SUPPORTING INFORMATIONS

Ultrasmall monodisperse NiO nanocrystals as a heterogeneous catalyst for the A3-coupling reaction toward propargylamines

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Experimental section

Materials and instrumentations

Nickle (acetylacetonate)$_2$ [Ni(acac)$_2$], oleylamine, triphenylphosphine (TPP), diphenylether (DE), and all other commercially available chemicals were purchased from Merck Chemical Company with high purity. The applied solvents were purified by standard procedure. Melting points were measured by a Yanagimoto Micro Melting Point apparatus in open capillary tubes. Fourier transform infrared (FT-IR) spectra were gained (in KBr) by Nicolet FT-IR spectrophotometer. The ^{1}H and ^{13}C NMR spectra were recorded on Bruker DRX-400 spectrometer with $CDCl_3$ as solvent at 25 °C and chemical shifts are given in ppm rel. to Me_4Si. The XRD patterns were obtained by an X'PertPro (Philips) instrument with 1.54 Ångström wavelengths of the X-ray beam and Cu anode material. Microscopic morphology of the nanoparticles was visualized by SEM (MIRA 3 TESCAN). Energy-dispersive X-ray spectroscopy (EDX) of the nanoparticles imagined by a Sigma ZEISS, Oxford Instruments Field Emission. The purity determination of the substrates and reaction

monitoring were accomplished by TLC on silicagel polygram SILG/UV 254 plates (from Merck Company).

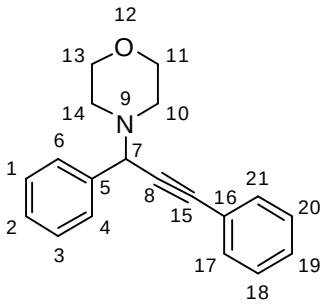
Synthesis of NiO nanoparticles

The synthesis protocol for preparation of ultrasmall NiO nanoparticles is a modified method which developed by Taeghwan and co-workers that employs the thermal decomposition of metal-surfactant complexes [24, 39]. Initially, $Ni(acac)_2$ (0.32 g) and Oleylamine (1.5 ml) were mixed under N_2 atmosphere at 100 °C. Afterwards, the fresh prepared Ni-Oleylamine complex was added to a balloon containing a solution of TPP (1.8 g) in DE (2.5 ml) with 200 °C temperature. After elapsing a short time the solution color altered from dark green to black due to formation of colloidal Ni nanoparticles. The resultant solution was kept in 280 °C for 1 hour and then the temperature decreased to the ambient temperature. Consequently pure ethanol (200 ml) was poured to the reaction chamber which caused Ni nanoparticles precipitation. In the following, the precipitate was centrifuged and washed with ethanol (3×50 mL) and then was exposed to the dry air for 24 hours to form NiO nanoparticles and the resultant product was kept at 60 °C temperature.

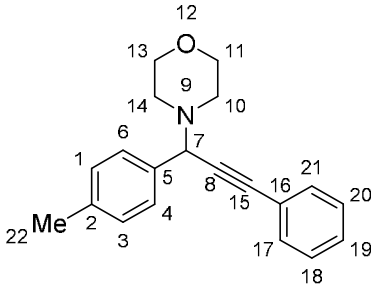
Synthesis of propargylamine derivatives by NiO nanoparticles catalyst

All of the reactions were carried out at 80 °C in a 25 ml one-capped balloon equipped with a magnetic stirring bar in a paraffin bath. Generally, a mixture of the selected aldehyde (1.0 mmol), secondary amine (1.1 mmol) and alkyne (1.2 mmol) was added in the balloon along with the catalytic amount of the NiO nanocrystals (3 mol %, 2.3 mg) as a catalyst. The reaction progress was examined by TLC, and after the completion of the reaction; absolute ethanol (10 mL) was added and the resulting mixture was centrifuged. The catalyst was separated from the reaction mixture by centrifugation and washed with CH_2Cl_2 (3×5 ml) and methanol (3×5 ml) for reusability and recycling in the next runs. The product was purified

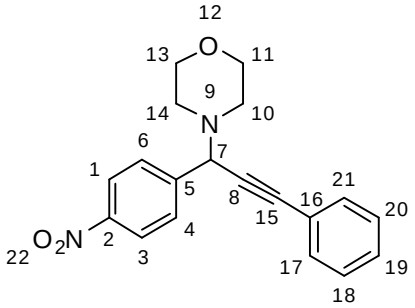
over silica gel by column chromatography (10% EtOAc in hexane) to give the desired propargylamines. All of the products are known compounds and have been reported already.



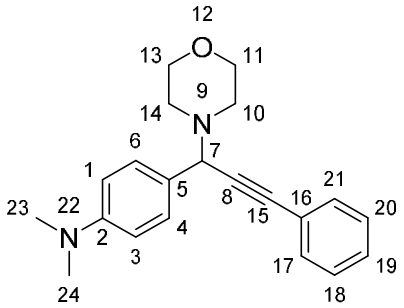
4-(1,3-diphenylprop-2-yn-1-yl)morpholine (4a) Light red oil; ^{13}C NMR ($CDCl_3$): δ 50.35 (C7), 57.21 (C10, C14), 68.54 (C11, C13), 84.08 (C8), 88.49 (C15), 115.17 (C16), 116.31 (C19), 121.96 (C21, C17), 123.67 (C2), 124.82 (C18, C20), 126.16 (C1, C3), 130.43 (C4), 131.87 (C6), 136.54 (C5); ^{1}H NMR ($CDCl_3$): δ 2.67-2.68 (m, 4H, C10H, C14H), 3.77-3.80 (m, 4H, C11H, C13H), 4.84 (s, 1H, C7H), 7.33-7.44 (m, 6H, ArH), 7.56-7.58 (m, 2H, ArH), 7.68-7.70 (m, 2H, ArH); FT-IR (KBr disk): ν cm^{-1} 3059, 3014, 2984, 2957, 2950, 1598, 1489, 1449, 1318, 1280.



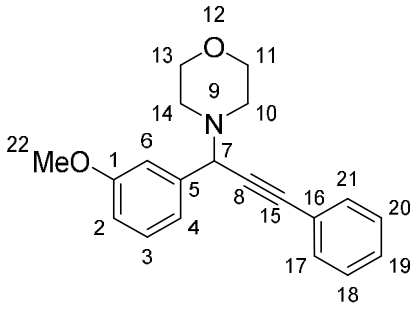
4-(3-phenyl-1-(p-tolyl)prop-2-yn-1-yl)morpholine (4b) Light orange oil; ^{13}C NMR ($CDCl_3$): δ 22.08 (C22), 49.14 (C7), 58.36 (C10, C14), 67.15 (C11, C13), 83.65 (C8), 87.91 (C15), 114.25 (C16), 117.55 (C19), 120.74 (C21, C17), 120.95 (C2), 122.33 (C18, C20), 123.85 (C1, C3), 126.80 (C4), 128.03 (C6), 132.17 (C5); ^{1}H NMR ($CDCl_3$): δ 2.39 (s, 3H, Me), 2.65-2.67 (m, 4H, C10H, C14H), 3.75-3.76 (m, 4H, C11H, C13H), 4.78 (s, 1H, C7H), 7.20-7.22 (m, 2H, ArH), 7.34-7.36 (m, 3H, ArH), 7.53-7.55 (m, 4H, ArH); FT-IR (KBr disk): ν cm^{-1} 3024, 2946, 2925, 2862, 2820, 2230, 1486, 1446, 1314, 1109.



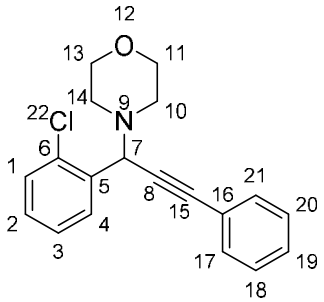
4-(1-(4-nitrophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4c) Yellowish oil; ^{13}C NMR ($CDCl_3$): δ 49.90 (C7), 61.45 (C10, C14), 67.04 (C11, C13), 83.16 (C8), 89.78 (C15), 122.31 (C16), 123.48 (C19), 128.45 (C21, C17), 128.72 (C2), 129.33 (C18, C20), 131.85 (C4, C6), 135.48 (C3, C1), 145.45 (C5), 149.23 (C2); ^{1}H NMR ($CDCl_3$): δ 2.63-2.66 (m, 4H, C10H, C14H), 3.75-3.76 (m, 4H, C11H, C13H), 4.89 (s, 1H, C7H), 7.38-7.39 (m, 3H, ArH), 7.53-7.55 (m, 2H, ArH), 7.87 (d, *J*=8.1 Hz, 2H, ArH), 8.24 (d, *J*=8.1 Hz, 2H, ArH); FT-IR (KBr disk): ν cm^{-1} 3067, 2958, 2854, 2216, 1690, 1522, 1450, 1347, 1275, 1113, 1006.



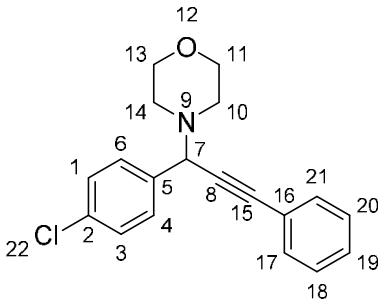
N,N-dimethyl-4-(1-morpholino-3-phenylprop-2-yn-1-yl)aniline (4d) Yellowish oil; ^{13}C NMR ($CDCl_3$): δ 44.11 (C23), 49.25 (C24), 61.70 (C10, C14), 67.03 (C11, C13), 84.15 (C8), 87.54 (C15), 113.85 (C1, C3), 118.80 (C16), 120.41 (C19), 123.74 (C21, C17), 130.40 (C18, C20), 131.21 (C4), 134.38 (C6), 148.01 (C2), 149.21 (C5); ^{1}H NMR ($CDCl_3$): δ 2.62-2.66 (m, 4H, C10H, C14H), 2.97 (s 6H, NMe_2), 3.73-3.74 (m, 4H, C11H, C13H), 4.70 (s, 1H, C7H), 6.73 (d, *J*=8.0 Hz, 2H, ArH), 7.32-7.33 (m, 3H, ArH), 7.45-7.51 (m, 4H, ArH); FT-IR (KBr disk): ν cm^{-1} 3084, 2955, 2892, 2854, 1965, 1611, 1521, 1150.



