SUPPLEMENTARY MATERIAL

for the
article
entitled

Synthesis of Amido-Spiro [2.2] Pentanes via Simmons-Smith Cyclopropanation of Allenamides.

authored by

Ting Lu, Ryuji Hayashi, Richard P. Hsung*, Kyle A. Dekorver, Andrew G. Lohse,
Zhenlei Song and Yu Tang

Division of Pharmaceutical Sciences and Department of Chemistry
7111 Rennebohm Hall, 777 Highland Avenue
University of Wisconsin at Madison
Madison, WI 53705-2222

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GENERAL PROCEDURES

All reactions were performed in flame-dried glassware under a nitrogen or argon atmosphere. Solvents were distilled prior to use. Reagents were used as purchased (Aldrich, Fluka), except where noted. Chromatographic separations were performed using Bodman 60 Å SiO_2. ^1H and ^{13}C NMR spectra were obtained on Varian VI-400 and VI-500 spectrometers using $CDCl_3$ as solvent. Melting points were determined using a Laboratory Devices MEL-TEMP and are uncorrected/calibrated. Infrared spectra were collected on a Bruker Equinox 55/S FT–IR Spectrophotometer, and relative intensities are expressed qualitatively as s (strong), m (medium), and w (weak). TLC analysis was performed using Aldrich 254 nm polyester-backed plates (60 Å, 250 μm) and visualized using UV and a suitable chemical stain. Low-resolution mass spectra were obtained using an Agilent-1100-HPLC/MSD and can be either APCI or ESI, or were performed at University of Wisconsin Mass Spectrometry Laboratories. High-resolution mass spectral analyses were performed at University of Wisconsin Mass Spectrometry Laboratories. All spectral data obtained for new compounds are reported. X-Ray analyses were performed at the X-Ray facility in University of Minnesota.

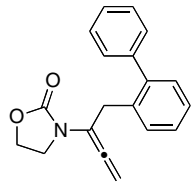
Simmons-Smith Cyclopropanation of Chiral Enamides. The enamides prepared and the following Simmons-Smith cyclopropanations shown in **Scheme 4** performed followed our previous reported procedures. [3]

Preparation of Allenamides. The α-unsubstituted allenamides were prepared with our group reported two-step protocol from the commercially available 2-oxazolidinone.[2] The α-substituted allenamides used in this work were prepared by α-alkylation of the corresponding unsubstituted allenamides following our group previous reported procedures.[3]

Simmons-Smith Cyclopropanation of Allenamides. To a solution of allenamide (0.20 mmol, 1.0 equiv) in anhyd $ClCH_2CH_2Cl$ (2 mL) was added carefully a solution of $ZnEt_2$ (1.0 mL, 1.0 *M* in hexanes, 1.0 mmol, 5.0 equiv) at 0 ˚C carefully under N_2. After stirring at 0 ˚C for 10 min, ICH_2Cl (150 μL, 2.0 mmol, 10.0 equiv) was added. After additional stirring at rt for the indicated time, or till the crude ^1H NMR indicated the completion of the reaction. The reaction was quenched with sat aq $NaHCO_3$ (5 mL), then extracted with CH_2Cl_2 (3 x 5 mL). The combined organic phases were washed with sat aq NaCl (2 x 5 mL), dried over Na_2SO_4, and concentrated under reduced pressure to afford the crude product determined by crude ^1H NMR. Purification of crude residue via silica gel flash column chromatography (Gradient eluent 10-50% of EtOAc/Hexanes) afforded the mono- and/or bis-cyclopropane products.

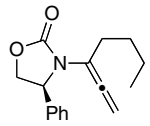
References
1. Song, Z.; Lu, T.; Hsung, R. P.; Al-Rashid, Z. F.; Ko, C.; Tang, Y. *Angew. Chem. Int. Ed.* **2007**, *46*, 4069.
2. Wei, L-L.; Mulder, J. A.; Xiong, H.; Zificsak, C. A.; Douglas, C. J.; Hsung, R. P. *Tetrahedron* **2001**, *57*, 459.
3. Xiong, H.; Hsung, R. P.; Wei, L-L.; Berry, C. R.; Mulder, J. A.; Stockwell, B. *Org. Lett.* **2000**, *2*, 2869.

Characterization.



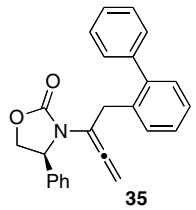
28

R_f = 0.45 [40% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.39 (t, *J* = 8.0 Hz, 2H), 3.86 (t, *J* = 3.5 Hz, 2H), 4.21 (t, *J* = 8.0 Hz, 2H), 4.96 (t, *J* = 3.0 Hz, 2H), 7.21-7.42 (m, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 34.1, 46.4, 62.0, 84.8, 85.0, 110.2, 127.0, 127.3, 127.6, 128.4, 128.4, 129.3, 129.3, 130.4, 135.3, 141.8, 142.6, 155.8, 204.3; IR (neat) cm^{-1} 3057w, 2361w, 2342w, 1749s, 1479m, 1451w, 1401m; mass spectrum (APCI): m/e (% relative intensity) 292 (100) $(M+H)^+$, 205 (100); HRMS (MALDI) m/e calcd for $C_{19}H_{17}NO_2Na$ 314.1152, found 314.1138.



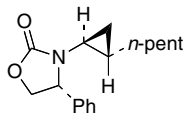
33

R_f = 0.38 [25% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.82 (t, *J* = 7.5 Hz, 3H), 1.18-1.37 (m, 4H), 2.40-2.44 (m, 2H), 4.14 (dd, *J* = 8.0, 8.5 Hz, 1H), 4.63 (dd, *J* = 8.5, 8.5 Hz, 1H), 4.82-4.95 (m, 2H), 5.00 (td, *J* = 3.0, 10.0 Hz, 1H), 7.23-7.42 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.1, 22.2, 29.1, 29.6, 61.5, 70.1, 84.6, 109.4, 127.3, 129.1, 129.2, 138.8, 156.3, 204.8; IR (neat) cm^{-1} 2958w, 2872w, 1756s, 1457m, 1394s; mass spectrum (ESI): m/e (% relative intensity) 258 (90) $(M+H)^+$; 232 (50); HRMS (ESI) m/e calcd for $C_{16}H_{20}NO_2$ 258.1489, found 258.1476.



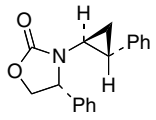
35

R_f = 0.36 [25% EtOAc/hexanes]; mp 124-126 ºC; $[\alpha]_D^{25}$ = + 11.6 º [*c* 0.30, CH_2Cl_2]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.63 (d, *J* = 15.0 Hz, 1H), 3.90 (td, *J* = 4.0, 15.0 Hz, 1H), 3.96 (t, *J* = 8.5 Hz, 1H), 4.49 (t, *J* = 8.5 Hz, 1H), 4.50 (m, 1H), 4.67 (t, *J* = 6.0, 8.8 Hz, 1H), 4.69 (m, 1H), 6.79-6.81(m, 4H), 7.11-7.38 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 34.4, 61.4, 70.2, 83.8, 107.8, 126.9, 127.0, 127.4, 127.6, 128.2, 128.9, 129.1, 129.3, 130.6, 131.0, 135.8, 138.2, 141.4, 143.0, 156.6, 204.8; IR (neat) cm^{-1} 3037w, 2900w, 1758s, 1536m, 1479s, 1442s; mass spectrum (ESI): m/e (% relative intensity) 390 (100) $(M+Na)^+$, 368 (90) $(M+H)^+$; HRMS (ESI) m/e calcd for $C_{25}H_{22}NO_2$ 368.1656, found 368.1671.



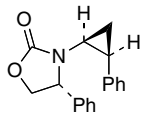
20

R_f = 0.47 [40% EtOAc/hexanes]; $[\alpha]_D^{23}$ = - 18.8 º [*c* 7.46, CH_2Cl_2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.62 (ddd, *J* = 1.2, 6.4, 11.6 Hz, 1H), 0.82 (t, *J* = 6.8 Hz, 3H), 0.86-0.91 (m, 2H), 0.99-1.04 (m, 2H), 1.06-1.20 (m, 6H), 2.10 (ddd, *J* = 3.2, 3.2, 6.4 Hz, 1H), 4.12 (dd, *J* = 5.2, 8.4 Hz, 1H), 4.54 (dd, *J* = 8.8, 8.8 Hz, 1H), 4.64 (dd, *J* = 5.2, 8.8 Hz, 1H), 7.29-7.32 (m, 2H), 7.35-7.43 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 14.2, 19.0, 22.5, 28.3, 30.8, 31.5, 32.3, 61.3, 69.6, 126.8, 128.8, 129.2, 138.9, 158.0; IR (neat) cm^{-1} 2940m, 2855w, 1752s, 1457m, 1408s, 1362w; mass spectrum (APCI): m/e (% relative intensity) 274 (100) $(M+H)^+$, 230 (10), 176 (27); HRMS (MALDI) m/e calcd for $C_{17}H_{24}NO_2$ 274.1807, found 274.1808.



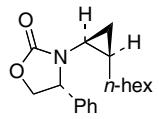
trans-**21**

R_f = 0.32 [40 % EtOAc/hexanes]; $[\alpha]_D^{23}$ = -25.5 º [*c* 0.40, CH_2Cl_2]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.23 (ddd, *J* = 6.5, 6.5, 13.5 Hz, 1H), 1.45 (ddd, *J* = 4.5, 6.5, 10.0 Hz, 1H), 2.17 (ddd, *J* = 3.5, 6.5, 10.0 Hz, 1H), 2.45 (ddd, *J* = 3.5, 3.5, 7.5 Hz, 1H), 4.21 (dd, *J* = 5.5, 9.0 Hz, 1H), 4.61 (dd, *J* = 8.5, 8.5 Hz, 1H), 4.78 (dd, *J* = 6.0, 9.0 Hz, 1H), 6.87 (d, *J* = 7.0 Hz, 2H), 7.10-7.19 (m, 3H), 7.32-7.42 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.7, 23.4, 34.0, 61.7, 69.9, 126.5, 127.0, 127.2, 128.5, 129.3, 129.5, 138.6, 139.8, 158.1; IR (neat) cm^{-1} 3030w, 2919w, 1750s, 1604w, 1480w, 1409s, 1360w; mass spectrum (APCI): m/e (% relative intensity) 280 (100) $(M+H)^+$, 236 (50), 176 (20), 164 (12); HRMS (MALDI) m/e calcd for $C_{18}H_{18}NO_2$ 280.1338, found 280.1344.



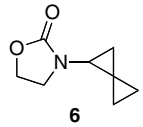
cis-**21**

R_f = 0.14 [20% EtOAc/hexanes]; mp 113-113.5 ºC; $[\alpha]_D^{23}$ = - 87.5 º [*c* 0.80, $CHCl_3$]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.43 (ddd, *J* = 7.5, 7.5, 9.0 Hz, 1H), 1.92 (ddd, *J* = 4.5, 7.5, 7.5 Hz, 1H), 2.11 (ddd, *J* = 7.5, 7.5, 9.0 Hz, 1H), 2.44 (ddd, *J* = 4.5, 7.5, 7.5 Hz, 1H), 3.61 (dd, *J* = 4.5, 9.0 Hz, 1H), 3.95 (dd, *J* = 4.5, 9.0 Hz, 1H), 4.05 (dd, *J* = 9.0, 9.0 Hz, 1H), 7.10-7.40 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 11.7, 22.7, 31.5, 60.0, 69.6, 126.9, 127.2, 127.8, 128.6, 129.2, 129.5, 136.9, 138.5, 158.8; IR (film) cm^{-1} 1740s, 1407m, 1126s, 1051m, 1031m, 1011m; mass spectrum (ESI): m/e (% relative intensity) 302 (100) $(M+Na)^+$, 280 (40) $(M+H)^+$; HRMS (ESI) m/e calcd for $C_{18}H_{17}NO_2Na$ 302.1157, found 302.1163.



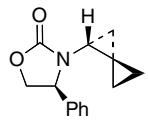
23

R_f = 0.16 [20% EtOAc/hexanes]; $[\alpha]_D^{23}$ = + 32.0 º [*c* 0.59, $CHCl_3$]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.54-0.58 (m, 1H), 0.67-0.73 (m, 1H), 0.76-0.84 (m, 4H), 0.97-1.05 (m, 1H), 1.15-1.30 (m, 6H), 1.32-1.40 (m, 2H), 1.68 (td, *J* = 7.5, 12.5 Hz, 1H), 2.11 (ddd, *J* = 4.0, 7.0, 7.0 Hz, 1H), 4.11 (dd, *J* = 5.0, 8.0, Hz, 1H), 4.49, (dd, *J* = 8.5, 8.5 Hz, 1H), 4.64 (dd, *J* = 5.0, 8.5 Hz, 1H), 7.20-7.40 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 11.0, 14.4, 17.7, 22.9, 28.2, 29.5, 29.6 30.1, 32.1, 62.1, 69.7, 127.3, 129.2, 129.5, 139.0, 159.2; IR (film) cm^{-1} 2854m, 2340s, 2271s, 1684s, 1370m; mass spectrum (ESI): m/e (% relative intensity) 310 (100) $(M+Na)^+$, 288 (30), 235 (12); HRMS (ESI) m/e calcd for $C_{18}H_{25}NO_2Na$ 310.1783, found 310.1790.



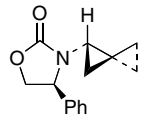
6

R_f = 0.30 [40% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.83 (dd, *J* = 4.0, 9.0 Hz, 1H), 0.91 (dd, *J* = 4.5, 9.0 Hz, 1H), 1.00 (dd, *J* = 5.0, 9.0 Hz, 1H), 1.09 (ddd, *J* = 4.5, 9.0, 13.5 Hz, 2H), 1.23 (t, *J* = 5.5 Hz, 1H), 2.97 (dd, *J* = 3.0, 7.0 Hz, 1H), 3.54 (dd, *J* = 7.0, 15.0 Hz, 1H), 3.61 (dd, *J* = 8.5, 17.0 Hz, 1H), 4.24-4.36 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 4.8, 6.5, 12.1, 15.3, 32.5, 45.2, 62.4, 159.2; IR (neat) cm^{-1} 2993w, 2921m, 1740s, 1534w, 1482m, 1416m; mass spectrum (APCI): m/e (% relative intensity) 154 (100) $(M+H)^+$, 140 (20), 110 (20), 100 (20); HRMS (MALDI) m/e calcd for $C_8H_{12}NO_2$ 154.0863, found: 154.0860.



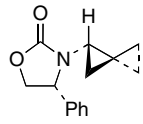
9a

R_f = 0.35 [25% EtOAc/hexanes]; mp 67-68 ºC; $[\alpha]_D^{25}$ = + 26.8 º [*c* 0.43, CH_2Cl_2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.62-0.65 (m, 2H), 0.81 (ddd, *J* = 5.2, 5.2, 9.2 Hz, 1H), 0.99 (ddd, *J* = 5.2, 5.2, 9.6 Hz, 1H), 1.06 (dd, *J* = 3.6, 5.2 Hz, 1H), 1.10 (dd, *J* = 5.2, 6.8 Hz, 1H), 2.63 (dd, *J* = 3.6, 6.8 Hz, 1H), 4.09 (dd, *J* = 6.0, 8.8 Hz, 1H), 4.53 (dd, *J* = 8.8, 8.8 Hz, 1H), 4.77 (dd, *J* = 6.0, 8.8 Hz, 1H), 7.20-7.22(m, 2H), 7.32-7.36 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.9, 6.4, 13.2, 14.9, 31.4, 60.8, 70.1, 126.9, 129.1, 129.4, 139.0, 159.0; IR (neat) cm^{-1} 3068w, 2998m, 2912w, 1755s, 1536m, 1458s; mass spectrum (APCI): m/e (% relative intensity) 230(100) $(M+H)^+$, 186 (60); HRMS (MALDI) m/e calcd for $C_{14}H_{15}NO_2Na$ 252.1000, found 252.1001.



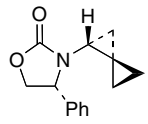
9b

R_f = 0.43 [25% EtOAc/hexanes]; mp 85-87 ºC; $[\alpha]_D^{25}$ = + 20.6 º [*c* 0.33, CH_2Cl_2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.73-0.82 (m, 2H), 0.95 (dd, *J* = 4.0, 4.8 Hz, 1H), 0.99-1.02 (m, 2H), 1.17 (ddd, *J* = 3.6, 4.8, 9.6 Hz, 1H), 2.68 (dd, *J* = 3.6, 6.8 Hz, 1H), 4.09 (dd, *J* = 4.4, 8.8 Hz, 1H), 4.53 (dd, *J* = 8.8, 8.8 Hz, 1H), 4.77 (dd, *J* = 4.4, 8.8 Hz, 1H), 7.30-7.44(m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.8, 6.6, 12.1, 17.1, 30.9, 60.9, 70.0, 126.9, 129.0, 129.5, 139.5, 158.9; IR (neat) cm^{-1} 3067w, 2998m, 2912w, 1754s, 1493m, 1409s; mass spectrum (APCI): m/e (% relative intensity) 230(100) $(M+H)^+$, 186 (45); HRMS (EI) m/e calcd for $C_{14}H_{15}NO_2$ 229.1098, found 229.1097.



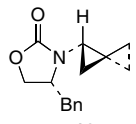
ent-**9a**

R_f = 0.10 [20% EtOAc/hexanes]; $[\alpha]_D^{20}$ = – 20.2 º [*c* 1.1, $CHCl_3$]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.62-0.71 (m, 2H), 0.86 (ddd, *J* = 5.0, 5.0, 9.0 Hz, 1H), 1.05 (ddd, *J* = 5.0, 5.0, 9.5 Hz, 1H), 1.10 (t, *J* = 5.5 Hz, 1H), 1.16 (t, *J* = 5.5 Hz, 1H), 2.69 (dd, *J* = 4.0, 8.5 Hz, 1H), 4.12 (dd, *J* = 6.0, 9.0 Hz, 1H), 4.55 (dd, *J* = 9.0, 9.0 Hz, 1H), 4.82 (dd, *J* = 6.0, 9.0 Hz, 1H), 7.24-7.30 (m, 2H), 7.32-7.45 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.9, 6.4, 13.2, 14.9, 31.4, 60.8, 70.1, 126.9, 129.1, 129.4, 139.0, 159.0; IR (neat) cm^{-1} 3004w, 2916m, 1748w, 1457s; mass spectrum (EI): m/e (% relative intensity) 228(100) $(M–H)^+$, 229 (70) $(M)^+$; HRMS (EI) m/e calcd for $C_{14}H_{15}NO_2$ 229.1098, found 229.1096.



ent-**9b**

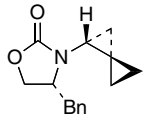
R_f = 0.17 [20% EtOAc/hexanes]; $[\alpha]_D^{20}$ = – 4.4 º [*c* 1.9, $CHCl_3$]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.71-0.81 (m, 2H), 0.91-1.10 (m, 3H), 1.17 (ddd, *J* = 3.6, 4.8, 9.6 Hz, 1H), 2.68 (dd, *J* = 3.2, 6.8 Hz, 1H), 4.19 (dd, *J* = 4.0, 8.4 Hz, 1H), 4.62 (dd, *J* = 8.4, 8.4 Hz, 1H), 4.70 (dd, *J* = 4.0, 8.4 Hz, 1H), 7.30-7.25 (m, 2H), 7.36-7.45(m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.8, 6.6, 12.1, 17.1, 30.9, 60.9, 70.1, 126.9, 129.5, 129.9, 139.5, 158.8; IR (neat) cm^{-1} 3004w, 2916m, 1747s, 1407s; mass spectrum (EI): m/e (% relative intensity) 228(100) $(M–H)^+$, 229 (65) $(M)^+$; HRMS (EI) m/e calcd for $C_{14}H_{15}NO_2$ 229.1098, found 229.1100.



10a

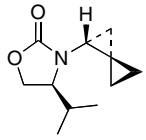
R_f = 0.13 [20% EtOAc/hexanes]; $[\alpha]_D^{20}$ = + 25.7 º [*c* 1.9, $CHCl_3$]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 0.75-0.82 (m, 2H), 0.98 (ddd, *J* = 5.0, 5.0, 9.5 Hz, 1H,), 1.13 (ddd, *J* = 5.0, 5.0, 9.5 Hz, 1H), 1.28 (t, *J* = 4.0 Hz, 1H), 1.39 (t, *J* = 6.0 Hz, 1H), 2.66 (dd, *J* = 8.5, 13.5 Hz, 1H), 2.84 (dd, *J* = 3.0, 6.5 Hz, 1H), 3.07 (dd, *J* = 4.5, 13.5 Hz, 1H), 4.00 (dd, *J* = 3.5, 7.5 Hz, 1H), 4.04-4.14 (m, 2H), 7.13 (d, *J* = 7.5 Hz, 2H), 7.26 (t, 1H, *J* = 6.5 Hz), 7.32 (t, *J* = 7.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 5.1, 6.5, 14.7,

15.0, 30.7, 39.1, 57.3, 67.0, 127.4, 129.2, 129.3, 136.2, 158.7; IR (neat) cm^{-1} 2996w, 1746s, 1413m; mass spectrum (EI) m/e (% relative intensity) 243 (100) $(M)^+$, 228 (65); HRMS (EI) m/e calcd for $C_{15}H_{17}NO_2$ 243.1254, found 243.1266.



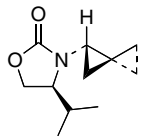
10b

R_f = 0.19 [20% EtOAc/hexanes]; $[\alpha]_D^{20}$ = – 11.9 º [*c* 1.6, $CHCl_3$]; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 0.79-0.87(m, 2H), 1.05 (t, *J* = 3.5 Hz, 1H,), 1.10 (dd, *J* = 4.5, 9.0 Hz, 1H), 1.18-1.28 (m, 2H), 2.75 (dd, *J* = 9.5, 13.5 Hz, 1H), 2.89 (dd, *J* = 2.5, 6.0 Hz, 1H), 3.22 (dd, *J* = 4.0, 13.5 Hz, 1H), 3.90 (m, 1H), 4.05 (dd, *J* = 2.5, 9.0 Hz, 1H), 4.14 (t, *J* = 9.0 Hz, 1H), 7.19 (d, *J* = 7.5 Hz, 2H), 7.27 (t, 1H, *J* = 6.5 Hz), 7.34 (t, *J* = 7.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 4.7, 6.8, 12.1, 18.1, 30.3, 38.8, 58.0, 66.6, 127.4, 129.2, 129.5, 136.2, 158.6; IR (neat) cm^{-1} 2999w, 1756s, 1418m; ; mass spectrum (EI) m/e (% relative intensity) 243 (100) $(M)^+$, 228 (40); HRMS (EI) m/e calcd for $C_{15}H_{17}NO_2$ 243.1254, found 243.1260.



11a

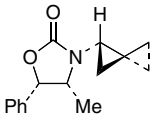
R_f = 0.24 [25% EtOAc/hexanes]; $[\alpha]_D^{25}$ = – 47.6 º [*c* 0.55, CH_2Cl_2]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.83 (ddd, *J* = 4.5, 9.0, 13.5 Hz, 1H), 0.88 (d, *J* = 6.0 Hz, 3H), 0.89 (d, *J* = 6.0 Hz, 3H), 0.91-0.99 (m, 2H), 1.04 (ddd, *J* = 4.5, 9.5, 14.0 Hz, 1H), 1.27 (dd, *J* = 3.0, 5.0 Hz, 1H), 1.39 (dd, *J* = 6.0, 6.0 Hz, 1H), 1.90 (dqq, *J* = 3.5, 7.0, 7.0 Hz, 1H), 2.80 (dd, *J* = 3.0, 7.0 Hz, 1H), 3.80 (ddd, *J* = 4.0, 4.5, 9.0 Hz, 1H), 4.05 (dd, *J* = 4.0, 8.5 Hz, 1H), 4.17 (dd, *J* = 8.5, 9.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.9, 6.5, 14.6, 15.0, 15.1, 18.0, 28.7, 30.4, 60.0, 63.3, 158.9; IR (neat) cm^{-1} 3100w, 2920m, 2800w, 2305m, 1750s, 1480m, 1400s; mass spectrum (APCI): m/e (% relative intensity) 196(100) $(M+H)^+$; HRMS (EI) m/e calcd for $C_{11}H_{17}NO_2$ 195.1254, found 195.1259.



11b

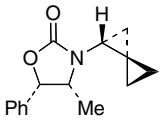
R_f = 0.30 [25% EtOAc/hexanes]; $[\alpha]_D^{25}$ = – 8.8 º [*c* 0.50, CH_2Cl_2]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.78-0.83 (m, 2H), 0.90 (m, 1H), 0.92 (d, *J* = 7.0 Hz, 3H), 0.94(d, *J* = 7.5 Hz, 3H), 1.10 (ddd, *J* = 5.0, 5.0, 9.5 Hz, 1H), 1.19 (dd, *J* = 5.5, 6.5 Hz, 1H), 1.26 (ddd, *J* = 4.0, 4.0, 9.0 Hz, 1H), 2.17 (dqq, *J* = 3.5, 7.0, 7.0 Hz, 1H), 2.80 (dd, *J* = 3.5, 7.0 Hz, 1H), 3.62 (ddd, *J* = 3.5, 3.5, 9.0 Hz, 1H), 4.08 (dd, *J* = 3.5, 9.0 Hz, 1H), 4.22 (dd, *J* = 9.0, 9.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.6, 6.8, 12.1, 15.3, 17.9, 18.3, 29.3, 30.2, 61.0, 63.5, 159.0; IR (neat) cm^{-1} 3110w, 2950s, 2750w, 2300w, 1760s, 1500m, 1400s; mass spectrum (APCI): m/e (% relative intensity) 196(100) $(M+H)^+$; HRMS (EI) m/e calcd for

$C_{11}H_{17}NO_2$ 195.1254, found 195.1252.



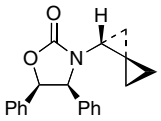
14a

R_f = 0.40 [25% EtOAc/hexanes]; $[\alpha]_D^{25}$ = + 60.4 º [*c* 0.50, CH_2Cl_2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.71 (d, *J* = 6.4 Hz, 3H), 0.82-0.87 (m, 2H), 1.00(ddd, *J* = 4.4, 5.2, 9.6 Hz, 1H), 1.10 (ddd, *J* = 4.8, 5.2, 9.2 Hz, 1H), 1.29 (dd, *J* = 3.2, 4.8 Hz, 1H), 1.42 (dd, *J* = 5.6, 6.4 Hz, 1H), 2.84 (dd, *J* = 3.2, 6.4 Hz, 1H), 4.13 (qd, *J* = 6.8, 7.6 Hz, 1H), 5.53 (d, *J* = 7.6 Hz, 1H), 7.26-7.29 (m, 2H), 7.33-7.40 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.8, 6.6, 14.4, 14.5, 14.8, 30.4, 56.3, 78.7, 126.2, 128.6 128.7, 135.3, 158.5; IR (neat) cm^{-1} 3150w, 3000m, 2750m, 1750s, 1490m, 1400s; mass spectrum (APCI): m/e (% relative intensity) 244(100) $(M+H)^+$, 200 (75); HRMS (EI) m/e calcd for $C_{15}H_{17}NO_2$ 243.1254, found 243.1253.



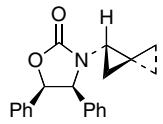
14b

R_f = 0.50 [25% EtOAc/hexanes]; mp 78-80 ºC; $[\alpha]_D^{25}$ = + 54.6 º [*c* 0.45, CH_2Cl_2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.83-0.85 (m, 5H), 1.02 (dd, *J* = 3.6, 4.8 Hz, 1H), 1.13 (dd, *J* = 2.8, 4.0 Hz, 1H), 1.20-1.30 (m, 2H), 2.84 (dd, *J* = 3.2, 6.4 Hz, 1H), 3.99(qd, *J* = 6.8, 8.0 Hz, 1H), 5.59 (d, *J* = 8.0 Hz, 1H), 7.27-7.41 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.7, 6.7, 12.0, 14.4, 17.8, 30.3, 57.4, 78.7, 126.1, 128.5 128.7, 135.3, 158.4; IR (neat) cm^{-1} 3100w, 3000m, 2800w, 1750s, 1490m, 1400s; mass spectrum (APCI): m/e (% relative intensity) 244(100) $(M+H)^+$, 200 (35) ; HRMS (EI) m/e calcd for $C_{15}H_{17}NO_2$ 243.1254, found 243.1250.



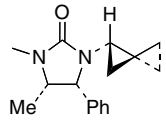
15a

R_f = 0.38 [33% EtOAc/hexanes]; mp 160-162 ºC; $[\alpha]_D^{22}$ = – 98.4 º [*c* 0.40, CH_2Cl_2]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.55 (ddd, *J* = 9.2, 4.4, 4.4 Hz, 1H); 0.69 (ddd, *J* = 9.2, 5.2, 4.4, 1H); 0.93 (ddd, *J* = 8.8, 5.6, 4.4 Hz, 1H); 1.00 (ddd, *J* = 9.2, 4.8, 4.8 Hz, 1H); 1.29-1.35 (m, 2H), 2.85 (dd, *J* = 6.8, 3.2 Hz, 1H); 5.03 (d, *J* = 8.0 Hz, 1H); 5.80 (d, *J* = 8.0 Hz, 1H); 6.83-6.85 (m, 2H), 6.97-7.00 (m, 2H), 7.06-7.10 (m, 6H); ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 4.8, 6.4, 14.2, 15.0, 31.4, 65.7, 80.0, 126.2, 127.7, 128.0, 128.1, 128.4, 128.5, 134.7, 134.8, 158.9; IR (neat) cm^{-1} 3064w, 1747s, 1403m, 1130m; mass spectrum (APCI): m/e (% relative intensity) 306 (100) $(M+H)^+$, 262 (40); HRMS (EI) m/e calcd for $C_{20}H_{19}NO_2$ 305.1416, found 305.1417.



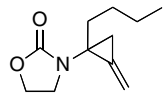
15b

R_f = 0.48 [33% EtOAc/hexanes]; mp 115-117 ºC; $[\alpha]_D^{22}$ = – 76.8 º [*c* 0.80, CH_2Cl_2; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.79-0.89 (m, 2H); 0.96-1.02 (m, 2H); 1.11 (ddd, *J* = 8.8, 4.4, 4.4 Hz, 1H); 1.33 (ddd, *J* = 8.8, 4.4, 3.6 Hz, 1H); 2.86 (dd, *J* = 6.8, 3.6 Hz, 1H); 4.91 (d, *J* = 7.6 Hz, 1H); 5.84 (d, *J* = 7.6 Hz, 1H); 6.91-6.94 (m, 2H), 6.99-7.02 (m, 2H), 7.07-7.14 (m, 6H); ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 4.7, 6.7, 12.4, 17.4, 31.4, 66.7, 80.2. 126.2, 127.8, 128.0, 128.1, 128.4, 128.5, 134.6, 134.9, 158.9;
IR (neat) cm^{-1} 2922w, 2853w, 1741s, 1405m; mass spectrum (APCI): m/e (% relative intensity) 306 (100) $(M+H)^+$, 262 (80); HRMS (MALDI) m/e calcd for $C_{20}H_{19}NO_2Na$ 328.1308, found 328.1314.



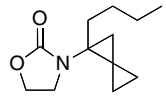
17a

R_f = 0.33 [50% EtOAc/hexanes]; mp 127-130 ºC; $[\alpha]_D^{25}$ = + 32.0 º [*c* 0.25, CH_2Cl_2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.70-0.75 (m, 5H), 0.86 (ddd, *J* = 3.6, 8.8, 9.2 Hz, 1H), 1.02 (ddd, *J* = 5.2, 6.0, 8.8 Hz, 1H), 1.19-1.28 (m, 2H), 2.69 (dd, *J* = 4.4, 8.4 Hz, 1H), 2.71 (s, 3H), 3.73(qd, *J* = 6.4, 8.4 Hz, 1H), 4.70 (d, *J* = 8.4 Hz, 1H), 7.16-7.19 (m, 2H), 7.29-7.37 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 5.1, 6.5, 12.1, 15.0, 17.3, 29.0, 31.3, 56.1, 63.5, 128.2, 128.4, 128.6, 136.9, 162.7; IR (neat) cm^{-1} 3065w, 2970m, 2800w, 2870w, 1757m, 1687s, 1492m, 1428s; mass spectrum (APCI): m/e (% relative intensity) 257(100) $(M+H)^+$; HRMS (EI) m/e calcd for $C_{16}H_{20}N_2O$ 256.1571, found 256.1569.



29-M

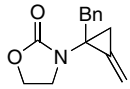
R_f = 0.50 [40% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.5 Hz, 3H), 1.28-1.52 (m, 5H), 1.56-1.65 (m, 2H), 1.87 (ddd, *J* = 5.5, 10.0, 15.5 Hz, 1H), 3.52-3.69 (m, 2 H), 4.23-4.29 (m, 2H), 5.51 (t, *J* = 2.0 Hz, 1H), 5.74 (t, *J* = 3.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.3, 17.3, 22.9, 28.8, 34.1, 44.9, 45.0, 61.9, 106.2, 135.7, 157.7; IR (neat) cm^{-1} 2958w, 2931w, 2872w, 1744s, 1482w, 1410s; mass spectrum (APCI): m/e (% relative intensity) 196 (100) $(M+H)^+$, 123 (20); HRMS (MALDI) m/e calcd for $C_{11}H_{17}NO_2Na$ 218.1152, found 218.1158.



29-B

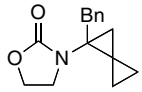
R_f = 0.55 [40% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.73 (dd, *J* = 4.5, 8.5 Hz, 1H), 0.85 (dd, *J* = 4.5, 9.0 Hz, 1H), 0.90 (t, *J* = 7.5 Hz, 3H), 0.99 (dd, *J* = 5.0, 9.5 Hz, 2H), 1.25 (ddd, *J* = 4.5, 9.0, 14.0 Hz, 2 H), 1.29-1.52 (m, 5H), 1.86 (ddd, *J* = 4.0, 11.0, 14.5 Hz, 1H), 3.55 (dd, *J* = 8.0, 15.5 Hz,

1H), 3.67 (dd, *J* = 9.0, 17.0 Hz, 1H), 4.29 (t, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 5.4, 5.8, 14.4, 18.1, 22.3, 23.2, 28.7, 34.2, 39.7, 44.9, 62.2, 158.1; IR (neat) cm^{-1} 2958w, 2932w, 2361m, 2342m, 1744s, 1482w, 1413m; mass spectrum (APCI): m/e (% relative intensity) 210 (100) $(M+H)^+$, 123 (20); HRMS (MALDI, m/z) calcd for $C_{12}H_{19}NO_2Na$ 232.1308, found 232.1299.



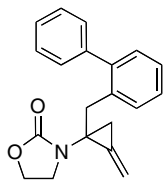
30-M

R_f = 0.47 [40% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.59-1.68 (m, 2H), 2.74 (d, *J* = 14.0 Hz, 1H), 2.84 (ddd, *J* = 7.0, 8.5, 17.0 Hz, 1H), 3.12 (ddd, *J* = 7.5, 9.0, 16.0 Hz, 1H), 3.36 (d, *J* = 13.5 Hz, 1H), 4.01-4.08 (m, 2H), 5.57 (t, *J* = 2.5 Hz, 1H), 5.76 (t, *J* = 3.0 Hz, 1H), 7.25-7.33 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.0, 37.4, 40.4, 45.4, 62.1, 107.1, 127.2, 128.8, 129.6, 135.1, 138.7, 157.8; IR (neat) cm^{-1} 3063w, 3029w, 2992w, 2917w, 2360w, 1743s, 1603w, 1494m, 1480w, 1454w, 1410m; mass spectrum (APCI): m/e (% relative intensity) 230 (100) $(M+H)^+$, 186 (10), 143 (70); HRMS (MALDI) m/e calcd for $C_{14}H_{15}NO_2Na$ 252.0995, found 252.0984.



