Supporting information

Single-pot triple catalytic transformations based on coupling of *in situ* generated allyl boronates with *in situ* hydrolyzed acetals

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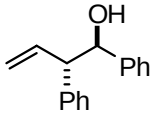
Contents:

Experimental Procedures and Characterization of the Prepared Compounds

The ^{1}H-NMR and ^{13}C-NMR spectra were recorded in $CDCl_3$ (internal standard: 7.26 ppm, ^{1}H; 77.00 ppm, ^{13}C) at room temperature using 400 MHz NMR spectrometer. Accurate mass data were obtained using ESI technique. The applied chemicals were obtained from commercial sources or synthesized according to literature procedures: **2a**[1], **2b**[2], **3e**[3], **3f**[4], **4c**[5], **5a**[5], **5b**[6] and **6**[7].

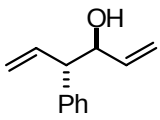
General Procedure for One-Pot Allylation of Acetals (Tables 1 and 2).

The corresponding allyl substrate **4-6** (0.15 mmol) was dissolved in a mixture of DMSO, MeOH and water (0.2/0.2/0.1 mL) followed by the addition of bis(pinacolato)diboron **1a** (0.18 mmol), palladium catalyst **2** (0.0075 mmol, 5 mol %), *p*-toluenesulfonic acid (0.03 mmol, 20 mol %), and acetal **3** (0.18 mmol). This reaction mixture was stirred for the allotted temperatures and times listed in Table 1 and 2. Thereafter, the reaction mixture was quenched by water and extracted with chloroform. After evaporation of the organic phase, product **7** was purified by silica gel chromatography. All one-pot reactions were performed without using inert atmosphere or application of carefully dried solvents.

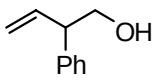


1,2-Diphenyl-3-buten-1-ol (7a). This compound was prepared according to the above general procedure except that water was not used and the amount of *p*-toluenesulfonic acid was reduced to 5 mol %. The NMR data obtained for **7a** are in agreement with the previously reported[1] values. ^{1}H NMR ($CDCl_3$): 7.18 (m, 8H), 7.07 (m, 2H), 6.27 (ddd, J = 8.7, 10.2, 17.0 Hz, 1H), 5.28 (d, J = 10.2 Hz, 1H), 5.24 (d, J = 17.0 Hz, 1H), 4.86 (d, J = 8.1 Hz, 1H), 3.56 (dd, J =

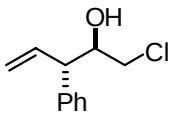
8.1, 8.7 Hz, 1H), 2.34 (br, 1H); ^{13}C NMR ($CDCl_3$): 141.8, 140.6, 137.8, 128.34, 128.31, 127.9, 127.4, 126.7, 126.6, 118.4, 77.2, 59.2; HRMS (ESI): calcd for $[C_{16}H_{16}O + Na]^+$, *m*/*z*, 247.1093; found, 247.1090.



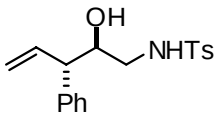
4-Phenyl-1,5-hexadien-3-ol (7b). This compound was prepared according to the above general procedure except that only 1.2 mmol of water was used and that all components, but acetal **3b**, were first stirred for 16 h at 50^{o}C, and. Thereafter, acetal **3b** was added and the stirring was continued for another 20 h at 50^{o}C. ^{1}H NMR ($CDCl_3$): 7.32 (m, 2H), 7.23 (m, 3H), 6.15 (ddd, *J* = 8.6, 10.4, 17.1 Hz, 1H), 5.75 (ddd, *J* = 5.9, 10.5, 17.1 Hz, 1H), 5.25 (d, *J* = 10.5 Hz, 1H), 5.21 (d, *J* = 17.1 Hz, 1H), 5.19 (d, *J* = 17.1 Hz, 1H), 5.08 (d, *J* = 10.4 Hz, 1H), 4.34 (m, 1H), 3.36 (dd, *J* = 8.6, 8.6 Hz, 1H), 1.90 (d, *J* = 3.6 Hz, 1H); ^{13}C NMR ($CDCl_3$): 140.6, 138.3, 137.9, 128.6, 128.4, 126.8, 118.1, 116.0, 75.2, 57.3; HRMS (ESI): calcd for $[C_{12}H_{14}O + Na]^+$, *m*/*z*, 197.0937; found, 197.0940.



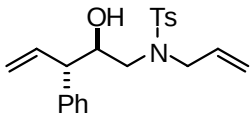
2-Phenyl-3-buten-1-ol (7c). This compound was prepared according to the above general procedure except that only 1.2 mmol of water was used. The NMR data obtained for **7c** are in agreement with the previously reported[8] values. ^{1}H NMR ($CDCl_3$): 7.34 (m, 2H), 7.25 (m, 3H), 6.02 (ddd, *J* = 7.6, 10.4, 17.2 Hz, 1H), 5.22 (d, *J* = 10.4 Hz, 1H), 5.19 (d, *J* = 17.2 Hz, 1H), 3.84 (m, 2H), 3.55 (dt, *J* = 7.6, 7.6 Hz, 1H), 1.51 (br, 1H); ^{13}C NMR ($CDCl_3$): 140.6, 138.2, 128.8, 127.9, 127.0, 117.1, 66.1, 52.5; HRMS (ESI): calcd for $[C_{10}H_{12}O + Na]^+$, *m*/*z*, 171.0780; found, 171.0781.



1-Chloro-3-phenyl-4-penten-2-ol (7d). This compound was prepared according to the above general procedure except that 50 mol % of *p*-toluenesulfonic acid was used. ^{1}H NMR ($CDCl_3$): 7.34 (m, 2H), 7.25 (m, 3H), 6.17 (ddd, *J* = 8.5, 10.3, 17.1 Hz, 1H), 5.26 (d, *J* = 10.3 Hz, 1H), 5.22 (d, *J* = 17.1 Hz, 1H), 4.06 (m, 1H), 3.50 (dd, *J* = 8.5, 8.5 Hz, 1H), 3.45 (m, 2H), 2.32 (br, 1H); ^{13}C NMR ($CDCl_3$): 140.2, 137.3, 128.9, 128.0, 127.2, 118.2, 73.7, 54.0, 48.2; HRMS (ESI): calcd for $[C_{11}H_{13}ClO + Na]^+$, *m*/*z*, 219.0547; found, 219.0547.

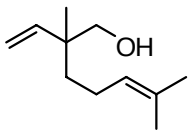


***N*1-(2-Hydroxy-3-phenyl-4-pentenyl)-4-methyl-1-benzenesulfonamide (7e).** This compound was prepared according to the above general procedure. ^{1}H NMR ($CDCl_3$): 7.64 (d, *J* = 8.2 Hz, 2H), 7.29 (m, 5H), 7.12 (d, *J* = 8.2 Hz, 2H), 6.03 (ddd, *J* = 8.8, 10.3, 17.0 Hz, 1H), 5.23 (d, *J* = 10.3 Hz, 1H), 5.20 (d, *J* = 17.0 Hz, 1H), 4.84 (dd, *J* = 5.4, 6.9 Hz, 1H), 3.84 (ddd, *J* = 3.1, 7.5, 8.7 Hz, 1H), 3.28 (dd, *J* = 8.7, 8.8 Hz, 1H), 2.83 (m, 2H), 2.42 (s, 3H), 2.23 (br, 1H); ^{13}C NMR ($CDCl_3$): 143.4, 139.7, 137.5, 136.6, 129.7, 129.0, 127.8, 127.2, 127.1, 118.7, 72.2, 54.8, 46.2, 21.5; HRMS (ESI): calcd for $[C_{18}H_{21}NO_3S + Na]^+$, *m*/*z*, 354.1134; found, 354.1133.

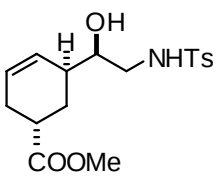


***N*1-allyl-*N*1-(2-hydroxy-3-phenyl-4-pentenyl)-4-methyl-1-benzenesulfonamide (7f).** This compound was prepared according to the above general procedure. ^{1}H NMR ($CDCl_3$): 7.57 (d, *J* = 8.3 Hz, 2H), 7.26 (m, 7H), 6.16 (ddd, *J* = 8.2, 10.3, 17.1 Hz, 1H), 5.56 (tdd, *J* = 6.6, 10.1, 17.0 Hz, 1H), 5.19 (d, *J* = 10.3 Hz, 1H), 5.12 (d, *J* = 17.1 Hz, 1H), 5.05 (d, *J* = 10.1 Hz, 1H), 4.99 (d, *J* = 17.0 Hz, 1H), 4.14 (m, 1H), 3.79 (m, 2H), 3.22 (dd, *J* = 8.2, 8.2 Hz, 1H), 2.96 (m, 2H), 2.72 (br, 1H), 2.41 (s, 3H); ^{13}C NMR ($CDCl_3$): 143.5, 140.8,

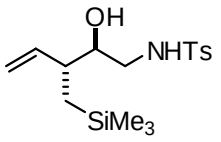
137.9, 136.1, 132.7, 129.7, 128.8, 128.1, 127.3, 126.9, 119.5, 117.5, 72.7, 54.9, 52.5, 52.0, 21.5; HRMS (ESI): calcd for $[C_{21}H_{25}NO_3S + Na]^+$, *m*/*z*, 394.1447; found, 394.1448.



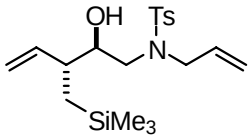
2,6-Dimetyl-2-vinyl-5-hepten-1-ol (7g). This compound was prepared according to the above general procedure except that 10 mol % of *p*-toluenesulfonic acid was used. The NMR data obtained for **7g** are in agreement with the previously reported[9] values. ^{1}H NMR ($CDCl_3$): 5.72 (dd, *J* = 10.9, 17.7 Hz, 1H), 5.18 (d, *J* = 10.9 Hz, 1H), 5.09 (m, 1H), 5.06 (d, *J* = 17.7 Hz, 1H), 3.37 (m, 2H), 1.91 (m, 2H), 1.67 (s, 3H), 1.59 (s, 3H), 1.34 (m, 1H), 1.33 (m, 2H), 1.03 (s, 3H); ^{13}C NMR ($CDCl_3$): 144.0, 131.5, 124.6, 114.7, 70.1, 42.3, 37.2, 25.7, 22.5, 19.5, 17.6; HRMS (ESI): calcd for $[C_{11}H_{20}O + Na]^+$, *m*/*z*, 191.1406; found, 191.1403.



Methyl 5-(1-hydroxy-2-(4-methylphenylsulfonamido)ethyl)-3-cyclohexene-1-carboxylate (7h). This compound was prepared according to the above general procedure. ^{1}H NMR ($CDCl_3$): 7.74 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 5.82 (m, 1H), 5.50 (m, 1H), 5.05 (m, 1H), 3.67 (s, 3H), 3.58 (m, 1H), 3.05 (m, 2H), 2.65 (m, 1H), 2.43 (s, 3H), 2.39 (m, 1H), 2.25 (m, 2H), 1.98 (dt, *J* = 3.7, 13.5 Hz, 1H), 1.77 (ddd, *J* = 5.9, 10.7, 13.5 Hz, 1H), 1.72 (br, 1H); ^{13}C NMR ($CDCl_3$): 175.8, 143.6, 136.6, 129.8, 128.7, 127.1, 125.9, 72.5, 51.8, 46.7, 37.5, 35.9, 27.1, 25.2, 21.5; HRMS (ESI): calcd for $[C_{17}H_{23}NO_5S + Na]^+$, *m*/*z*, 376.1189; found, 376.1191.

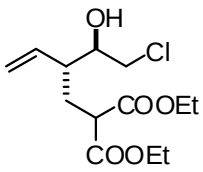


***N*1-(2-Hydroxy-3-((1,1,1-trimethylsilyl)methyl)-4-pentenyl)-4-methyl-1-benzenesulfonamide (7i).** This compound was prepared according to the above general procedure except that first, catalyst **2b** was dissolved in a MeOH/DMSO (0.2 mL / 0.2 mL) mixture. To this solution, 20 mol% LiOAc was added and the solution stirred at rt for 10 min. Thereafter, substrate **5b** and diboronic reagent **1b** were added and the mixture was stirred for 16 h at 40 ^{o}C. Thereafter, the acetal **3e**, 0.1 mL H_2O and 20 mol % PTS were added and the stirring was continued for another 20 h at 70^{o}C. ^{1}H NMR ($CDCl_3$): 7.74 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 5.51 (ddd, *J* = 9.9, 9.9, 17.1 Hz, 1H), 5.15 (d, *J* = 9.9 Hz, 1H), 5.10 (d, *J* = 17.1 Hz, 1H), 4.87 (m, 1H), 3.38 (m, 1H), 3.05 (m, 2H), 2.43 (s, 3H), 2.17 (m, 1H), 2.06 (br, 1H), 0.56 (m, 2H), -0.03 (s, 9H); ^{13}C NMR ($CDCl_3$): 143.4, 139.9, 136.8, 129.7, 127.1, 118.3, 73.7, 46.0, 44.7, 21.5, 18.0, -0.8; HRMS (ESI): calcd for $[C_{16}H_{27}NO_3SSi + Na]^+$, *m*/*z*, 364.1373; found, 364.1372.

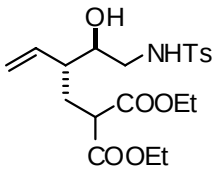


***N*1-Allyl-*N*1-(2-hydroxy-3-((1,1,1-trimethylsilyl)methyl)-4-pentenyl)-4-methyl-1-benzenesulfonamide (7j).** This compound was prepared according to the above general procedure except that first, the catalyst **2b** was dissolved in a MeOH/DMSO (0.2 mL / 0.2 mL) mixture. To this solution, 20 mol% LiOAc was added and the solution stirred at rt for 10 min. Thereafter, substrate **5b** and the diboronic reagent **1b** were added and the mixture was stirred for 16 h at 40 ^{o}C. Thereafter, the acetal **3f**, 0.1 mL H_2O and 20 mol % PTS were added and the stirring was continued for another 20 h at 70^{o}C. ^{1}H NMR ($CDCl_3$): 7.70 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 5.66 (m, 2H), 5.16 (d, *J* = 18.5 Hz, 1H), 5.15 (d, *J* = 9.0 Hz, 1H), 5.08 (d, *J* = 10.3 Hz, 1H), 5.03 (d, *J* = 17.1 Hz, 1H), 3.87 (m, 2H), 3.67 (m, 1H), 3.10 (m, 2H), 2.44 (br,

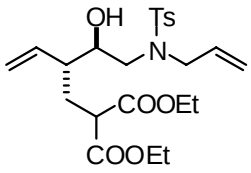
1H), 2.43 (s, 3H), 2.20 (m, 1H), 0.72 (m, 2H), -0.01 (s, 9H); ^{13}C NMR ($CDCl_3$): 143.5, 139.7, 136.5, 133.1, 129.8, 127.3, 119.3, 116.7, 73.7, 52.2, 51.7, 44.3, 21.5, 18.2, -0.8; HRMS (ESI): calcd for $[C_{19}H_{31}NO_3SSi + Na]^+$, *m*/*z*, 404.1686; found, 404.1683.



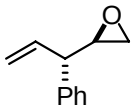
Diethyl 2-(2-(2-chloro-1-hydroxyethyl)-3-butenyl)malonate (7k). This compound was prepared according to the above general procedure. ^{1}H NMR ($CDCl_3$): 5.68 (ddd, *J* = 10.0, 10.0, 17.3 Hz, 1H), 5.24 (d, *J* = 10.0 Hz, 1H), 5.12 (d, *J* = 17.3 Hz, 1H), 4.18 (m, 4H), 3.77 (m, 1H), 3.54 (m, 2H), 3.39 (dd, *J* = 5.0, 10.0 Hz, 1H), 2.28 (m, 1H), 2.27 (br, 1H), 2.08 (m, 2H), 1.25 (m, 6H); ^{13}C NMR ($CDCl_3$): 169.4, 169.2, 135.2, 119.9, 73.4, 61.5, 61.4, 49.7, 48.2, 45.7, 30.3, 14.1, 14.0; HRMS (ESI): calcd for $[C_{13}H_{21}ClO_5 + Na]^+$, *m*/*z*, 315.0970; found, 315.0967.



Diethyl 2-(2-(1-hydroxy-2-(4-metylphenylsulfonamido)ethyl)-3-butenyl)malonate (7l). This compound was prepared according to the above general procedure. ^{1}H NMR ($CDCl_3$): 7.74 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 5.58 (ddd, *J* = 10.0, 10.0, 17.2 Hz, 1H), 5.21 (d, *J* = 10.0 Hz, 1H), 5.07 (d, *J* = 17.2 Hz, 1H), 4.97 (m, 1H), 4.19 (m, 4H), 3.58 (m, 1H), 3.35 (m, 1H), 2.98 (m, 2H), 2.43 (s, 3H), 2.35 (br, 1H), 2.07 (m, 1H), 1.95 (m, 2H), 1.26 (m, 6H); ^{13}C NMR ($CDCl_3$): 169.4, 169.3, 143.5, 136.7, 136.0, 129.8, 127.1, 120.1, 71.7, 61.6, 49.7, 46.7, 46.5, 29.5, 21.5, 14.1, 14.0; HRMS (ESI): calcd for $[C_{20}H_{29}NO_7S + Na]^+$, *m*/*z*, 450.1557; found, 450.1555.

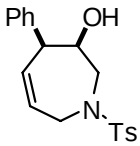


Diethyl 2-(2-(2-(*N*-allyl-4-metylphenylsulfonamido)-1-hydroxyethyl)-3-butenyl)malonate (7m). This compound was prepared according to the above general procedure. ^{1}H NMR ($CDCl_3$): 7.68 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 5.69 (ddd, *J* = 8.9, 10.2, 17.3 Hz, 1H), 5.63 (m, 1H), 5.17 (m, 3H), 5.02 (d, *J* = 17.3 Hz, 1H), 4.18 (m, 4H), 3.84 (m, 3H), 3.39 (m, 1H), 3.09 (m, 2H), 2.66 (d, *J* = 3.3 Hz, 1H), 2.42 (s, 3H), 2.05 (m, 3H), 1.25 (m, 6H); ^{13}C NMR ($CDCl_3$): 169.5, 169.4, 143.6, 136.2, 135.8, 132.9, 129.8, 127.3, 119.5, 119.2, 71.4, 61.4, 61.3, 52.4, 52.3, 49.7, 46.1, 30.2, 21.5, 14.1, 14.0; HRMS (ESI): calcd for $[C_{23}H_{33}NO_7S + Na]^+$, *m*/*z*, 490.1870; found, 450.1867.

