*Supporting Information*

# *m*CPBA-mediated metal-free intramolecular aminohydroxylation and dioxygenation of unfunctionalized olefins

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## 1. General information

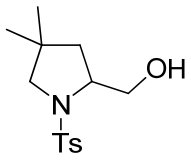
Reagents were used as received without further purification unless otherwise specified. Solvents were dried and distilled prior to use. Reactions were monitored with thin layer chromatography using silica gel $GF_{254}$ plates. Organic solutions were concentrated in *vacuo* using a rotary evaporator. Flash column chromatography was performed using silica gel (200−300 meshes). Petroleum ether used had a boiling point range of 60−90 °C. Melting points were measured on a digital melting point apparatus without correction of the thermometer. Nuclear magnetic resonance spectra were recorded at ambient temperature (unless otherwise stated) at 400 MHz (100 MHz for $^{13}C$) in $CDCl_3$. Chemical shifts are reported in ppm (δ) using TMS as internal standard, and spin−spin coupling constants (*J*) are given in Hz. High resolution mass spectrometry (HRMS) analyses were carried out on IonSpec 7.0T FTICR HR-ESI-MS.

Substrates **1a**−**1u** and **1w** were prepared according to our previous works.[1] Substrates **1v** were synthesized according to Takaki's method.[2] Substrates **8a-8f** were synthesized according to Waser's method.[3]

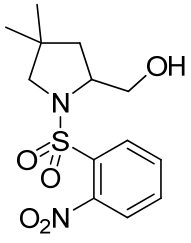
## 2. General procedures for aminohydroxylation and dioxygenation

In a 100 mL round-bottomed flask, alkenylamine (0.5mmol), *m*CPBA( 0.6mmol) were added in dry $CH_2Cl_2$ (20 mL). The resulting mixture was stirred at room temperature for a given time. Then $CH_2Cl_2$ (10 mL) was added and the mixture was washed with aqueous $Na_2S_2O_3$ and aqueous $NaHCO_3$, dried with $MgSO_4$, and concentrated to give crude residue, which was purified by flash column chromatography to give the corresponding products.

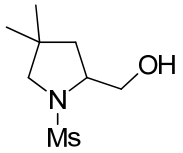
## 3. Compounds characterization



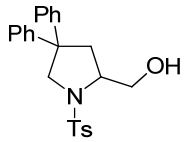

**(4,4-Dimethyl-1-tosylpyrrolidin-2-yl)methanol**(**2a**). Colorless oil.$^1H$ NMR (400 MHz, $CDCl_3$) δ = 7.68 – 7.66 (m, 2H), 7.27 – 7.25 (m, 2H), 3.73 (dd, *J* = 11.9, 3.1 Hz, 1H), 3.66 (dd, *J* = 11.9, 5.1 Hz, 1H), 3.59 – 3.52 (m, 1H), 3.26 (brs, 1H), 3.14 (d, *J* = 10.8 Hz, 1H), 3.06 (d, *J* = 10.8 Hz, 1H), 2.35 (d, *J* = 7.8 Hz, 3H), 1.64 – 1.47 (m, 2H), 0.95 (s, 3H), 0.36 (s, 3H).$^{13}C$ NMR (100 MHz, $CDCl_3$) δ = 143.8, 134.1, 129.7, 127.6, 65.9, 62.4, 62.3, 43.3, 36.9, 26.1, 25.7, 21.6. The NMR data are in accordance with those reported in the previous literature.[4]



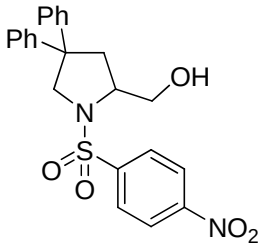
**(4,4-Dimethyl-1-(2-nitrophenylsulfonyl)pyrrolidin-2-yl)methanol**(**2b**). Colorless oil.[1]H NMR (400 MHz, $CDCl_3$) δ = 8.01 (d, *J* = 7.3 Hz, 1H), 7.64 (p, *J* = 8.0 Hz, 2H), 7.54 (d, *J* = 7.3 Hz, 1H), 4.04 (dd, *J* = 9.9, 6.3 Hz, 1H), 3.72 (dd, *J* = 11.9, 2.7 Hz, 1H), 3.54 (dd, *J* = 11.9, 4.0 Hz, 1H), 3.41 (t, *J* = 15.2 Hz, 1H), 3.07 (d, *J* = 10.7 Hz, 1H), 2.40 (brs, 1H), 1.80 – 1.58 (m, 2H), 1.05 – 0.95 (m, 3H), 0.72 (s, 3H).[13]C NMR (100 MHz, $CDCl_3$) δ = 148.1, 133.8, 132.2, 131.7, 130.7, 123.9, 64.6, 62.1, 61.9, 42.8, 37.7, 25.4.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{13}H_{18}N_2O_5S$, 315.1015; found: 315.1012.



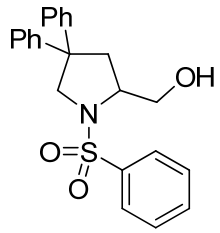

**(4,4-Dimethyl-1-(methylsulfonyl)pyrrolidin-2-yl)methanol**(**2c**). Colorless oil.[1]H NMR (400 MHz, $CDCl_3$) δ = 3.86 – 3.77 (m, 1H), 3.68 (dd, *J* = 11.7, 3.4 Hz, 1H), 3.58 (dd, *J* = 11.7, 5.2 Hz, 1H), 3.19 (d, *J* = 10.4 Hz, 1H), 3.18 (brs, 1H), 3.08 (d, *J* = 10.4 Hz, 1H), 2.87 (s, 3H), 1.80 (dd, *J* = 12.7, 7.5 Hz, 1H), 1.61 (dd, *J* = 12.7, 8.9 Hz, 1H), 1.06 (s, 3H), 1.02 (s, 3H).[13]C NMR (100 MHz, $CDCl_3$) δ = 65.4, 61.9, 61.8, 43.1, 37.5, 37.1, 26.1, 26.0.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_8H_{17}NO_3S$, 208.1007; found: 208.1003.



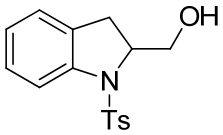

**(4,4-Diphenyl-1-tosylpyrrolidin-2-yl)methanol**(**2d**). Colorless oil.[1]HNMR (400 MHz, $CDCl_3$) δ = 7.45 (d, *J* = 8.3 Hz, 2H), 7.18 – 6.90 (m, 12H), 4.19 (d, *J* = 10.6 Hz, 1H), 3.90(d, *J* = 10.6 Hz, 1H), 3.67 – 3.64 (m, 1H), 3.59 – 3.47 (m, 2), 3.05 (brs, 1H), 2.69 (dd, *J* = 12.3, 7.5 Hz, 1H), 2.38 (dd, *J* = 13.0, 7.5 Hz, 1H), 2.28 (s, 3H).[13]C NMR (100 MHz, $CDCl_3$) δ = 145.3, 144.3, 143.6, 134.0, 129.7, 128.6, 128.6, 127.3, 126.6, 126.6, 126.4, 126.3, 65.4, 61.7, 59.7, 52.2, 40.8, 21.5.The NMR data are in accordance with those reported in the previous literature.[5]



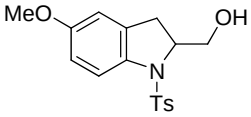

**(1-(4-Nitrophenylsulfonyl)-4,4-diphenylpyrrolidin-2-yl)methanol**(**2e**). White solid, Mp=155-156 °C.[1]H NMR (400 MHz, $CDCl_3$) δ =7.96 – 7.94 (m, 2H), 7.62 – 7.58 (m, 2H), 7.22 – 6.85 (m, 10H), 4.20 (q, *J* = 11.1 Hz, 2H), 3.77 – 3.70 (m, 3H),, 2.95 (s, 1H), 2.94 – 2.90 (m, 1H), 2.34 (dd, *J* = 13.3, 8.9 Hz, 1H).[13]C NMR (100 MHz, $CDCl_3$) δ = 148.7, 143.9, 142.4, 142.0, 127.7, 127.6, 126.9, 125.8, 125.6, 125.4, 125.3, 123.0, 64.3, 61.1, 59.9, 51.5, 38.9.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{23}H_{22}N_2O_5S$, 439.1328; found: 439.1323.



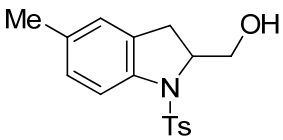
**(4,4-Diphenyl-1-(phenylsulfonyl)pyrrolidin-2-yl)methanol**(**2f**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.64 – 7.55 (m, 2H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.8 Hz, 2H), 7.18 (dd, *J* = 9.8, 5.2 Hz, 2H), 7.15 – 7.08 (m, 3H), 7.03 – 6.91 (m, 5H), 4.20 (d, *J* = 10.6 Hz, 1H), 3.98 (dd, *J* = 10.6, 0.9 Hz, 1H), 3.70 – 3.62 (m, 1H), 3.59 – 3.55 (m, 2H), 2.85 (t, *J* = 6.6 Hz, 1H), 2.73 – 2.67 (m, 1H), 2.42 (dd, *J* = 13.0, 7.6 Hz, 1H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 144.1, 143.2, 136.2, 131.7, 128.1, 127.6, 127.6, 126.2, 125.7, 125.6, 125.5, 125.3, 64.3, 60.7, 58.7, 51.1, 39.9.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{23}H_{23}NO_3S$, 394.1477; found: 394.1478.



**(1-Tosylindolin-2-yl)methanol**(**2g**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.63 – 7.60 (m, 2H), 7.46 – 7.44 (m, 2H),, 7.18 – 7.12 (m, 1H), 7.09 (d, *J* = 8.1 Hz, 2H), 7.01 – 6.92 (m, 2H), 4.22 (td, *J* = 9.1, 5.6 Hz, 1H), 3.65 (d, *J* = 5.6 Hz, 2H), 2.72 (dd, *J* = 16.4, 9.7 Hz, 1H), 2.57 (dd, *J* = 16.4, 2.9 Hz, 1H), 2.46 (brs, 1H), 2.27 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 143.2, 1403, 133.3, 131.0, 128.7, 126.8, 126.1, 124.1, 124.0, 116.6, 64.5, 62.5, 30.2, 20.5.The NMR data are in accordance with those reported in the previous literature.[6]

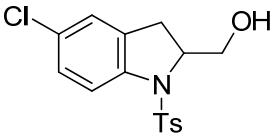


**(5-Methoxy-1-tosylindolin-2-yl)methanol**(**2h**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.52 – 7.49 (m, 2H), 7.42 (d, *J* = 7.9 Hz, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 6.69 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.51 (d, *J* = 2.2 Hz, 1H), 4.28 – 4.14 (m, 1H), 3.68 (s, 3H), 3.60 (d, *J* = 6.4 Hz, 2H), 2.60 (dd, *J* = 16.5, 9.4 Hz, 1H), 2.48 (brs, 1H),2.46 (dd, *J* = 16.5, 2.7 Hz, 1H), 2.28 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 157.7, 144.2, 134.5, 134.1, 134.0, 129.7, 127.2, 118.9, 113.0, 110.7, 65.3, 63.9, 55.6, 31.4, 21.6.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for$C_{17}H_{19}NO_4S$, 334.1113; found: 334.1111.

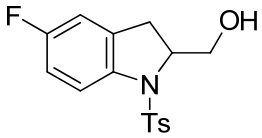


**(5-Methyl-1-tosylindolin-2-yl)methanol**(**2i**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.47 (dd, *J* = 17.2, 8.2 Hz, 3H), 7.09 (d, *J* = 8.2 Hz, 2H), 6.94 (d, *J* = 8.2 Hz, 1H), 6.78 (s, 1H), 4.22 – 4.18 (m, 1H), 3.62 (d, *J* = 5.7 Hz, 2H), 2.66 (dd, *J* = 16.3, 9.6 Hz, 1H), 2.49 (dd, *J* = 16.5, 2.7 Hz, 1H), 2.45 (brs, 1H), 2.28 (s, 3H), 2.20 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 144.1, 138.9, 134.9, 134.3, 132.2, 129.7, 128.5,

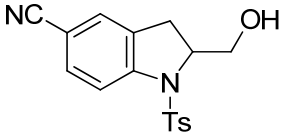
127.2, 125.7, 117.5, 65.5, 63.7, 31.2, 21.6, 21.0.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{19}NO_3S$, 318.1164; found: 318.1160.