30-B

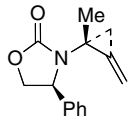
R_f = 0.55 [40% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.87 (dddd, *J* = 3.0, 4.5, 8.5, 12.5 Hz, 2H), 1.16 (dt, *J* = 3.0, 6.5 Hz, 1H), 1.21 (d, *J* = 5.5 Hz, 1H), 1.25 (dd, *J* = 6.5, 12.0 Hz, 2H), 2.61 (d, *J* = 14.0 Hz, 1 H), 2.70 (dd, *J* = 8.5, 16.0 Hz, 1H), 3.17 (dd, *J* = 6.0, 8.0 Hz, 1H), 3.34 (d, *J* = 14.0 Hz, 1H), 3.90 – 4.09 (m, 2H), 7.23 - 7.32 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 5.6, 5.7, 18.4, 22.8, 40.2, 41.0, 45.2, 62.4, 127.0, 128.8, 129.0, 129.6, 129.7, 139. 2, 158.2; IR (neat) cm^{-1} 3061w, 2993w, 2918w, 1742s, 1526m, 1485m, 1411s; mass spectrum (APCI): m/e (% relative intensity) 244 (100) $(M+H)^+$, 157 (60); HRMS (MALDI, m/z) calcd for $C_{15}H_{17}NO_2Na$: 266.1152, found 266.1165.



31-M

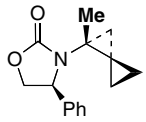
R_f = 0.53 [40% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.12 (dt, *J* = 2.5, 10.5 Hz, 1H), 1.39 (dt, *J* = 2.5, 10.5 Hz, 1H), 2.85 (dd, *J* = 8.5, 17.0 Hz, 1H), 2.92 (dd, *J* = 7.0, 15.5 Hz, 1H), 3.05 (d, *J* = 14.0 Hz, 1H), 3.30 (d, *J* = 14.0 Hz, 1H), 3.95-4.02 (m, 2H), 5.42 (t, *J* = 2.5 Hz, 1H), 5.65 (t, *J* = 3.0 Hz, 1H), 7.24-7.46 (m, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 17.4, 36.1, 36.9, 44.3, 61.8, 106.8, 127.3, 127.4, 127.7, 128.6, 129.7, 130.6, 131.1, 134.8, 135.6, 141.9, 142.7, 157.3; IR (neat) cm^{-1} 3058w, 2361w, 2342w, 1749s, 1703m, 1479m, 1411m; mass spectrum (APCI): m/e (% relative intensity) 306 (100) $(M+H)^+$, 219 (80), 165 (30); HRMS (MALDI) m/e calcd for $C_{20}H_{19}NO_2Na$ 328.1308, found

328.1309.



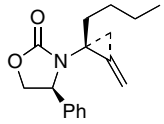
36-M

R_f = 0.33 [25% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.22 (s, 3H), 1.33 (dd, *J* = 2.0, 11.0 Hz, 1H), 1.48 (dd, *J* = 2.0, 10.5 Hz, 1H), 4.13 (ddd, *J* = 3.0, 9.0, 9.0 Hz, 1H), 4.52 (ddd, *J* = 3.0, 9.0, 9.0 Hz, 1H), 4.70 (ddd, *J* = 3.0, 9.0, 9.0 Hz, 1H), 5.47 (b, 1H), 5.50 (d, *J* = 3.0 Hz, 1H), 7.39-7.45 (m, 5H); IR (neat) cm^{-1} 3034w, 2998m, 2906w, 1752s, 1710m, 1495m, 1400s; mass spectrum (APCI): m/e (% relative intensity) 230(100) $(M+H)^+$, 186 (45); HRMS (EI) m/e calcd for $C_{14}H_{14}NO_2$ 228.1020, found 228.1015.



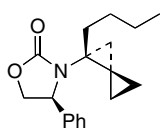
36-B

R_f = 0.38 [25% EtOAc/hexanes]; $[\alpha]_D^{25}$ = + 100.0° [*c* 0.25, CH_2Cl_2]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.61 (ddd, *J* = 3.5, 5.5, 8.5 Hz, 1H), 0.63 (d, *J* = 5.0 Hz, 1H), 0.82 (d, *J* = 5.0 Hz, 1H), 0.83-0.87 (m, 2H), 1.22 (s, 3H), 1.33 (ddd, *J* = 3.0, 5.5, 8.5 Hz, 1H), 4.14 (dd, *J* = 7.0, 8.5 Hz, 1H), 4.56 (dd, *J* = 8.5, 8.5 Hz, 1H), 4.90 (dd, *J* = 7.0 8.5 Hz, 1H), 7.32-7.40 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 4.8, 6.7, 19.2, 19.7, 21.1, 36.5, 59.9, 70.4, 127.5, 129.2, 129.3, 139.9, 158; IR (neat) cm^{-1} 3064w, 2997m, 2911w, 1749m, 1687s, 1525m, 1480s; mass spectrum (APCI): m/e (% relative intensity) 244(100) $(M+H)^+$, 200 (20); HRMS (EI) m/e calcd for $C_{15}H_{17}NO_2$ 243.1254, found 243.1261.



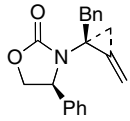
37-M

R_f = 0.31 [25% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.78 (t, *J* = 7.0 Hz, 3H), 1.12-1.35 (m, 7H), 1.45 (dd, *J* = 2.0, 11.0 Hz, 1H), 4.14 (dd, *J* = 7.0, 9.0 Hz, 1H), 4.52 (dd, *J* = 9.0, 9.0 Hz, 1H), 4.70 (dd, *J* = 3.0, 9.0, 9.0 Hz, 1H), 5.49 (b, 1H), 5.84 (d, *J* = 2.5, 3.0 Hz, 1H), 7.39-7.45 (m, 5H); IR (neat) cm^{-1} 3065m, 2956s, 2872m, 1752s, 1709m, 1495m, 1458s; mass spectrum (APCI): m/e (% relative intensity) 272 (100) $(M+H)^+$, 228 (15); HRMS (EI) m/e calcd for $C_{17}H_{21}NO_2$ 271.1568, found 271.1572.



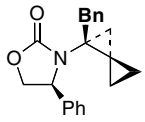
37-B

R_f = 0.38 [25% EtOAc/hexanes]; $[\alpha]_D^{25}$ = + 90.5° [*c* 0.55, CH_2Cl_2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.61-0.65 (m, 2H), 0.78 (ddd, *J* = 4.8, 5.2, 9.2 Hz, 1H), 0.82 (d, *J* = 4.8 Hz, 1H), 0.88 (t, *J* = 7.2 Hz, 3H), 0.92 (ddd, *J* = 3.2, 3.2, 8.8 Hz, 1H), 1.14-1.31 (m, 3H), 1.35-1.45 (m, 3H), 1.73 (ddd, *J* = 4.0, 4.8, 7.2 Hz, 1H), 4.12 (dd, *J* = 5.6, 8.8 Hz, 1H), 4.59 (dd, *J* = 8.8, 8.8 Hz, 1H), 4.89 (dd, *J* = 5.6, 8.8 Hz, 1H), 7.27-7.40 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 5.1, 6.2, 14.3, 17.1, 21.3, 23.1, 28.5, 35.0, 40.3, 60.9, 70.5, 127.1, 129.0, 129.2, 140.7, 158.4; IR (neat) cm^{-1} 3098w, 2910m, 2775m, 1750s, 1687s, 1505m, 1425s; mass spectrum (APCI): m/e (% relative intensity) 286 (100) $(M+H)^+$, 242(10); HRMS (EI) m/e calcd for $C_{18}H_{23}NO_2$ 285.1824, found 285.1721.



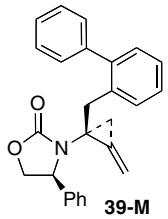
38-M

R_f = 0.26 [25% EtOAc/hexanes]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.24-1.27 (m, 2H), 2.83 (d, *J* =14.0 Hz, 1H), 3.08 (d, *J* =13.6 Hz, 1H), 3.99 (ddd, *J* =1.2, 4.0, 5.6 Hz, 1H), 4.11 (ddd, *J* = 1.2, 6.8, 14.4 Hz, 1H), 4.39 (ddd, *J* = 9.0, 9.0 Hz, 1H), 5.44 (b, 1H), 5.59 (dd, *J* = 2.4, 3.2 Hz, 1H), 7.02-7.30 (m, 10H); IR (neat) cm^{-1} 3030s, 2910m, 1752s, 1603m, 1495m, 1478s; mass spectrum (APCI): m/e (% relative intensity) 306 (100) $(M+H)^+$; HRMS (EI) m/e calcd for $C_{20}H_{19}NO_2$ 305.1411, found 305.1426.



38-B

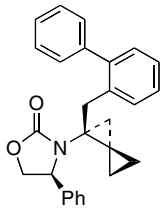
R_f = 0.43 [25% EtOAc/hexanes]; $[\alpha]_D^{25}$ = + 132.7° [*c* 2.80, CH_2Cl_2]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.67 (d, *J* = 5.0Hz, 1H), 0.71-0.72 (m, 2H), 0.88 (d, *J* = 5.5 Hz, 1H), 1.13 (m, 1H), 1.14 (m, 1H), 2.33 (d, *J* = 14.5 Hz, 1H), 3.52 (dd, *J* = 1.0, 14.0 Hz, 1H), 3.73 (dd, *J* = 5.0, 9.0 Hz, 1H), 3.82 (dd, *J* = 5.0, 9.0 Hz, 1H), 4.16 (dd, *J* = 9.0, 9.0 Hz, 1H), 7.03-7.05(m, 2H) 7.29-7.40 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 5.3, 5.5, 16.5, 21.6, 40.4, 41.9, 61.3, 71.0, 126.6, 127.1, 128.7, 128.8, 129.1, 129.7, 139.6, 141.2, 158.8; IR (neat) cm^{-1} 3000m, 2800m, 2775w, 1750s, 1500m, 1400m; mass spectrum (APCI): m/e (% relative intensity) 320 (100) $(M+H)^+$; HRMS (EI, m/z) calcd for $C_{21}H_{21}NO_2$ 319.1567, found 319.1552.



39-M

R_f = 0.28 [25% EtOAc/hexanes]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.64 (td, *J* =2.0, 10.5 Hz, 1H), 0.87 (td, *J* =2.5, 10.5 Hz, 1H), 3.08 (d, *J* =14.0 Hz, 1H), 3.18 (d, *J* =14.0 Hz, 1H), 3.88 (dd, *J* =7.0, 8.5 Hz, 1H), 4.07 (dd, *J* = 7.0, 8.5 Hz, 1H), 4.27 (dd, *J* = 8,5, 8.5 Hz, 1H), 5.35 (b, 1H), 5.63 (b, 1H), 6.82-6.98 (m,

2H), 7.20-7.44 (m, 12H); IR (neat) cm^{-1} 3038m, 2974m, 2917w, 1956w, 1752s, 1598w, 1447s, 1450s; mass spectrum (APCI): m/e (% relative intensity) 382 (100) $(M+H)^+$, 219 (25); HRMS (EI) m/e calcd for $C_{26}H_{23}NO_2$ 381.1724, found 381.1710.



39-B

.

R_f = 0.41 [25% EtOAc/hexanes]; $[\alpha]_D^{25}$ = + 28.5 ° [*c* 2.80, CH_2Cl_2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.01 (d, *J* = 5.6 Hz, 1H), 0.06 (d, *J* = 5.2 Hz, 1H), 0.08 - 0.09 (m, 2H), 1.32 (ddd, *J* = 3.6, 4.0, 9.6 Hz, 1H), 1.60 (ddd, *J* = 3.6, 4.0, 8.8 Hz, 1H), 3.10 (d, *J* = 14.4 Hz, 1H), 3.82 (d, *J* = 14.4 Hz, 1H), 3.99 (dd, *J* = 4.8, 9.2 Hz, 1H), 4.09 (dd, *J* = 5.2, 8.4 Hz, 1H), 4.21 (dd, *J* = 8.4, 9.2 Hz, 1H), 7.33-7.36 (m, 2H) 7.59-7.75 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 5.1, 5.3, 16.5, 20.7, 36.8, 40.7, 61.1, 71.0 126.5, 127.28, 127.34, 127.7 128.5, 128.6, 129.1, 129.6, 130.6, 131.5, 136.8, 141.5, 142.2, 143.1, 158.8 ; IR (neat) cm^{-1} 3000s, 2900m, 2775w, 1750s, 1490m, 1400m; mass spectrum (APCI): m/e (% relative intensity) 396 (100) $(M+H)^+$; HRMS (EI) m/e calcd for $C_{27}H_{25}NO_2$ 395.1880, found: 395.1884.

SUPPORTING INFORMATION

PROTON NMR AND SELECTED CARBON NMR SPECTRA

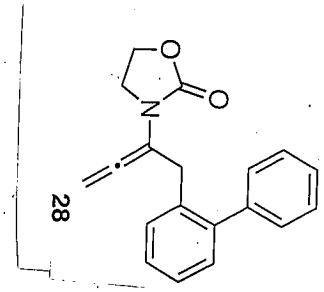
for the
article
entitled

Synthesis of Amido-Spiro [2.2] Pentanes via Simmons-Smith Cyclopropanation of Allenamides.

authored by

Ting Lu, Ryuji Hayashi, Richard P. Hsung*, Kyle A. Dekorver, Andrew G. Lohse, Zhenlei Song and Yu Tang

Division of Pharmaceutical Sciences and Department of Chemistry, 7111 Rennebohm Hall, 777 Highland Avenue
University of Wisconsin at Madison, Madison, WI 53705-2222



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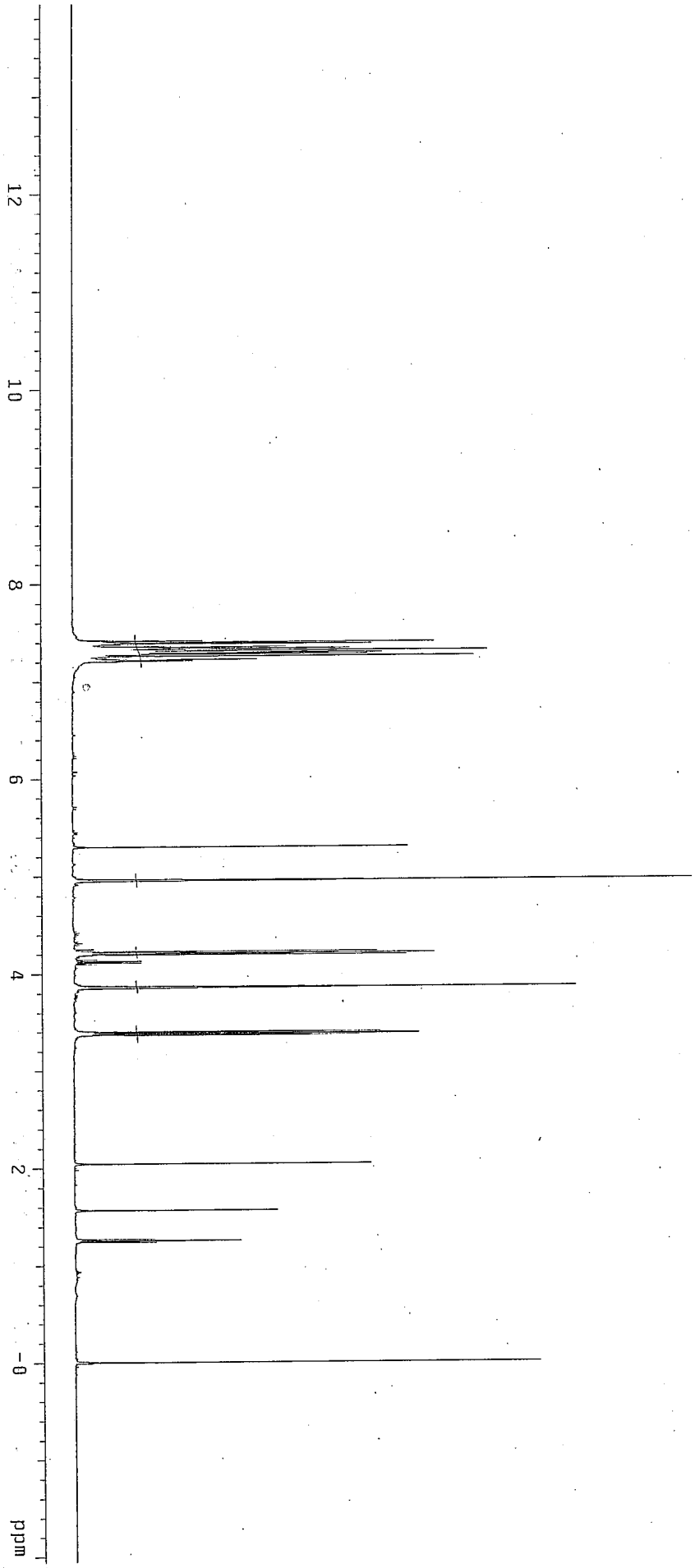

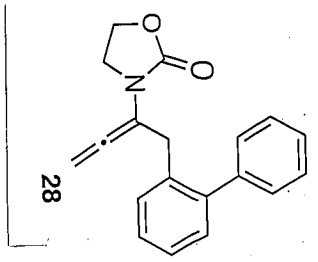

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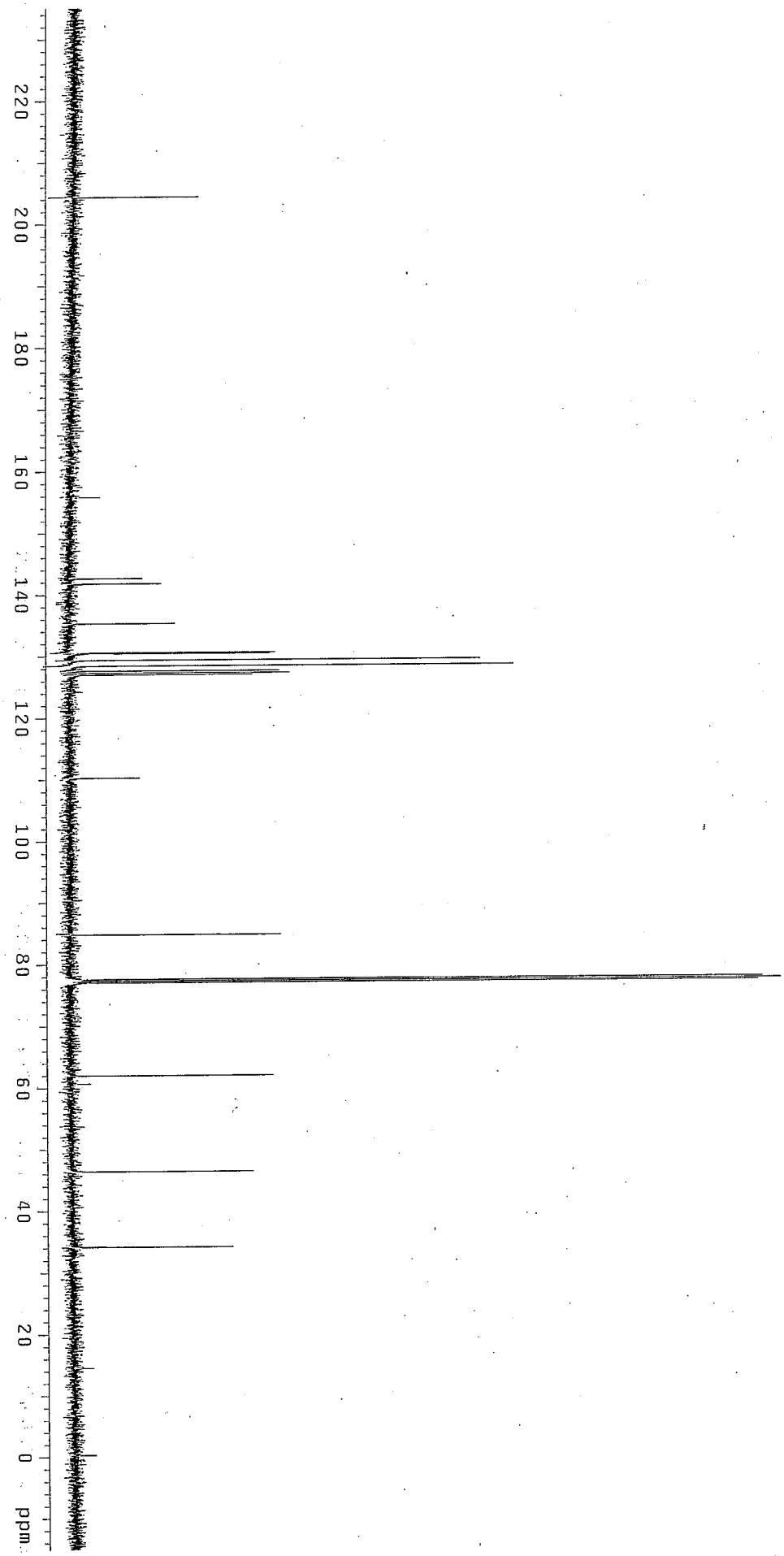

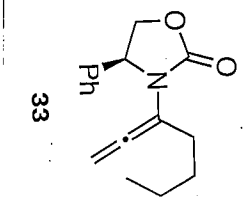

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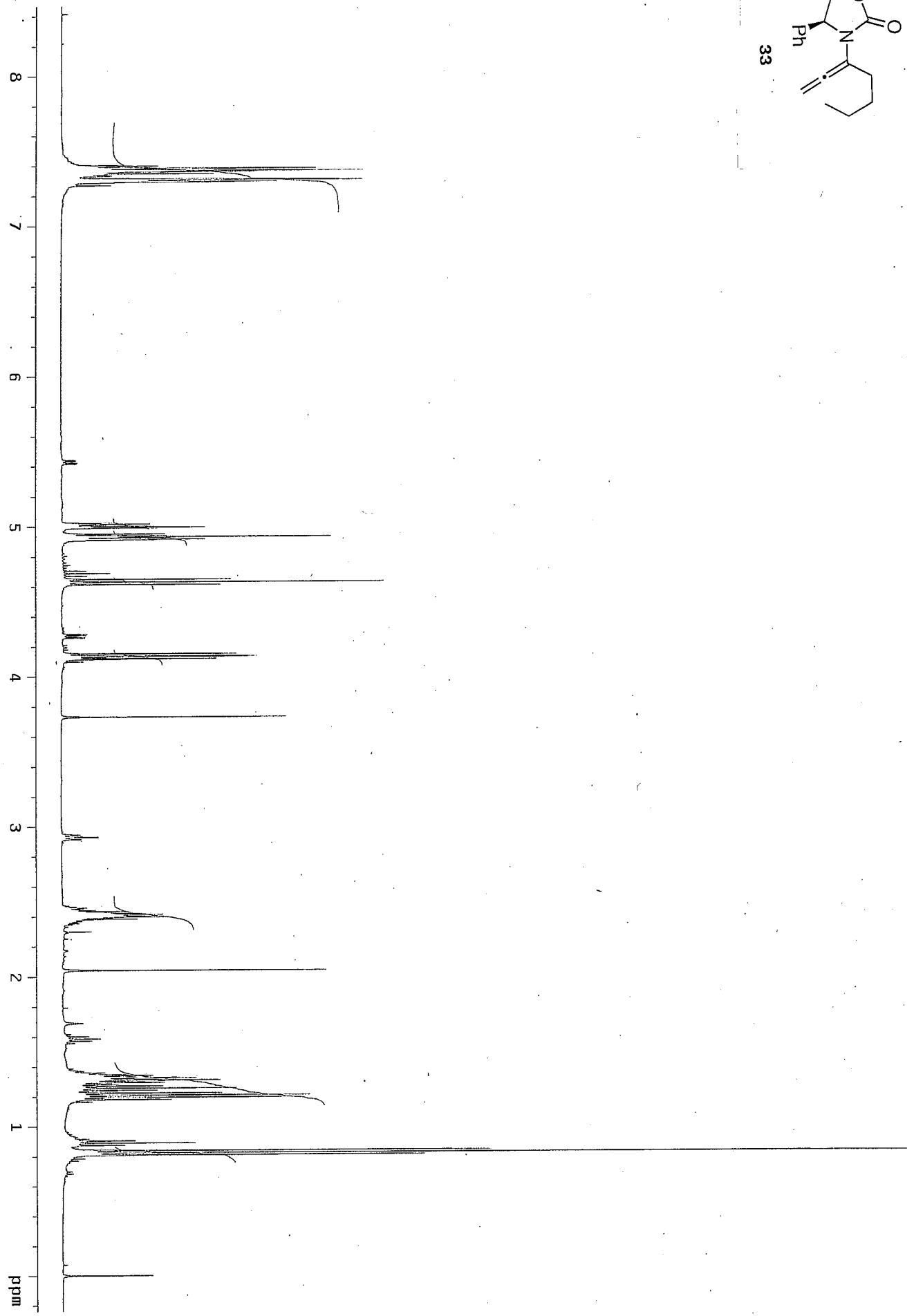

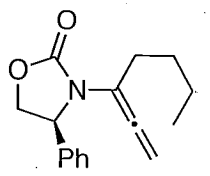

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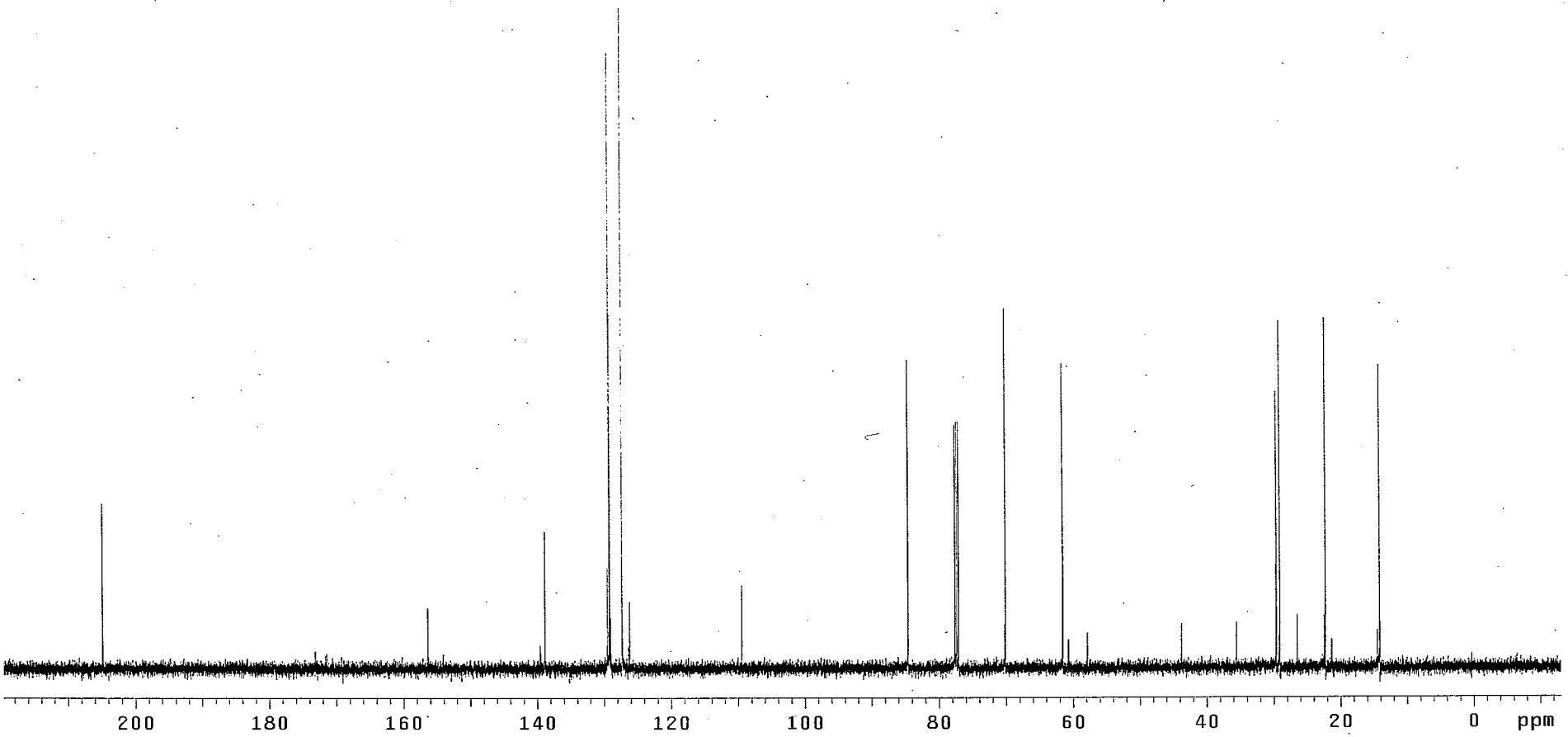

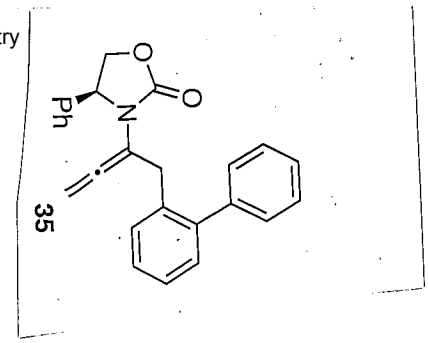

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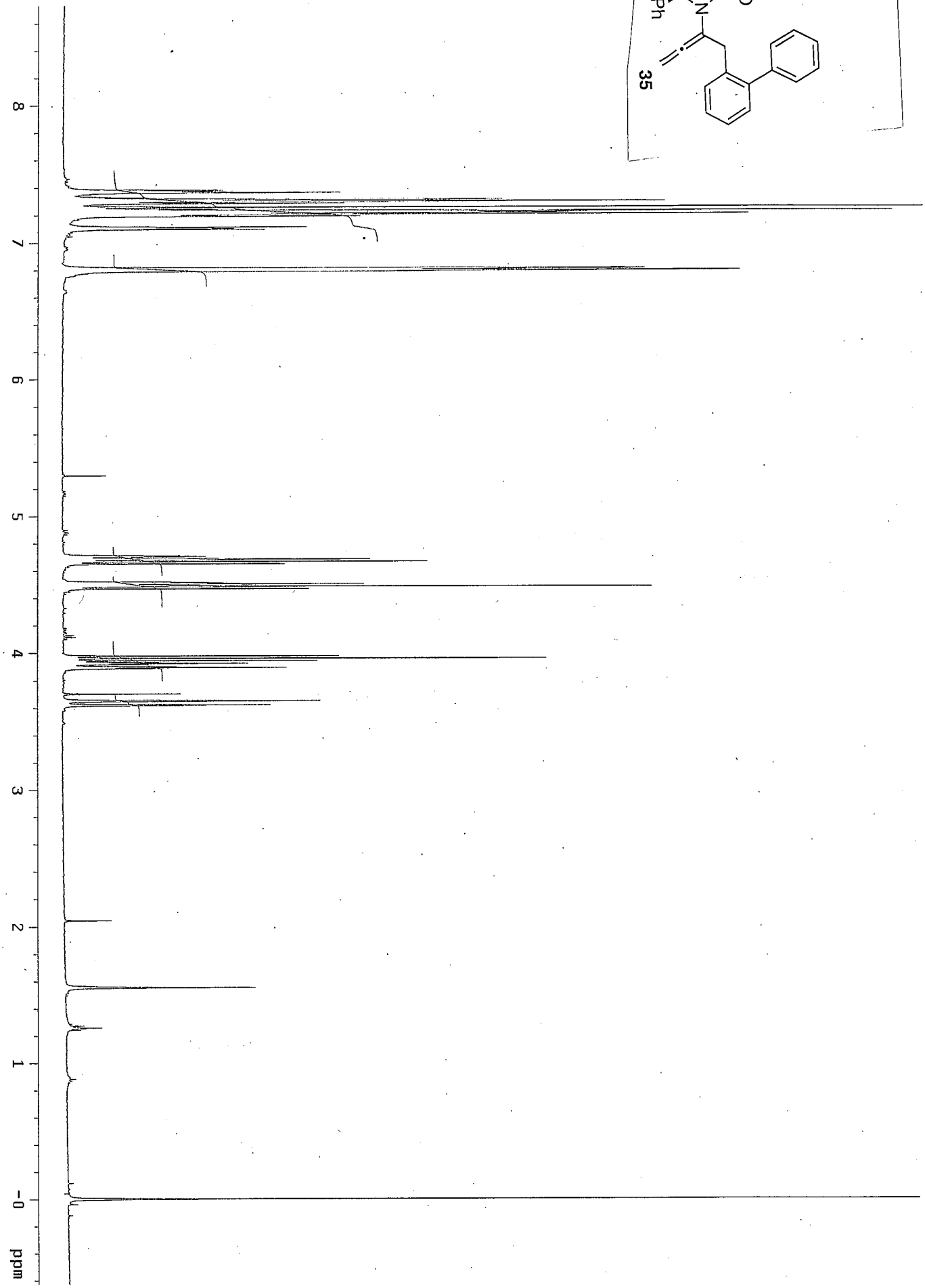
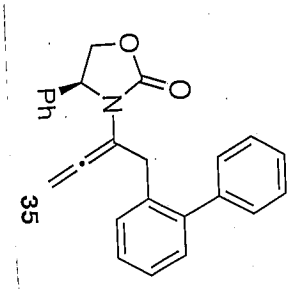
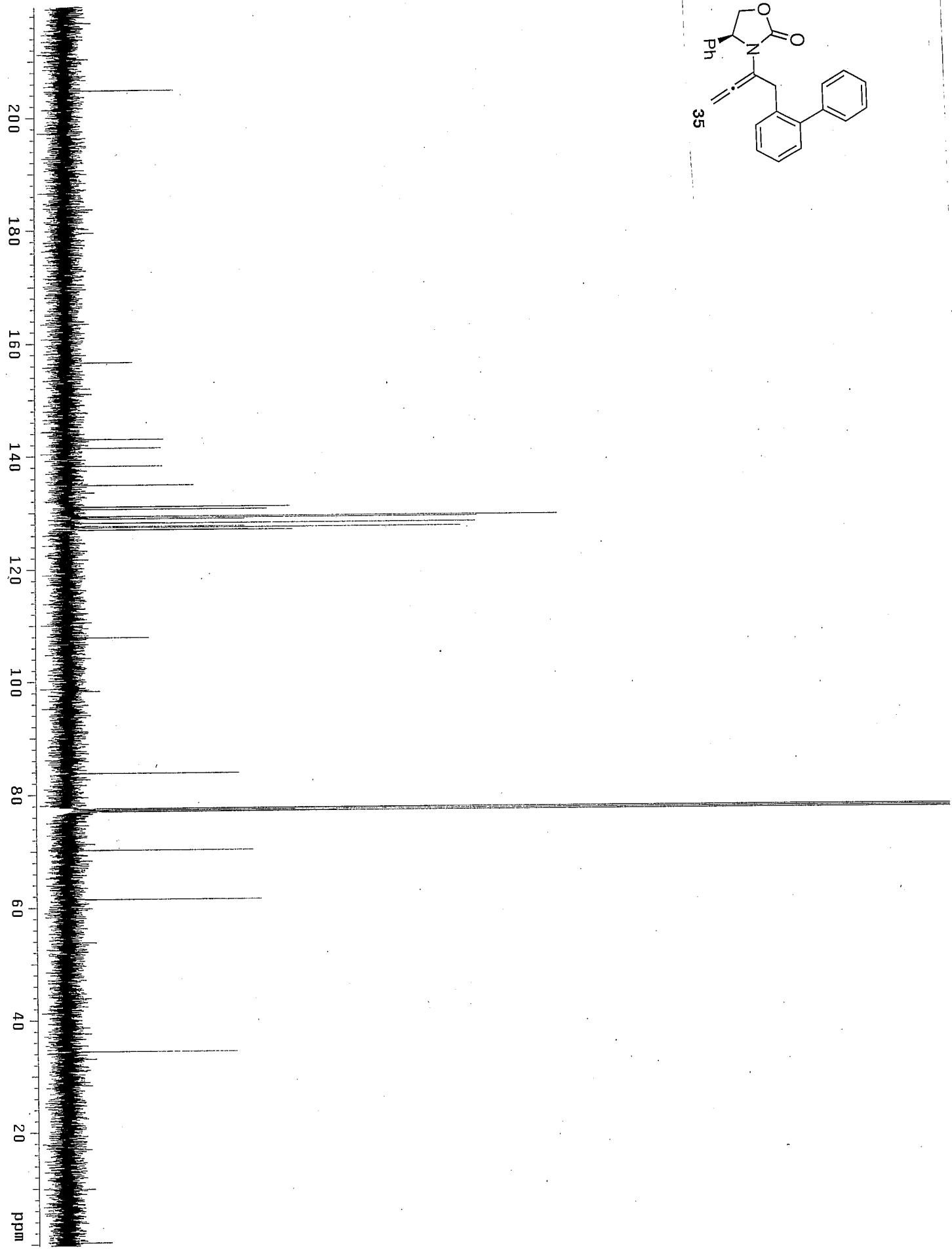
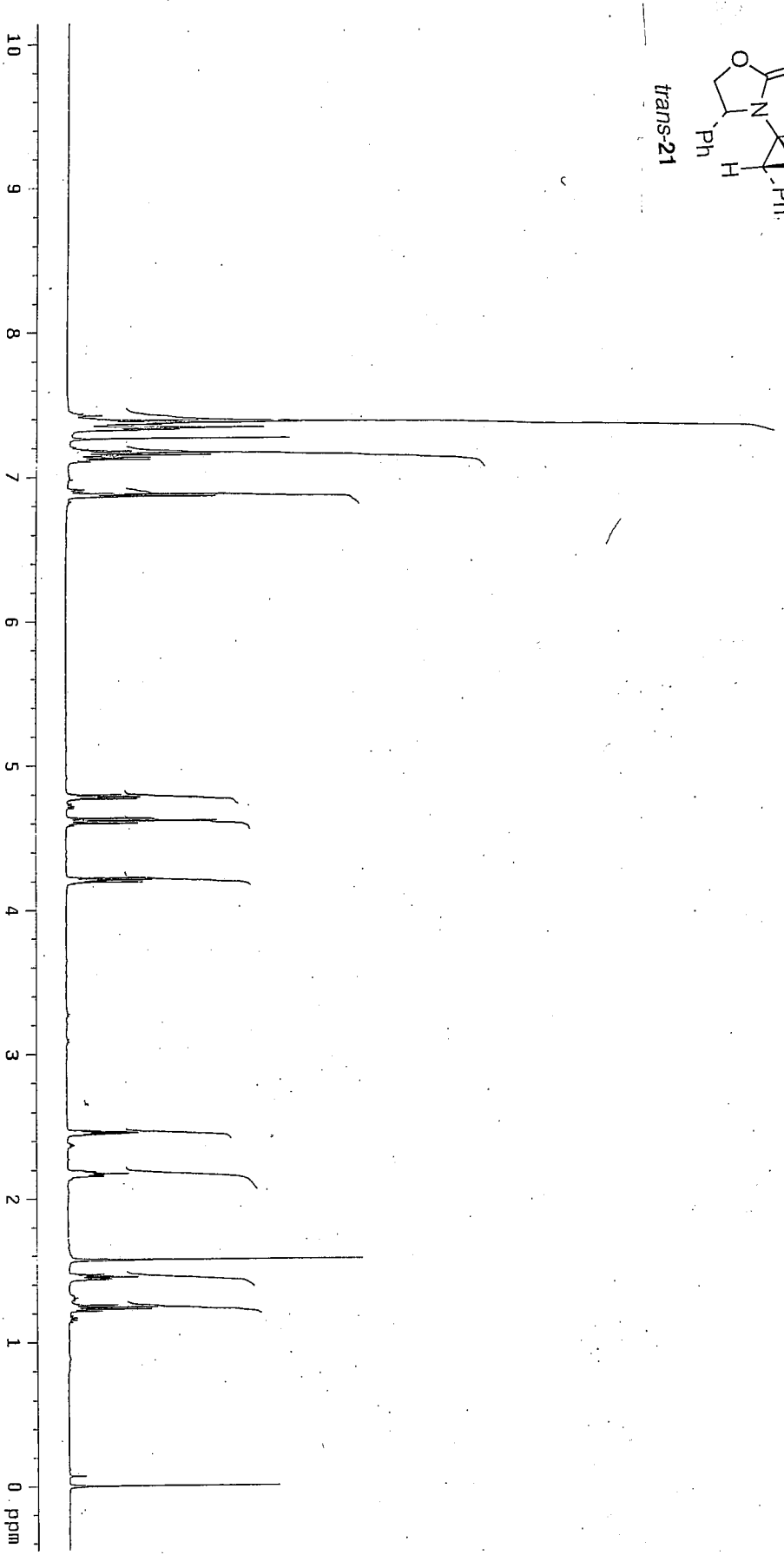
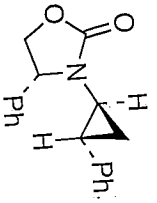