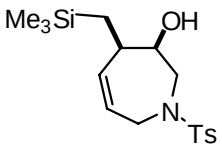


2-(1-Phenylallyl)oxirane (8). This compound was prepared according to the above general procedure except that five equivalents of KOH were added to the in situ formed chlorohydrin **7d**. After stirring at rt for 16 h, epoxide **8** was isolated. ^{1}H NMR ($CDCl_3$): 7.35 (m, 2H), 7.27 (m, 3H), 6.05 (ddd, *J* = 6.8, 10.4, 17.2 Hz, 1H), 5.25 (d, *J* = 17.2 Hz, 1H), 5.24 (d, *J* = 10.4 Hz, 1H), 3.22 (m, 2H), 2.79 (m, 1H), 2.61 (m, 1H); ^{13}C NMR ($CDCl_3$): 140.1, 137.2, 128.7, 128.0, 127.0, 117.0, 54.7, 51.8, 46.1; HRMS (ESI): calcd for $[C_{11}H_{12}O + Na]^+$, *m*/*z*, 183.0780; found, 183.0782.

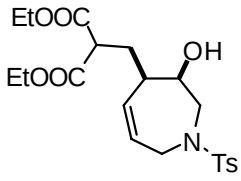
Ring Closing Metathesis of Dienes 7f, j and m (Scheme 3). Dienes **7f, j** or **m** (0.1 mmol) and catalyst **9** (5 mol%, 0.005 mmol) were dissolved in freshly distilled dichloromethane (7.0 mL) under Ar atmosphere. This reaction mixture was stirred at 50 °C for 7 hours, the solvent was evaporated and the product **10** was purified by column chromatography.



1-(4-Methylphenyl)sulfonyl-4-phenyl-2,3,4,7-tetrahydro-1*H*-3-azepinol (10a). ^{1}H NMR ($CDCl_3$): 7.70 (d, *J* = 8.3 Hz, 2H), 7.32 (m, 7H), 5.82 (m, 2H), 4.09 (m, 1H), 4.00 (m, 1H), 3.93 (m, 2H), 3.48 (m, 2H), 2.44 (s, 3H), 2.11 (d, *J* = 7.4 Hz, 1H); ^{13}C NMR ($CDCl_3$): 143.6, 142.0, 135.3, 132.8, 129.8, 128.7, 128.3, 127.2, 126.9, 126.8, 72.1, 54.3, 48.9, 48.4, 21.5; HRMS (ESI): calcd for $[C_{19}H_{21}NO_3S + Na]^+$, *m*/*z*, 366.1134; found, 366.1135.



1-(4-Methylphenyl)sulfonyl-4-(1,1,1-trimethylsilyl)methyl-2,3,4,7-tetrahydro-1*H*-3-azepinol (10b). ^{1}H NMR ($CDCl_3$): 7.67 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 5.73 (m, 1H), 5.40 (dd, *J* = 4.5, 11.0 Hz, 1H), 3.79 (m, 1H), 3.78 (m, 2H), 3.40 (m, 2H), 2.75 (m, 1H), 2.42 (s, 3H), 1.98 (d, *J* = 8.4 Hz, 1H), 0.82 (m, 2H), 0.00 (s, 9H); ^{13}C NMR ($CDCl_3$): 143.5, 136.3, 135.4, 129.7, 127.2, 126.1, 73.0, 54.6, 48.0, 38.7, 21.5, 20.6, -0.9; HRMS (ESI): calcd for $[C_{17}H_{27}NO_3SSi + Na]^+$, *m*/*z*, 376.1373; found, 376.1373.



Diethyl 2-((3-hydroxy-1-((4-methylphenyl)sulfonyl)-2,3,4,7-tetrahydro-1*H*-4-azepinyl)methyl)malonate (10c). ^{1}H NMR ($CDCl_3$): 7.66 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 5.82 (m, 1H), 5.42 (m, 1H), 4.19 (m, 4H), 3.92 (m, 1H), 3.78 (m, 2H), 3.42 (dd, *J* = 6.1, 8.4 Hz, 1H), 3.40 (m, 2H), 2.68 (m, 1H), 2.43 (s, 3H), 2.35 (d, *J* = 8.6 Hz, 1H), 2.18 (m, 2H), 1.27 (m, 6H); ^{13}C NMR ($CDCl_3$): 169.5, 169.4, 143.6, 135.3, 132.8, 129.8, 127.5, 127.2, 69.5, 61.7, 61.6, 54.7, 49.9, 48.4, 40.4, 31.4, 21.5, 14.0; HRMS (ESI): calcd for $[C_{21}H_{29}NO_7S + Na]^+$, *m*/*z*, 462.1557; found, 462.1558.

References:

(1) N. Selander, A. Kipke, S. Sebelius and K. J. Szabó, *J. Am. Chem. Soc.*, 2007, **129**, 13723.

(2) J. Aydin, K. S. Kumar, L. Eriksson and K. J. Szabó, *Adv. Synth. Catal.*, 2007, **349**, 2585.

(3) M. Sander, E. V. Dehmlow, B. Neumann and H. G. Stammler, H. *Tetrahedron*, 1999, **55**, 13395.

(4) G. H. Lee, E. B. Choi, E. Lee and C. S. Pak, *J. Org. Chem.*, 1994, **59**, 1428.

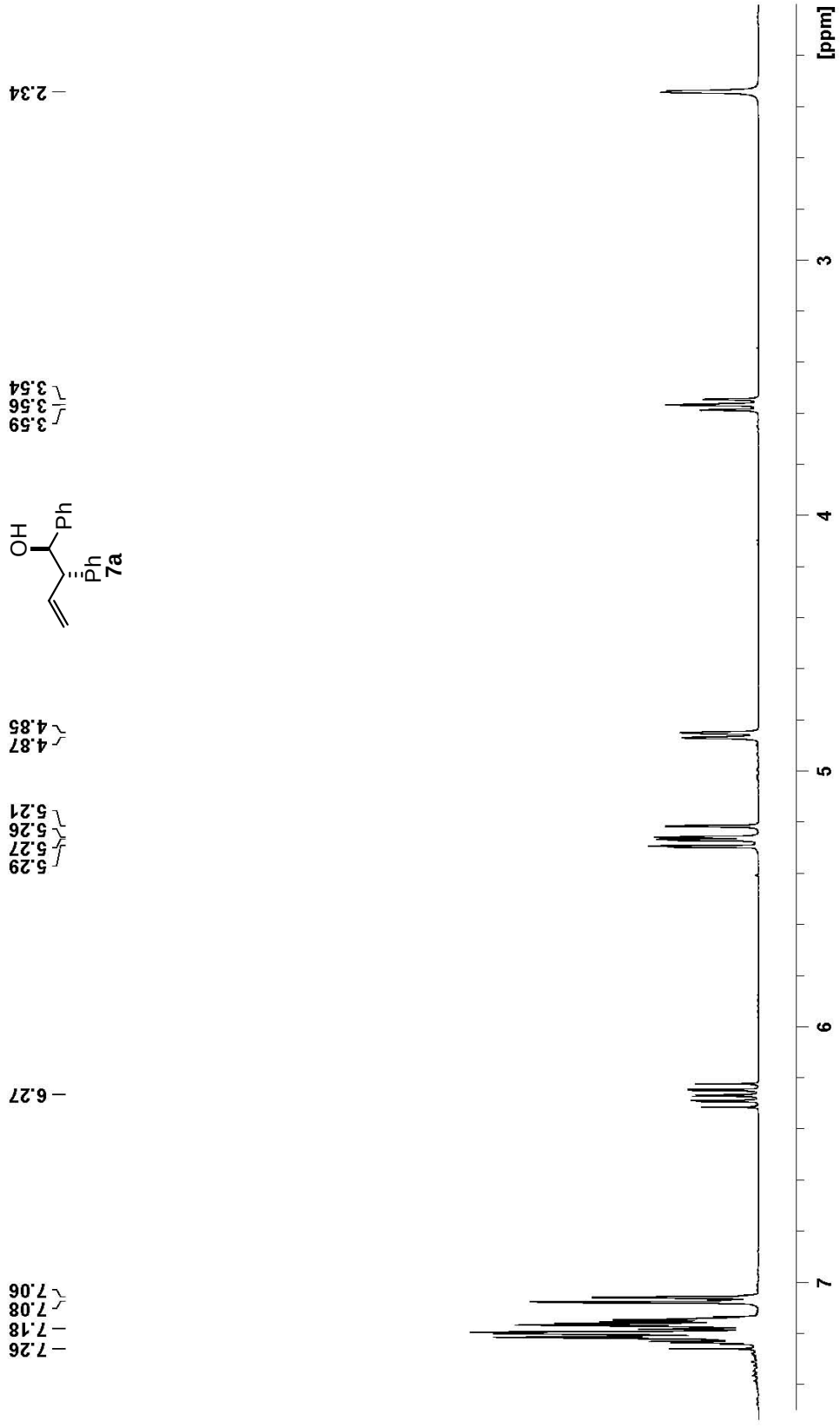
(5) J. A. Marshall and S. Xie, *J. Org. Chem.*, 1995, **60**, 7230.

(6) J. S. Panek and M. A. Sparks, *Tetrahedron Asymmetry*, 1990, **1**, 801.

(7) G. S. Skinner, G. Limperos and R. H. Petterbone, *J. Am. Chem. Soc.*, 1950, **72**, 1648.

(8) T. Miura, Y. Takahashi and M. Murakami, *J. Chem. Soc., Chem. Commun.*, 2007, 595.