4-(1-(3-methoxyphenyl)-3-phenylprop-2-yn-1-yl)morpholine (4e) Yellowish oil; ^{13}C NMR ($CDCl_3$): δ 49.99 (C7), 55.24 (C22), 62.01 (C10, C14), 67.20 (C11, C13), 85.15 (C8), 88.54 (C15), 113.10 (C2), 114.39 (C6), 120.99 (C4), 123.03 (C16), 128.36 (C21), 128.42 (C17), 129.28 (C18, C20), 131.88 (C19), 132.17 (C3), 139.57 (C5), 159.71 (C1); ^{1}H NMR ($CDCl_3$): δ 2.66-2.67 (m, 4H, C10H, C14H), 3.77-3.79 (m, 4H, C11H, C13H), 3.86 (s, 4H, OCH_3), 4.79 (s, 1H, C7H), 6.86 (s, 1H, C6H), 7.25-7.36 (m, 6H, ArH), 7.53-7.54 (m, 2H, ArH); FT-IR (KBr disk): ν cm^{-1} 3057, 2995, 2851, 1965, 1599, 1486, 1449, 1317, 1150, 1048.

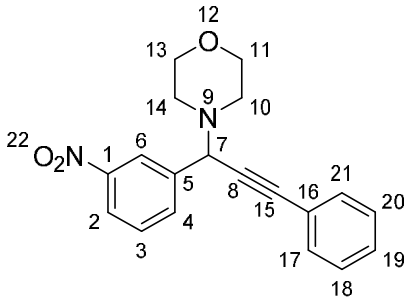


4-(1-(2-chlorophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4f) Yellow oil; ^{13}C NMR ($CDCl_3$): δ 49.87 (C7), 58.96 (C10, C14), 67.14 (C11, C13), 84.70 (C8), 88.40 (C15), 122.82 (C16), 125.58 (C3), 126.39 (C18, C20), 128.38 (C21), 128.41 (C17), 129.18 (C19), 130.58 (C2), 130.93 (C1), 131.85 (C4), 134.69 (C6), 135.56 (C5); ^{1}H NMR ($CDCl_3$): δ 2.66-2.67 (m, 4H, C10H, C14H), 3.77-3.79 (m, 4H, C11H, C13H), 3.86 (s, 4H, OCH_3), 4.79 (s, 1H, C7H), 6.86 (s, 1H, C6H), 7.25-7.36 (m, 6H, ArH), 7.53-7.54 (m, 2H, ArH); FT-IR (KBr disk): ν cm^{-1} 3047, 2997, 2897, 2750, 1562, 1472, 1452, 1324, 1274, 1117, 1055.

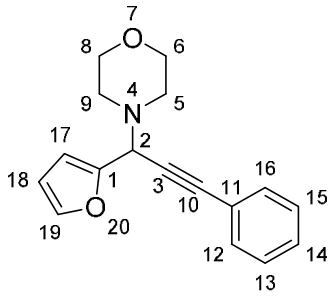


4-(1-(4-chlorophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4g) Yellow oil; ^{13}C NMR ($CDCl_3$): δ 49.85 (C7), 61.40 (C10, C14), 67.15 (C11, C13), 84.43 (C8), 88.98 (C15), 122.77 (C16), 128.43 (C1, C3), 128.48 (C18, C20), 129.70 (C19), 129.94 (C17, C21), 131.88 (C4, C6), 133.61 (C2), 136.53 (C5); ^{1}H NMR ($CDCl_3$): δ 2.61-2.62 (m, 4H, C10H, C14H), 3.73-

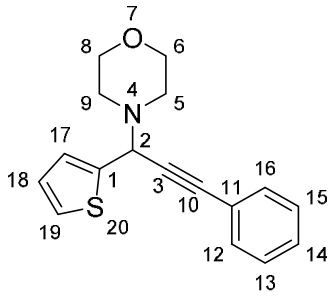
3.75 (m, 4H, C11H, C13H), 4.77 (s, 1H, C7H), 7.36-7.37 (m, 5H, ArH), 7.51-7.52 (m, 2H, ArH), 7.58-7.60 (m, 2H, ArH); FT-IR (KBr disk): ν cm^{-1} 3070, 3029, 2957, 2857, 1494, 1454, 1428, 1113, 1075, 1034.



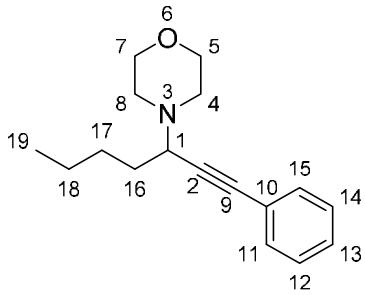
4-(1-(3-nitrophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4h) Light yellow oil; ^{13}C NMR ($CDCl_3$): δ 49.78 (C7), 61.24 (C10, C14), 66.95 (C11, C13), 83.17 (C8), 89.76 (C15), 122.36 (C16), 122.85 (C21, C17), 123.41 (C18, C20), 128.39 (C19), 128.71 (C6), 129.15 (C2), 131.83 (C3), 134.48 (C4), 140.36 (C5), 148.39 (C1); ^{1}H NMR ($CDCl_3$): δ 2.63-2.69 (m, 4H, C10H, C14H), 3.76-3.78 (m, 4H, C11H, C13H), 4.90 (s, 1H, C7H), 7.34-7.38 (m, 3H, ArH), 7.54-7.62 (m, 3H, ArH), 8.02 (d, *J*=7.6 Hz, 1H, C2H), 8.18 (d, *J*=7.7 Hz, 1H, C4H), 8.56 (s, 1H, C6H); FT-IR (KBr disk): ν cm^{-1} 3085, 3028, 3002, 2986, 2882, 1506, 1473, 1419, 1263, 1208, 1168, 1121, 1045, 1014.



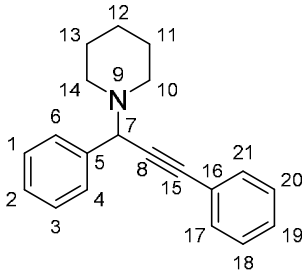
4-(1-(furan-2-yl)-3-phenylprop-2-yn-1-yl)morpholine (4i) Yellowish white oil; ^{13}C NMR ($CDCl_3$): δ 49.61 (C2), 56.12 (C5, C9), 66.95 (C6, C8), 82.85 (C3), 87.02 (C10), 109.76 (C17), 110.13 (C18), 122.57 (C11), 128.35 (C13, C15), 128.50 (C12, C16), 131.87 (C14), 142.87 (C19), 150.76 (C1); ^{1}H NMR ($CDCl_3$): δ 2.63-2.72 (m, 4H, C5H, C9H), 3.74-3.83 (m, 4H, C6H, C8H), 4.89 (s, 1H, C2H), 6.37 (t, *J*=3 Hz, 1H, C18H), 6.52 (d, J=2.8 Hz, 1H, C17H), 7.31-7.35 (m, 3H, ArH), 7.45-7.52 (m, 3H, ArH); FT-IR (KBr disk): ν cm^{-1} 3063, 3028, 2932, 1604, 1495, 1453, 1261, 1152, 1028.



4-(3-phenyl-1-(thiophen-2-yl)prop-2-yn-1-yl)morpholine (4j) White oil; ^{13}C NMR ($CDCl_3$): δ 49.69 (C2), 57.83 (C5, C9), 67.15 (C6, C8), 84.29 (C3), 87.63 (C10), 122.69 (C11), 125.57 (C18), 125.87 (C17), 126.36 (C16), 126.44 (C12), 128.39 (C13), 128.48 (C15), 128.84 (C14), 131.89 (C19), 142.80 (C1); ^{1}H NMR ($CDCl_3$): δ 2.66-2.74 (m, 4H, C5H, C9H), 3.73-3.82 (m, 4H, C6H, C8H), 5.01 (s, 1H, C2H), 6.97-6.99 (m, 1H, C18H), 7.25-7.27 (m, 1H, C17H), 7.30-7.31 (m, 1H, C19H), 7.34-7.36 (m, 3H, ArH), 7.51-7.54 (m, 2H, ArH); FT-IR (KBr disk): ν cm^{-1} 3062, 3028, 2955, 2934, 2248, 1607, 1490, 1454, 1125, 1109, 1065, 1016.



4-(1-phenylhept-1-yn-3-yl)morpholine (4k) White oil; ^{13}C NMR ($CDCl_3$): δ 14.21 (C19), 21.70 (C18), 25.24 (C17), 34.47 (C16), 54.14 (C1), 57.30 (C4, C8), 67.79 (C5, C7), 87.45 (C2), 88.21 (C9), 123.64 (C10), 126.87 (C15, C11), 128.45 (C13), 129.70 (C12, C14); ^{1}H NMR ($CDCl_3$): δ 1.02 (t, J=7.0 Hz, 3H, C19H), 1.38-1.39 (m, 4H, C17H, C18H), 1.60-1.62 (m, 2H, C16H), 2.95-2.96 (m, 4H, C4H, C8H), 3.63-3.68 (m, 4H, C5H, C7H), 3.81-3.82 (m, 1H, C1H), 7.45-7.48 (m, 3H, ArH), 7.69-7.72 (m, 2H, ArH); FT-IR (KBr disk): ν cm^{-1} 3035, 3020, 2964, 2874, 2234, 1568, 1479, 1439, 1328, 1263, 1184, 1120, 1064.