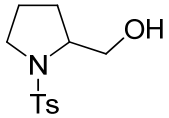

**(5-Chloro-1-tosylindolin-2-yl)methanol**(**2j**). Colorless oil.$^1$H NMR (400 MHz, $CDCl_3$) δ = 7.50 (t, *J* = 9.0 Hz, 1H), 7.47 – 7.44 (m, 2H), 7.10 (dd, *J* = 13.7, 5.1 Hz, 3H), 6.93 (s, 1H), 4.26 – 4.21 (m, 1H), 3.65 (d, *J* = 5.6 Hz, 2H), 2.70 (dd, *J* = 16.7, 9.6 Hz, 1H), 2.65 (brs, 1H), 2.60 (dd, *J* = 16.7, 3.3 Hz, 1H), 2.28 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 144.6, 140.2, 134.1, 130.3, 129.9, 127.9, 127.1, 125.4, 118.3, 65.4, 63.8, 31.1, 21.6.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{16}H_{16}ClNO_3S$, 338.0618; found: 338.0614




**(5-Fluoro-1-tosylindolin-2-yl)methanol** (**2k**). Colorless oil.$^1$H NMR (400 MHz, $CDCl_3$) δ = 7.56 (dd, *J* = 8.8, 4.6 Hz, 1H), 7.44 (d, *J* = 8.3 Hz, 2H), 7.12 (d, *J* = 5.4 Hz, 2H), 6.85 (td, *J* = 8.8, 2.5 Hz, 1H), 6.68 (dd, *J* = 8.3, 2.3 Hz, 1H), 4.26 – 7.22 (m, 1H), 3.67 – 3.60 (m, 2H), 2.67 (dd, *J* = 16.7, 9.5 Hz, 1H), 2.54 (dd, *J* = 16.7, 2.9 Hz, 1H), 2.32 (s, 1H), 2.30 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 162.8(d, J = 242.2 Hz), 144.4, 137.5, 134.4 (d, *J* = 8.8 Hz), 134.0, 129.8, 127.2, 118.8 (d, *J* = 8.7 Hz), 114.5 (d, *J* = 23.4 Hz), 112.3 (d, *J* = 24.0 Hz), 65.4, 63.9, 31.2, 21.6.$^{19}$F NMR (376 MHz, $CDCl_3$) δ = -117.7 (m).HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{16}H_{16}FNO_3S$, 322.0913; found:322.0911.

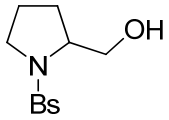



**2-(Hydroxymethyl)-1-tosylindoline-5-carbonitrile**(**2l**). Colorless oil.$^1$H NMR (400 MHz, $CDCl_3$) δ= 7.66 – 7.64 (m, 2H), 7.51 (d, *J* = 8.3 Hz, 2H), 7.43 (d, *J* = 8.3 Hz, 1H), 7.25 (s, 1H), 7.16 (d, *J* = 8.2 Hz, 2H), 4.31 – 4.28 (m, 1H),, 3.76 (dd, *J* = 11.5, 5.0 Hz, 1H), 3.71 (dd, *J* = 11.5, 5.0 Hz, 1H), 2.90 – 2.77 (m, 2H), 2.66 (brs, 1H), 2.30 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ= 144.7, 144.0, 133.1, 131.9, 131.6, 129.0, 127.9, 125.9, 117.8, 115.6, 106.7, 64.3, 62.7, 29.9, 20.6.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{16}N_2O_3S$, 329.0960 found:329.0953.

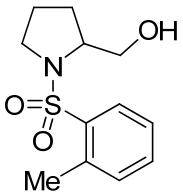



**(1-Tosylpyrrolidin-2-yl)methanol**(**2m**). Colorless oil.$^1$H NMR (400 MHz, $CDCl_3$) δ = 7.67 – 7.65 (m, 2H), 7.27 – 7.5 (m, 2H), 3.63 – 3.48 (m, 3H), 3.38 (dt, *J* = 10.7, 6.7 Hz, 1H), 3.18 (dt, *J* = 10.0, 6.7 Hz, 1H), 2.90 (brs, 1H), 2.37 (s, 3H), 1.78 – 1.64 (m, 1H), 1.65 – 1.53 (m, 2H), 1.38 (td, *J* = 12.5, 6.3 Hz, 1H).$^{13}$C NMR (100 MHz, $CDCl_3$)

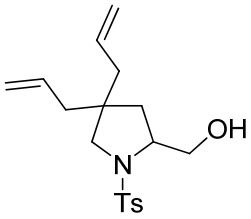
δ = 143.8, 133.8, 129.8, 127.6, 65.8, 61.8, 50.0, 28.8, 24.2, 21.6. The NMR data are in accordance with those reported in the previous literature.[6]



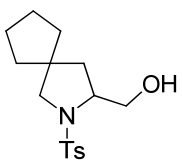

**(1-(Phenylsulfonyl)pyrrolidin-2-yl)methanol**(**2n**).Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.79 (d, *J* = 7.6 Hz, 2H), 7.56 (d, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 3.70 – 3.54 (m, 3H), 3.40 (dt, *J* = 10.4, 6.2 Hz, 1H), 3.19 (dt, *J* = 10.4, 6.9 Hz, 1H), 2.87 (brs, 1H), 1.81 – 1.68 (m, 1H), 1.69 – 1.54 (m, 2H), 1.43 – 1.33 (m, 1H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 136.8, 133.0, 129.2, 127.6, 65.7, 61.9, 50.0, 28.8, 24.2.The NMR data are in accordance with those reported in the previous literature.[7]




**(1-(*o*-Tolylsulfonyl)pyrrolidin-2-yl)methanol**(**2o**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.85 (d, *J* = 8.2 Hz, 1H), 7.38 (q, *J* = 7.4 Hz, 1H), 7.28 – 7.20 (m, 2H), 3.83 (dq, *J* = 9.6, 4.8 Hz, 1H), 3.61 – 3.51 (m, 2H), 3.28 – 3.23 (m, 2H), 2.69 (brs, 1H), 2.60 (s, 3H), 1.93 – 1.73 (m, 3H), 1.71 – 1.64 (m, 1H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 136.9, 135.6, 131.9, 131.9, 128.8, 125.3, 64.31, 60.1, 48.4, 28.0, 23.5,19.8.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{12}H_{17}NO3S$, 256.1007; found: 256.1006.

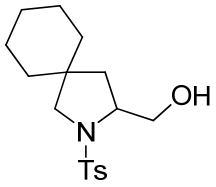



**(4,4-Diallyl-1-tosylpyrrolidin-2-yl)methanol**(**2p**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.69 – 7.65 (m, 2H), 7.28 – 7.26 (m, 2H), 5.62 – 5.59 (m, 1H),, 5.48 – 5.36 (m, 2H), 4.99 (dd, *J* = 12.3, 10.0 Hz, 1H), 4.89 (d, *J* = 10.0 Hz, 1H), 4.66 (d, *J* = 16.9 Hz, 1H), 3.74 (dd, *J* = 9.7, 6.9 Hz, 1H), 3.68 – 3.61 (m, 1H), 3.56 – 3.47 (m, 1H), 3.20 – 3.08 (m, 3H), 2.36 (s, 3H), 2.02 (d, *J* = 7.5 Hz, 2H), 1.69 (dd, *J* = 13.0, 7.5 Hz, 1H), 1.57 – 1.49 (m, 2H), 1.32 (dd, *J* = 14.1, 8.0 Hz, 1H).$^{13}$C NMR (100MHz, $CDCl_3$) δ = 142.9, 133.1, 132.5, 132.1, 128.8, 126.5, 117.6, 117.5, 64.7, 60.7, 58.2, 41.9, 39.6, 38.2, 37.9, 20.5.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{25}NO_3S$, 336.1633; found: 336.1632.

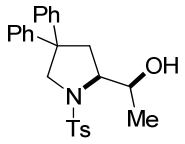



**(2-Tosyl-2-azaspiro[4.4]nonan-3-yl)methanol**(**2q**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.68 – 7.66 (m, 2H), 7.29 – 7.26 (m, 2H), 3.53 – 7.49 (m, 1H), 3.24 (d, *J* =

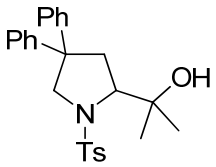
10.6 Hz, 2H), 3.17 (brs, 1H), 3.08 (d, *J* = 10.6 Hz, 1H), 2.37 (s, 3H), 1.72 – 1.32 (m, 8H), 0.87 – 0.78 (m, 1H), 0.75 – 0.58 (m, 1H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 143.8, 133.9, 129.7, 127.6, 66.0, 62.5, 60.7, 42.8, 41.4, 36.5, 36.1, 24.3, 24.3, 21.6.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for$C_{16}H_{23}NO_3S$, 310.1477; found: 310.1475.



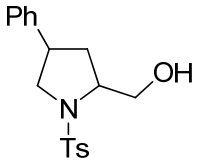

**(2-Tosyl-2-azaspiro[4.5]decan-3-yl)methanol**(**2r**). Colorless oil.$^1$H NMR (400 MHz, $CDCl_3$) δ = 7.69 – 7.67 (m, 2H), 7.26 – 7.24 (m, 2H), 3.71 (dd, *J* = 11.8, 3.2 Hz, 1H), 3.65 (dd, *J* = 11.8, 5.1 Hz, 1H), 3.48 (tdd, *J*= 8.6, 5.1, 3.4 Hz, 1H), 3.35 (brs, 1H), 3.25 (d, *J* = 11.1 Hz, 1H), 3.08 (d, *J* = 11.1 Hz, 1H), 2.35 (s, 3H), 1.66 (dd, *J* = 12.7, 7.4 Hz, 1H), 1.45 (dd, *J* = 12.8, 9.5 Hz, 1H), 1.38 – 1.23 (m, 4H), 1.19 – 1.00 (m, 4H), 0.63 (ddd, *J* = 13.0, 9.0, 3.9 Hz, 1H), 0.54 – 0.42 (m, 1H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 142.8, 133.0, 128.7, 126.5, 64.9, 60.4, 58.5, 40.5, 39.8, 35.2, 32.9, 24.7, 22.7, 21.7, 20.5. The NMR data are in accordance with those reported in the previous literature.[6]



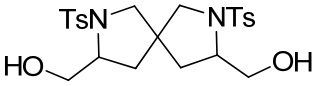

**1-(4,4-Diphenyl-1-tosylpyrrolidin-2-yl)ethanol**(**2s**). White solid, mp=180°C, $^1$H NMR (400 MHz, $CDCl_3$) δ = 7.37 – 7.34 (m, 2H),, 7.20 – 7.13 (m, 2H), 7.10 – 7.01 (m, 3H), 7.02 – 6.92 (m, 7H), 4.36 (dd, *J* = 10.9, 1.6 Hz, 1H), 4.27 (brs, 1H), 4.00 (d, *J* = 10.9 Hz, 1H), 3.65 – 3.56 (m, 1H), 2.79 – 2.68 (m, 1H), 2.46 (dd, *J* = 13.0, 10.3 Hz, 2H), 2.28 (s, 3H), 1.12 (d, *J* = 6.5 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ = 144.7, 142.9, 142.1, 134.2, 128.6, 127.5, 127.5, 125.8, 125.5, 125.5, 125.5, 125.1, 66.2, 64.3, 59.50, 51.2, 36.3, 20.4, 16.9.The NMR data are in accordance with those reported in the previous literature.[5]



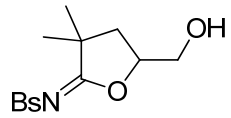

**2-(4,4-Diphenyl-1-tosylpyrrolidin-2-yl)propan-2-ol** (**2t**). Colorless oil.$^1$H NMR (400 MHz, $CDCl_3$) δ = 7.20 – 7.14 (m, 4H), 7.08 (t, *J* = 7.3 Hz, 1H), 7.00 – 6.81 (m, 9H), 4.81 (s, 1H), 4.53 (dd, *J* = 11.6, 2.1 Hz, 1H), 4.15 (d, *J* = 11.6 Hz, 1H), 3.75 (dd, *J* = 10.5, 6.9 Hz, 1H), 3.06 –3.02 (m, 1H),, 2.25 (s, 3H), 1.97 (dd, *J* = 13.6, 10.5 Hz, 1H), 1.28 (s, 3H), 1.17 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 145.0, 142.3, 141.7, 133.3, 128.3, 127.6, 127.5, 125.9, 125.6, 125.5, 125.2, 125.1, 71.8, 68.9, 61.5, 51.0, 40.8, 25.7, 21.4, 20.4.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{26}H_{29}NO_3S$, 436.1946; found: 436.1943.