(9) J. Mulzer and B. List, *Tetrahedron Lett.*, 1996, **37**, 2403.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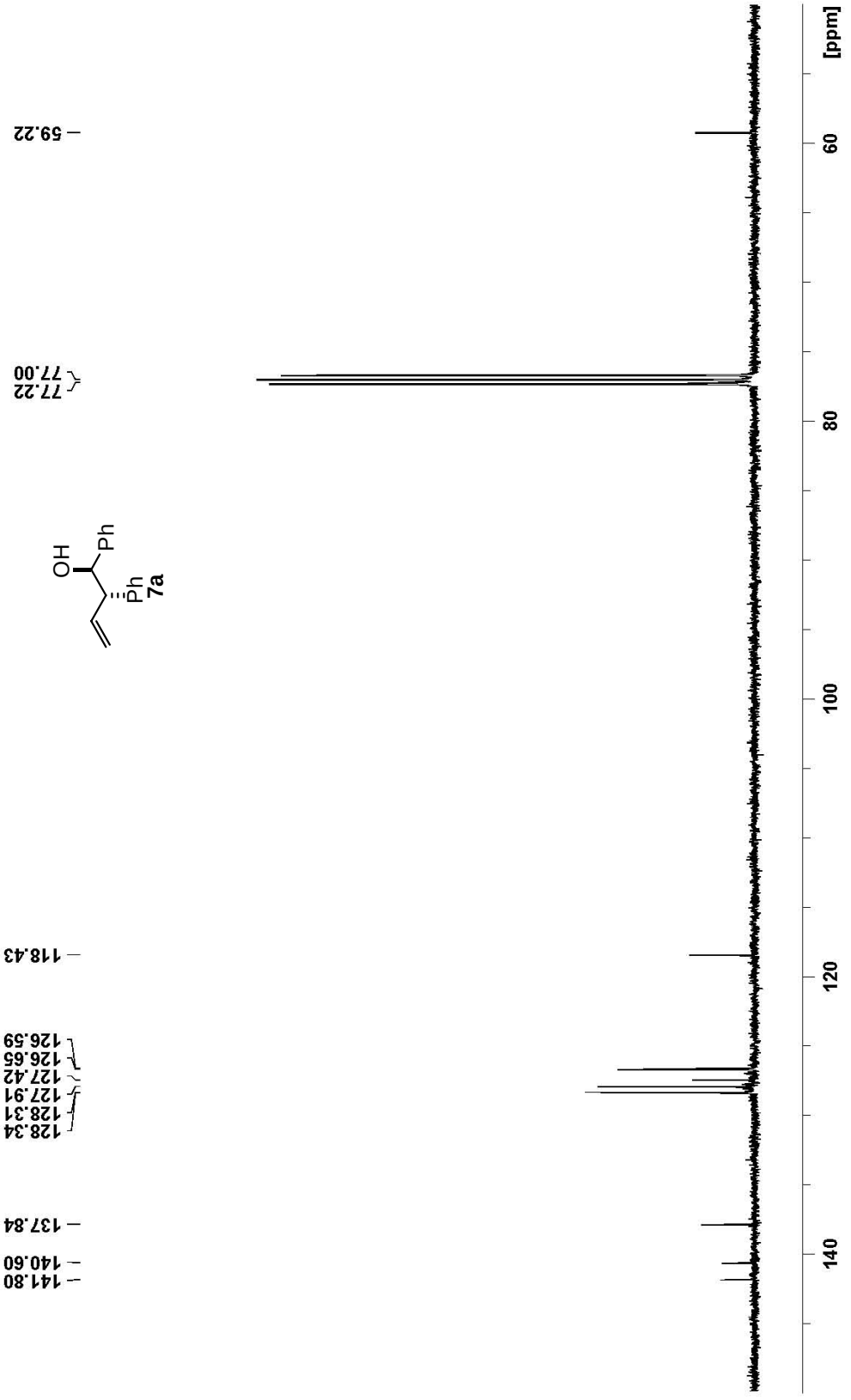
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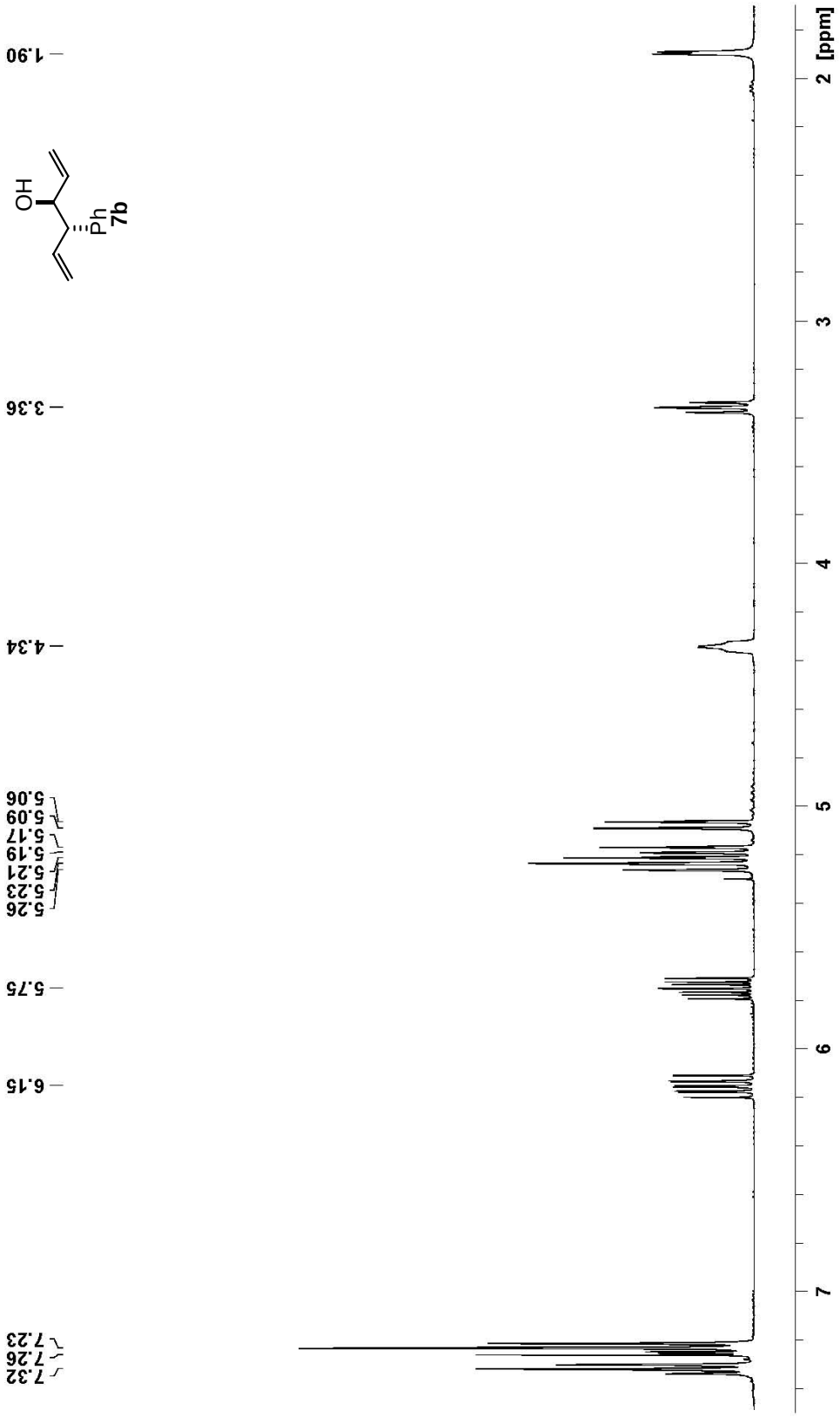
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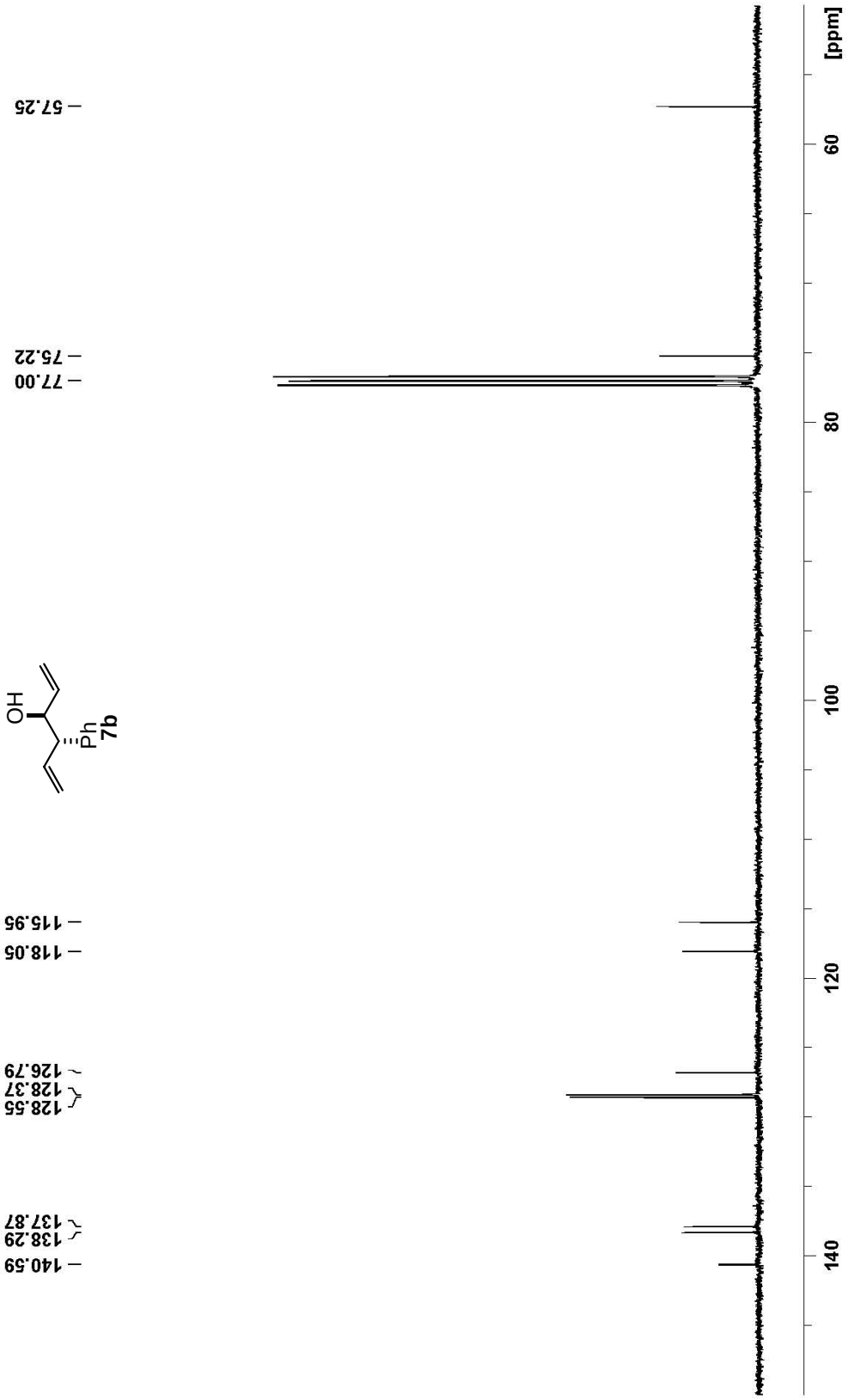
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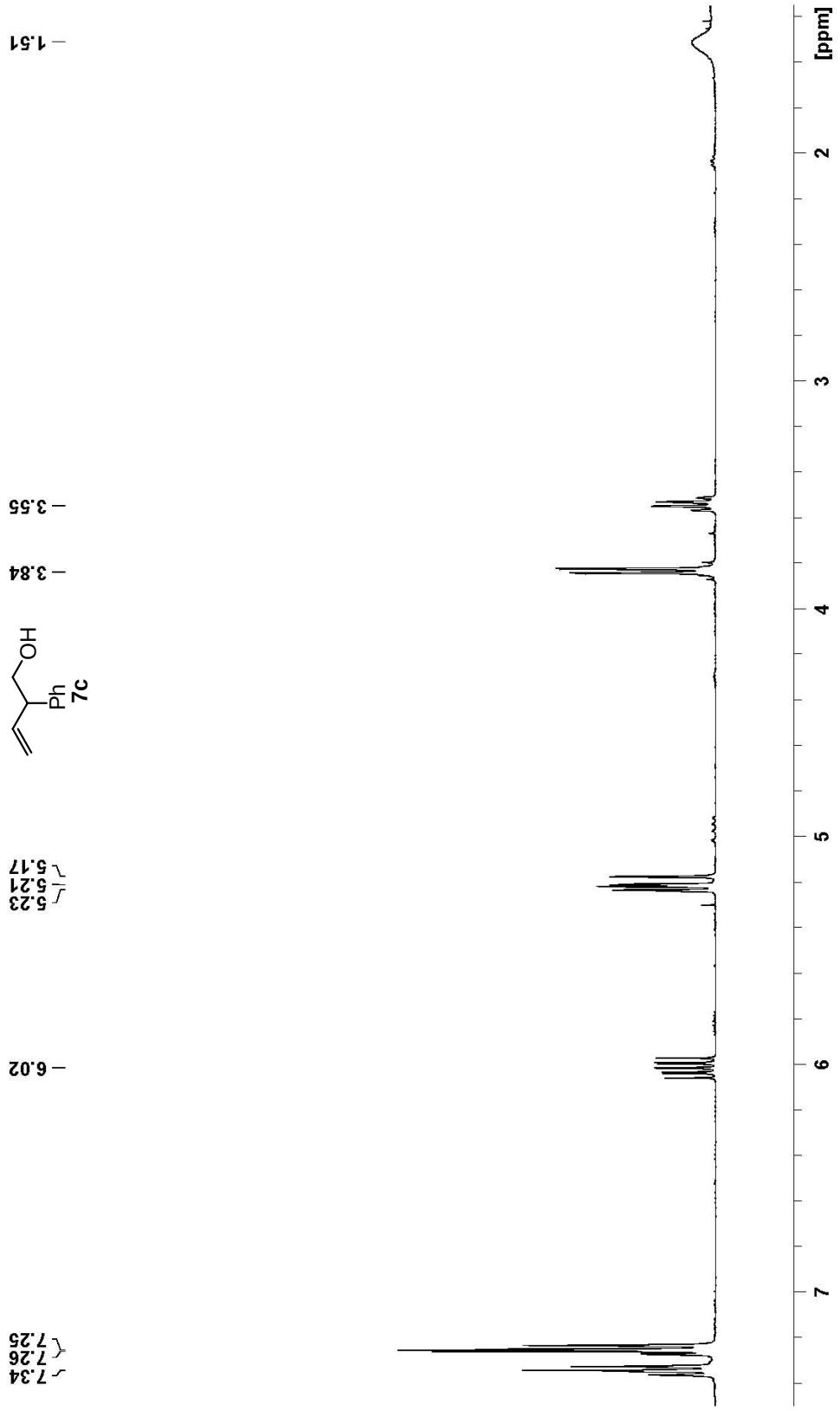
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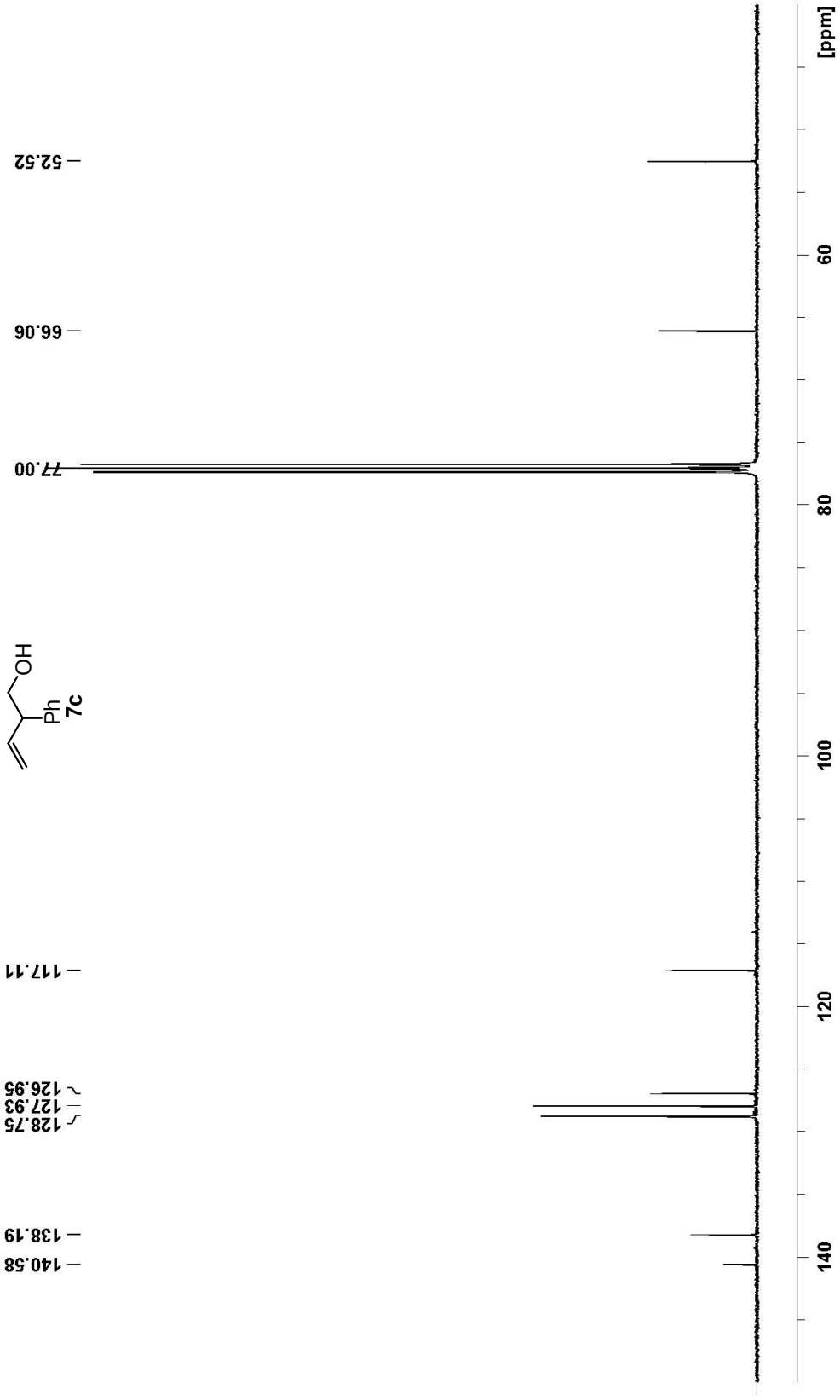
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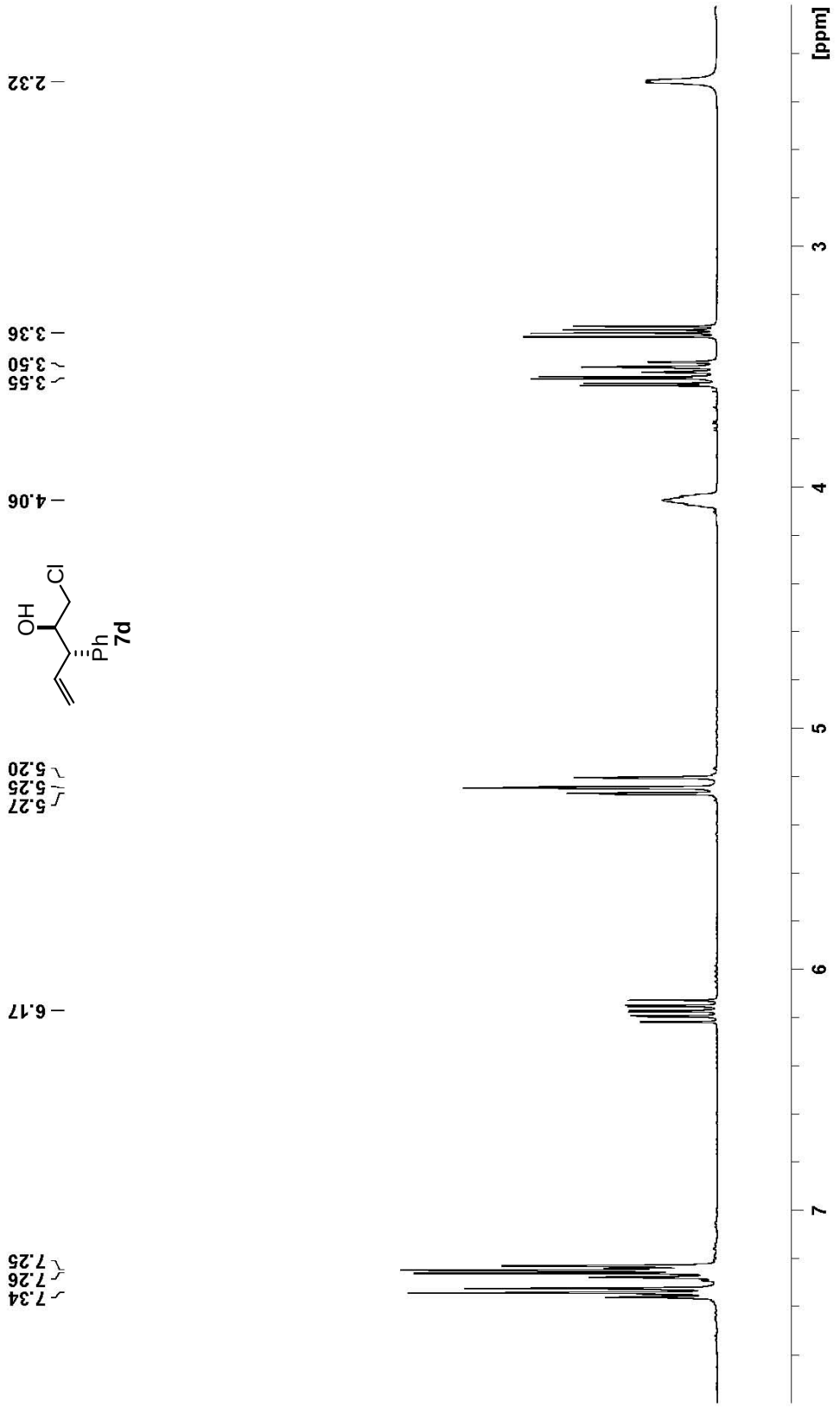
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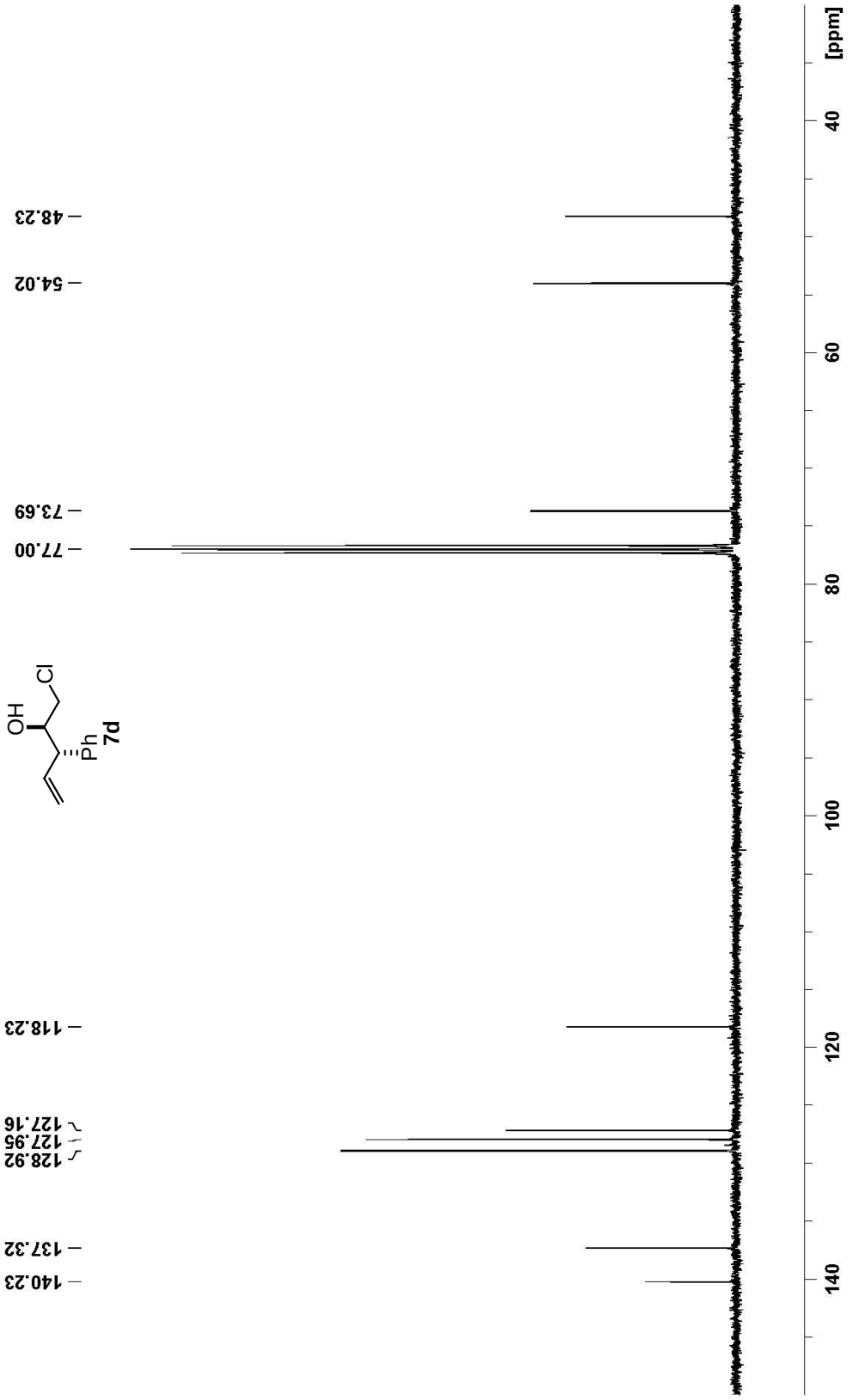