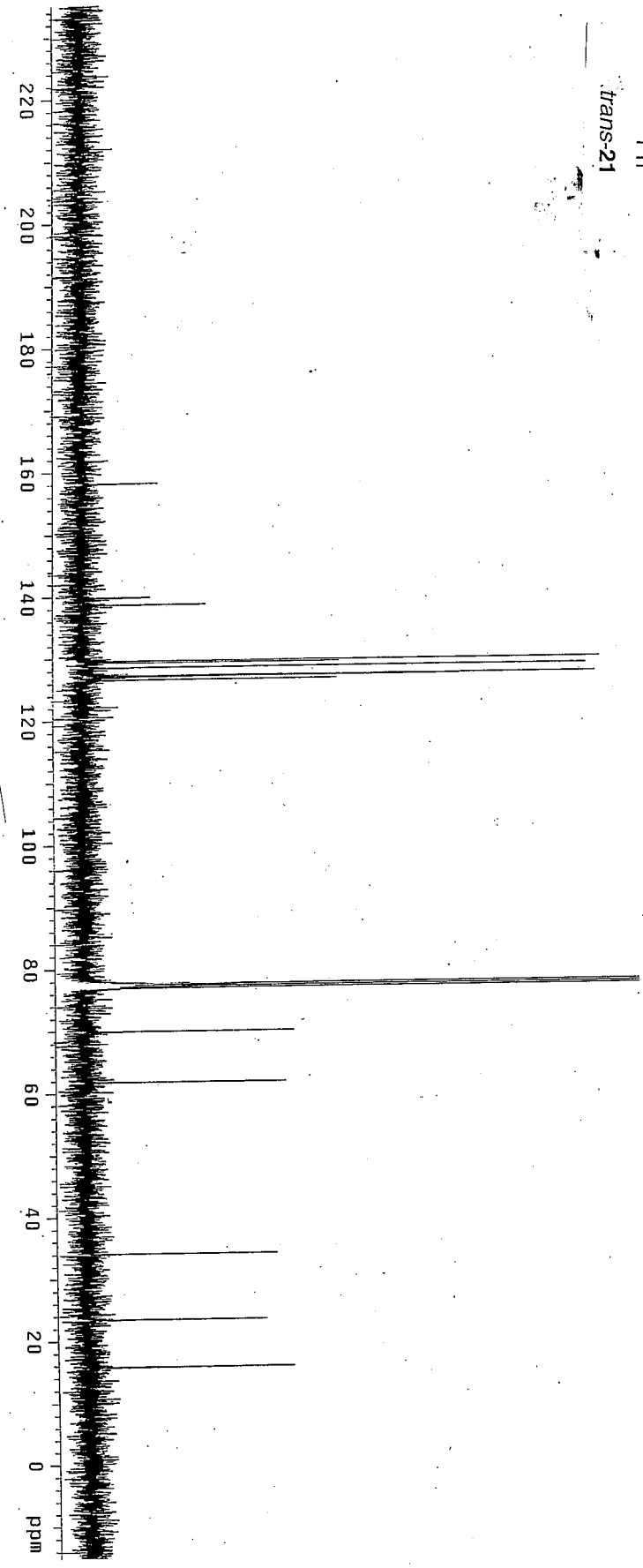








trans-**21**



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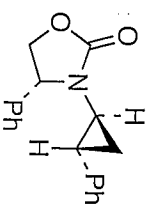

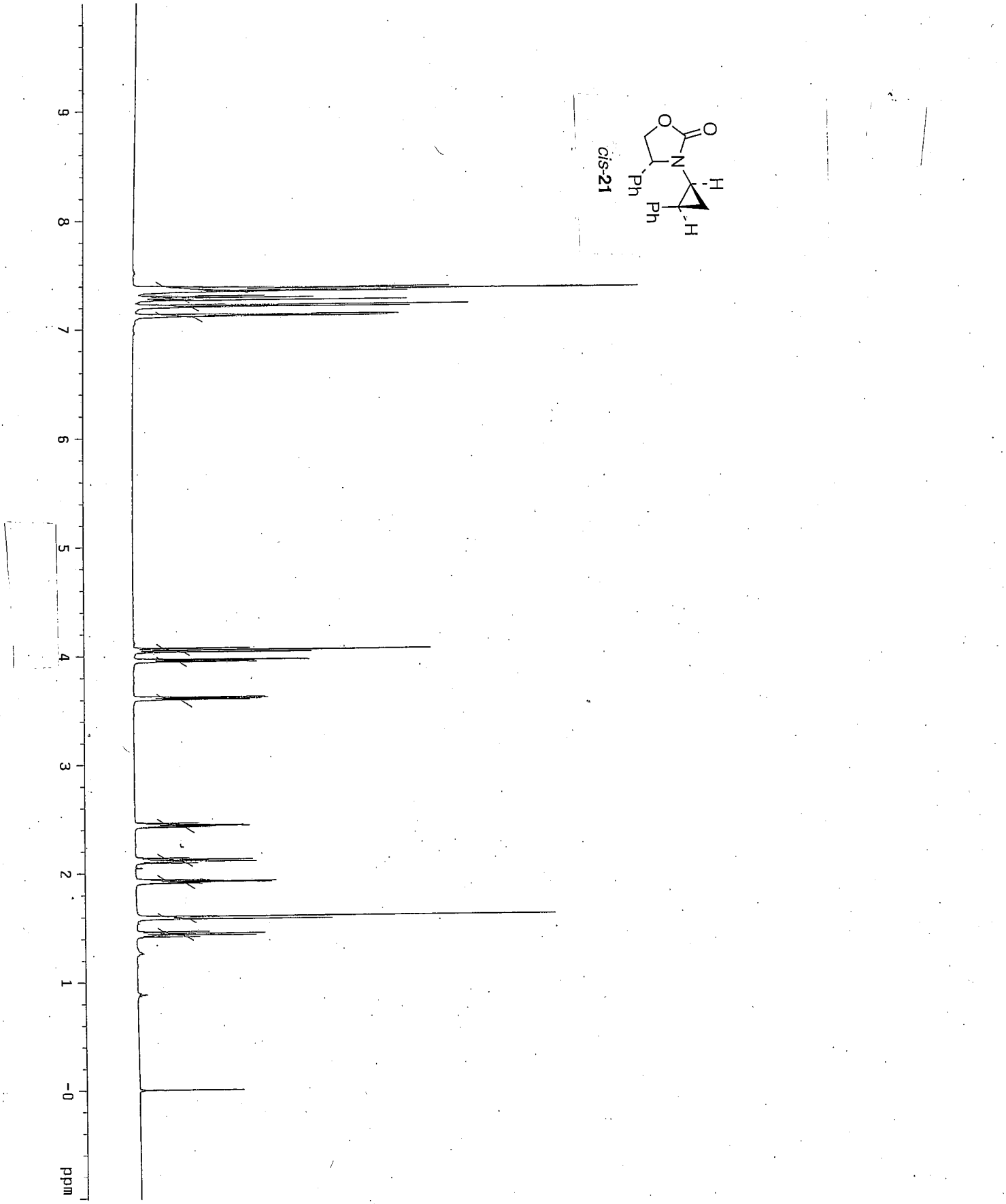

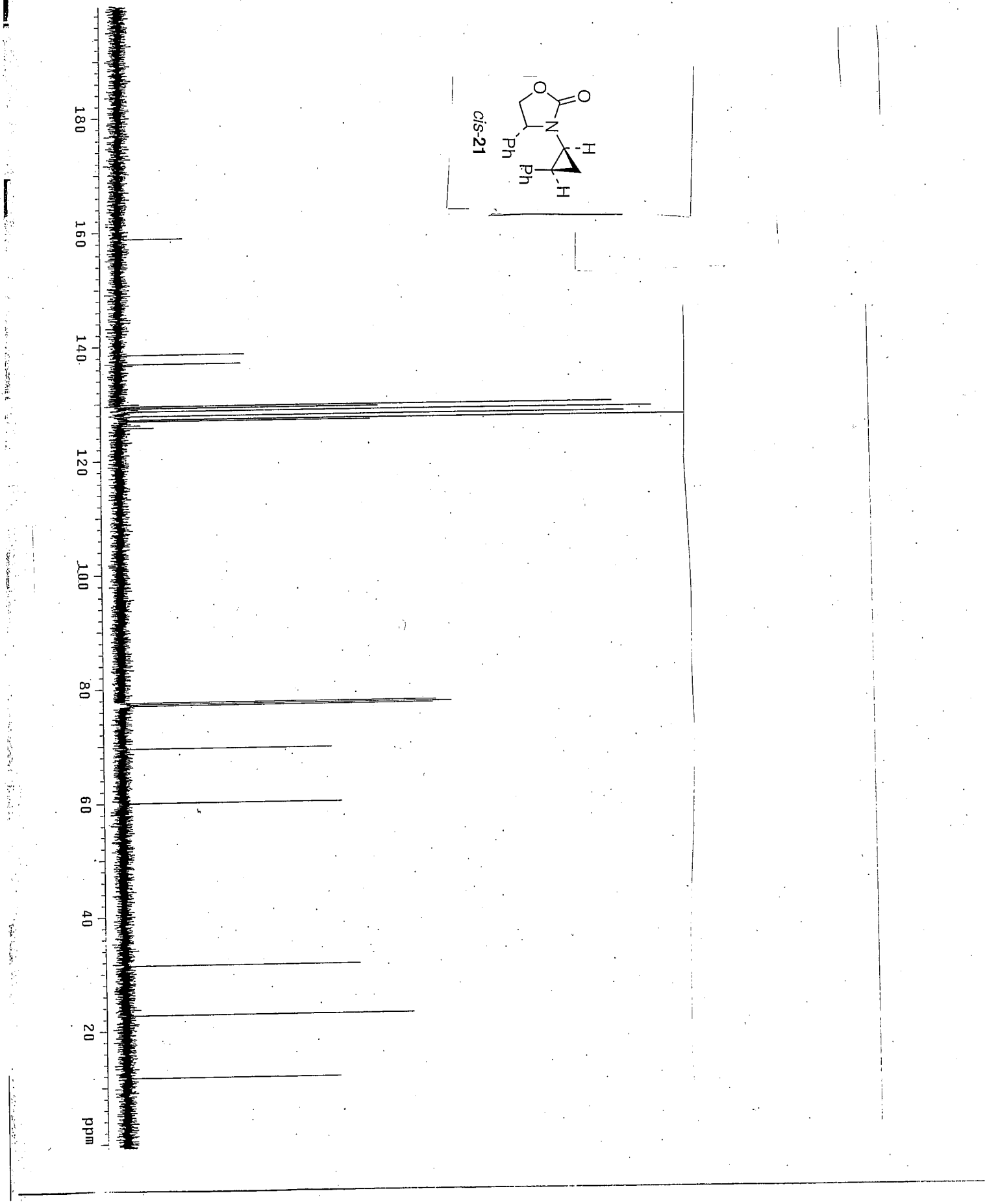

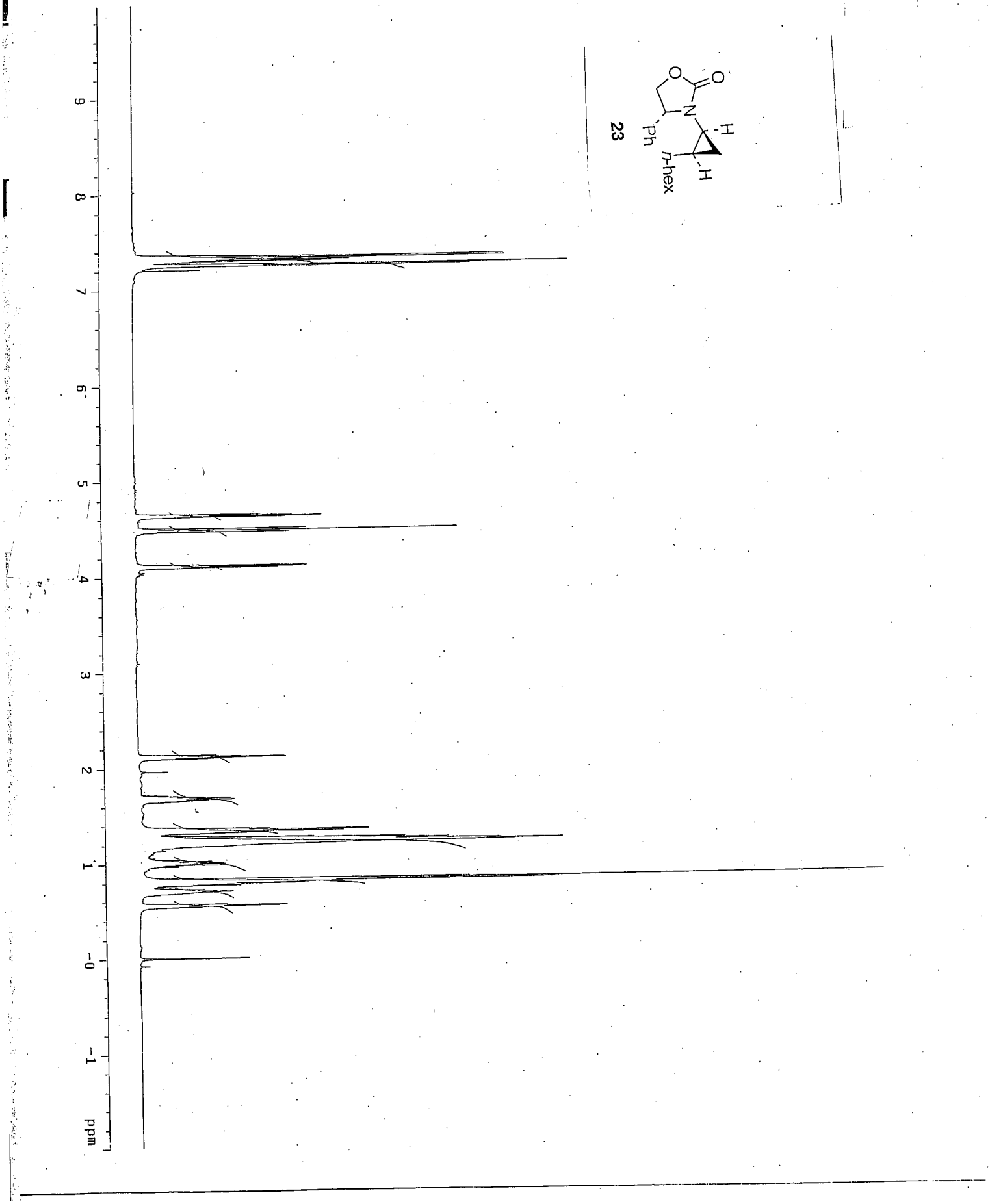
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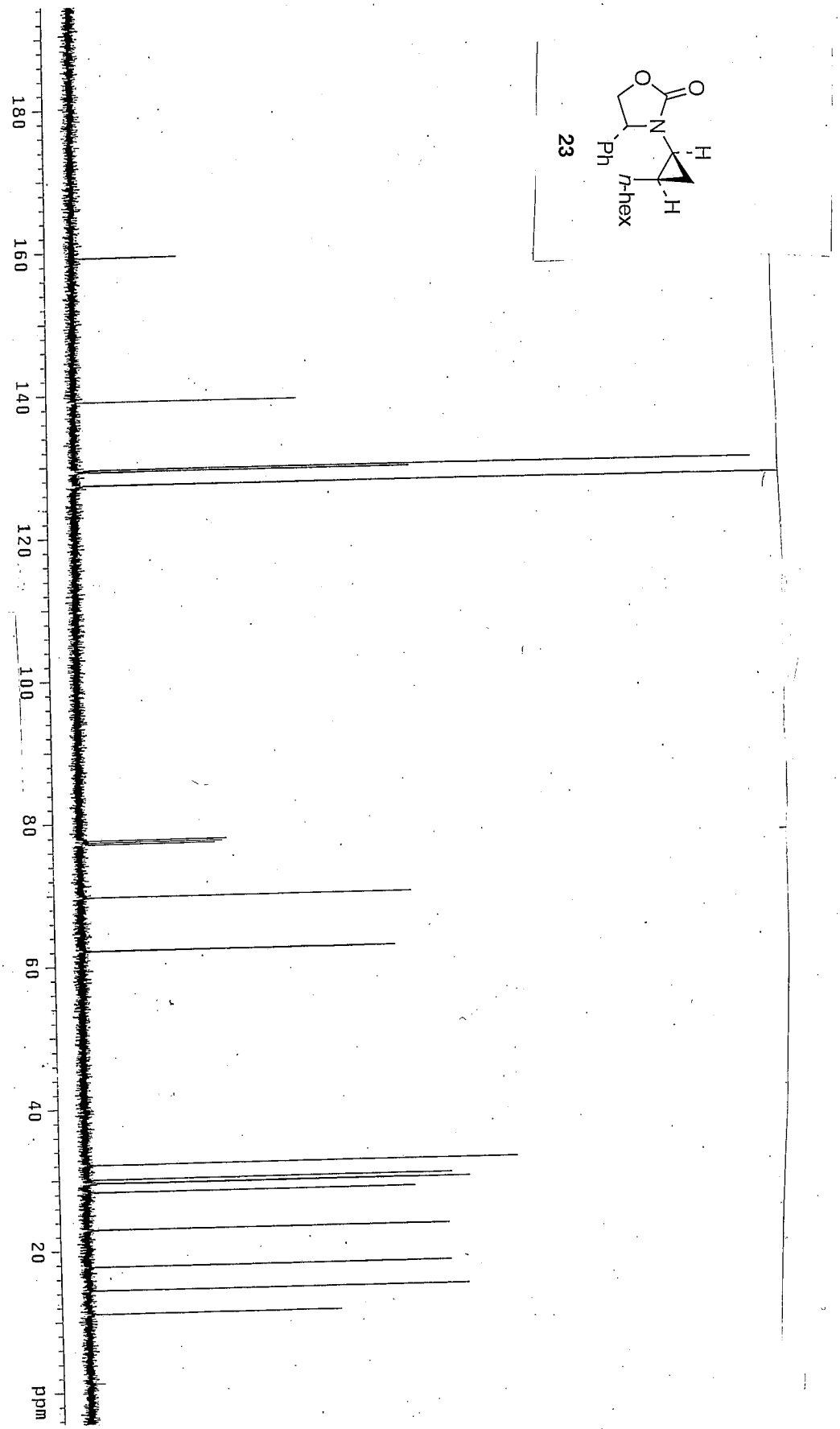
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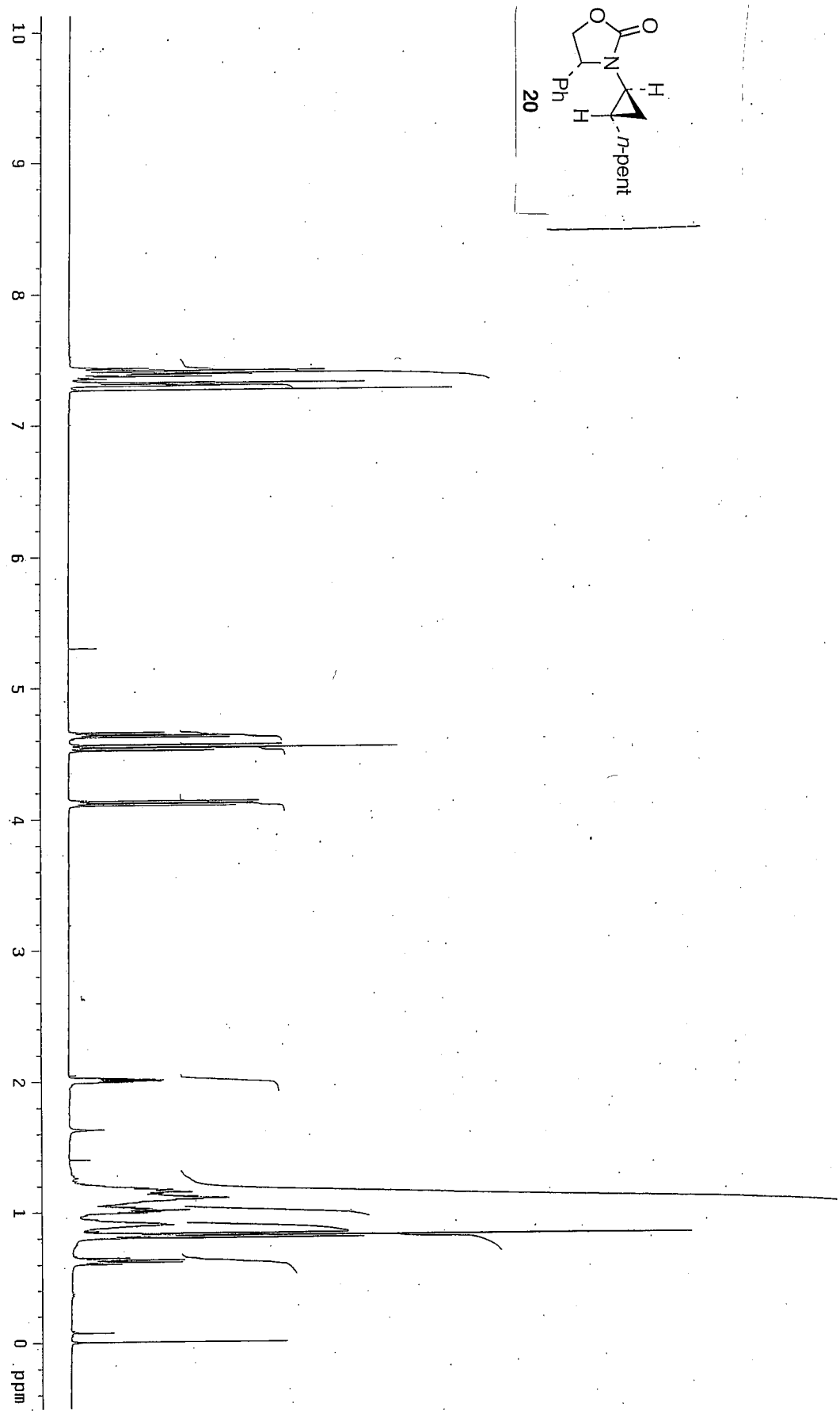

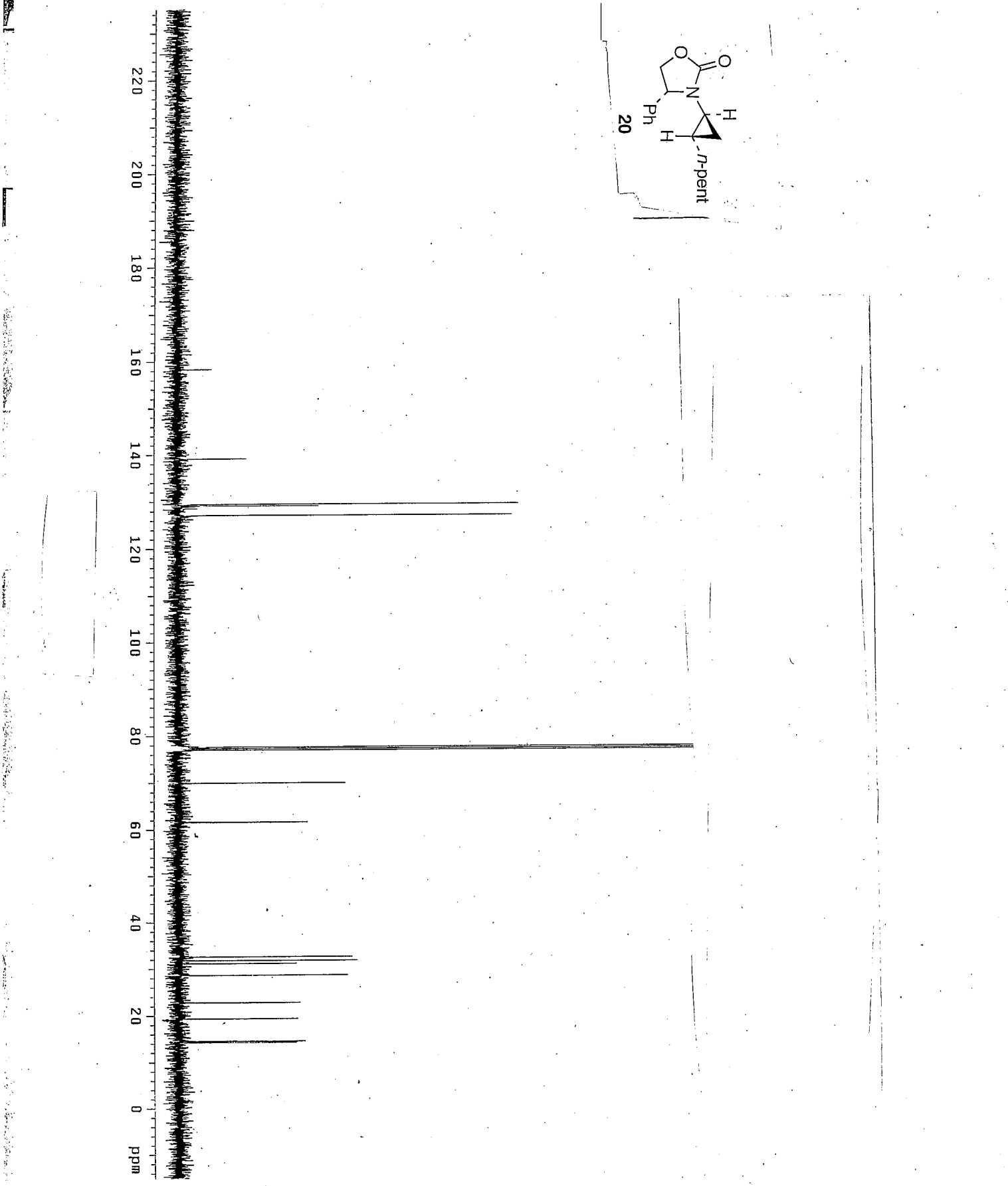
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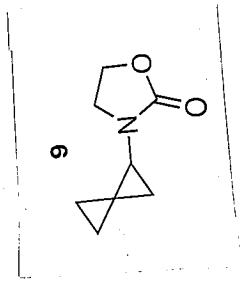

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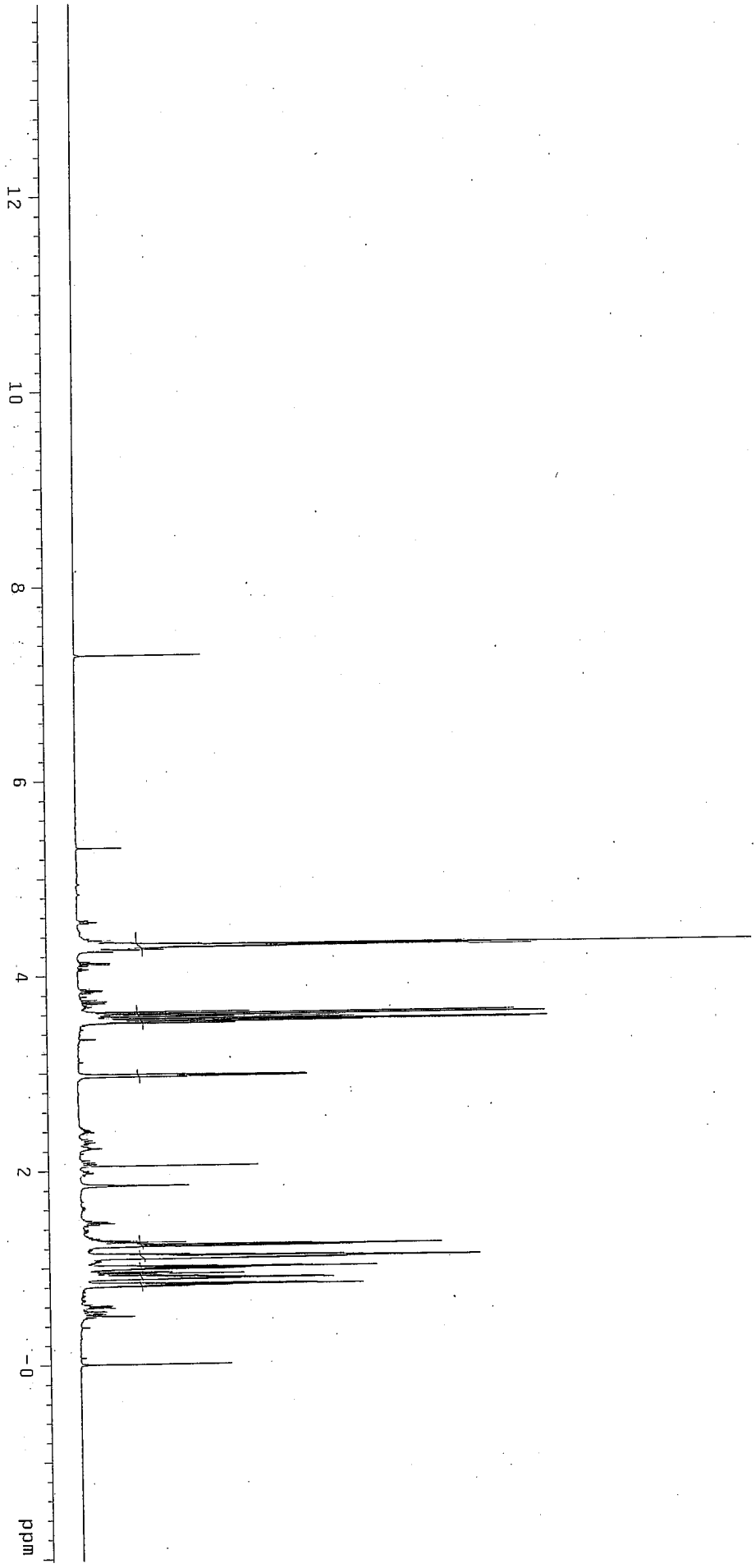

