009
7.006
5.260
5.146
5.098
4.523
4.187
4.137
3.955
3.751
3.705
3.673
3.390
3.340
3.318
3.183
3.182
3.170
3.166
3.163
3.133
2.821
2.803
2.800
2.364
2.190
1t
Bn
Ph
NHTs


ppm (t1)
200
150
100
50
0
166.341
165.521
144.654
144.604
136.327
135.762
135.719
135.552
135.201
135.049
132.925
132.631
131.941
131.917
129.487
129.341
129.237
129.139
128.976
128.923
128.843
128.784
128.696
128.629
128.560
128.468
128.417
128.301
128.246
128.144
128.045
127.994
127.981
127.926
127.898
127.863
127.784
127.733
127.700
127.610
126.686
121.862
121.059
119.755
119.304
117.526
116.775
111.273
110.413
107.737
107.132
55.477
54.751
51.976
48.867
47.883
N
H
Bn
N
O
O
Ph
NHTs
1t


ppm (t1)
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0.0
0.90
6.07
1.03
0.84
0.87
0.95
1.93
0.94
0.93
1.06
1.00
1.04
8.491
7.381
7.358
7.334
7.299
7.269
7.244
7.214
7.189
7.046
7.022
6.998
6.679
6.656
6.630
6.557
6.531
6.506
5.194
5.188
4.633
4.584
4.571
4.521
3.531
3.526
3.522
3.518
3.485
3.481
3.478
3.476
3.444
3.438
3.434
3.366
3.247
3.209
3.165
2.037
2.034
2.021
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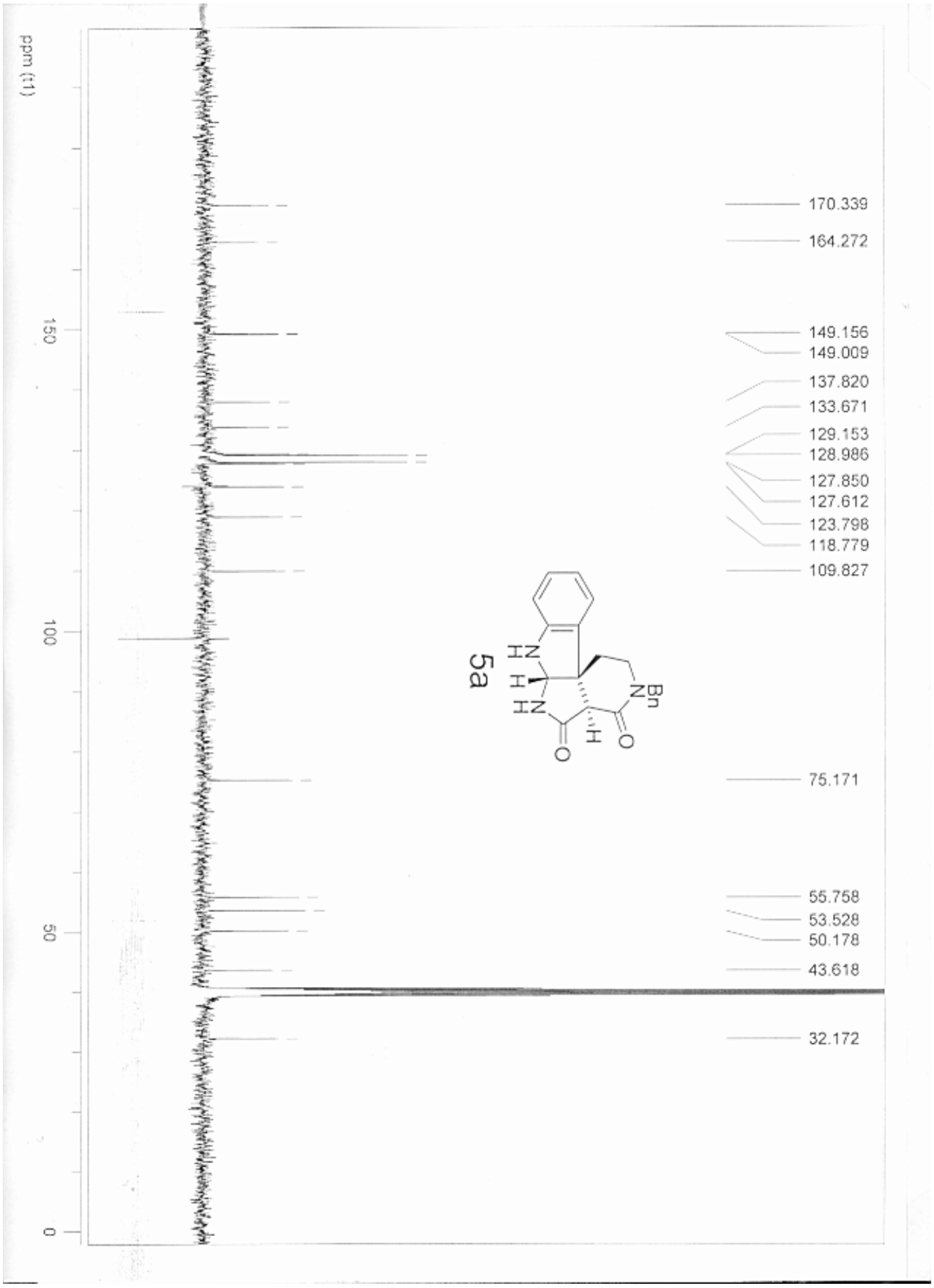
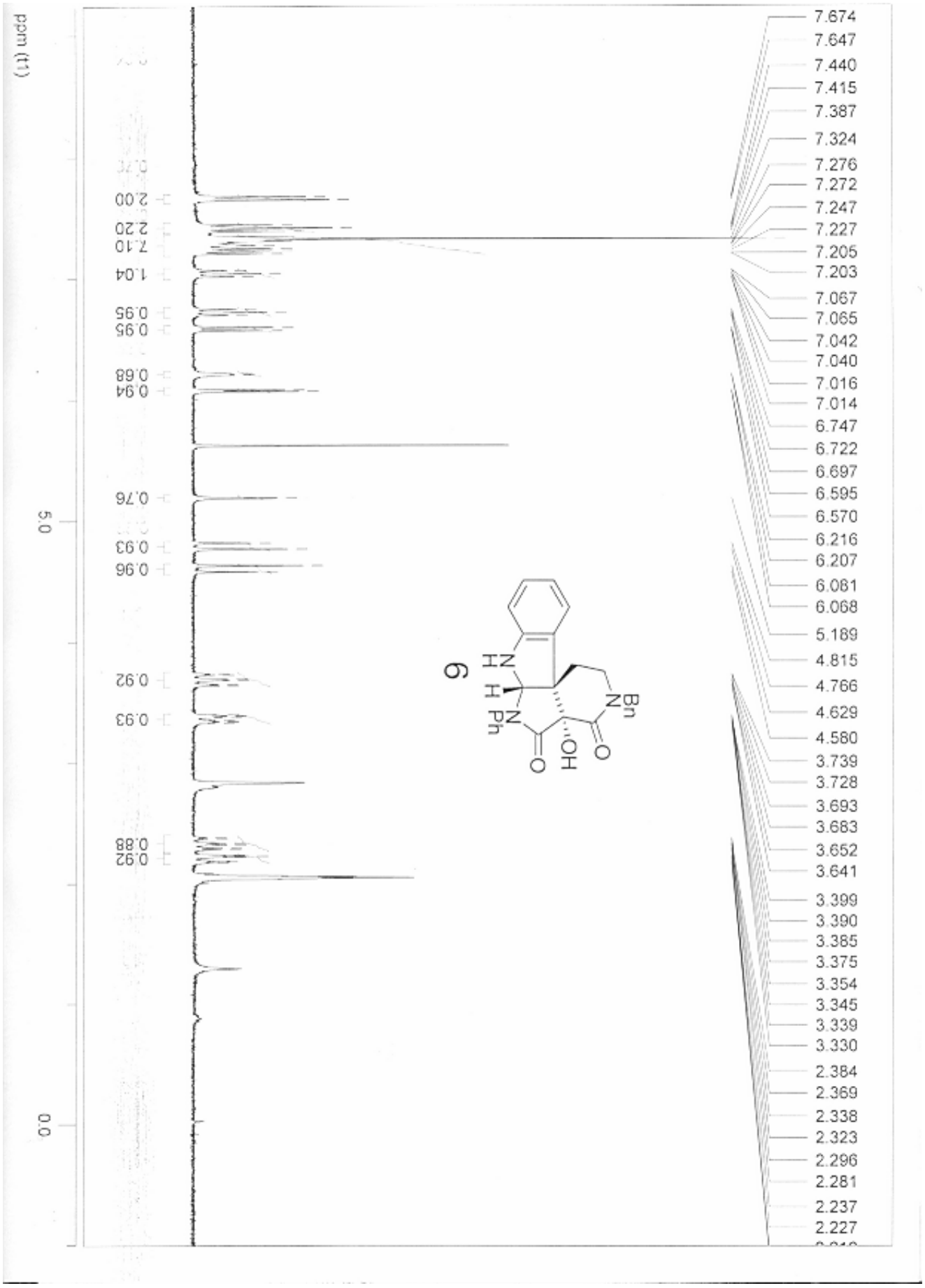
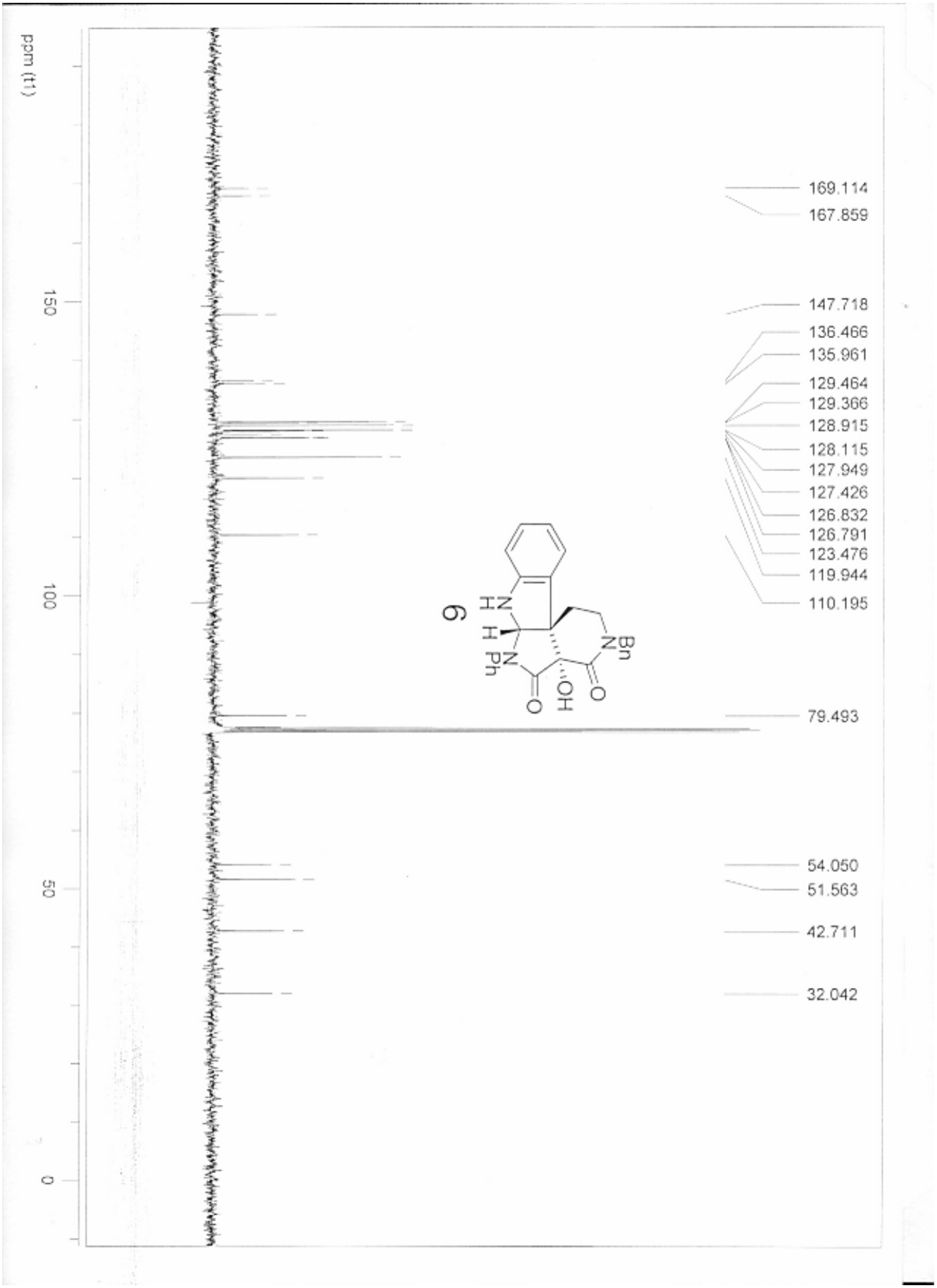
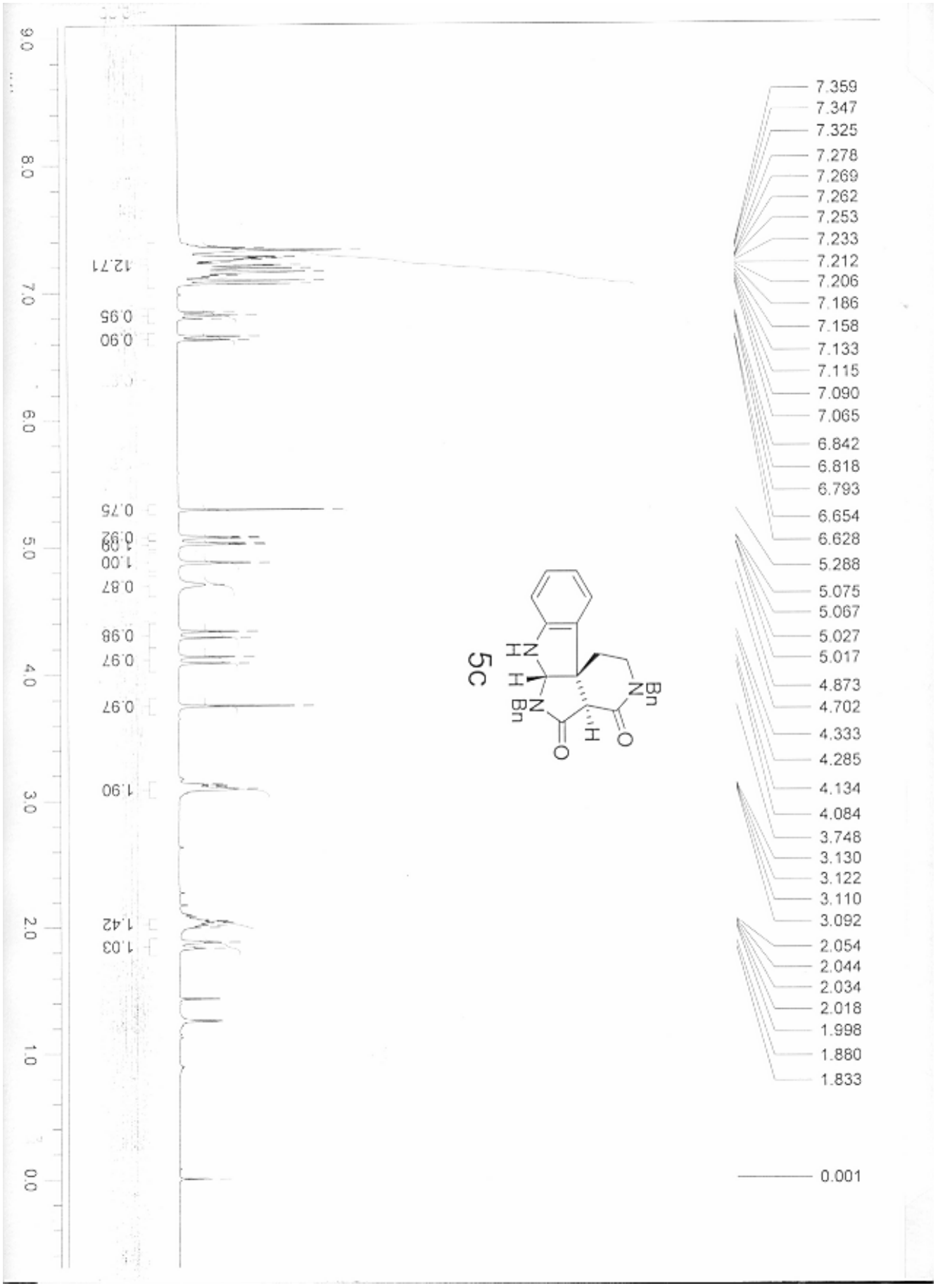
1.988
1.980
1.975
1.950
1.937
1.890
1.845
5a
Bn


ppm (t1)
150
100
50
0
170.339
164.272
149.156
149.009
137.820
133.671
129.153
128.986
127.850
127.612
123.798
118.779
109.827
75.171
55.758
53.528
50.178
43.618
32.172
5a
Bn


7.674
7.647
7.440
7.415
7.387
7.324
7.276
7.272
7.247
7.227
7.205
7.203
7.067
7.065
7.042
7.040
7.016
7.014
6.747
6.722
6.697
6.595
6.570
6.216
6.207
6.081
6.068
5.189
4.815
4.766
4.629
4.580
3.739
3.728
3.693
3.683
3.652
3.641
3.399
3.390
3.385
3.375
3.354
3.345
3.339
3.330
2.384
2.369
2.338
2.323
2.296
2.281
2.237
2.227
2.00
2.20
2.10
1.04
0.95
0.95
0.68
0.94
0.76
0.93
0.96
0.92
0.93
0.88
0.92
5.0
0.0
ppm (t1)
6
Bn
Ph
OH


ppm (t1)
150
100
50
0
169.114
167.859
147.718
136.466
135.961
129.464
129.366
128.915
128.115
127.949
127.426
126.832
126.791
123.476
119.944
110.195
79.493
54.050
51.563
42.711
32.042
6
N
H
H
N
Ph
Bn
N
OH
O
O


7.359
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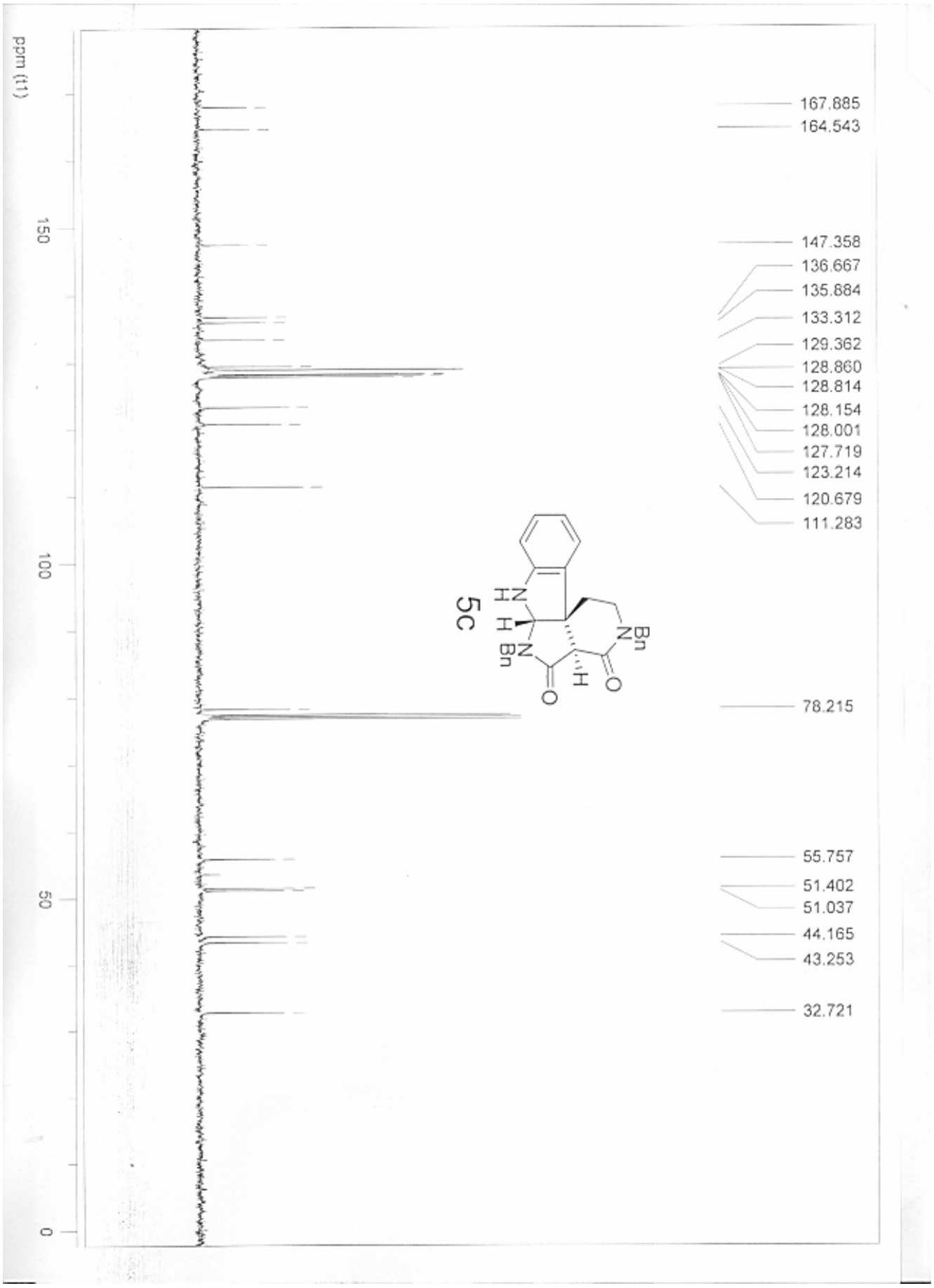
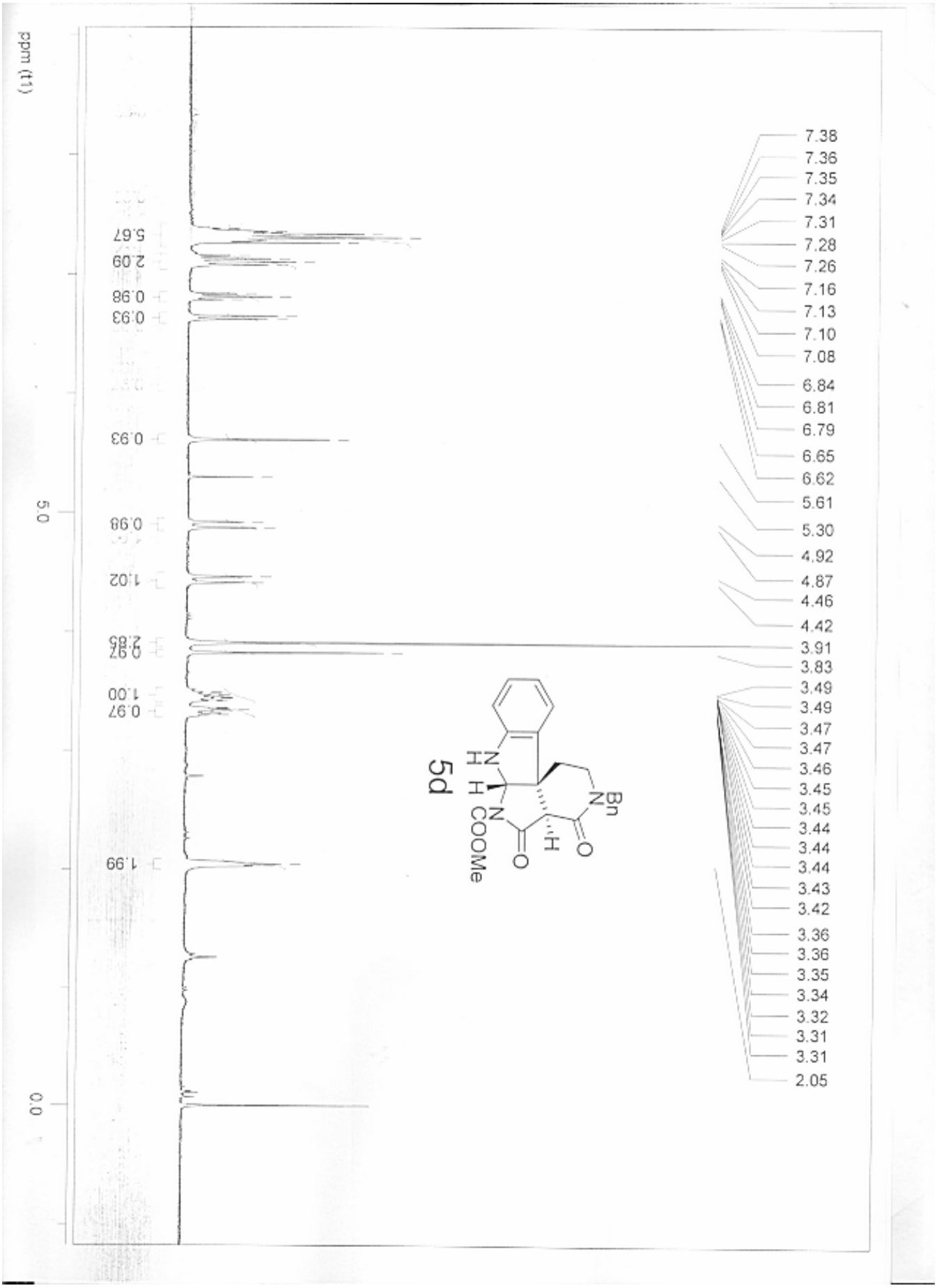
7.325
7.278
7.269
7.262
7.253
7.233
7.212
7.206
7.186
7.158
7.133
7.115
7.090
7.065
6.842
6.818
6.793
6.654
6.628
5.288
5.075
5.067
5.027
5.017
4.873
4.702
4.333
4.285
4.134
4.084
3.748
3.130
3.122
3.110
3.092
2.054
2.044
2.034
2.018
1.998
1.880
1.833
0.001
12.71
0.95
0.90
0.75
1.09
1.00
0.87
0.98
0.97
0.97
1.90
1.42
1.03
5C
N
H
H
N
Bn
Bn
N
O
O
H