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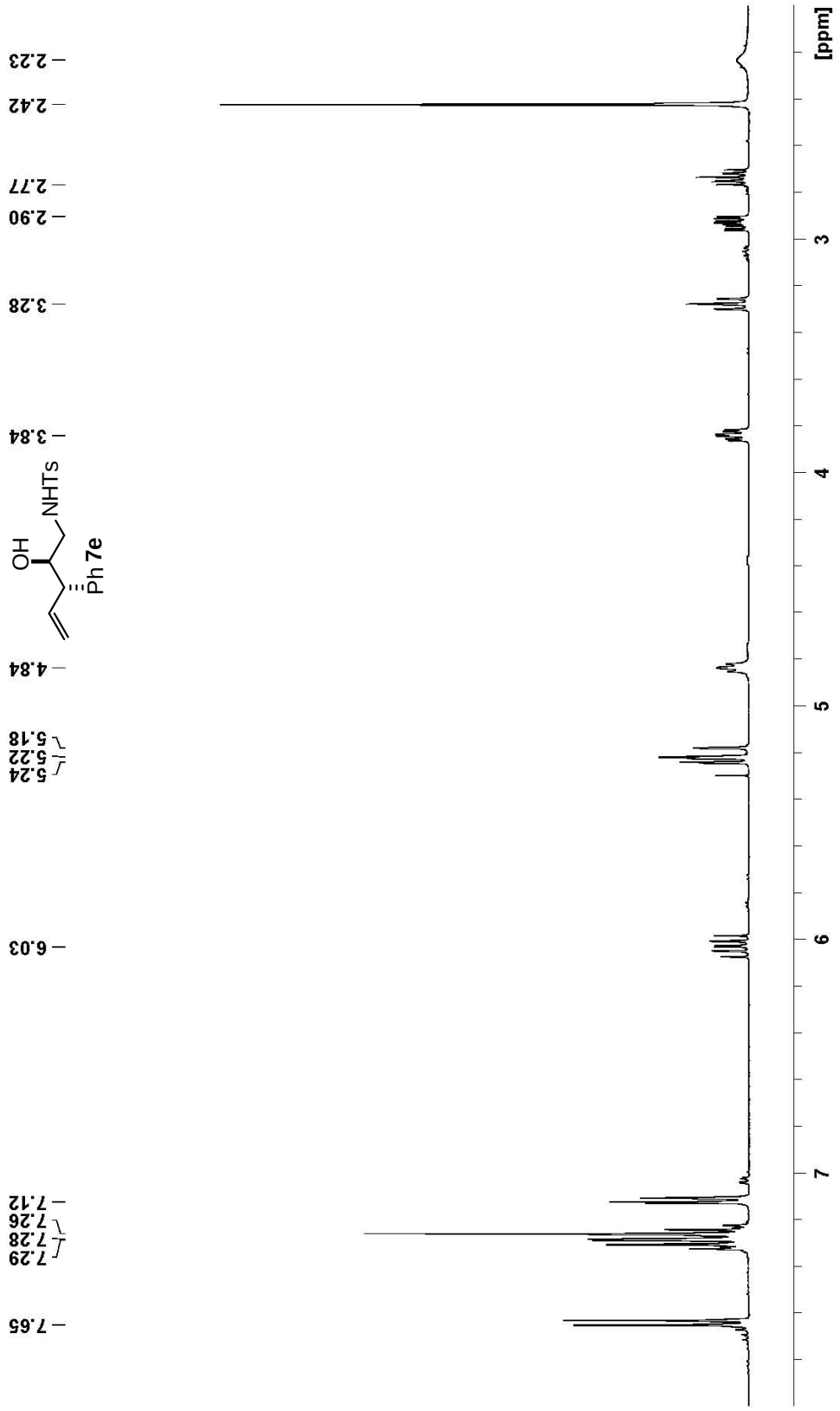
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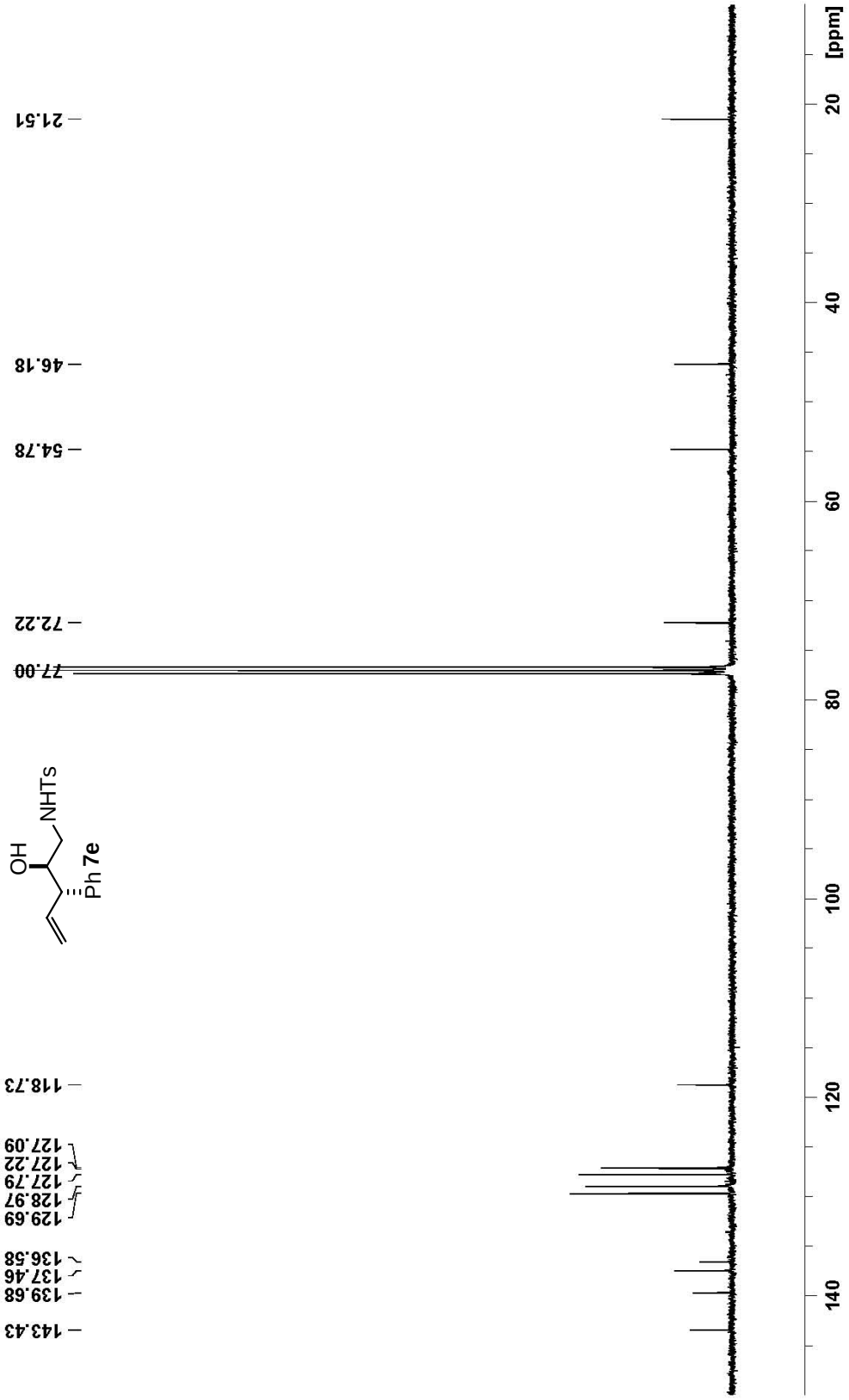
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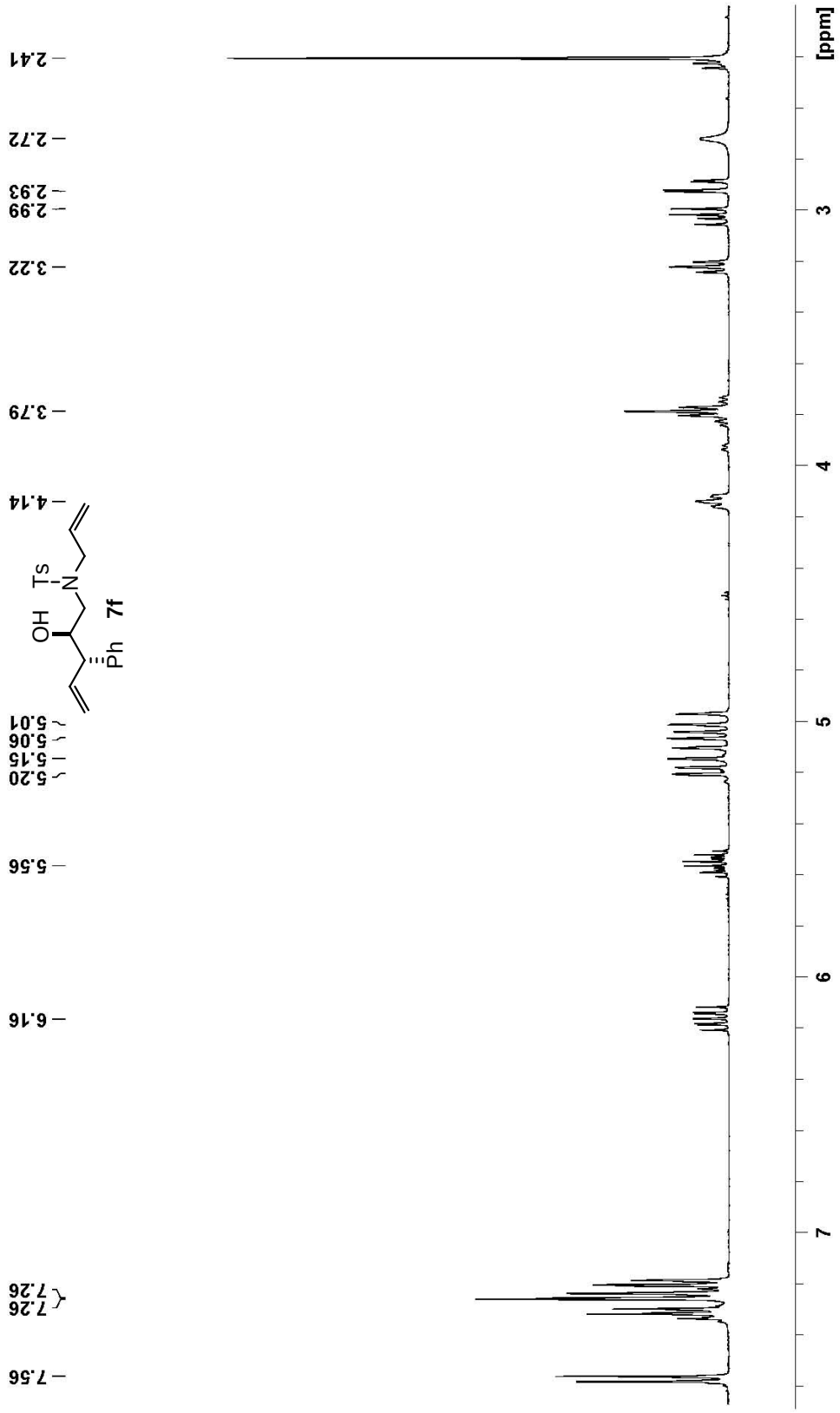
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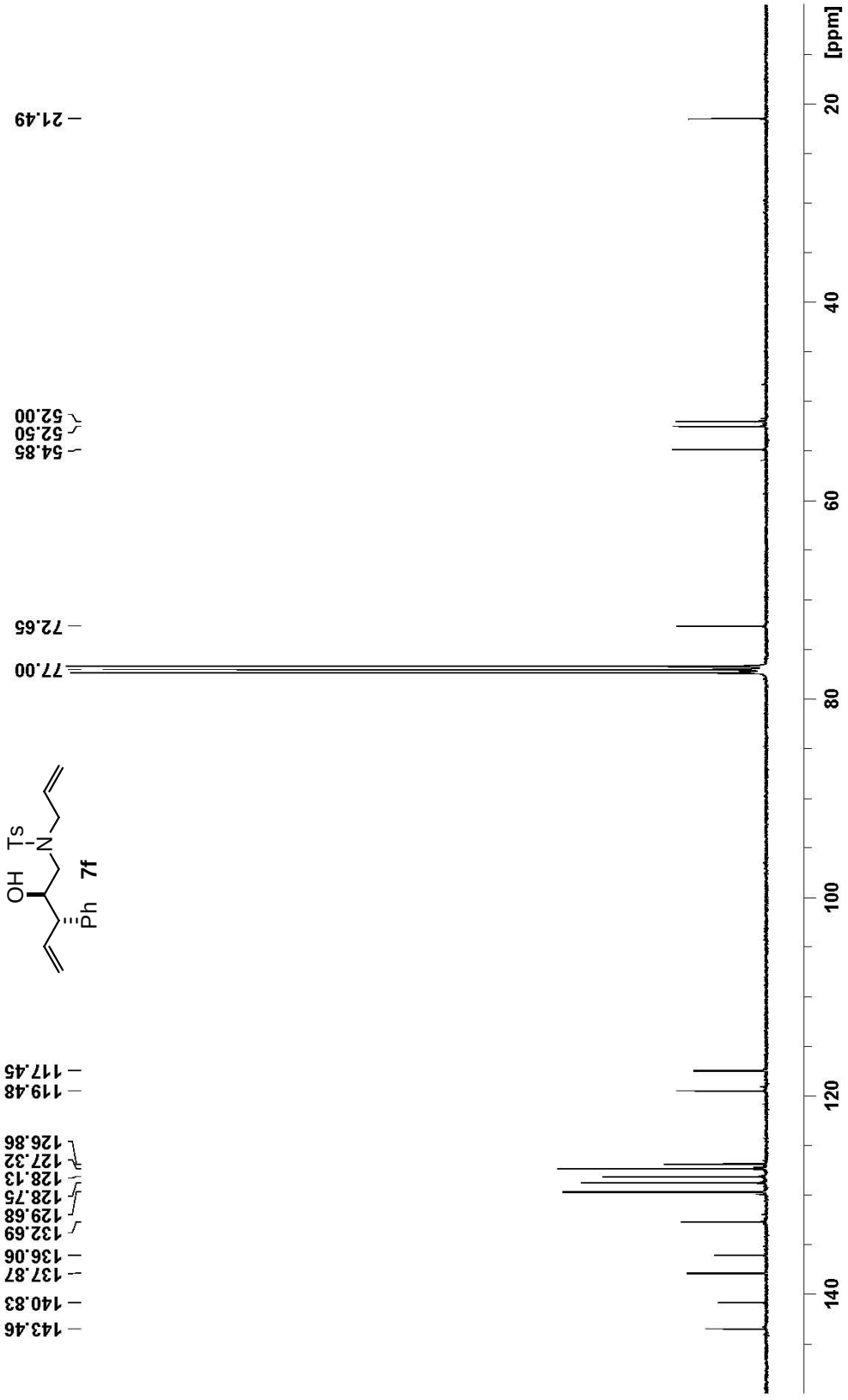
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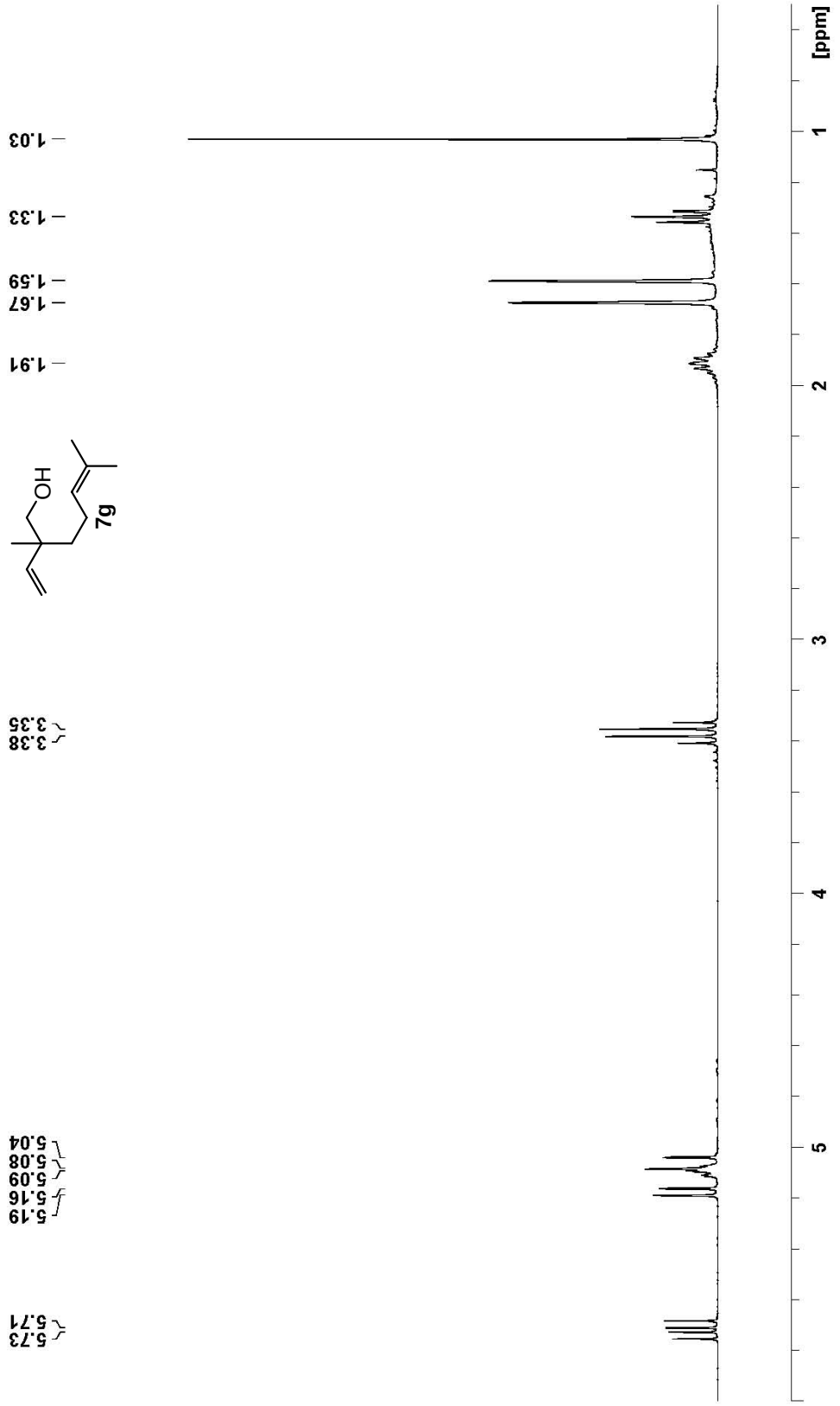
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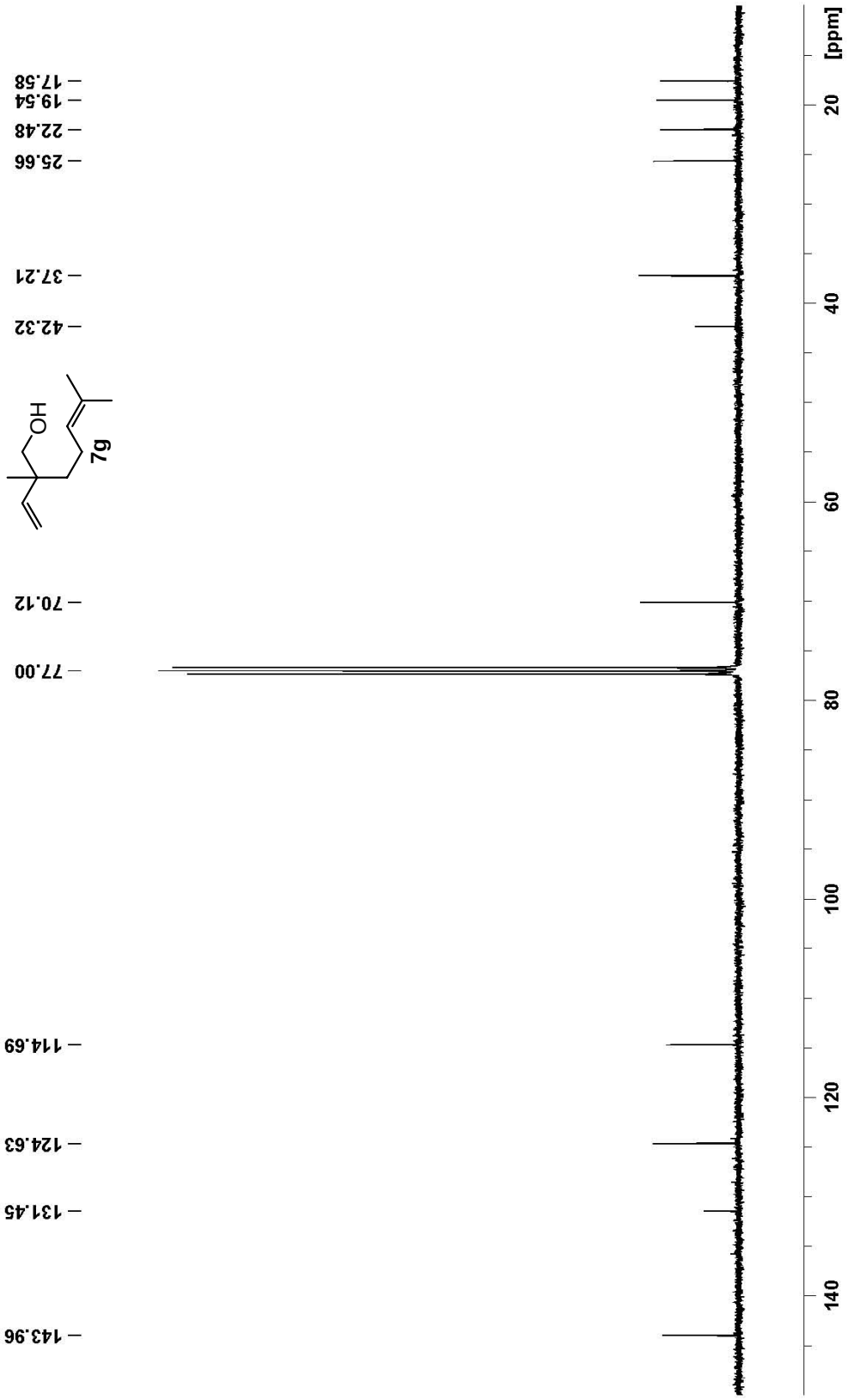
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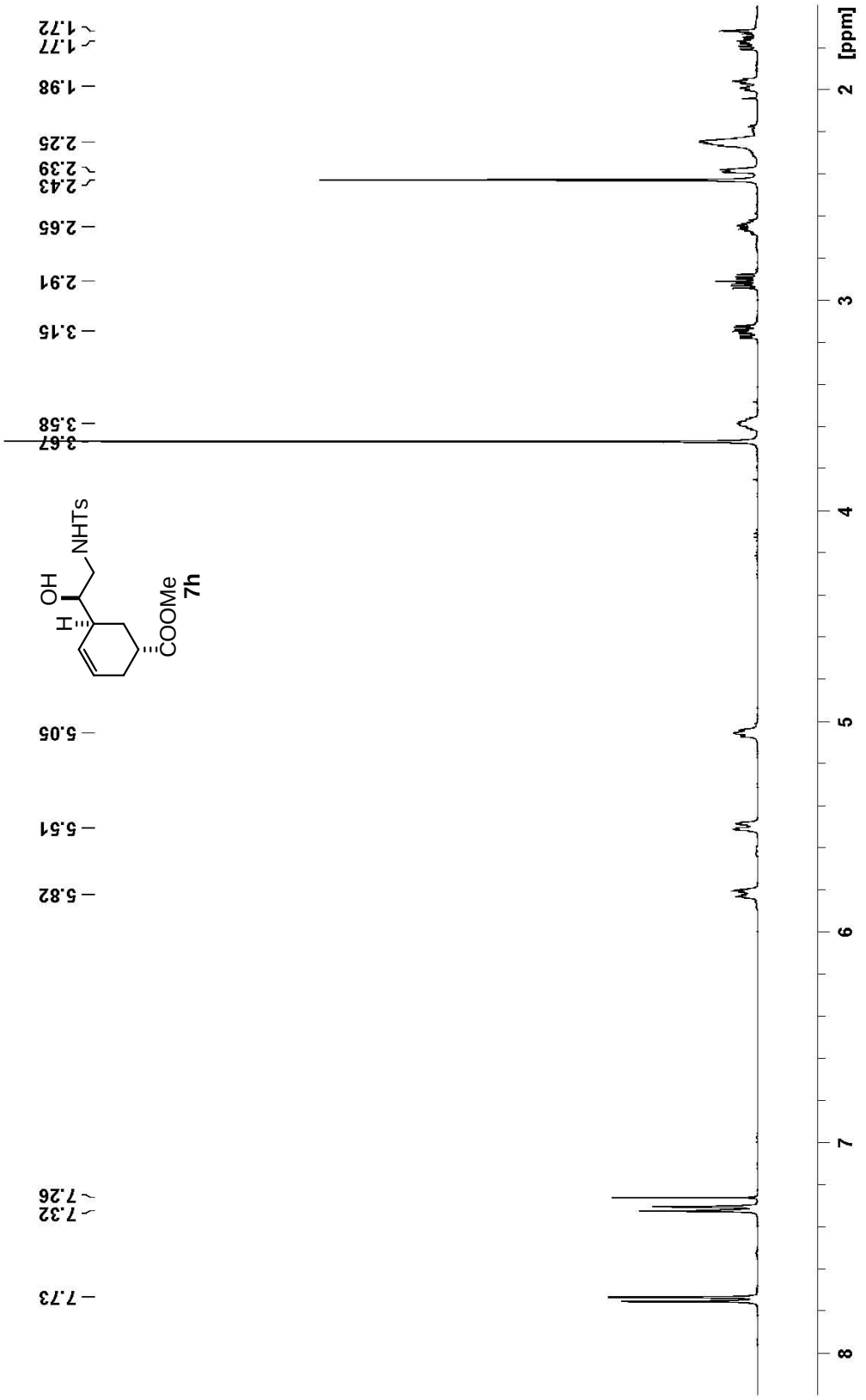