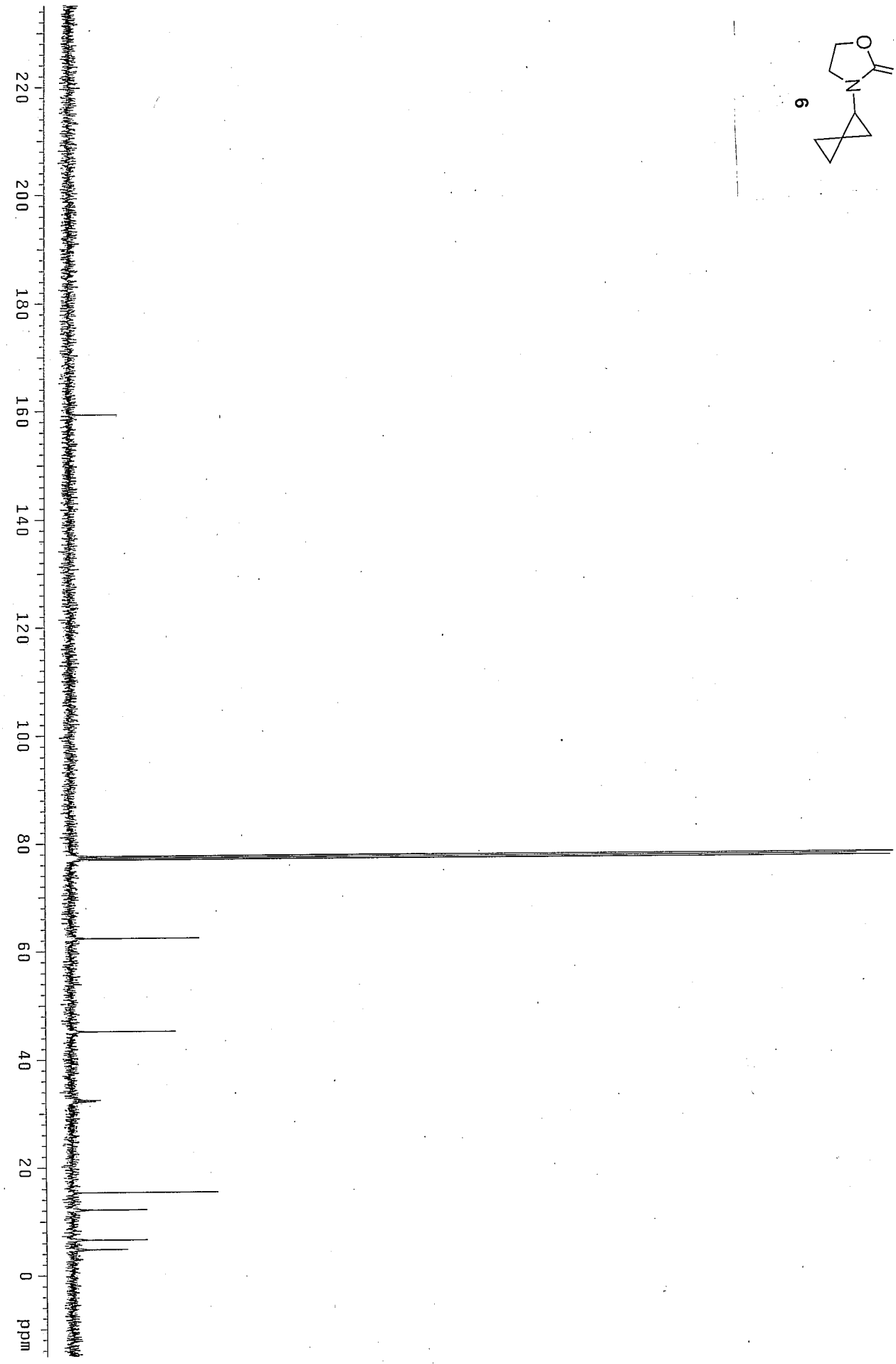

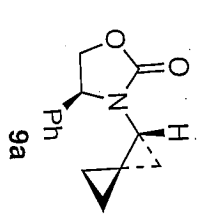

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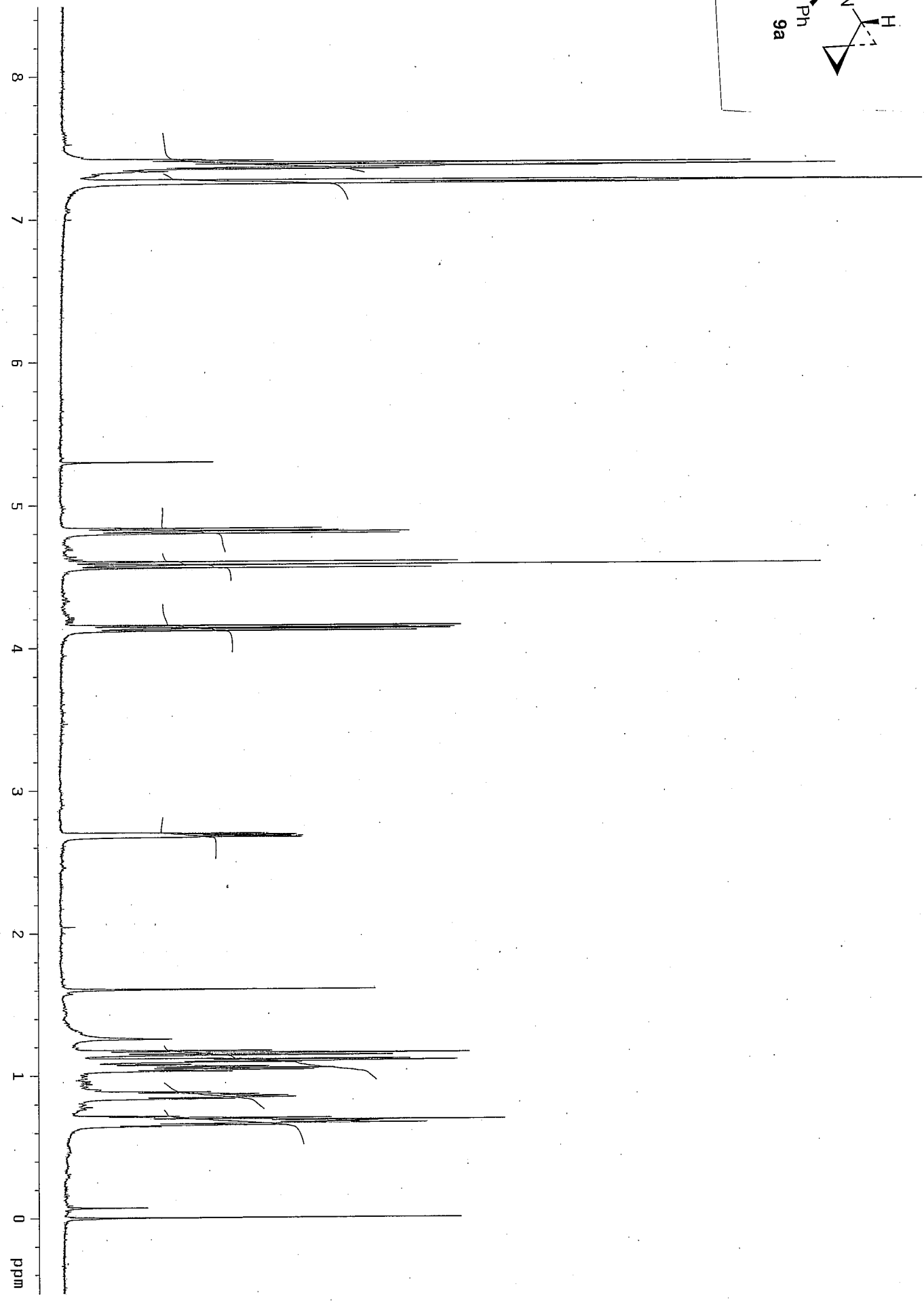

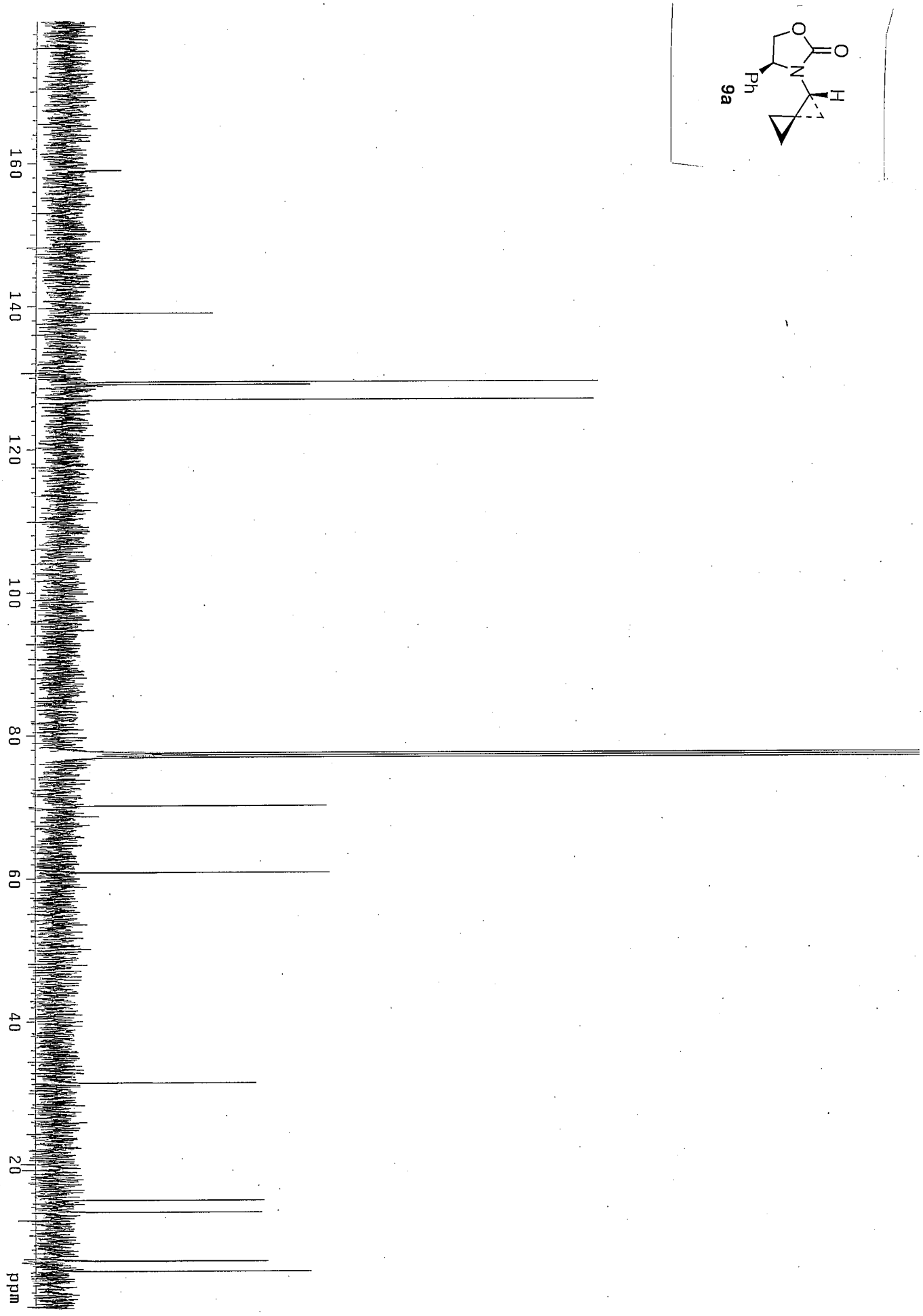

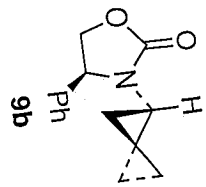

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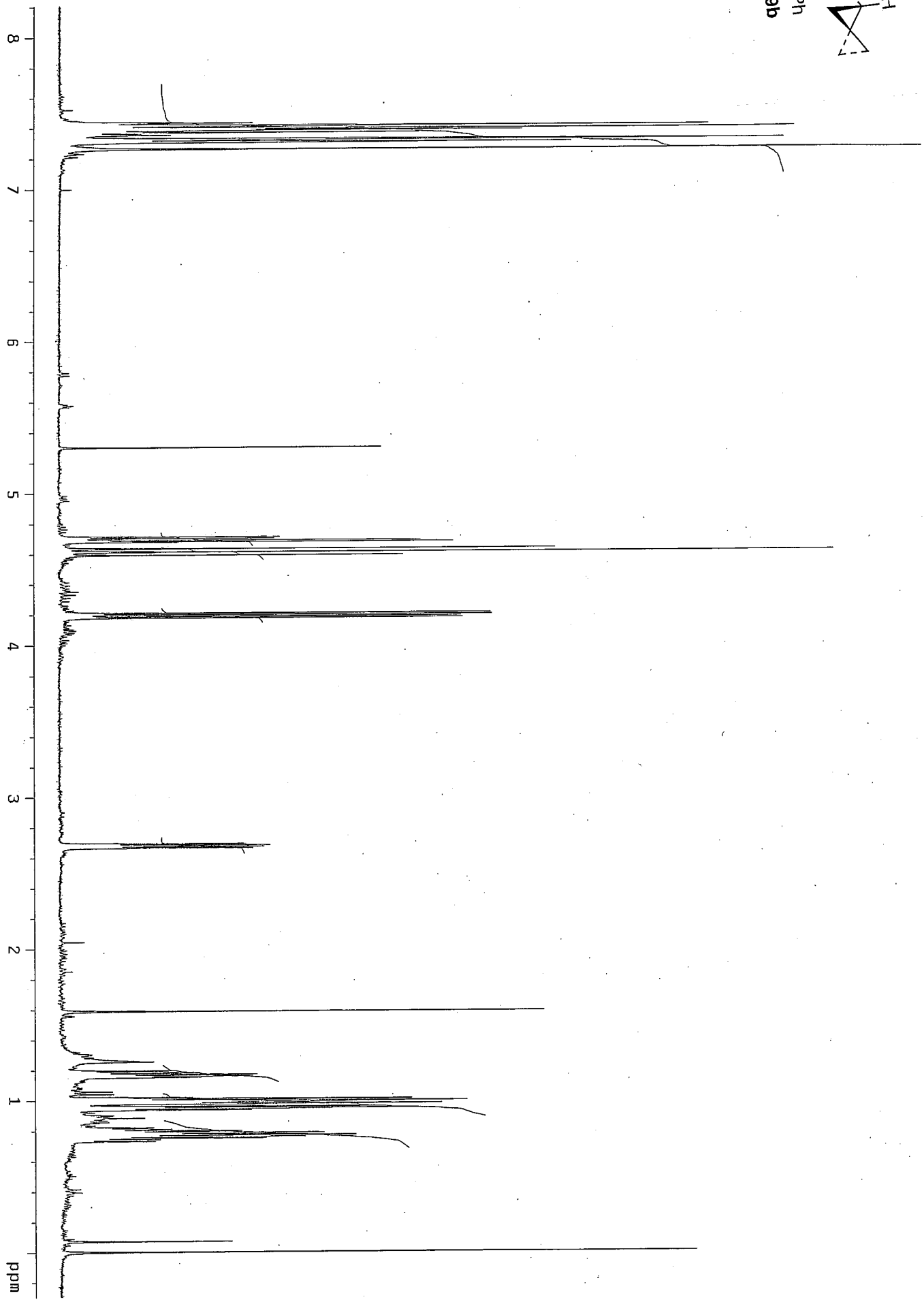

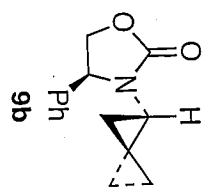

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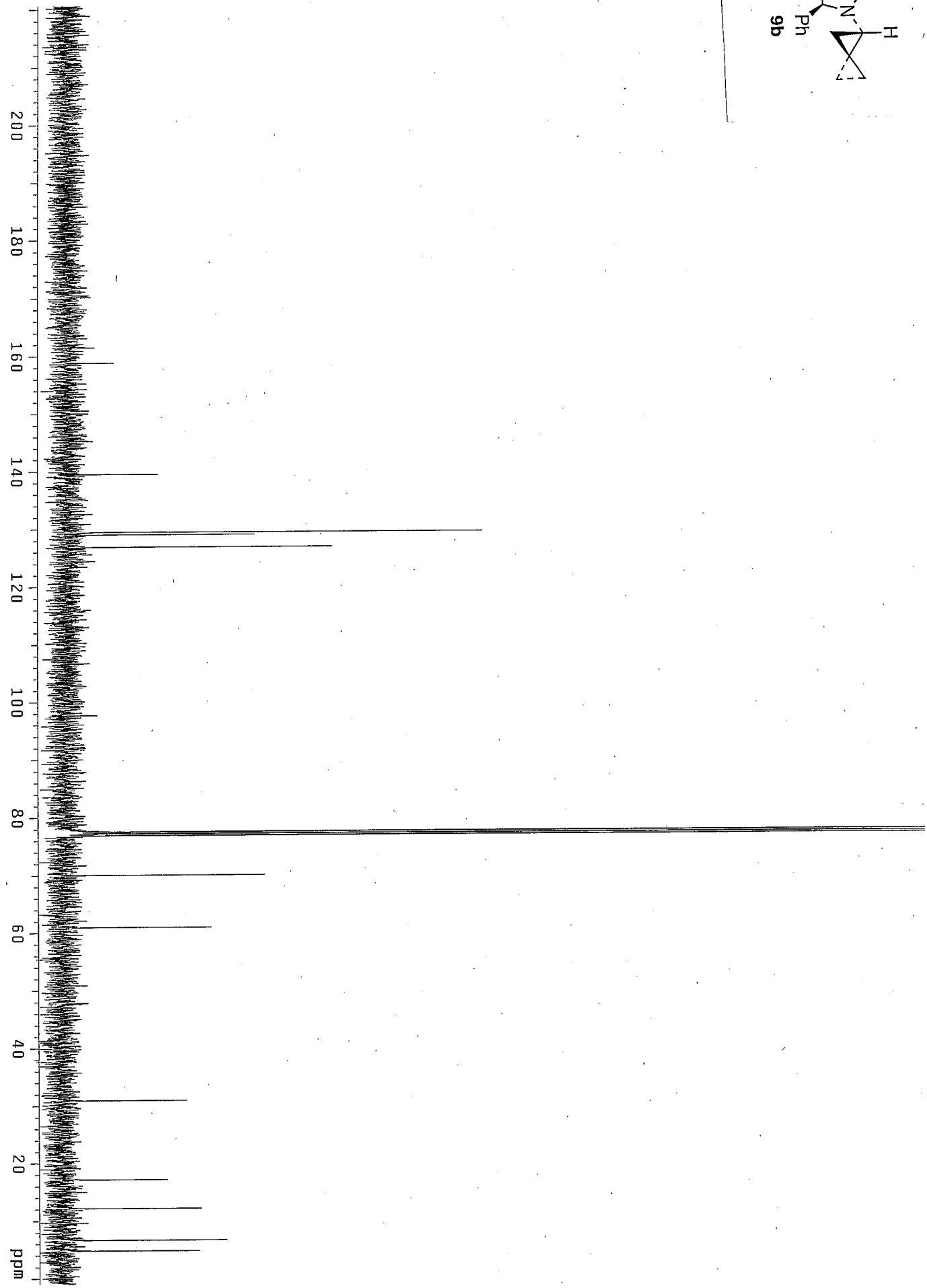

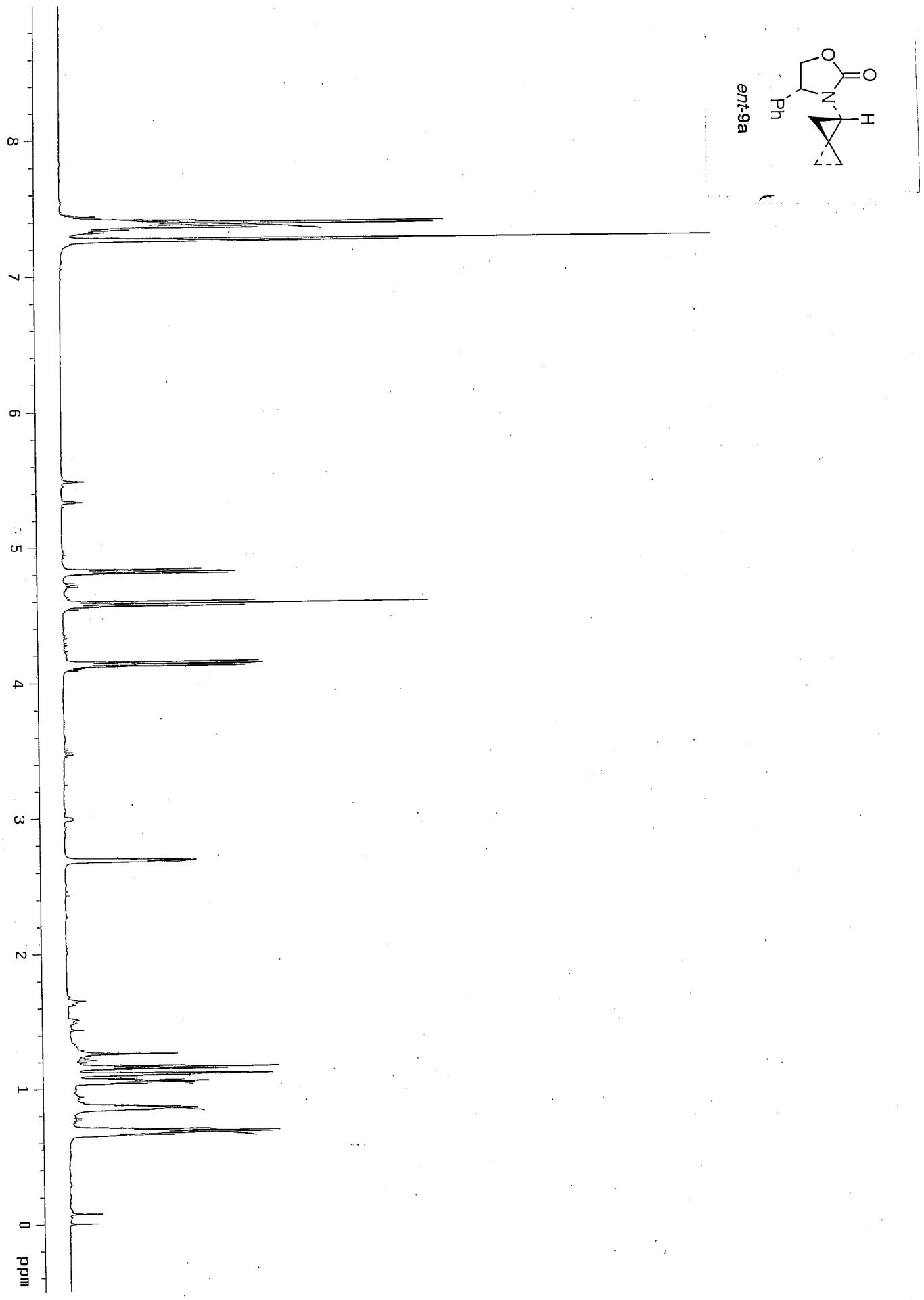

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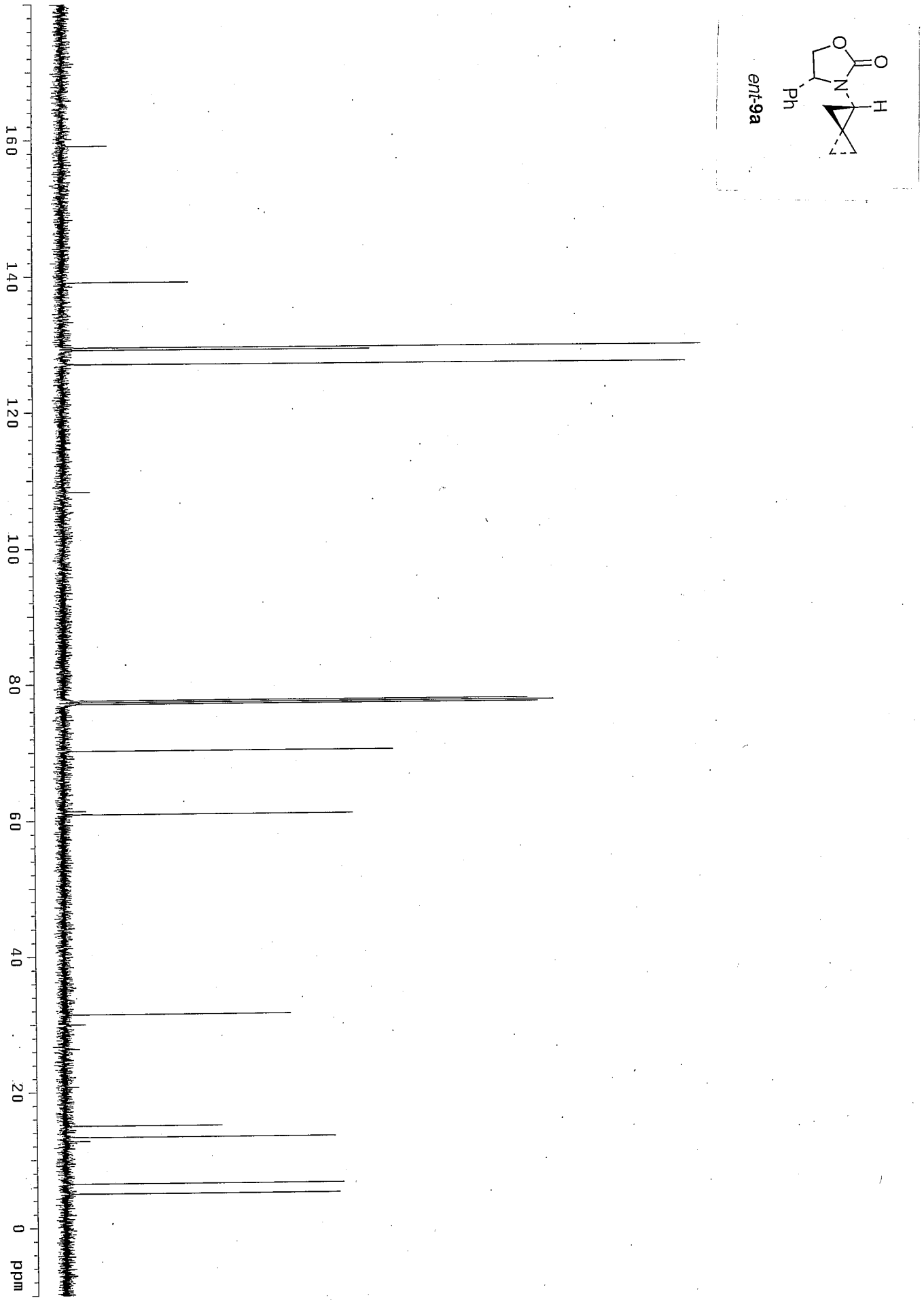

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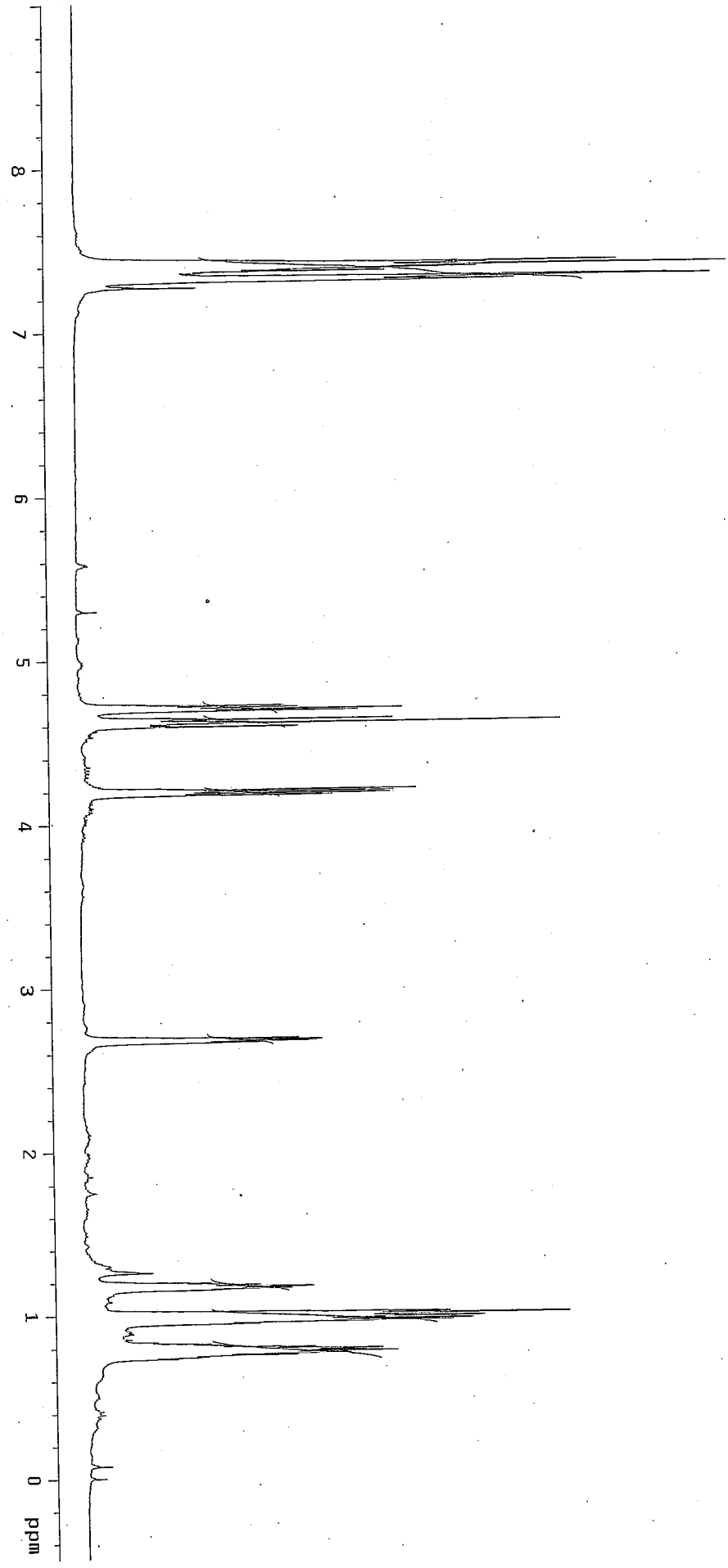

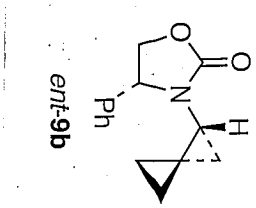

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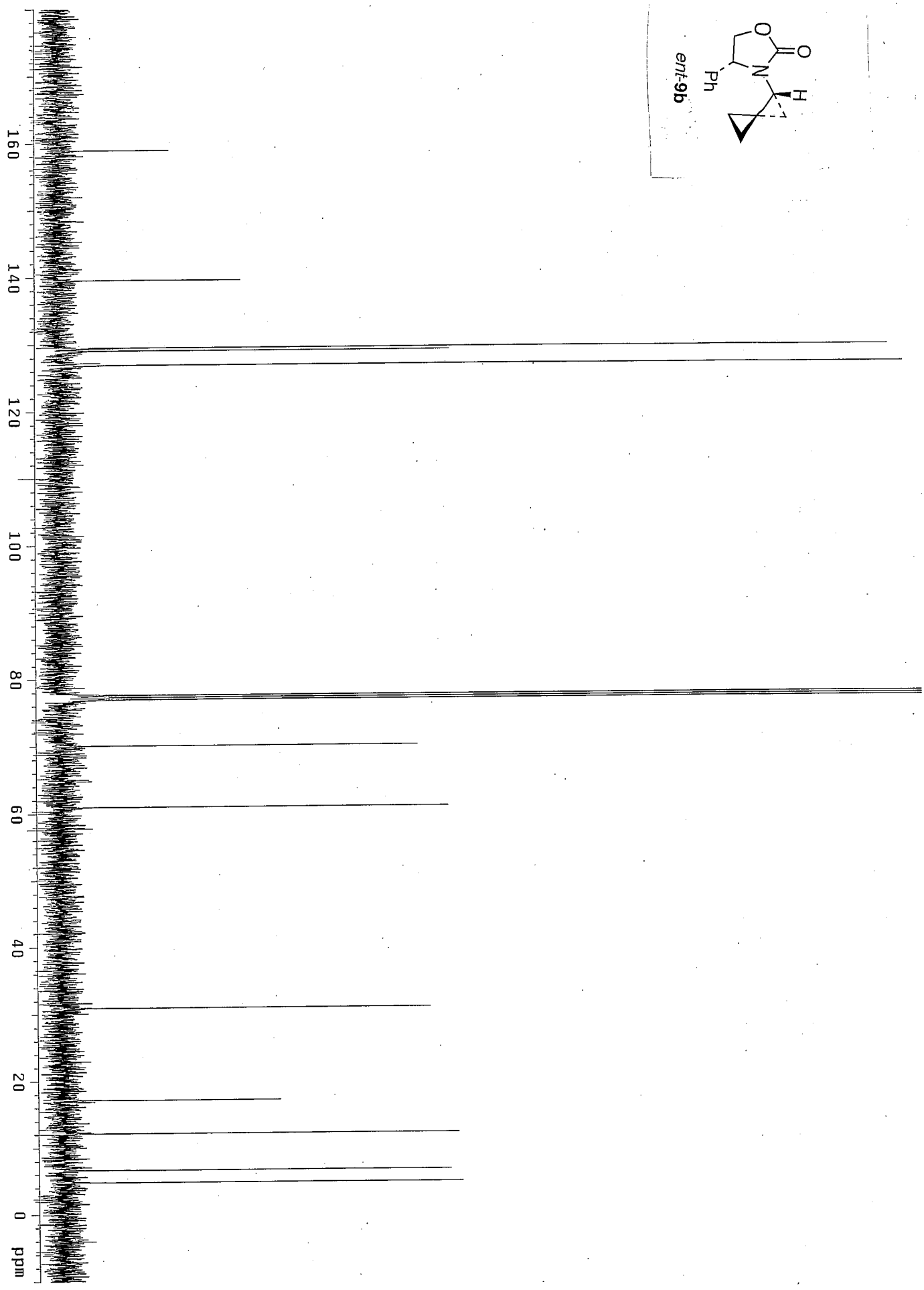
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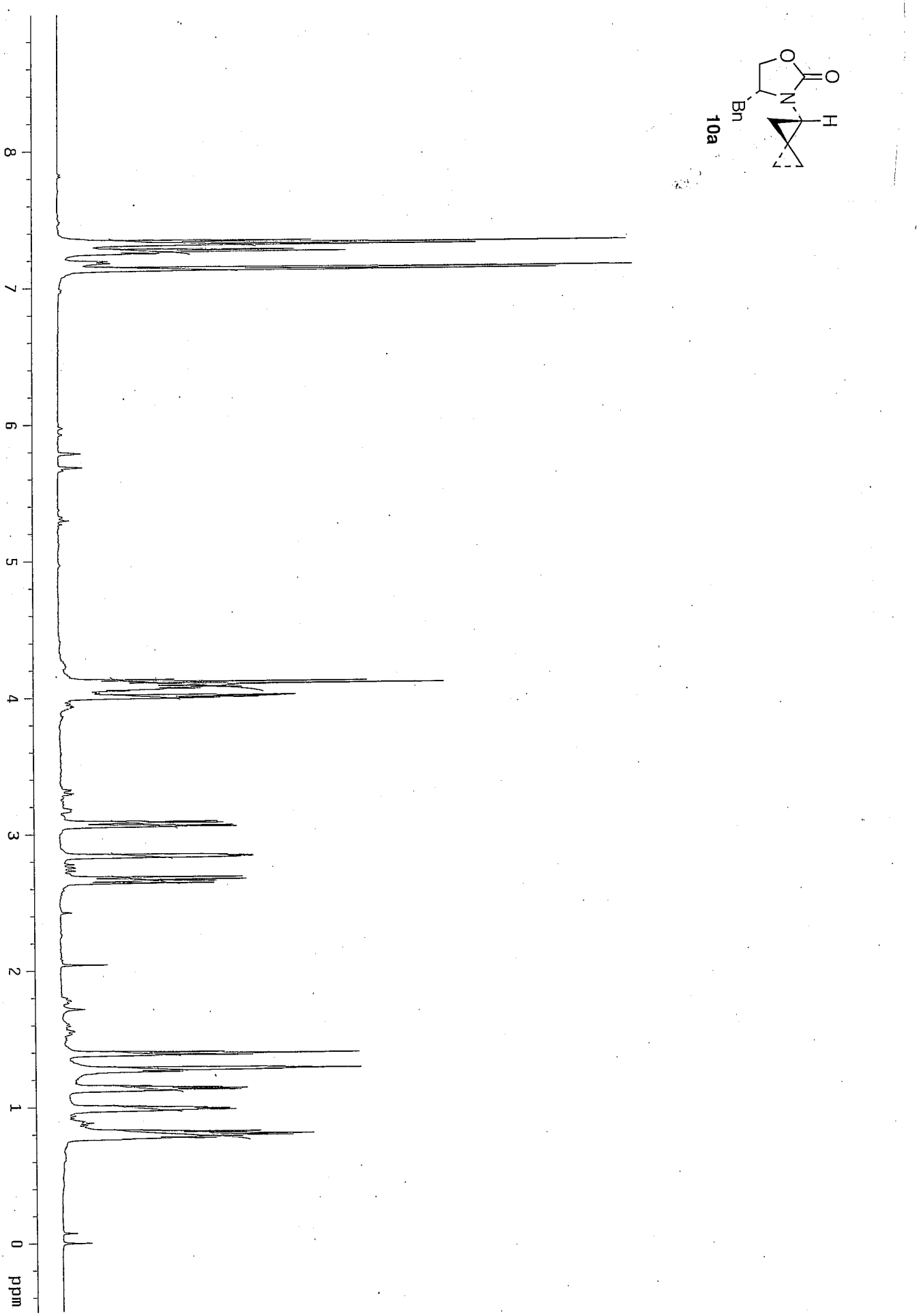