1-(1,3-diphenylprop-2-yn-1-yl)piperidine (4l) Red oil; ^{13}C NMR ($CDCl_3$): δ 24.15 (C12), 26.07 (C11, C13), 52.47 (C7), 56.11 (C10, C14), 82.18 (C15), 87.19 (C8), 121.10 (C16), 126.01 (C19), 126.86 (C21, C17), 127.24 (C2), 127.60 (C18, C20), 128.74 (C1, C3), 129.03 (C4), 129.17 (C6), 138.41 (C5); ^{1}H NMR ($CDCl_3$): δ 1.45-1.58 (m, 6H, C11H, C12H, C13H), 2.38-2.41 (m, 4H, C10H, C14H), 4.94 (s, 1H, C7H), 7.33-7.94 (m, 10H, ArH); FT-IR (KBr disk): ν cm^{-1} 3084, 3020, 2994, 2967, 1452, 1408, 1349, 1319, 1300.

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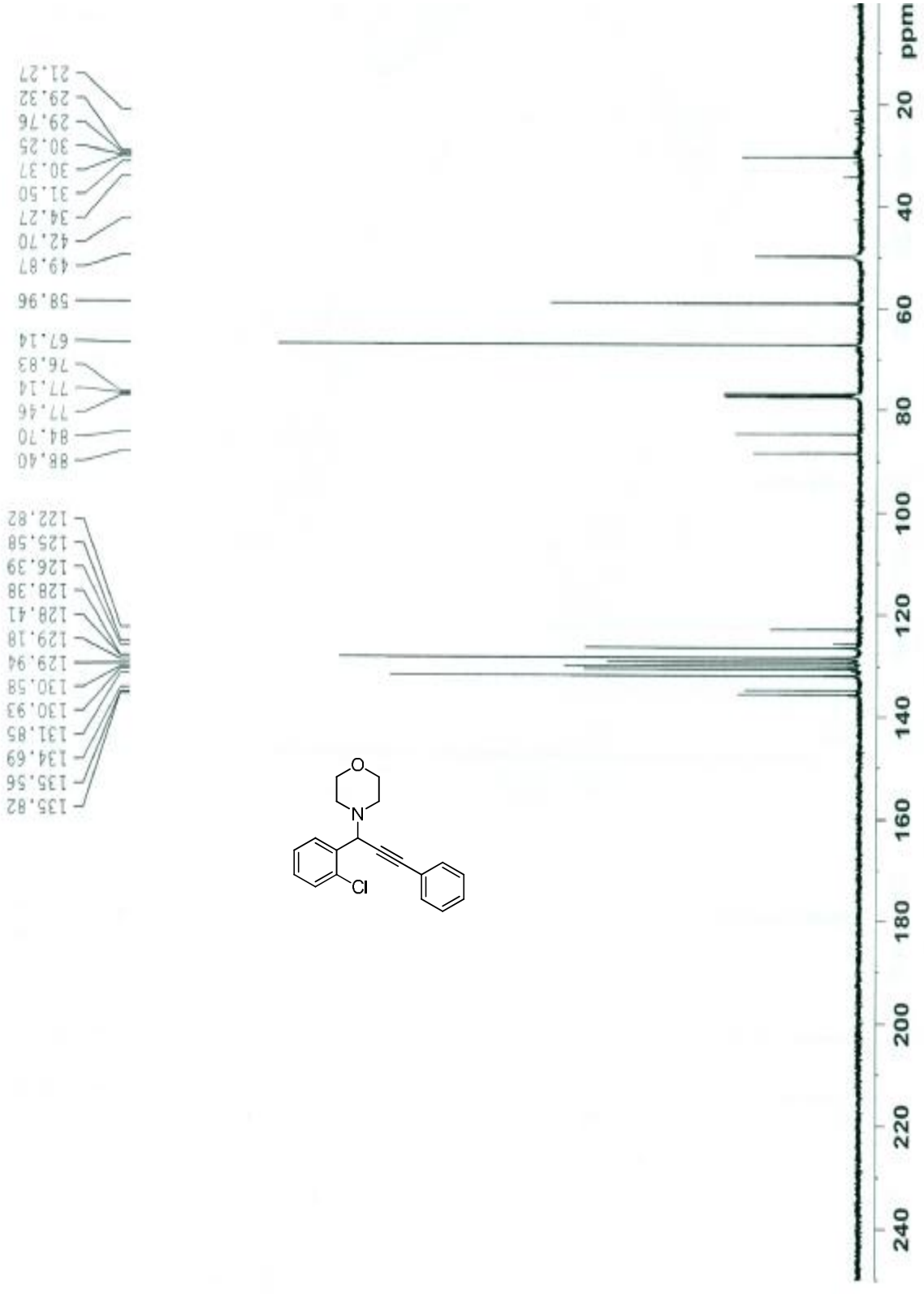
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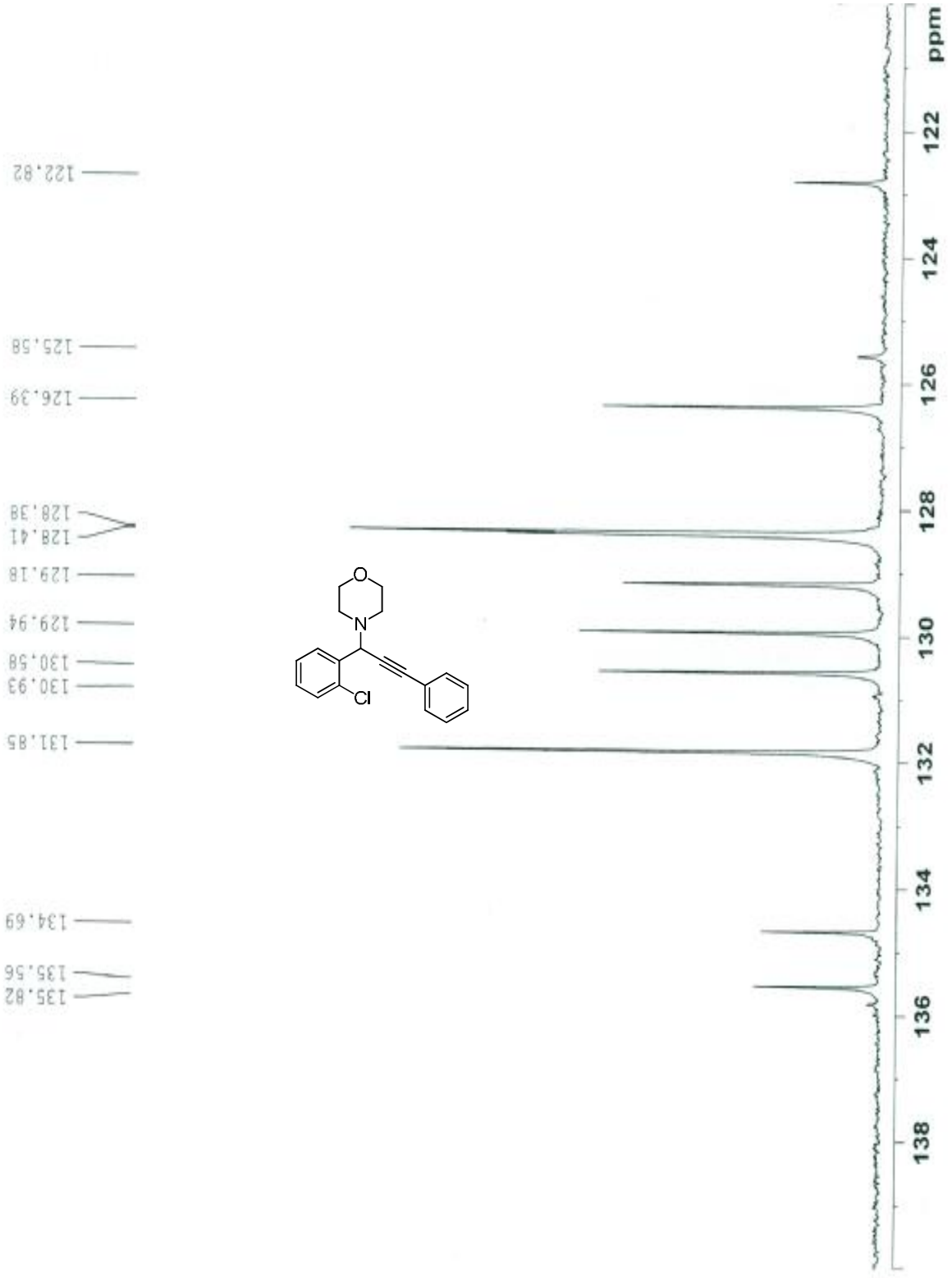
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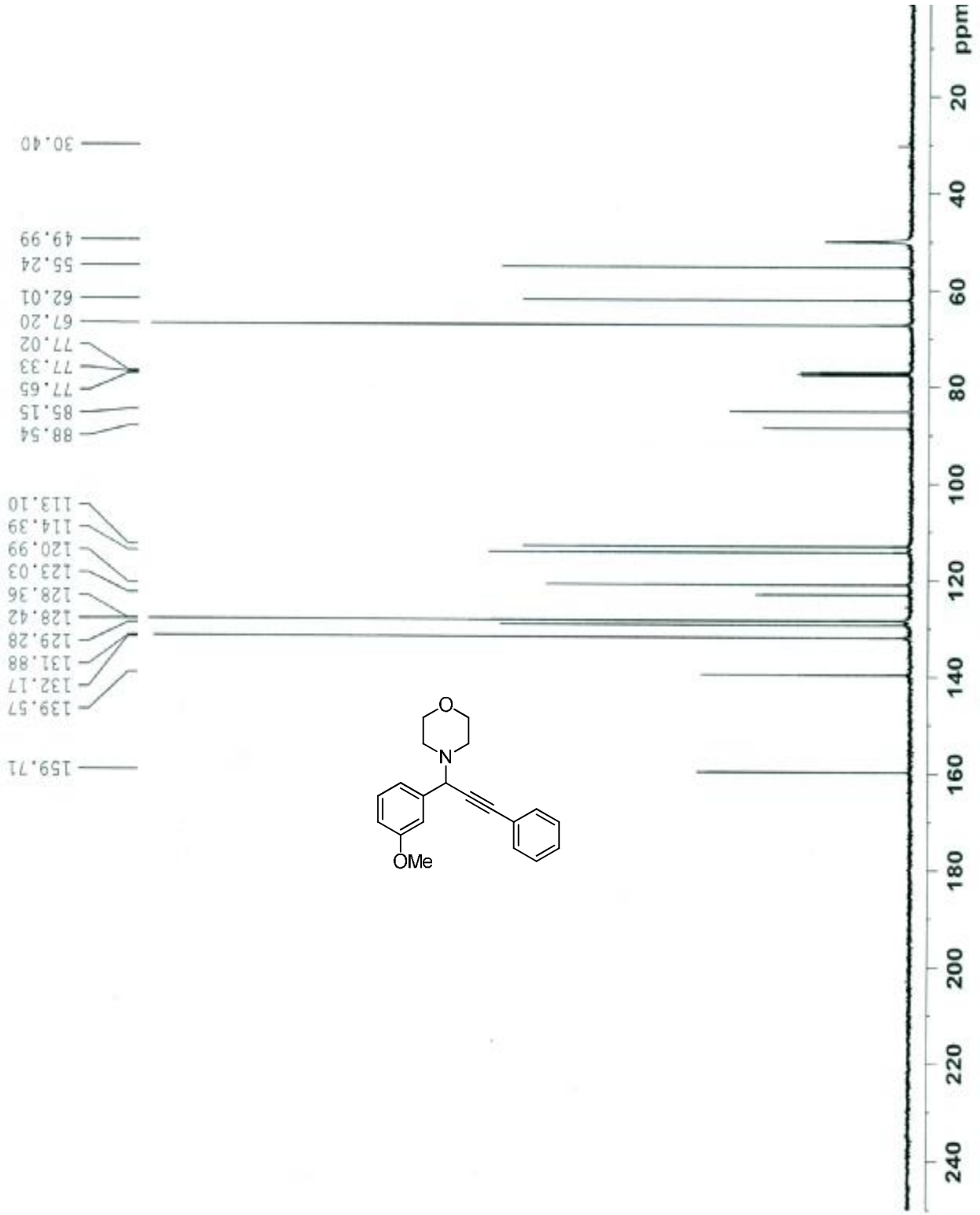