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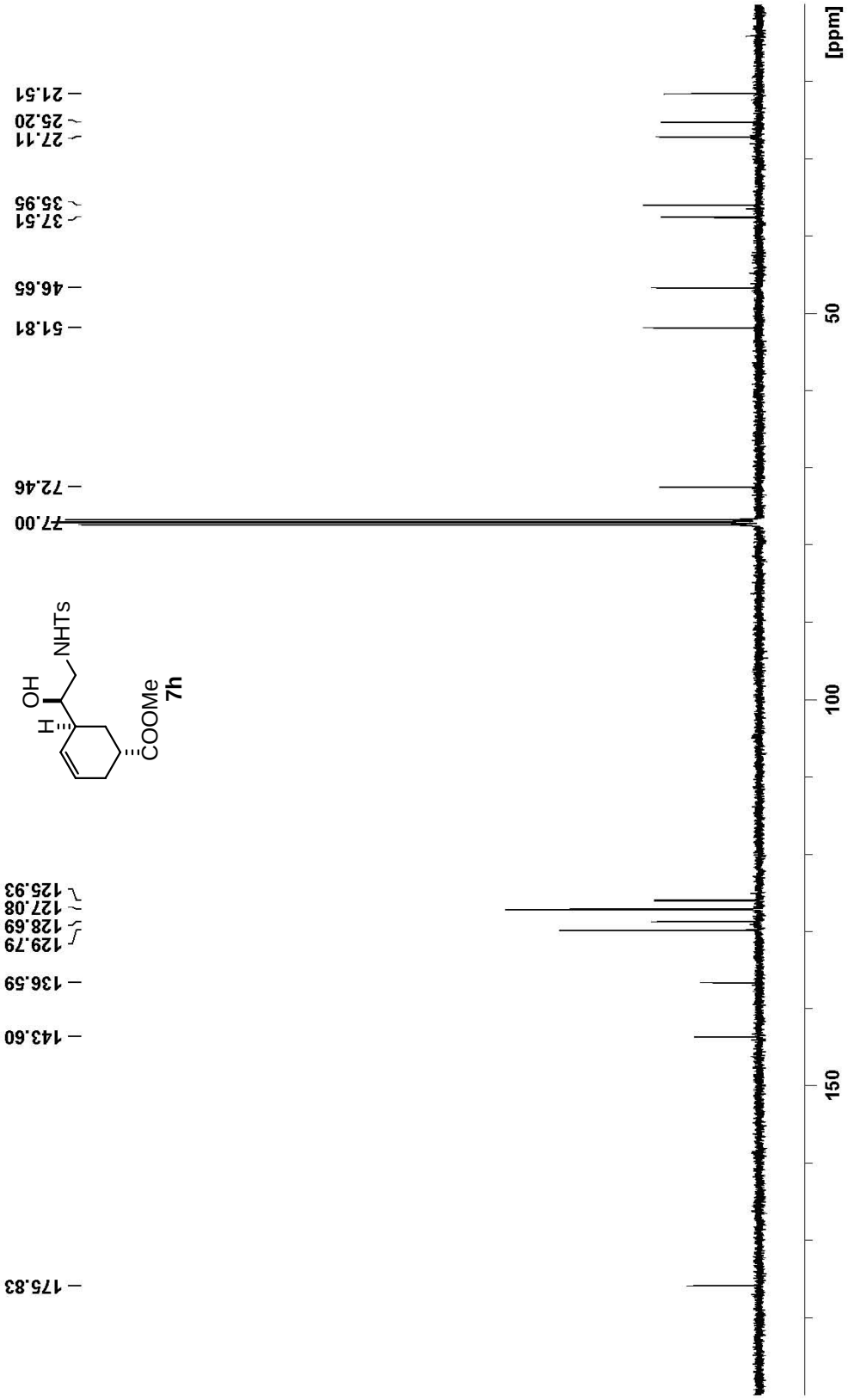
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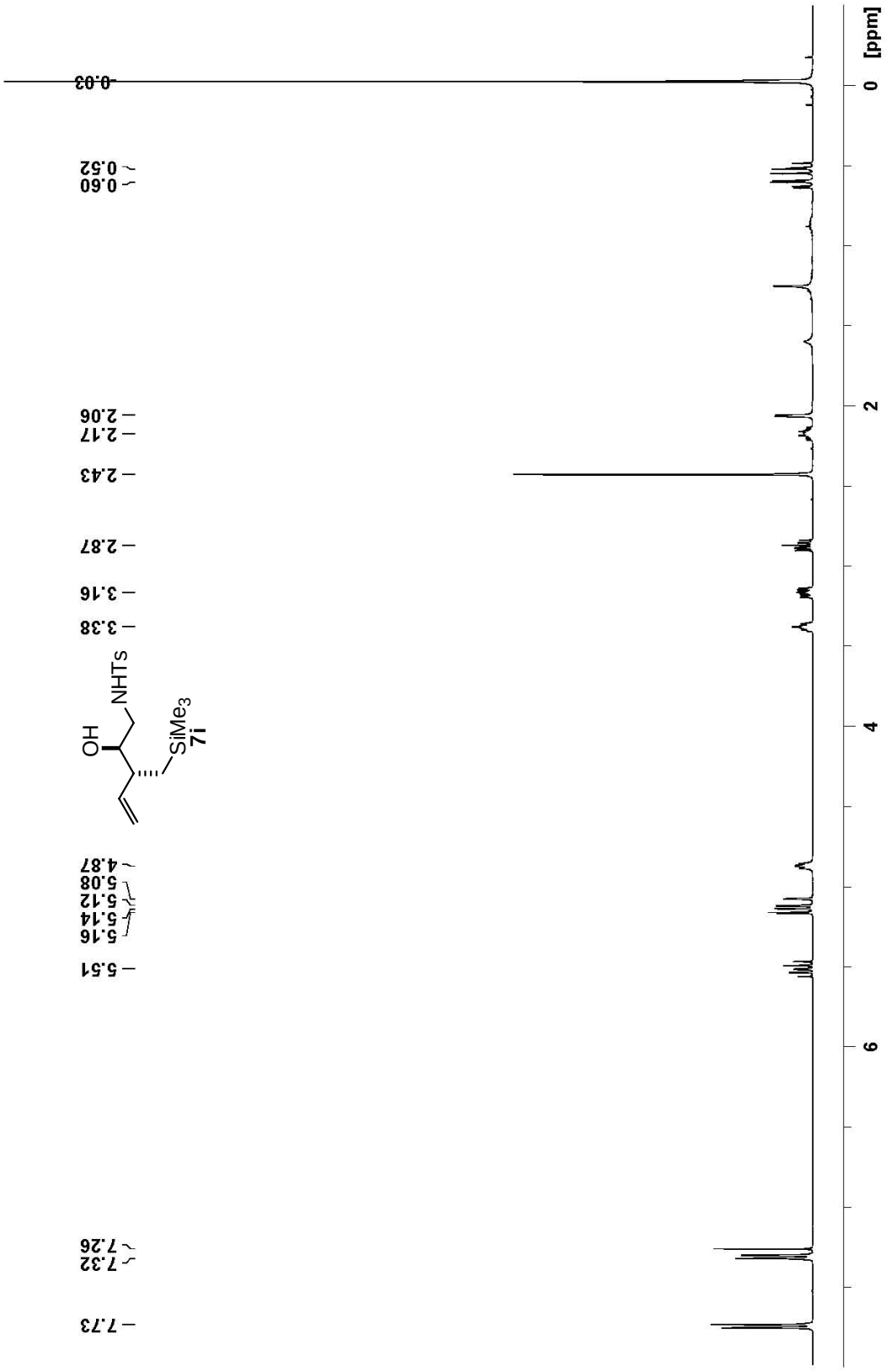
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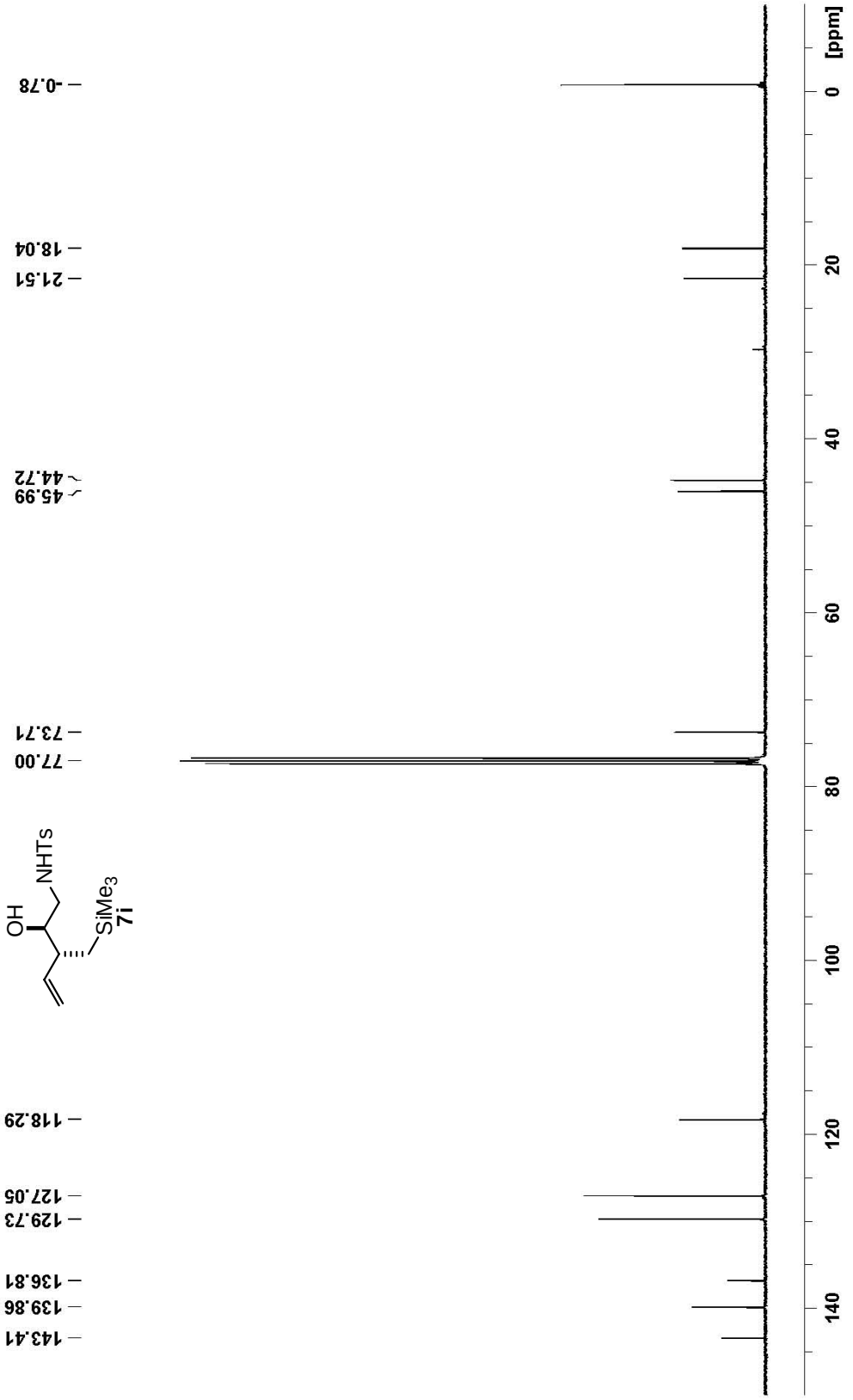
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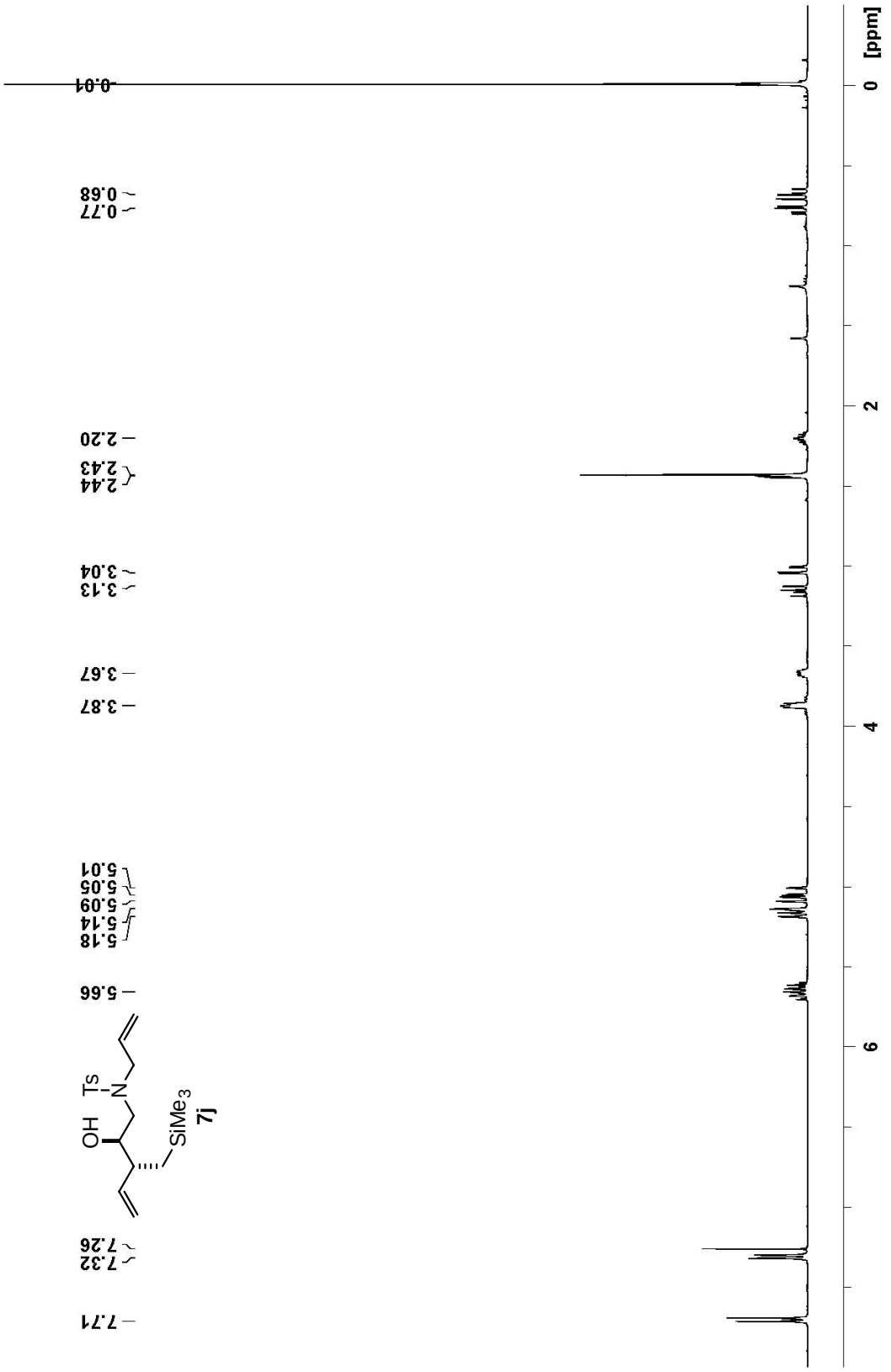
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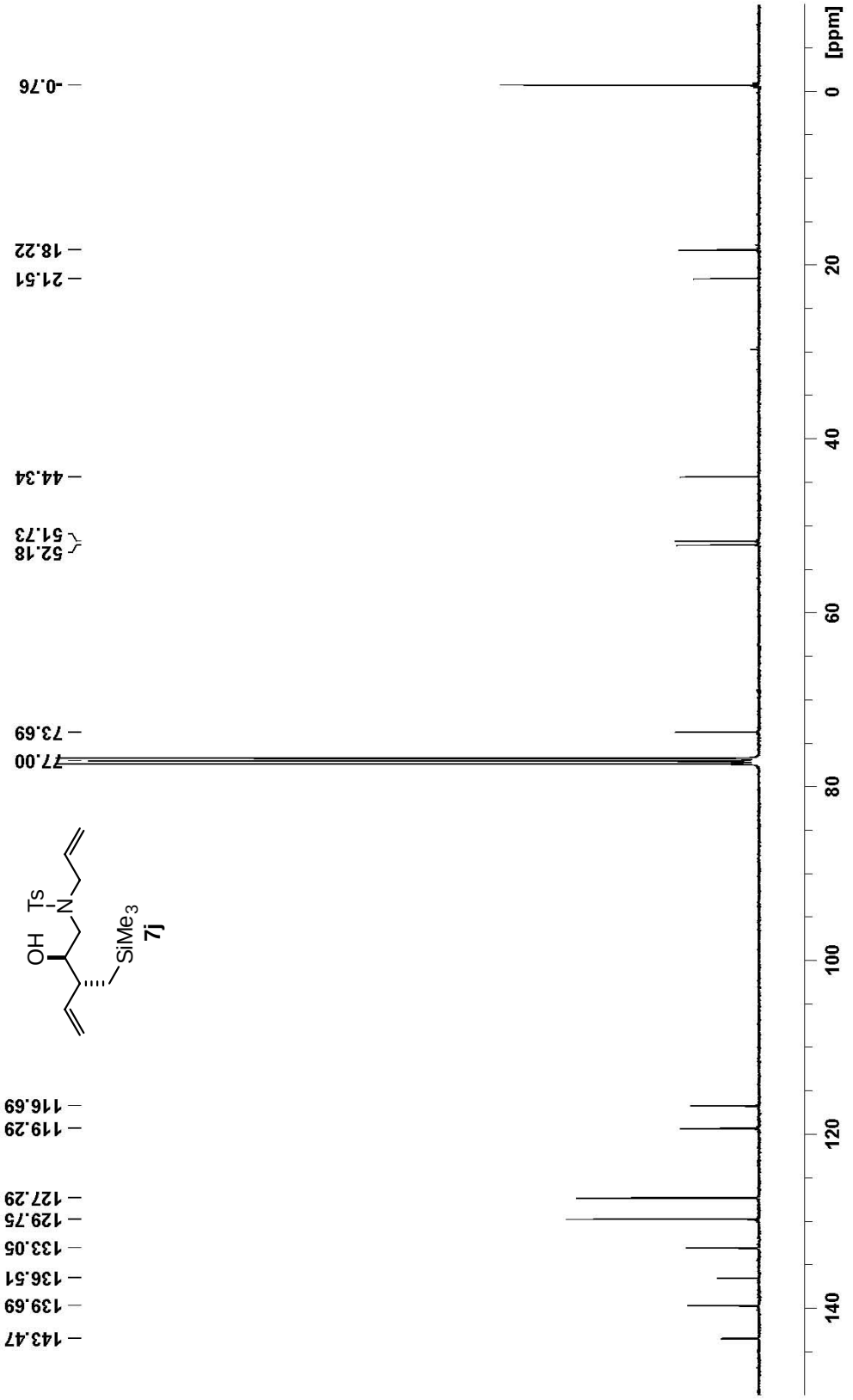
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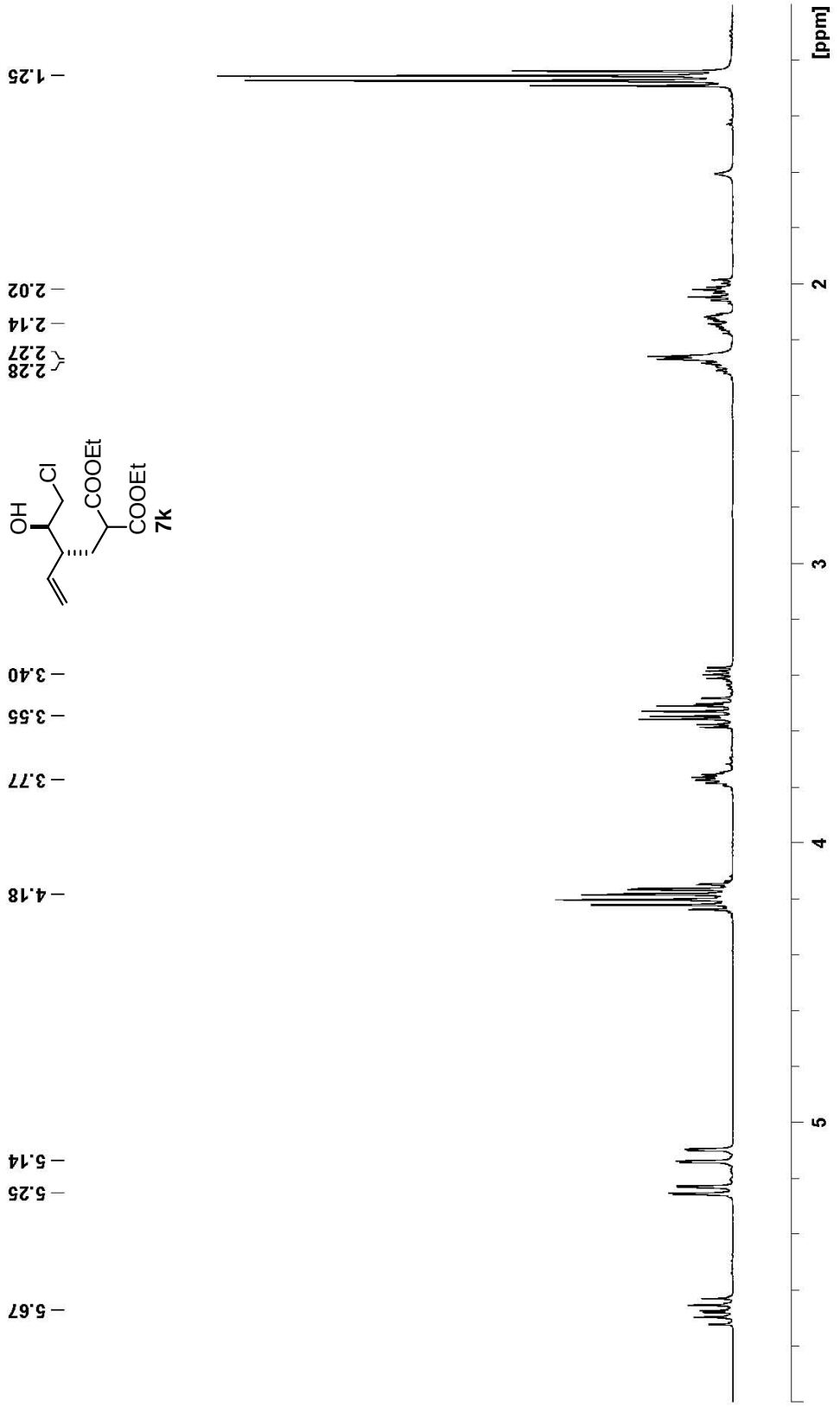
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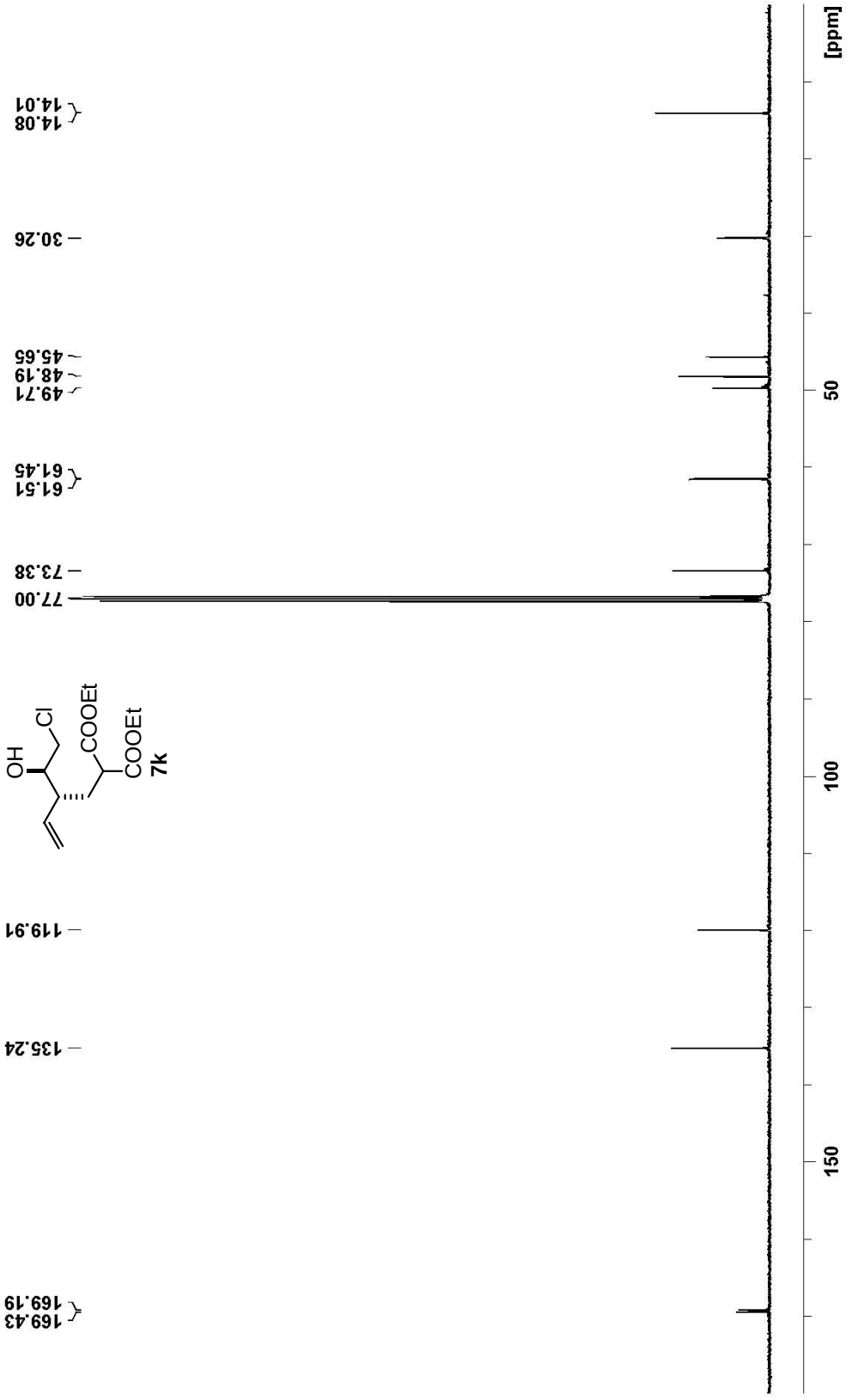