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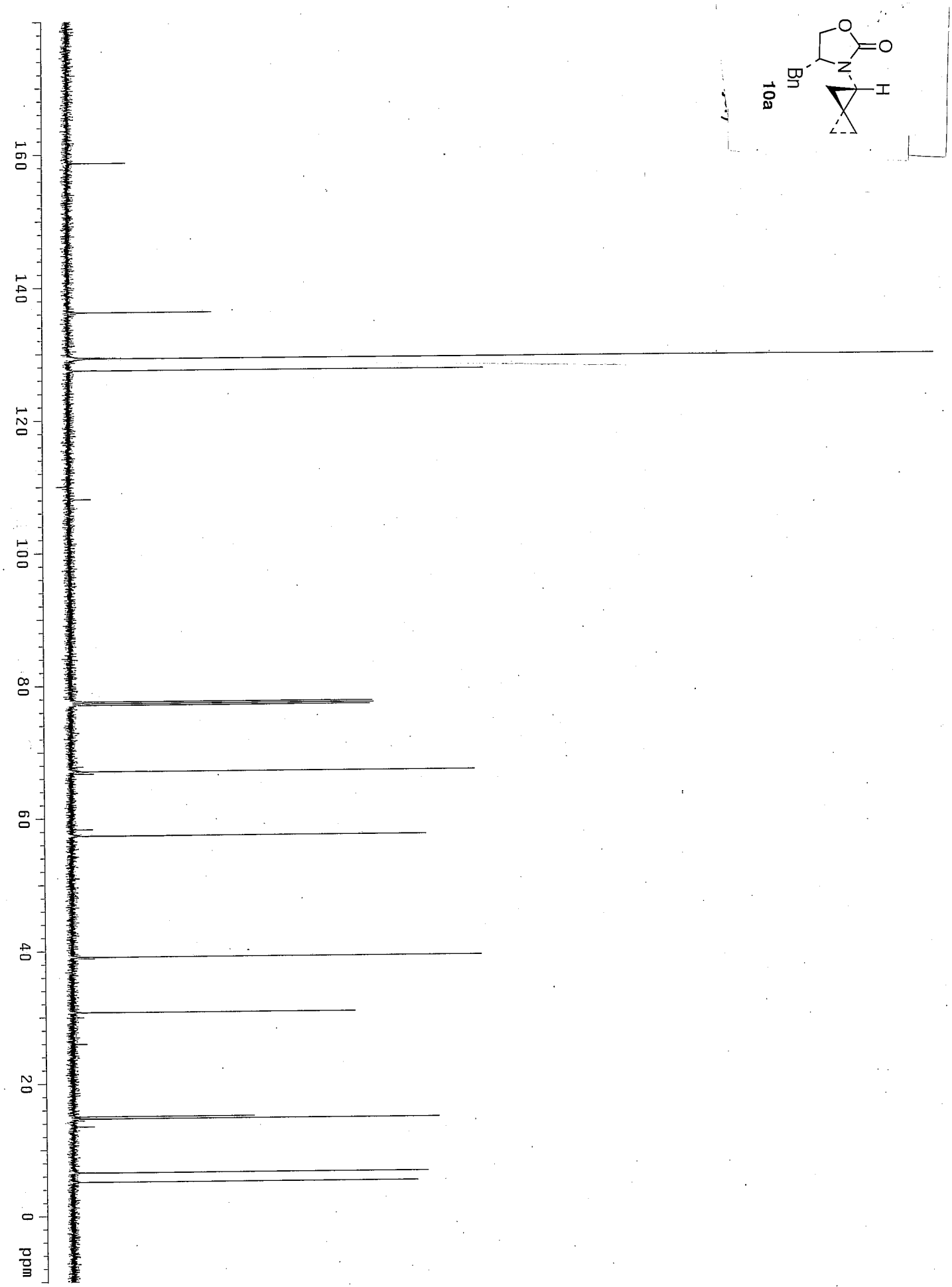
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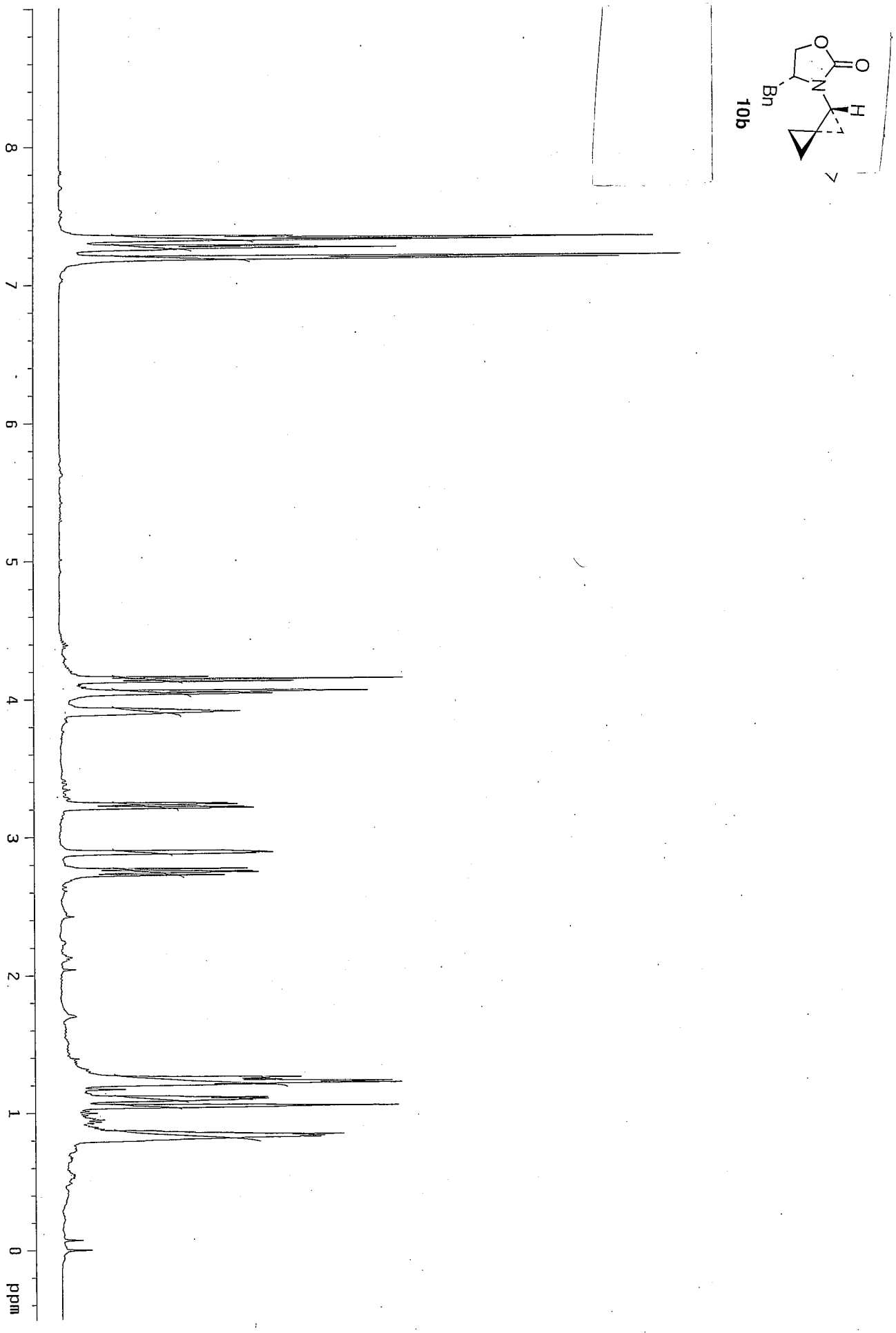
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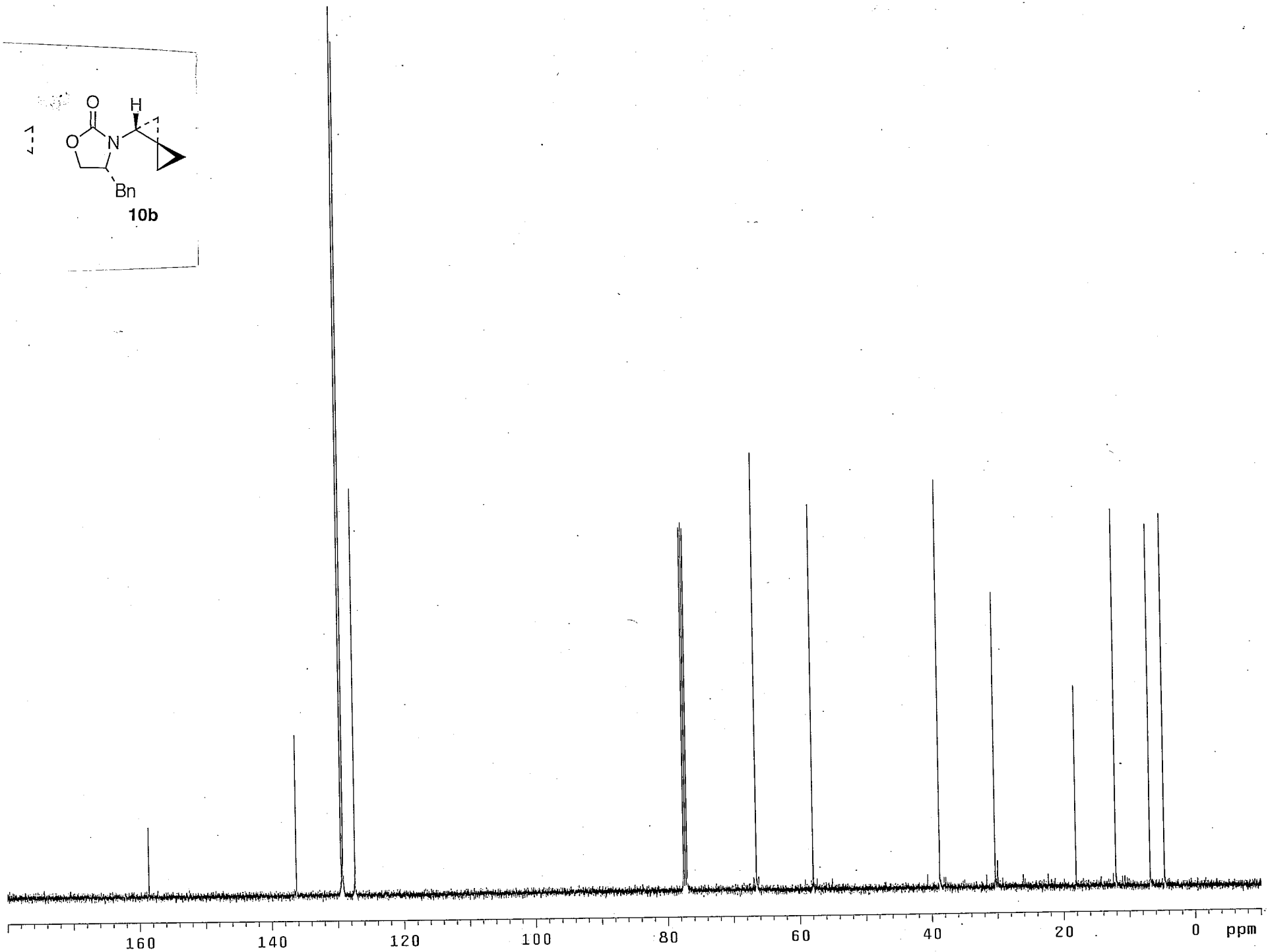
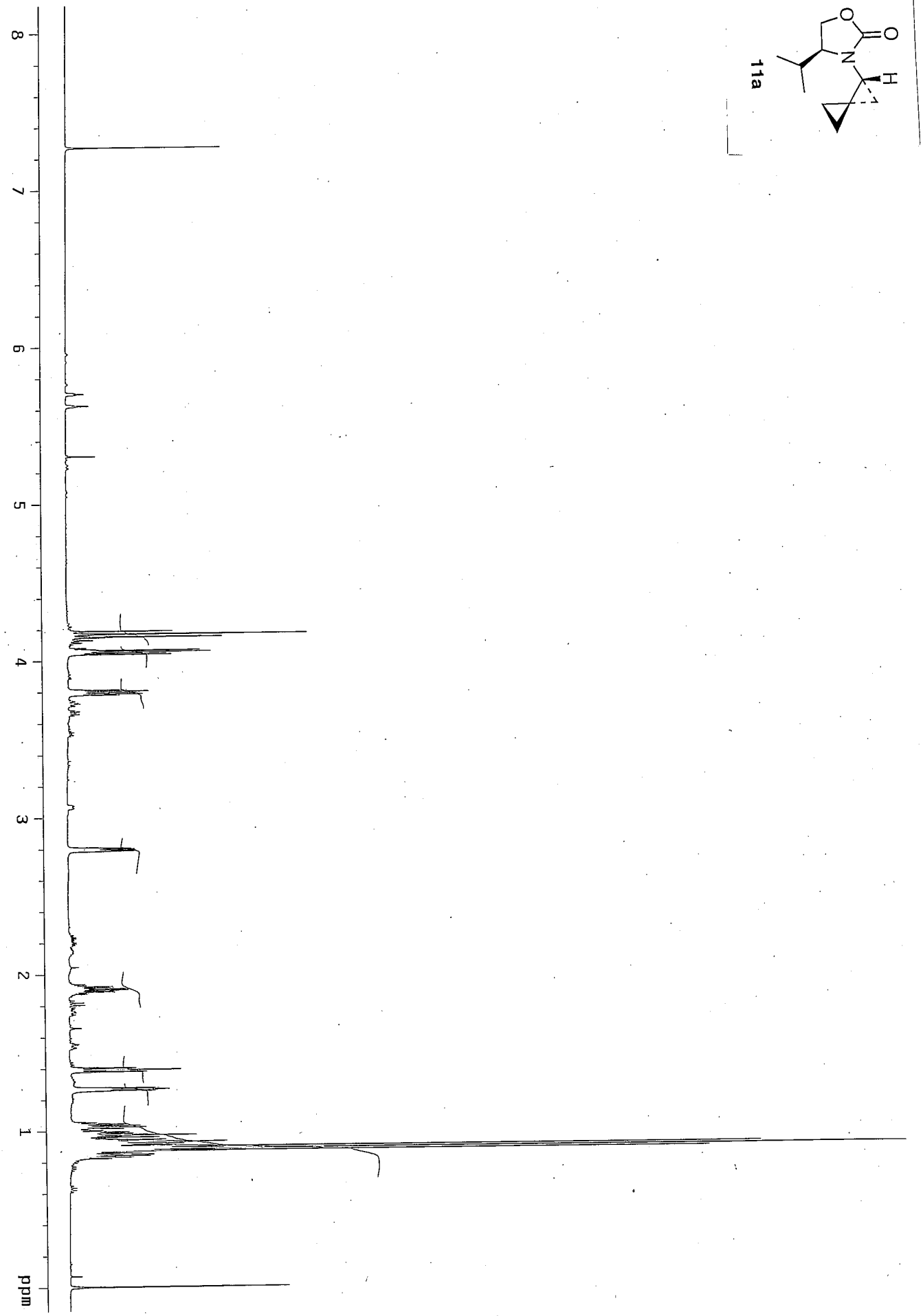
10b

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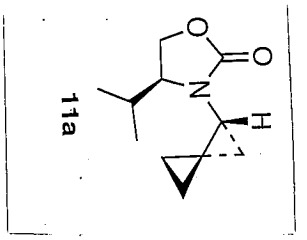

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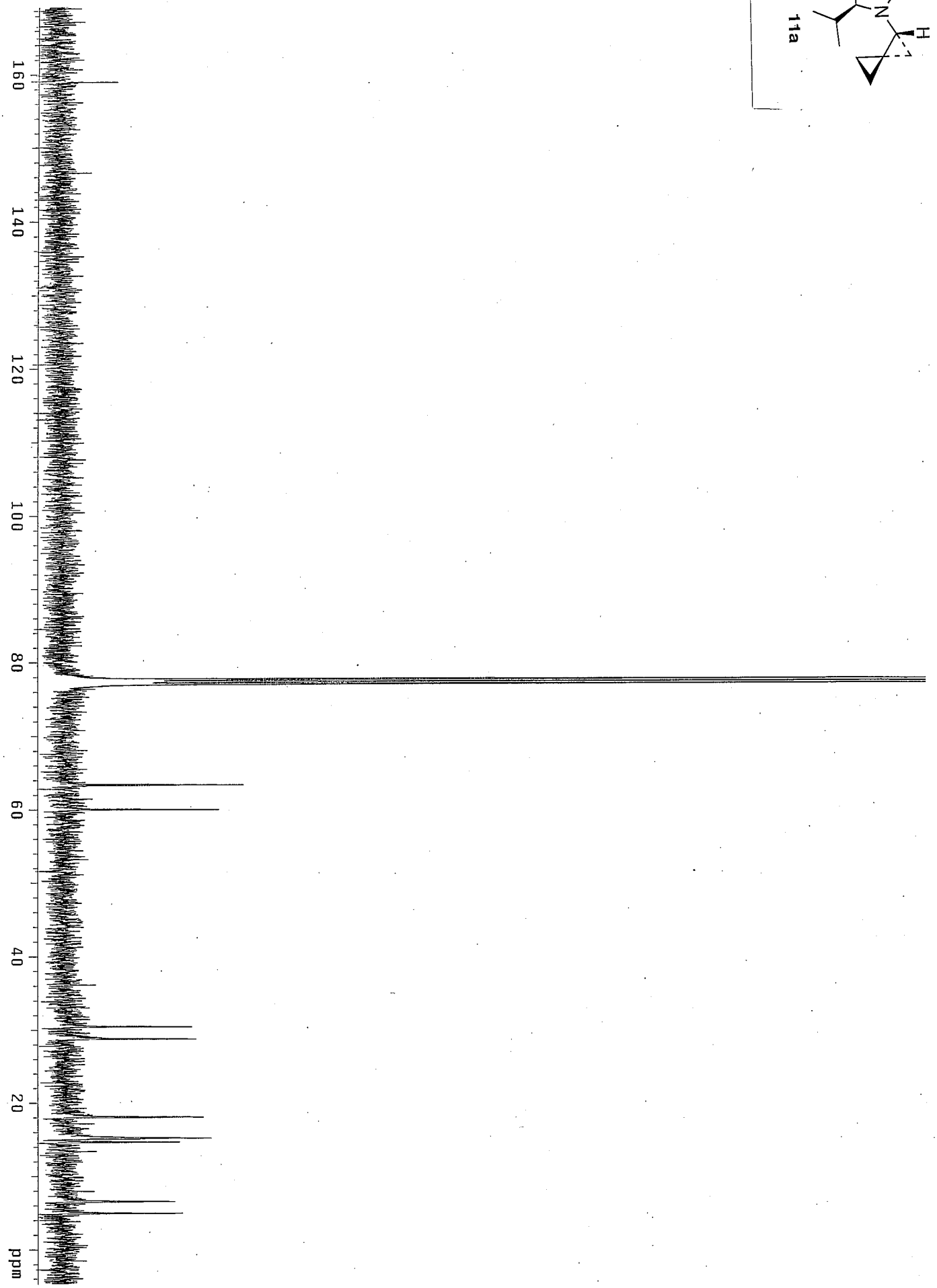

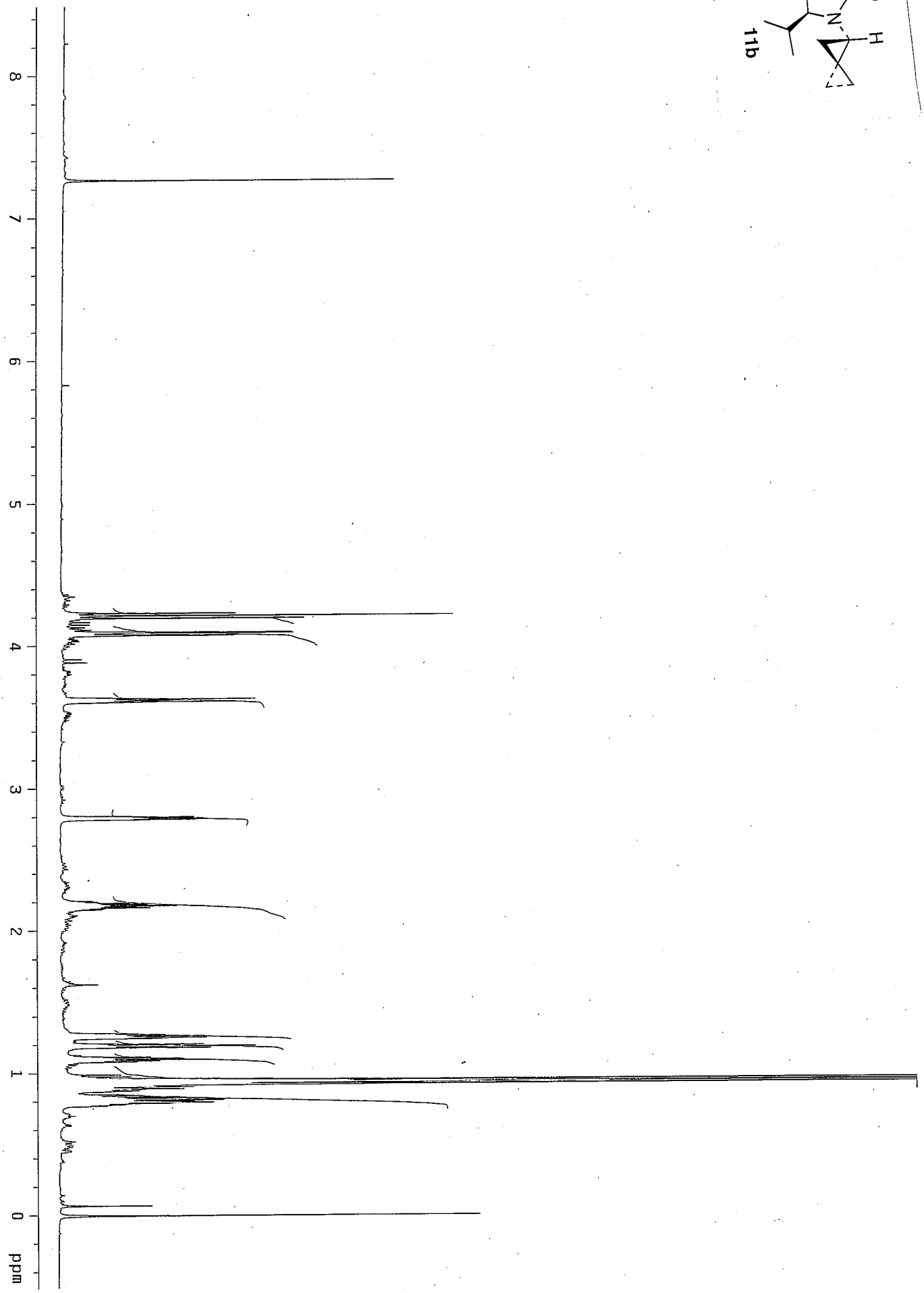
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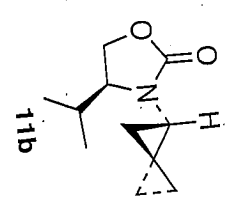

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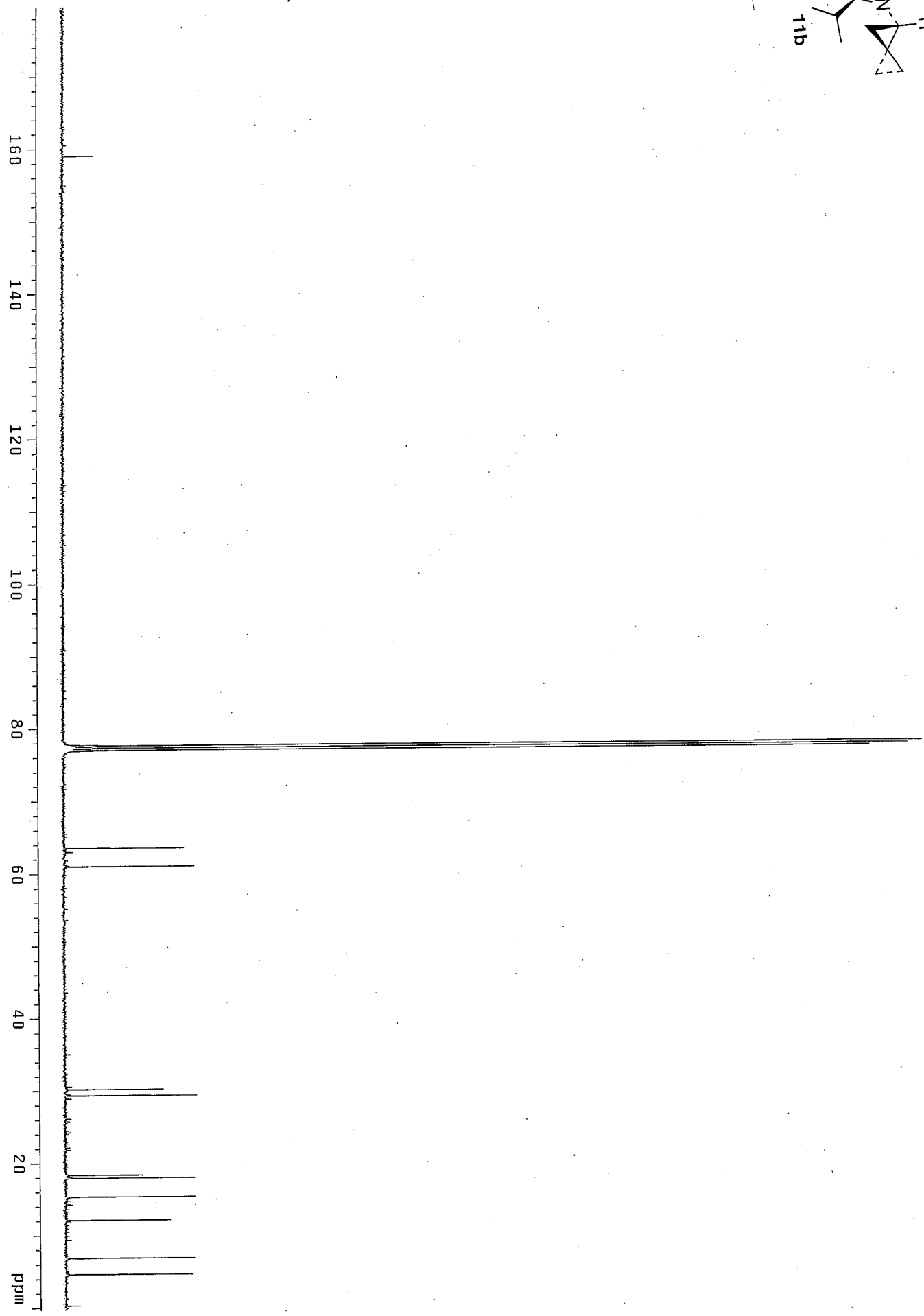

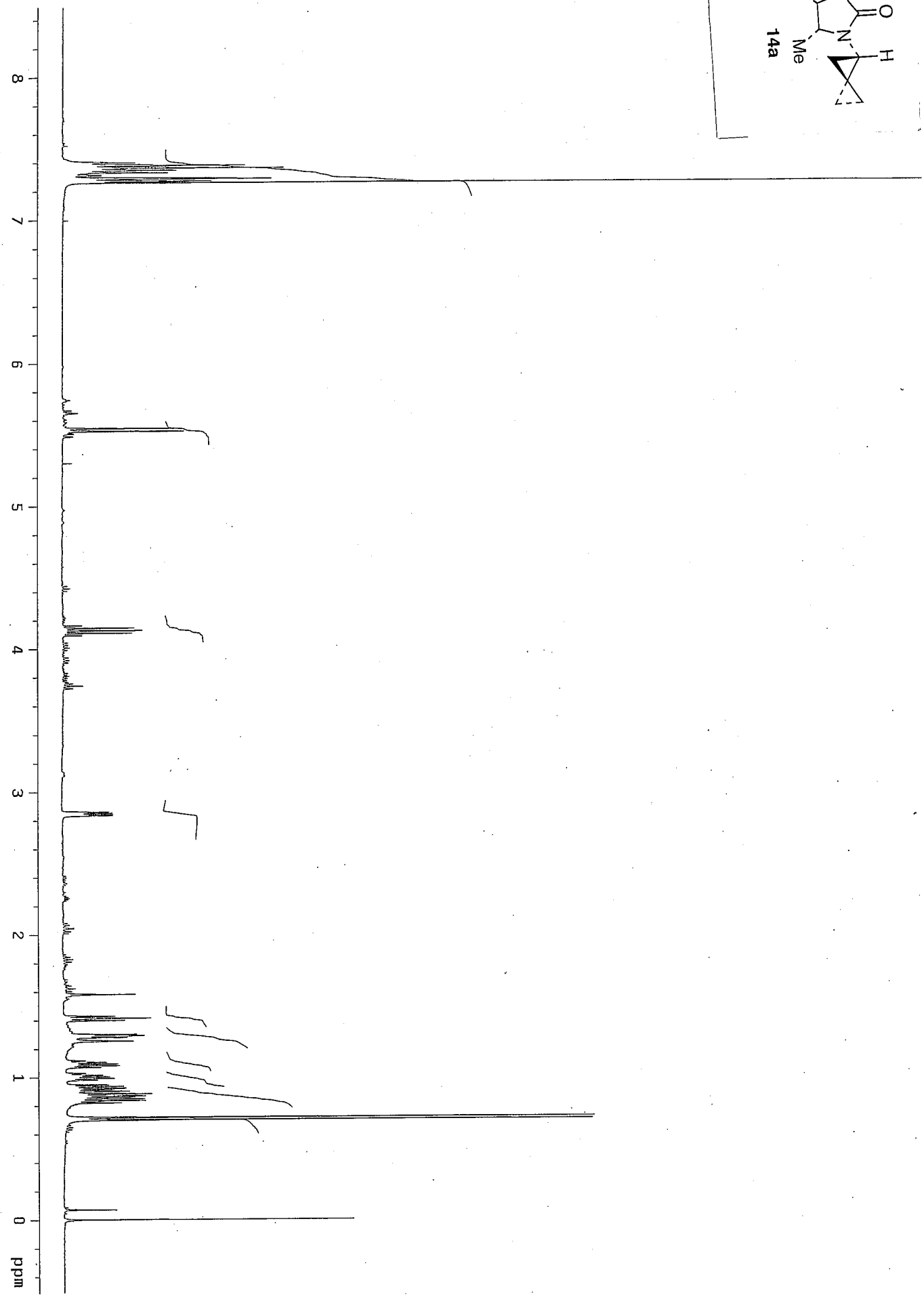
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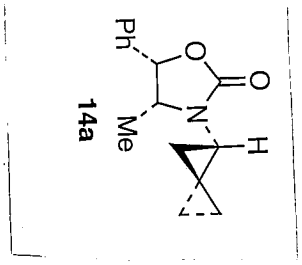

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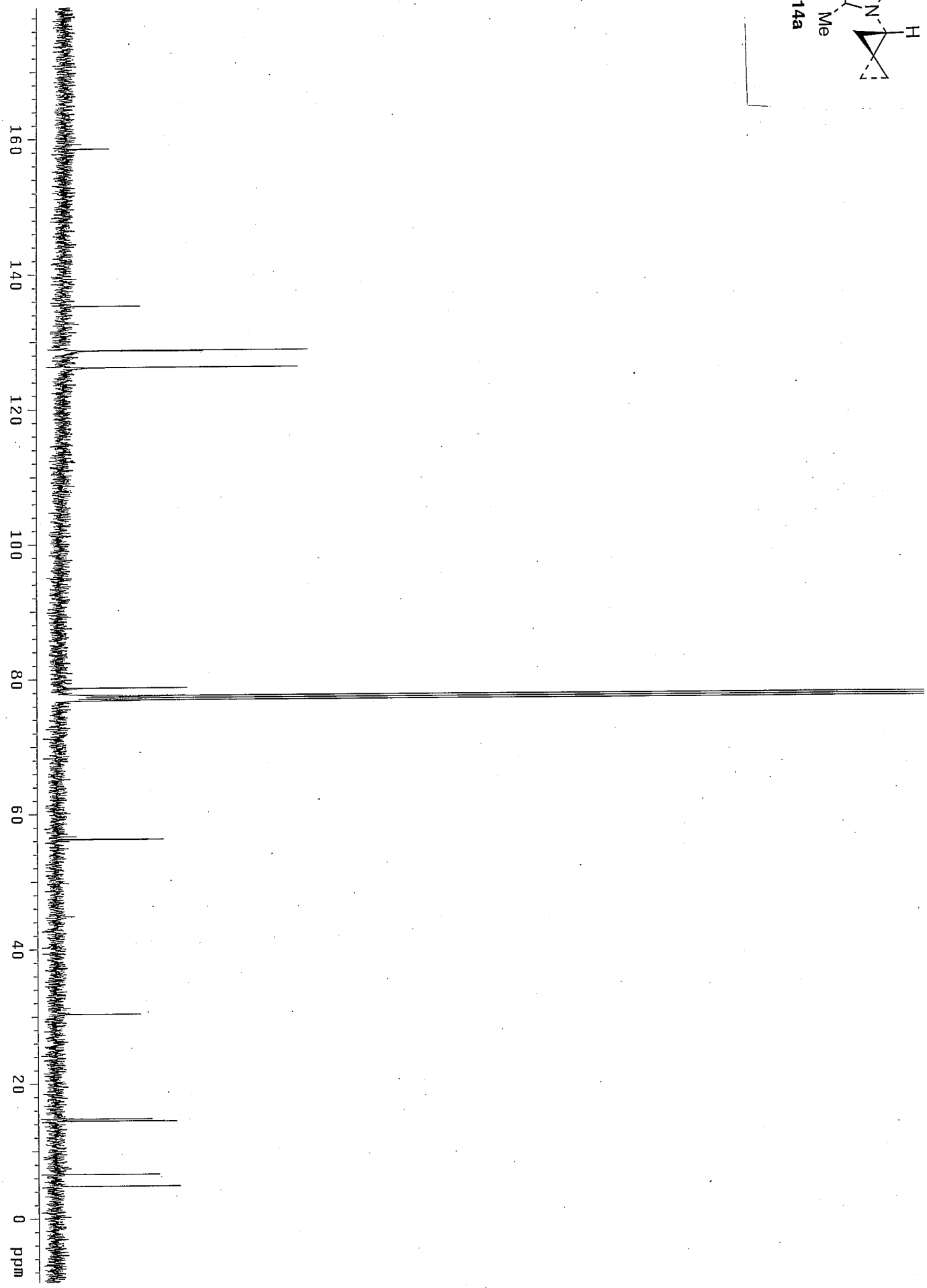