OMe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128.42
129.28
131.88
132.17
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OMe
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140
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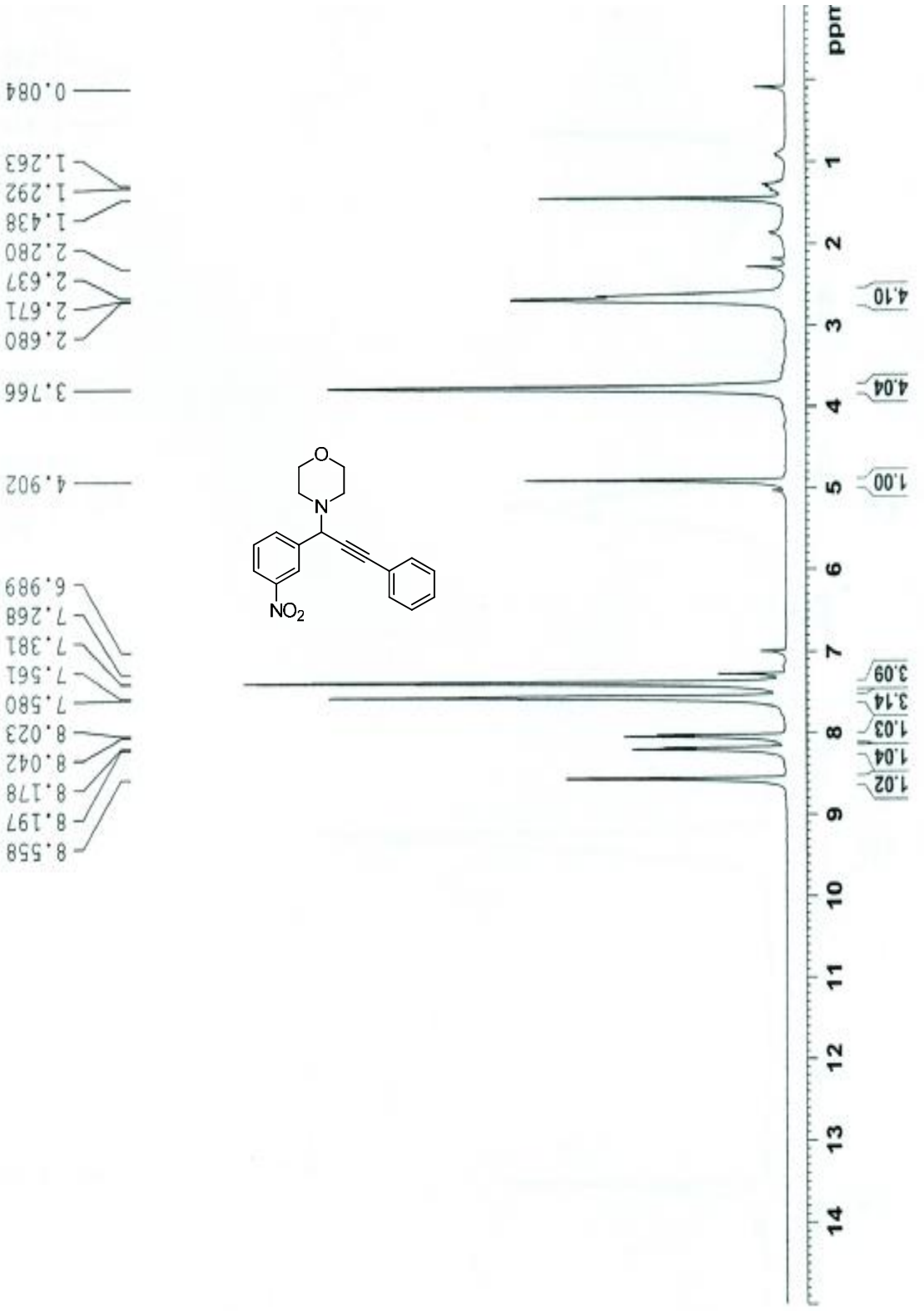
20
ppm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7.561
7.580
8.023
8.042
8.178
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3.09
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1.04
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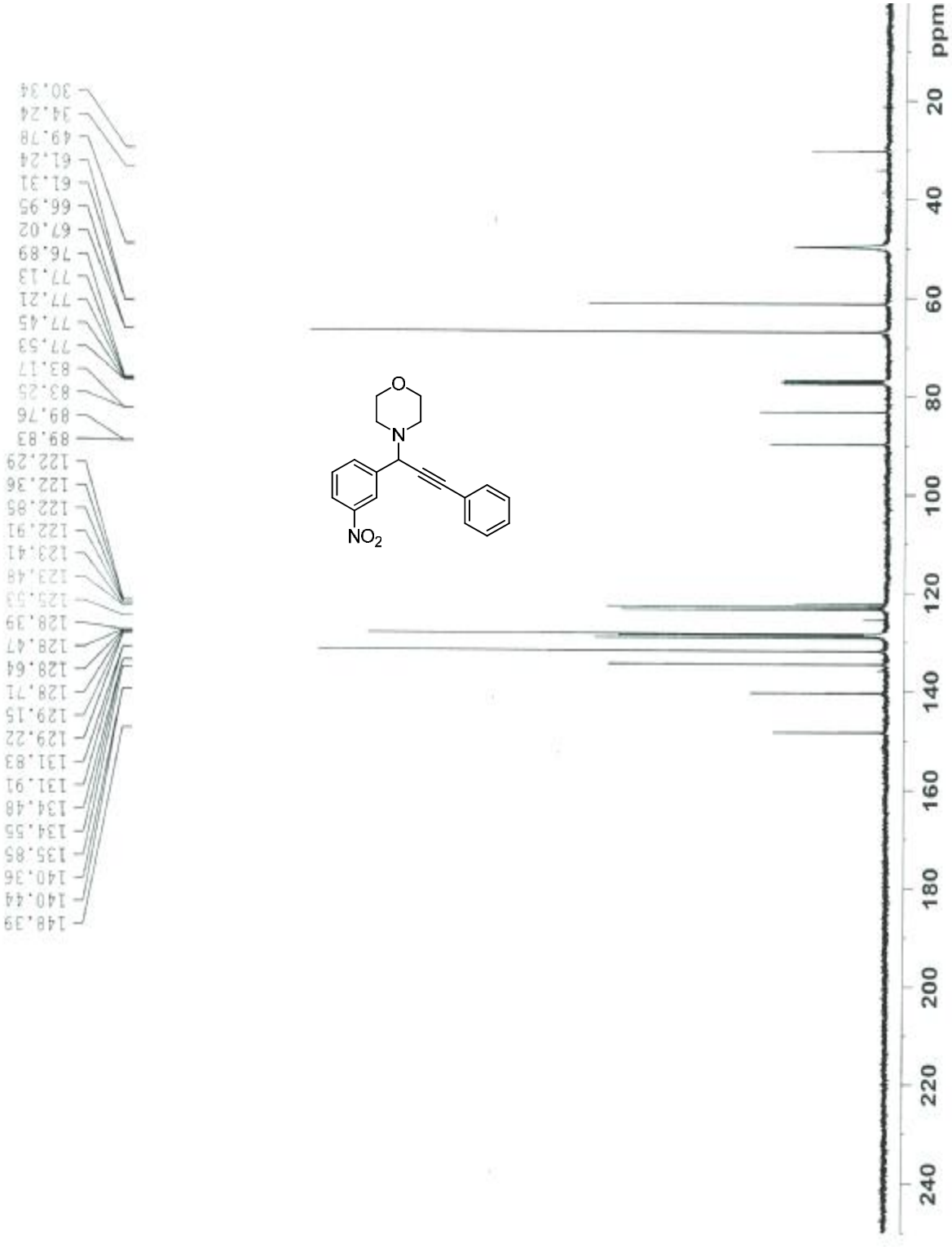
NO2
ppm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83.25
89.76
89.83
122.29
122.36
122.85
122.91
123.41
123.48
125.53