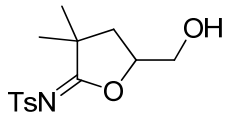

**(4-Phenyl-1-tosylpyrrolidin-2-yl)methanol**(**2u**). (DiastereomerMixtures, dr =60: 40). Colorless oil. Majordiastereomer:$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.67 – 7.65 (m, 2H), 7.24 (d, *J* = 7.8 Hz, 2H), 7.20 – 7.08 (m, 5H), 3.81 (dd, *J* = 15.6, 7.4 Hz, 2H), 3.76 – 3.63 (m, 2H), 3.50 – 3.39 (m, 1H), 2.94 (brs, 1H), 2.93 (t, *J* = 9.9 Hz, 1H), 2.35 (s, 3H), 2.08 (dd, *J* = 12.7, 6.5 Hz, 1H), 1.67 (dt, *J* = 20.5, 10.3 Hz, 1H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 144.0, 139.7, 133.2, 129.9, 128.7, 127.8, 127.0, 126.9, 66.1, 61.6, 55.9, 41.9, 35.7, 21.6.The NMR data are in accordance with those reported in the previous literature.[6]



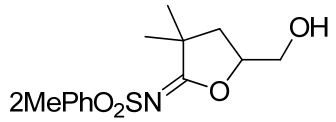

**(2,7-Ditosyl-2,7-diazaspiro[4.4]nonane-3,8-diyl)dimethanol**(**2v**).
(DiastereomerMixtures, dr =2 : 1: 1). Colorless oil.Majordiastereomer:$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.56 – 7.52 (m, 4H), 7.30 – 7.24 (m, 4H), 3.69 – 3.55 (m, 2H), 3.49 – 3.37 (m, 4H), 2.98 (brs, 2H), 2.74 (d, *J* = 10.8 Hz, 2H), 2.52 (d, *J* = 10.8 Hz, 2H), 2.45 (s, 6H), 1.87 (dd, *J* = 13.1, 8.6 Hz, 2H), 1.66 (dd, *J* = 12.9, 6.4 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ = 145.1, 144.5, 129.9, 127.7, 65.2, 61.3, 57.9, 46.3, 38.5, 21.7.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{23}H_{30}N2O_6S_2$ 495.1624; found:495.1615.



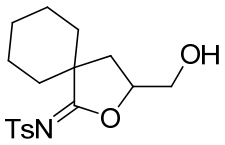

**N-(5-(Hydroxymethyl)-3,3-dimethyldihydrofuran-2(3H)-ylidene)benzenesulfonamide**(**9a**). White solid, mp=77-79 °C.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.98 – 7.82 (m, 2H), 7.55 – 7.47 (m, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 4.82 – 4.68 (m, 1H), 3.81 (d, *J* = 12.7 Hz, 1H), 3.61 – 3.49 (m, 1H), 3.31 (brs, 1H), 1.99 – 1.84 (m, 2H), 1.21 (s, 3H), 1.20 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 180.7, 141.2, 132.7, 128.6, 127.2, 85.2, 62.7, 44.7, 37.6, 26.1, 25.4.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{13}H_{17}NO_4S$, 284.0957; found:284.0957.



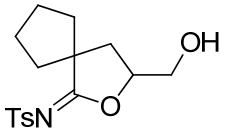

**N-(5-(Hydroxymethyl)-3,3-dimethyldihydrofuran-2(3H)-ylidene)-4-methylbenzenesulfonamide**(**9b**).White solid, mp=131-132°C.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.80 – 7.78(m, 2H), 7.23 – 7.20 (m, 2H), 4.76 – 4.68 (m, 1H), 3.84 (dd, *J* = 12.9, 2.0 Hz, 1H), 3.51 (dd, *J* = 12.9, 4.1 Hz, 1H), 3.17 (brs, 1H), 2.34 (s, 3H), 1.93 (d, *J* = 8.1 Hz, 2H), 1.21 (s, 3H), 1.20 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 180.4, 143.5, 138.2, 129.2, 127.3, 85.1, 62.7, 44.6, 37.6, 26.1, 25.4, 21.6.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{19}NO_4S$, 298.1113; found:298.1111.



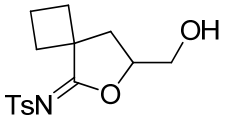

**N-(5-(Hydroxymethyl)-3,3-dimethyldihydrofuran-2(3H)-ylidene)-2-methylbenzenesulfonamide**(**9c**).White solid, mp=94-95°C.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 8.01 – 7.91 (m, 1H), 7.37 (td, *J* = 7.5, 1.2 Hz, 1H), 7.28 – 7.19 (m, 2H), 4.75 – 4.70(m, 1H), 3.77 (d, *J* = 12.7 Hz, 1H), 3.48 (d, *J* = 12.4 Hz, 1H), 3.18 (s, 1H), 2.58 (s, 3H), 1.94 (d, *J* = 8.1 Hz, 2H), 1.22 (s, 6H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 180.4, 139.7, 137.6, 132.7, 132.1, 128.5, 125.7, 84.9, 62.6, 44.6, 37.5, 26.1, 25.5, 20.4.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{19}NO_4S$, 298.1113; found:298.1113.



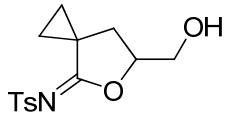

**N-(3-(Hydroxymethyl)-2-oxaspiro[4.5]decan-1-ylidene)-4-methylbenzenesulfonamide**(**9d**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.78 – 7.76 (m, 2H), 7.22–7.20(m, 2H),, 4.72 – 4.68 (m, 1H), 3.81 (dd, *J* = 12.8, 2.5 Hz, 1H), 3.55 (brs, 1H), 3.52 (dd, *J* = 12.8, 4.3 Hz, 1H), 2.33 (s, 3H), 2.15 (dd, *J* = 12.8, 6.9 Hz, 1H), 1.79 (dd, *J* = 12.7, 9.6 Hz, 1H), 1.72 – 1.59 (m, 3H), 1.54 – 1.41 (m, 4H), 1.29 – 1.05 (m, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 180.4, 143.4, 138.4, 129.2, 127.2, 85.6, 62.9, 49.1, 34.4, 33.2, 33.2, 24.9, 22.2, 22.1, 21.5.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{23}NO_4S$, 338.1426; found:338.1426.



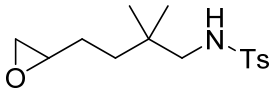

**N-(3-(Hydroxymethyl)-2-oxaspiro[4.4]nonan-1-ylidene)-4-methylbenzenesulfonamide**(**9e**). Colorless oil.$^{1}$H NMR (400 MHz, $CDCl_3$) δ =7.80 – 7.77 (m, 2H), 7.23 – 7.21 (m, 2H),, 4.70 – 4.65 (m, 1H), 3.83 (d, *J* = 12.7 Hz, 1H), 3.53 (d, *J* = 9.9 Hz, 1H), 3.05 (brs, 1H), 2.34 (s, 3H), 2.14 – 2.04 (m, 1H), 2.02 – 1.89 (m, 2H), 1.84 – 1.63 (m, 4H), 1.63 – 1.52 (m, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 180.8, 143.3, 138.5, 129.2 127.2, 85.6, 62.8, 54.5, 39.3, 37.8, 37.5, 25.4, 25.2, 21.5.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{16}H_{21}NO_4S$, 324.1270; found:324.1268.




**N-(7-(Hydroxymethyl)-6-oxaspiro[3.4]octan-5-ylidene)-4-methylbenzenesulfonamide**(**9f**). White solid, mp=101-102°C.$^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.81 – 7.79 (m, 2H), 7.24 – 7.22 (m, 2H), 4.66 – 4.64 (m, 1H), 3.78 (dd, *J* = 12.8, 1.9 Hz, 1H), 3.49 (dd, *J* = 12.8, 3.8 Hz, 1H), 3.15 (brs, 1H), 2.53 – 2.45 (m, 1H), 2.35 (s, 3H), 2.34 – 2.32 (m, 1H), 2.27 (dd, *J* = 13.0, 6.8 Hz, 1H), 2.13 (dd, *J* = 13.0, 8.2 Hz, 1H), 2.04 – 1.87 (m, 4H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 179.2, 143.4, 138.4, 129.2, 127.3, 85.3, 62.8, 48.6, 36.6, 33.4, 30.8, 21.6, 15.9.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{19}NO_4S$, 310.1113; found:310.1108.

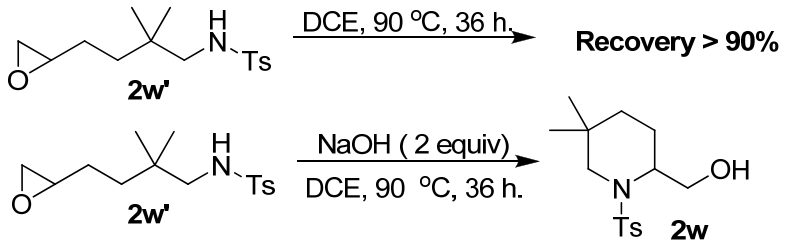



**N-(6-(Hydroxymethyl)-5-oxaspiro[2.4]heptan-4-ylidene)-4-methylbenzenesulfonamide**(**9d**).White solid, mp=154-155°C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.77 – 7.75 (m, 2H), 7.23 – 7.21 (m, 2H), 4.91 – 4.89 (m, 1H) 3.81 (d, *J* = 12.6 Hz, 1H), 3.57 (dd, *J* = 12.6, 4.1 Hz, 1H), 2.95 (brs, 1H), 2.35 (s, 3H), 2.16 (t, *J* = 7.7 Hz, 2H), 1.36 – 1.24 (m, 2H), 1.07 – 1.00 (m, 2H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 179.1, 143.4, 138.4, 129.2, 127.3, 85.8, 63.3, 30.7, 24.8, 21.6, 19.1, 17.8.HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{17}NO_4S$, 296.0957; found:296.0957.




**N-(2,2-dimethyl-4-(oxiran-2-yl)butyl)-4-methylbenzenesulfonamide**(**2w'**).Oil,$^{1}$H NMR (400 MHz, $CDCl_3$) δ =7.69 – 7.67 (m, 2H), 7.24 – 7.22 (m, 2H), 5.08 (t, *J* = 6.7 Hz, 1H), 2.76 (s, 1H), 2.64 (t, *J* = 4.5 Hz, 1H), 2.61 – 2.52 (m, 2H), 2.39 – 2.32 (m, 4H), 1.45 – 1.22 (m, 4H), 0.77 (s, 6H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ =143.3, 137.0, 129.7, 127.0, 52.7, 52.6, 47.2, 34.9, 33.5, 26.9, 24.9, 24.7, 21.5. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{23}NO_3S$, 298.1477; found:298.1477.

## 4. Refluxing of epoxide in DCE

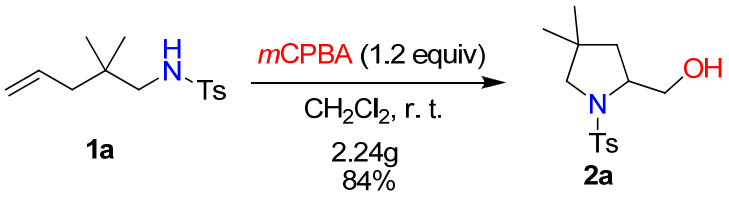



N-(2,2-dimethyl-4-(oxiran-2-yl)butyl)-4-methylbenzenesulfonamide (1 mmol) was dissolved in 20 mL of dry DCE in a 50 mL flame-dried round-bottom flask. The mixture was heated for 36 h at 90 °C in an oil bath. Then DCE was removed and almost quantitative epoxide could be recovered.

To 1 mmol of N-(2,2-dimethyl-4-(oxiran-2-yl)butyl)-4-methylbenzenesulfonamide in 20 mL of DCE in a 50 mL flame-dried round-bottom flask was added 2 equiv of NaOH. The resulting mixture was heated for 36 h at 90 °C in an oil bath. The mixture was then filtered, diluted with DCM (20 mL) and was washed with H2O. The organic layer was dried over MgSO4, filtered and concentrated to give crude residue which was purified by flash column chromatography to give **2w**. **(5,5-dimethyl-1-tosylpiperidin-2-yl)methanol** (**2w**), $^{1}$H NMR (400 MHz, $CDCl_3$) δ = 7.66 – 7.62 (m, 2H), 7.22 – 7.17(m, 2H), 3.90 (d, *J* = 6.5 Hz, 1H), 3.59 (dd, *J* = 11.2, 7.3 Hz, 1H), 3.49 (dd, *J* = 11.2, 6.8 Hz, 1H), 3.17 (d, *J* = 13.2 Hz, 1H), 2.72 (d, *J* = 13.2 Hz, 1H), 2.55 (brs, 1H), 2.32 (s, 3H), 1.69 – 1.47 (m, 2H), 1.27 (td, *J* = 13.3, 4.7 Hz, 1H), 1.20 – 1.09 (m, 1H), 0.79 (s, 3H), 0.67 (s, 3H). $^{13}$C NMR (101 MHz, $CDCl_3$)

δ = 143.0, 138.0, 129.6, 127.0, 59.8, 54.0, 51.9, 32.4, 30.2, 28.5, 23.6, 21.8, 21.5. HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{23}NO_3S$, 298.1477; found: 298.1477.