ppm (t1)
150
100
50
0
167.885
164.543
147.358
136.667
135.884
133.312
129.362
128.860
128.814
128.154
128.001
127.719
123.214
120.679
111.283
78.215
55.757
51.402
51.037
44.165
43.253
32.721
5c
N
H
H
N
Bn
Bn
N
O
O
H


ppm (t1)
5.0
0.0
5.67
2.09
0.98
0.93
0.93
0.99
1.02
2.85
0.97
1.00
0.97
1.99
7.38
7.36
7.35
7.34
7.31
7.28
7.26
7.16
7.13
7.10
7.08
6.84
6.81
6.79
6.65
6.62
5.61
5.30
4.92
4.87
4.46
4.42
3.91
3.83
3.49
3.49
3.47
3.47
3.46
3.45
3.45
3.44
3.44
3.44
3.43
3.42
3.36
3.36
3.35
3.34
3.32
3.31
3.31
2.05
5d
Bn
N
O
H
O
N
H
H
N
COOMe


ppm (t1)
150
100
50
166.18
162.64
152.64
146.79
136.26
130.99
129.67
128.83
128.27
127.85
123.08
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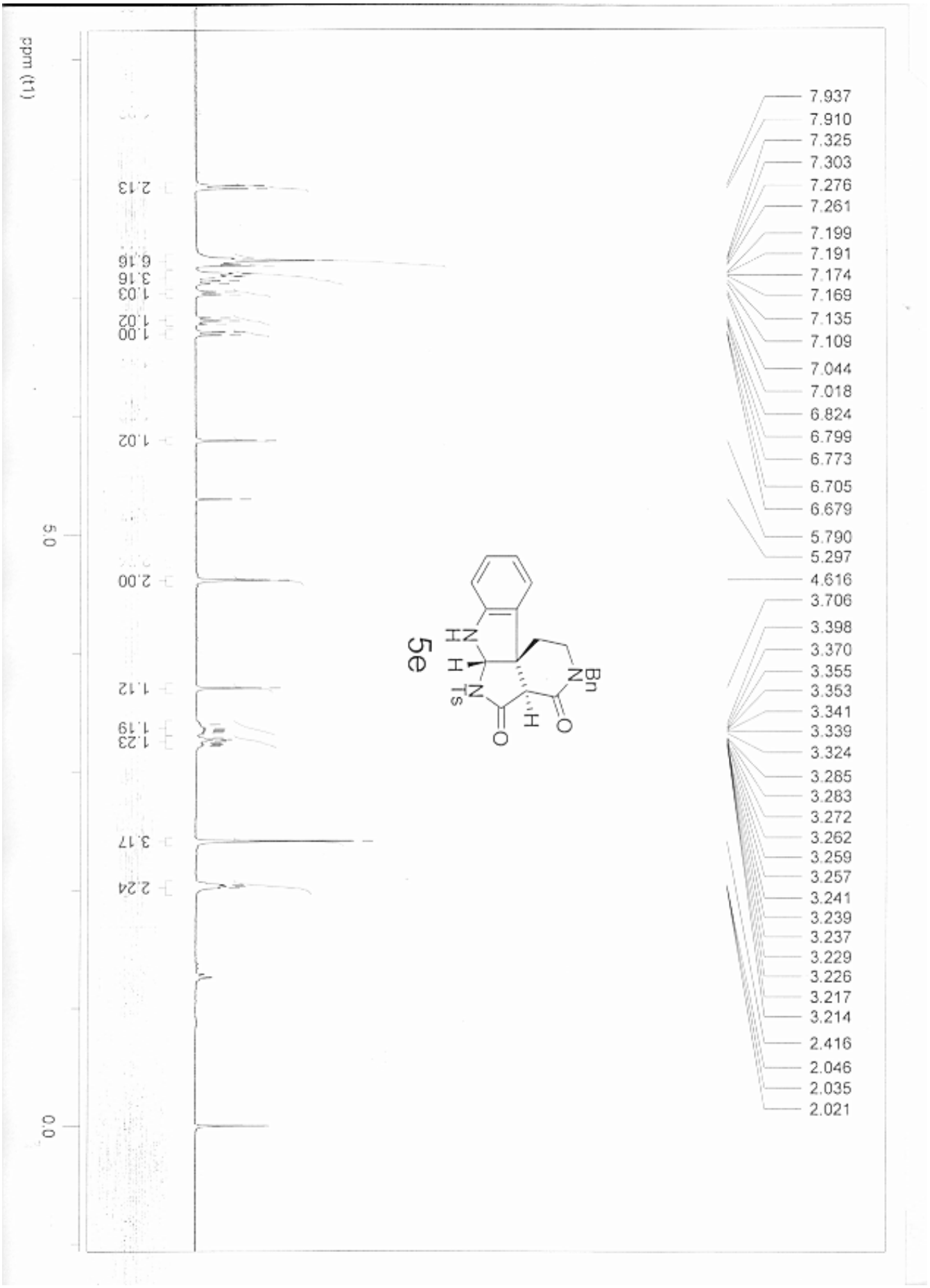
78.81
56.08
53.99
50.89
49.06
43.13
31.02
5d
Bn
COOMe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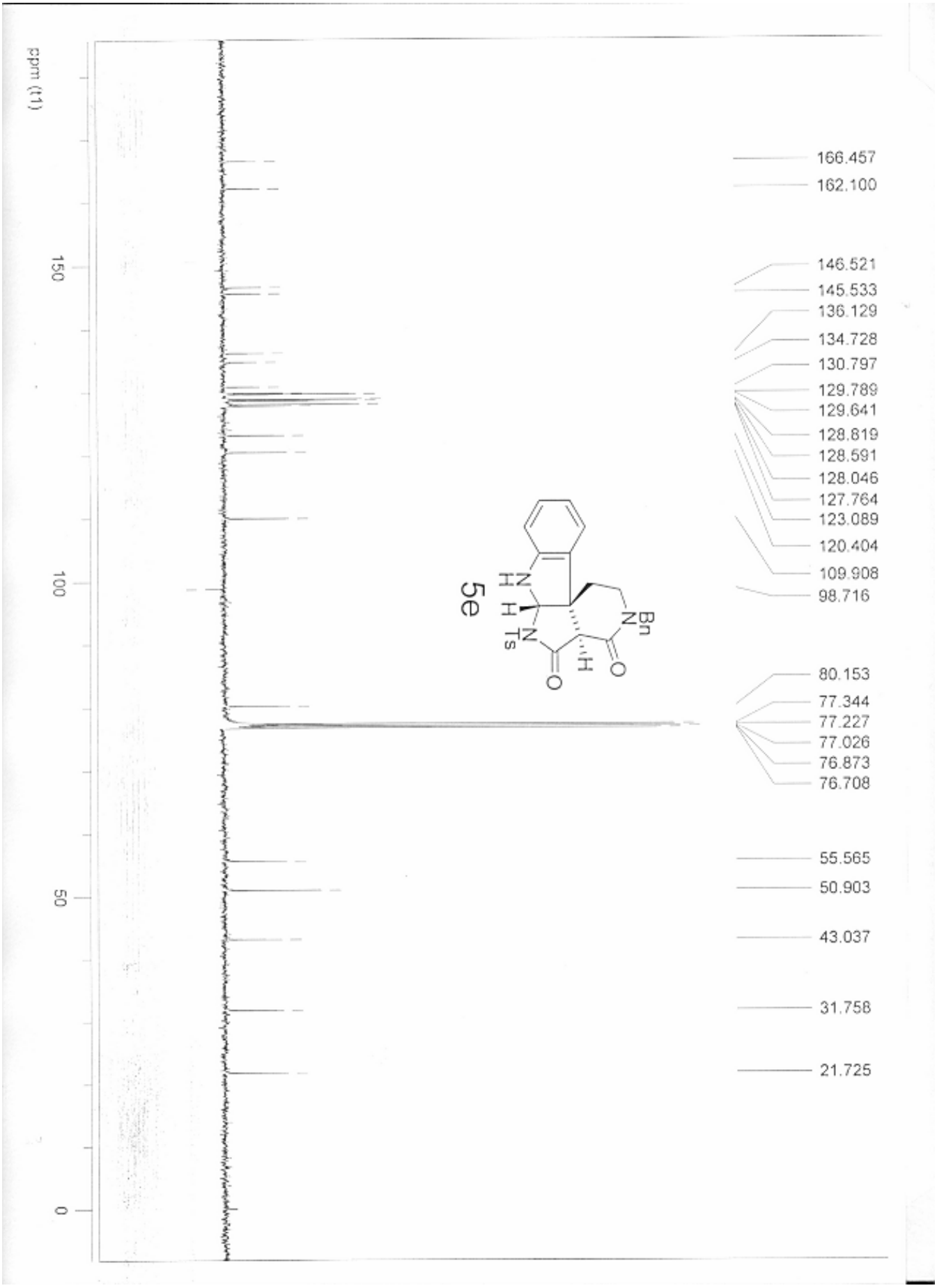
3.16
1.03
1.02
1.00
1.02
2.00
1.12
1.19
1.23
3.17
2.24
7.937
7.910
7.325
7.303
7.276
7.261
7.199
7.191
7.174
7.169
7.135
7.109
7.044
7.018
6.824
6.799
6.773
6.705
6.679
5.790
5.297
4.616
3.706
3.398
3.370
3.355
3.353
3.341
3.339
3.324
3.285
3.283
3.272
3.262
3.259
3.257
3.241
3.239
3.237
3.229
3.226
3.217
3.214
2.416
2.046
2.035
2.021
5e
Bn
Ts
H
O
O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0
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146.521
145.533
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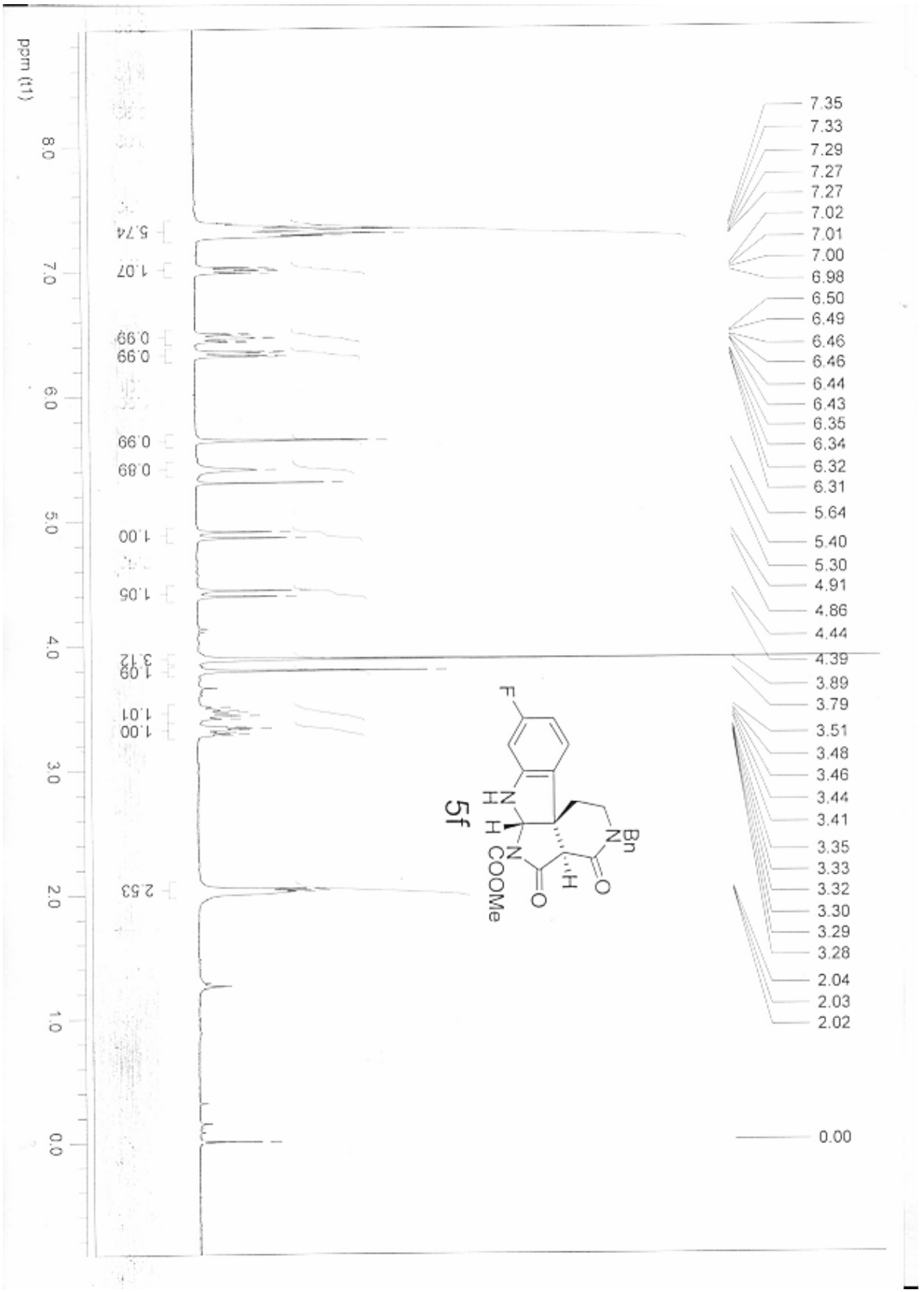
134.728
130.797
129.789
129.641
128.819
128.591
128.046
127.764
123.089
120.404
109.908
98.716
80.153
77.344
77.227
77.026
76.873
76.708
55.565
50.903
43.037
31.758
21.725
5e
Bn
Ts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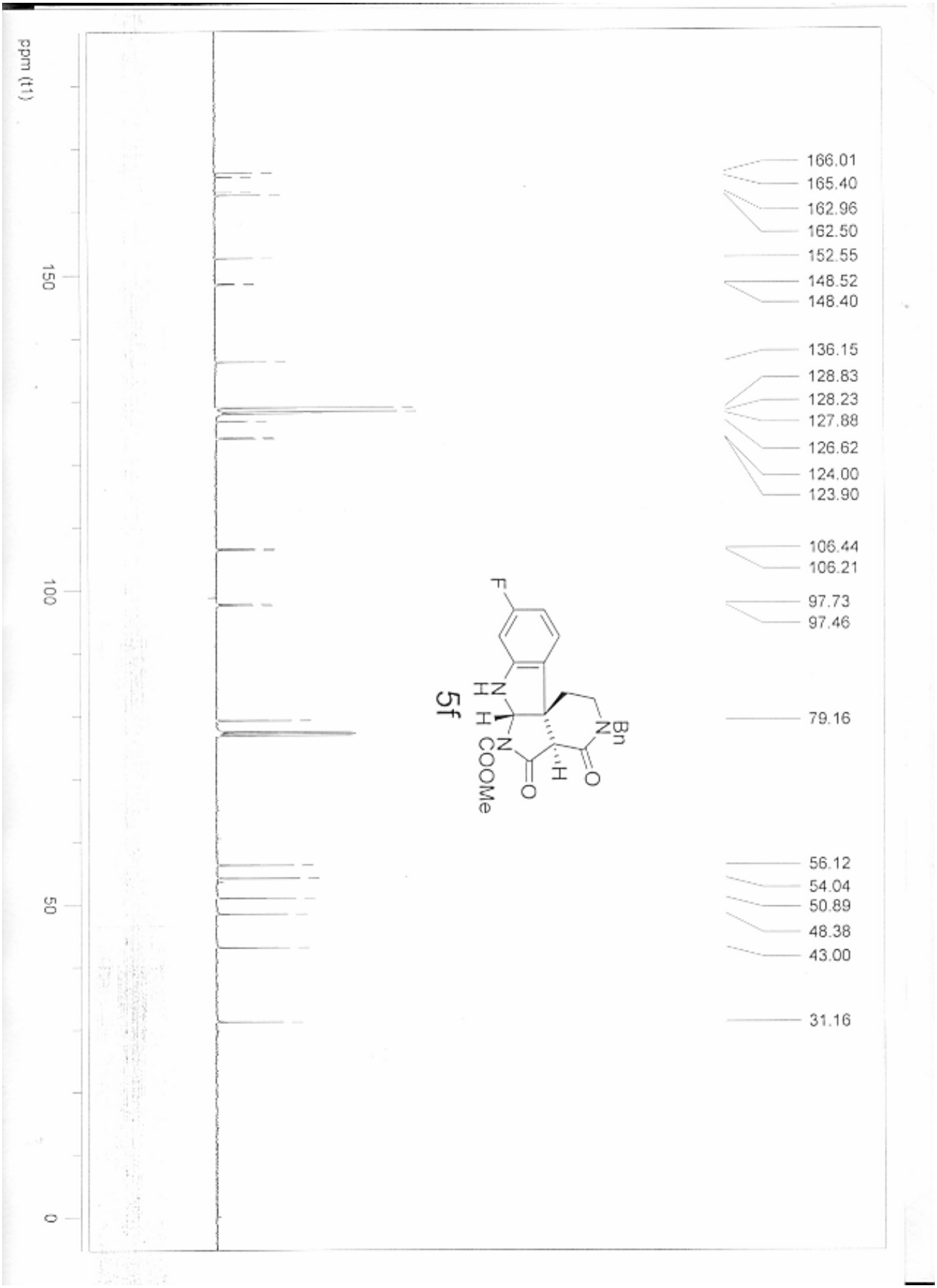
4.0
3.0
2.0
1.0
0.0
5.74
1.07
0.99
0.99
0.99
0.89
1.00
1.05
3.12
1.09
1.01
1.00
2.53
7.35
7.33
7.29
7.27
7.27
7.02
7.01
7.00
6.98
6.50
6.49
6.46
6.46
6.44
6.43
6.35
6.34
6.32
6.31
5.64
5.40
5.30
4.91
4.86
4.44
4.39
3.89
3.79
3.51
3.48
3.46
3.44
3.41
3.35
3.33
3.32
3.30
3.29
3.28
2.04
2.03
2.02
0.00
F
N
H
H
N
COOMe
O
O
H
N
Bn
5f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162.50
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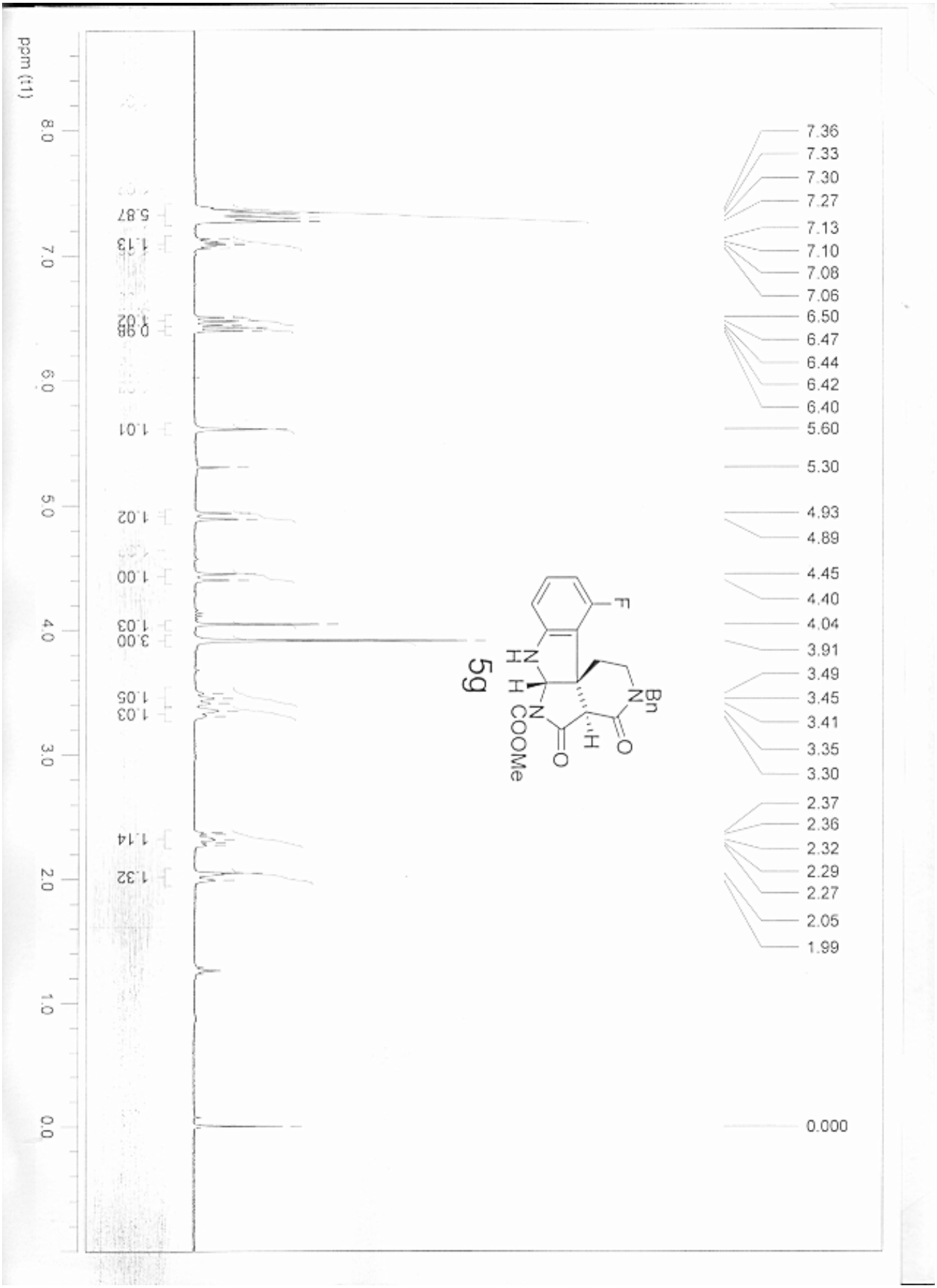
148.52
148.40
136.15
128.83
128.23
127.88
126.62
124.00
123.90
106.44
106.21
97.73
97.46
79.16
56.12
54.04
50.89
48.38
43.00
31.16
F
N
H
H
N
COOMe
O
H
N
Bn
O
5f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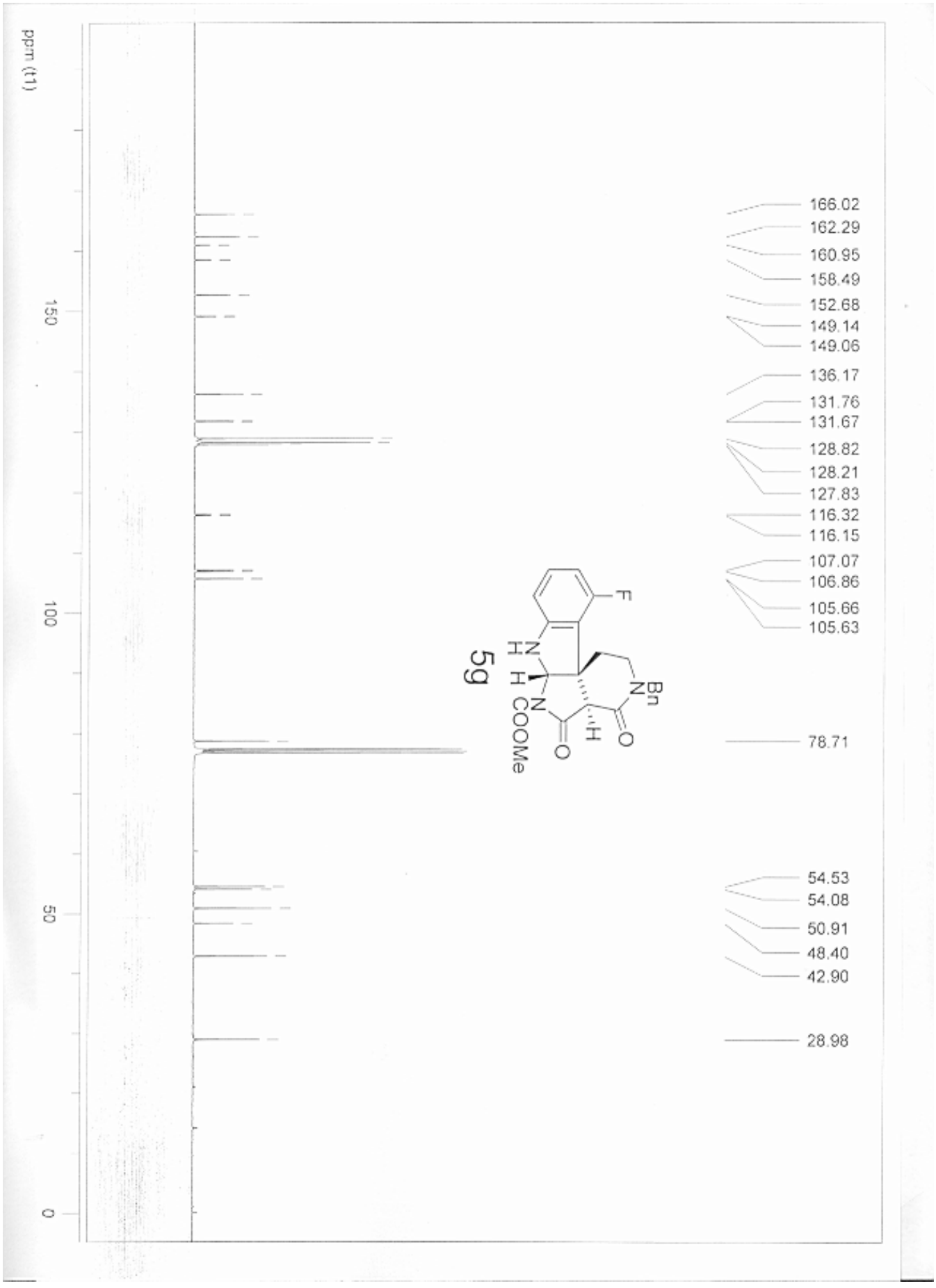
5.0
4.0
3.0
2.0
1.0
0.0
5.87
1.13
0.98
1.02
1.01
1.02
1.00
3.00
1.03
1.05
1.03
1.14
1.32
7.36
7.33
7.30
7.27
7.13
7.10
7.08
7.06
6.50
6.47
6.44
6.42
6.40
5.60
5.30
4.93
4.89
4.45
4.40
4.04
3.91
3.49
3.45
3.41
3.35
3.30
2.37
2.36
2.32
2.29
2.27
2.05
1.99
0.000
5g
F
N
H
H
N
COOMe
O
H
O
N
Bn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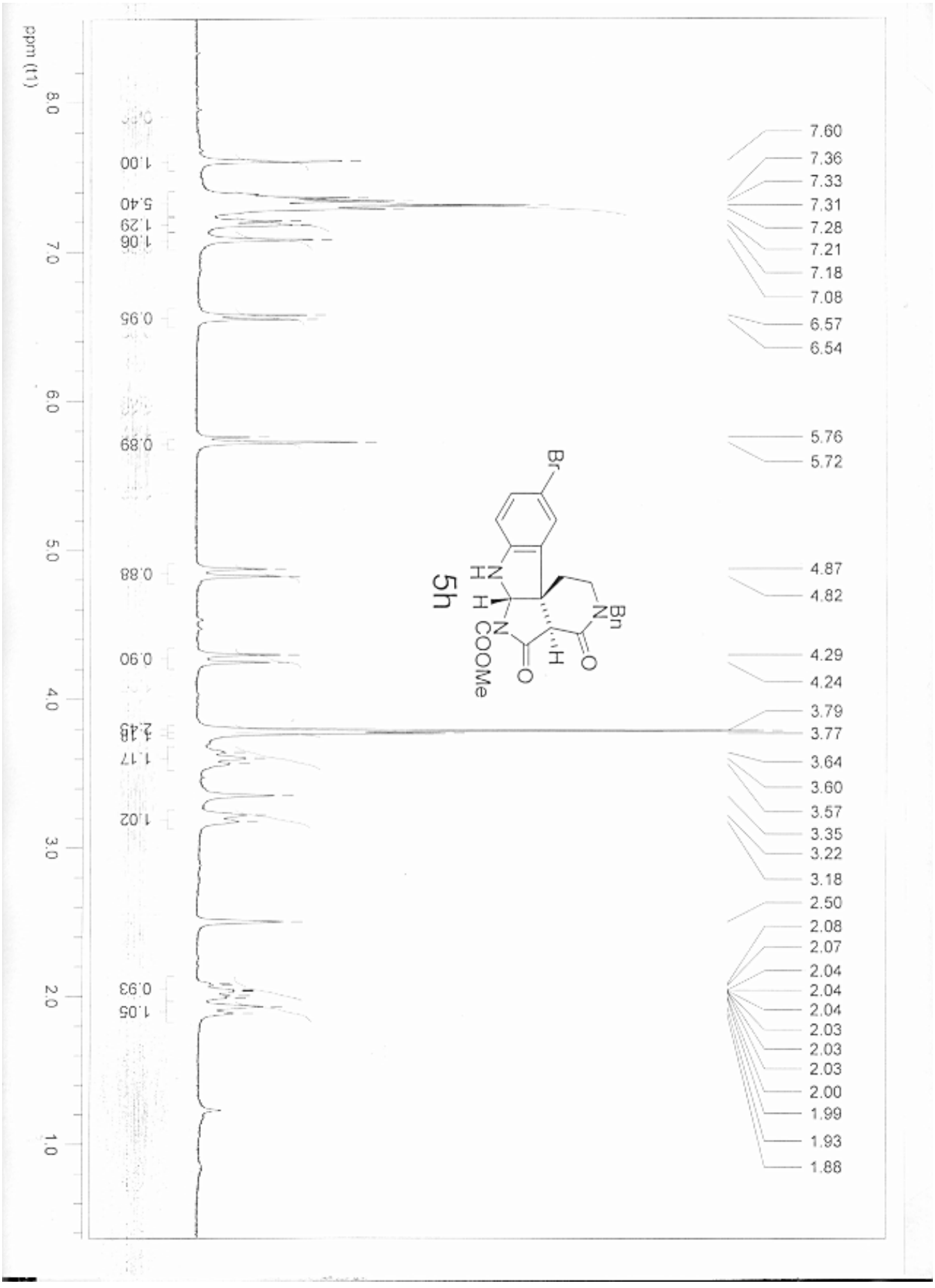
100
50
0
166.02
162.29
160.95
158.49
152.68
149.14
149.06
136.17
131.76
131.67
128.82
128.21
127.83
116.32
116.15
107.07
106.86
105.66
105.63
78.71
54.53
54.08
50.91
48.40
42.90
28.98
5g
F
H
N
N
H
COOMe
O
H
N
Bn
O