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128.47
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129.15
129.22
131.83
131.91
134.48
134.55
135.85
140.36
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200
220
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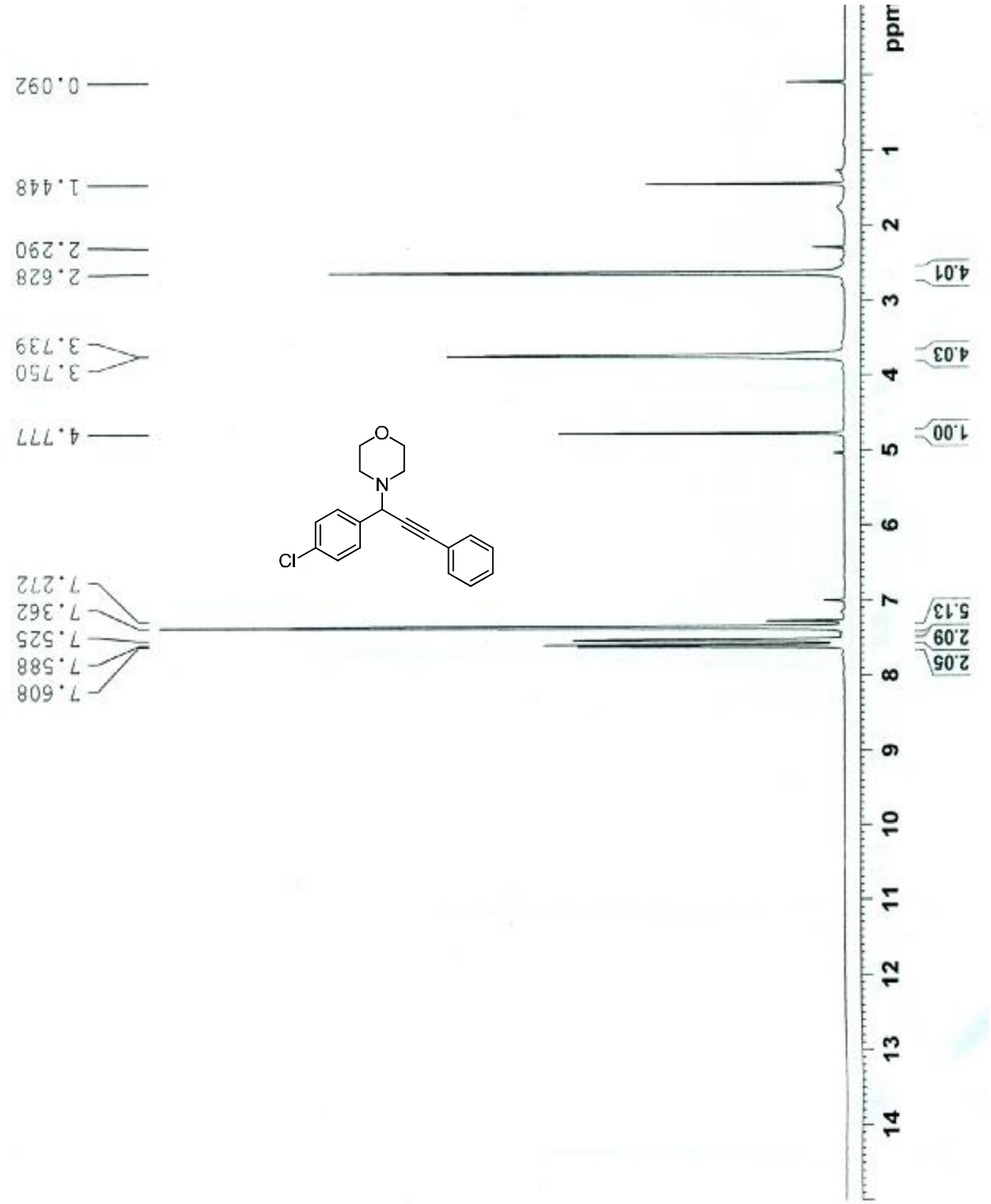
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7.272
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4.01
4.03
1.00
5.13
2.09
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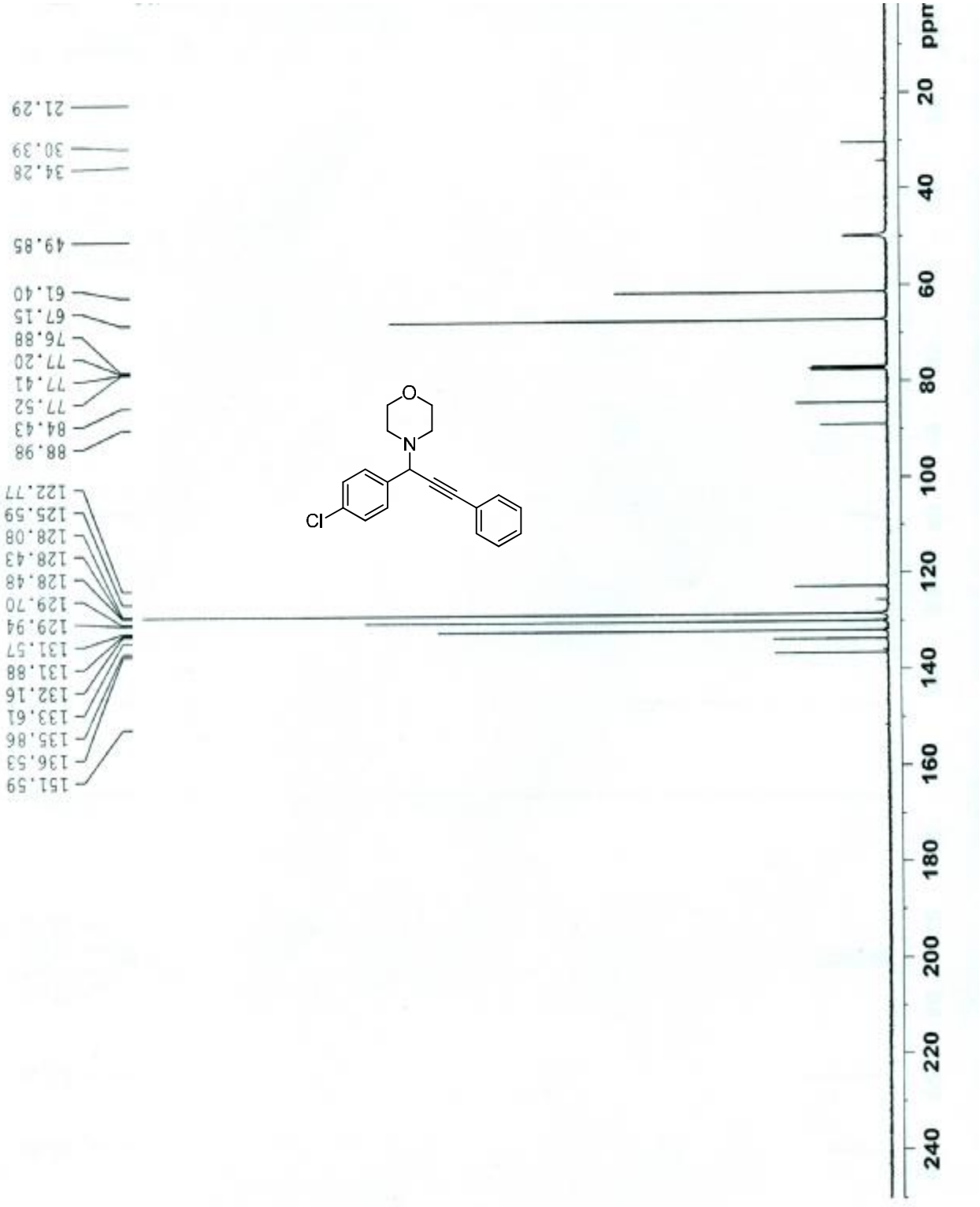
N
Cl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125.59
128.08
128.43
128.48
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129.94
131.57
131.88
132.16
133.61
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Cl
ppm