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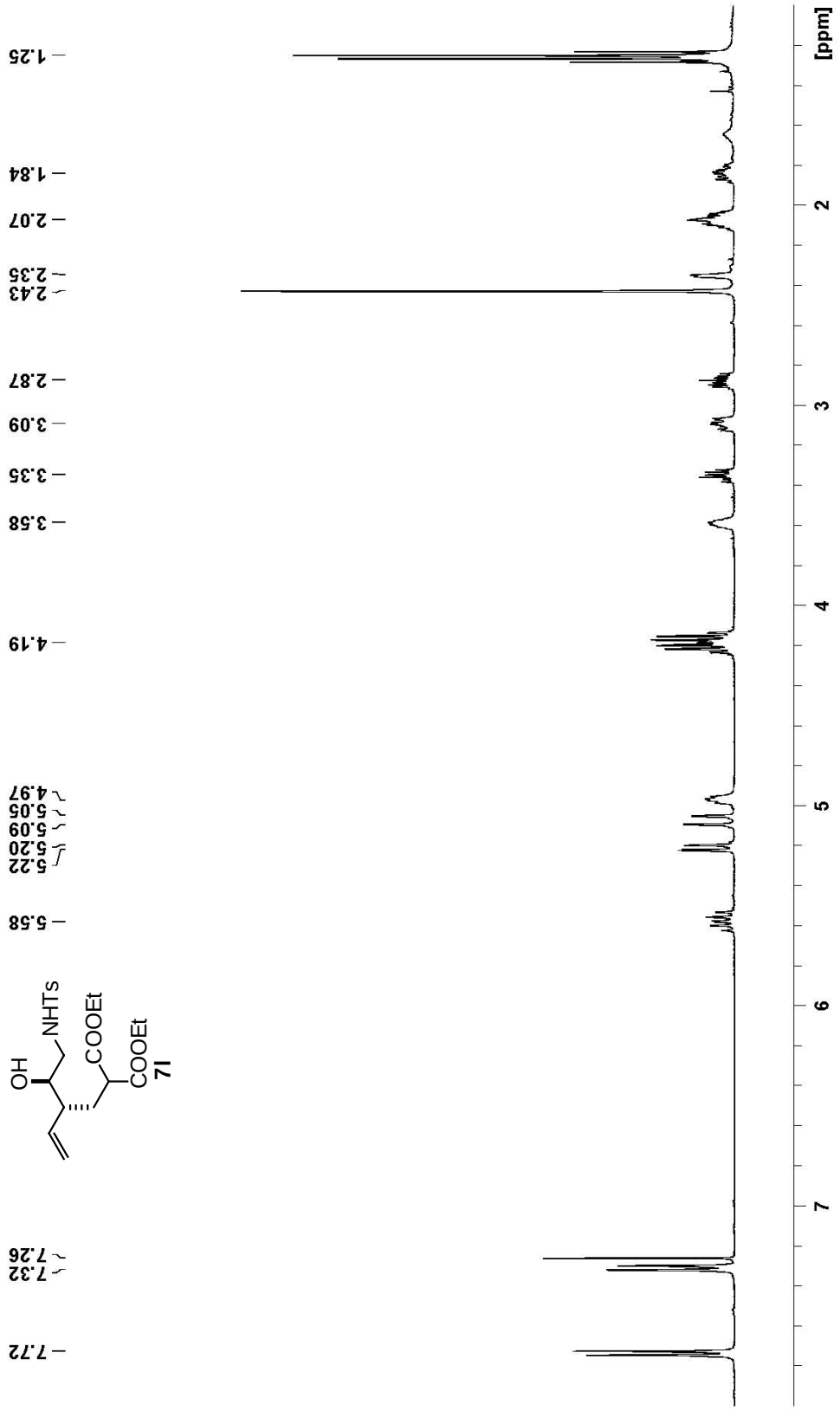
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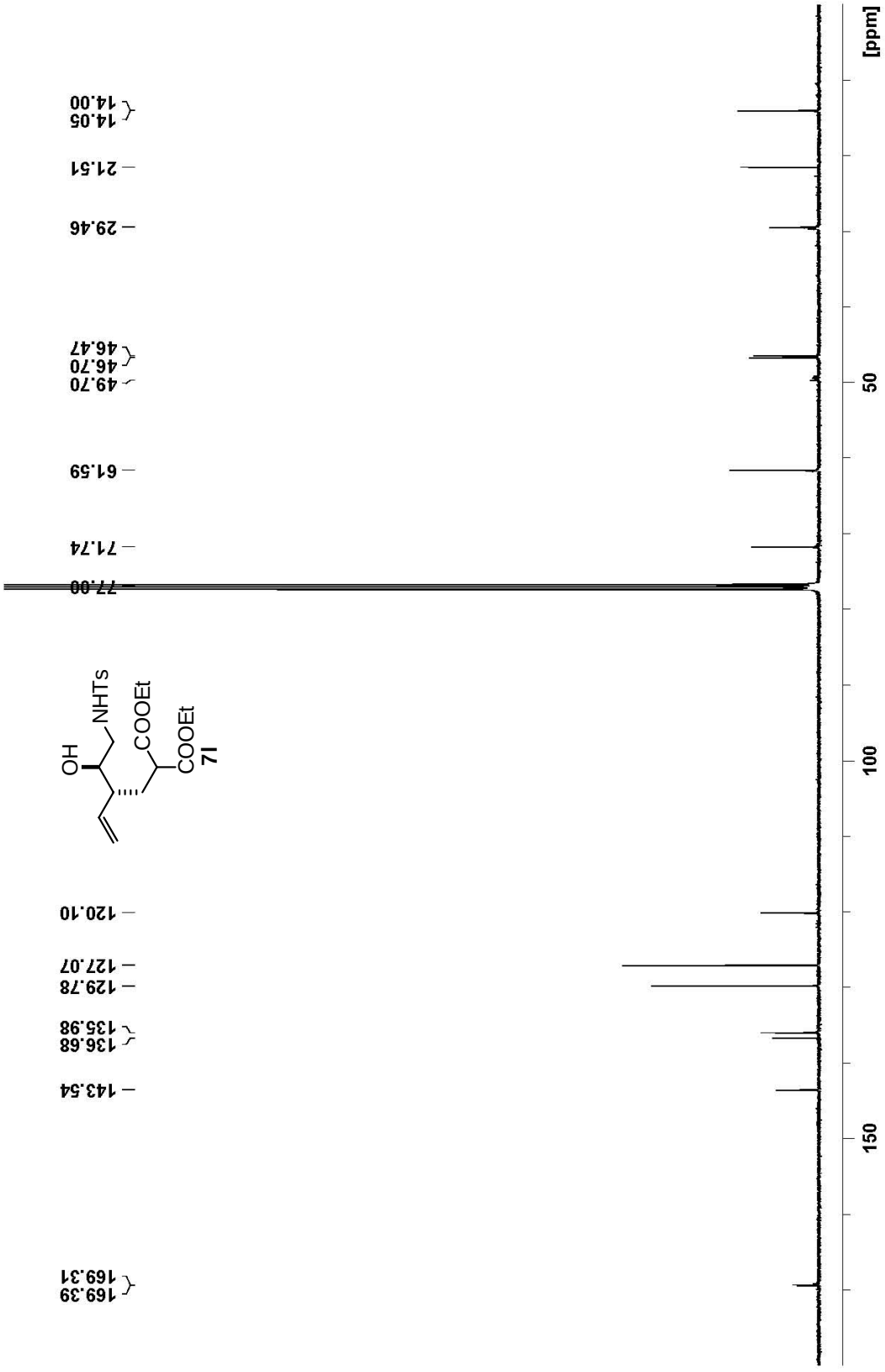
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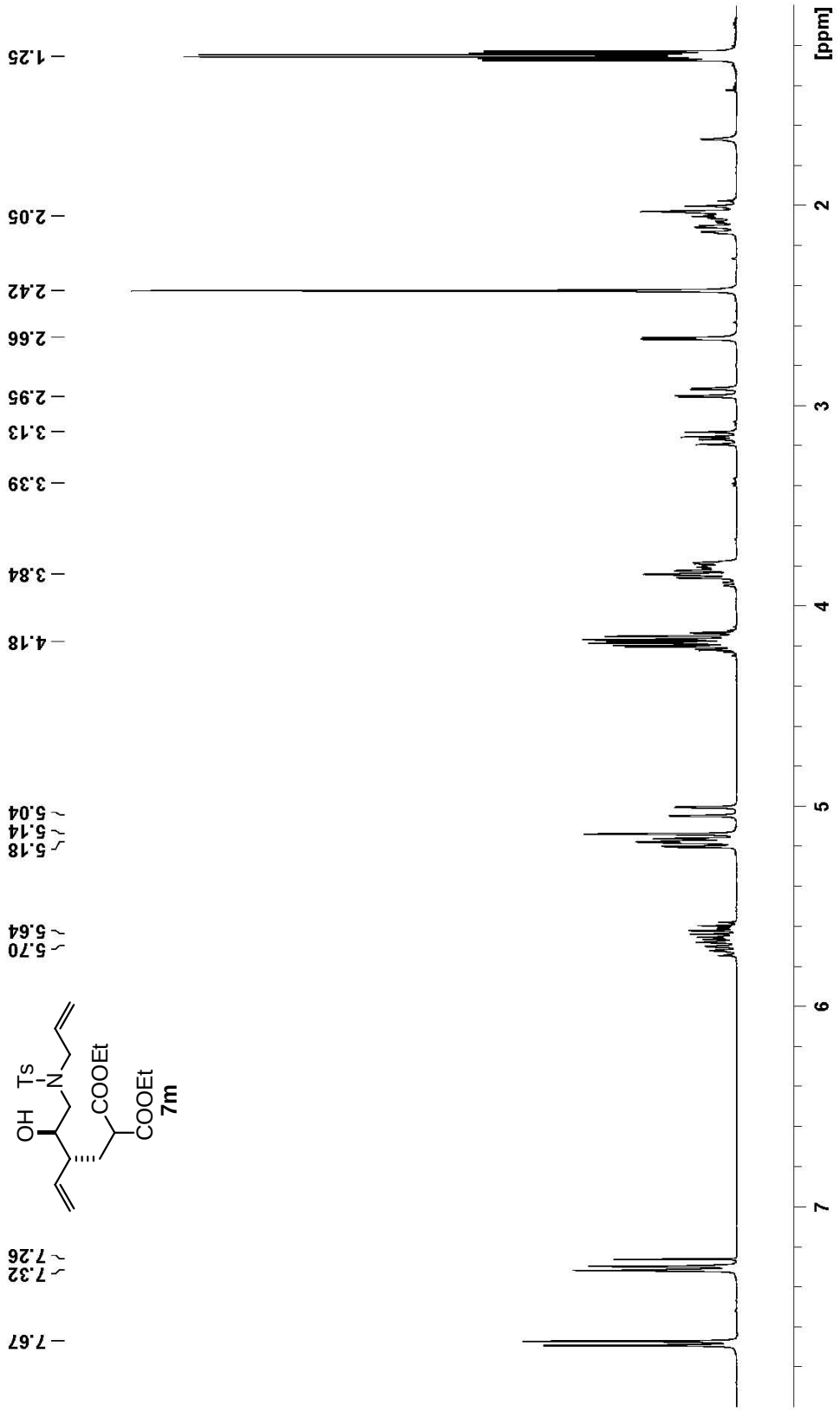
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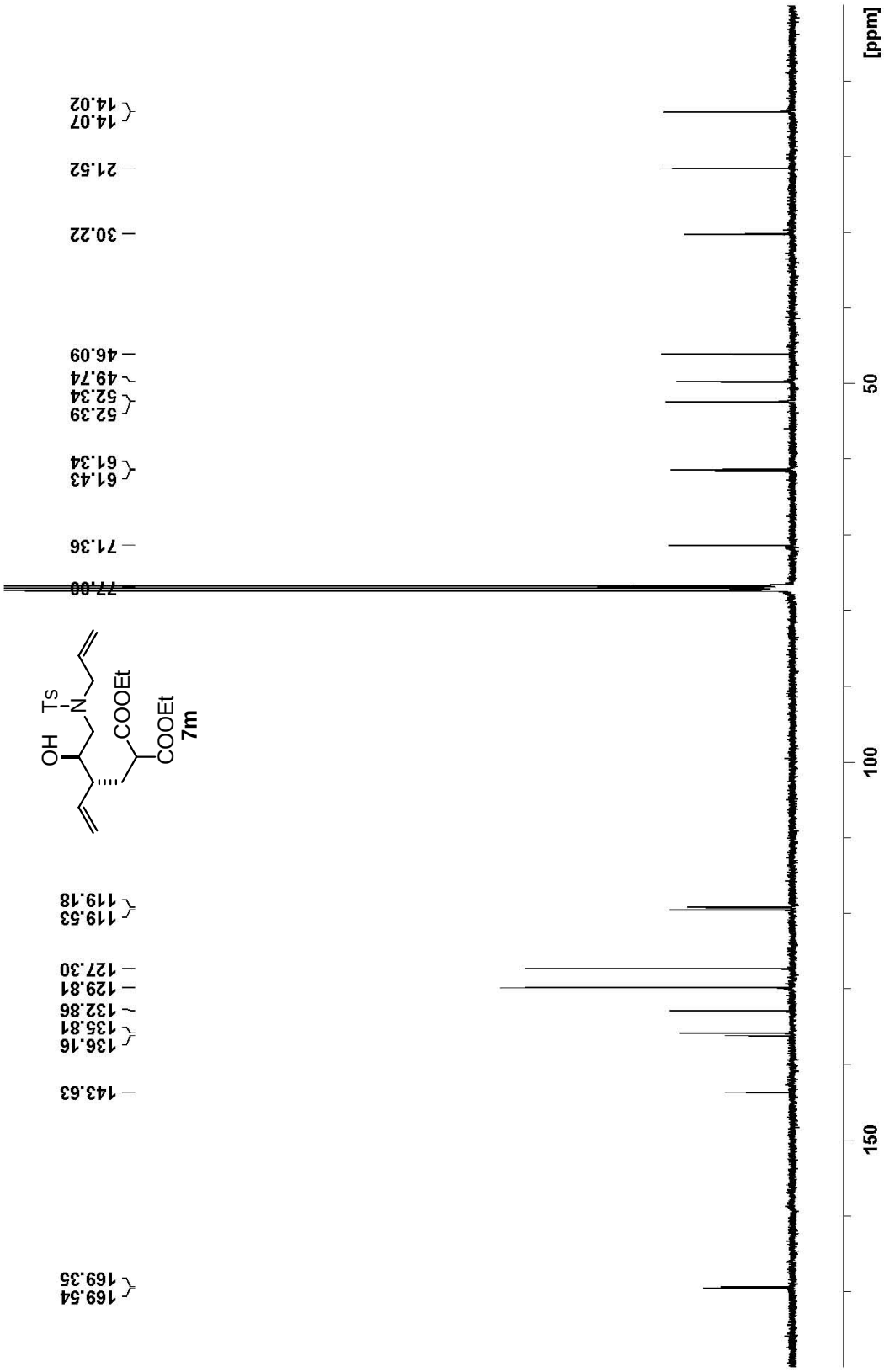
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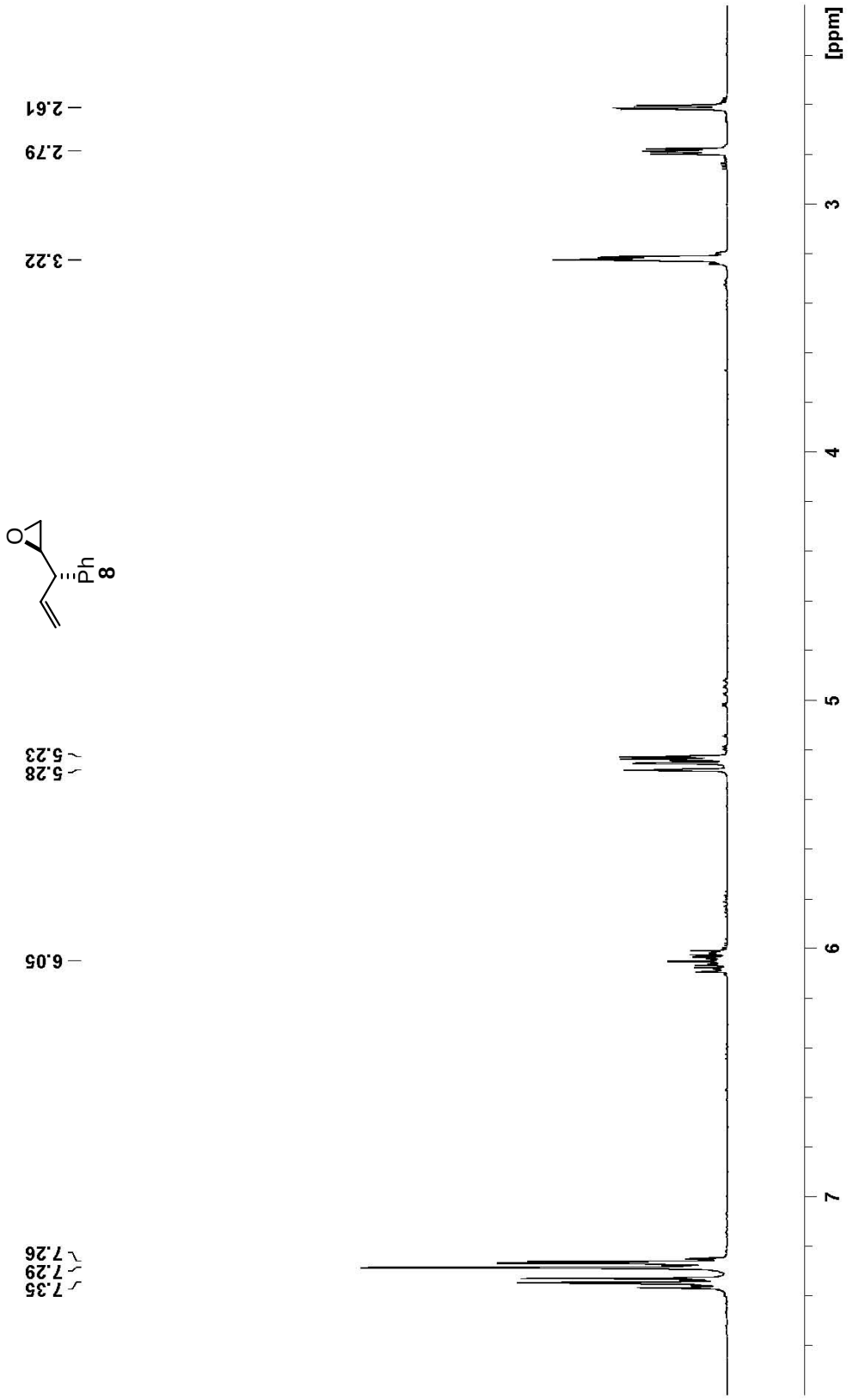
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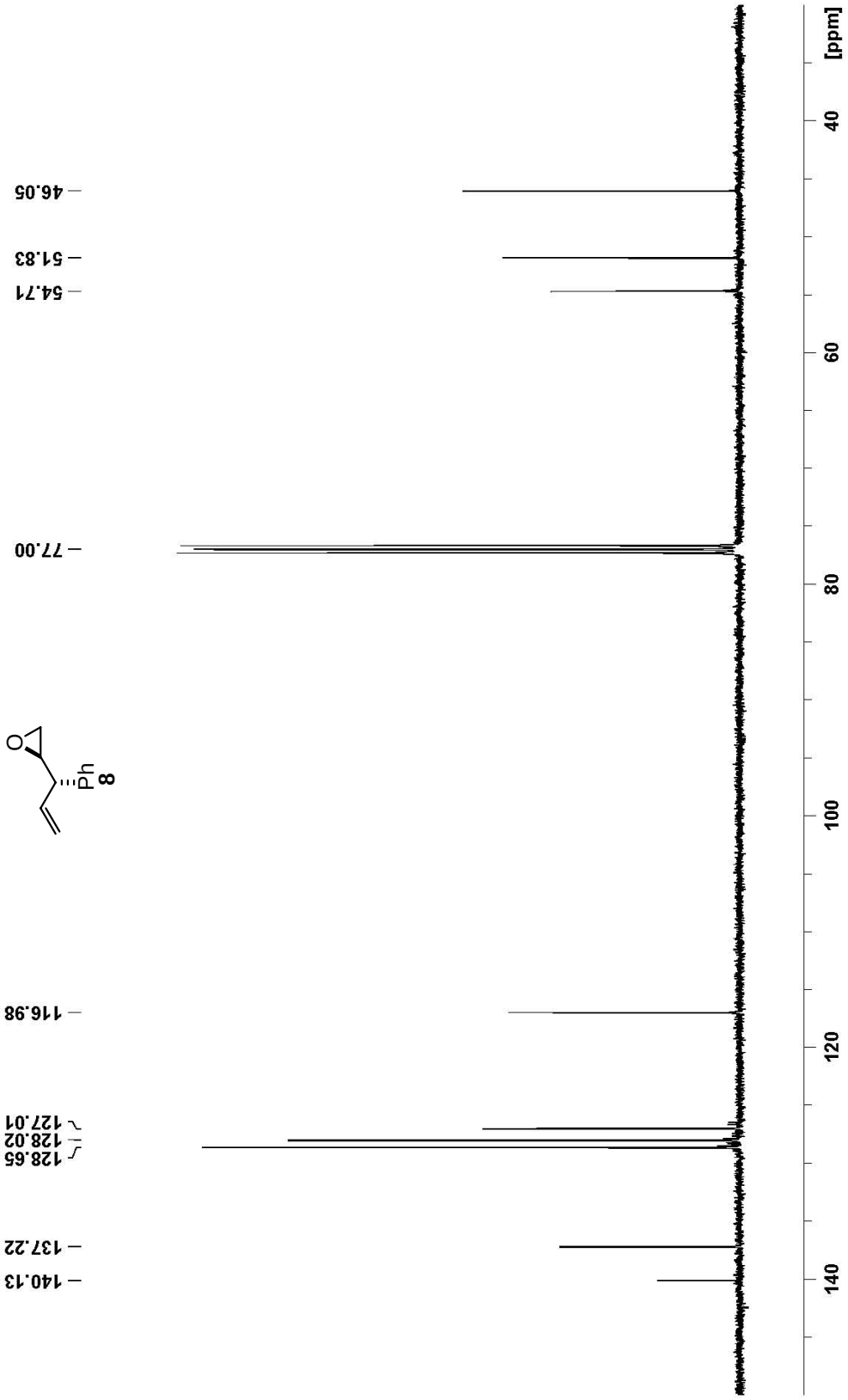
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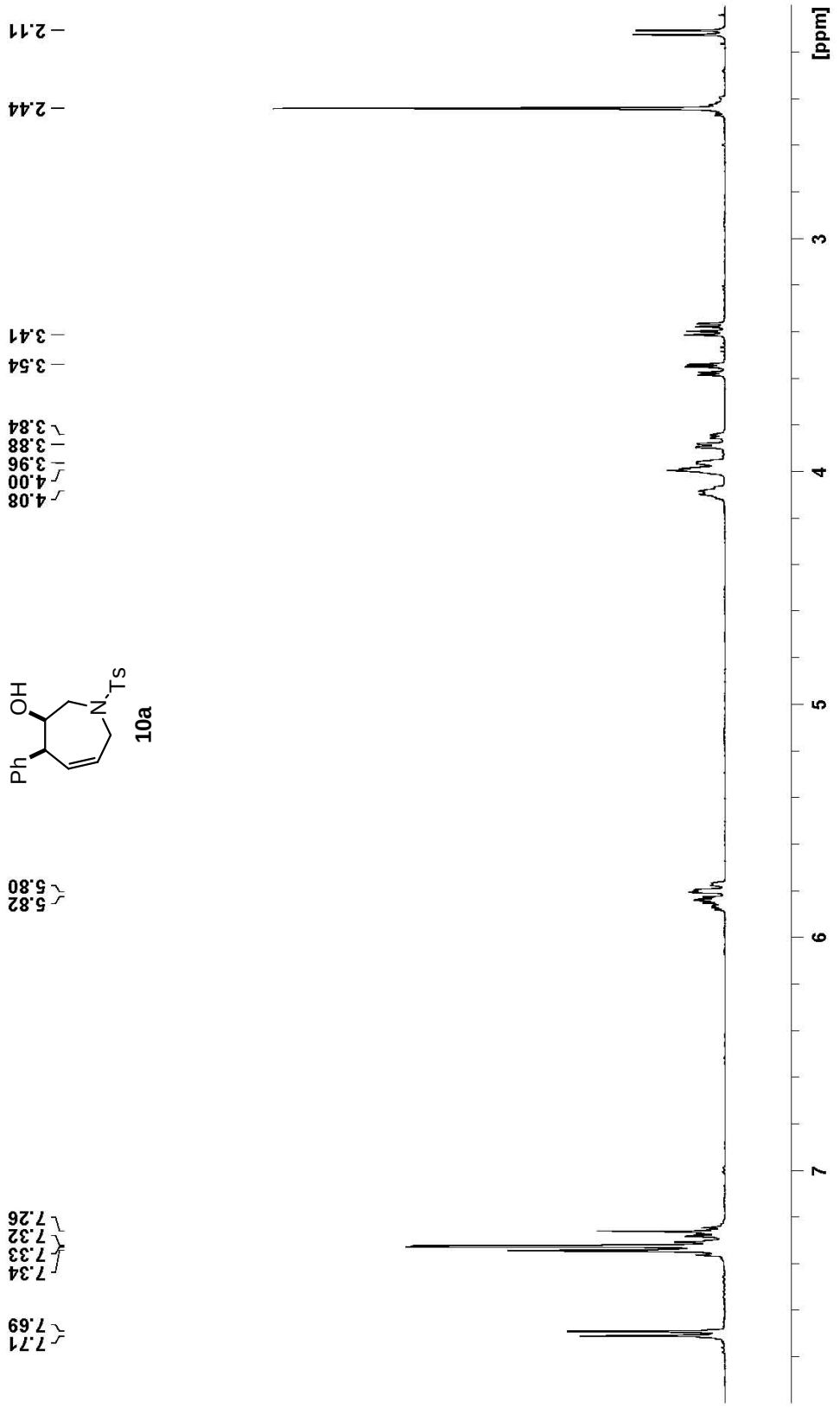
Ph
8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OH
Ph
N
Ts
10a