ppm (t1)
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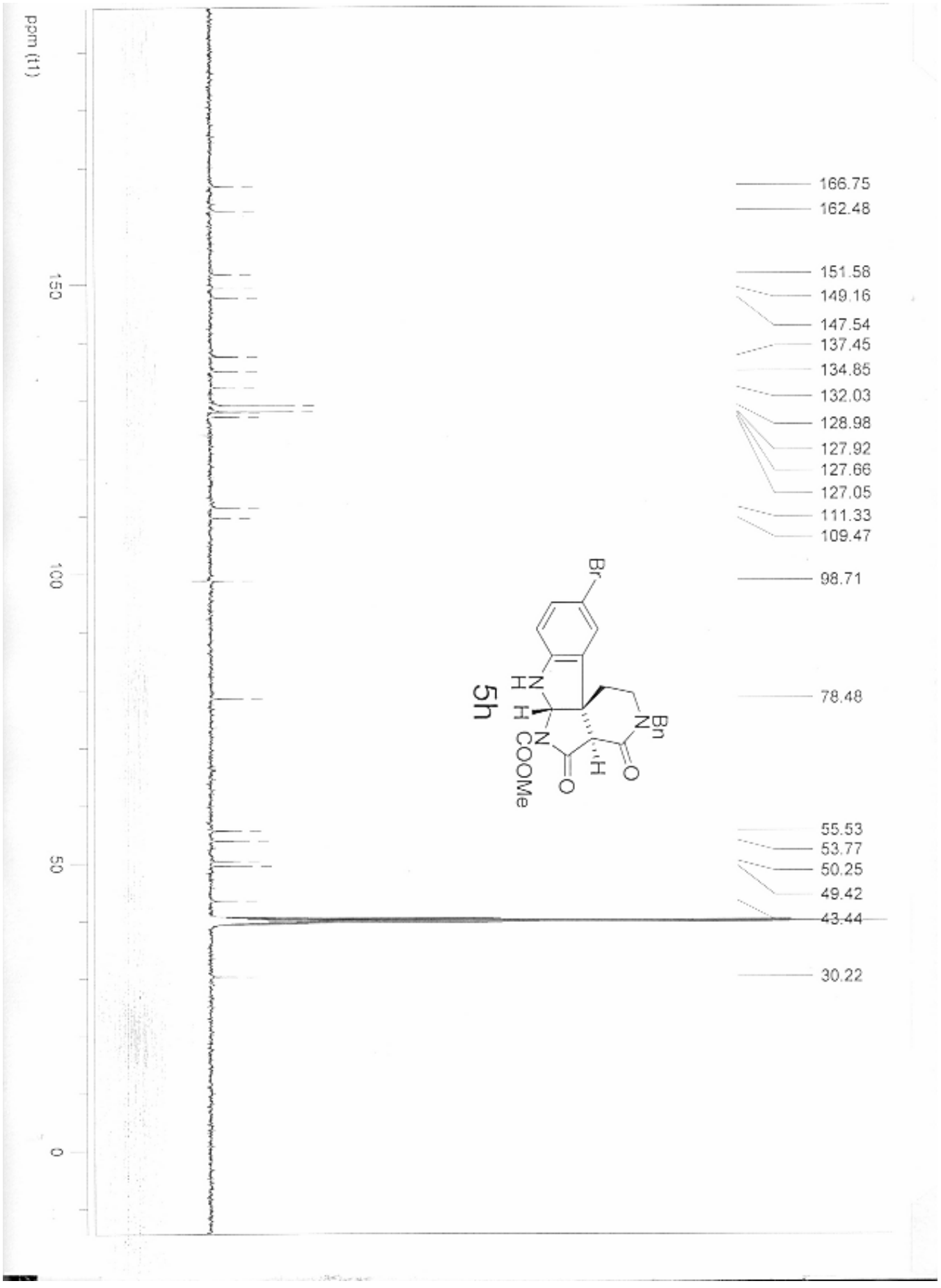
7.0
6.0
5.0
4.0
3.0
2.0
1.0
1.00
5.40
1.29
1.06
0.95
0.89
0.88
0.90
2.48
1.17
1.02
0.93
1.05
7.60
7.36
7.33
7.31
7.28
7.21
7.18
7.08
6.57
6.54
5.76
5.72
4.87
4.82
4.29
4.24
3.79
3.77
3.64
3.60
3.57
3.35
3.22
3.18
2.50
2.08
2.07
2.04
2.04
2.04
2.03
2.03
2.03
2.00
1.99
1.93
1.88
Br
N
H
H
N
COOMe
O
H
N
Bn
O
5h


ppm (t1)
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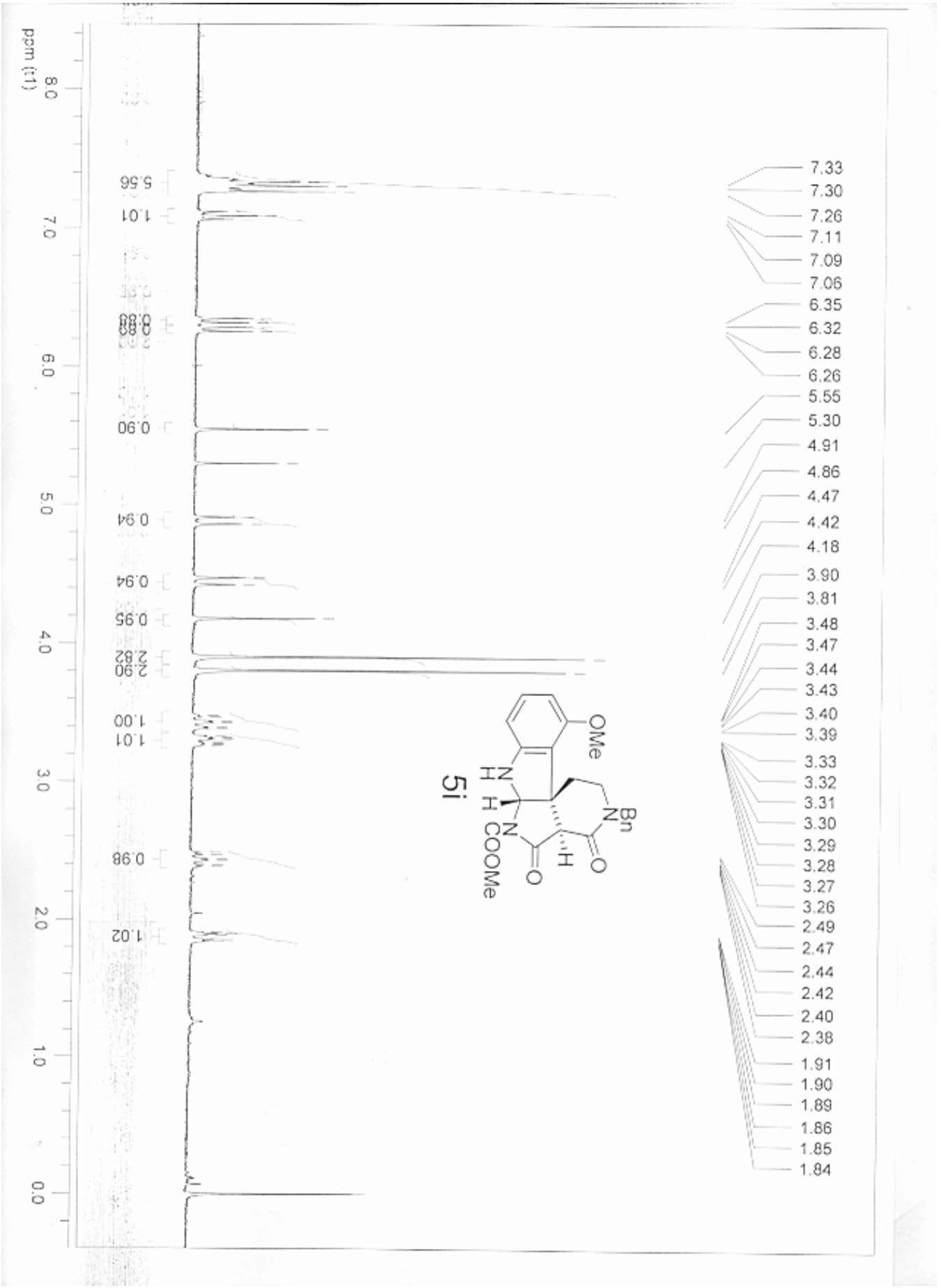
100
50
0
166.75
162.48
151.58
149.16
147.54
137.45
134.85
132.03
128.98
127.92
127.66
127.05
111.33
109.47
98.71
78.48
55.53
53.77
50.25
49.42
43.44
30.22
Br
N
H
H
N
COOMe
O
H
O
N
Bn
5h


ppm (t1)
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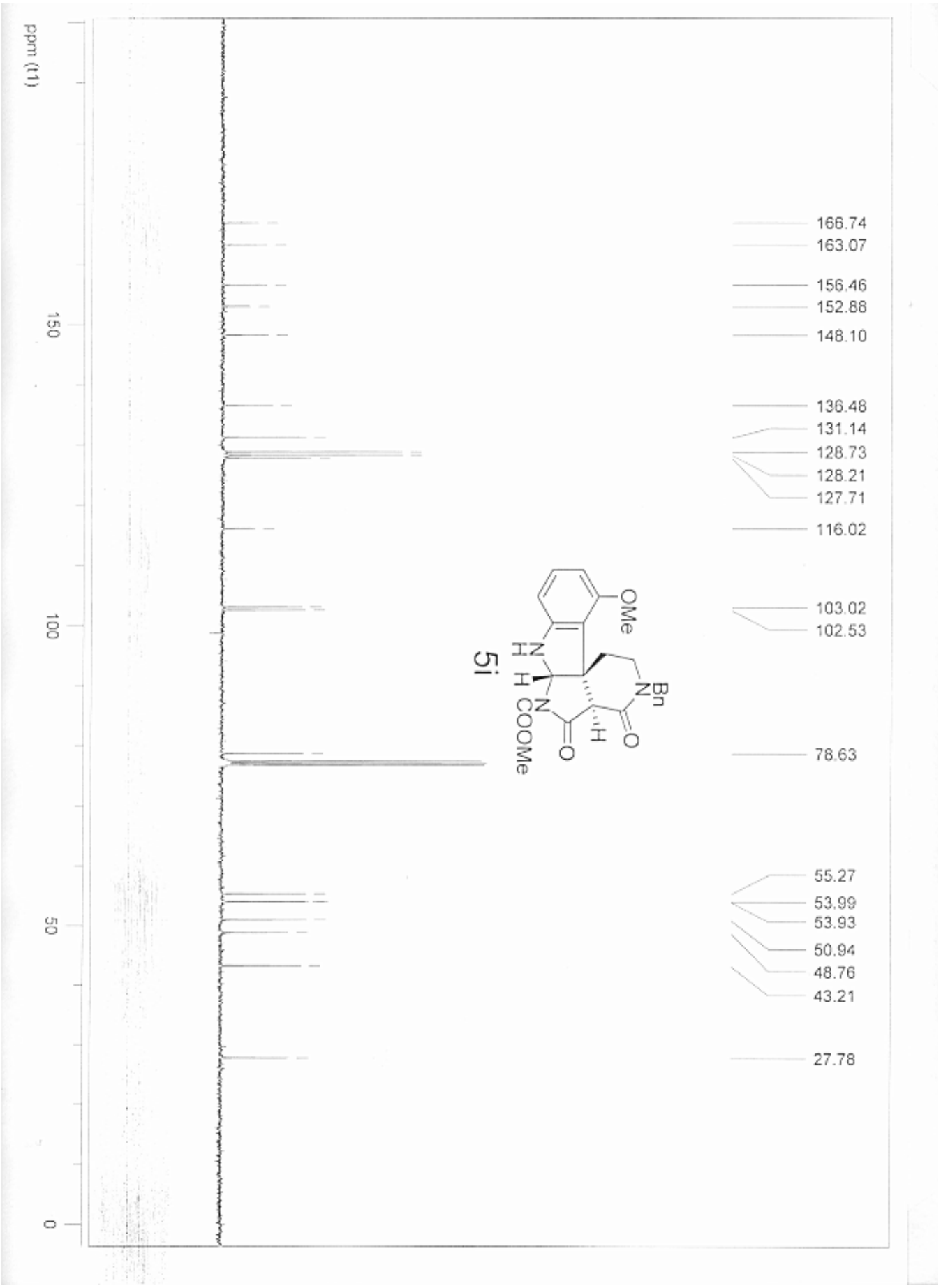
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
5.56
1.01
0.90
0.94
0.94
0.95
2.82
2.90
1.00
1.01
0.98
1.02
7.33
7.30
7.26
7.11
7.09
7.06
6.35
6.32
6.28
6.26
5.55
5.30
4.91
4.86
4.47
4.42
4.18
3.90
3.81
3.48
3.47
3.44
3.43
3.40
3.39
3.33
3.32
3.31
3.30
3.29
3.28
3.27
3.26
2.49
2.47
2.44
2.42
2.40
2.38
1.91
1.90
1.89
1.86
1.85
1.84
OMe
N
H
H
N
COOMe
O
Bn
N
O
H
5i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0
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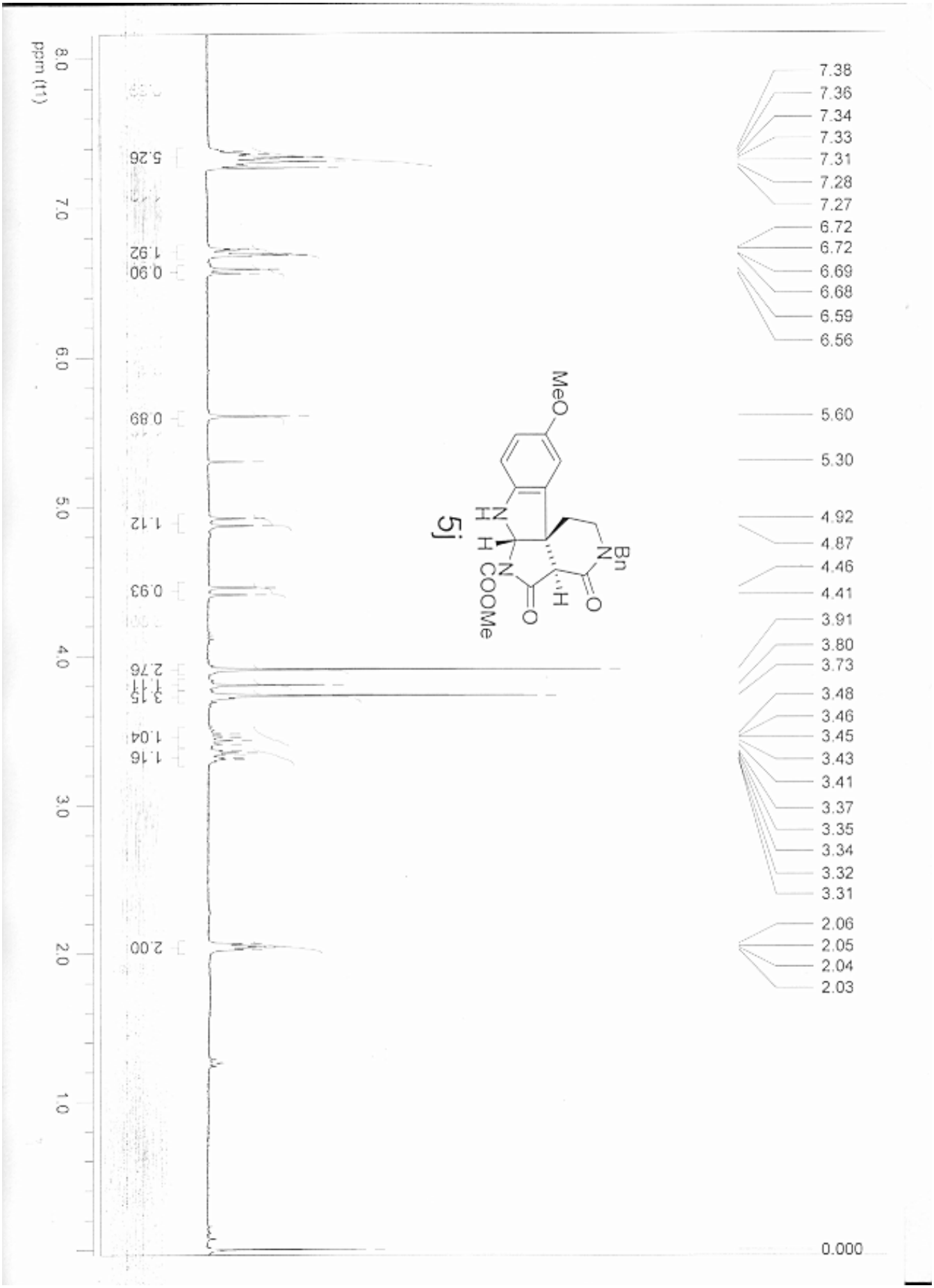
136.48
131.14
128.73
128.21
127.71
116.02
103.02
102.53
78.63
55.27
53.99
53.93
50.94
48.76
43.21
27.78
OMe
Bn
N
H
H
COOMe
5i


ppm (t1)
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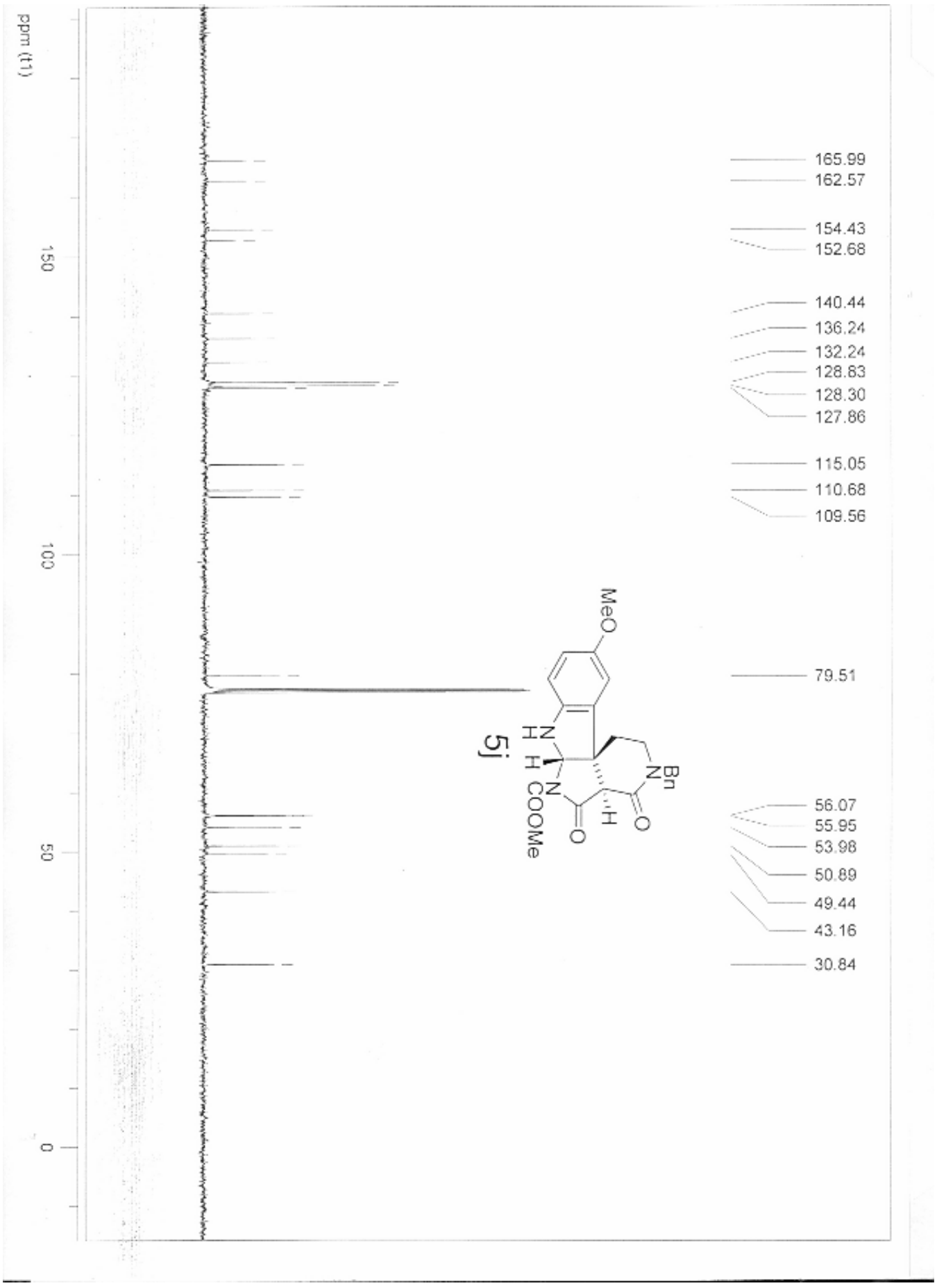
7.0
6.0
5.0
4.0
3.0
2.0
1.0
5.26
1.92
0.90
0.89
1.12
0.93
2.76
3.11
1.04
1.16
2.00
MeO
Bn
N
H
H
N
N
O
O
COOMe
5j
7.38
7.36
7.34
7.33
7.31
7.28
7.27
6.72
6.72
6.69
6.68
6.59
6.56
5.60
5.30
4.92
4.87
4.46
4.41
3.91
3.80
3.73
3.48
3.46
3.45
3.43
3.41
3.37
3.35
3.34
3.32
3.31
2.06
2.05
2.04
2.03
0.000