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100
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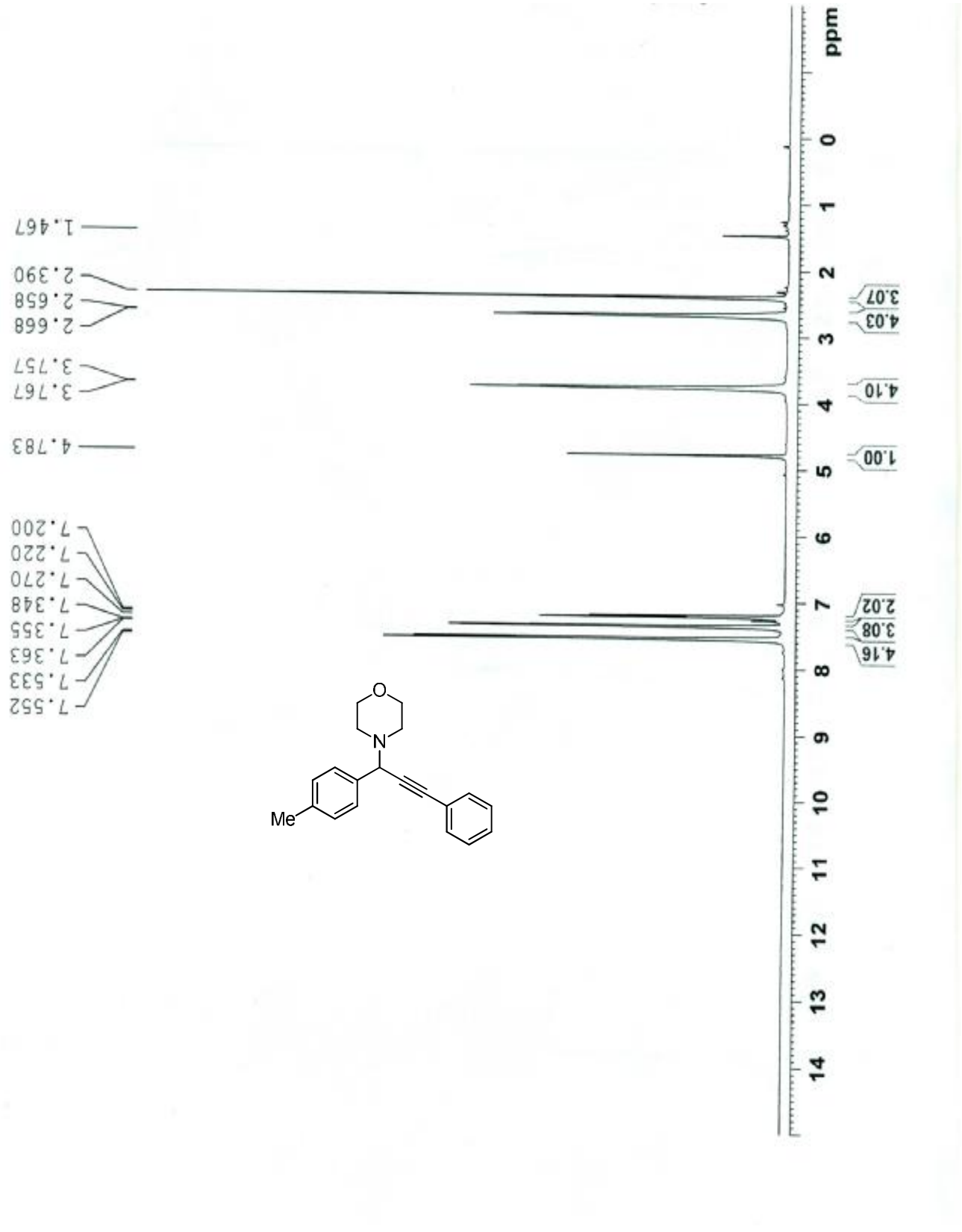
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2.02
3.08
4.16
1.467
2.390
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2.668
3.757
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7.220
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7.363
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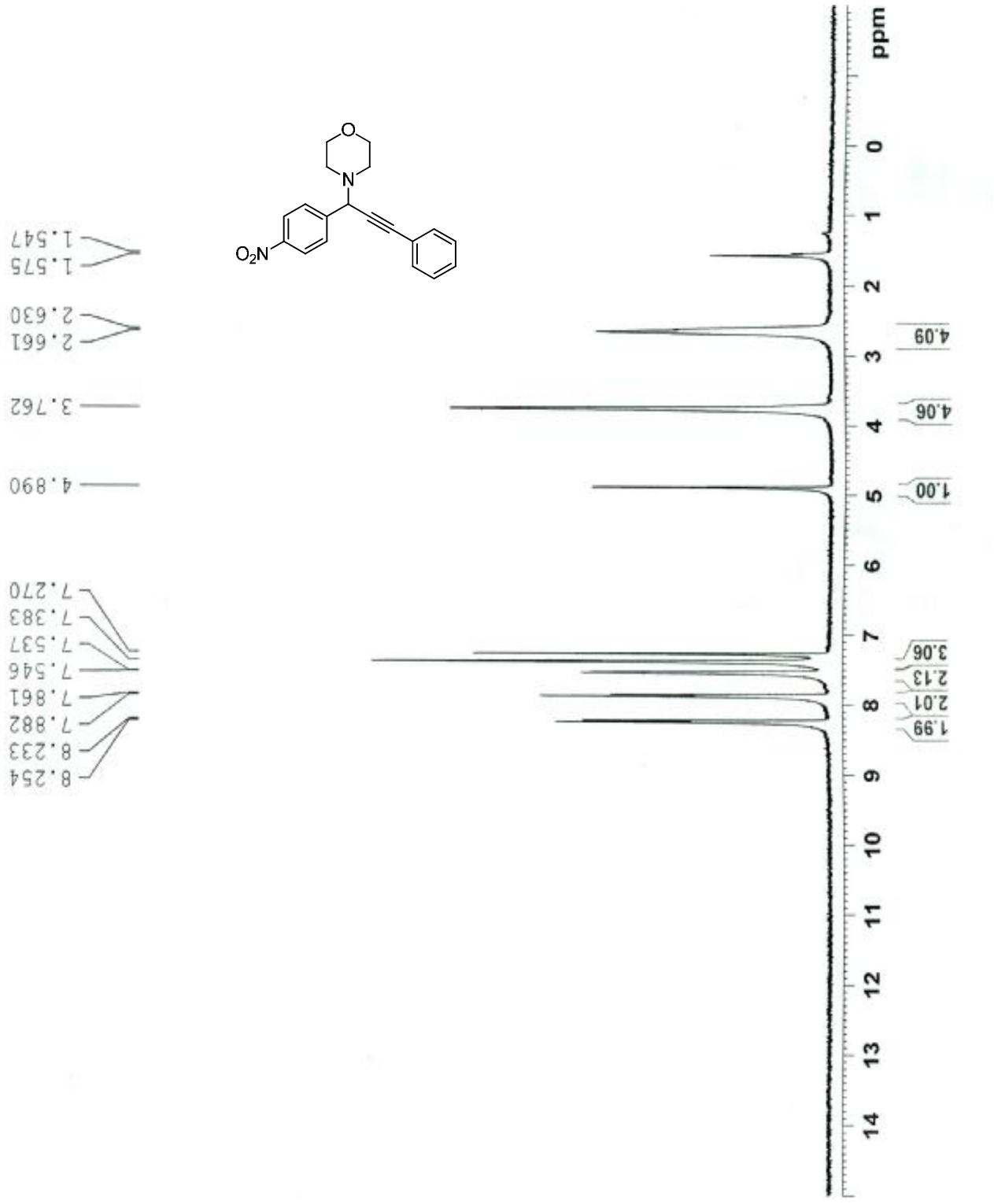
O
N
Me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7.383
7.537
7.546
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7.882
8.233
8.254
O
N
O2N
ppm
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2
3
4
5
6
7
8
9
10
11
12
13
14
4.09
4.06
1.00
3.06
2.13
2.01
1.99