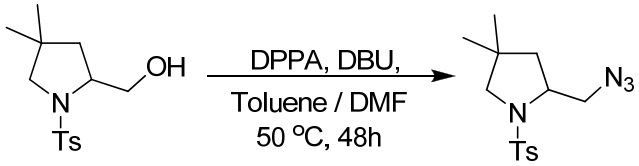
**5. Scale-up of the reaction**




**1a** **2a**

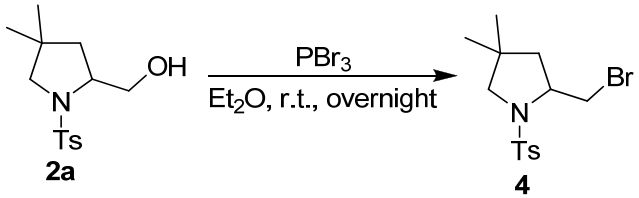
In a 100 mL round-bottomed flask were added 2.5 g **1a**, 1.9 g *m*CPBA and dry $CH_2Cl_2$ (60 mL). The resulting mixture was stirred at room temperature for 24 hours. Then $CH_2Cl_2$ (20 mL) was added and the mixture was washed with aqueous $Na_2S_2O_3$ and aqueous $NaHCO_3$, dried with $MgSO_4$, and concentrated to give crude residue, which was purified by flash column chromatography to give **2a** (2.24 g, 84%).

**6. Derivatization of 2a**




**2a** **3**

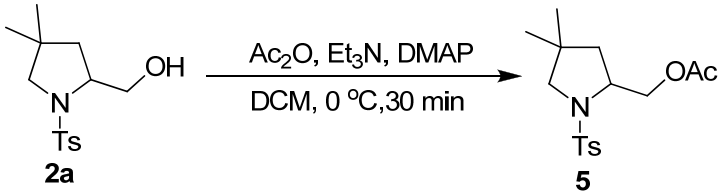
To 1mmol **2a** in 2 mL dry toluene/DMF(9:1) was added diphenylphosphorylazide (DPPA, 2.5mmol) and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU, 2.5mmol) in a 5 mL flame-dried round-bottom flask. The reaction mixture was stirred for 48 h at 50 °C in an oil bath under argon. The reaction mixture was then diluted with $H_2O$ and extracted with $CH_2Cl_2$. The organic layers were collected dried over anhydrous$MgSO_4$, and concentrated to give crude residue. The crude product was purified by silica gel flash chromatography to afford **3**.White solid. mp =71°C;$^1$H NMR (400 MHz, $CDCl_3$) δ = 7.68 – 7.62 (m, 2H), 7.28 – 7.22 (m, 2H), 3.71 – 3.60 (m, 2H), 3.54 (dt, *J* = 9.3, 5.6 Hz, 1H), 3.08 (q, *J* = 10.8 Hz, 2H), 2.36 (s, 3H), 1.63 (dd, *J* = 7.5, 2.7 Hz, 2H), 0.98 (s, 3H), 0.41 (s, 3H);$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 143.8, 134.6, 129.8, 127.5, 61.6, 59.0, 55.0, 43.8, 37.5, 26.1, 25.6, 21.6; HRMS–ESI (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{20}N_4O_2S$, 309.1385 found: 309.1382.



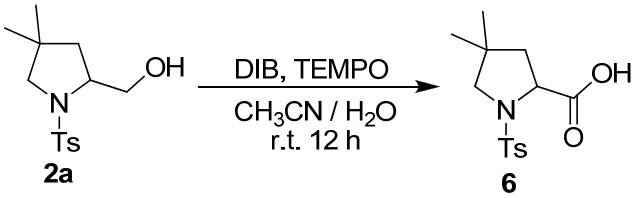

**2a** **4**

In a 50 mL flask, **2a** (1mmol) was added to the solution of anhydrous $Et_2O$ (10mL). After the mixture was cooled to 0°C, $PBr_3$ (1mmol) was added dropwise in 10 minutes. The mixture was stirred at room temperature overnight under argon. The mixture was then poured into ice water (50 mL). The organic layer was extracted with $CH_2Cl_2$. The organic layers were collected dried over anhydrous $MgSO_4$, and

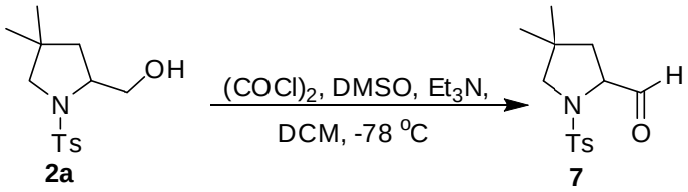
concentrated to give crude residue. The crude product was purified by silica gel flash chromatography to afford **4**.White solid.mp =94-96°C; $^1$H NMR (400 MHz, $CDCl_3$) δ = 7.66 – 7.64 (m, 2H), 7.28 – 7.24 (m, 2H), 3.86 (dd, *J* = 9.6, 3.0 Hz, 1H), 3.80 (ddd, *J* = 15.9, 8.3, 3.0 Hz, 1H), 3.45 (t, *J* = 9.1 Hz, 1H), 3.10 (q, *J* = 10.9 Hz, 2H), 2.36 (s, 3H), 1.81 (dd, *J* = 12.9, 7.2 Hz, 1H), 1.63 (dd, *J* = 12.9, 8.3 Hz, 1H), 0.98 (s, 3H), 0.46 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ = 143.7, 134.9, 129.7, 127.5, 61.9, 60.0, 45.9, 37.5, 37.5, 26.1, 25.8, 21.6. The NMR data are in accordance with those reported in the previous literature.[8]




In a 50 mL flask, TFAA (1.3mmol) was added dropwise to a solution of **2a** (1mmol) and $Et_3N$ (2mmol) in $CH_2Cl_2$ (10 mL) at 0°C. After stirring for 30 min at 0°C, the reaction mixture was quenched with cold water (10 mL) and extracted with $CH_2Cl_2$. The organic layers were collected dried over anhydrous $MgSO_4$, and concentrated to give crude residue. The crude product was purified by silica gel flash chromatography to afford **5**. White solid; mp = 79-80 °C; $^1$H NMR (400 MHz, $CDCl_3$) δ = 7.66 – 7.64 (m, 2H), 7.24 – 7.22 (m, 2H), 4.38 (dd, *J* = 11.0, 3.8 Hz, 1H), 4.12 (dd, *J* = 11.0, 7.1 Hz, 1H), 3.77 (qd, *J* = 7.7, 3.8 Hz, 1H), 3.06 (s, 2H), 2.34 (s, 3H), 1.94 (s, 1H), 1.65 (dd, *J* = 12.8, 7.7 Hz, 3H), 1.53 (dd, *J* = 12.8, 8.1 Hz, 1H), 0.98 (s, 3H), 0.51 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 170.6, 143.5, 134.9, 129.6, 127.4, 66.7, 61.5, 57.8, 43.6, 37.5, 26.3, 25.9, 21.5, 20.8. HRMS–ESI (*m/z*): $[M+H]^+$calcd for $C_{16}H_{23}NO_4S$, 326.1426 found: 326.1422.




To a solution of **2a** (1mmol) and $PhI(OAc)_2$ (2.2 mmol) in a 1:1 mixture (2 mL) of MeCN and$H_2O$was added TEMPO (0.1 mmol), and the solution was stirred at room temperature for 12 h. Saturated $NaHCO_3$ aqueous solution (10 mL) was added to the reaction mixture. The product was basified to pH 10 with 1N NaOH, and washed with AcOEt. The aqueous layer was acidified to pH3 with 1N HCl, extracted with $CH_2Cl_2$. The organic layers were collected dried over anhydrous $MgSO_4$, and concentrated to give crude residue. The crude product was purified by silica gel flash chromatography to afford **6**. Colorlessoil.$^1$H NMR (400 MHz, $CDCl_3$) δ = 9.96 (s, 1H), 7.718 – 7.69 (m, 2H), 7.27 – 7.25 (m, 2H), 4.22 (t, *J* = 8.1 Hz, 1H), 3.13 (d, *J* = 9.9 Hz, 1H), 3.03 (d, *J* = 9.9 Hz, 1H), 2.35 (s, 3H), 1.93 (dd, *J* = 12.6, 8.3 Hz, 1H), 1.84 (dd, *J* = 12.6, 8.3 Hz, 1H), 1.01 (s, 3H), 0.64 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 177.7, 143.9, 134.6, 129.8, 127.7, 60.7, 60.4, 44.3, 38.9, 25.7, 25.5, 21.6.The NMR data are in accordance with those reported in the previous literature.[6]




**2a** **7**

To a solution of oxalyl chloride (1.2mmol) in $CH_2Cl_2$(10 mL) cooled at -78 °C was added dropwise a solution of DMSO (1.1mmol) in $CH_2Cl_2$ (10 mL). After5 min, a solution of **2a** (1mmol) in $CH_2Cl_2$ (5 mL) was added. The reaction mixture was then stirred for 30 min at -78 °C and triethylamine (5mmol) was added in one portion. After 10 min at -78 °C, the mixture was allowed to warm to room temperature and diluted with $CH_2Cl_2$(10 mL). The organic layer was successively washed with saturated aqueous solution of $NH_4Cl$ (20 mL) extracted with $CH_2Cl_2$. The organic layers were collected dried over anhydrous $MgSO_4$, and concentrated to give crude residue. The crude product was purified by silica gel flash chromatography to afford **7**.Colorless oil.$^1$H NMR (400 MHz, $CDCl_3$) δ = 9.64 (d, *J* = 2.9 Hz, 1H), 7.65 – 7.63 (m, 2H), 7.30 – 7.27 (m, 2H), 3.76 (td, *J* = 8.1, 2.9 Hz, 1H), 3.23 (d, *J* = 9.8 Hz, 1H), 2.89 (d, *J* = 9.8 Hz, 1H), 2.37 (s, 3H), 1.74 (dd, *J* = 12.8, 7.6 Hz, 1H), 1.63 (dd, *J* = 12.8, 8.6 Hz, 1H), 0.98 (s, 3H), 0.57 (s, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ = 200.4, 144.2, 133.3, 129.9, 127.7, 66.4, 61.1, 41.6, 38.5, 26.2, 25.4, 21.6.The NMR data are in accordance with those reported in the previous literature.[4]

## 7. ORTEP drawing of 2s and 9a

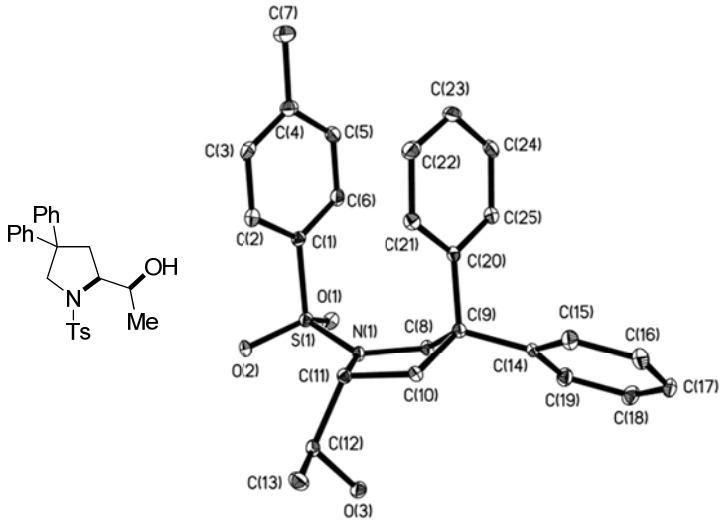



Table 1. Crystal data and structure refinement for **2s**

| Properties | Data |
|---|---|
| Empirical | $C_{25}H_{27}NO_3S$ |
| Formula weight | 421.54 |
| Temperature | 113(2) K |
| Wavelength | 0.71073 A |
| Cell length a | 4.198(3) A |
| Cell length b | 6.0639(12) |

| | |
|---|---|
| Cell length c | 25.178(5) |
| Cell angle alpha | 90 deg |
| Cell angle beta | 100.14(3) |
| Cell angle gamma | 90 deg |
| Cell volume | 2133.9(7) A^3 |
| Cell formula units Z | 4, 1.312 Mg/m^3 |
| Crystal Size | 0.20 x 0.18 x 0.12 mm |