ppm (t1)
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100
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152.68
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136.24
132.24
128.83
128.30
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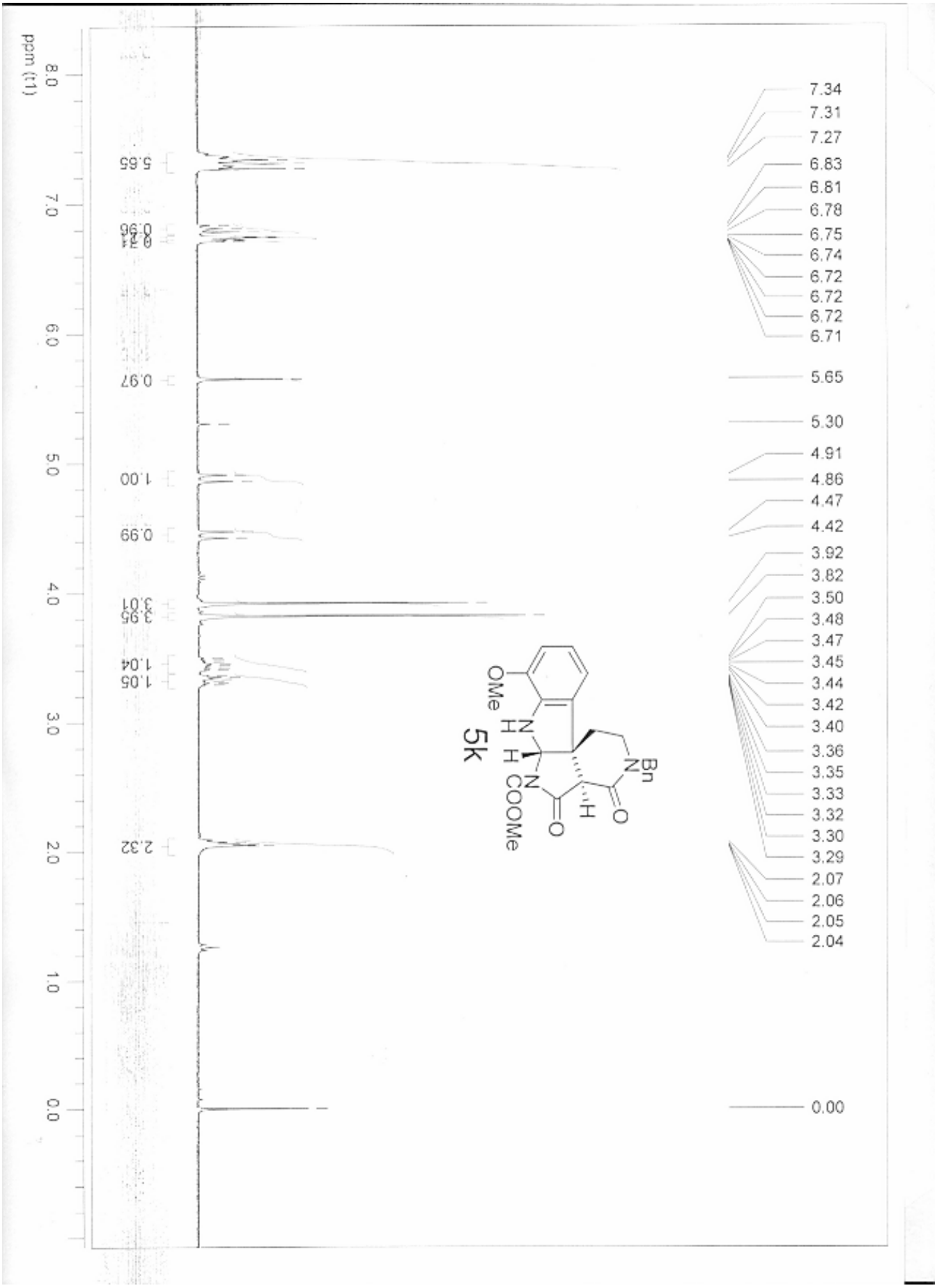
115.05
110.68
109.56
79.51
56.07
55.95
53.98
50.89
49.44
43.16
30.84
MeO
N
H
H
N
COOMe
Bn
N
O
O
H
5j


ppm (t1)
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2.0
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0.0
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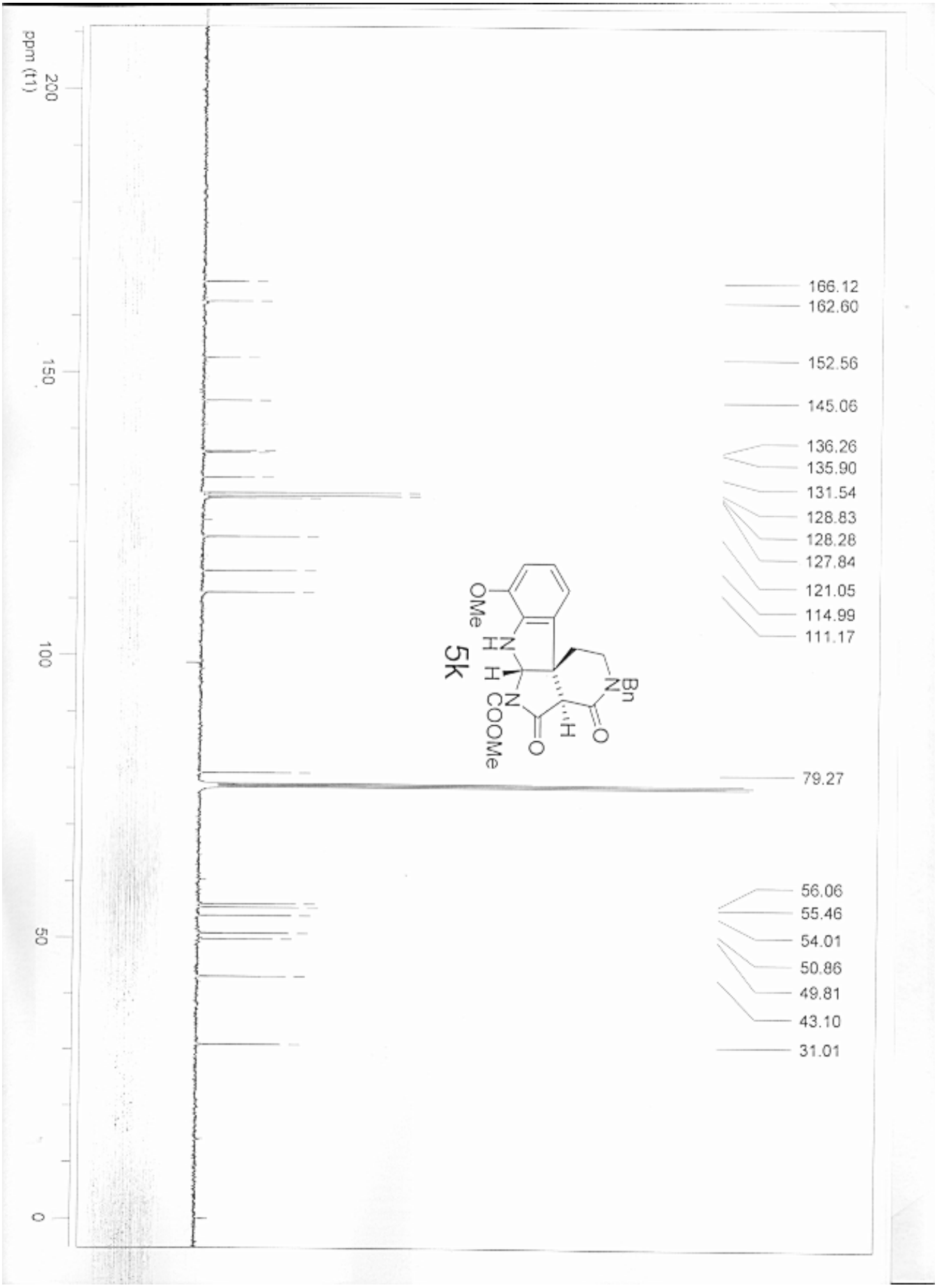
0.96
0.97
1.00
0.99
3.01
1.04
1.05
2.32
7.34
7.31
7.27
6.83
6.81
6.78
6.75
6.74
6.72
6.72
6.72
6.71
5.65
5.30
4.91
4.86
4.47
4.42
3.92
3.82
3.50
3.48
3.47
3.45
3.44
3.42
3.40
3.36
3.35
3.33
3.32
3.30
3.29
2.07
2.06
2.05
2.04
0.00
OMe
N
H
H
N
COOMe
5k
Bn
N
O
O
H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145.06
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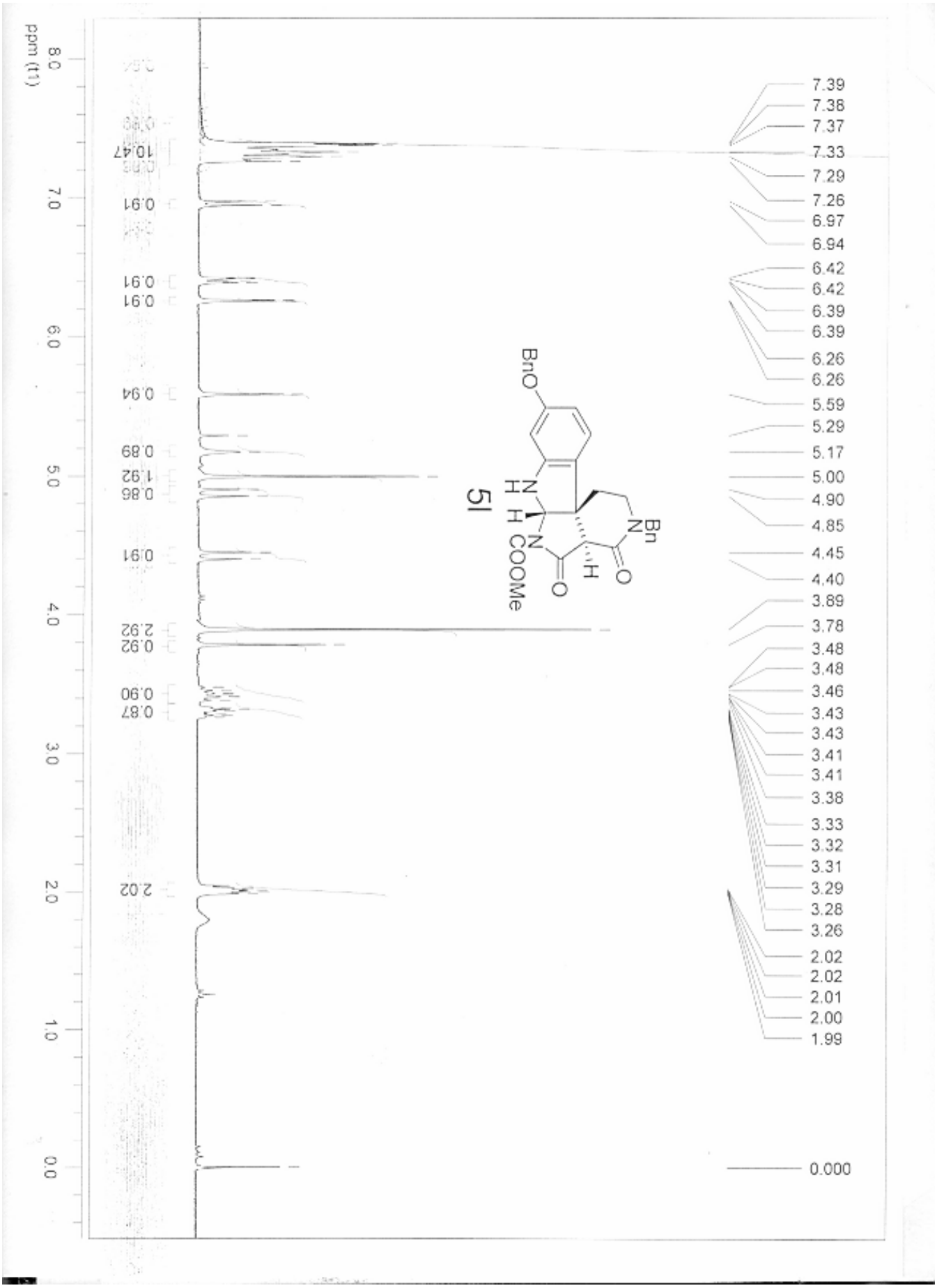
135.90
131.54
128.83
128.28
127.84
121.05
114.99
111.17
79.27
56.06
55.46
54.01
50.86
49.81
43.10
31.01
OMe
H
N
H
N
COOMe
5k
Bn
N
O
O
H


ppm (t1)
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2.0
1.0
0.0
10.47
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0.91
0.91
0.94
0.89
1.92
0.86
0.91
2.92
0.92
0.90
0.87
2.02
BnO
N
H
N
H
COOMe
Bn
N
O
O
H
5l
7.39
7.38
7.37
7.33
7.29
7.26
6.97
6.94
6.42
6.42
6.39
6.39
6.26
6.26
5.59
5.29
5.17
5.00
4.90
4.85
4.45
4.40
3.89
3.78
3.48
3.48
3.46
3.43
3.43
3.41
3.41
3.38
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3.31
3.29
3.28
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2.02
2.01
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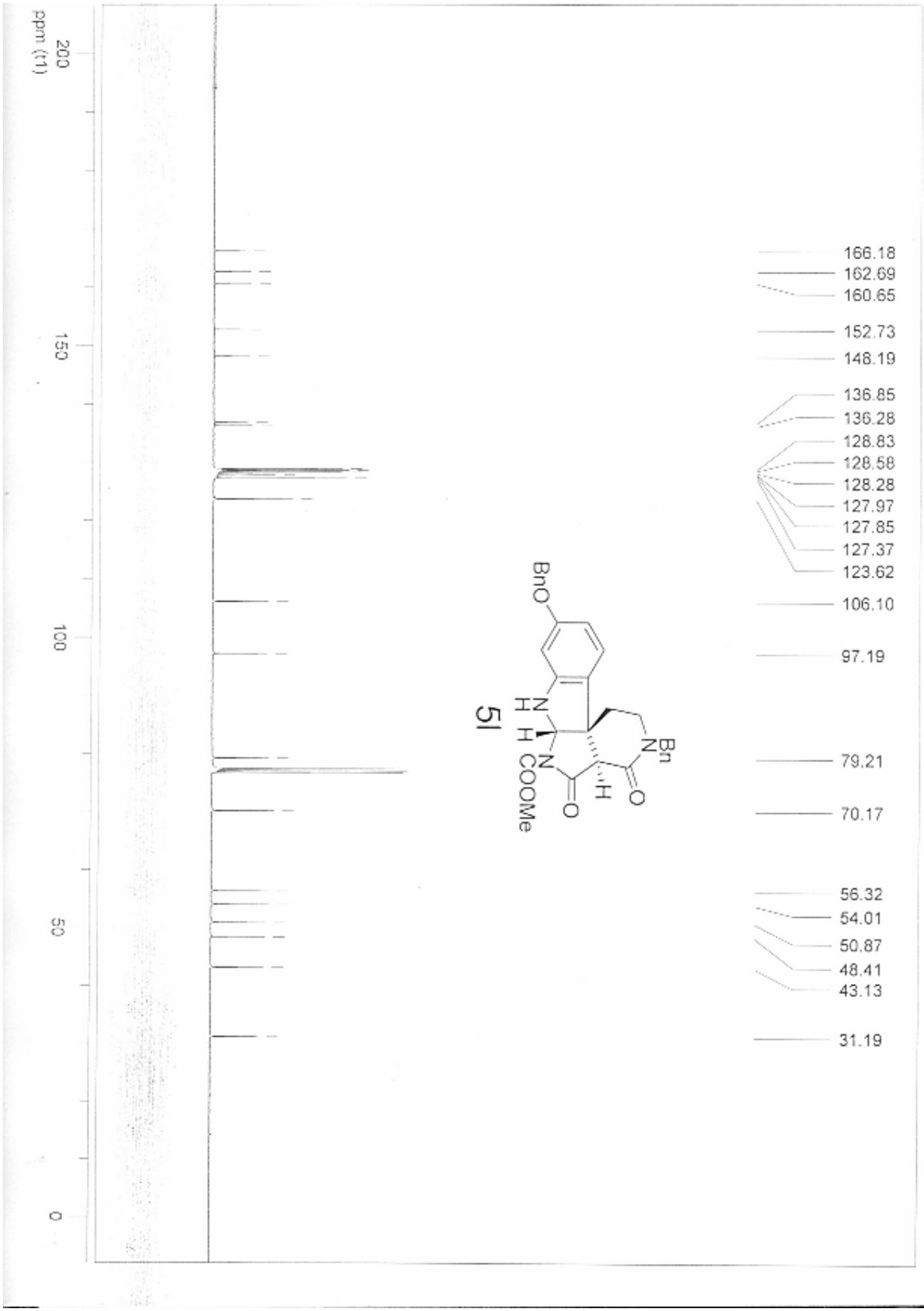
1.99
0.000


ppm (t1)
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100
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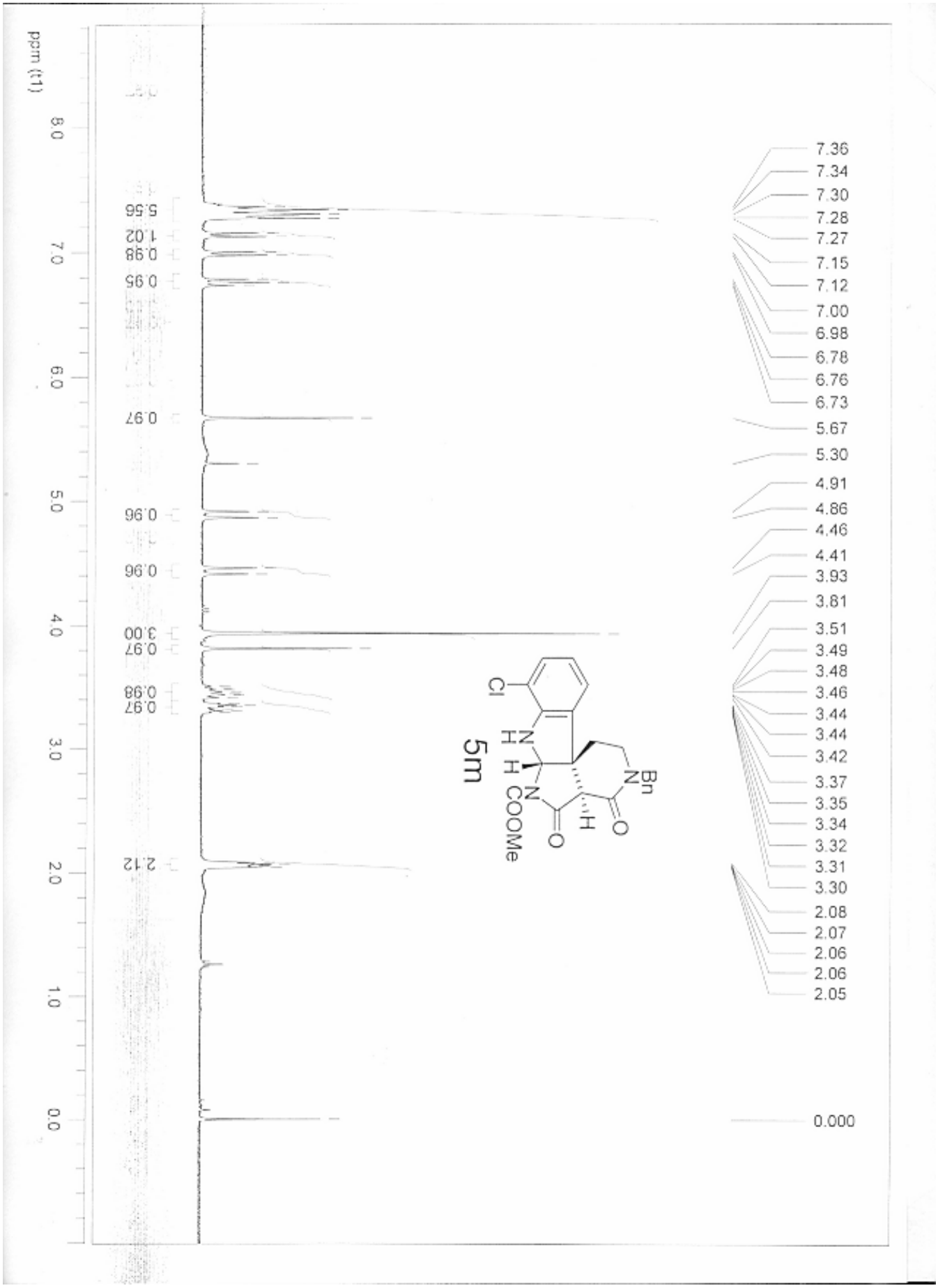
160.65
152.73
148.19
136.85
136.28
128.83
128.58
128.28
127.97
127.85
127.37
123.62
106.10
97.19
79.21
70.17
56.32
54.01
50.87
48.41
43.13
31.19
BnO
N
H
H
N
COOMe
O
H
O
N
Bn
O
5l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3.0
2.0
1.0
0.0
5.56
1.02
0.98
0.95
0.97
0.96
0.96
3.00
0.97
0.98
0.97
2.12
7.36
7.34
7.30
7.28
7.27
7.15
7.12
7.00
6.98
6.78
6.76
6.73
5.67
5.30
4.91
4.86
4.46
4.41
3.93
3.81
3.51
3.49
3.48
3.46
3.44
3.44
3.42
3.37
3.35
3.34
3.32
3.31
3.30
2.08
2.07
2.06
2.06
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0.000
5m
Cl
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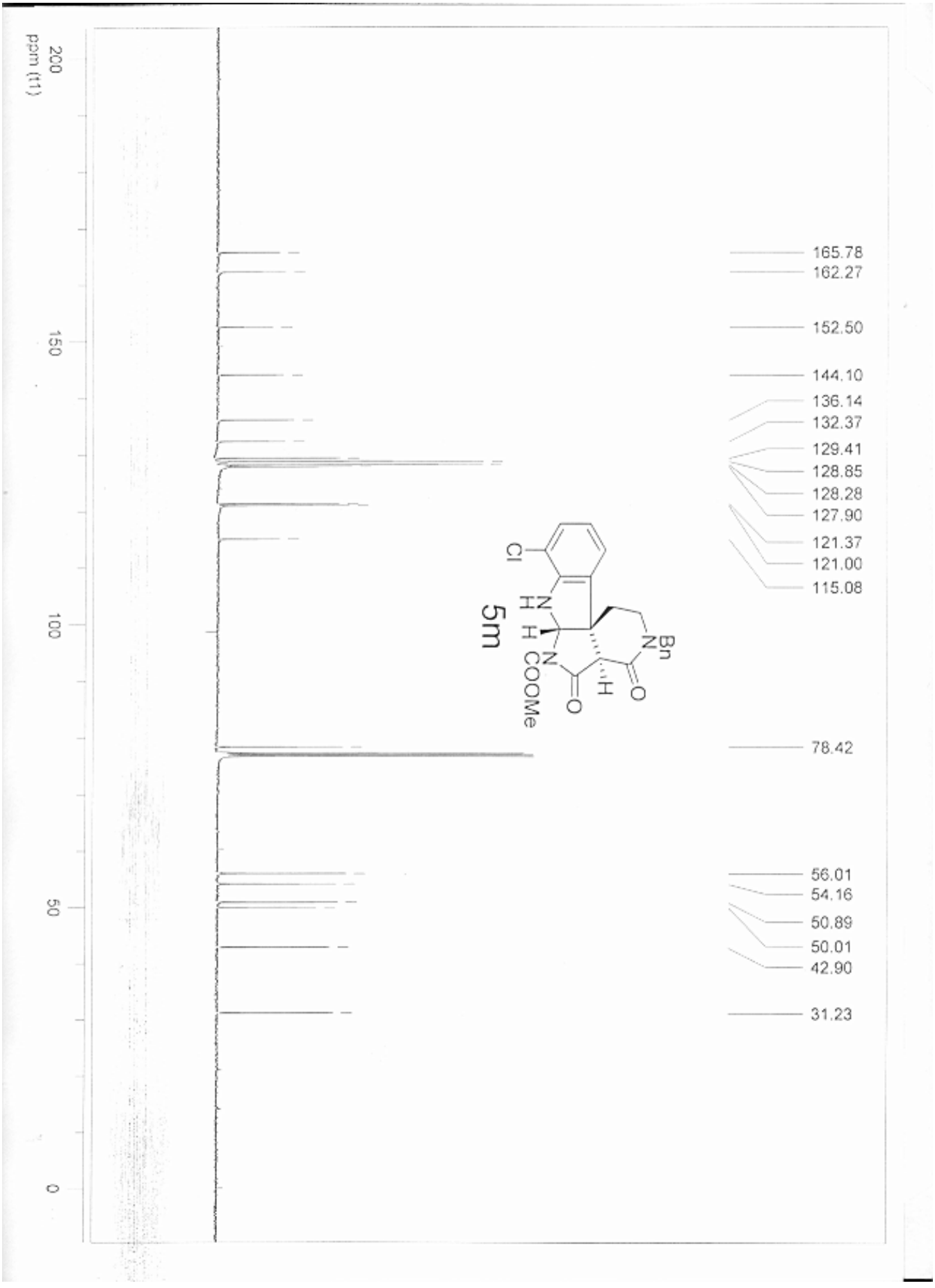
N
COOMe
O
H
N
Bn
O