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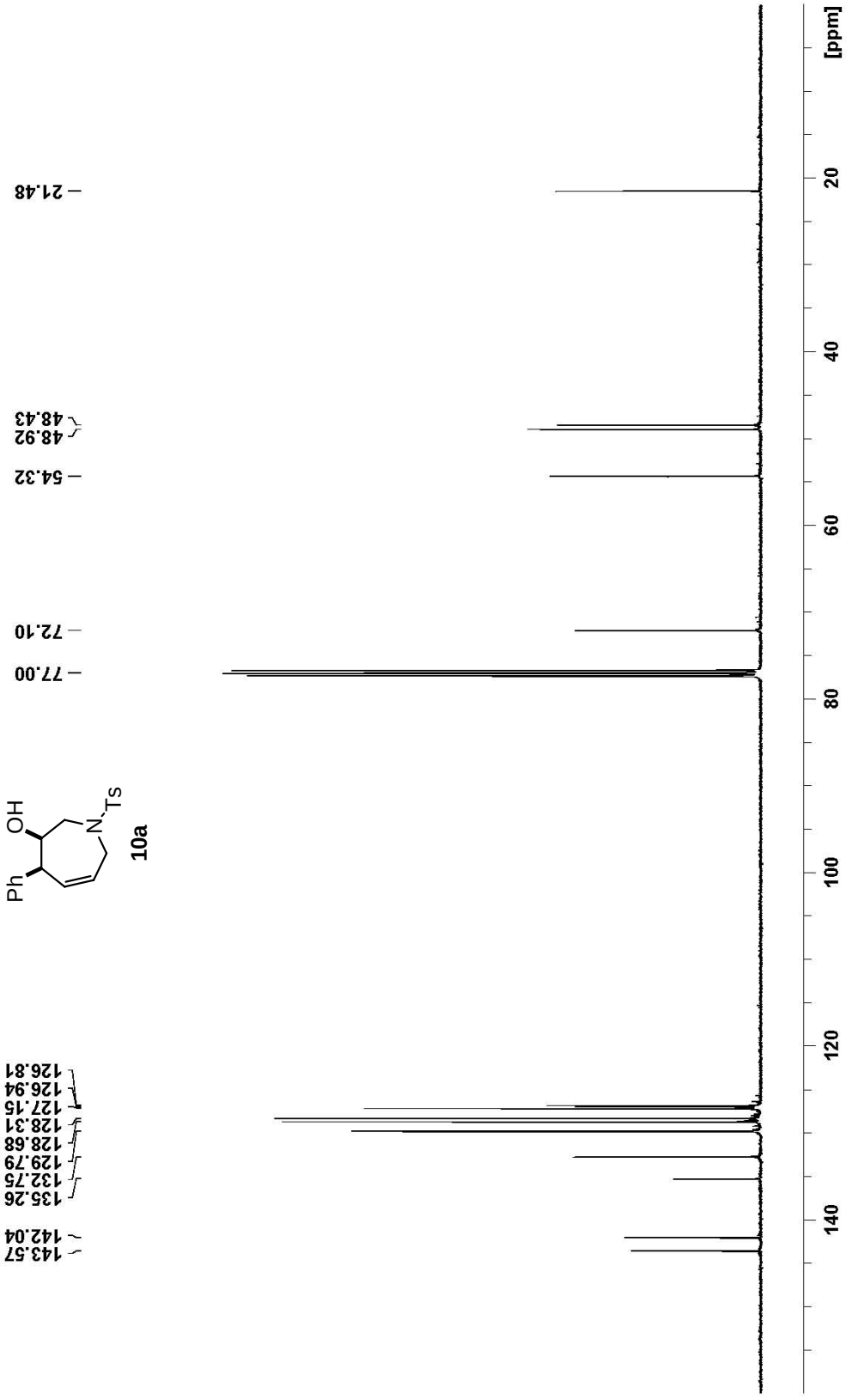
7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Ph
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Ts
10a
[ppm]
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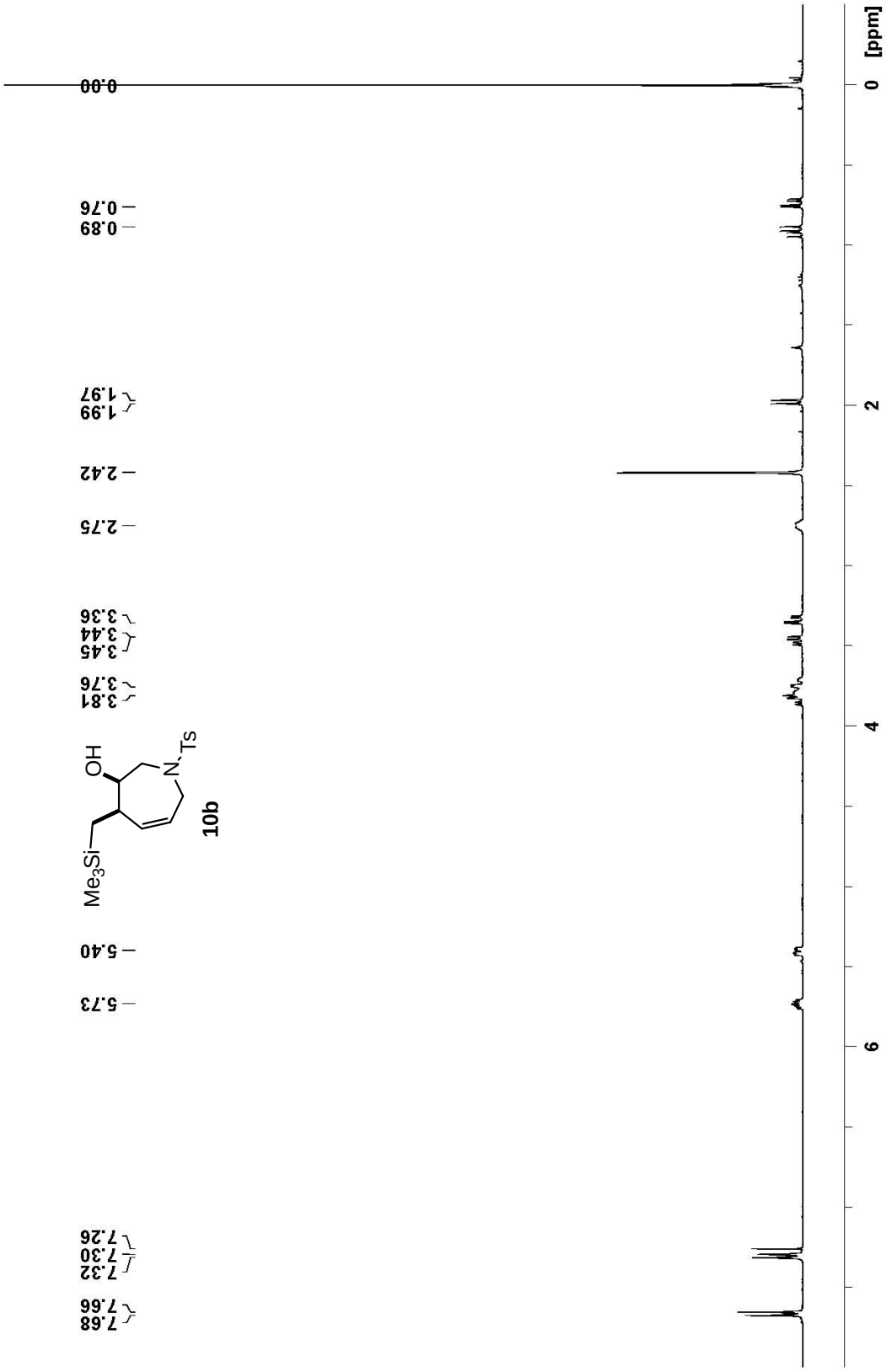
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5.73
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OH
N
Ts
Me3Si
10b
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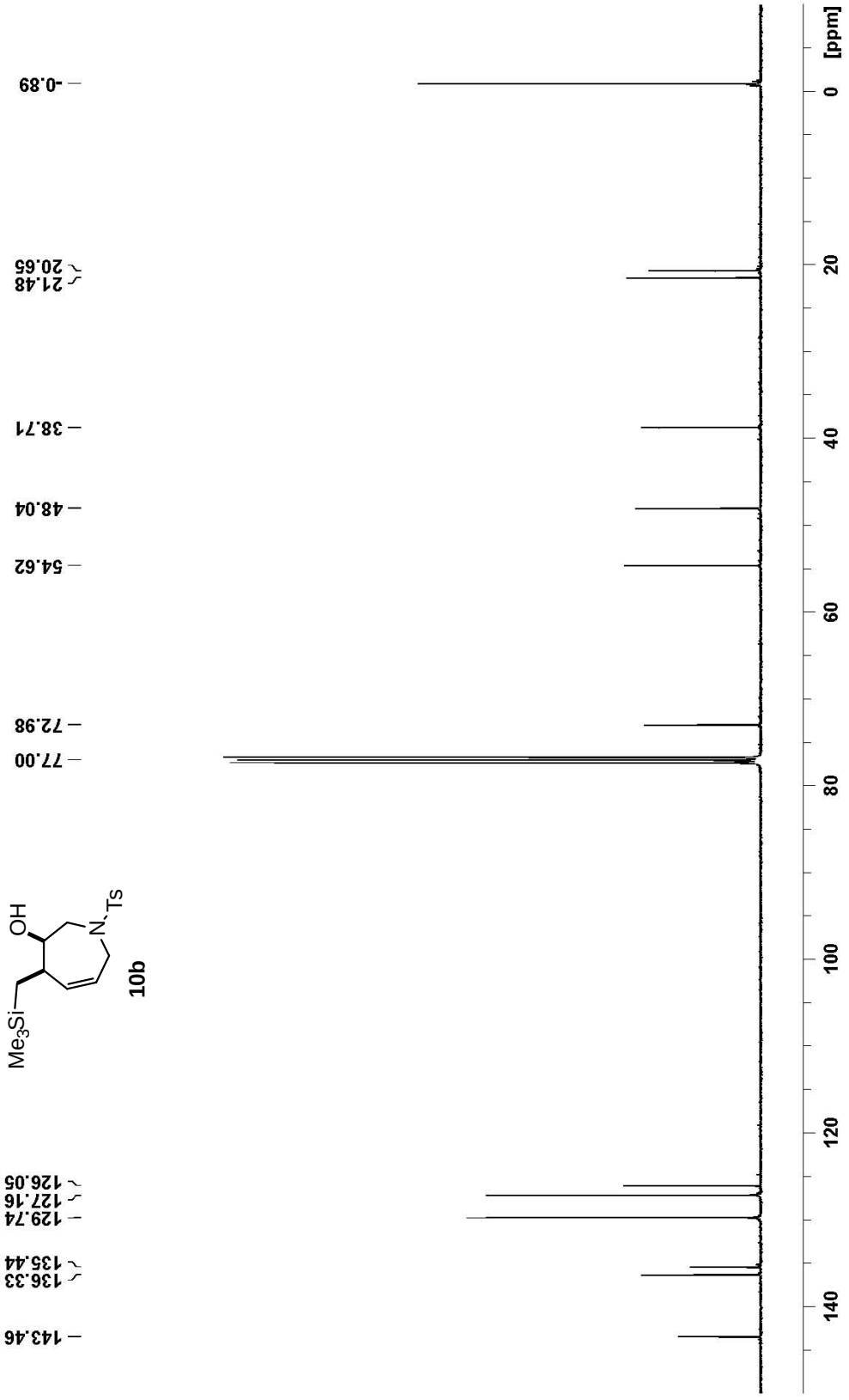
2
4
6
[ppm]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N
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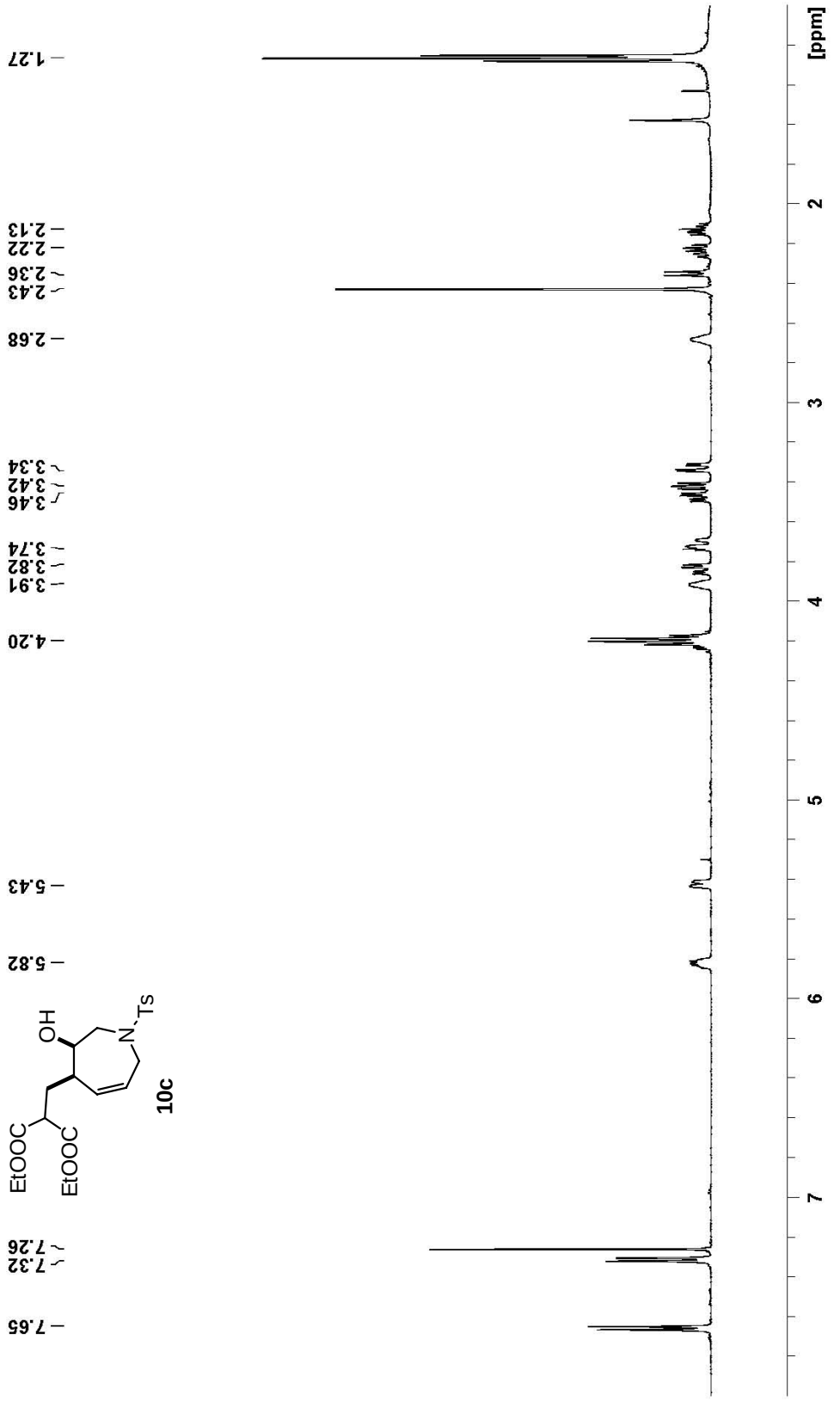