Table 2. Atomic coordination and equivalent isotropic displacement parameter

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 856(1) | 5392(1) | 1087(1) | 16(1) |
| O(1) | 876(1) | 7765(2) | 1095(1) | 21(1) |
| O(2) | 26(1) | 4244(2) | 807(1) | 21(1) |
| O(3) | 1676(1) | 3570(2) | -285(1) | 20(1) |
| N(1) | 1749(1) | 4595(2) | 816(1) | 15(1) |
| C(1) | 1019(1) | 4508(2) | 1765(1) | 16(1) |
| C(2) | 681(1) | 2441(2) | 1890(1) | 20(1) |
| C(3) | 832(1) | 1762(3) | 2428(1) | 22(1) |
| C(4) | 1316(1) | 3103(3) | 2838(1) | 24(1) |
| C(5) | 1638(1) | 5163(3) | 2698(1) | 24(1) |
| C(6) | 1494(1) | 5885(3) | 2167(1) | 21(1) |
| C(7) | 1489(2) | 2355(4) | 3418(1) | 38(1) |
| C(8) | 2678(1) | 5734(2) | 898(1) | 17(1) |
| C(9) | 3418(1) | 3884(2) | 1058(1) | 15(1) |
| C(10) | 2938(1) | 2008(2) | 685(1) | 16(1) |
| C(11) | 1845(1) | 2282(2) | 632(1) | 15(1) |
| C(12) | 1316(1) | 1974(2) | 50(1) | 17(1) |
| C(13) | 1452(1) | -339(2) | -161(1) | 24(1) |
| C(14) | 4414(1) | 4367(2) | 928(1) | 17(1) |
| C(15) | 5111(1) | 2706(3) | 1024(1) | 24(1) |
| C(16) | 6020(1) | 3028(3) | 904(1) | 30(1) |
| C(17) | 6260(1) | 5015(3) | 686(1) | 29(1) |
| C(18) | 5585(1) | 6677(3) | 595(1) | 27(1) |
| C(19) | 4668(1) | 6355(3) | 715(1) | 22(1) |
| C(20) | 3519(1) | 3397(2) | 1666(1) | 16(1) |
| C(21) | 3203(1) | 1472(2) | 1880(1) | 20(1) |
| C(22) | 3354(1) | 1115(3) | 2437(1) | 25(1) |
| C(23) | 3816(1) | 2685(3) | 2787(1) | 26(1) |
| C(24) | 4127(1) | 4633(3) | 2582(1) | 26(1) |
| C(25) | 3979(1) | 4975(3) | 2027(1) | 21(1) |

Table 3.Bond lengths

| | | | |
|---|---|---|---|
| S(1)-O(1) | 1.4394(11) | C(16)-C(17) | 1.391(3) |
| S(1)-O(2) | 1.4426(12) | C(16)-H(16) | 0.9500 |
| S(1)-N(1) | 1.6149(13) | C(17)-C(18) | 1.382(3) |
| S(1)-C(1) | 1.7653(15) | C(17)-H(17) | 0.9500 |
| O(3)-C(12) | 1.4353(17) | C(18)-C(19) | 1.401(2) |
| O(3)-H(3) | 0.8400 | C(18)-H(18) | 0.9500 |
| N(1)-C(8) | 1.4722(18) | C(19)-H(19) | 0.9500 |
| N(1)-C(11) | 1.4906(17) | C(20)-C(21) | 1.392(2) |
| C(1)-C(6) | 1.391(2) | C(20)-C(25) | 1.400(2) |
| C(1)-C(2) | 1.397(2) | C(21)-C(22) | 1.397(2) |
| C(2)-C(3) | 1.396(2) | C(21)-H(21) | 0.9500 |
| C(2)-H(2) | 0.9500 | C(22)-C(23) | 1.383(2) |
| C(3)-C(4) | 1.396(2) | C(22)-H(22) | 0.9500 |
| C(3)-H(3A) | 0.9500 | C(23)-C(24) | 1.392(2) |
| C(4)-C(5) | 1.396(2) | C(23)-H(23) | 0.9500 |
| C(4)-C(7) | 1.508(2) | C(24)-C(25) | 1.390(2) |
| C(5)-C(6) | 1.388(2) | C(24)-H(24) | 0.9500 |
| C(5)-H(5) | 0.9500 | C(25)-H(25) | 0.9500 |
| C(6)-H(6) | 0.9500 | | |
| C(7)-H(7A) | 0.9800 | O(1)-S(1)-O(2) | 120.08(7) |
| C(7)-H(7B) | 0.9800 | O(1)-S(1)-N(1) | 106.87(6) |
| C(7)-H(7C) | 0.9800 | O(2)-S(1)-N(1) | 106.46(7) |
| C(8)-C(9) | 1.541(2) | O(1)-S(1)-C(1) | 106.96(7) |
| C(8)-H(8A) | 0.9900 | O(2)-S(1)-C(1) | 106.45(7) |
| C(8)-H(8B) | 0.9900 | N(1)-S(1)-C(1) | 109.81(7) |
| C(9)-C(14) | 1.534(2) | C(12)-O(3)-H(3) | 109.5 |
| C(9)-C(20) | 1.541(2) | C(8)-N(1)-C(11) | 110.85(11) |
| C(9)-C(10) | 1.554(2) | C(8)-N(1)-S(1) | 123.43(10) |
| C(10)-C(11) | 1.543(2) | C(11)-N(1)-S(1) | 122.32(10) |
| C(10)-H(10A) | 0.9900 | C(6)-C(1)-C(2) | 121.08(14) |
| C(10)-H(10B) | 0.9900 | C(6)-C(1)-S(1) | 119.02(11) |
| C(11)-C(12) | 1.538(2) | C(2)-C(1)-S(1) | 119.90(11) |
| C(11)-H(11) | 1.0000 | C(3)-C(2)-C(1) | 118.87(14) |
| C(12)-C(13) | 1.523(2) | C(3)-C(2)-H(2) | 120.6 |
| C(12)-H(12) | 1.0000 | C(1)-C(2)-H(2) | 120.6 |
| C(13)-H(13A) | 0.9800 | C(2)-C(3)-C(4) | 121.13(15) |
| C(13)-H(13B) | 0.9800 | C(2)-C(3)-H(3A) | 119.4 |
| C(13)-H(13C) | 0.9800 | C(4)-C(3)-H(3A) | 119.4 |
| C(14)-C(19) | 1.392(2) | C(5)-C(4)-C(3) | 118.40(14) |
| C(14)-C(15) | 1.404(2) | C(5)-C(4)-C(7) | 120.57(16) |
| C(15)-C(16) | 1.391(2) | C(3)-C(4)-C(7) | 121.03(16) |
| C(15)-H(15) | 0.9500 | C(6)-C(5)-C(4) | 121.71(15) |

| | |
|---|---|
| C(6)-C(5)-H(5) | 119.1 |
| C(4)-C(5)-H(5) | 119.1 |
| C(5)-C(6)-C(1) | 118.81(15) |
| C(5)-C(6)-H(6) | 120.6 |
| C(1)-C(6)-H(6) | 120.6 |
| C(4)-C(7)-H(7A) | 109.5 |
| C(4)-C(7)-H(7B) | 109.5 |
| H(7A)-C(7)-H(7B) | 109.5 |
| C(4)-C(7)-H(7C) | 109.5 |
| H(7A)-C(7)-H(7C) | 109.5 |
| H(7B)-C(7)-H(7C) | 109.5 |
| N(1)-C(8)-C(9) | 104.37(11) |
| N(1)-C(8)-H(8A) | 110.9 |
| C(9)-C(8)-H(8A) | 110.9 |
| N(1)-C(8)-H(8B) | 110.9 |
| C(9)-C(8)-H(8B) | 110.9 |
| H(8A)-C(8)-H(8B) | 108.9 |
| C(14)-C(9)-C(20) | 108.69(12) |
| C(14)-C(9)-C(8) | 114.60(12) |
| C(20)-C(9)-C(8) | 109.86(12) |
| C(14)-C(9)-C(10) | 109.46(12) |
| C(20)-C(9)-C(10) | 114.44(12) |
| C(8)-C(9)-C(10) | 99.72(12) |
| C(11)-C(10)-C(9) | 107.50(12) |
| C(11)-C(10)-H(10A) | 110.2 |
| C(9)-C(10)-H(10A) | 110.2 |
| C(11)-C(10)-H(10B) | 110.2 |
| C(9)-C(10)-H(10B) | 110.2 |
| H(10A)-C(10)-H(10B) | 108.5 |
| N(1)-C(11)-C(12) | 110.72(12) |
| N(1)-C(11)-C(10) | 102.75(11) |
| C(12)-C(11)-C(10) | 112.60(12) |
| N(1)-C(11)-H(11) | 110.2 |
| C(12)-C(11)-H(11) | 110.2 |
| C(10)-C(11)-H(11) | 110.2 |
| O(3)-C(12)-C(13) | 109.70(12) |
| O(3)-C(12)-C(11) | 108.29(12) |
| C(13)-C(12)-C(11) | 111.88(12) |
| O(3)-C(12)-H(12) | 109.0 |
| C(13)-C(12)-H(12) | 109.0 |
| C(11)-C(12)-H(12) | 109.0 |
| C(12)-C(13)-H(13A) | 109.5 |
| C(12)-C(13)-H(13B) | 109.5 |
| H(13A)-C(13)-H(13B) | 109.5 |
| C(12)-C(13)-H(13C) | 109.5 |
| H(13A)-C(13)-H(13C) | 109.5 |
| H(13B)-C(13)-H(13C) | 109.5 |
| C(19)-C(14)-C(15) | 117.72(14) |
| C(19)-C(14)-C(9) | 123.95(14) |
| C(15)-C(14)-C(9) | 118.33(13) |
| C(16)-C(15)-C(14) | 121.00(15) |
| C(16)-C(15)-H(15) | 119.5 |
| C(14)-C(15)-H(15) | 119.5 |
| C(15)-C(16)-C(17) | 120.61(16) |
| C(15)-C(16)-H(16) | 119.7 |
| C(17)-C(16)-H(16) | 119.7 |
| C(18)-C(17)-C(16) | 119.05(15) |
| C(18)-C(17)-H(17) | 120.5 |
| C(16)-C(17)-H(17) | 120.5 |
| C(17)-C(18)-C(19) | 120.44(15) |
| C(17)-C(18)-H(18) | 119.8 |
| C(19)-C(18)-H(18) | 119.8 |
| C(14)-C(19)-C(18) | 121.16(15) |
| C(14)-C(19)-H(19) | 119.4 |
| C(18)-C(19)-H(19) | 119.4 |
| C(21)-C(20)-C(25) | 117.85(14) |
| C(21)-C(20)-C(9) | 124.38(13) |
| C(25)-C(20)-C(9) | 117.75(13) |
| C(20)-C(21)-C(22) | 120.94(15) |
| C(20)-C(21)-H(21) | 119.5 |
| C(22)-C(21)-H(21) | 119.5 |
| C(23)-C(22)-C(21) | 120.42(15) |
| C(23)-C(22)-H(22) | 119.8 |
| C(21)-C(22)-H(22) | 119.8 |
| C(22)-C(23)-C(24) | 119.50(15) |
| C(22)-C(23)-H(23) | 120.2 |
| C(24)-C(23)-H(23) | 120.2 |
| C(25)-C(24)-C(23) | 119.86(15) |
| C(25)-C(24)-H(24) | 120.1 |
| C(23)-C(24)-H(24) | 120.1 |
| C(24)-C(25)-C(20) | 121.42(15) |
| C(24)-C(25)-H(25) | 119.3 |
| C(20)-C(25)-H(25) | 119.3 |

Table 4.Anisotropic displacement parameters

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S(1) | 14(1) | 16(1) | 17(1) | 2(1) | 3(1) | 1(1) |
| O(1) | 23(1) | 16(1) | 26(1) | 3(1) | 8(1) | 3(1) |
| O(2) | 12(1) | 27(1) | 22(1) | 0(1) | 1(1) | -1(1) |
| O(3) | 17(1) | 26(1) | 18(1) | 6(1) | 3(1) | 0(1) |
| N(1) | 12(1) | 15(1) | 19(1) | -2(1) | 3(1) | -3(1) |
| C(1) | 13(1) | 18(1) | 17(1) | 2(1) | 4(1) | 2(1) |
| C(2) | 16(1) | 19(1) | 24(1) | 0(1) | 5(1) | -1(1) |
| C(3) | 18(1) | 24(1) | 26(1) | 8(1) | 9(1) | 2(1) |
| C(4) | 16(1) | 38(1) | 20(1) | 6(1) | 6(1) | 7(1) |
| C(5) | 18(1) | 34(1) | 19(1) | -4(1) | 3(1) | -3(1) |
| C(6) | 16(1) | 24(1) | 25(1) | -2(1) | 5(1) | -3(1) |
| C(7) | 31(1) | 59(1) | 23(1) | 11(1) | 4(1) | 1(1) |
| C(8) | 14(1) | 17(1) | 20(1) | 1(1) | 2(1) | -3(1) |
| C(9) | 14(1) | 17(1) | 15(1) | 1(1) | 2(1) | -2(1) |
| C(10) | 14(1) | 18(1) | 15(1) | -1(1) | 1(1) | -2(1) |
| C(11) | 15(1) | 14(1) | 16(1) | -1(1) | 3(1) | -2(1) |
| C(12) | 13(1) | 22(1) | 17(1) | 2(1) | 2(1) | -3(1) |
| C(13) | 26(1) | 24(1) | 19(1) | -3(1) | 0(1) | -5(1) |
| C(14) | 13(1) | 24(1) | 14(1) | -3(1) | 2(1) | -4(1) |
| C(15) | 18(1) | 29(1) | 25(1) | 2(1) | 3(1) | 0(1) |
| C(16) | 16(1) | 42(1) | 30(1) | 0(1) | 2(1) | 3(1) |
| C(17) | 17(1) | 47(1) | 24(1) | -8(1) | 7(1) | -9(1) |
| C(18) | 26(1) | 30(1) | 28(1) | -3(1) | 10(1) | -10(1) |
| C(19) | 20(1) | 23(1) | 25(1) | -2(1) | 6(1) | -4(1) |
| C(20) | 11(1) | 22(1) | 16(1) | 0(1) | 1(1) | 1(1) |
| C(21) | 18(1) | 22(1) | 20(1) | 2(1) | 2(1) | 0(1) |
| C(22) | 20(1) | 32(1) | 23(1) | 8(1) | 6(1) | 5(1) |
| C(23) | 18(1) | 45(1) | 15(1) | 4(1) | 3(1) | 7(1) |
| C(24) | 16(1) | 41(1) | 19(1) | -7(1) | 0(1) | -3(1) |
| C(25) | 16(1) | 27(1) | 20(1) | -1(1) | 2(1) | -4(1) |