ppm (t1)
200
150
100
50
0
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162.27
152.50
144.10
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132.37
129.41
128.85
128.28
127.90
121.37
121.00
115.08
78.42
56.01
54.16
50.89
50.01
42.90
31.23
5m
Cl
H
N
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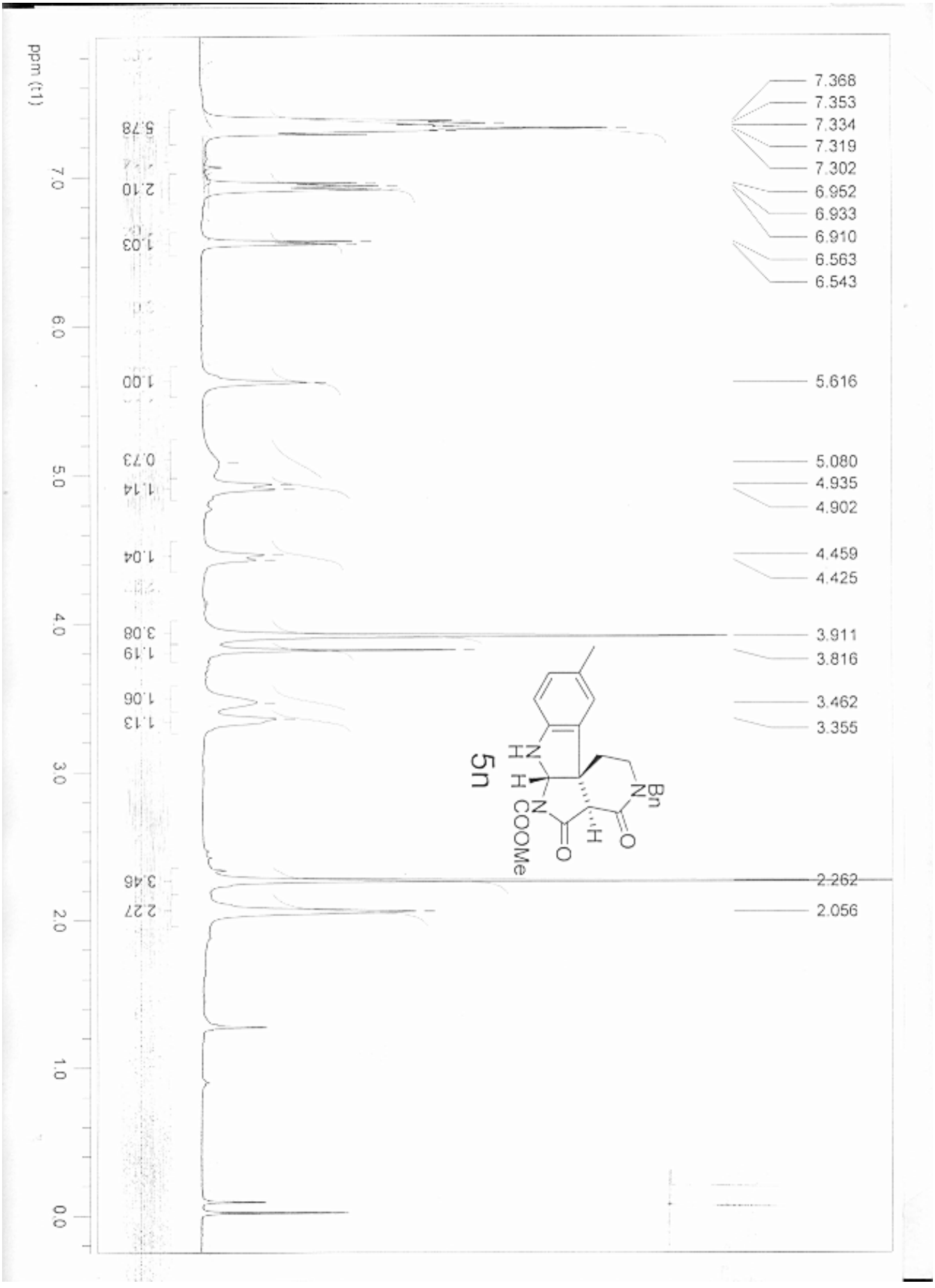
N
COOMe
O
O
H
N
Bn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4.0
3.0
2.0
1.0
0.0
5.78
2.10
1.03
1.00
0.73
1.14
1.04
3.08
1.19
1.06
1.13
3.46
2.27
7.368
7.353
7.334
7.319
7.302
6.952
6.933
6.910
6.563
6.543
5.616
5.080
4.935
4.902
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4.425
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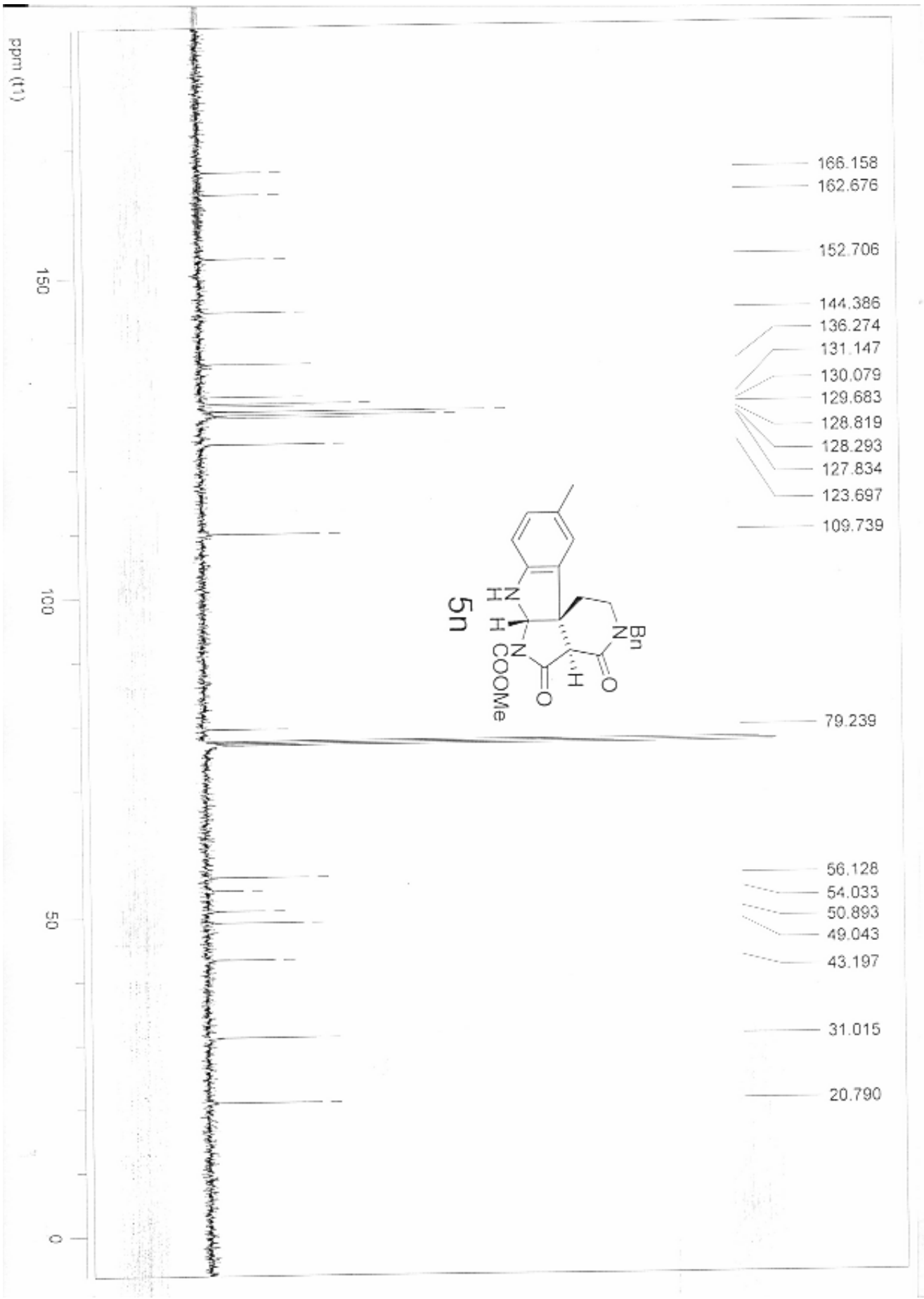
3.816
3.462
3.355
2.262
2.056
5n
Bn
COOMe


ppm (t1)
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144.386
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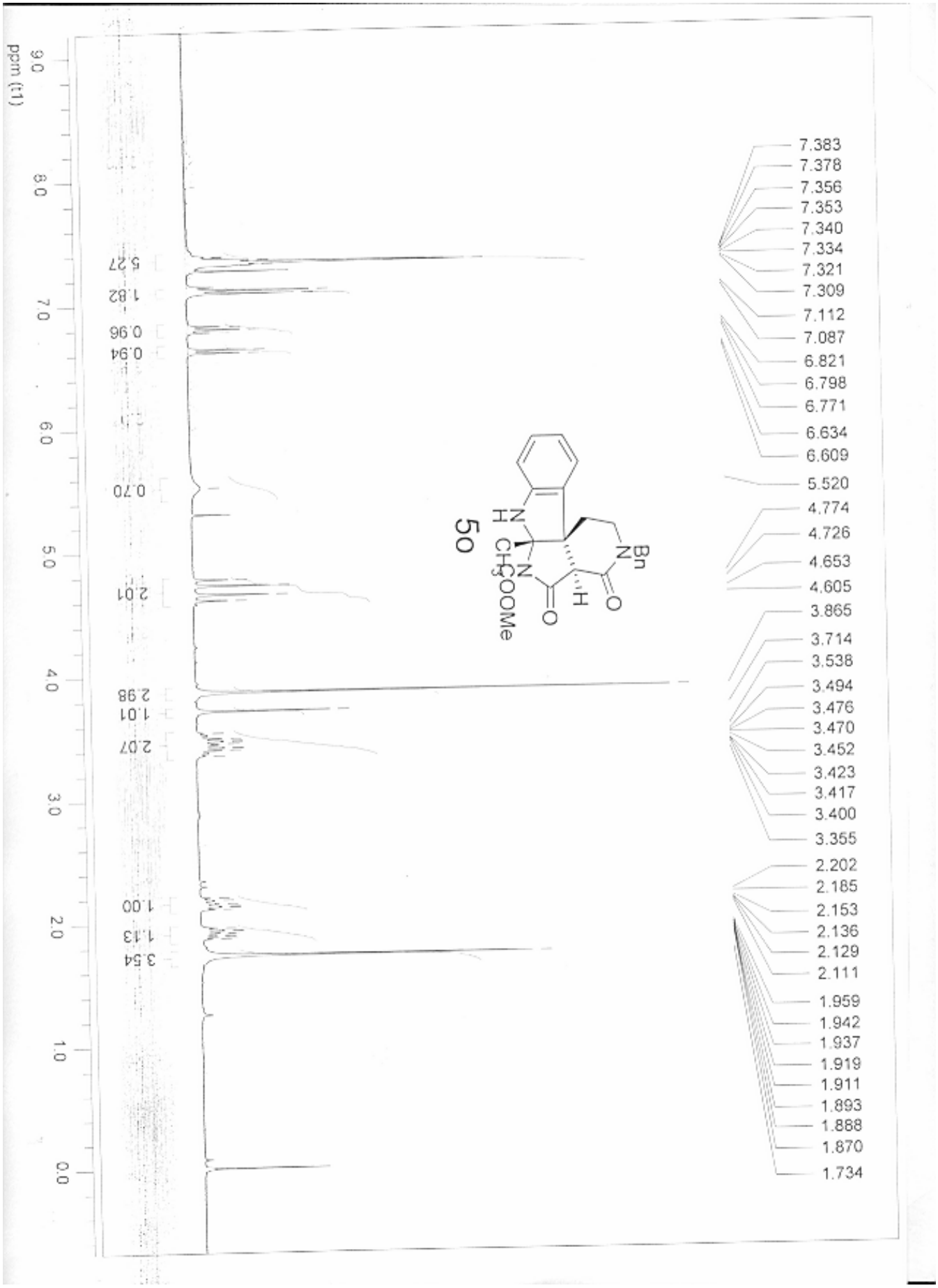
131.147
130.079
129.683
128.819
128.293
127.834
123.697
109.739
79.239
56.128
54.033
50.893
49.043
43.197
31.015
20.790
5n
Bn
COOMe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6.0
5.0
4.0
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5.27
1.82
0.96
0.94
0.70
2.01
2.98
1.01
2.07
1.00
1.13
3.54
50
Bn
N
O
O
H
N
N
H
CH3COOMe
7.383
7.378
7.356
7.353
7.340
7.334
7.321
7.309
7.112
7.087
6.821
6.798
6.771
6.634
6.609
5.520
4.774
4.726
4.653
4.605
3.865
3.714
3.538
3.494
3.476
3.470
3.452
3.423
3.417
3.400
3.355
2.202
2.185
2.153
2.136
2.129
2.111
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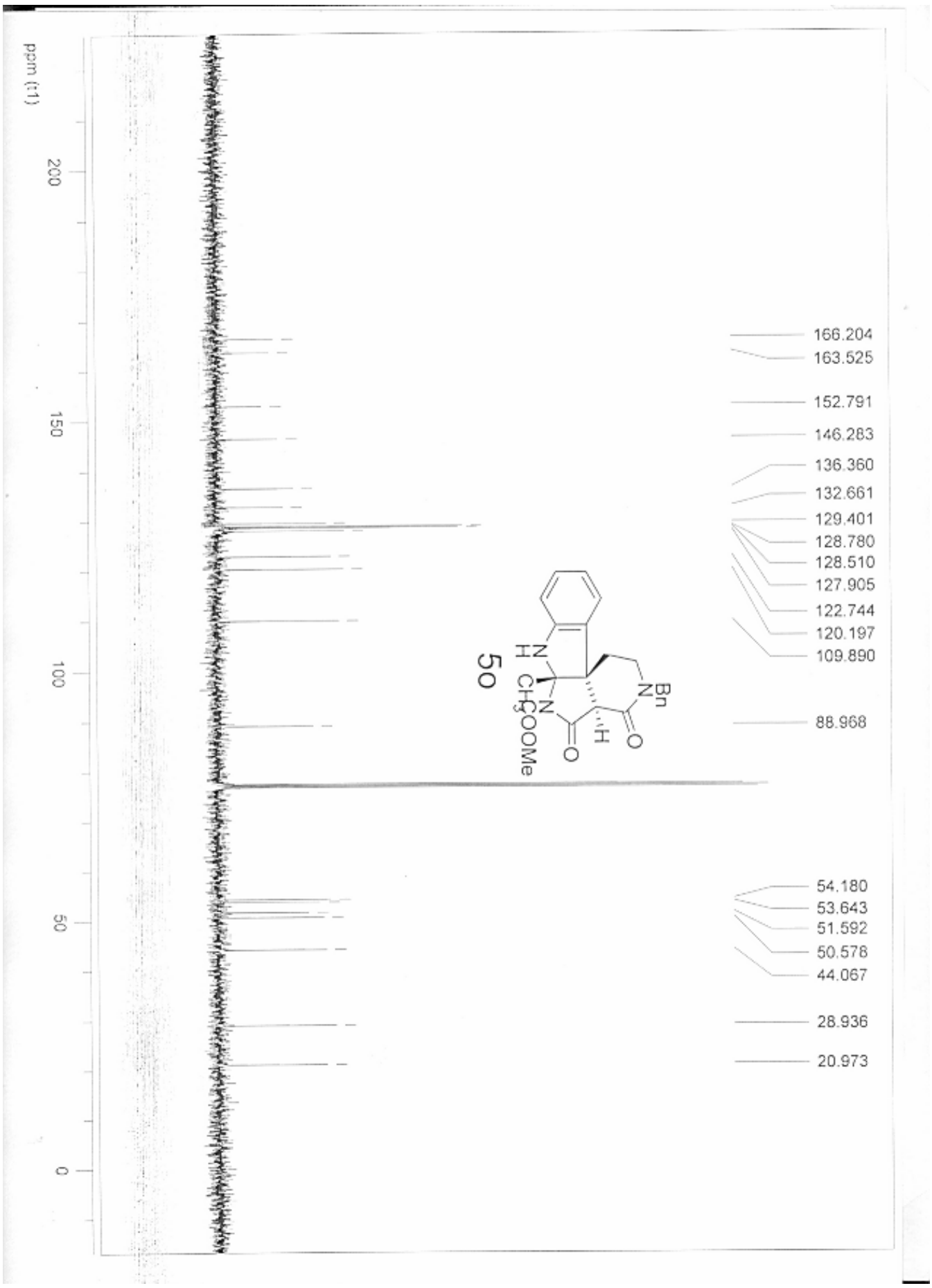
1.942
1.937
1.919
1.911
1.893
1.888
1.870
1.734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132.661
129.401
128.780
128.510
127.905
122.744
120.197
109.890
88.968
54.180
53.643
51.592
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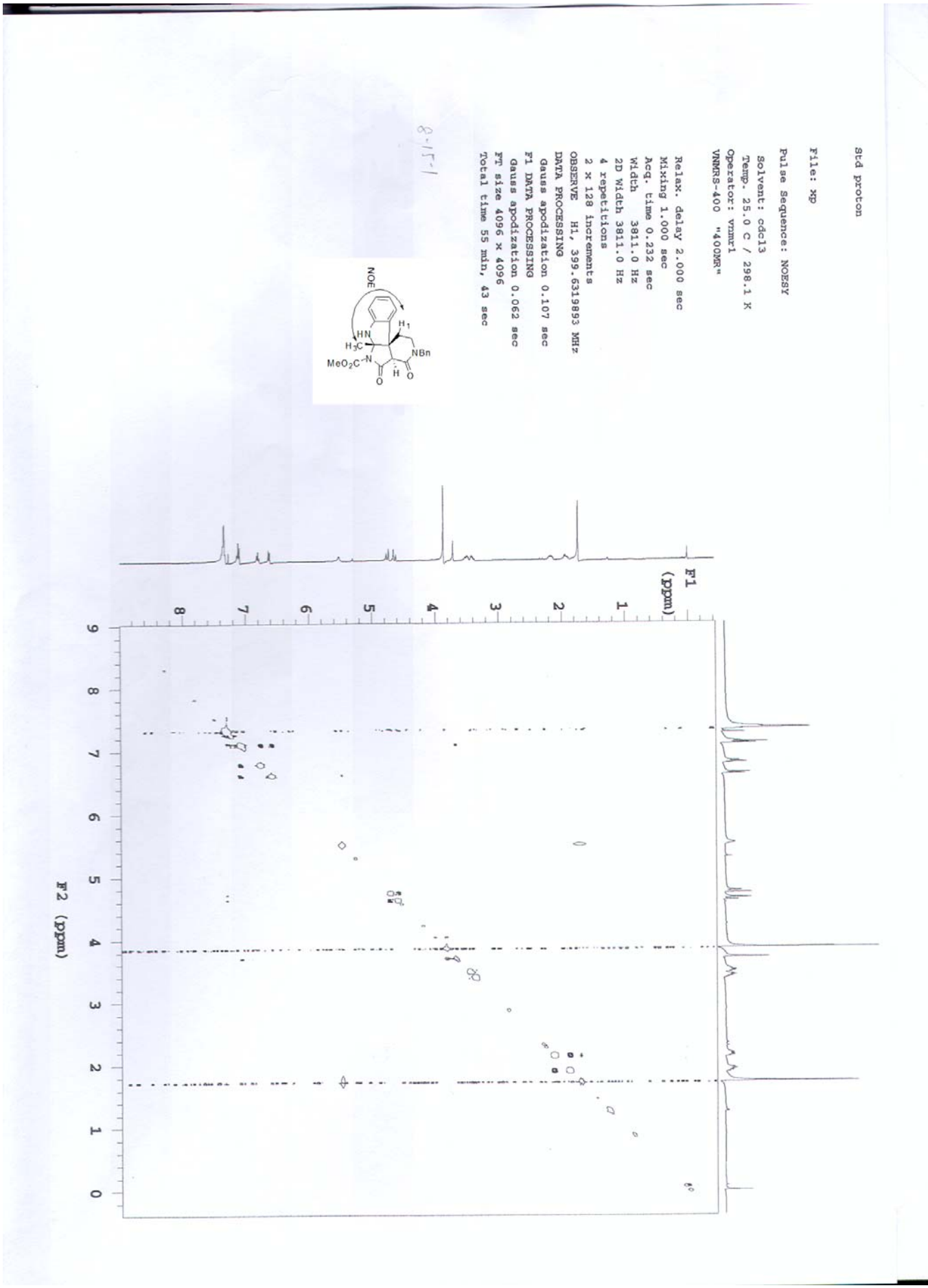
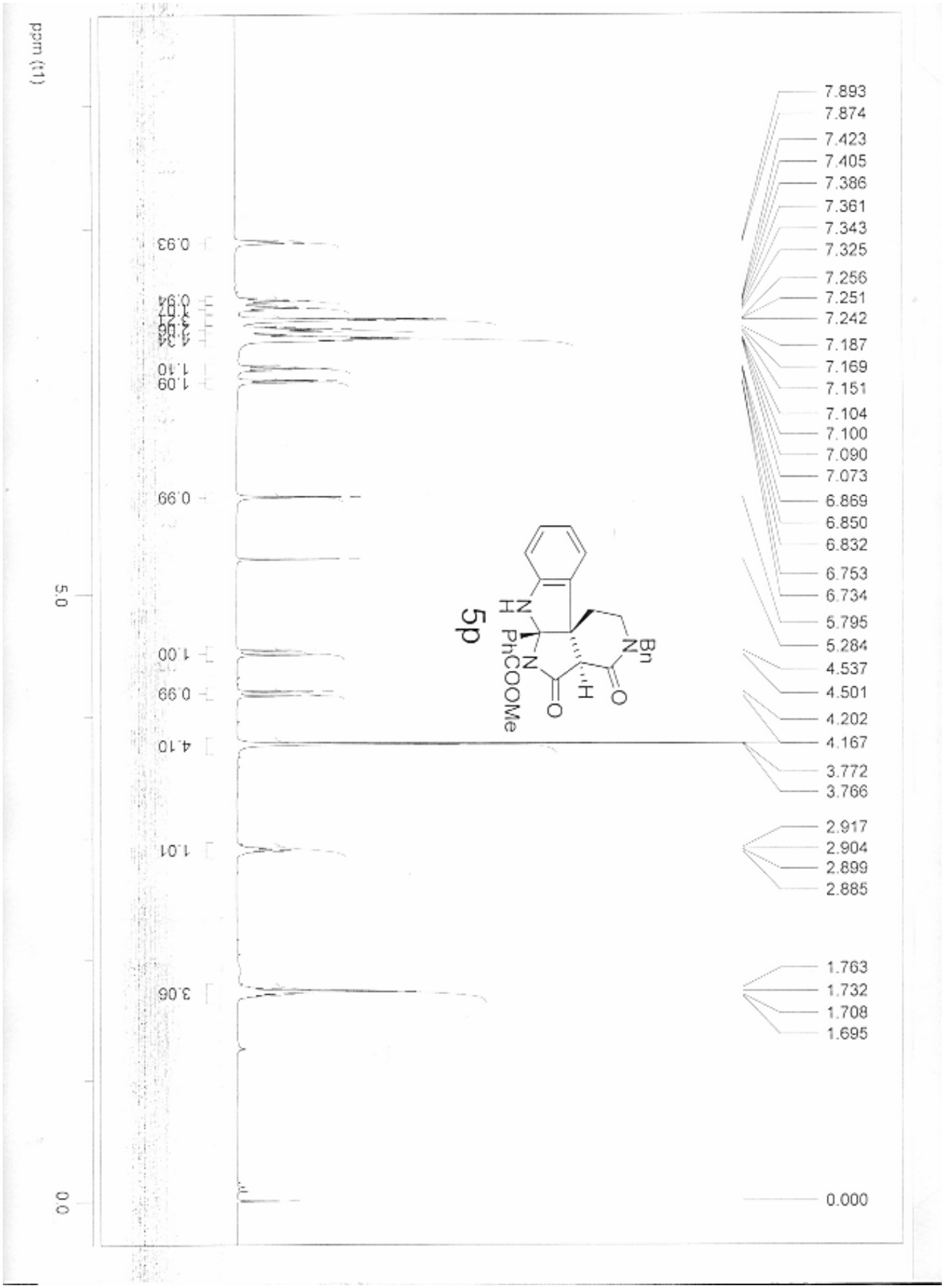
28.936
20.973
5o
Bn
N
H
N
CH3OOMe
H
O
O