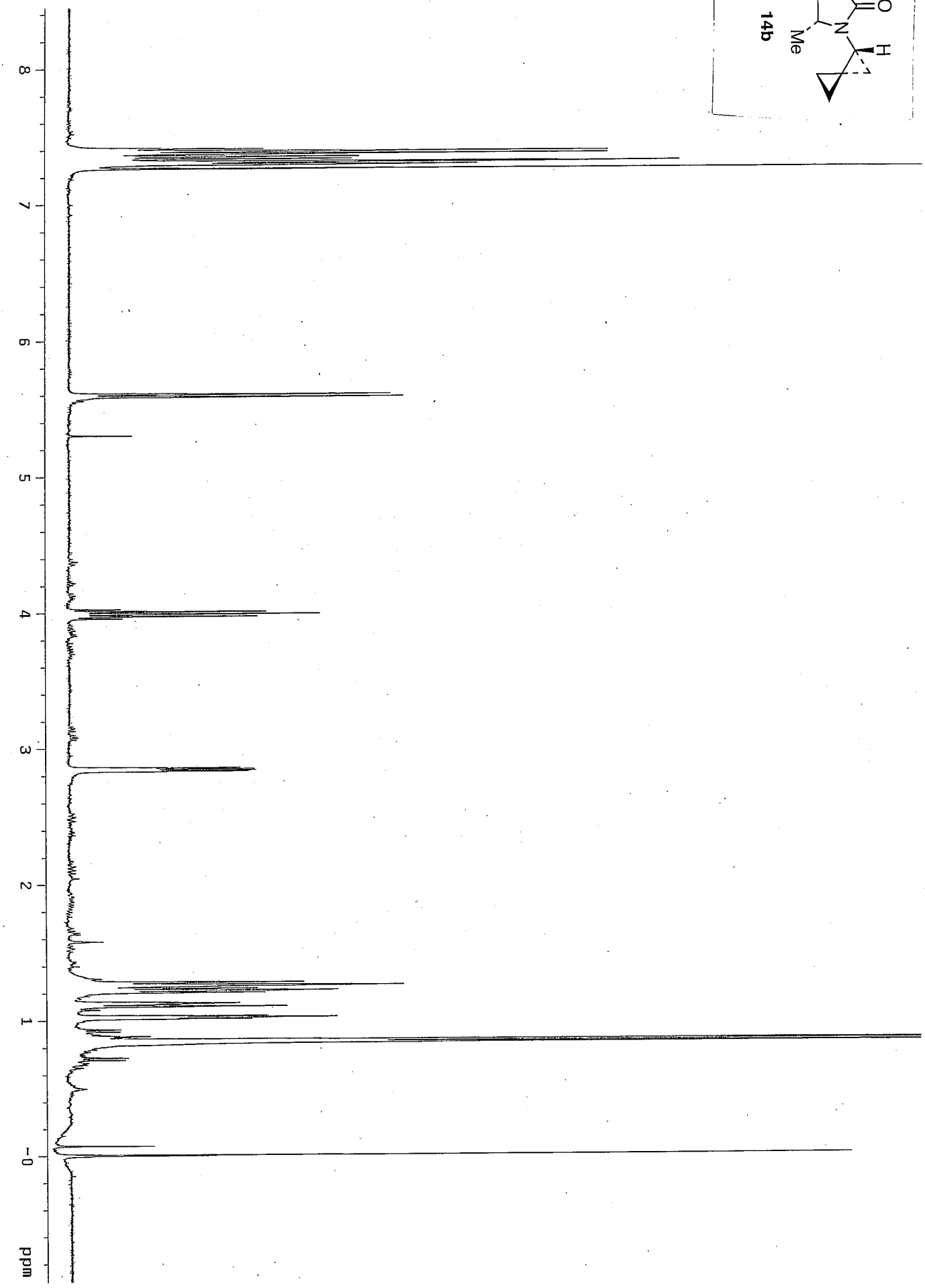
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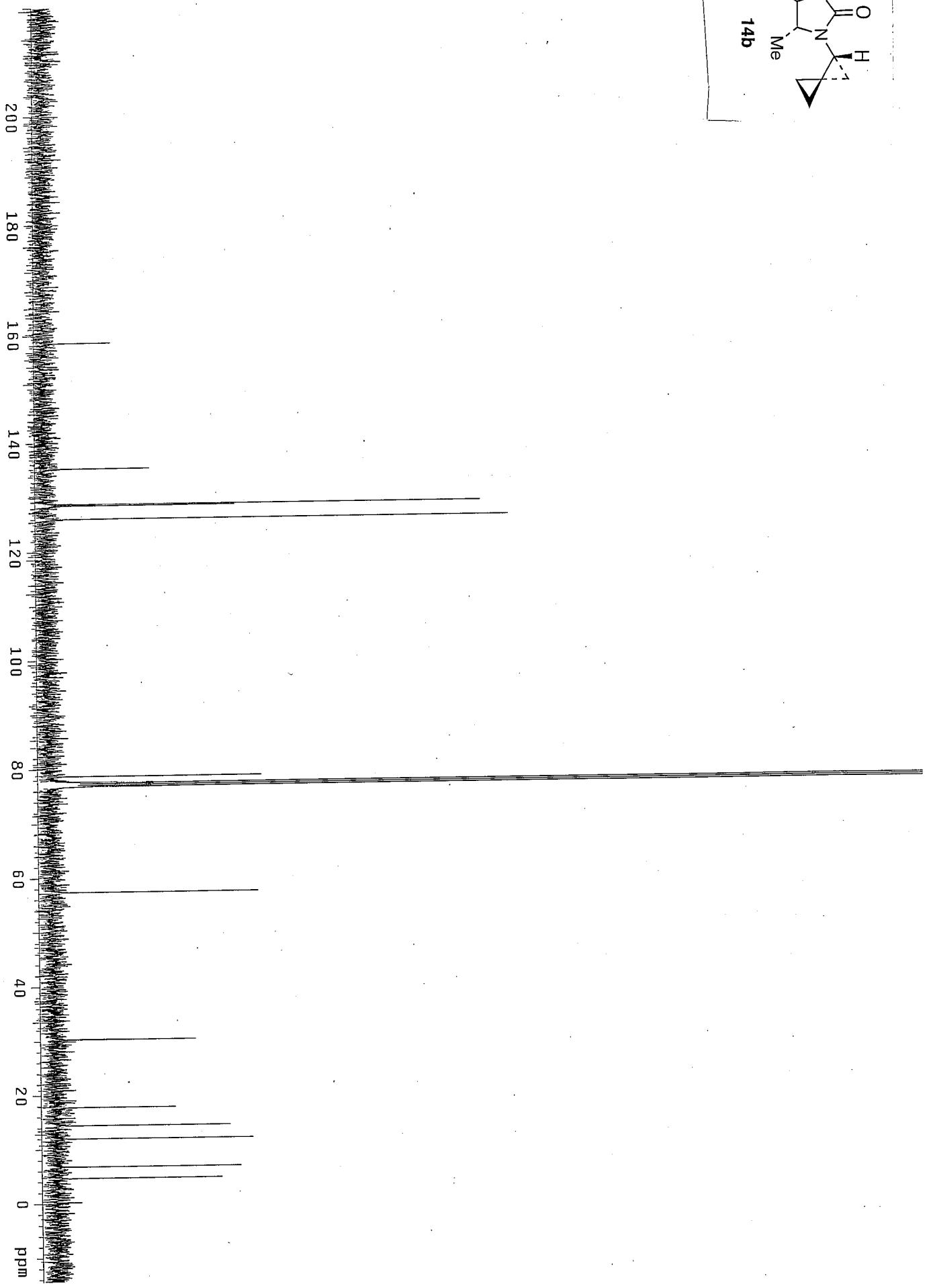
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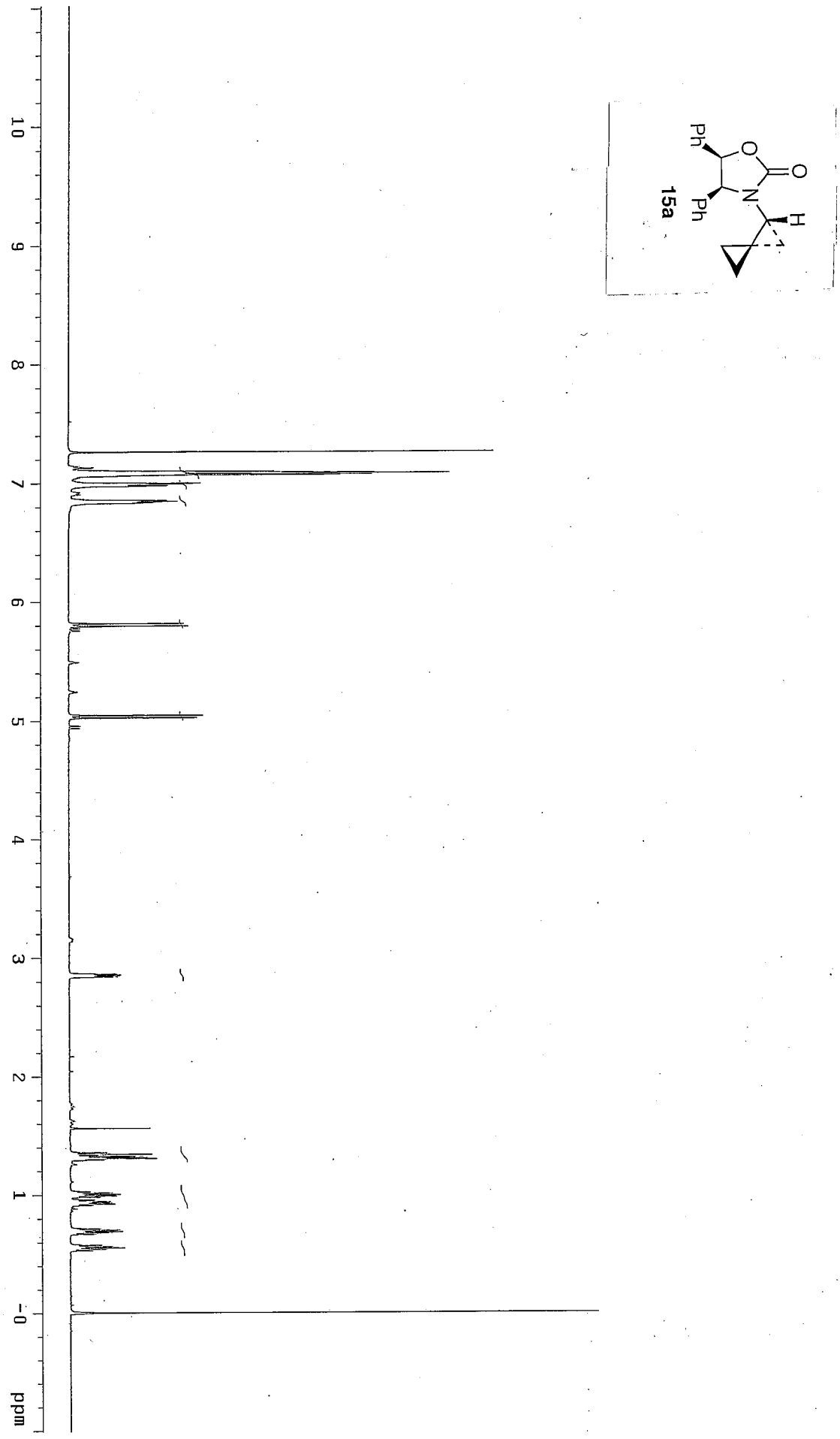
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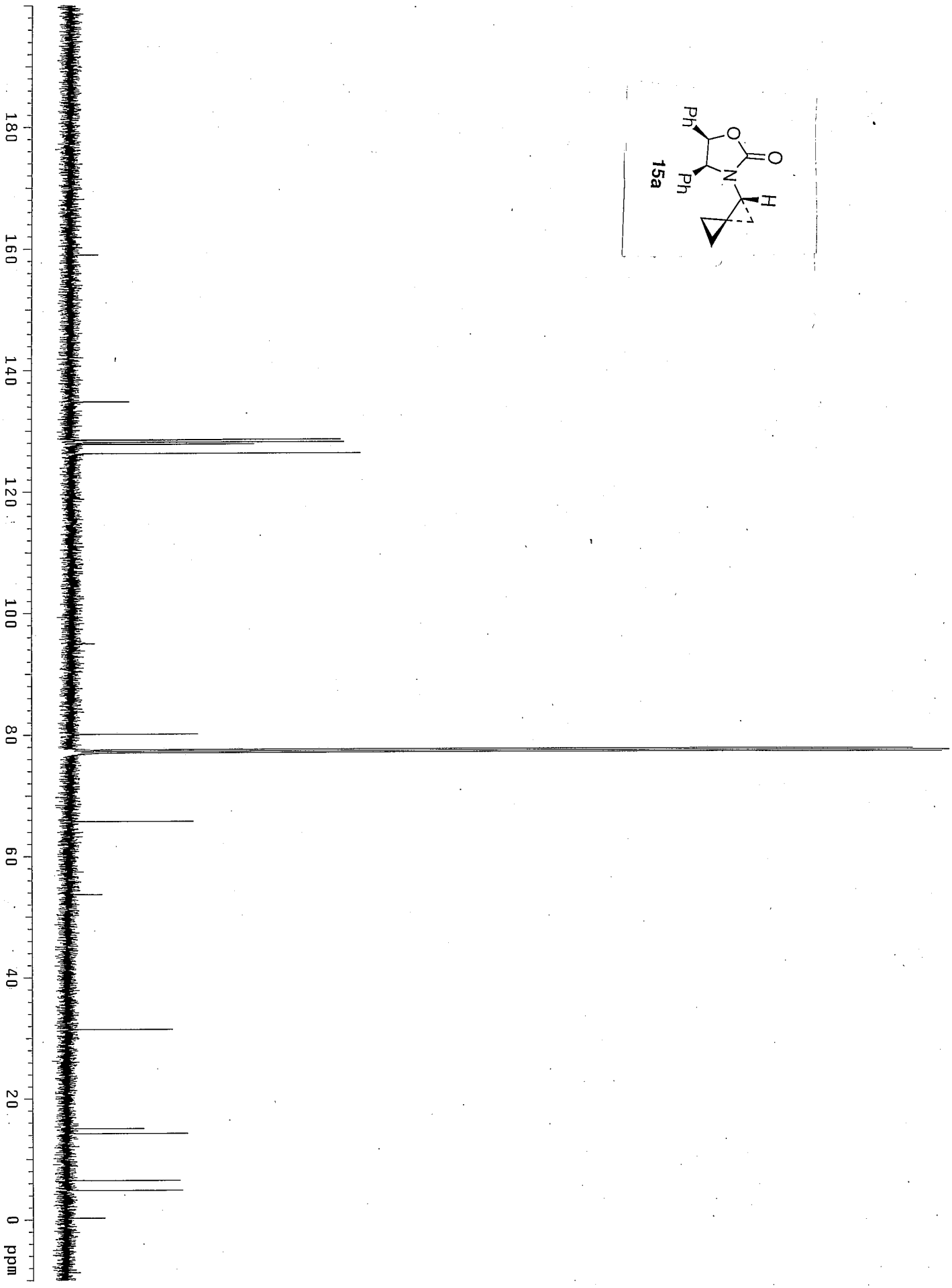
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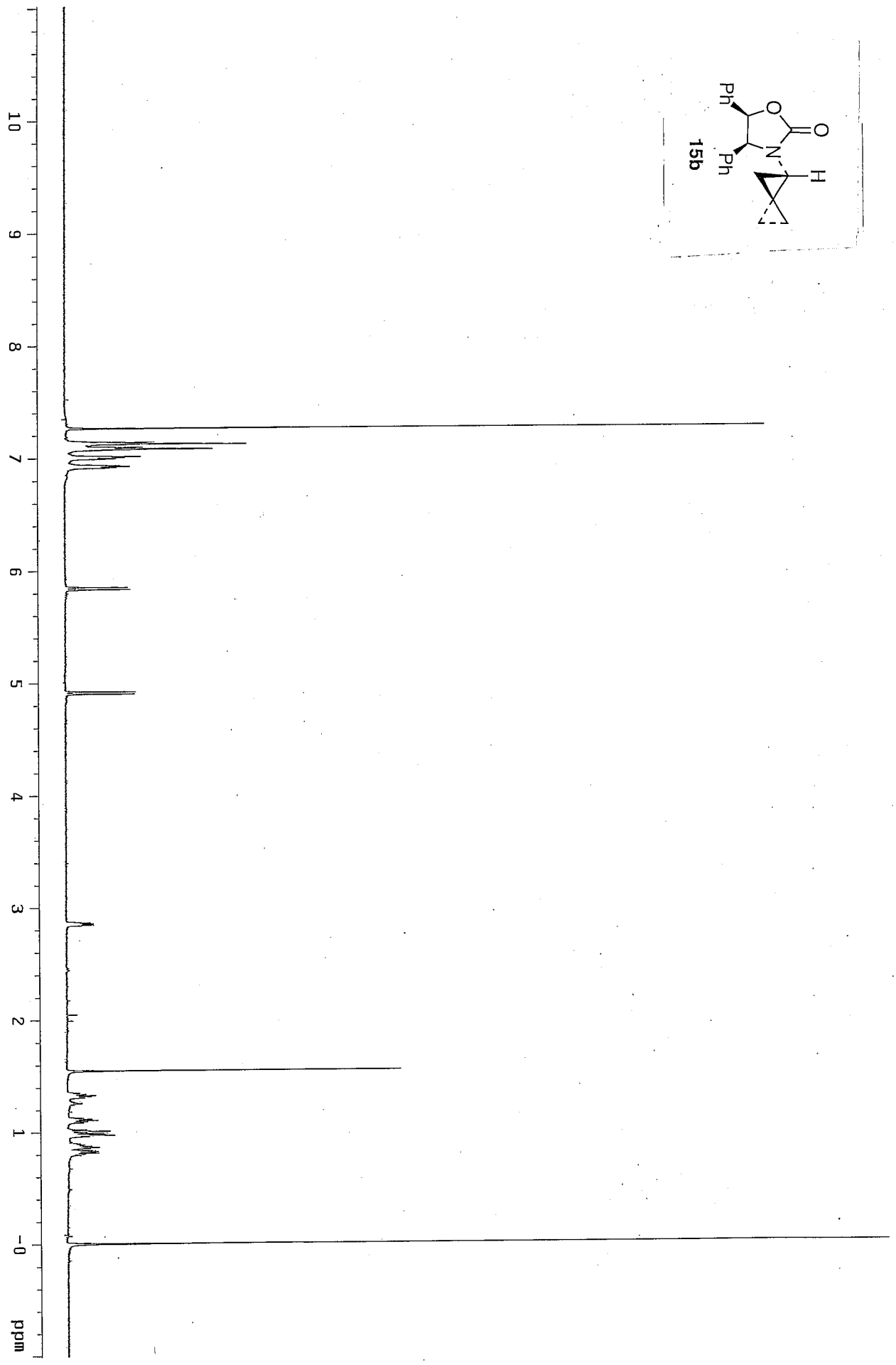
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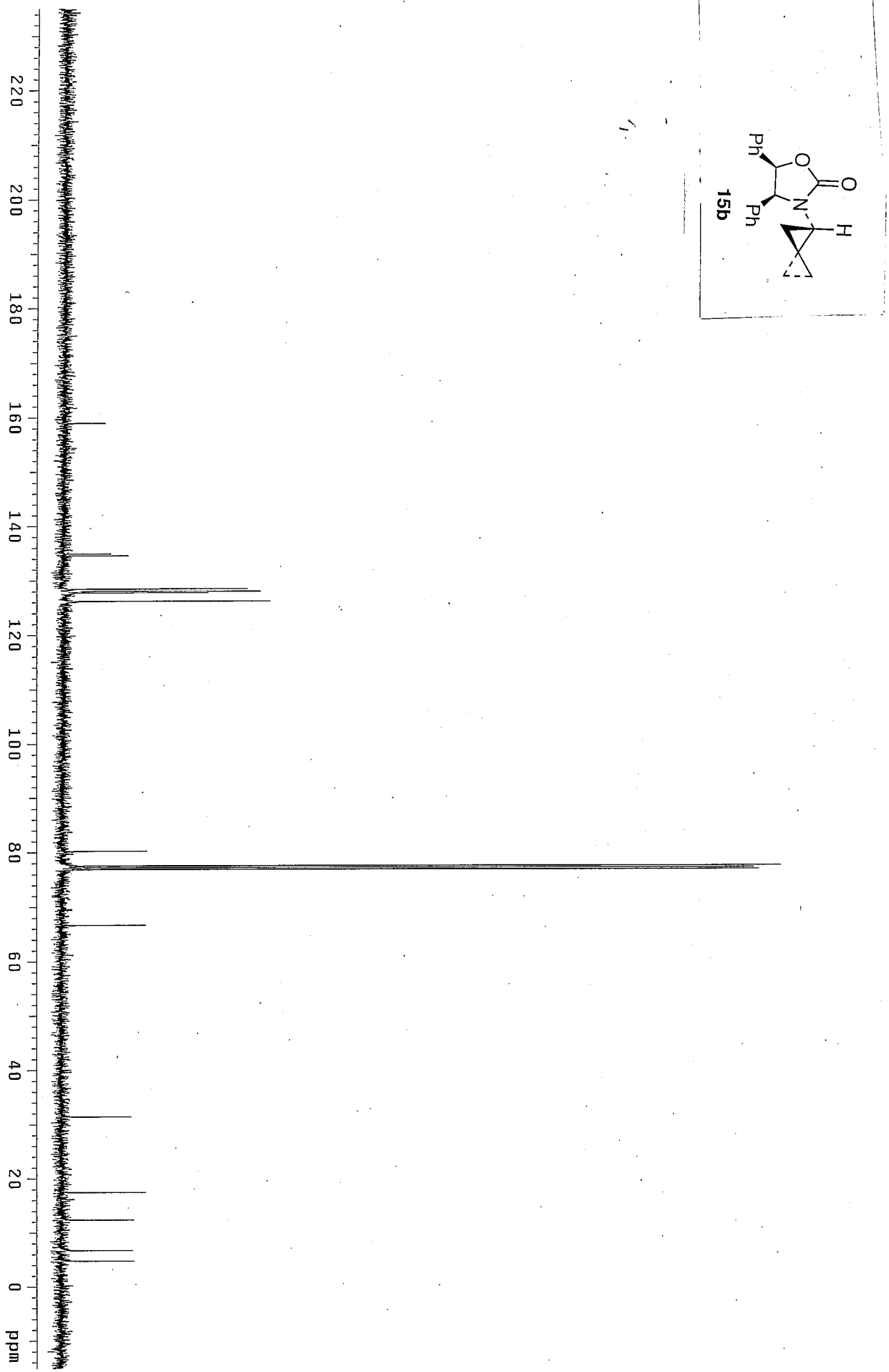
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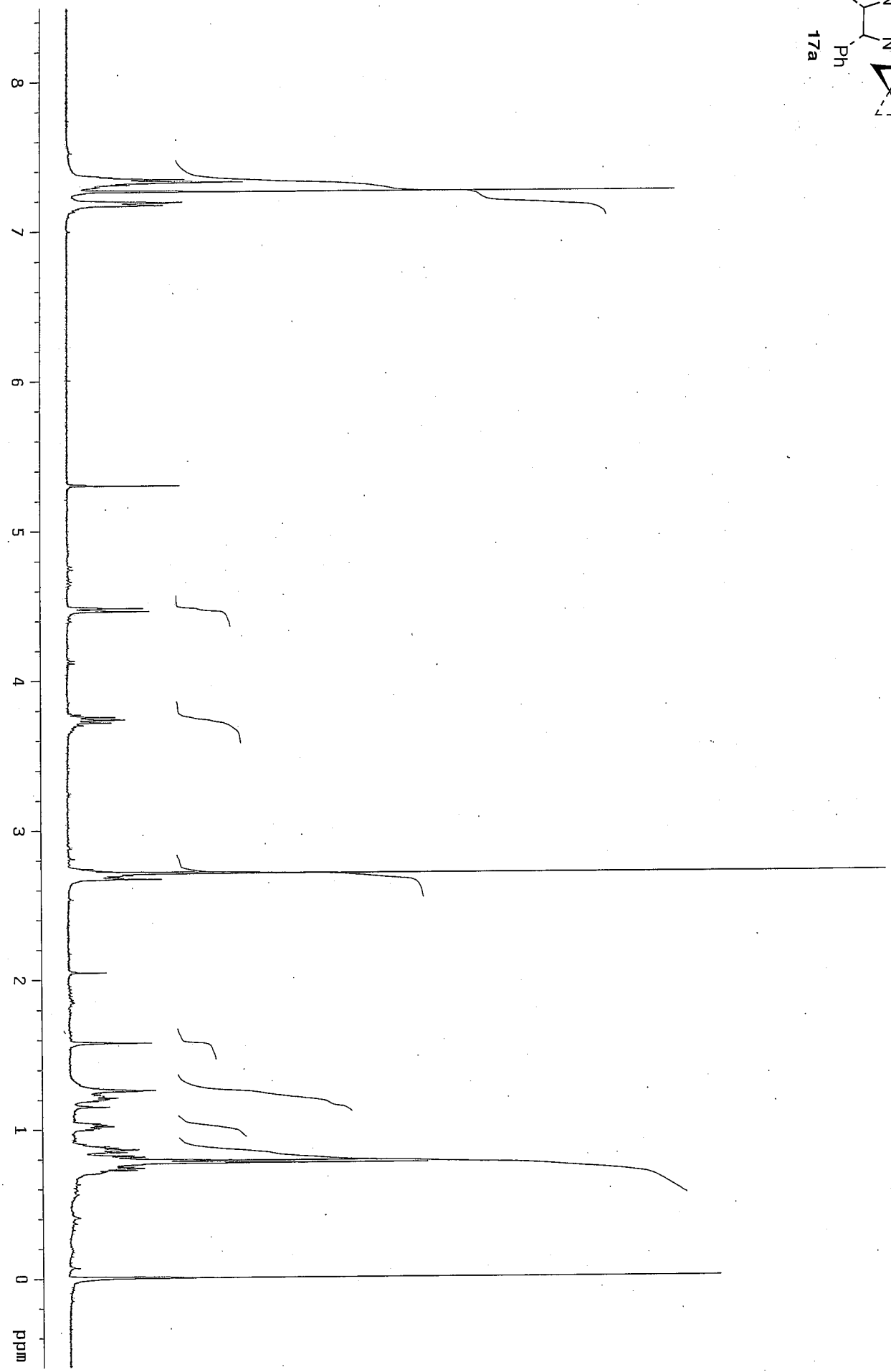
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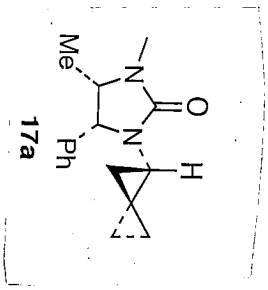
ppm



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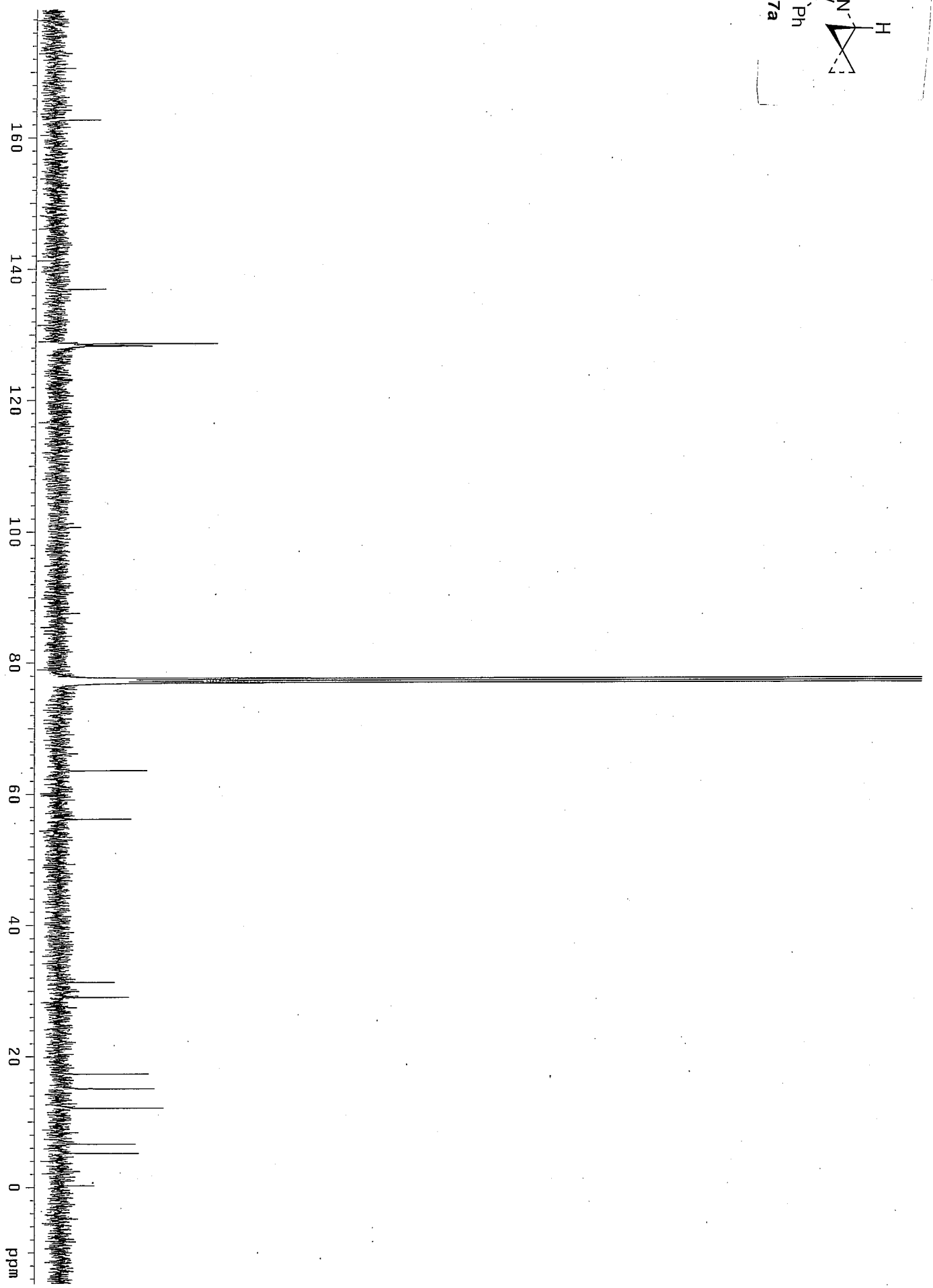
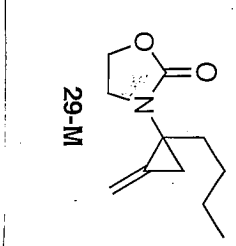
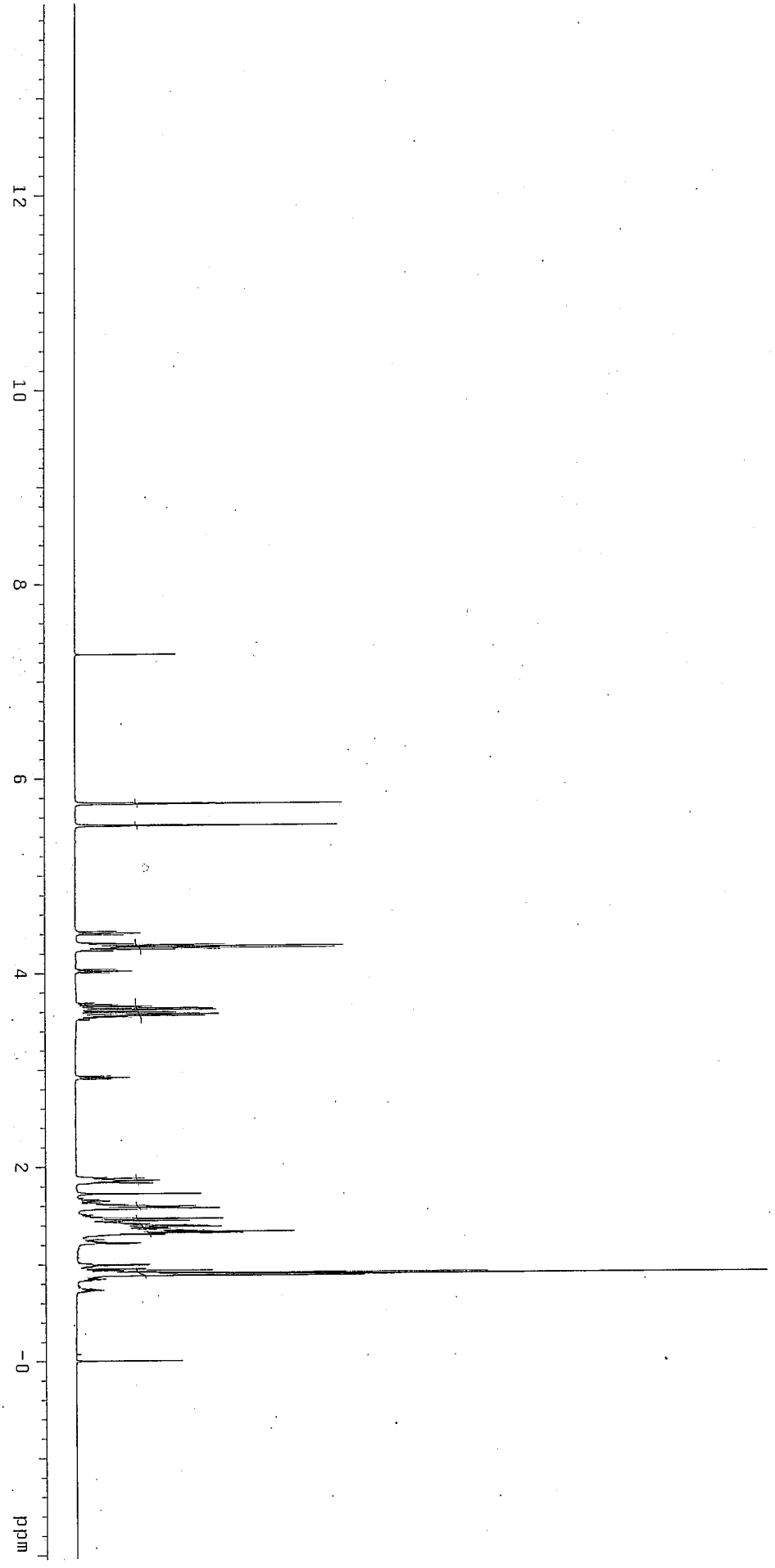
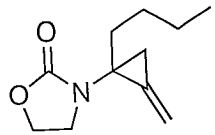