Table 5.Hydrogen coordinates and isotropic displacement parameters

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(3) | 1225 | 4372 | -436 | 30 |
| H(2) | 355 | 1513 | 1613 | 23 |
| H(3A) | 601 | 364 | 2517 | 27 |
| H(5) | 1964 | 6095 | 2974 | 28 |
| H(6) | 1715 | 7295 | 2079 | 25 |
| H(7A) | 1002 | 2999 | 3603 | 56 |
| H(7B) | 2125 | 2837 | 3596 | 56 |
| H(7C) | 1451 | 743 | 3431 | 56 |

| | | | | |
|---|---|---|---|---|
| H(8A) | 2718 | 6848 | 1188 | 20 |
| H(8B) | 2781 | 6473 | 562 | 20 |
| H(10A) | 3128 | 2109 | 326 | 19 |
| H(10B) | 3139 | 551 | 843 | 19 |
| H(11) | 1591 | 1230 | 879 | 18 |
| H(12) | 618 | 2243 | 37 | 21 |
| H(13A) | 1046 | -528 | -515 | 35 |
| H(13B) | 1273 | -1430 | 92 | 35 |
| H(13C) | 2124 | -549 | -193 | 35 |
| H(15) | 4960 | 1340 | 1174 | 29 |
| H(16) | 6482 | 1882 | 971 | 35 |
| H(17) | 6879 | 5225 | 600 | 34 |
| H(18) | 5744 | 8048 | 451 | 33 |
| H(19) | 4211 | 7512 | 650 | 27 |
| H(21) | 2879 | 385 | 1643 | 24 |
| H(22) | 3139 | -215 | 2575 | 30 |
| H(23) | 3920 | 2437 | 3166 | 31 |
| H(24) | 4440 | 5726 | 2820 | 31 |
| H(25) | 4194 | 6309 | 1891 | 25 |

Table 6.Torsion angles

| | | | |
|---|---|---|---|
| O(1)-S(1)-N(1)-C(8) | 36.02(13) | C(11)-N(1)-C(8)-C(9) | -29.51(15) |
| O(2)-S(1)-N(1)-C(8) | 165.50(11) | S(1)-N(1)-C(8)-C(9) | 130.02(11) |
| C(1)-S(1)-N(1)-C(8) | -79.65(13) | N(1)-C(8)-C(9)-C(14) | 154.27(12) |
| O(1)-S(1)-N(1)-C(11) | -166.73(11) | N(1)-C(8)-C(9)-C(20) | -83.04(14) |
| O(2)-S(1)-N(1)-C(11) | -37.25(13) | N(1)-C(8)-C(9)-C(10) | 37.51(13) |
| C(1)-S(1)-N(1)-C(11) | 77.60(12) | C(14)-C(9)-C(10)-C(11) | -154.44(12) |
| O(1)-S(1)-C(1)-C(6) | -25.10(14) | C(20)-C(9)-C(10)-C(11) | 83.29(15) |
| O(2)-S(1)-C(1)-C(6) | -154.63(12) | C(8)-C(9)-C(10)-C(11) | -33.88(14) |
| N(1)-S(1)-C(1)-C(6) | 90.51(13) | C(8)-N(1)-C(11)-C(12) | -112.91(14) |
| O(1)-S(1)-C(1)-C(2) | 155.57(12) | S(1)-N(1)-C(11)-C(12) | 87.29(14) |
| O(2)-S(1)-C(1)-C(2) | 26.04(14) | C(8)-N(1)-C(11)-C(10) | 7.53(14) |
| N(1)-S(1)-C(1)-C(2) | -88.81(13) | S(1)-N(1)-C(11)-C(10) | -152.26(10) |
| C(6)-C(1)-C(2)-C(3) | -0.3(2) | C(9)-C(10)-C(11)-N(1) | 17.32(14) |
| S(1)-C(1)-C(2)-C(3) | 178.97(12) | C(9)-C(10)-C(11)-C(12) | 136.46(12) |
| C(1)-C(2)-C(3)-C(4) | -0.5(2) | N(1)-C(11)-C(12)-O(3) | 55.59(15) |
| C(2)-C(3)-C(4)-C(5) | 0.9(2) | C(10)-C(11)-C(12)-O(3) | -58.80(15) |
| C(2)-C(3)-C(4)-C(7) | -179.25(16) | N(1)-C(11)-C(12)-C(13) | 176.61(12) |
| C(3)-C(4)-C(5)-C(6) | -0.5(2) | C(10)-C(11)-C(12)-C(13) | 62.23(16) |
| C(7)-C(4)-C(5)-C(6) | 179.60(16) | C(20)-C(9)-C(14)-C(19) | -119.30(15) |
| C(4)-C(5)-C(6)-C(1) | -0.3(2) | C(8)-C(9)-C(14)-C(19) | 4.0(2) |
| C(2)-C(1)-C(6)-C(5) | 0.7(2) | C(10)-C(9)-C(14)-C(19) | 115.05(15) |
| S(1)-C(1)-C(6)-C(5) | -178.63(12) | C(20)-C(9)-C(14)-C(15) | 61.23(17) |

| | | | |
|---|---|---|---|
| C(8)-C(9)-C(14)-C(15) | -175.45(13) | C(10)-C(9)-C(20)-C(21) | -1.0(2) |
| C(10)-C(9)-C(14)-C(15) | -64.42(17) | C(14)-C(9)-C(20)-C(25) | 54.90(17) |
| C(19)-C(14)-C(15)-C(16) | -0.8(2) | C(8)-C(9)-C(20)-C(25) | -71.22(17) |
| C(9)-C(14)-C(15)-C(16) | 178.66(14) | C(10)-C(9)-C(20)-C(25) | 177.59(13) |
| C(14)-C(15)-C(16)-C(17) | 0.1(3) | C(25)-C(20)-C(21)-C(22) | -1.1(2) |
| C(15)-C(16)-C(17)-C(18) | 0.7(3) | C(9)-C(20)-C(21)-C(22) | 177.50(14) |
| C(16)-C(17)-C(18)-C(19) | -0.9(3) | C(20)-C(21)-C(22)-C(23) | 0.6(2) |
| C(15)-C(14)-C(19)-C(18) | 0.7(2) | C(21)-C(22)-C(23)-C(24) | 0.2(2) |
| C(9)-C(14)-C(19)-C(18) | -178.78(14) | C(22)-C(23)-C(24)-C(25) | -0.5(2) |
| C(17)-C(18)-C(19)-C(14) | 0.2(3) | C(23)-C(24)-C(25)-C(20) | 0.0(2) |
| C(14)-C(9)-C(20)-C(21) | -123.70(15) | C(21)-C(20)-C(25)-C(24) | 0.8(2) |
| C(8)-C(9)-C(20)-C(21) | 110.18(16) | C(9)-C(20)-C(25)-C(24) | -177.90(14) |

Table 7. Hydrogen bonds

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| O(3)-H(3)...O(2)#1 | 0.84 | 2.04 | 2.8634(17) | 168.6 |

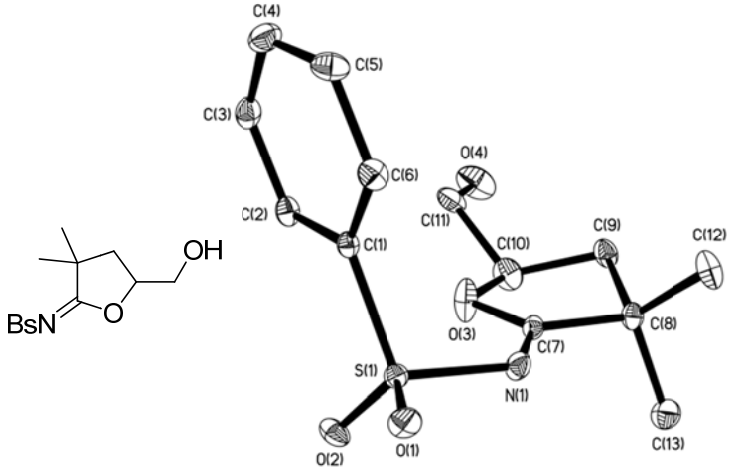



Table 1.Crystal data and structure refinement for **9a**

| Properties | Data |
|---|---|
| Empirical | $C_{13}H_{17}NO_4S$ |
| Formula weight | 283.34 |
| Temperature | 113(2) K |
| Wavelength | 0.71073 A |
| Cell length a | 8.6906(17) A |
| Cell length b | 11.953(2) A |
| Cell length c | 13.546(3) A |
| Cell angle alpha | 90 deg |
| Cell angle beta | 90 deg |
| Cell angle gamma | 90 deg |
| Cell volume | 1407.1(5) A^3 |
| Cell formula units Z | 4, 1.337 Mg/m^3 |

| Crystal Size | 0.20 x 0.18 x 0.12 mm |
|---|---|

Table 2. Atomic coordination and equivalent isotropic displacement parameter

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 10282(1) | 9626(1) | 7866(1) | 17(1) |
| O(1) | 10614(3) | 8790(2) | 8601(2) | 24(1) |
| O(2) | 10860(3) | 10732(2) | 8033(2) | 25(1) |
| O(3) | 8367(3) | 10907(2) | 6553(2) | 34(1) |
| O(4) | 7036(4) | 12412(2) | 4391(2) | 51(1) |
| N(1) | 8388(3) | 9608(2) | 7783(2) | 22(1) |
| C(1) | 10975(3) | 9132(2) | 6727(2) | 16(1) |
| C(2) | 11536(4) | 9886(3) | 6033(2) | 20(1) |
| C(3) | 12140(4) | 9479(3) | 5154(2) | 25(1) |
| C(4) | 12169(4) | 8328(3) | 4966(3) | 28(1) |
| C(5) | 11568(5) | 7597(3) | 5671(3) | 28(1) |
| C(6) | 10981(4) | 7985(3) | 6553(2) | 22(1) |
| C(7) | 7685(3) | 10213(2) | 7143(2) | 17(1) |
| C(8) | 5948(3) | 10198(2) | 7023(2) | 20(1) |
| C(9) | 5788(4) | 10805(3) | 6026(3) | 25(1) |
| C(10) | 7206(5) | 11534(3) | 5973(3) | 33(1) |
| C(11) | 7917(5) | 11739(3) | 4963(3) | 31(1) |
| C(12) | 5360(4) | 8991(3) | 7011(3) | 31(1) |
| C(13) | 5232(4) | 10858(3) | 7877(3) | 31(1) |