Std proton
File: xp
Pulse Sequence: NOESY
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: vnmr1
VNMRS-400 "400MR"
Relax. delay 2.000 sec
Mixing 1.000 sec
Acq. time 0.232 sec
Width 3811.0 Hz
2D Width 3811.0 Hz
4 repetitions
2 x 128 increments
OBSERVE H1, 399.6319893 MHz
DATA PROCESSING
Gauss apodization 0.107 sec
F1 DATA PROCESSING
Gauss apodization 0.062 sec
FT size 4096 x 4096
Total time 55 min, 43 sec
8-15-1
NOE
HN
H3C
MeO2C
N
H1
NBn
O
H
O
F1 (ppm)
F2 (ppm)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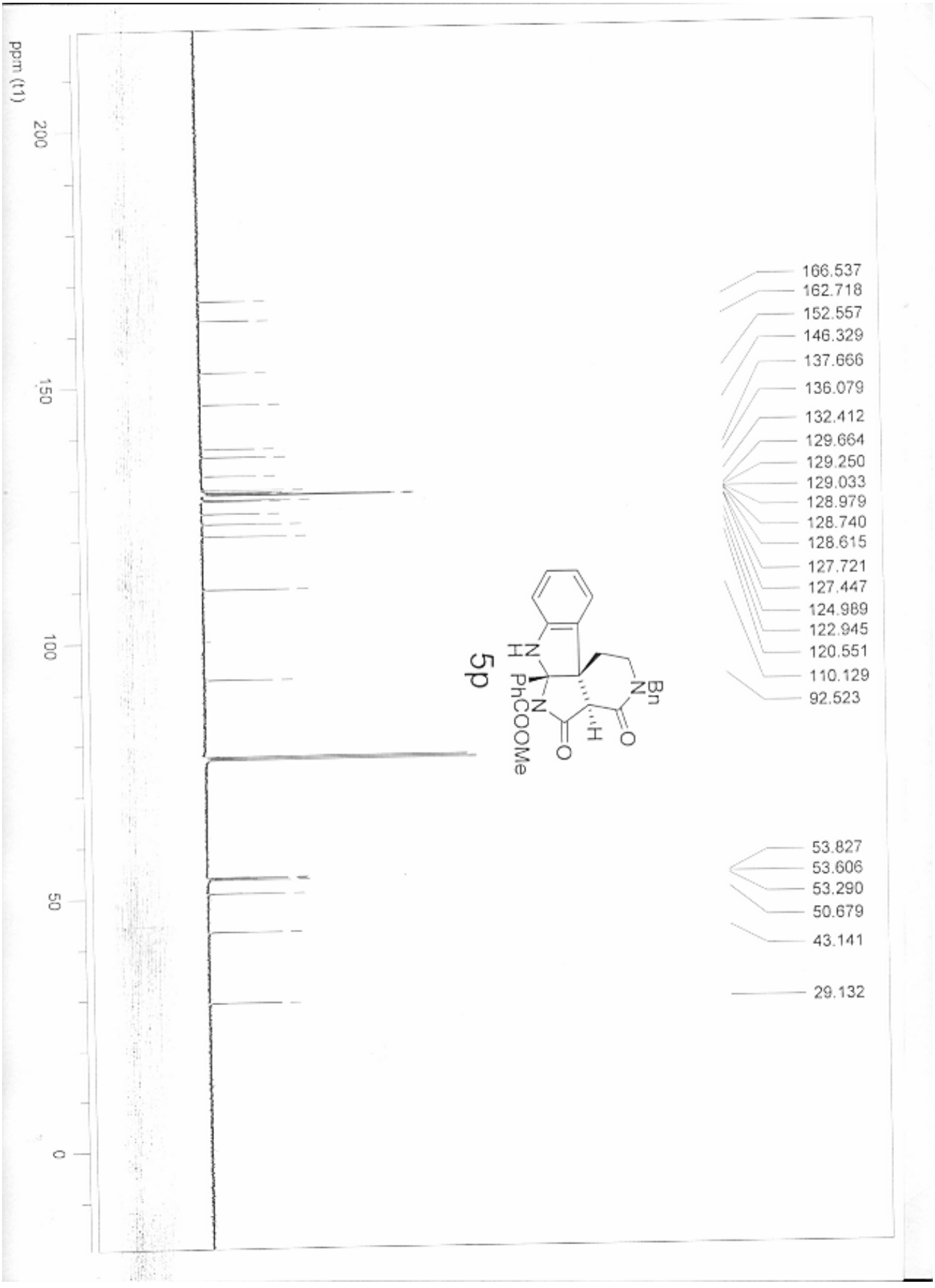
1.09
1.10
0.99
1.00
0.99
4.10
1.01
3.06
7.893
7.874
7.423
7.405
7.386
7.361
7.343
7.325
7.256
7.251
7.242
7.187
7.169
7.151
7.104
7.100
7.090
7.073
6.869
6.850
6.832
6.753
6.734
5.795
5.284
4.537
4.501
4.202
4.167
3.772
3.766
2.917
2.904
2.899
2.885
1.763
1.732
1.708
1.695
0.000
5p
Bn
PhCOOMe
H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132.412
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129.250
129.033
128.979
128.740
128.615
127.721
127.447
124.989
122.945
120.551
110.129
92.523
53.827
53.606
53.290
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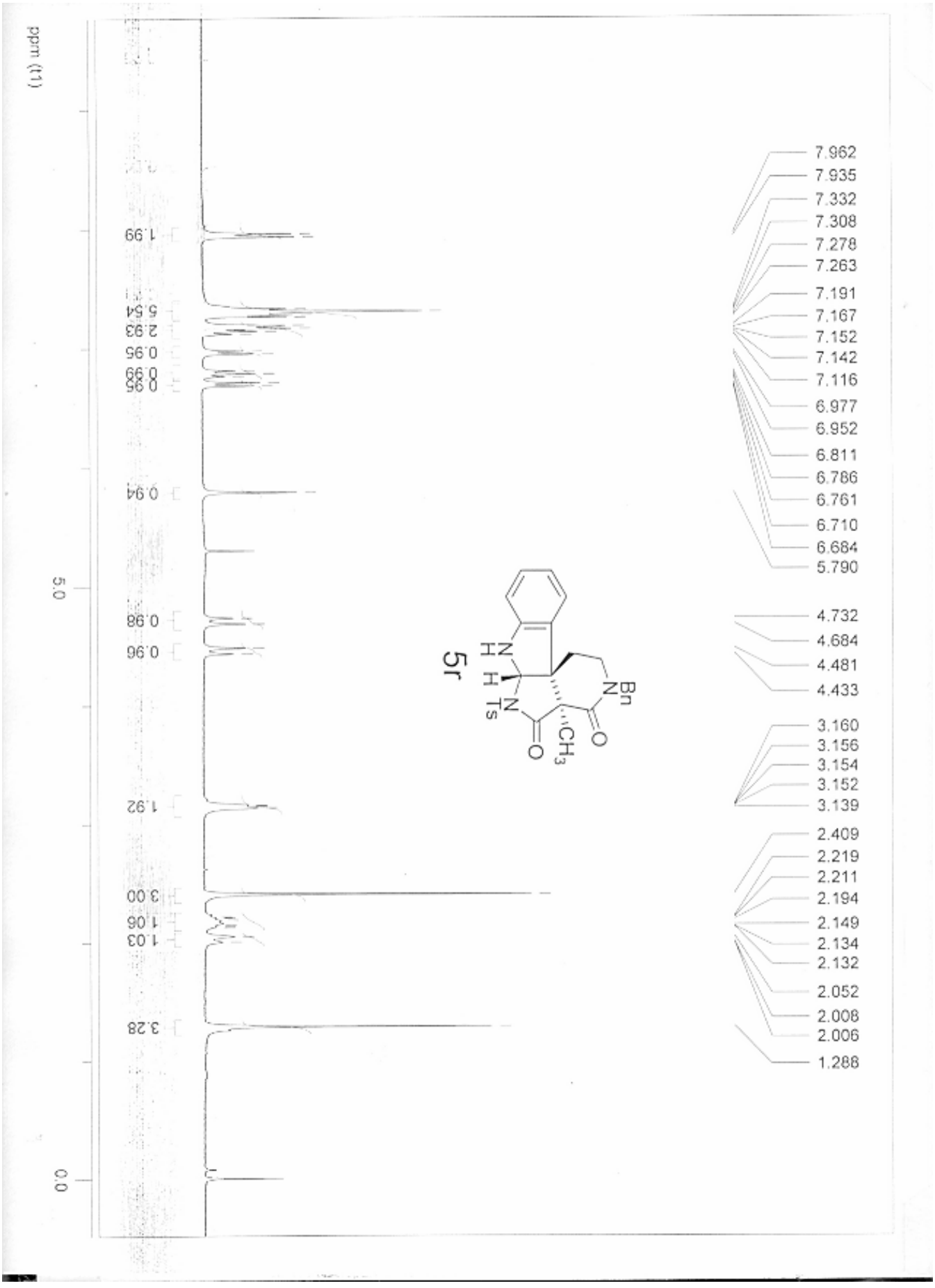
43.141
29.132
5p
N
H
N
PhCOOMe
Bn
N
H
O
O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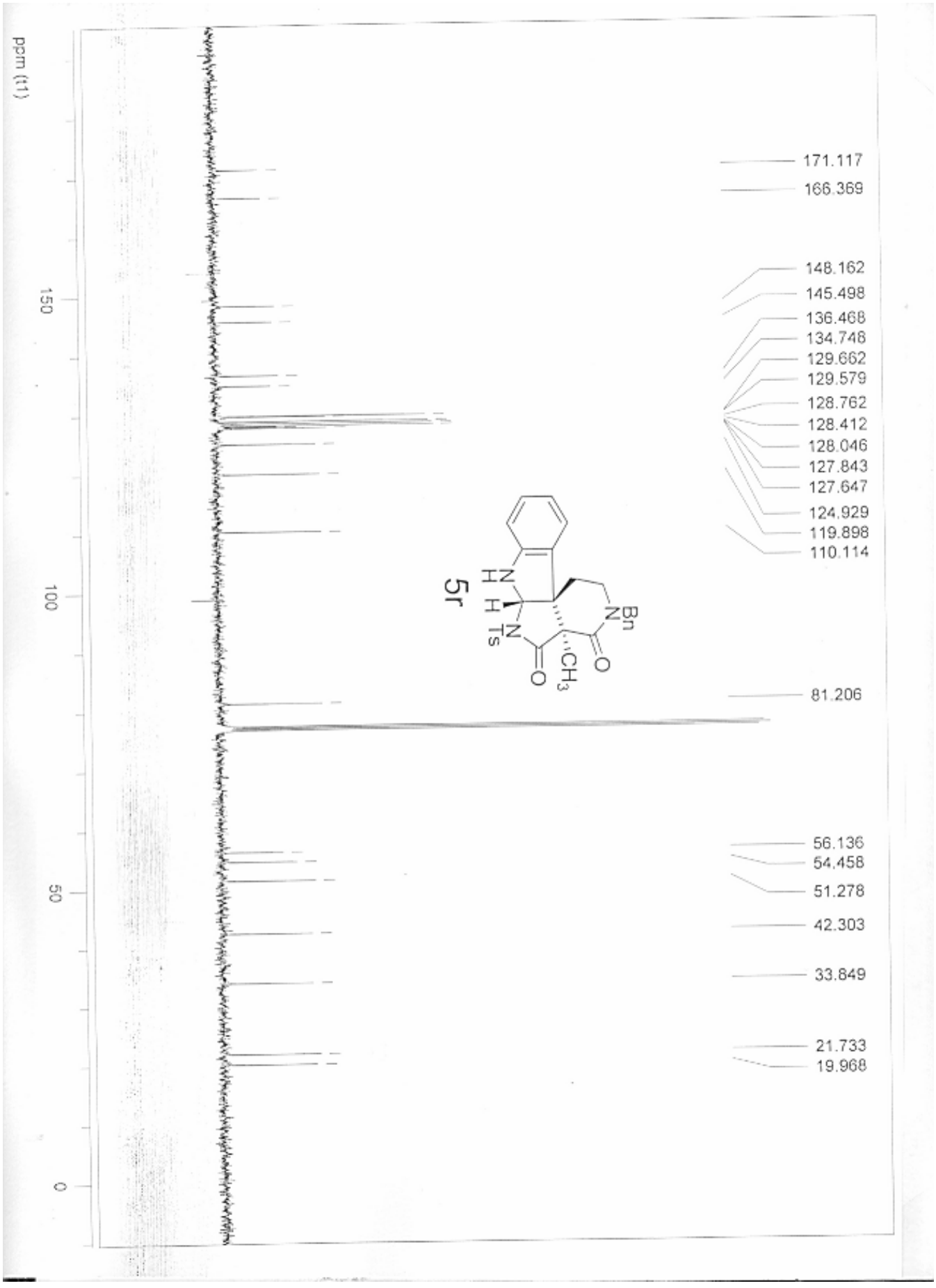
5.54
2.93
0.95
0.99
0.95
0.94
0.98
0.96
1.92
3.00
1.06
1.03
3.28
7.962
7.935
7.332
7.308
7.278
7.263
7.191
7.167
7.152
7.142
7.116
6.977
6.952
6.811
6.786
6.761
6.710
6.684
5.790
4.732
4.684
4.481
4.433
3.160
3.156
3.154
3.152
3.139
2.409
2.219
2.211
2.194
2.149
2.134
2.132
2.052
2.008
2.006
1.288
5r
Bn
Ts
CH3


ppm (t1)
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100
50
0
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166.369
148.162
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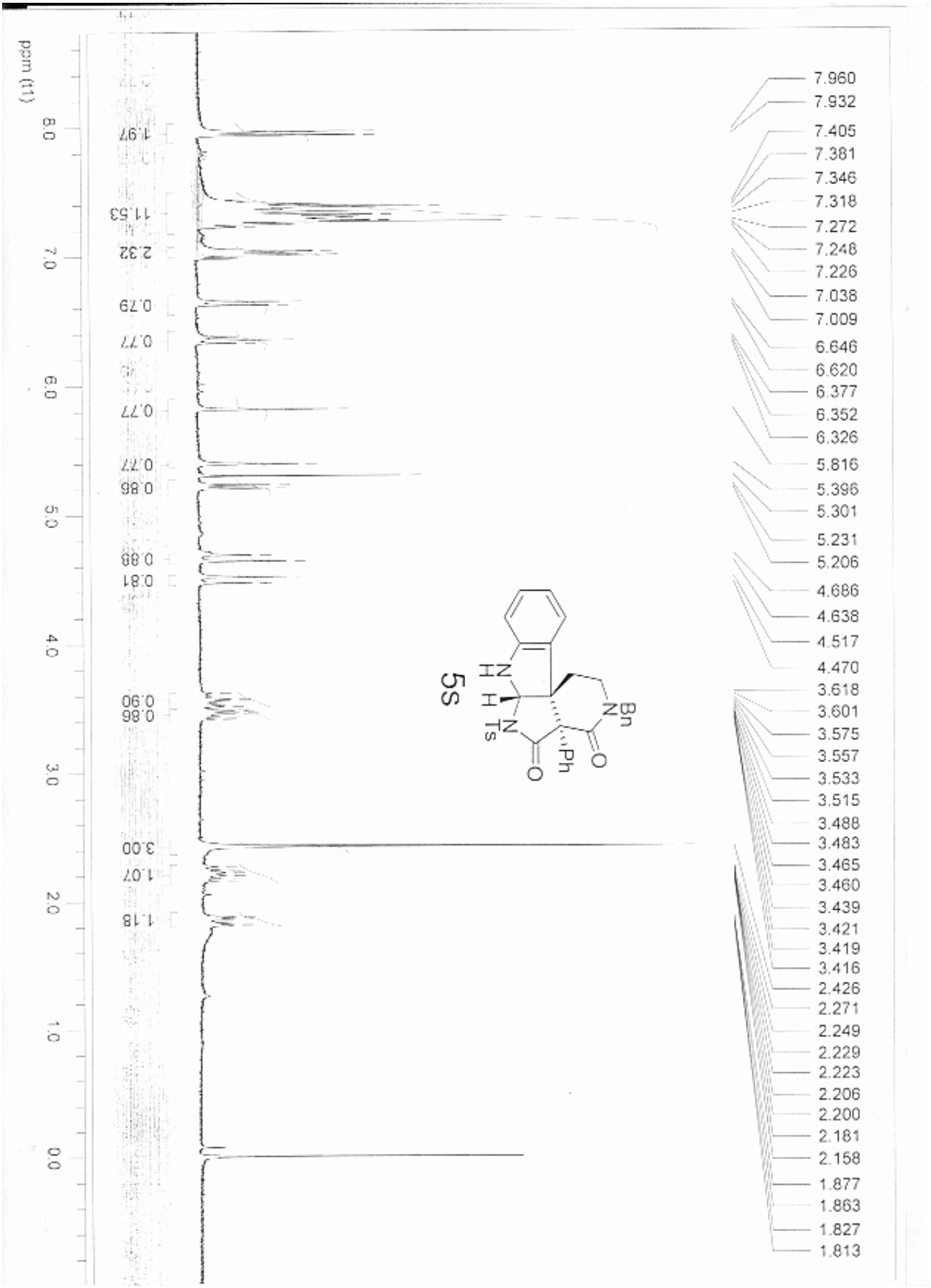
136.468
134.748
129.662
129.579
128.762
128.412
128.046
127.843
127.647
124.929
119.898
110.114
81.206
56.136
54.458
51.278
42.303
33.849
21.733
19.968
5r
Bn
Ts
CH3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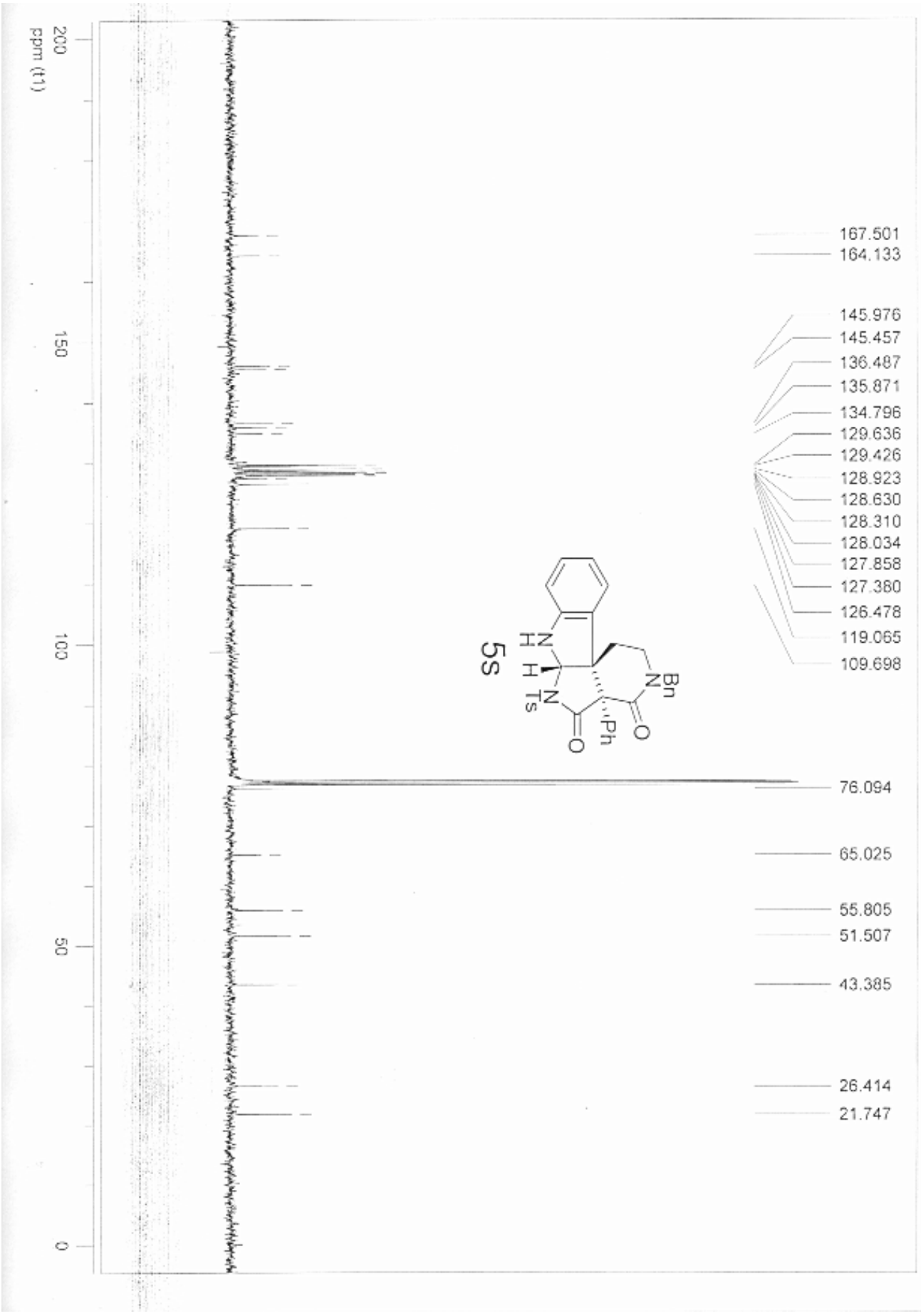
7.405
7.381
7.346
7.318
7.272
7.248
7.226
7.038
7.009
6.646
6.620
6.377
6.352
6.326
5.816
5.396
5.301
5.231
5.206
4.686
4.638
4.517
4.470
3.618
3.601
3.575
3.557
3.533
3.515
3.488
3.483
3.465
3.460
3.439
3.421
3.419
3.416
2.426
2.271
2.249
2.229
2.223
2.206
2.200
2.181
2.158
1.877
1.863
1.827
1.813
1.97
11.53
2.32
0.79
0.77
0.77
0.77
0.86
0.89
0.81
0.90
0.86
3.00
1.07
1.18
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
5s
N
H
H
N
Ts
Bn
N
O
O
Ph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100
50
0
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164.133
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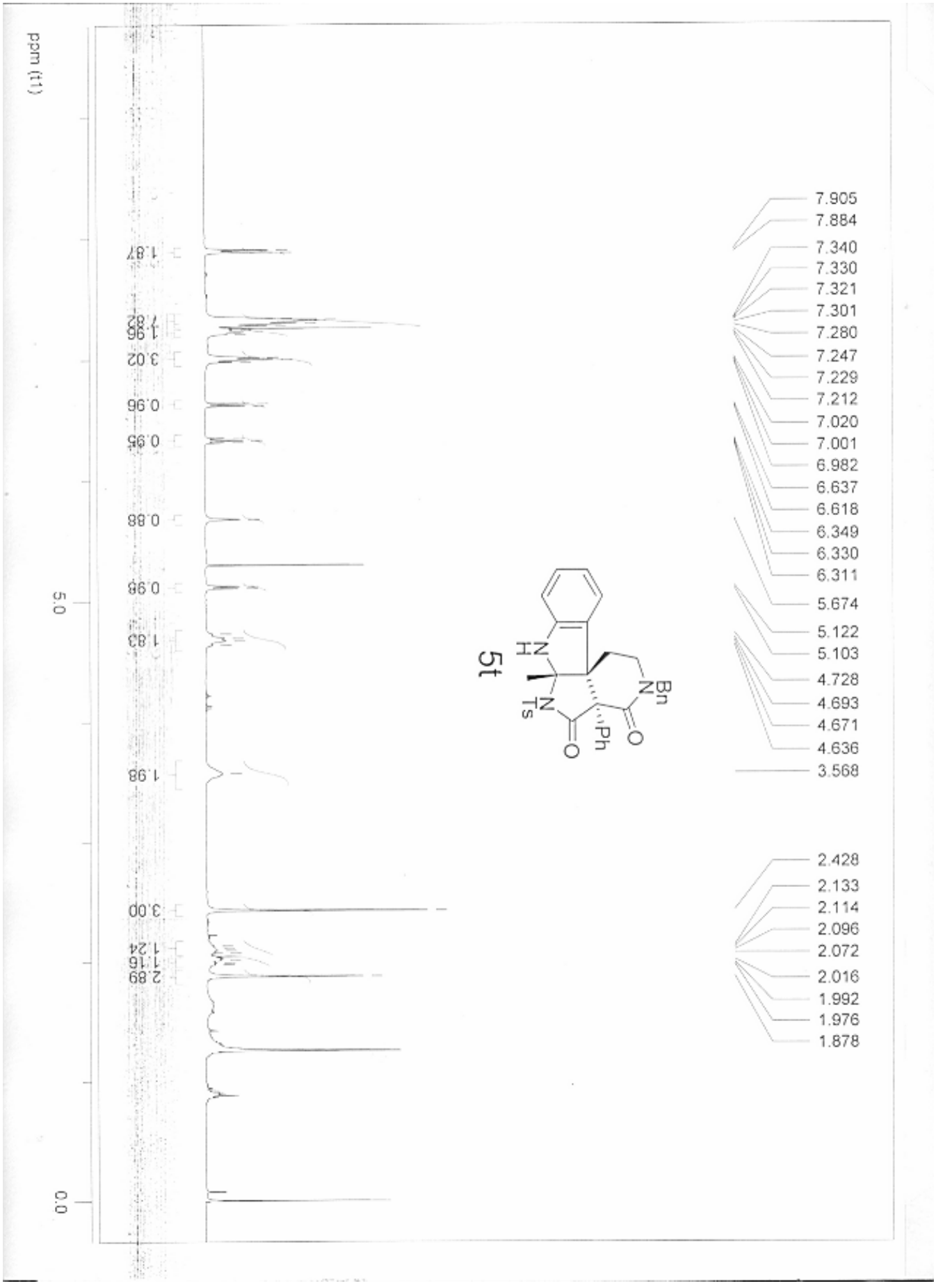
145.457
136.487
135.871
134.796
129.636
129.426
128.923
128.630
128.310
128.034
127.858
127.360
126.478
119.065
109.698
76.094
65.025
55.805
51.507
43.385
26.414
21.747
5s
N
H
H
N
Ts
Bn
N
O
O
Ph


ppm (t1)
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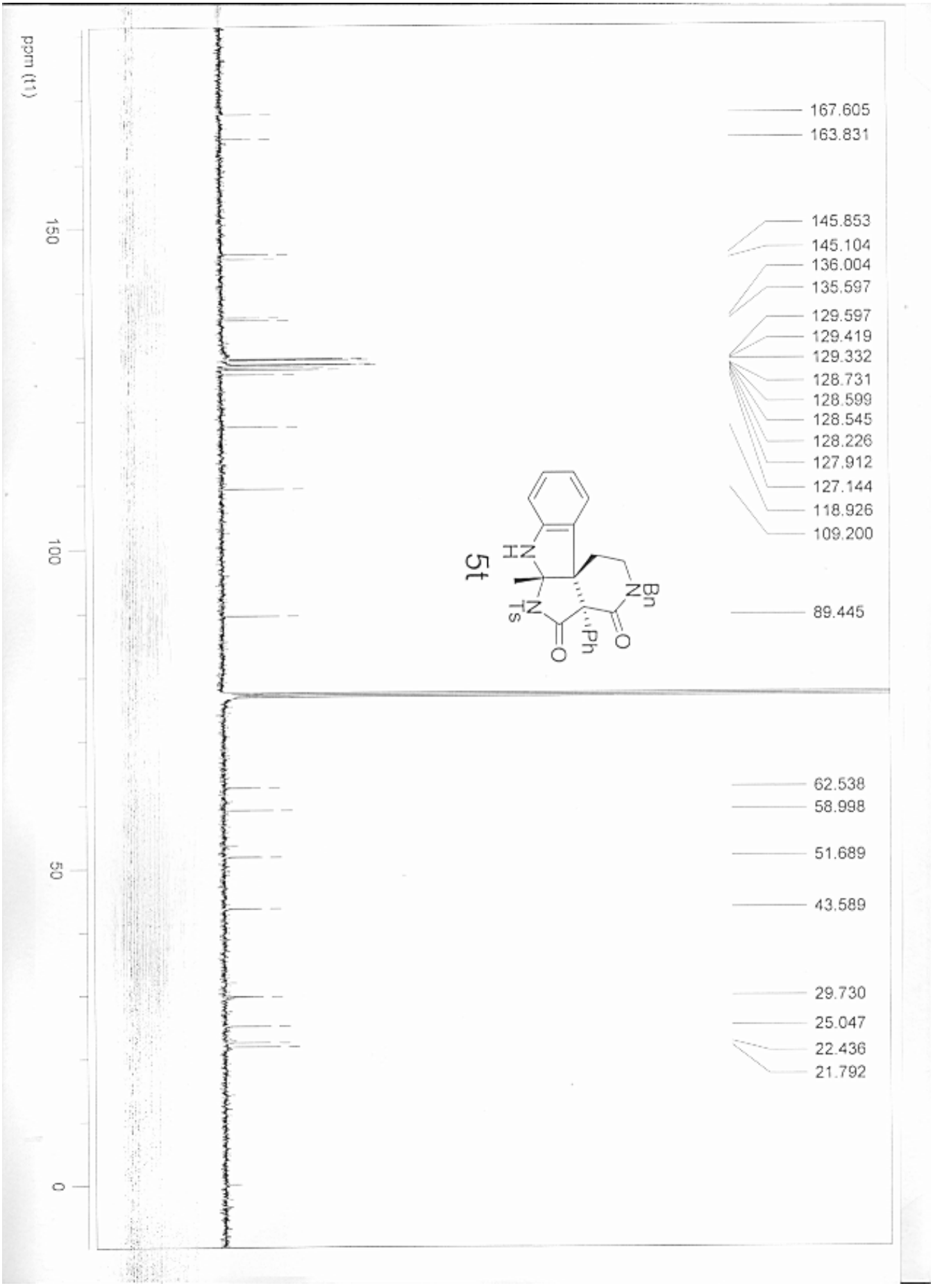
1.96
7.82
3.02
0.96
0.95
0.88
0.98
1.83
1.98
3.00
1.24
1.16
2.89
5t
Bn
N
O
Ph
O
N
Ts
N
H
7.905
7.884
7.340
7.330
7.321
7.301
7.280
7.247
7.229
7.212
7.020
7.001
6.982
6.637
6.618
6.349
6.330
6.311
5.674
5.122
5.103
4.728
4.693
4.671
4.636
3.568
2.428
2.133
2.114
2.096
2.072
2.016
1.992
1.976
1.878


ppm (t1)
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100
50
0
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163.831
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145.104
136.004
135.597
129.597
129.419
129.332
128.731
128.599
128.545
128.226
127.912
127.144
118.926
109.200
89.445
62.538
58.998
51.689
43.589
29.730
25.047
22.436
21.792
5t
N
H
N
Ts
Bn
N
O
Ph
O