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2.22
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2.43
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3.42
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5.43
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7.26
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EtOOC
EtOOC
OH
N
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3
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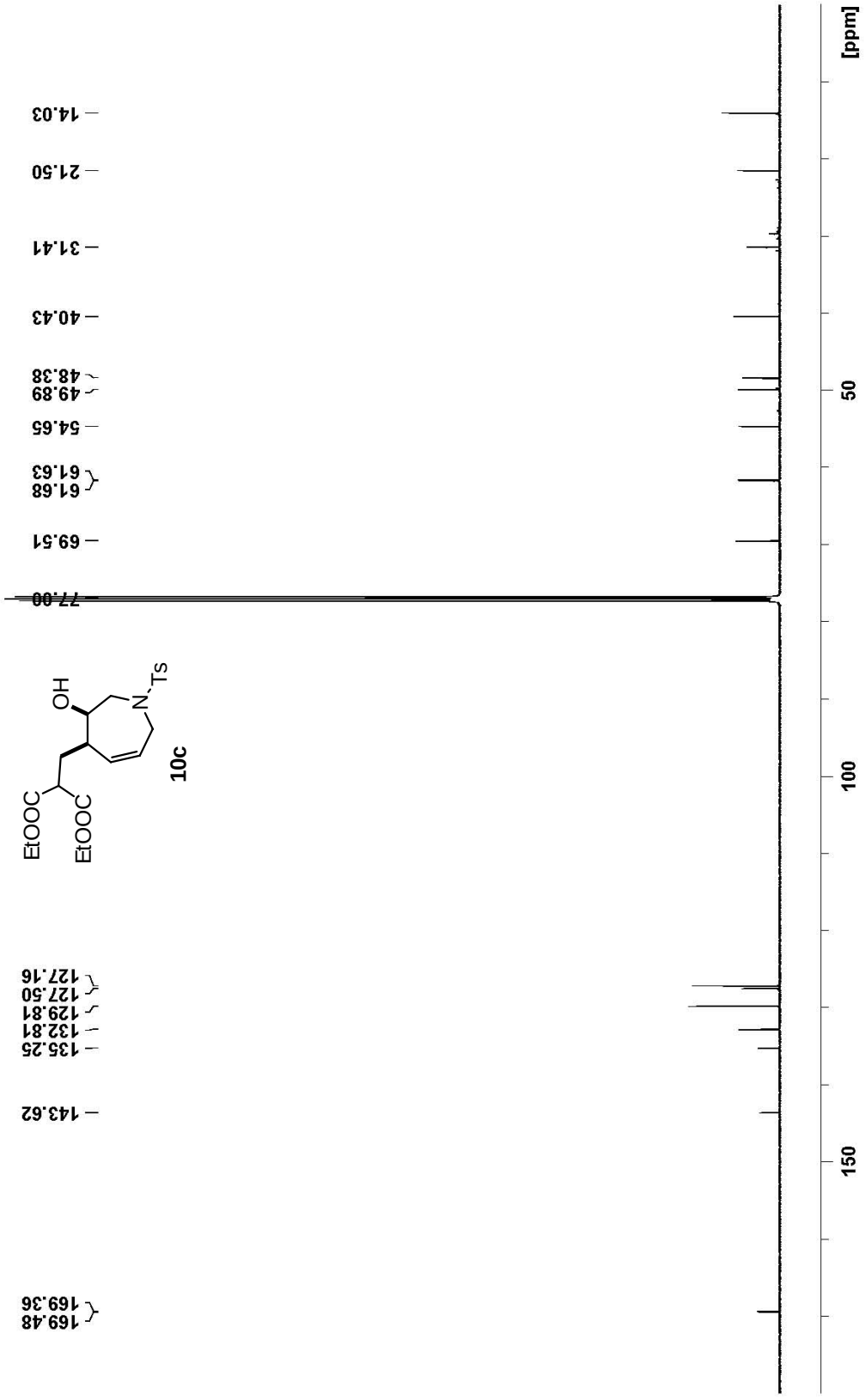
7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132.81
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150
OH
N
Ts
EtOOC
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