Table 3. Bond lengths

| | | | |
|---|---|---|---|
| S(1)-O(2) | 1.432(2) | C(4)-H(4A) | 0.9500 |
| S(1)-O(1) | 1.441(2) | C(5)-C(6) | 1.380(5) |
| S(1)-N(1) | 1.650(3) | C(5)-H(5) | 0.9500 |
| S(1)-C(1) | 1.758(3) | C(6)-H(6) | 0.9500 |
| O(3)-C(7) | 1.297(4) | C(7)-C(8) | 1.518(4) |
| O(3)-C(10) | 1.481(4) | C(8)-C(12) | 1.531(4) |
| O(4)-C(11) | 1.354(5) | C(8)-C(13) | 1.532(5) |
| O(4)-H(4) | 0.8400 | C(8)-C(9) | 1.539(4) |
| N(1)-C(7) | 1.284(4) | C(9)-C(10) | 1.512(5) |
| C(1)-C(2) | 1.391(4) | C(9)-H(9A) | 0.9900 |
| C(1)-C(6) | 1.392(4) | C(9)-H(9B) | 0.9900 |
| C(2)-C(3) | 1.389(5) | C(10)-C(11) | 1.522(5) |
| C(2)-H(2) | 0.9500 | C(10)-H(10) | 1.0000 |
| C(3)-C(4) | 1.400(5) | C(11)-H(11A) | 0.9900 |
| C(3)-H(3) | 0.9500 | C(11)-H(11B) | 0.9900 |
| C(4)-C(5) | 1.395(5) | C(12)-H(12A) | 0.9800 |

| | |
|---|---|
| C(12)-H(12B) | 0.9800 |
| C(12)-H(12C) | 0.9800 |
| C(13)-H(13A) | 0.9800 |
| C(13)-H(13B) | 0.9800 |
| C(13)-H(13C) | 0.9800 |
| | |
| O(2)-S(1)-O(1) | 117.42(14) |
| O(2)-S(1)-N(1) | 111.92(15) |
| O(1)-S(1)-N(1) | 103.73(14) |
| O(2)-S(1)-C(1) | 109.19(15) |
| O(1)-S(1)-C(1) | 107.74(14) |
| N(1)-S(1)-C(1) | 106.16(14) |
| C(7)-O(3)-C(10) | 109.8(3) |
| C(11)-O(4)-H(4) | 109.5 |
| C(7)-N(1)-S(1) | 120.8(2) |
| C(2)-C(1)-C(6) | 121.5(3) |
| C(2)-C(1)-S(1) | 119.7(2) |
| C(6)-C(1)-S(1) | 118.8(2) |
| C(3)-C(2)-C(1) | 119.0(3) |
| C(3)-C(2)-H(2) | 120.5 |
| C(1)-C(2)-H(2) | 120.5 |
| C(2)-C(3)-C(4) | 120.5(3) |
| C(2)-C(3)-H(3) | 119.8 |
| C(4)-C(3)-H(3) | 119.8 |
| C(5)-C(4)-C(3) | 119.0(3) |
| C(5)-C(4)-H(4A) | 120.5 |
| C(3)-C(4)-H(4A) | 120.5 |
| C(6)-C(5)-C(4) | 121.3(3) |
| C(6)-C(5)-H(5) | 119.3 |
| C(4)-C(5)-H(5) | 119.3 |
| C(5)-C(6)-C(1) | 118.7(3) |
| C(5)-C(6)-H(6) | 120.7 |
| C(1)-C(6)-H(6) | 120.7 |
| N(1)-C(7)-O(3) | 124.0(3) |
| N(1)-C(7)-C(8) | 122.6(3) |
| O(3)-C(7)-C(8) | 113.3(3) |
| C(7)-C(8)-C(12) | 110.1(2) |
| C(7)-C(8)-C(13) | 108.5(3) |
| C(12)-C(8)-C(13) | 111.0(3) |
| C(7)-C(8)-C(9) | 100.3(2) |
| C(12)-C(8)-C(9) | 113.8(3) |
| C(13)-C(8)-C(9) | 112.5(3) |
| C(10)-C(9)-C(8) | 103.8(3) |
| C(10)-C(9)-H(9A) | 111.0 |
| C(8)-C(9)-H(9A) | 111.0 |
| C(10)-C(9)-H(9B) | 111.0 |
| C(8)-C(9)-H(9B) | 111.0 |
| H(9A)-C(9)-H(9B) | 109.0 |
| O(3)-C(10)-C(9) | 103.8(3) |
| O(3)-C(10)-C(11) | 106.3(3) |
| C(9)-C(10)-C(11) | 117.8(3) |
| O(3)-C(10)-H(10) | 109.5 |
| C(9)-C(10)-H(10) | 109.5 |
| C(11)-C(10)-H(10) | 109.5 |
| O(4)-C(11)-C(10) | 112.4(4) |
| O(4)-C(11)-H(11A) | 109.1 |
| C(10)-C(11)-H(11A) | 109.1 |
| O(4)-C(11)-H(11B) | 109.1 |
| C(10)-C(11)-H(11B) | 109.1 |
| H(11A)-C(11)-H(11B) | 107.9 |
| C(8)-C(12)-H(12A) | 109.5 |
| C(8)-C(12)-H(12B) | 109.5 |
| H(12A)-C(12)-H(12B) | 109.5 |
| C(8)-C(12)-H(12C) | 109.5 |
| H(12A)-C(12)-H(12C) | 109.5 |
| H(12B)-C(12)-H(12C) | 109.5 |
| C(8)-C(13)-H(13A) | 109.5 |
| C(8)-C(13)-H(13B) | 109.5 |
| H(13A)-C(13)-H(13B) | 109.5 |
| C(8)-C(13)-H(13C) | 109.5 |
| H(13A)-C(13)-H(13C) | 109.5 |
| H(13B)-C(13)-H(13C) | 109.5 |

Table 4. Anisotropic displacement parameters

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S(1) | 15(1) | 22(1) | 16(1) | -1(1) | -2(1) | 3(1) |
| O(1) | 23(1) | 33(1) | 15(1) | 7(1) | -4(1) | 6(1) |
| O(2) | 23(1) | 21(1) | 30(1) | -9(1) | -8(1) | 4(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O(3) | 18(1) | 36(1) | 49(2) | 27(1) | -3(1) | 0(1) |
| O(4) | 70(2) | 39(2) | 44(2) | 7(1) | -18(2) | -4(2) |
| N(1) | 15(1) | 32(1) | 20(1) | 3(1) | 0(1) | 4(1) |
| C(1) | 13(1) | 17(1) | 17(1) | 2(1) | -1(1) | 0(1) |
| C(2) | 18(2) | 18(1) | 23(2) | 5(1) | -2(1) | -2(1) |
| C(3) | 22(2) | 34(2) | 20(2) | 7(1) | 1(1) | -3(1) |
| C(4) | 28(2) | 37(2) | 20(2) | -2(1) | 3(1) | 3(2) |
| C(5) | 38(2) | 19(2) | 27(2) | -6(1) | -1(2) | 0(1) |
| C(6) | 25(2) | 17(1) | 23(2) | 4(1) | -1(1) | -3(1) |
| C(7) | 15(1) | 14(1) | 22(1) | -2(1) | 3(1) | 0(1) |
| C(8) | 13(1) | 21(1) | 24(2) | 3(1) | -1(1) | 2(1) |
| C(9) | 22(2) | 30(2) | 25(2) | 6(1) | -8(1) | -4(1) |
| C(10) | 31(2) | 32(2) | 37(2) | 5(2) | -8(2) | 2(2) |
| C(11) | 41(2) | 30(2) | 21(2) | 1(1) | -7(2) | -15(2) |
| C(12) | 21(2) | 25(2) | 46(2) | 5(2) | -8(2) | -5(1) |
| C(13) | 22(2) | 38(2) | 33(2) | -4(2) | 1(2) | 8(1) |

Table 5. Hydrogen coordinates and isotropic displacement parameters

| | | | | |
|---|---|---|---|---|
| H(4) | 6415 | 12020 | 4065 | 76 |
| H(2) | 11506 | 10668 | 6158 | 24 |
| H(3) | 12536 | 9986 | 4678 | 31 |
| H(4A) | 12591 | 8049 | 4368 | 34 |
| H(5) | 11563 | 6816 | 5540 | 34 |
| H(6) | 10589 | 7479 | 7032 | 26 |
| H(9A) | 5763 | 10263 | 5473 | 30 |
| H(9B) | 4840 | 11263 | 6008 | 30 |
| H(10) | 6994 | 12267 | 6301 | 40 |
| H(11A) | 8943 | 12086 | 5049 | 37 |
| H(11B) | 8063 | 11013 | 4624 | 37 |
| H(12A) | 5795 | 8596 | 6442 | 46 |
| H(12B) | 4234 | 8992 | 6963 | 46 |
| H(12C) | 5672 | 8613 | 7620 | 46 |
| H(13A) | 5470 | 10486 | 8503 | 47 |
| H(13B) | 4113 | 10893 | 7789 | 47 |
| H(13C) | 5654 | 11619 | 7884 | 47 |

Table 6. Torsion angles

| | | | |
|---|---|---|---|
| O(2)-S(1)-N(1)-C(7) | 56.7(3) | O(2)-S(1)-C(1)-C(6) | 159.8(3) |
| O(1)-S(1)-N(1)-C(7) | -175.8(3) | O(1)-S(1)-C(1)-C(6) | 31.2(3) |
| C(1)-S(1)-N(1)-C(7) | -62.3(3) | N(1)-S(1)-C(1)-C(6) | -79.4(3) |
| O(2)-S(1)-C(1)-C(2) | -18.7(3) | C(6)-C(1)-C(2)-C(3) | -1.2(5) |
| O(1)-S(1)-C(1)-C(2) | -147.3(2) | S(1)-C(1)-C(2)-C(3) | 177.3(2) |
| N(1)-S(1)-C(1)-C(2) | 102.1(3) | C(1)-C(2)-C(3)-C(4) | 0.7(5) |

| | | | |
|---|---|---|---|
| C(2)-C(3)-C(4)-C(5) | 0.6(5) | O(3)-C(7)-C(8)-C(13) | -103.0(3) |
| C(3)-C(4)-C(5)-C(6) | -1.4(6) | N(1)-C(7)-C(8)-C(9) | -166.2(3) |
| C(4)-C(5)-C(6)-C(1) | 1.0(5) | O(3)-C(7)-C(8)-C(9) | 15.1(3) |
| C(2)-C(1)-C(6)-C(5) | 0.3(5) | C(7)-C(8)-C(9)-C(10) | -26.5(3) |
| S(1)-C(1)-C(6)-C(5) | -178.1(3) | C(12)-C(8)-C(9)-C(10) | -144.0(3) |
| S(1)-N(1)-C(7)-O(3) | -3.1(5) | C(13)-C(8)-C(9)-C(10) | 88.7(3) |
| S(1)-N(1)-C(7)-C(8) | 178.3(2) | C(7)-O(3)-C(10)-C(9) | -21.1(4) |
| C(10)-O(3)-C(7)-N(1) | -175.3(3) | C(7)-O(3)-C(10)-C(11) | -146.0(3) |
| C(10)-O(3)-C(7)-C(8) | 3.4(4) | C(8)-C(9)-C(10)-O(3) | 29.4(4) |
| N(1)-C(7)-C(8)-C(12) | -45.9(4) | C(8)-C(9)-C(10)-C(11) | 146.6(3) |
| O(3)-C(7)-C(8)-C(12) | 135.3(3) | O(3)-C(10)-C(11)-O(4) | -173.4(3) |
| N(1)-C(7)-C(8)-C(13) | 75.7(4) | C(9)-C(10)-C(11)-O(4) | 70.7(4) |

Table 7.Hydrogen bonds

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| O(4)-H(4)...O(1)#1 | 0.84 | 2.11 | 2.917(4) | 161.8 |
| O(4)-H(4)...N(1)#1 | 0.84 | 2.61 | 3.272(4) | 136.3 |
| O(4)-H(4)...S(1)#1 | 0.84 | 2.95 | 3.776(3) | 169.6 |