7.270
7.383
7.537
7.546
7.861
7.882
8.233
8.254
O
N
O2N
ppm
7.2
7.3
7.4
7.5
7.6
7.7
7.8
7.9
8.0
8.1
8.2
8.3
8.4
3.06
2.13
2.01
1.99

49.90
61.45
67.04
76.72
77.04
77.36
83.16
89.78
122.31
123.48
128.45
128.72
129.33
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N
O2N
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220
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140
120
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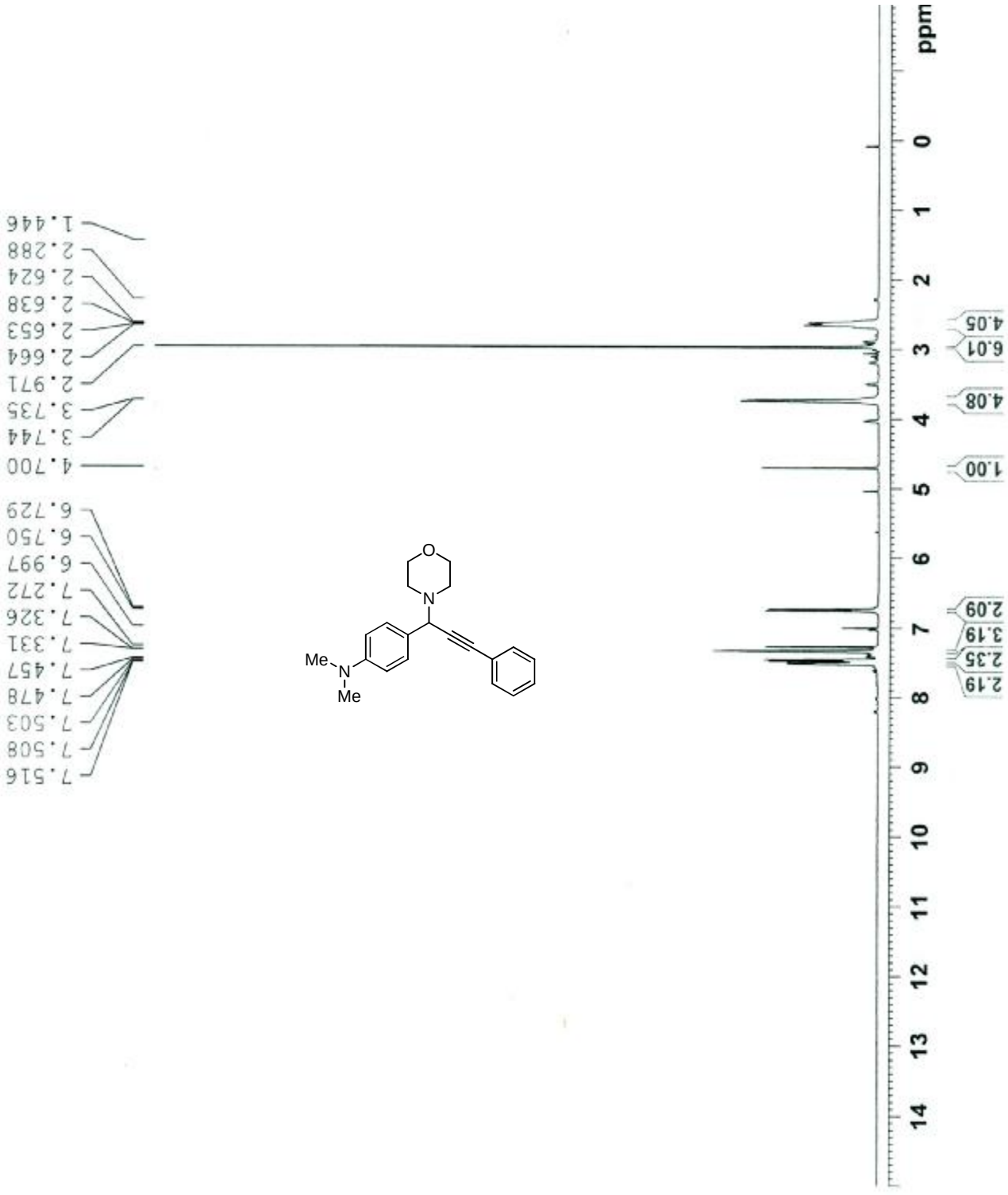
80
60
40
20
ppm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4.05
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2.288
2.624
2.638
2.653
2.664
2.971
3.735
3.744
4.700
6.729
6.750
6.997
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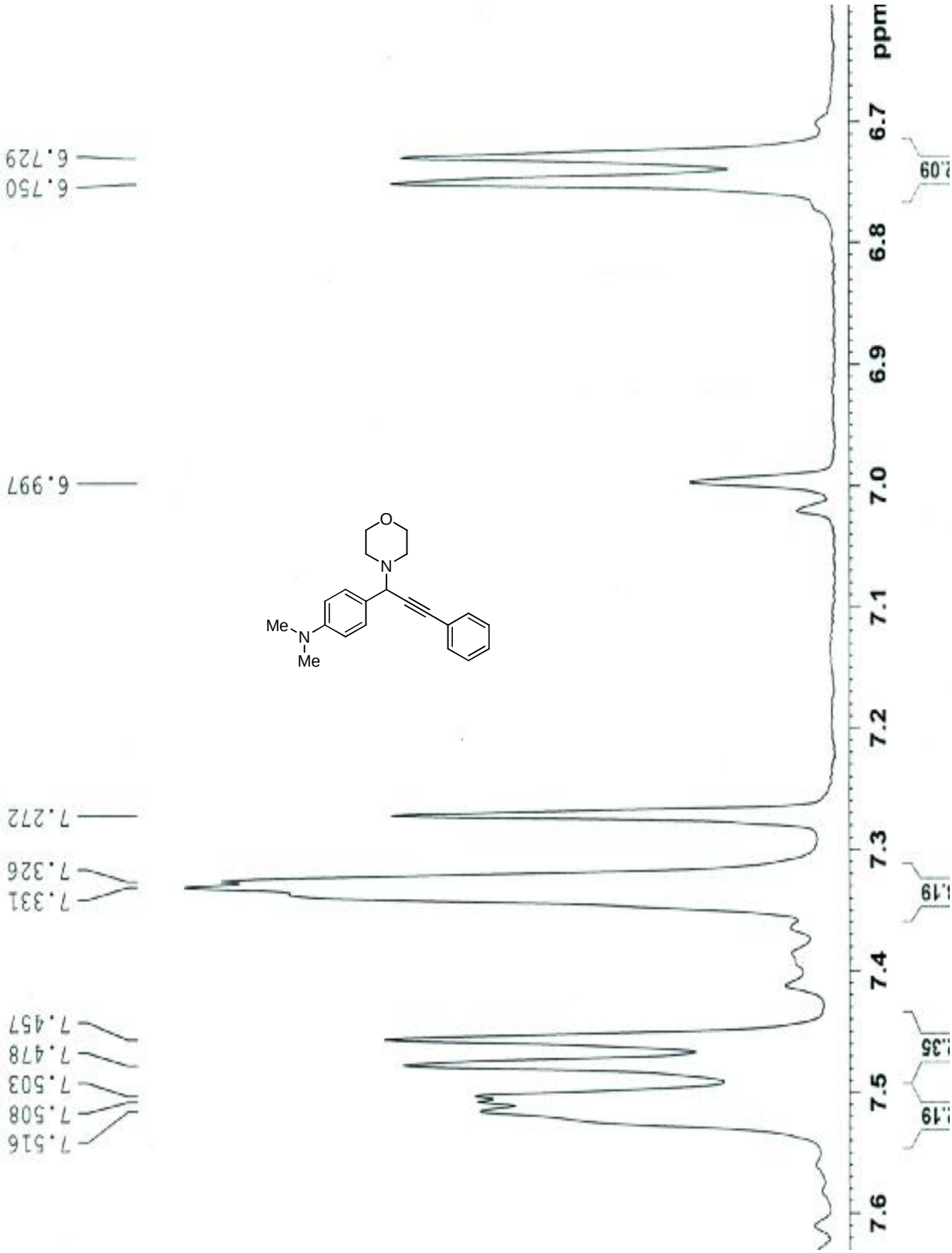
7.326
7.331
7.457
7.478
7.503
7.508
7.516
O
N
Me
N
Me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6.997
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7.326
7.331
7.457
7.478
7.503
7.508
7.516
O
N
Me
N
Me
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6.8
6.9
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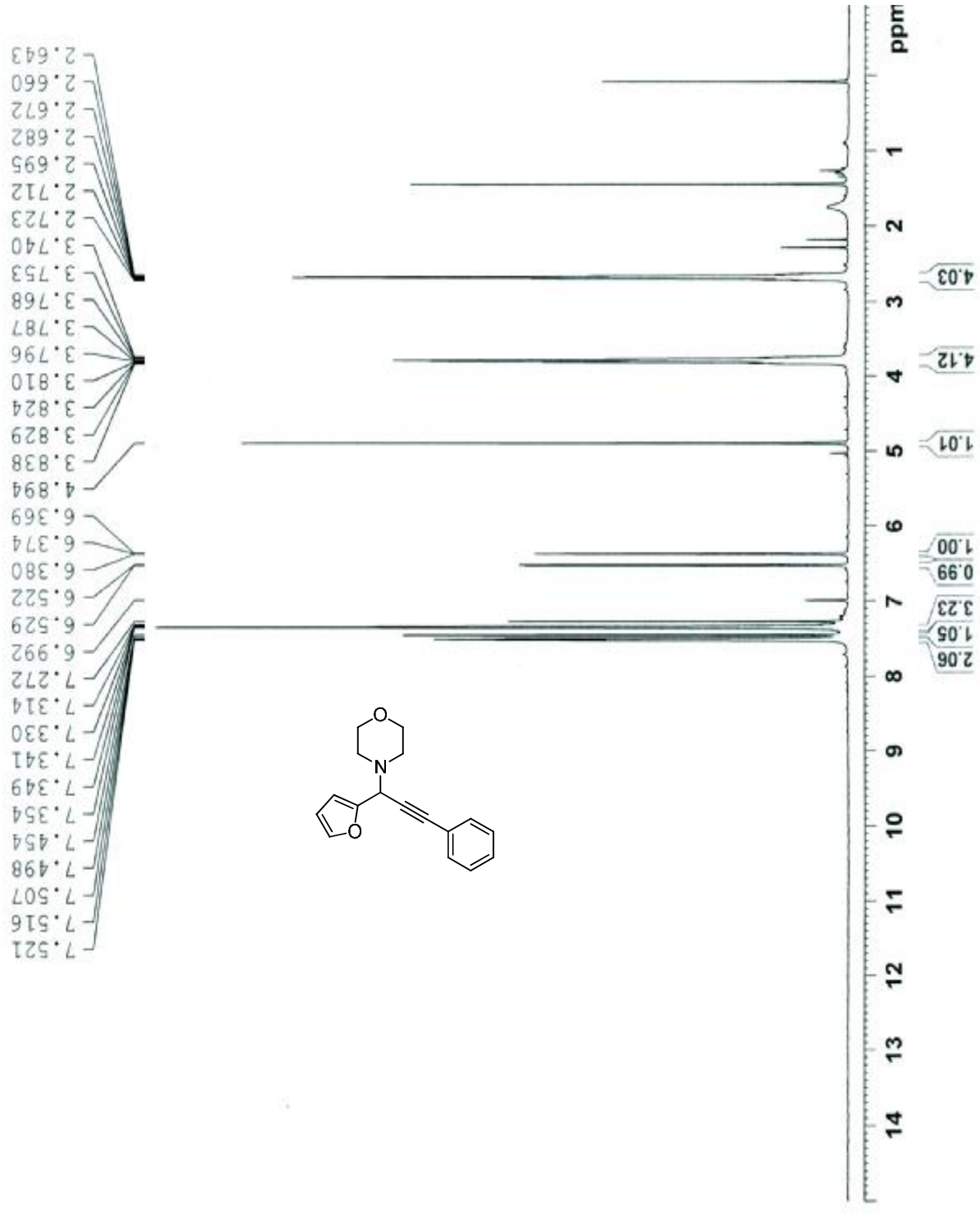
7.3
7.4
7.5
7.6
ppm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3.796
3.810
3.824
3.829
3.838
4.894
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6.374
6.380
6.522
6.529
6.992
7.272
7.314
7.330
7.341
7.349
7.354
7.454
7.498
7.507
7.516
7.521
4.03
4.12
1.01
1.00
0.99
3.23
1.05
2.06
ppm
1
2
3
4
5
6
7
8
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10
11
12
13
14
O
N
O