ORTEP draing of **5d**

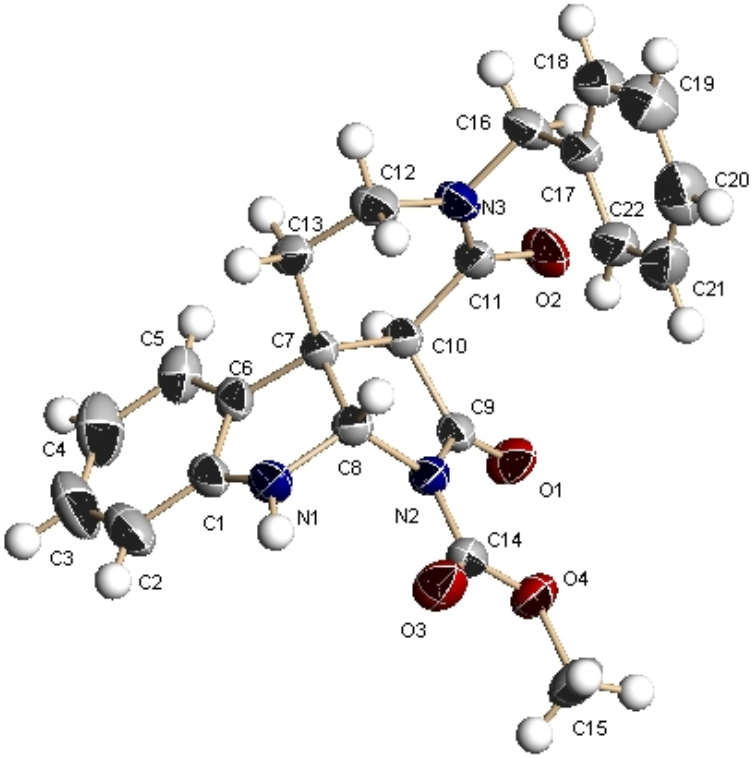

Table 1. Crystal data and structure refinement for **5d**.

Identification code	cd211522
Empirical formula	C22 H21 N3 O4
Formula weight	391.42
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 13.4875(9) A alpha = 90 deg. b = 10.5732(7) A beta = 103.886(2) deg. c = 13.9427(9) A gamma = 90 deg.
Volume	1930.2(2) A^3
Z, Calculated density	4, 1.347 Mg/m^3
Absorption coefficient	0.094 mm^-1
F(000)	824
Crystal size	0.165 x 0.123 x 0.112 mm
Theta range for data collection	1.89 to 26.00 deg.
Limiting indices	-16<=h<=16, -12<=k<=13, -10<=l<=17
Reflections collected / unique	11475 / 3797 [R(int) = 0.0223]
Completeness to theta = 26.00	99.9 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.86561
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3797 / 0 / 267
Goodness-of-fit on F^2	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0411, wR2 = 0.1015
R indices (all data)	R1 = 0.0520, wR2 = 0.1095
Largest diff. peak and hole	0.211 and -0.179 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd211522. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
N(1)	5440(1)	8243(1)	-1280(1)	46(1)
N(2)	6629(1)	6756(1)	-253(1)	37(1)
N(3)	7242(1)	8800(1)	2143(1)	42(1)
O(1)	8345(1)	6217(1)	261(1)	51(1)
O(2)	8638(1)	7608(1)	2187(1)	58(1)
O(3)	5235(1)	5580(1)	-951(1)	60(1)
O(4)	6788(1)	4681(1)	-538(1)	53(1)
C(1)	6046(1)	9016(2)	-1706(1)	44(1)
C(2)	5890(2)	9389(2)	-2683(1)	66(1)
C(3)	6574(2)	10235(3)	-2915(2)	82(1)
C(4)	7388(2)	10705(2)	-2207(2)	77(1)
C(5)	7538(1)	10332(2)	-1221(1)	54(1)
C(6)	6868(1)	9479(1)	-984(1)	40(1)
C(7)	6792(1)	8975(1)	15(1)	34(1)
C(8)	5987(1)	7907(1)	-306(1)	37(1)
C(9)	7660(1)	6957(1)	190(1)	35(1)
C(10)	7753(1)	8303(1)	593(1)	34(1)
C(11)	7910(1)	8215(1)	1719(1)	38(1)
C(12)	6357(1)	9539(2)	1625(1)	47(1)
C(13)	6473(1)	10012(1)	638(1)	40(1)
C(14)	6141(1)	5628(1)	-617(1)	42(1)
C(15)	6367(2)	3480(2)	-924(2)	75(1)
C(16)	7345(1)	8664(2)	3209(1)	50(1)
C(17)	6541(1)	7813(2)	3454(1)	43(1)
C(18)	6074(1)	8149(2)	4195(1)	54(1)
C(19)	5353(2)	7372(2)	4446(2)	69(1)
C(20)	5090(2)	6249(2)	3952(2)	71(1)
C(21)	5547(2)	5903(2)	3210(2)	65(1)
C(22)	6266(1)	6676(2)	2963(1)	53(1)

Table 3. Bond lengths [A] and angles [deg] for **5d**.

N(1)-C(1)	1.386(2)
N(1)-C(8)	1.4266(19)
N(1)-H(1)	0.851(19)
N(2)-C(9)	1.3963(18)
N(2)-C(14)	1.3974(18)
N(2)-C(8)	1.4844(17)
N(3)-C(11)	1.3411(19)
N(3)-C(12)	1.4642(19)
N(3)-C(16)	1.4658(19)
O(1)-C(9)	1.1967(16)
O(2)-C(11)	1.2222(17)
O(3)-C(14)	1.2008(18)
O(4)-C(14)	1.3153(18)
O(4)-C(15)	1.441(2)
C(1)-C(2)	1.384(2)
C(1)-C(6)	1.395(2)
C(2)-C(3)	1.378(3)
C(2)-H(2)	0.9300
C(3)-C(4)	1.381(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.397(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.373(2)
C(5)-H(5)	0.9300
C(6)-C(7)	1.519(2)
C(7)-C(13)	1.5231(19)
C(7)-C(10)	1.5276(18)
C(7)-C(8)	1.5547(19)
C(8)-H(8)	0.9800
C(9)-C(10)	1.5241(19)
C(10)-C(11)	1.537(2)
C(10)-H(10)	0.9800
C(12)-C(13)	1.508(2)
C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600

C(15)-H(15C)	0.9600
C(16)-C(17)	1.509(2)
C(16)-H(16A)	0.9700
C(16)-H(16B)	0.9700
C(17)-C(18)	1.380(2)
C(17)-C(22)	1.390(2)
C(18)-C(19)	1.380(3)
C(18)-H(18)	0.9300
C(19)-C(20)	1.376(3)
C(19)-H(19)	0.9300
C(20)-C(21)	1.375(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.374(3)
C(21)-H(21)	0.9300
C(22)-H(22)	0.9300

C(1)-N(1)-C(8)	109.34(12)
C(1)-N(1)-H(1)	122.7(13)
C(8)-N(1)-H(1)	119.6(13)
C(9)-N(2)-C(14)	128.48(12)
C(9)-N(2)-C(8)	113.84(11)
C(14)-N(2)-C(8)	117.66(11)
C(11)-N(3)-C(12)	125.65(12)
C(11)-N(3)-C(16)	119.45(13)
C(12)-N(3)-C(16)	114.82(12)
C(14)-O(4)-C(15)	116.48(13)
C(2)-C(1)-N(1)	128.54(16)
C(2)-C(1)-C(6)	121.11(17)
N(1)-C(1)-C(6)	110.25(13)
C(3)-C(2)-C(1)	117.62(19)
C(3)-C(2)-H(2)	121.2
C(1)-C(2)-H(2)	121.2
C(2)-C(3)-C(4)	121.87(19)
C(2)-C(3)-H(3)	119.1
C(4)-C(3)-H(3)	119.1
C(3)-C(4)-C(5)	120.3(2)
C(3)-C(4)-H(4)	119.9
C(5)-C(4)-H(4)	119.9
C(6)-C(5)-C(4)	118.29(19)
C(6)-C(5)-H(5)	120.9
C(4)-C(5)-H(5)	120.9
C(5)-C(6)-C(1)	120.81(15)
C(5)-C(6)-C(7)	130.20(15)
C(1)-C(6)-C(7)	108.77(13)

C(6)-C(7)-C(13)	111.14(12)
C(6)-C(7)-C(10)	114.24(11)
C(13)-C(7)-C(10)	110.75(11)
C(6)-C(7)-C(8)	100.77(11)
C(13)-C(7)-C(8)	114.48(11)
C(10)-C(7)-C(8)	105.07(11)
N(1)-C(8)-N(2)	114.41(12)
N(1)-C(8)-C(7)	105.03(12)
N(2)-C(8)-C(7)	102.88(10)
N(1)-C(8)-H(8)	111.3
N(2)-C(8)-H(8)	111.3
C(7)-C(8)-H(8)	111.3
O(1)-C(9)-N(2)	127.19(13)
O(1)-C(9)-C(10)	125.92(13)
N(2)-C(9)-C(10)	106.87(11)
C(9)-C(10)-C(7)	104.80(11)
C(9)-C(10)-C(11)	107.35(11)
C(7)-C(10)-C(11)	117.23(11)
C(9)-C(10)-H(10)	109.1
C(7)-C(10)-H(10)	109.1
C(11)-C(10)-H(10)	109.1
O(2)-C(11)-N(3)	123.07(14)
O(2)-C(11)-C(10)	118.20(13)
N(3)-C(11)-C(10)	118.73(12)
N(3)-C(12)-C(13)	112.13(12)
N(3)-C(12)-H(12A)	109.2
C(13)-C(12)-H(12A)	109.2
N(3)-C(12)-H(12B)	109.2
C(13)-C(12)-H(12B)	109.2
H(12A)-C(12)-H(12B)	107.9
C(12)-C(13)-C(7)	112.76(12)
C(12)-C(13)-H(13A)	109.0
C(7)-C(13)-H(13A)	109.0
C(12)-C(13)-H(13B)	109.0
C(7)-C(13)-H(13B)	109.0
H(13A)-C(13)-H(13B)	107.8
O(3)-C(14)-O(4)	126.39(14)
O(3)-C(14)-N(2)	121.57(14)
O(4)-C(14)-N(2)	112.04(12)
O(4)-C(15)-H(15A)	109.5
O(4)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
O(4)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5

H(15B)-C(15)-H(15C)	109.5
N(3)-C(16)-C(17)	112.89(13)
N(3)-C(16)-H(16A)	109.0
C(17)-C(16)-H(16A)	109.0
N(3)-C(16)-H(16B)	109.0
C(17)-C(16)-H(16B)	109.0
H(16A)-C(16)-H(16B)	107.8
C(18)-C(17)-C(22)	118.40(16)
C(18)-C(17)-C(16)	119.85(15)
C(22)-C(17)-C(16)	121.74(15)
C(17)-C(18)-C(19)	120.83(17)
C(17)-C(18)-H(18)	119.6
C(19)-C(18)-H(18)	119.6
C(20)-C(19)-C(18)	120.00(19)
C(20)-C(19)-H(19)	120.0
C(18)-C(19)-H(19)	120.0
C(21)-C(20)-C(19)	119.85(19)
C(21)-C(20)-H(20)	120.1
C(19)-C(20)-H(20)	120.1
C(22)-C(21)-C(20)	120.09(18)
C(22)-C(21)-H(21)	120.0
C(20)-C(21)-H(21)	120.0
C(21)-C(22)-C(17)	120.82(17)
C(21)-C(22)-H(22)	119.6
C(17)-C(22)-H(22)	119.6

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **5d**.
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
N(1)	42(1)	45(1)	42(1)	-3(1)	-11(1)	2(1)
N(2)	38(1)	33(1)	37(1)	-2(1)	2(1)	3(1)
N(3)	42(1)	49(1)	31(1)	-3(1)	2(1)	3(1)
O(1)	43(1)	44(1)	62(1)	-8(1)	4(1)	13(1)
O(2)	54(1)	67(1)	41(1)	1(1)	-9(1)	23(1)
O(3)	49(1)	51(1)	71(1)	-8(1)	-6(1)	-6(1)
O(4)	59(1)	33(1)	59(1)	-6(1)	1(1)	3(1)
C(1)	47(1)	47(1)	36(1)	0(1)	2(1)	15(1)
C(2)	73(1)	83(1)	38(1)	4(1)	4(1)	23(1)
C(3)	93(2)	109(2)	48(1)	30(1)	24(1)	31(2)
C(4)	78(1)	81(1)	80(2)	35(1)	38(1)	15(1)
C(5)	50(1)	52(1)	62(1)	14(1)	15(1)	7(1)
C(6)	41(1)	39(1)	38(1)	5(1)	7(1)	12(1)
C(7)	32(1)	33(1)	32(1)	0(1)	1(1)	4(1)
C(8)	33(1)	37(1)	37(1)	-2(1)	3(1)	5(1)
C(9)	36(1)	37(1)	30(1)	1(1)	5(1)	4(1)
C(10)	30(1)	35(1)	34(1)	0(1)	3(1)	3(1)
C(11)	37(1)	38(1)	34(1)	-3(1)	-3(1)	1(1)
C(12)	43(1)	54(1)	41(1)	-8(1)	5(1)	12(1)
C(13)	37(1)	37(1)	41(1)	-4(1)	-1(1)	9(1)
C(14)	49(1)	38(1)	35(1)	-1(1)	3(1)	-1(1)
C(15)	87(1)	36(1)	87(2)	-13(1)	-9(1)	-1(1)
C(16)	53(1)	60(1)	31(1)	-5(1)	3(1)	-4(1)
C(17)	43(1)	44(1)	35(1)	0(1)	-1(1)	5(1)
C(18)	58(1)	52(1)	51(1)	-5(1)	13(1)	1(1)
C(19)	62(1)	81(1)	69(1)	-1(1)	24(1)	-3(1)
C(20)	58(1)	75(1)	76(1)	13(1)	7(1)	-16(1)
C(21)	70(1)	48(1)	65(1)	1(1)	-10(1)	-7(1)
C(22)	58(1)	50(1)	46(1)	-5(1)	2(1)	5(1)

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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd211522.

	x	y	z	U(eq)
H(2)	5343	9080	-3165	79
H(3)	6485	10496	-3567	99
H(4)	7839	11272	-2387	92
H(5)	8078	10654	-738	65
H(8)	5531	7848	144	44
H(10)	8353	8705	443	40
H(12A)	6269	10256	2032	56
H(12B)	5749	9019	1527	56
H(13A)	6981	10680	744	48
H(13B)	5829	10371	278	48
H(15A)	5719	3356	-767	113
H(15B)	6825	2813	-634	113
H(15C)	6277	3470	-1628	113
H(16A)	7298	9493	3493	60
H(16B)	8015	8323	3509	60
H(18)	6247	8909	4530	64
H(19)	5046	7608	4948	83
H(20)	4605	5725	4120	86
H(21)	5369	5145	2875	78
H(22)	6573	6434	2461	64
H(1)	5006(14)	7729(18)	-1614(14)	59(5)

Table 6. Torsion angles [deg] for **5d**.

C(8)-N(1)-C(1)-C(2)	168.47(16)
C(8)-N(1)-C(1)-C(6)	-15.19(17)
N(1)-C(1)-C(2)-C(3)	175.89(17)
C(6)-C(1)-C(2)-C(3)	-0.1(3)
C(1)-C(2)-C(3)-C(4)	-0.2(3)
C(2)-C(3)-C(4)-C(5)	-0.2(3)
C(3)-C(4)-C(5)-C(6)	1.0(3)
C(4)-C(5)-C(6)-C(1)	-1.3(2)
C(4)-C(5)-C(6)-C(7)	-175.25(16)
C(2)-C(1)-C(6)-C(5)	0.9(2)
N(1)-C(1)-C(6)-C(5)	-175.78(13)
C(2)-C(1)-C(6)-C(7)	176.00(14)
N(1)-C(1)-C(6)-C(7)	-0.66(17)
C(5)-C(6)-C(7)-C(13)	67.12(19)
C(1)-C(6)-C(7)-C(13)	-107.39(14)
C(5)-C(6)-C(7)-C(10)	-59.1(2)
C(1)-C(6)-C(7)-C(10)	126.40(13)
C(5)-C(6)-C(7)-C(8)	-171.16(15)
C(1)-C(6)-C(7)-C(8)	14.33(14)
C(1)-N(1)-C(8)-N(2)	-88.10(15)
C(1)-N(1)-C(8)-C(7)	23.94(15)
C(9)-N(2)-C(8)-N(1)	122.64(13)
C(14)-N(2)-C(8)-N(1)	-58.94(17)
C(9)-N(2)-C(8)-C(7)	9.31(15)
C(14)-N(2)-C(8)-C(7)	-172.26(12)
C(6)-C(7)-C(8)-N(1)	-22.54(13)
C(13)-C(7)-C(8)-N(1)	96.80(14)
C(10)-C(7)-C(8)-N(1)	-141.49(12)
C(6)-C(7)-C(8)-N(2)	97.49(12)
C(13)-C(7)-C(8)-N(2)	-143.18(12)
C(10)-C(7)-C(8)-N(2)	-21.46(13)
C(14)-N(2)-C(9)-O(1)	7.6(2)
C(8)-N(2)-C(9)-O(1)	-174.21(14)
C(14)-N(2)-C(9)-C(10)	-171.32(13)
C(8)-N(2)-C(9)-C(10)	6.89(15)
O(1)-C(9)-C(10)-C(7)	160.59(14)
N(2)-C(9)-C(10)-C(7)	-20.49(14)
O(1)-C(9)-C(10)-C(11)	-74.07(18)
N(2)-C(9)-C(10)-C(11)	104.85(12)
C(6)-C(7)-C(10)-C(9)	-83.69(14)

C(13)-C(7)-C(10)-C(9)	149.90(12)
C(8)-C(7)-C(10)-C(9)	25.79(14)
C(6)-C(7)-C(10)-C(11)	157.44(12)
C(13)-C(7)-C(10)-C(11)	31.03(17)
C(8)-C(7)-C(10)-C(11)	-93.08(14)
C(12)-N(3)-C(11)-O(2)	179.93(15)
C(16)-N(3)-C(11)-O(2)	-3.3(2)
C(12)-N(3)-C(11)-C(10)	-0.3(2)
C(16)-N(3)-C(11)-C(10)	176.48(13)
C(9)-C(10)-C(11)-O(2)	57.68(17)
C(7)-C(10)-C(11)-O(2)	175.17(13)
C(9)-C(10)-C(11)-N(3)	-122.09(14)
C(7)-C(10)-C(11)-N(3)	-4.59(19)
C(11)-N(3)-C(12)-C(13)	-22.2(2)
C(16)-N(3)-C(12)-C(13)	160.92(13)
N(3)-C(12)-C(13)-C(7)	49.28(17)
C(6)-C(7)-C(13)-C(12)	178.24(12)
C(10)-C(7)-C(13)-C(12)	-53.65(16)
C(8)-C(7)-C(13)-C(12)	64.91(16)
C(15)-O(4)-C(14)-O(3)	2.3(3)
C(15)-O(4)-C(14)-N(2)	-177.97(15)
C(9)-N(2)-C(14)-O(3)	177.27(15)
C(8)-N(2)-C(14)-O(3)	-0.9(2)
C(9)-N(2)-C(14)-O(4)	-2.5(2)
C(8)-N(2)-C(14)-O(4)	179.39(12)
C(11)-N(3)-C(16)-C(17)	-106.52(16)
C(12)-N(3)-C(16)-C(17)	70.61(18)
N(3)-C(16)-C(17)-C(18)	-137.74(16)
N(3)-C(16)-C(17)-C(22)	43.5(2)
C(22)-C(17)-C(18)-C(19)	0.2(3)
C(16)-C(17)-C(18)-C(19)	-178.59(17)
C(17)-C(18)-C(19)-C(20)	-0.2(3)
C(18)-C(19)-C(20)-C(21)	0.0(3)
C(19)-C(20)-C(21)-C(22)	0.2(3)
C(20)-C(21)-C(22)-C(17)	-0.1(3)
C(18)-C(17)-C(22)-C(21)	0.0(2)
C(16)-C(17)-C(22)-C(21)	178.75(16)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd211522 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(3)	0.851(19)	2.445(19)	2.8772(19)	112.2(15)
N(1)-H(1)...O(2)#1	0.851(19)	2.20(2)	2.9655(17)	149.4(17)

Symmetry transformations used to generate equivalent atoms:
#1 x-1/2,-y+3/2,z-1/2

OTEP Drawing of **5s**

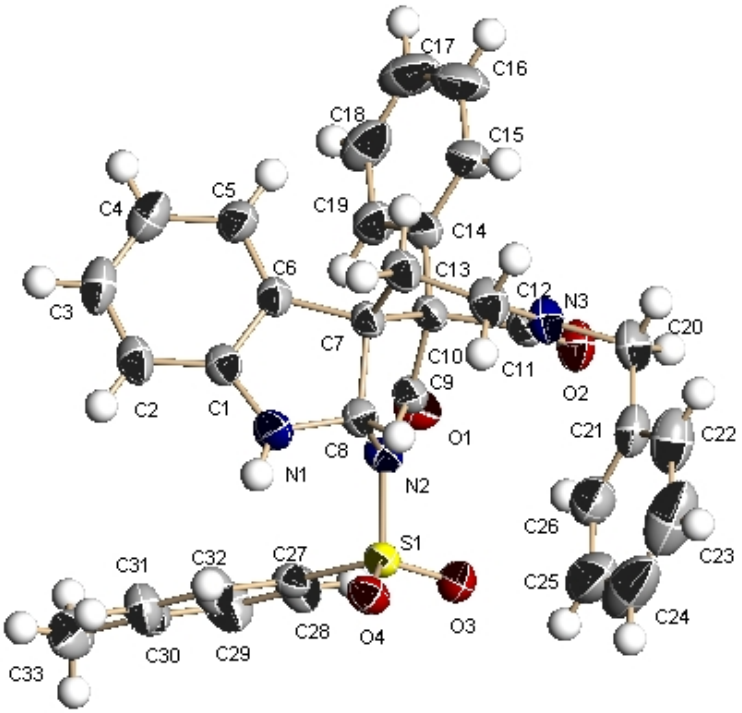

Table 1. Crystal data and structure refinement for **5s**.