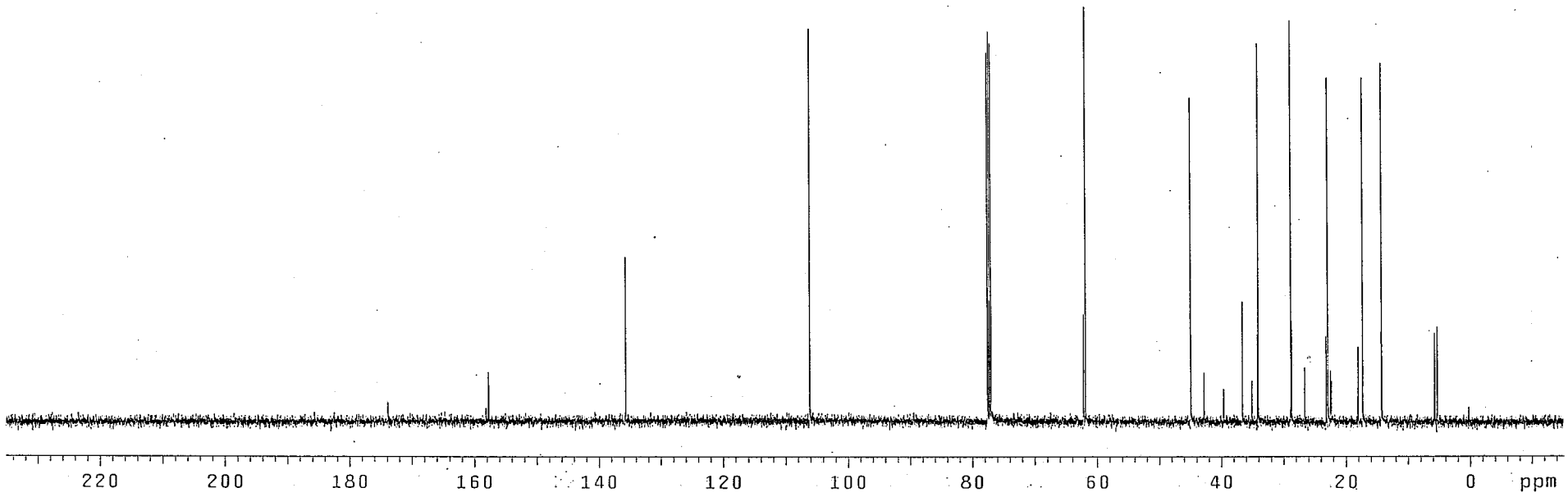


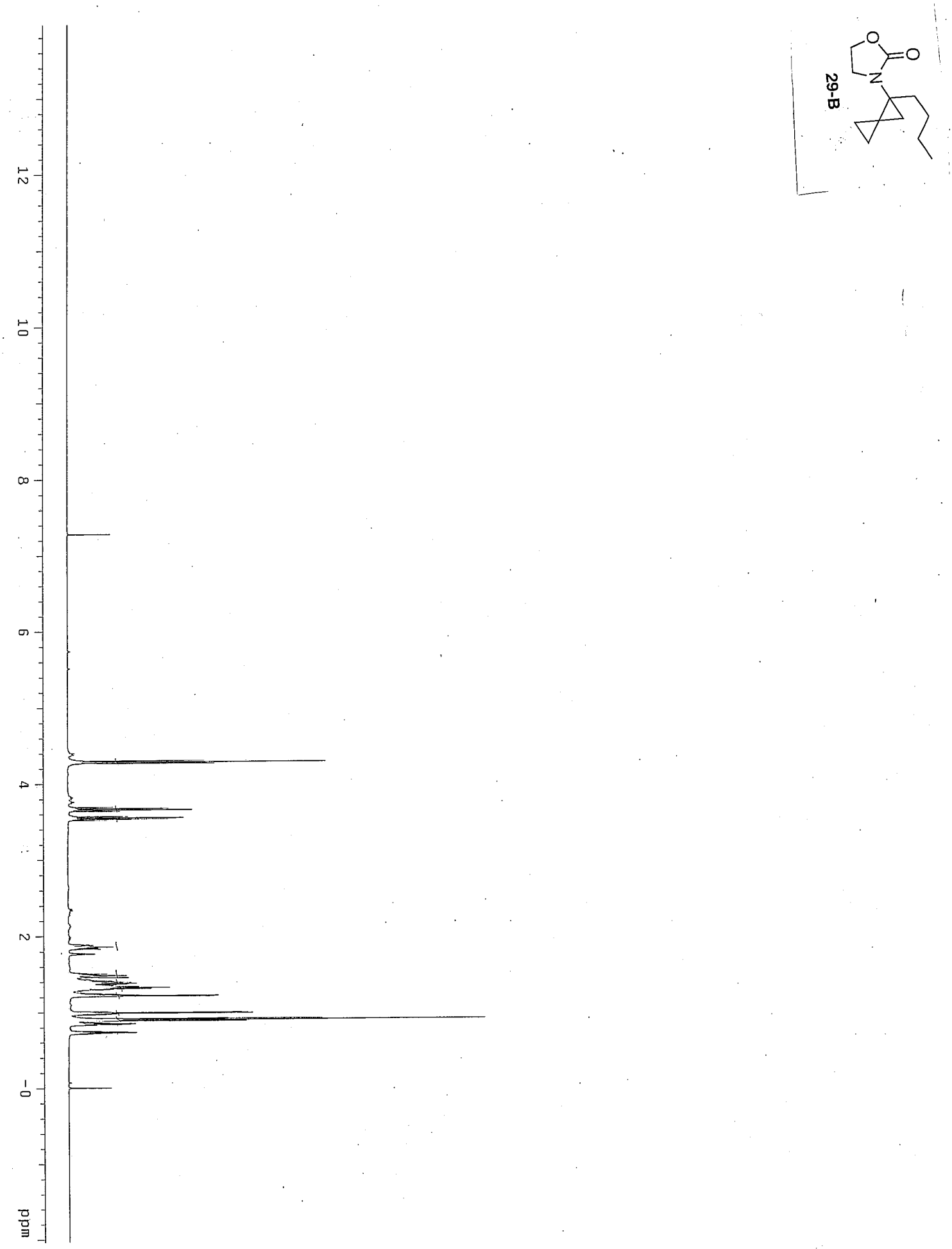
29-M





29-M




29-B
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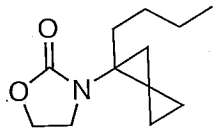
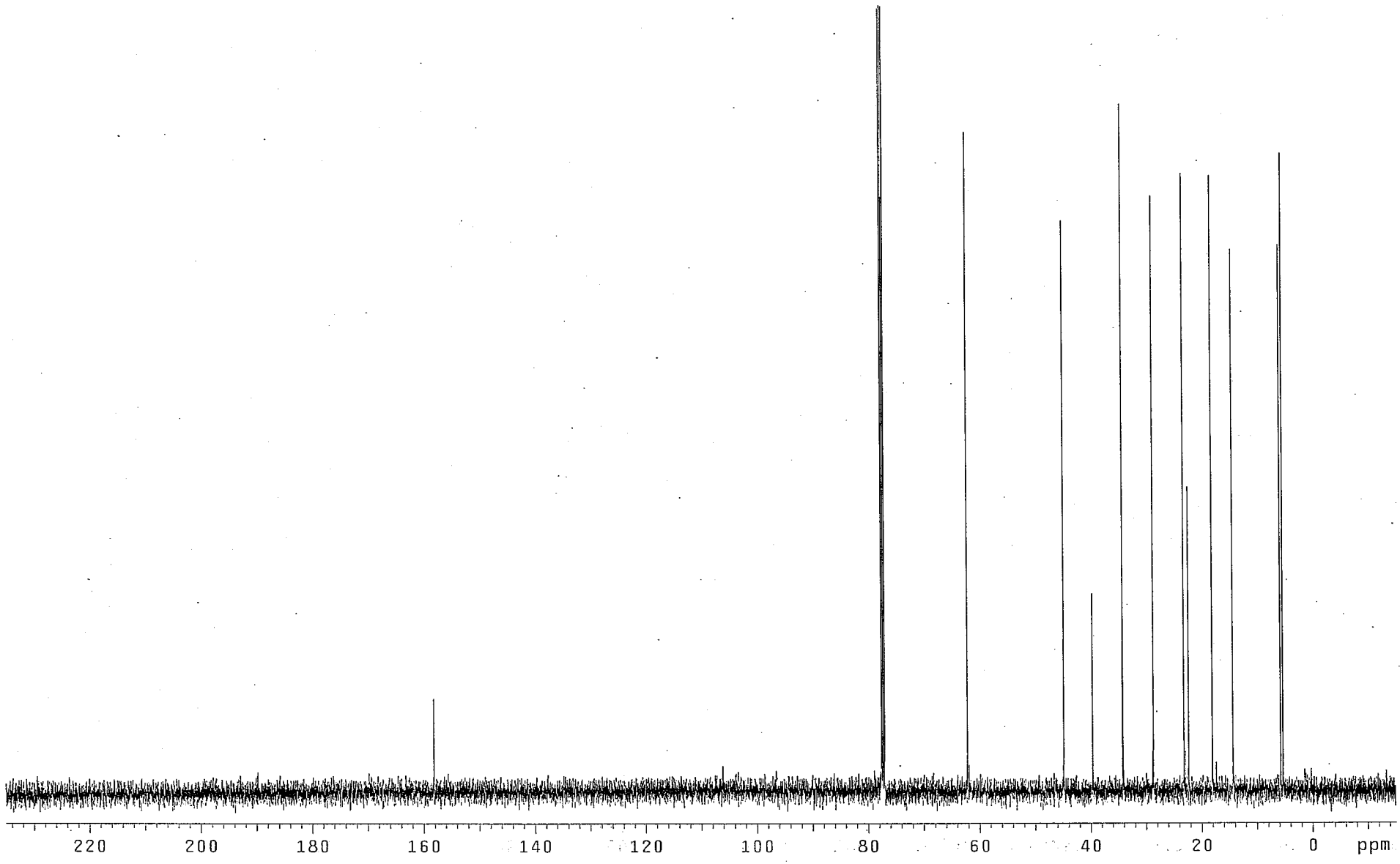
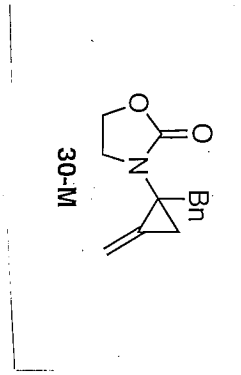
ppm