30.33
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66.95
76.80
77.12
77.44
82.85
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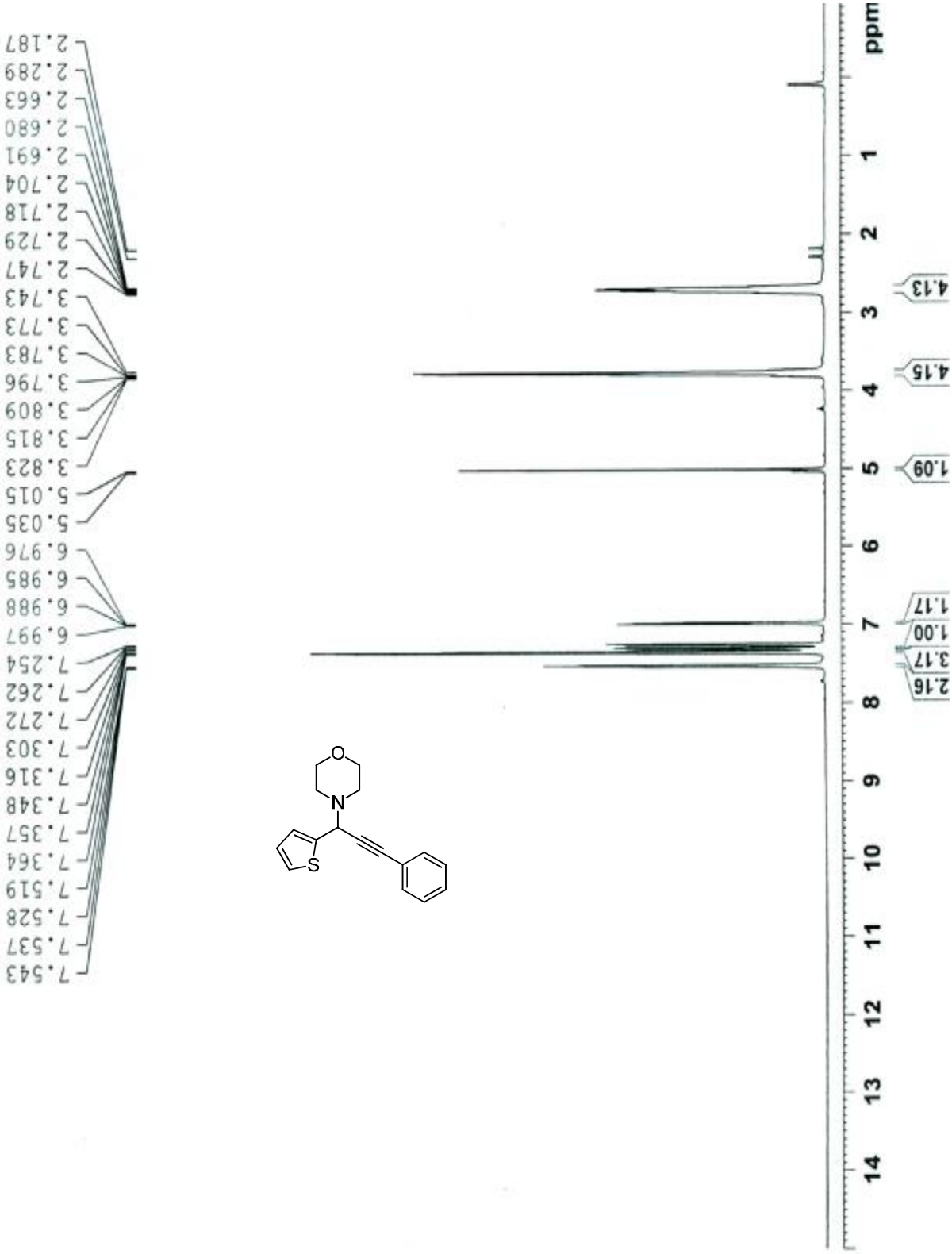
O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3.796
3.809
3.815
3.823
5.015
5.035
6.976
6.985
6.988
6.997
7.254
7.262
7.272
7.303
7.316
7.348
7.357
7.364
7.519
7.528
7.537
7.543
O
N
S
ppm
1
2
3
4
5
6
7
8
9
10
11
12
13
14
4.13
4.15
1.09
1.17
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3.809
3.815
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ppm
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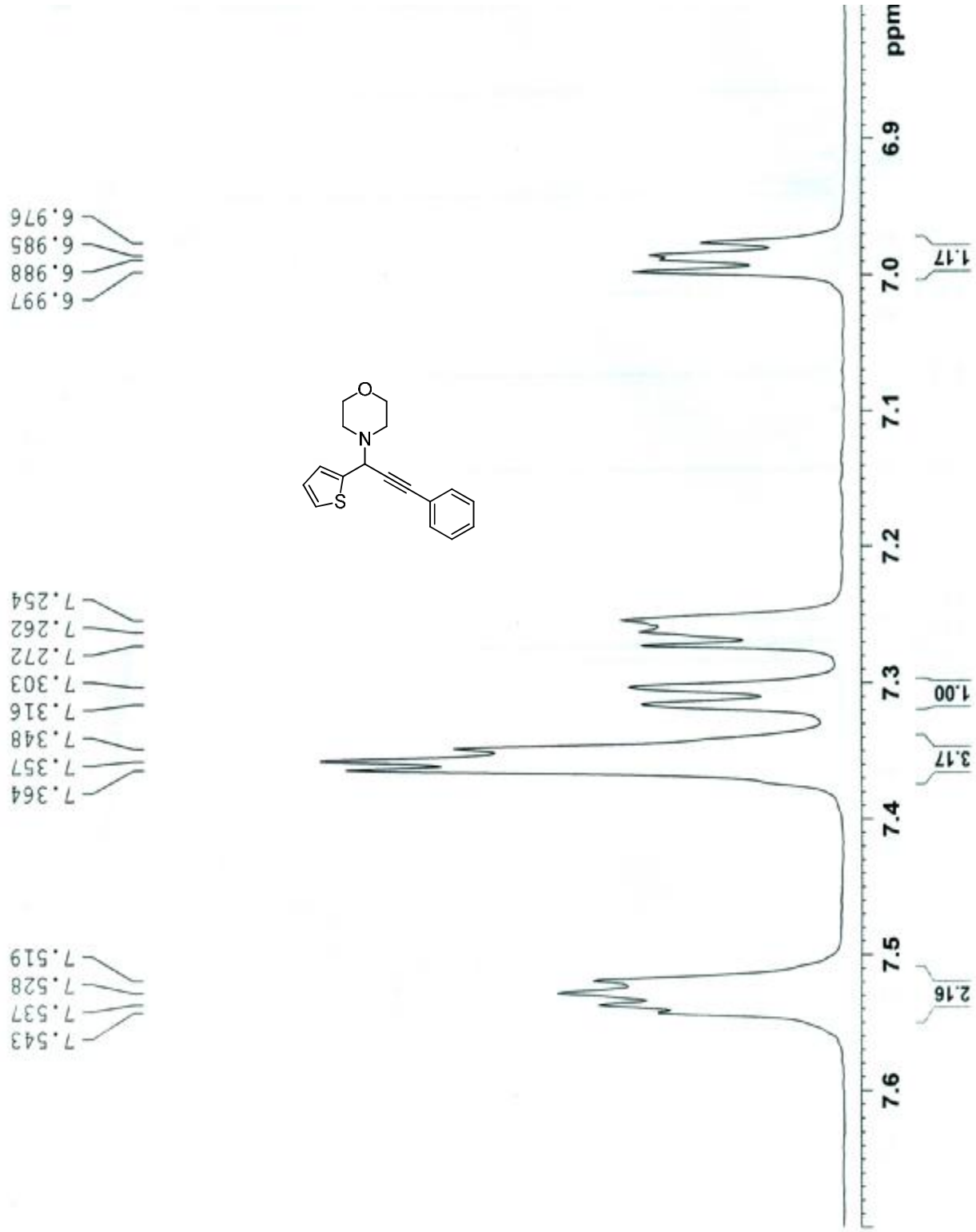
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7.519
7.528
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O
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S
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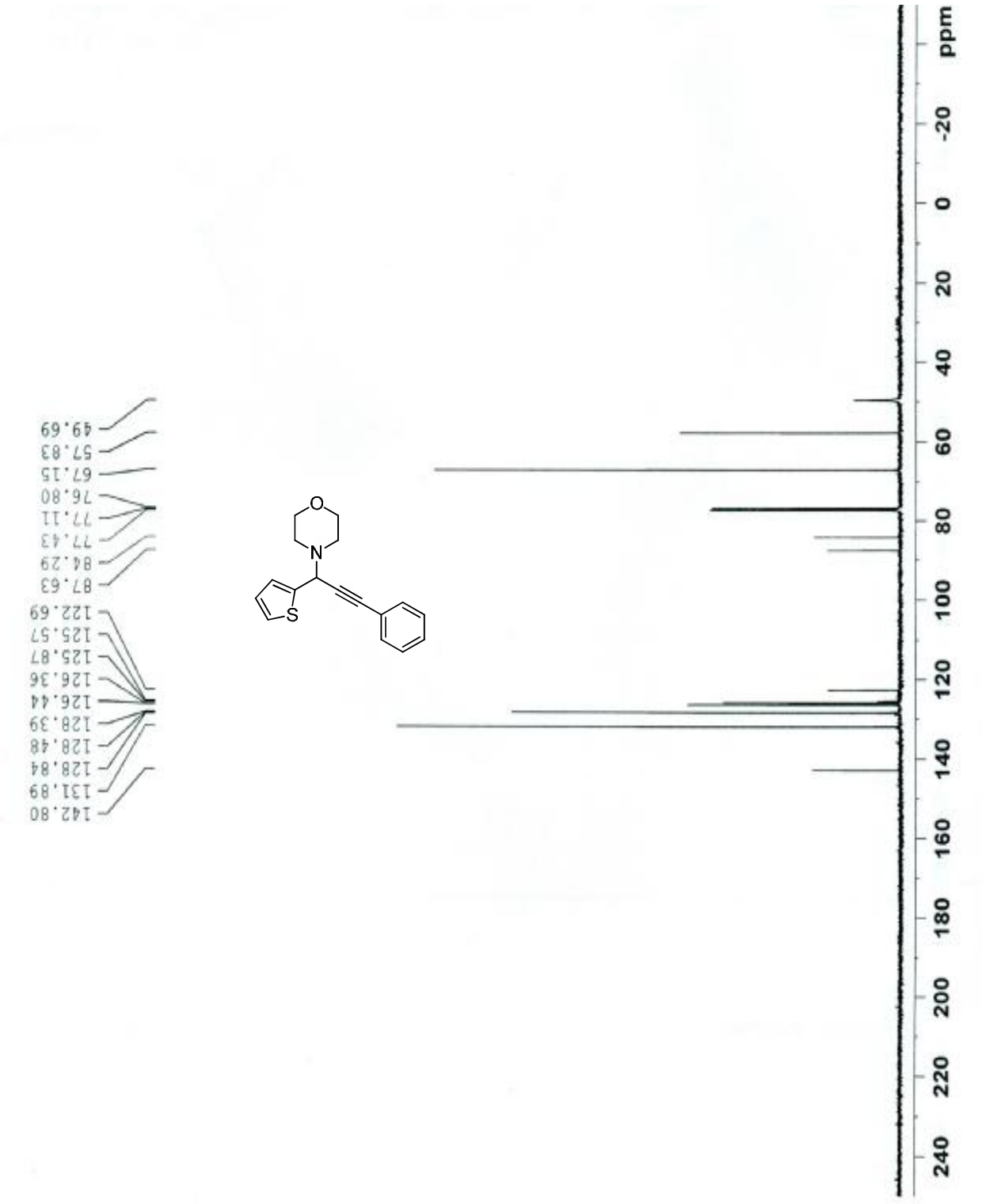
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131.89
142.80
O
N
S
ppm
240
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200
180
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140
120
100
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60
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