Identification code	cd212115
Empirical formula	C36 H35 N3 O5 S
Formula weight	621.73
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 10.4770(6) A alpha = 90 deg.
	b = 11.2649(7) A beta = 100.4940(10) deg.
	c = 27.4274(16) A gamma = 90 deg.
Volume	3182.9(3) A^3
Z, Calculated density	4, 1.297 Mg/m^3
Absorption coefficient	0.149 mm^-1
F(000)	1312
Crystal size	0.321 x 0.213 x 0.146 mm
Theta range for data collection	1.96 to 26.00 deg.
Limiting indices	-12<=h<=9, -13<=k<=13, -31<=l<=33
Reflections collected / unique	18279 / 6245 [R(int) = 0.0359]
Completeness to theta = 26.00	100.0 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.42722
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6245 / 0 / 413
Goodness-of-fit on F^2	1.032
Final R indices [I>2sigma(I)]	R1 = 0.0522, wR2 = 0.1267
R indices (all data)	R1 = 0.0702, wR2 = 0.1366
Largest diff. peak and hole	0.331 and -0.319 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd212115. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S(1)	5634(1)	6148(1)	908(1)	35(1)
N(1)	2852(2)	5646(2)	156(1)	48(1)
N(2)	4084(2)	6257(2)	960(1)	37(1)
N(3)	2187(2)	4532(2)	1706(1)	45(1)
O(1)	4229(1)	7866(1)	1479(1)	46(1)
O(2)	3336(2)	5980(2)	2153(1)	53(1)
O(3)	6403(1)	6220(1)	1388(1)	49(1)
O(4)	5664(1)	5090(1)	621(1)	44(1)
O(5)	1025(3)	7637(3)	8309(1)	122(1)
C(1)	1950(2)	6523(2)	17(1)	40(1)
C(2)	1633(2)	7086(2)	-437(1)	50(1)
C(3)	634(3)	7893(2)	-501(1)	60(1)
C(4)	-58(3)	8117(2)	-129(1)	62(1)
C(5)	254(2)	7547(2)	327(1)	50(1)
C(6)	1287(2)	6773(2)	402(1)	38(1)
C(7)	1806(2)	5954(2)	838(1)	35(1)
C(8)	3057(2)	5471(2)	676(1)	36(1)
C(9)	3630(2)	7034(2)	1286(1)	36(1)
C(10)	2295(2)	6562(2)	1345(1)	35(1)
C(11)	2646(2)	5648(2)	1773(1)	39(1)
C(12)	1390(2)	4045(2)	1257(1)	51(1)
C(13)	833(2)	4979(2)	885(1)	45(1)
C(14)	1384(2)	7488(2)	1495(1)	40(1)
C(15)	379(2)	7125(3)	1727(1)	55(1)
C(16)	-514(3)	7932(4)	1837(1)	77(1)
C(17)	-410(3)	9102(4)	1731(1)	81(1)
C(18)	596(3)	9487(3)	1515(1)	68(1)
C(19)	1485(2)	8677(2)	1397(1)	52(1)
C(20)	2669(3)	3642(2)	2086(1)	55(1)
C(21)	3624(3)	2815(2)	1913(1)	52(1)
C(22)	3475(3)	1604(3)	1938(1)	71(1)
C(23)	4356(5)	853(3)	1779(1)	97(1)
C(24)	5374(5)	1313(5)	1593(1)	105(2)

C(25)	5534(3)	2524(4)	1567(1)	86(1)
C(26)	4671(3)	3258(3)	1727(1)	64(1)
C(27)	5977(2)	7366(2)	556(1)	37(1)
C(28)	6562(2)	8365(2)	789(1)	50(1)
C(29)	6840(2)	9303(2)	506(1)	56(1)
C(30)	6544(2)	9274(2)	-5(1)	47(1)
C(31)	5964(2)	8261(2)	-228(1)	53(1)
C(32)	5679(2)	7307(2)	46(1)	48(1)
C(33)	6830(3)	10305(2)	-316(1)	65(1)
C(34)	1386(4)	8078(5)	7531(1)	125(2)
C(35)	1222(3)	7244(4)	7920(1)	90(1)
C(36)	1347(7)	5980(5)	7841(3)	202(3)

Table 3. Bond lengths [A] and angles [deg] for **5s**.

S(1)-O(3)	1.4152(15)
S(1)-O(4)	1.4319(15)
S(1)-N(2)	1.6608(17)
S(1)-C(27)	1.752(2)
N(1)-C(1)	1.373(3)
N(1)-C(8)	1.417(3)
N(1)-H(1)	0.81(3)
N(2)-C(9)	1.395(3)
N(2)-C(8)	1.497(3)
N(3)-C(11)	1.347(3)
N(3)-C(12)	1.462(3)
N(3)-C(20)	1.469(3)
O(1)-C(9)	1.197(2)
O(2)-C(11)	1.216(2)
O(5)-C(35)	1.208(4)
C(1)-C(2)	1.381(3)
C(1)-C(6)	1.394(3)
C(2)-C(3)	1.374(4)
C(2)-H(2)	0.9300
C(3)-C(4)	1.378(4)
C(3)-H(3)	0.9300
C(4)-C(5)	1.390(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.376(3)
C(5)-H(5)	0.9300
C(6)-C(7)	1.530(3)
C(7)-C(13)	1.520(3)
C(7)-C(10)	1.552(3)
C(7)-C(8)	1.557(3)
C(8)-H(8)	0.9800
C(9)-C(10)	1.533(3)
C(10)-C(14)	1.519(3)
C(10)-C(11)	1.553(3)
C(12)-C(13)	1.508(3)
C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-C(19)	1.375(3)
C(14)-C(15)	1.388(3)

C(15)-C(16)	1.377(4)
C(15)-H(15)	0.9300
C(16)-C(17)	1.357(5)
C(16)-H(16)	0.9300
C(17)-C(18)	1.370(5)
C(17)-H(17)	0.9300
C(18)-C(19)	1.385(4)
C(18)-H(18)	0.9300
C(19)-H(19)	0.9300
C(20)-C(21)	1.506(4)
C(20)-H(20A)	0.9700
C(20)-H(20B)	0.9700
C(21)-C(22)	1.376(4)
C(21)-C(26)	1.385(4)
C(22)-C(23)	1.380(5)
C(22)-H(22)	0.9300
C(23)-C(24)	1.366(6)
C(23)-H(23)	0.9300
C(24)-C(25)	1.377(6)
C(24)-H(24)	0.9300
C(25)-C(26)	1.356(4)
C(25)-H(25)	0.9300
C(26)-H(26)	0.9300
C(27)-C(32)	1.379(3)
C(27)-C(28)	1.380(3)
C(28)-C(29)	1.373(3)
C(28)-H(28)	0.9300
C(29)-C(30)	1.379(3)
C(29)-H(29)	0.9300
C(30)-C(31)	1.381(3)
C(30)-C(33)	1.503(3)
C(31)-C(32)	1.374(3)
C(31)-H(31)	0.9300
C(32)-H(32)	0.9300
C(33)-H(33A)	0.9600
C(33)-H(33B)	0.9600
C(33)-H(33C)	0.9600
C(34)-C(35)	1.454(5)
C(34)-H(34A)	0.9600
C(34)-H(34B)	0.9600
C(34)-H(34C)	0.9600
C(35)-C(36)	1.450(7)
C(36)-H(36A)	0.9600
C(36)-H(36B)	0.9600

C(36)-H(36C)	0.9600
O(3)-S(1)-O(4)	119.62(10)
O(3)-S(1)-N(2)	108.42(9)
O(4)-S(1)-N(2)	103.18(9)
O(3)-S(1)-C(27)	109.21(10)
O(4)-S(1)-C(27)	108.75(9)
N(2)-S(1)-C(27)	106.88(9)
C(1)-N(1)-C(8)	110.50(19)
C(1)-N(1)-H(1)	127.9(18)
C(8)-N(1)-H(1)	120.6(18)
C(9)-N(2)-C(8)	114.10(16)
C(9)-N(2)-S(1)	123.61(14)
C(8)-N(2)-S(1)	122.21(13)
C(11)-N(3)-C(12)	126.86(18)
C(11)-N(3)-C(20)	118.21(19)
C(12)-N(3)-C(20)	114.28(19)
N(1)-C(1)-C(2)	128.4(2)
N(1)-C(1)-C(6)	110.38(19)
C(2)-C(1)-C(6)	121.2(2)
C(3)-C(2)-C(1)	118.1(2)
C(3)-C(2)-H(2)	120.9
C(1)-C(2)-H(2)	120.9
C(2)-C(3)-C(4)	121.2(2)
C(2)-C(3)-H(3)	119.4
C(4)-C(3)-H(3)	119.4
C(3)-C(4)-C(5)	120.8(2)
C(3)-C(4)-H(4)	119.6
C(5)-C(4)-H(4)	119.6
C(6)-C(5)-C(4)	118.5(2)
C(6)-C(5)-H(5)	120.8
C(4)-C(5)-H(5)	120.8
C(5)-C(6)-C(1)	120.1(2)
C(5)-C(6)-C(7)	131.1(2)
C(1)-C(6)-C(7)	108.37(18)
C(13)-C(7)-C(6)	110.71(16)
C(13)-C(7)-C(10)	111.05(16)
C(6)-C(7)-C(10)	116.57(17)
C(13)-C(7)-C(8)	112.62(17)
C(6)-C(7)-C(8)	100.69(16)
C(10)-C(7)-C(8)	104.65(15)
N(1)-C(8)-N(2)	113.87(17)
N(1)-C(8)-C(7)	104.90(17)
N(2)-C(8)-C(7)	101.95(15)

N(1)-C(8)-H(8)	111.8
N(2)-C(8)-H(8)	111.8
C(7)-C(8)-H(8)	111.8
O(1)-C(9)-N(2)	124.28(19)
O(1)-C(9)-C(10)	129.75(19)
N(2)-C(9)-C(10)	105.93(16)
C(14)-C(10)-C(9)	114.88(17)
C(14)-C(10)-C(7)	115.13(16)
C(9)-C(10)-C(7)	102.47(15)
C(14)-C(10)-C(11)	108.96(16)
C(9)-C(10)-C(11)	102.42(16)
C(7)-C(10)-C(11)	112.24(17)
O(2)-C(11)-N(3)	123.1(2)
O(2)-C(11)-C(10)	117.8(2)
N(3)-C(11)-C(10)	119.12(18)
N(3)-C(12)-C(13)	113.51(19)
N(3)-C(12)-H(12A)	108.9
C(13)-C(12)-H(12A)	108.9
N(3)-C(12)-H(12B)	108.9
C(13)-C(12)-H(12B)	108.9
H(12A)-C(12)-H(12B)	107.7
C(12)-C(13)-C(7)	112.27(18)
C(12)-C(13)-H(13A)	109.2
C(7)-C(13)-H(13A)	109.2
C(12)-C(13)-H(13B)	109.2
C(7)-C(13)-H(13B)	109.2
H(13A)-C(13)-H(13B)	107.9
C(19)-C(14)-C(15)	118.0(2)
C(19)-C(14)-C(10)	122.8(2)
C(15)-C(14)-C(10)	119.2(2)
C(16)-C(15)-C(14)	120.6(3)
C(16)-C(15)-H(15)	119.7
C(14)-C(15)-H(15)	119.7
C(17)-C(16)-C(15)	120.7(3)
C(17)-C(16)-H(16)	119.7
C(15)-C(16)-H(16)	119.7
C(16)-C(17)-C(18)	119.8(3)
C(16)-C(17)-H(17)	120.1
C(18)-C(17)-H(17)	120.1
C(17)-C(18)-C(19)	119.8(3)
C(17)-C(18)-H(18)	120.1
C(19)-C(18)-H(18)	120.1
C(14)-C(19)-C(18)	121.1(3)
C(14)-C(19)-H(19)	119.4

C(18)-C(19)-H(19)	119.4
N(3)-C(20)-C(21)	111.20(19)
N(3)-C(20)-H(20A)	109.4
C(21)-C(20)-H(20A)	109.4
N(3)-C(20)-H(20B)	109.4
C(21)-C(20)-H(20B)	109.4
H(20A)-C(20)-H(20B)	108.0
C(22)-C(21)-C(26)	118.7(3)
C(22)-C(21)-C(20)	120.6(3)
C(26)-C(21)-C(20)	120.6(2)
C(21)-C(22)-C(23)	120.2(3)
C(21)-C(22)-H(22)	119.9
C(23)-C(22)-H(22)	119.9
C(24)-C(23)-C(22)	119.9(4)
C(24)-C(23)-H(23)	120.1
C(22)-C(23)-H(23)	120.1
C(23)-C(24)-C(25)	120.3(4)
C(23)-C(24)-H(24)	119.8
C(25)-C(24)-H(24)	119.8
C(26)-C(25)-C(24)	119.6(4)
C(26)-C(25)-H(25)	120.2
C(24)-C(25)-H(25)	120.2
C(25)-C(26)-C(21)	121.2(3)
C(25)-C(26)-H(26)	119.4
C(21)-C(26)-H(26)	119.4
C(32)-C(27)-C(28)	120.5(2)
C(32)-C(27)-S(1)	119.37(16)
C(28)-C(27)-S(1)	120.16(17)
C(29)-C(28)-C(27)	119.2(2)
C(29)-C(28)-H(28)	120.4
C(27)-C(28)-H(28)	120.4
C(28)-C(29)-C(30)	121.6(2)
C(28)-C(29)-H(29)	119.2
C(30)-C(29)-H(29)	119.2
C(29)-C(30)-C(31)	117.9(2)
C(29)-C(30)-C(33)	121.8(2)
C(31)-C(30)-C(33)	120.3(2)
C(32)-C(31)-C(30)	121.8(2)
C(32)-C(31)-H(31)	119.1
C(30)-C(31)-H(31)	119.1
C(31)-C(32)-C(27)	119.0(2)
C(31)-C(32)-H(32)	120.5
C(27)-C(32)-H(32)	120.5
C(30)-C(33)-H(33A)	109.5

C(30)-C(33)-H(33B)	109.5
H(33A)-C(33)-H(33B)	109.5
C(30)-C(33)-H(33C)	109.5
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(35)-C(34)-H(34A)	109.5
C(35)-C(34)-H(34B)	109.5
H(34A)-C(34)-H(34B)	109.5
C(35)-C(34)-H(34C)	109.5
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
O(5)-C(35)-C(36)	121.8(5)
O(5)-C(35)-C(34)	118.2(4)
C(36)-C(35)-C(34)	120.0(4)
C(35)-C(36)-H(36A)	109.5
C(35)-C(36)-H(36B)	109.5
H(36A)-C(36)-H(36B)	109.5
C(35)-C(36)-H(36C)	109.5
H(36A)-C(36)-H(36C)	109.5
H(36B)-C(36)-H(36C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **5s**.
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
S(1)	35(1)	39(1)	33(1)	-1(1)	8(1)	1(1)
N(1)	50(1)	65(1)	32(1)	-7(1)	10(1)	9(1)
N(2)	36(1)	39(1)	36(1)	-5(1)	11(1)	-4(1)
N(3)	54(1)	45(1)	37(1)	6(1)	8(1)	-6(1)
O(1)	47(1)	43(1)	50(1)	-13(1)	13(1)	-8(1)
O(2)	63(1)	57(1)	36(1)	1(1)	-1(1)	-5(1)
O(3)	43(1)	62(1)	39(1)	4(1)	2(1)	2(1)
O(4)	48(1)	40(1)	47(1)	-4(1)	18(1)	2(1)
O(5)	137(2)	167(3)	73(2)	1(2)	47(2)	-4(2)
C(1)	41(1)	44(1)	33(1)	-3(1)	3(1)	-8(1)
C(2)	56(1)	60(2)	33(1)	1(1)	4(1)	-13(1)
C(3)	73(2)	57(2)	41(1)	11(1)	-9(1)	-10(1)
C(4)	62(2)	60(2)	57(2)	6(1)	-8(1)	10(1)
C(5)	46(1)	57(2)	43(1)	0(1)	2(1)	5(1)
C(6)	38(1)	41(1)	34(1)	2(1)	5(1)	-5(1)
C(7)	36(1)	40(1)	29(1)	-1(1)	7(1)	-3(1)
C(8)	39(1)	36(1)	32(1)	-4(1)	5(1)	-3(1)
C(9)	40(1)	36(1)	32(1)	-1(1)	9(1)	0(1)
C(10)	37(1)	40(1)	31(1)	-1(1)	10(1)	-2(1)
C(11)	39(1)	47(1)	32(1)	1(1)	11(1)	-2(1)
C(12)	58(2)	50(1)	45(1)	4(1)	9(1)	-16(1)
C(13)	43(1)	51(1)	40(1)	0(1)	8(1)	-11(1)
C(14)	39(1)	49(1)	30(1)	-4(1)	5(1)	3(1)
C(15)	48(1)	69(2)	51(1)	0(1)	17(1)	4(1)
C(16)	52(2)	109(3)	75(2)	-11(2)	28(1)	11(2)
C(17)	62(2)	97(3)	82(2)	-24(2)	7(2)	31(2)
C(18)	72(2)	58(2)	70(2)	-5(1)	-2(2)	21(2)
C(19)	53(1)	51(2)	50(1)	0(1)	8(1)	8(1)
C(20)	71(2)	53(2)	43(1)	13(1)	12(1)	-5(1)
C(21)	65(2)	48(1)	38(1)	8(1)	-4(1)	-4(1)
C(22)	99(2)	50(2)	55(2)	4(1)	-6(2)	-10(2)
C(23)	143(4)	58(2)	75(2)	-12(2)	-23(2)	20(2)

C(24)	117(3)	120(4)	65(2)	-23(2)	-17(2)	59(3)
C(25)	71(2)	114(3)	68(2)	1(2)	-1(2)	22(2)
C(26)	61(2)	68(2)	61(2)	7(1)	5(1)	3(1)
C(27)	33(1)	40(1)	38(1)	-1(1)	9(1)	-2(1)
C(28)	54(1)	55(2)	41(1)	-6(1)	7(1)	-17(1)
C(29)	62(2)	48(1)	58(2)	-7(1)	10(1)	-23(1)
C(30)	40(1)	46(1)	57(1)	7(1)	12(1)	-5(1)
C(31)	64(2)	56(2)	40(1)	4(1)	9(1)	-9(1)
C(32)	59(1)	43(1)	40(1)	-2(1)	8(1)	-12(1)
C(33)	61(2)	56(2)	78(2)	20(1)	15(1)	-11(1)
C(34)	103(3)	214(5)	59(2)	27(3)	16(2)	47(3)
C(35)	57(2)	151(4)	63(2)	-12(2)	15(2)	-10(2)
C(36)	210(7)	128(5)	255(8)	-49(5)	8(6)	-50(5)

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

	x	y	z	U(eq)
H(2)	2084	6923	-691	60
H(3)	421	8295	-801	71
H(4)	-742	8656	-184	74
H(5)	-225	7686	575	59
H(8)	3208	4633	764	43
H(12A)	683	3593	1350	61
H(12B)	1914	3503	1102	61
H(13A)	74	5329	985	53
H(13B)	556	4608	564	53
H(15)	307	6329	1809	66
H(16)	-1195	7674	1985	92
H(17)	-1020	9640	1805	97
H(18)	681	10289	1449	82
H(19)	2162	8942	1247	62
H(20A)	1944	3187	2162	66
H(20B)	3085	4039	2387	66
H(22)	2778	1291	2063	85
H(23)	4256	35	1798	117
H(24)	5963	807	1482	126
H(25)	6230	2835	1442	103
H(26)	4785	4075	1711	77
H(28)	6766	8402	1133	60
H(29)	7238	9974	662	67
H(31)	5762	8223	-572	64
H(32)	5291	6632	-111	57
H(33A)	6513	11024	-193	97
H(33B)	6409	10184	-654	97
H(33C)	7750	10366	-301	97
H(34A)	836	8755	7545	188
H(34B)	1156	7700	7214	188
H(34C)	2275	8331	7579	188
H(36A)	1334	5564	8145	303
H(36B)	2152	5825	7732	303
H(36C)	638	5714	7592	303

H(1)	3330(20)	5340(20)	-9(9)	49(7)

Table 6. Torsion angles [deg] for cd212115.

O(3)-S(1)-N(2)-C(9)	40.06(19)
O(4)-S(1)-N(2)-C(9)	167.86(16)
C(27)-S(1)-N(2)-C(9)	-77.55(18)
O(3)-S(1)-N(2)-C(8)	-136.52(16)
O(4)-S(1)-N(2)-C(8)	-8.72(17)
C(27)-S(1)-N(2)-C(8)	105.87(16)
C(8)-N(1)-C(1)-C(2)	168.7(2)
C(8)-N(1)-C(1)-C(6)	-14.3(3)
N(1)-C(1)-C(2)-C(3)	176.0(2)
C(6)-C(1)-C(2)-C(3)	-0.7(3)
C(1)-C(2)-C(3)-C(4)	-1.6(4)
C(2)-C(3)-C(4)-C(5)	1.2(4)
C(3)-C(4)-C(5)-C(6)	1.4(4)
C(4)-C(5)-C(6)-C(1)	-3.6(3)
C(4)-C(5)-C(6)-C(7)	-175.0(2)
N(1)-C(1)-C(6)-C(5)	-173.9(2)
C(2)-C(1)-C(6)-C(5)	3.3(3)
N(1)-C(1)-C(6)-C(7)	-0.8(2)
C(2)-C(1)-C(6)-C(7)	176.47(19)
C(5)-C(6)-C(7)-C(13)	66.3(3)
C(1)-C(6)-C(7)-C(13)	-105.8(2)
C(5)-C(6)-C(7)-C(10)	-61.9(3)
C(1)-C(6)-C(7)-C(10)	125.99(19)
C(5)-C(6)-C(7)-C(8)	-174.4(2)
C(1)-C(6)-C(7)-C(8)	13.5(2)
C(1)-N(1)-C(8)-N(2)	-88.0(2)
C(1)-N(1)-C(8)-C(7)	22.6(2)
C(9)-N(2)-C(8)-N(1)	117.5(2)
S(1)-N(2)-C(8)-N(1)	-65.6(2)
C(9)-N(2)-C(8)-C(7)	5.1(2)
S(1)-N(2)-C(8)-C(7)	-178.02(13)
C(13)-C(7)-C(8)-N(1)	96.8(2)
C(6)-C(7)-C(8)-N(1)	-21.1(2)
C(10)-C(7)-C(8)-N(1)	-142.44(17)

C(13)-C(7)-C(8)-N(2)	-144.21(16)
C(6)-C(7)-C(8)-N(2)	97.85(17)
C(10)-C(7)-C(8)-N(2)	-23.48(19)
C(8)-N(2)-C(9)-O(1)	-166.3(2)
S(1)-N(2)-C(9)-O(1)	16.9(3)
C(8)-N(2)-C(9)-C(10)	15.8(2)
S(1)-N(2)-C(9)-C(10)	-161.04(14)
O(1)-C(9)-C(10)-C(14)	26.9(3)
N(2)-C(9)-C(10)-C(14)	-155.28(16)
O(1)-C(9)-C(10)-C(7)	152.5(2)
N(2)-C(9)-C(10)-C(7)	-29.7(2)
O(1)-C(9)-C(10)-C(11)	-91.0(3)
N(2)-C(9)-C(10)-C(11)	86.76(18)
C(13)-C(7)-C(10)-C(14)	-80.2(2)
C(6)-C(7)-C(10)-C(14)	47.8(2)
C(8)-C(7)-C(10)-C(14)	158.03(17)
C(13)-C(7)-C(10)-C(9)	154.37(17)
C(6)-C(7)-C(10)-C(9)	-77.6(2)
C(8)-C(7)-C(10)-C(9)	32.6(2)
C(13)-C(7)-C(10)-C(11)	45.2(2)
C(6)-C(7)-C(10)-C(11)	173.24(17)
C(8)-C(7)-C(10)-C(11)	-76.56(19)
C(12)-N(3)-C(11)-O(2)	-177.9(2)
C(20)-N(3)-C(11)-O(2)	-7.7(3)
C(12)-N(3)-C(11)-C(10)	1.7(3)
C(20)-N(3)-C(11)-C(10)	171.89(19)
C(14)-C(10)-C(11)-O(2)	-69.7(2)
C(9)-C(10)-C(11)-O(2)	52.4(2)
C(7)-C(10)-C(11)-O(2)	161.62(19)
C(14)-C(10)-C(11)-N(3)	110.8(2)
C(9)-C(10)-C(11)-N(3)	-127.1(2)
C(7)-C(10)-C(11)-N(3)	-18.0(3)
C(11)-N(3)-C(12)-C(13)	-13.2(3)
C(20)-N(3)-C(12)-C(13)	176.3(2)
N(3)-C(12)-C(13)-C(7)	41.1(3)
C(6)-C(7)-C(13)-C(12)	170.95(19)
C(10)-C(7)-C(13)-C(12)	-57.9(2)
C(8)-C(7)-C(13)-C(12)	59.1(2)
C(9)-C(10)-C(14)-C(19)	25.0(3)
C(7)-C(10)-C(14)-C(19)	-93.7(2)
C(11)-C(10)-C(14)-C(19)	139.2(2)
C(9)-C(10)-C(14)-C(15)	-157.12(19)
C(7)-C(10)-C(14)-C(15)	84.2(2)
C(11)-C(10)-C(14)-C(15)	-42.9(3)

C(19)-C(14)-C(15)-C(16)	2.3(4)
C(10)-C(14)-C(15)-C(16)	-175.6(2)
C(14)-C(15)-C(16)-C(17)	-1.5(4)
C(15)-C(16)-C(17)-C(18)	-0.4(5)
C(16)-C(17)-C(18)-C(19)	1.4(5)
C(15)-C(14)-C(19)-C(18)	-1.3(3)
C(10)-C(14)-C(19)-C(18)	176.5(2)
C(17)-C(18)-C(19)-C(14)	-0.5(4)
C(11)-N(3)-C(20)-C(21)	-104.7(2)
C(12)-N(3)-C(20)-C(21)	66.7(3)
N(3)-C(20)-C(21)-C(22)	-128.6(2)
N(3)-C(20)-C(21)-C(26)	51.5(3)
C(26)-C(21)-C(22)-C(23)	-0.1(4)
C(20)-C(21)-C(22)-C(23)	180.0(3)
C(21)-C(22)-C(23)-C(24)	-0.4(5)
C(22)-C(23)-C(24)-C(25)	0.6(5)
C(23)-C(24)-C(25)-C(26)	-0.2(5)
C(24)-C(25)-C(26)-C(21)	-0.4(5)
C(22)-C(21)-C(26)-C(25)	0.5(4)
C(20)-C(21)-C(26)-C(25)	-179.6(2)
O(3)-S(1)-C(27)-C(32)	159.53(18)
O(4)-S(1)-C(27)-C(32)	27.4(2)
N(2)-S(1)-C(27)-C(32)	-83.37(19)
O(3)-S(1)-C(27)-C(28)	-19.2(2)
O(4)-S(1)-C(27)-C(28)	-151.31(18)
N(2)-S(1)-C(27)-C(28)	97.92(19)
C(32)-C(27)-C(28)-C(29)	0.3(4)
S(1)-C(27)-C(28)-C(29)	178.98(19)
C(27)-C(28)-C(29)-C(30)	0.3(4)
C(28)-C(29)-C(30)-C(31)	-0.6(4)
C(28)-C(29)-C(30)-C(33)	179.1(2)
C(29)-C(30)-C(31)-C(32)	0.4(4)
C(33)-C(30)-C(31)-C(32)	-179.4(2)
C(30)-C(31)-C(32)-C(27)	0.2(4)
C(28)-C(27)-C(32)-C(31)	-0.5(3)
S(1)-C(27)-C(32)-C(31)	-179.24(19)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **5s** [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(4)#1	0.81(3)	2.20(3)	2.975(3)	163(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z