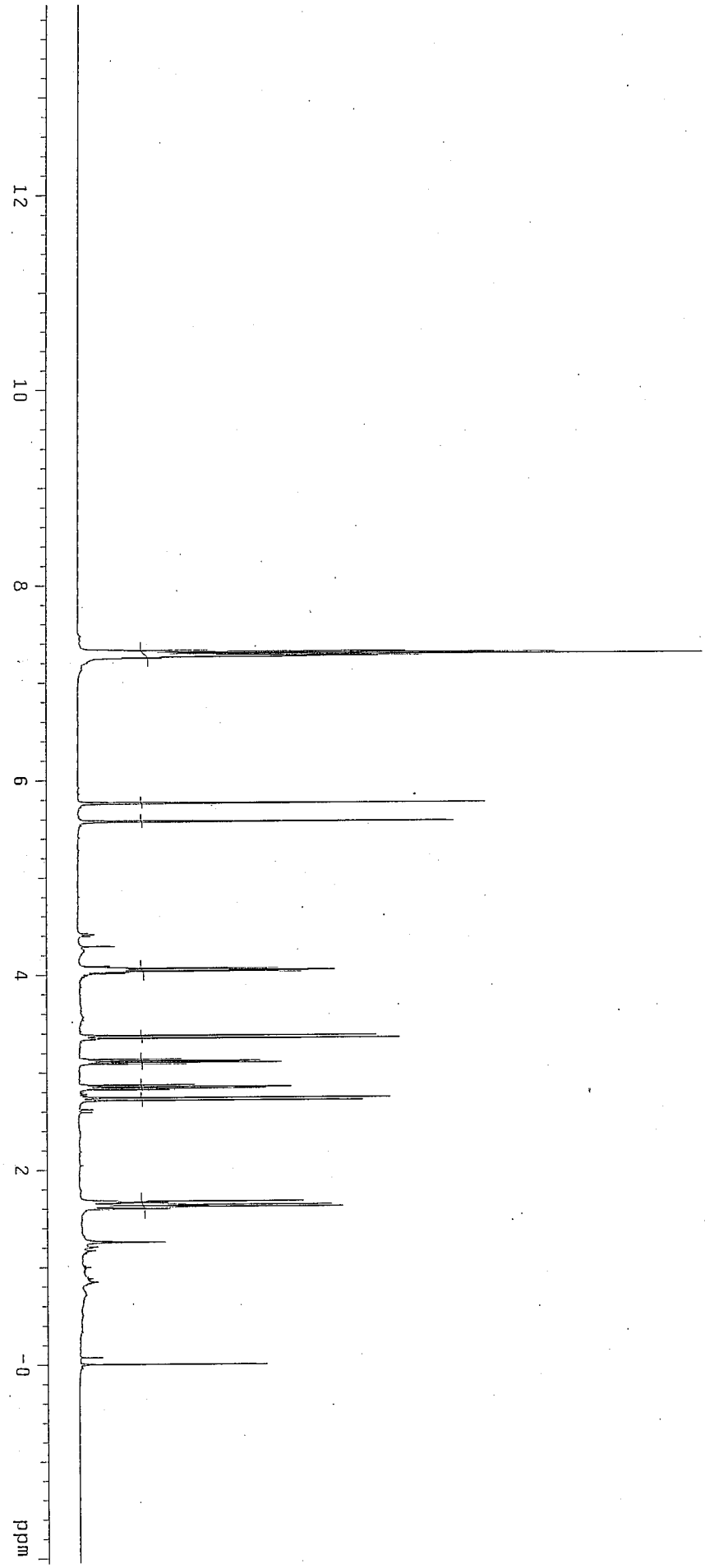


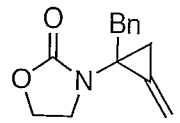
29-B



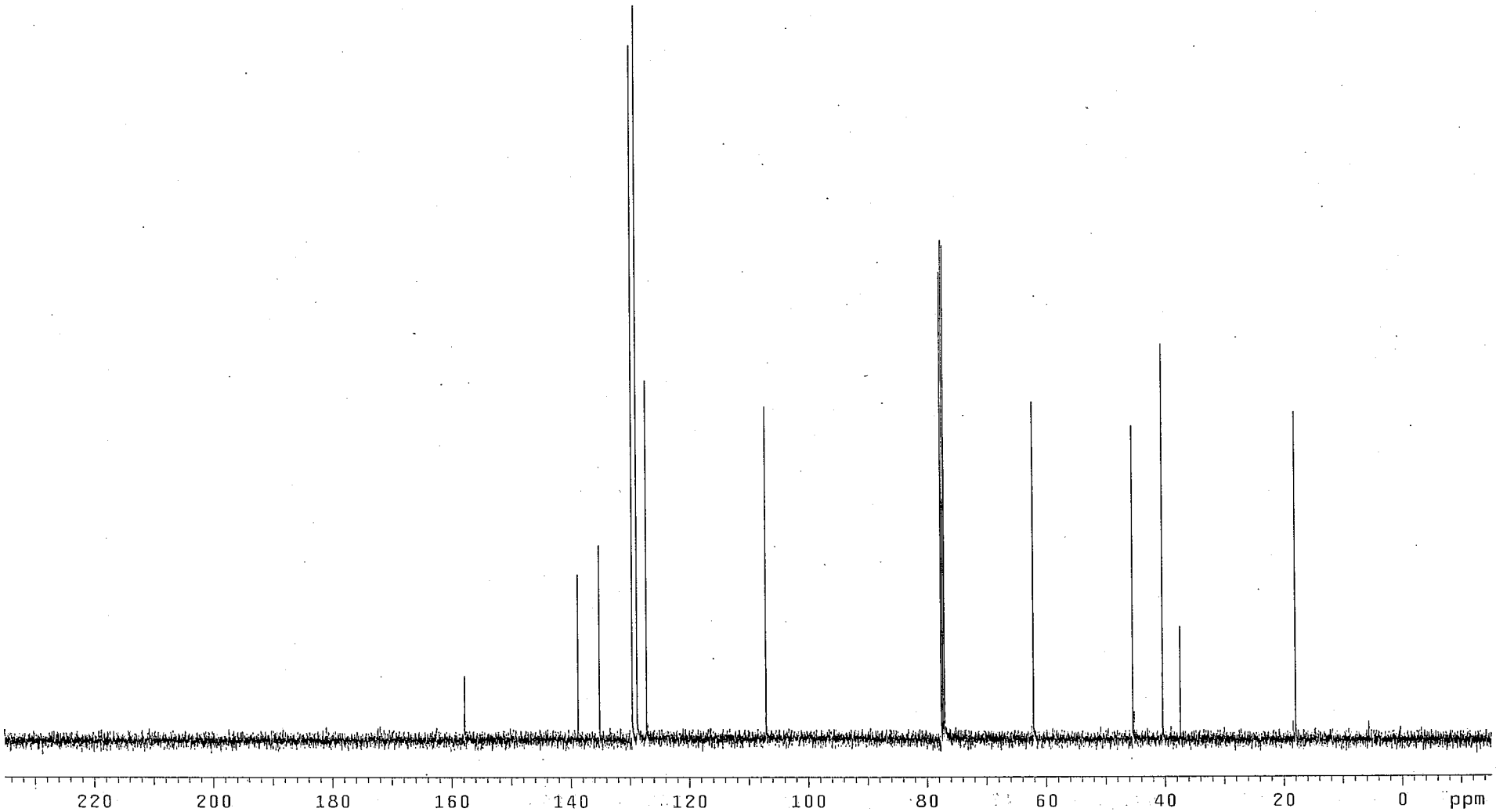


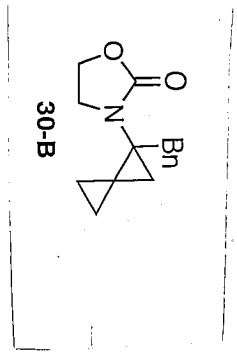
3O-M



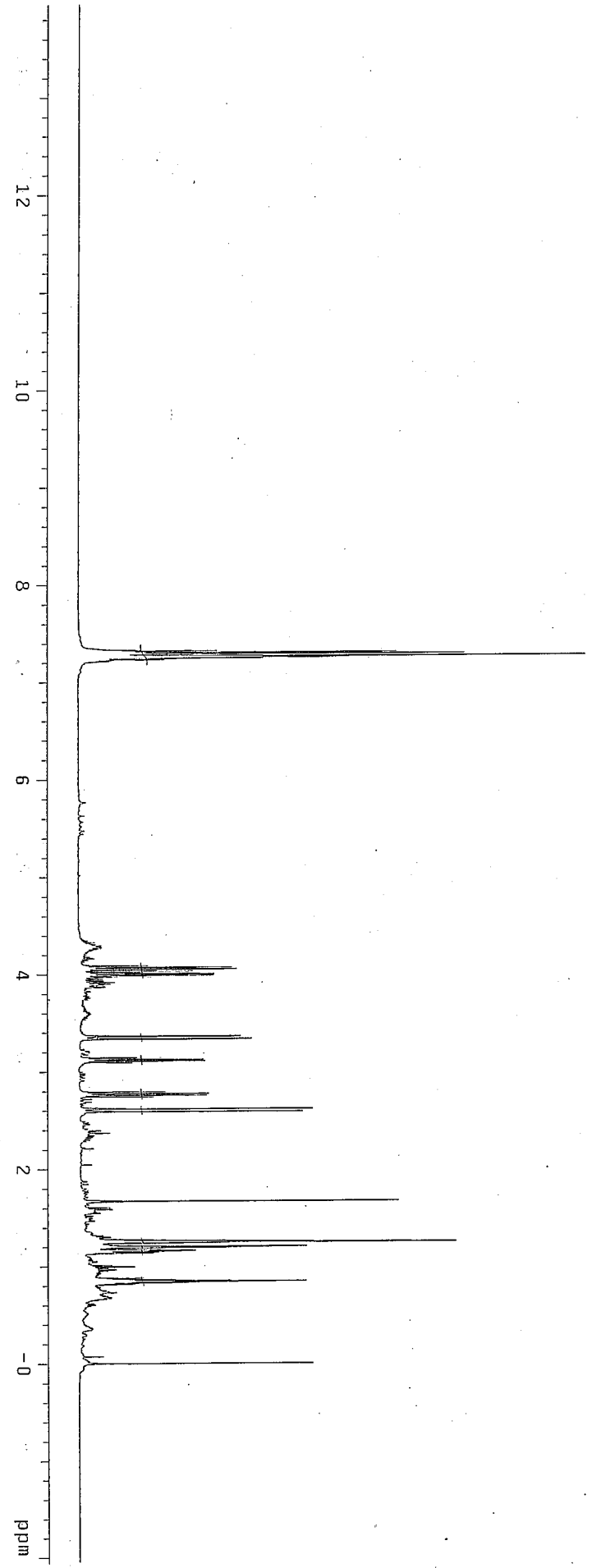


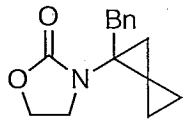
30-M



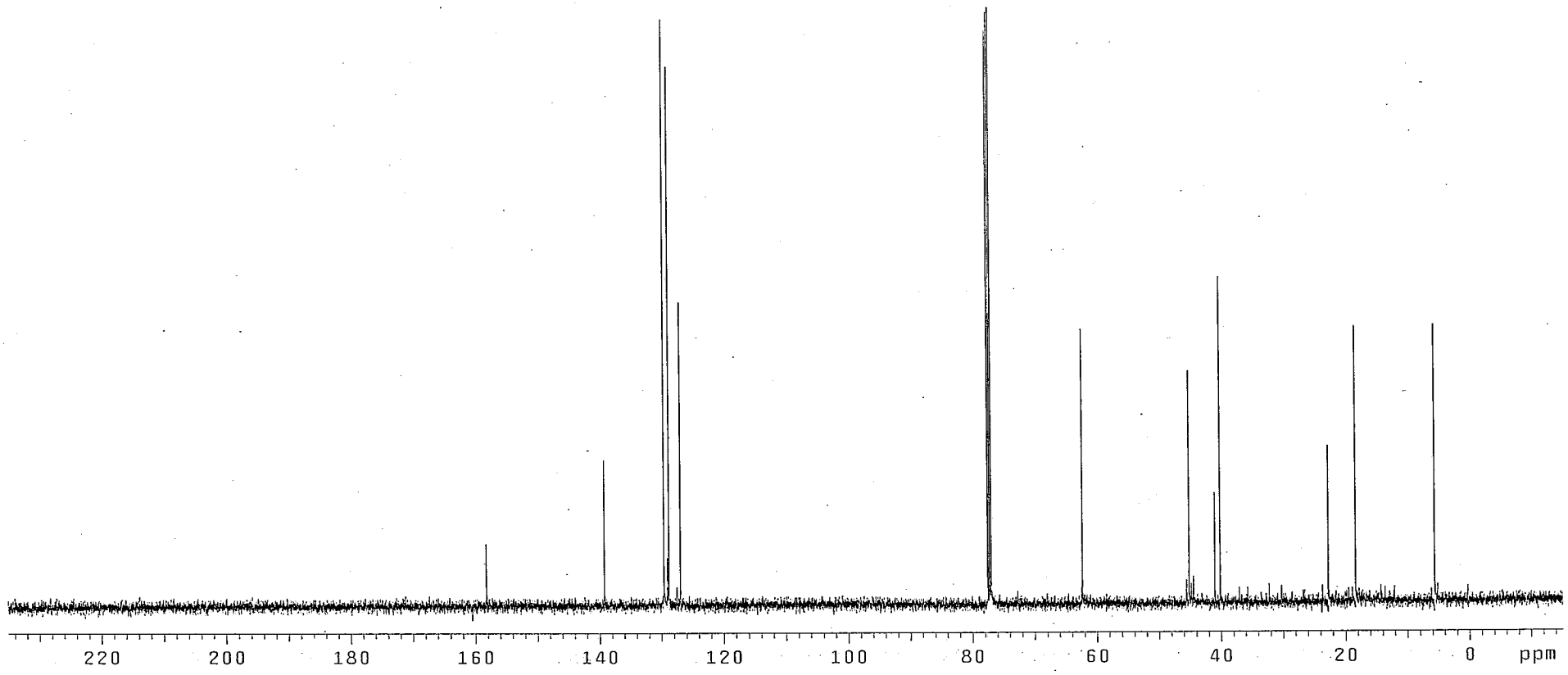


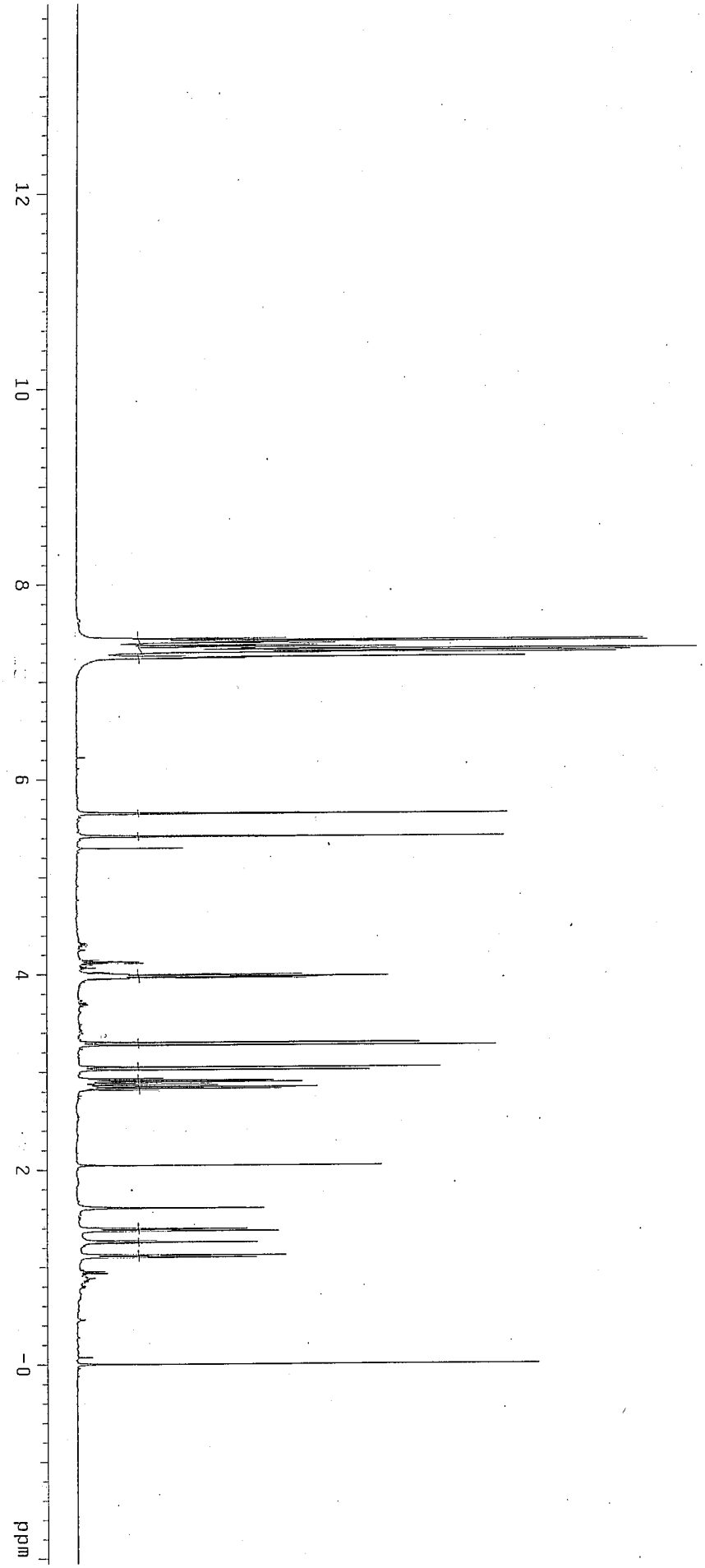
30-B

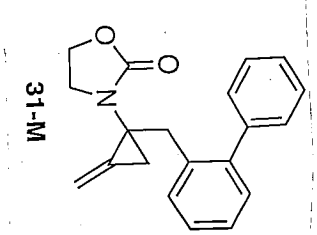




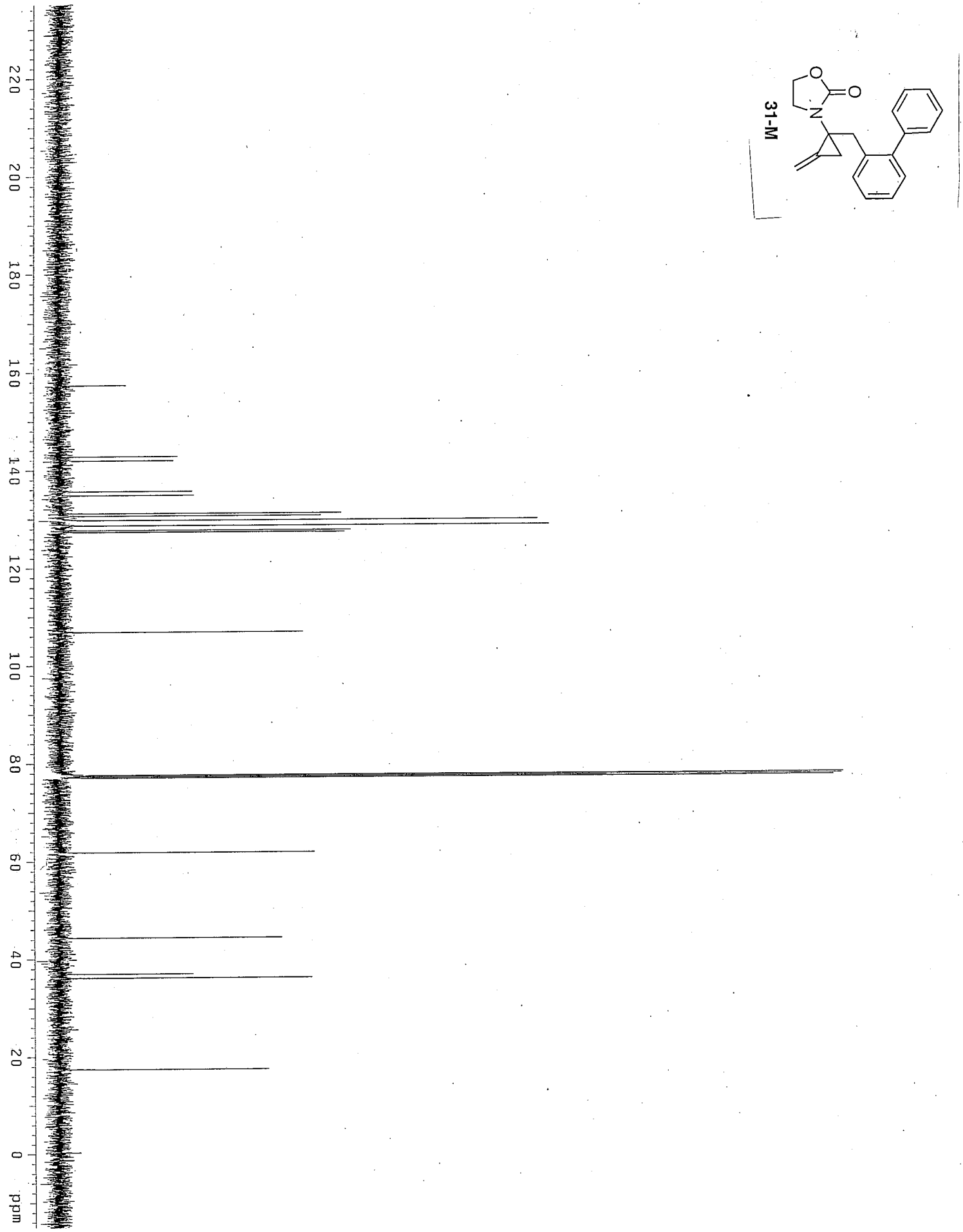
30-B

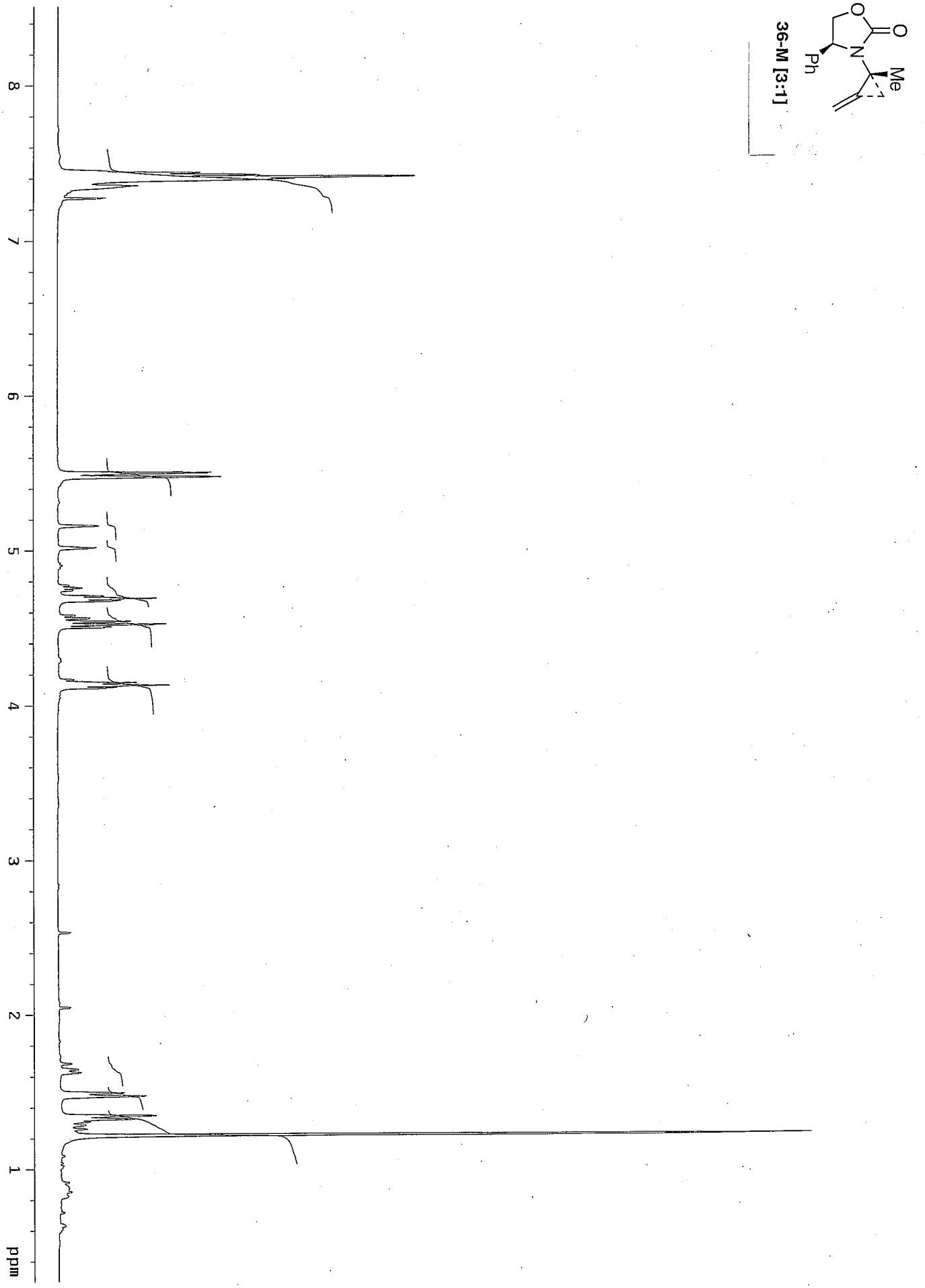






31-M




36-M [3:1]
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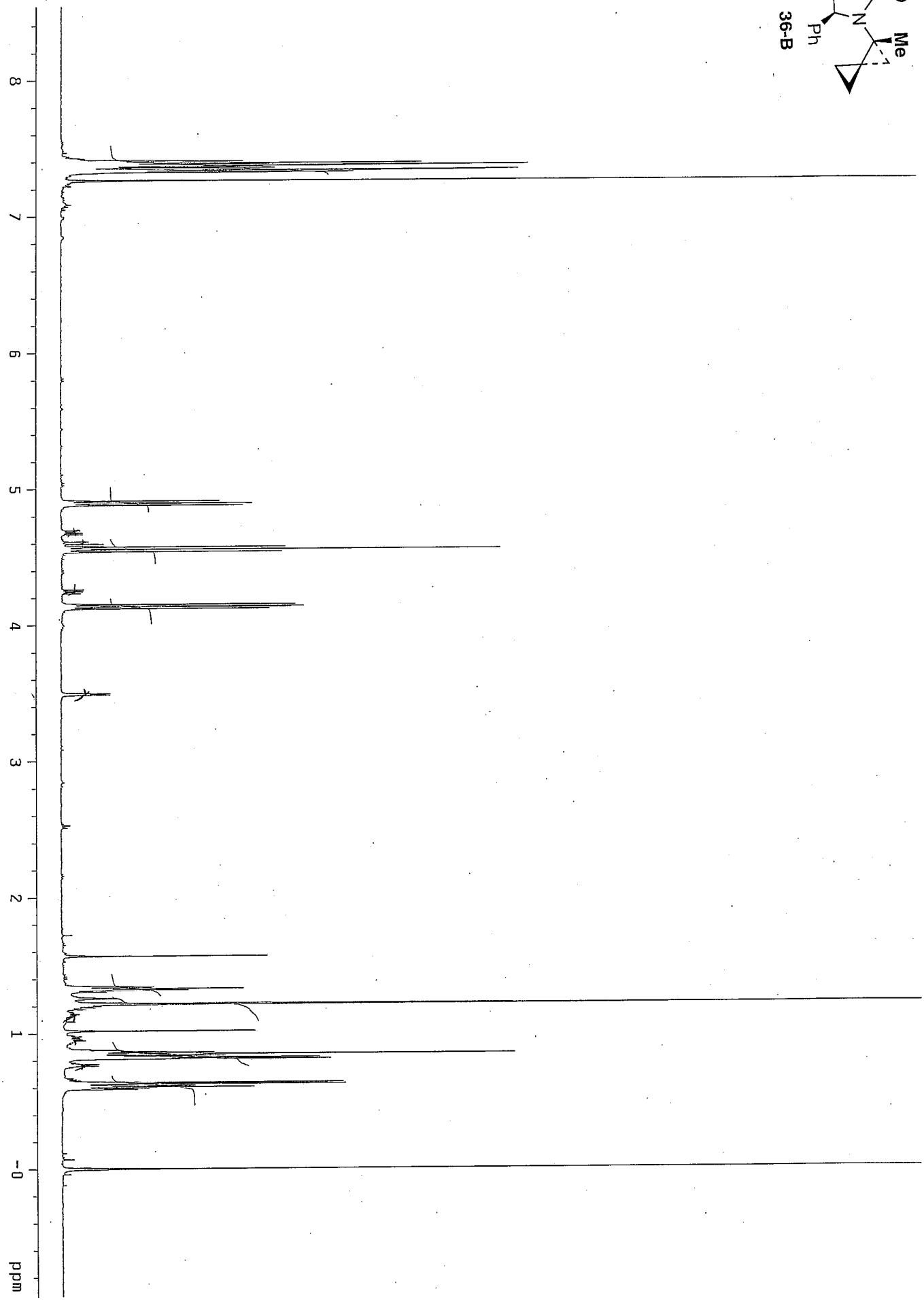
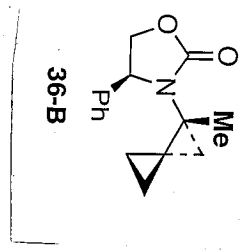
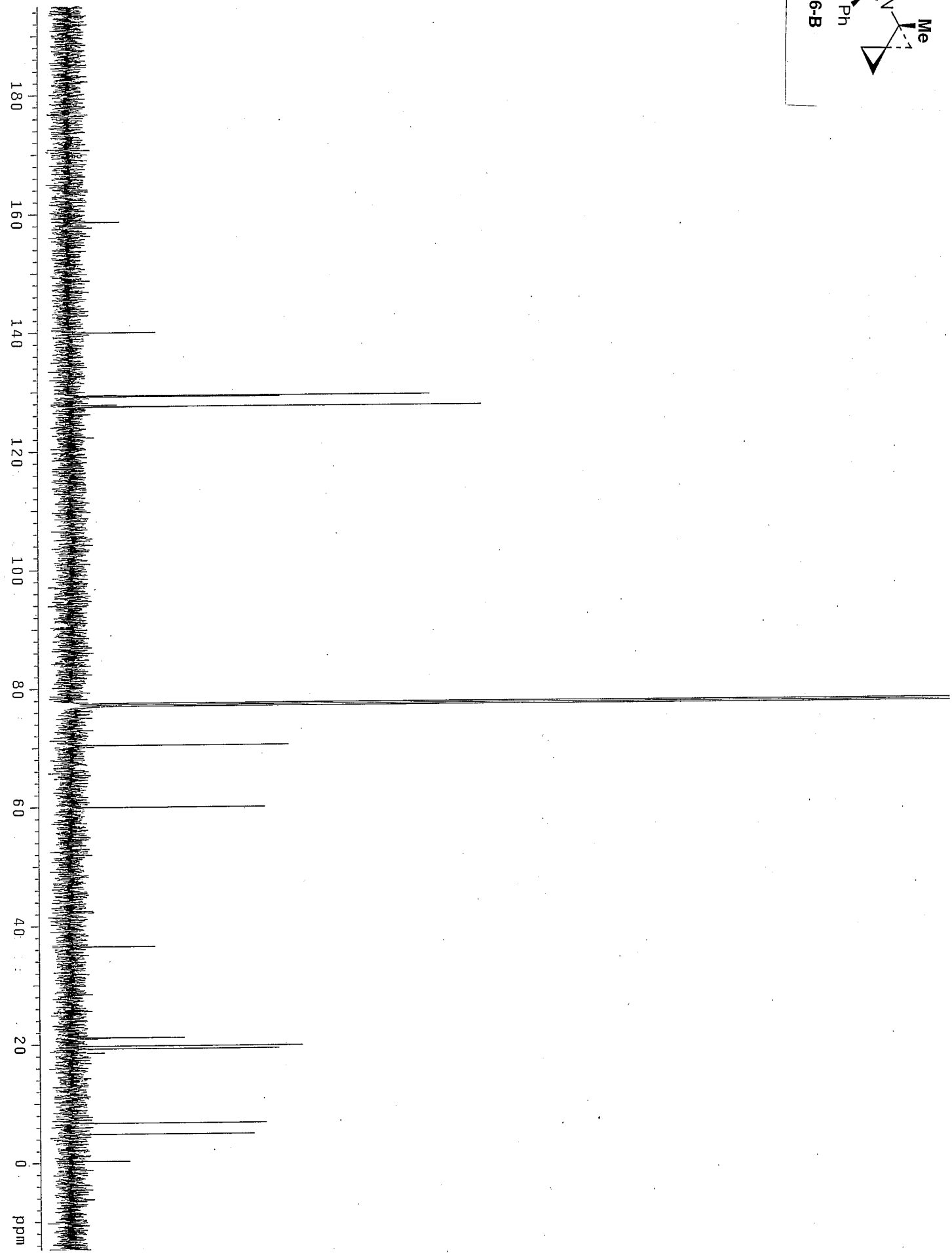
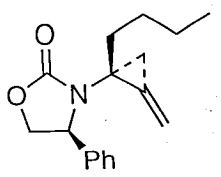
1
ppm



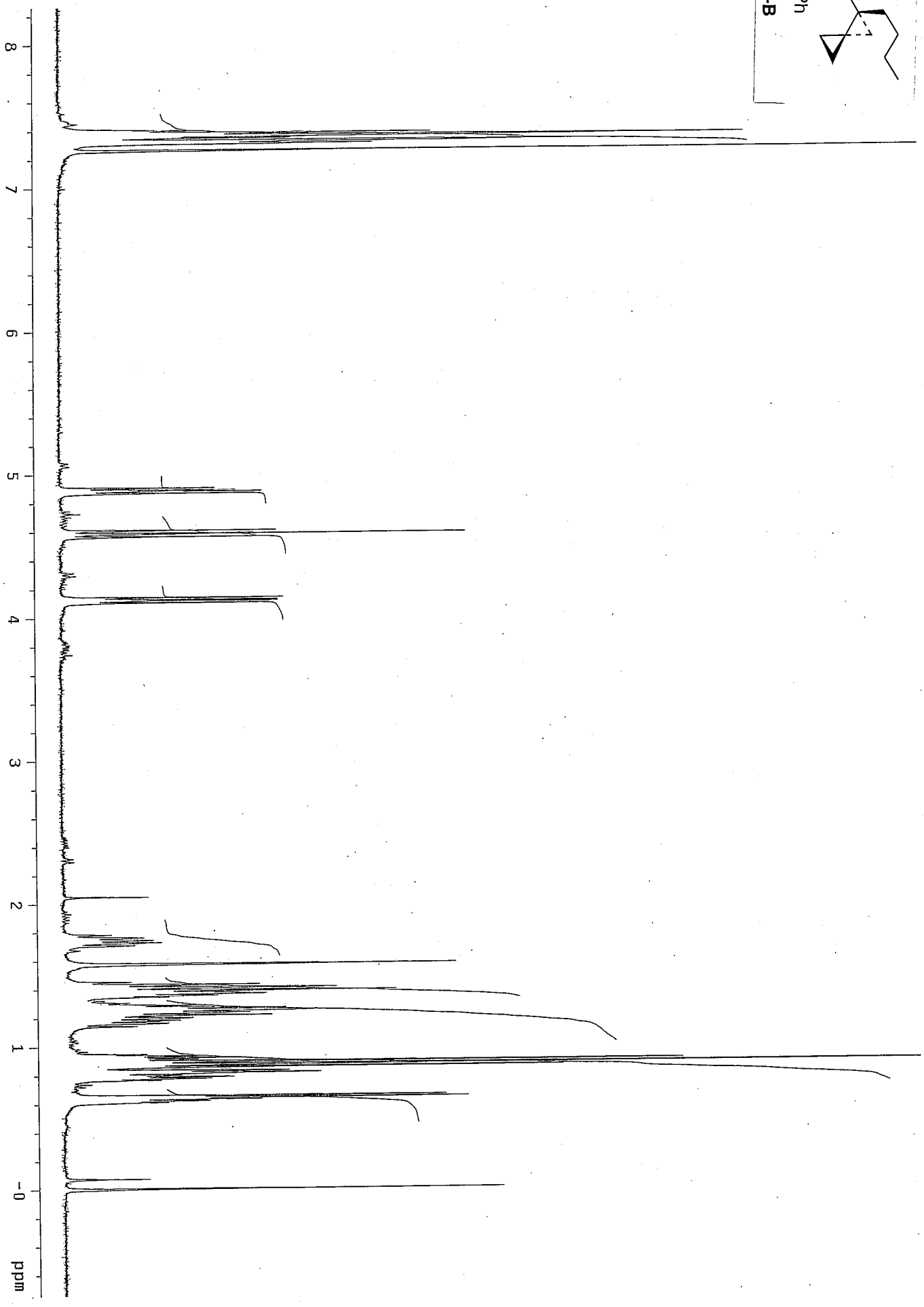


36-B





37-M [5:1]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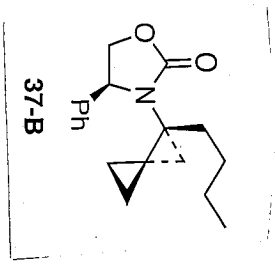
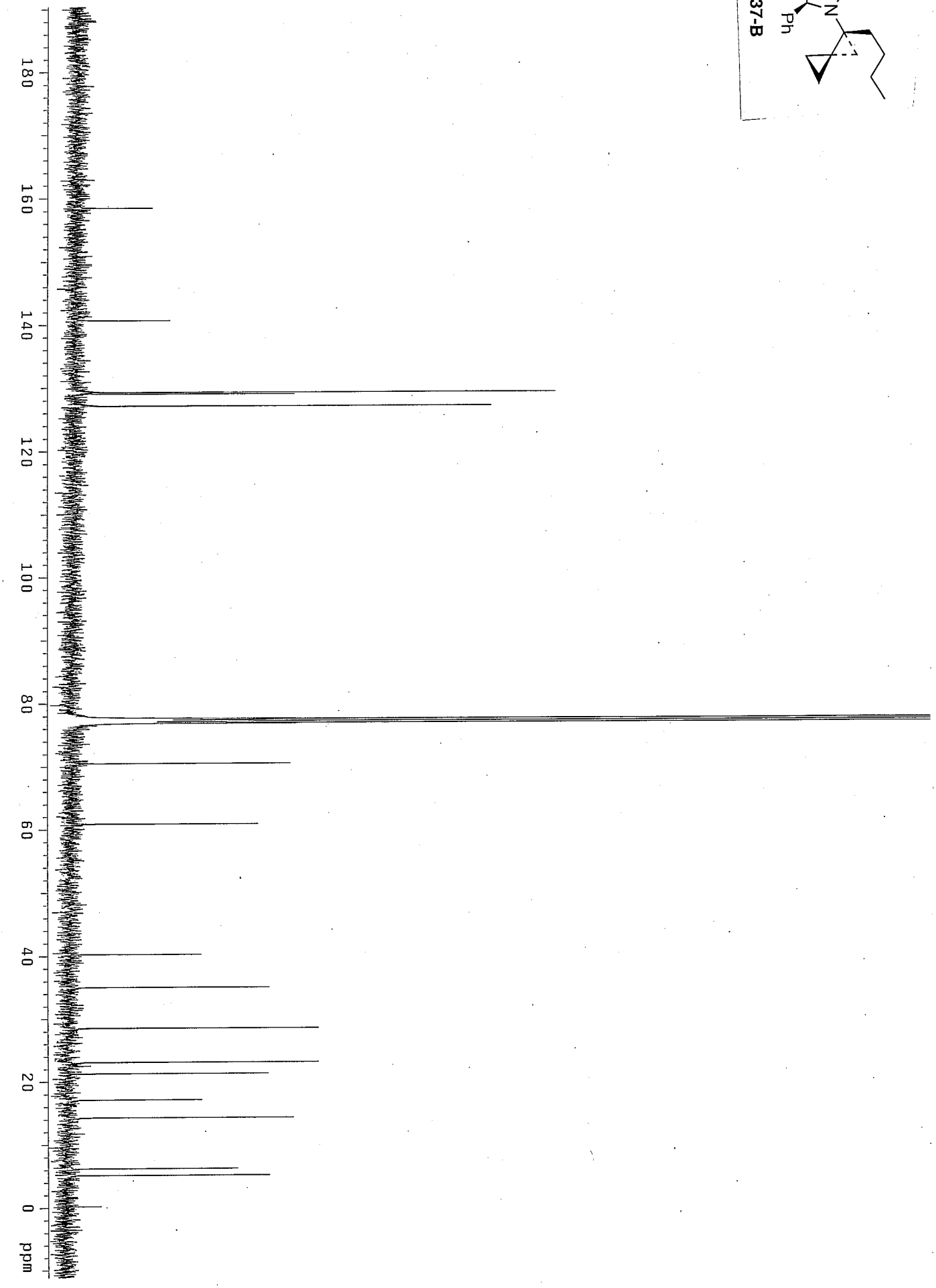
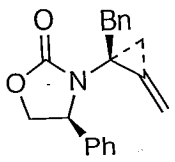
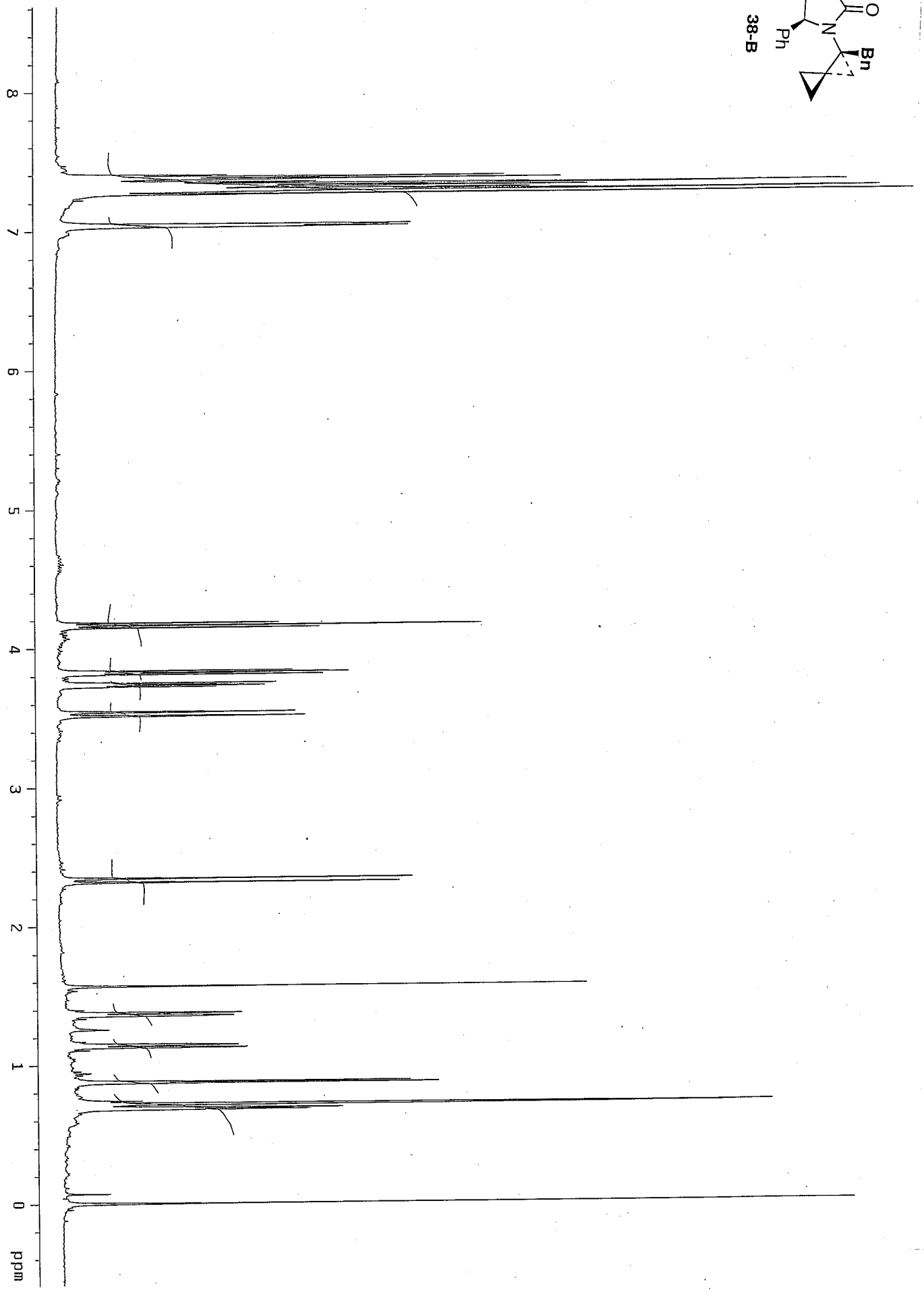


37-B





38-M [4:1]


38-B
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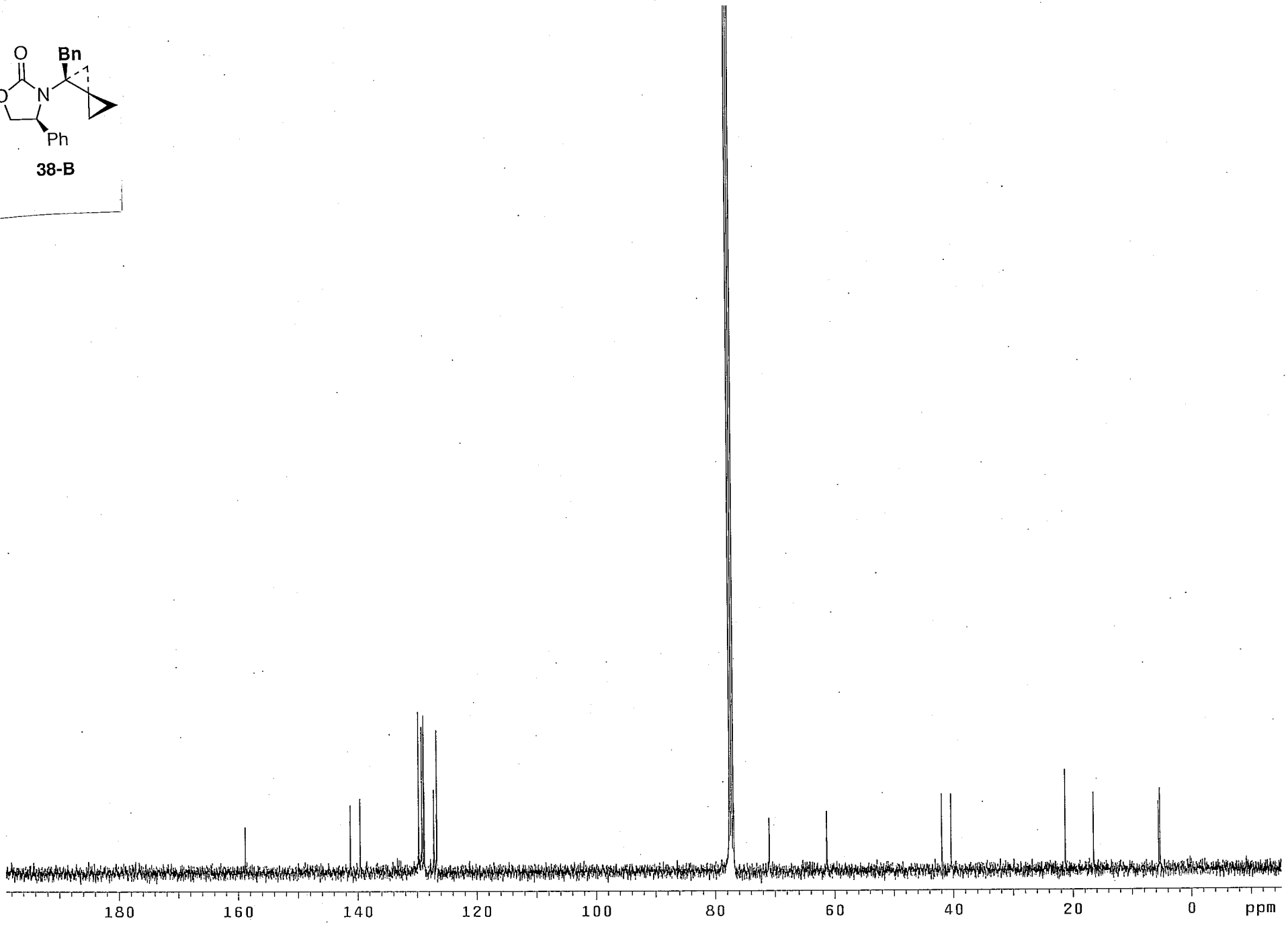
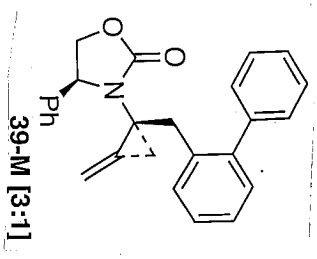
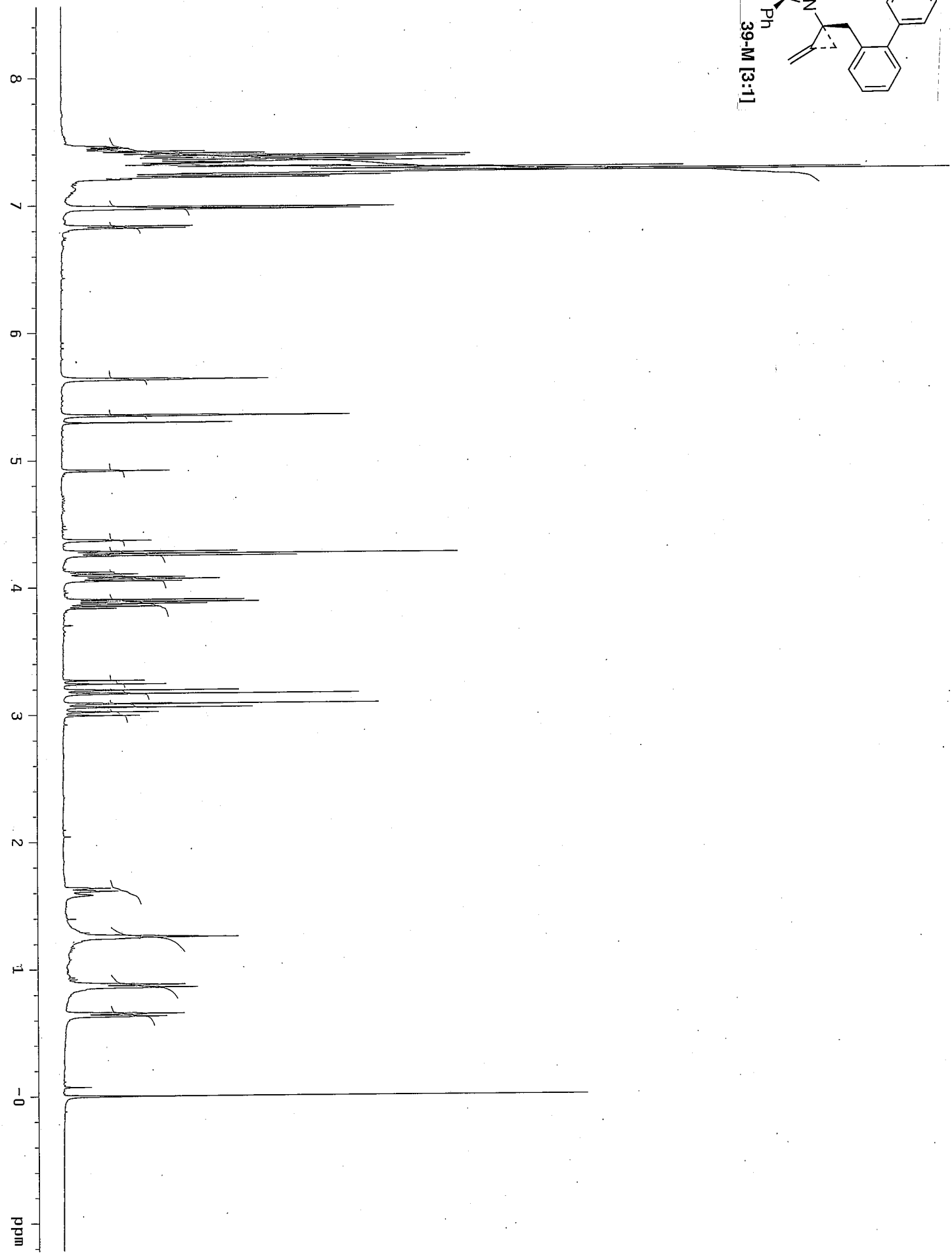
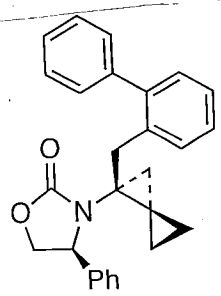
38-B



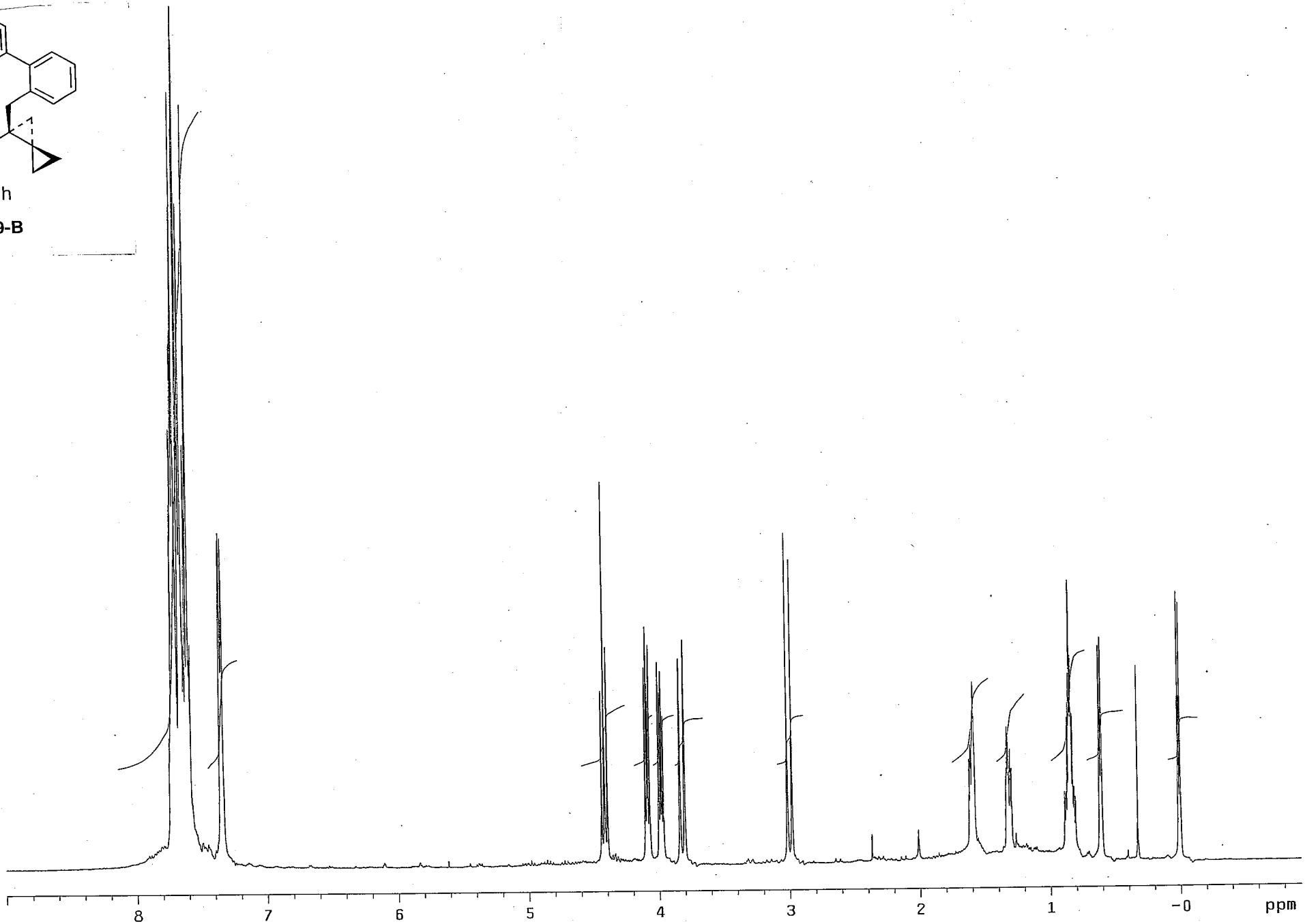


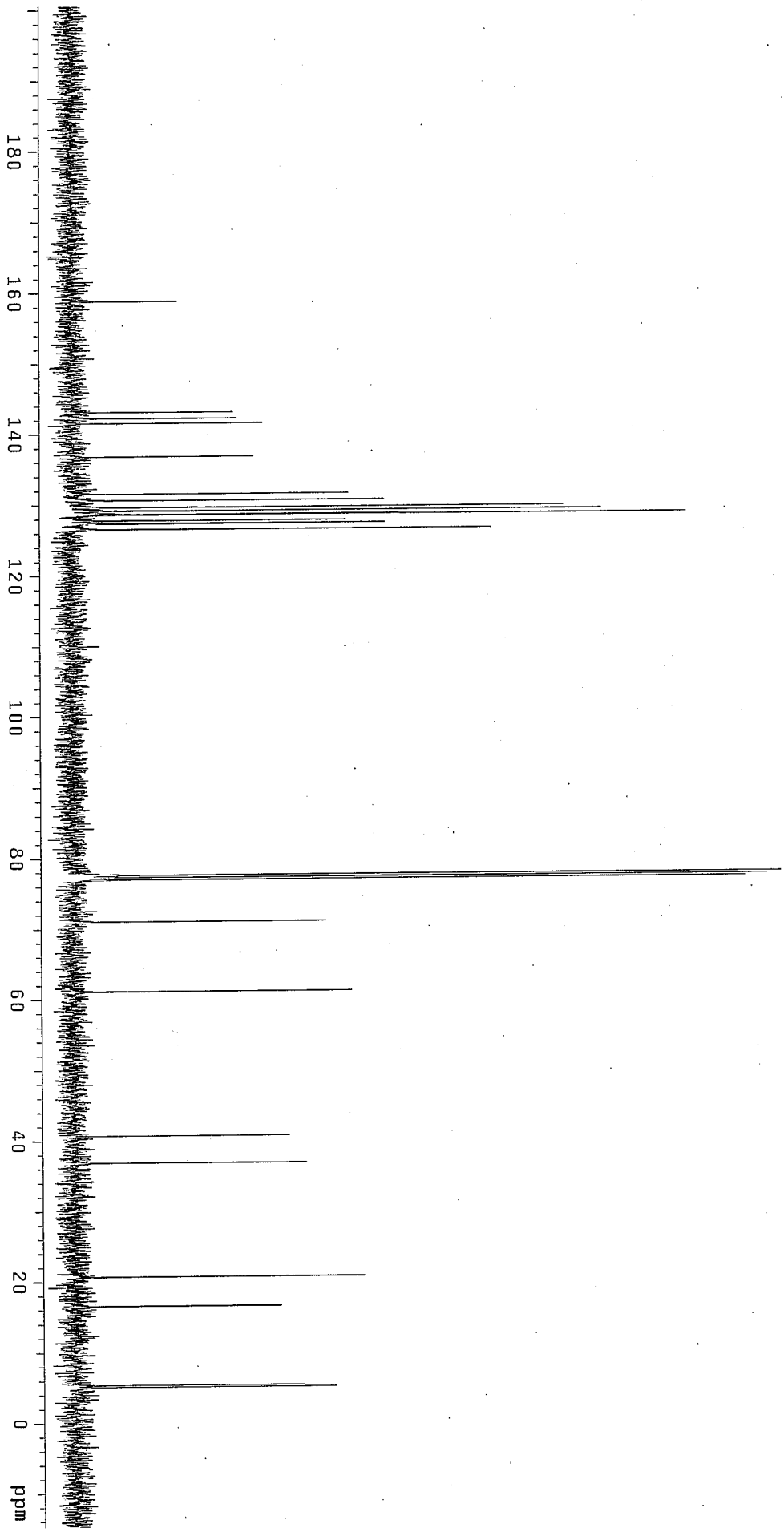
39-M [3:1]

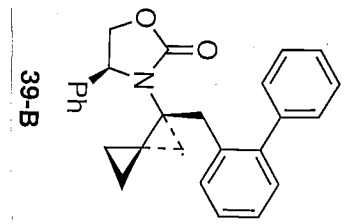




39-B







39-B