## 8. References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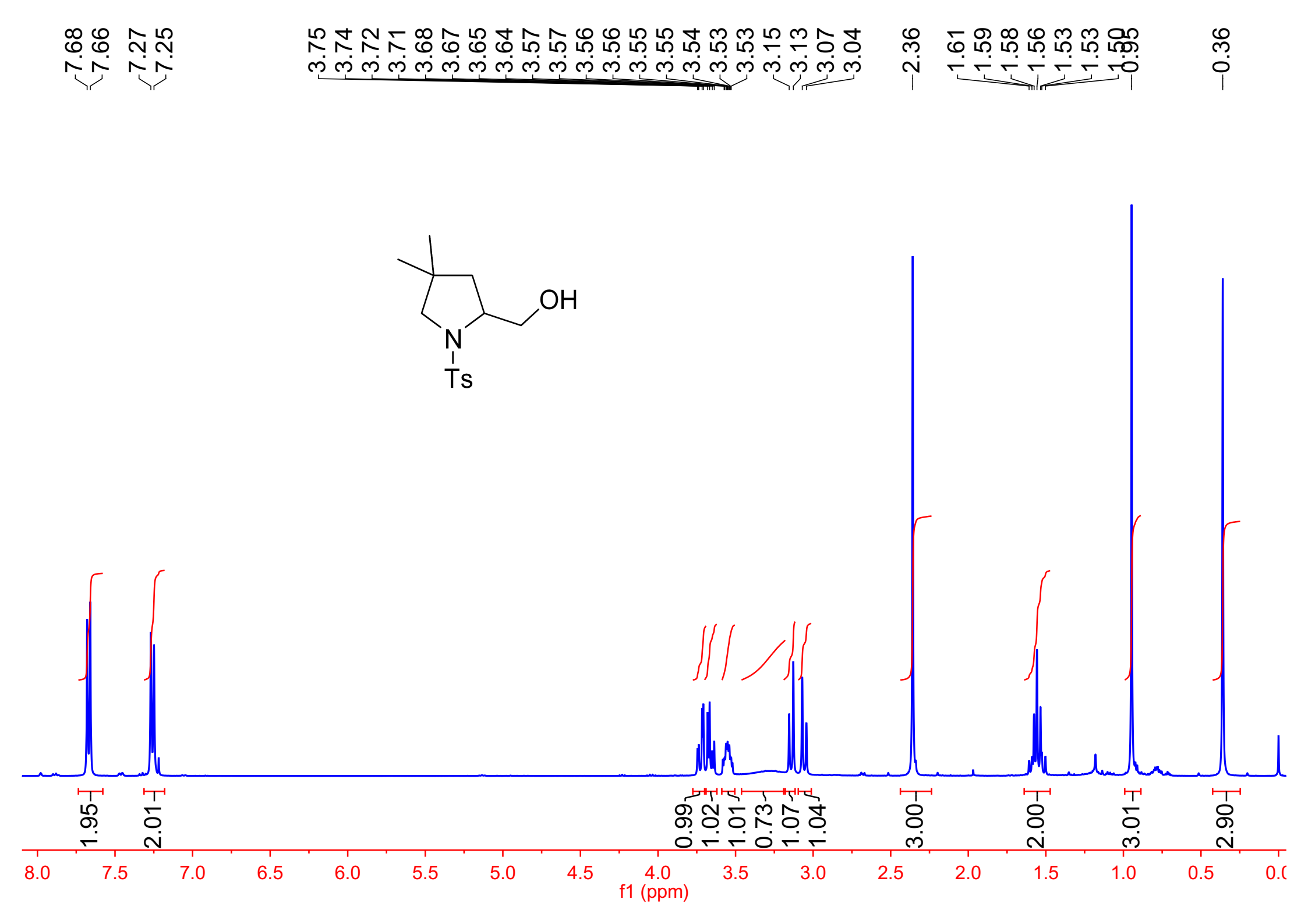
## 9. Copies of NMR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3.13
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3.04
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0.99
1.02
1.01
0.73
1.07
1.04
3.00
2.00
3.01
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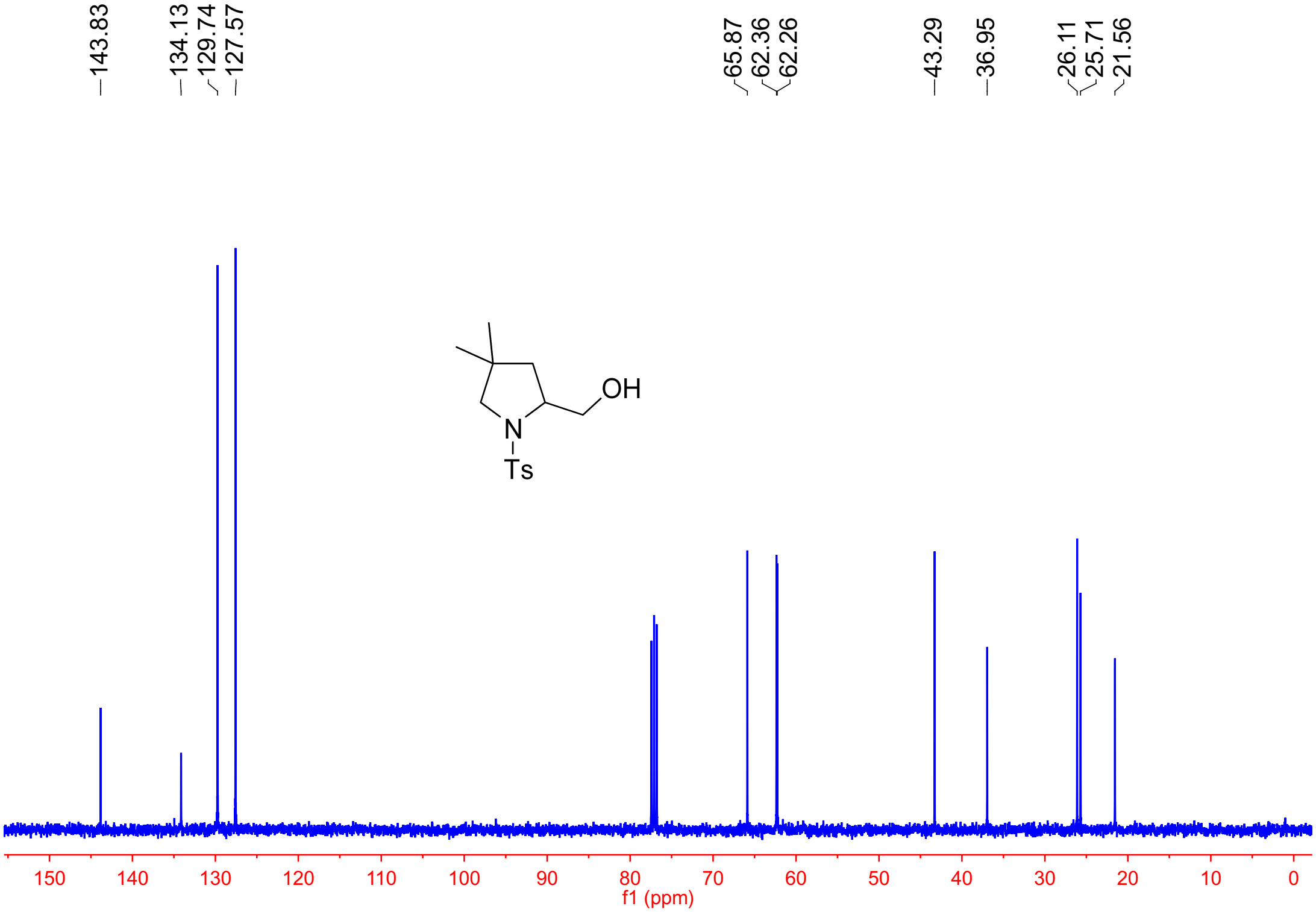
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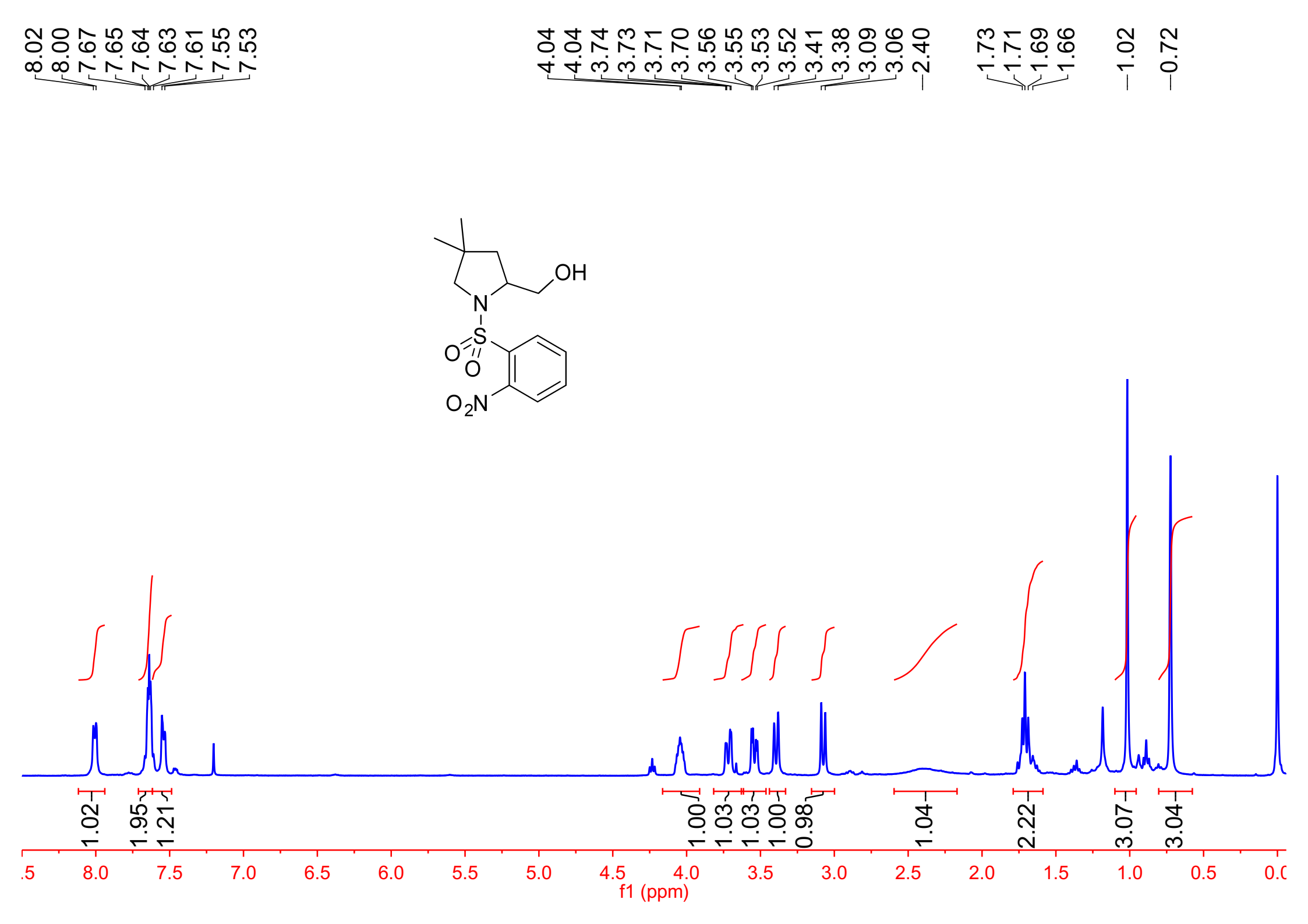
f1 (ppm)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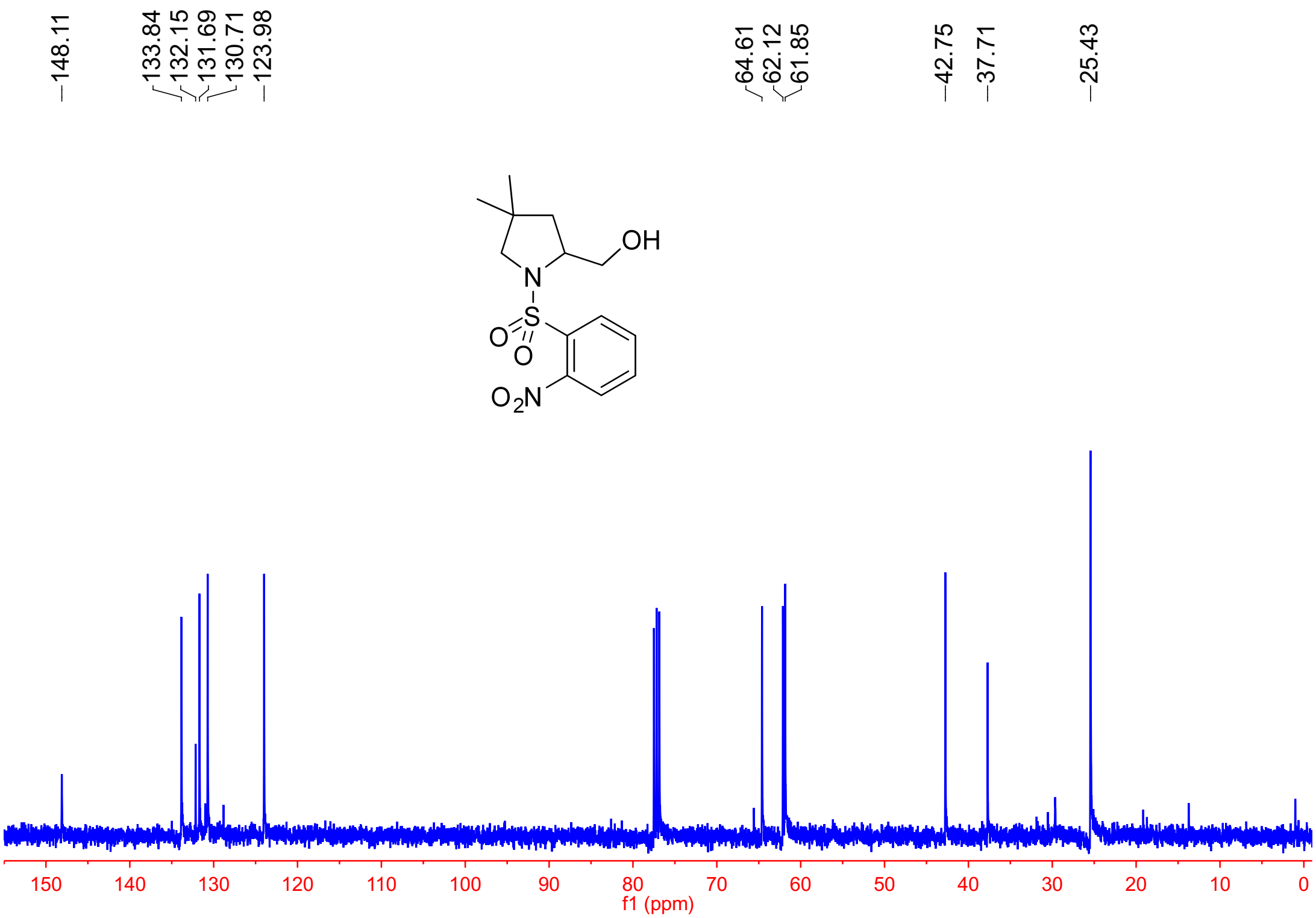
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S
O
O
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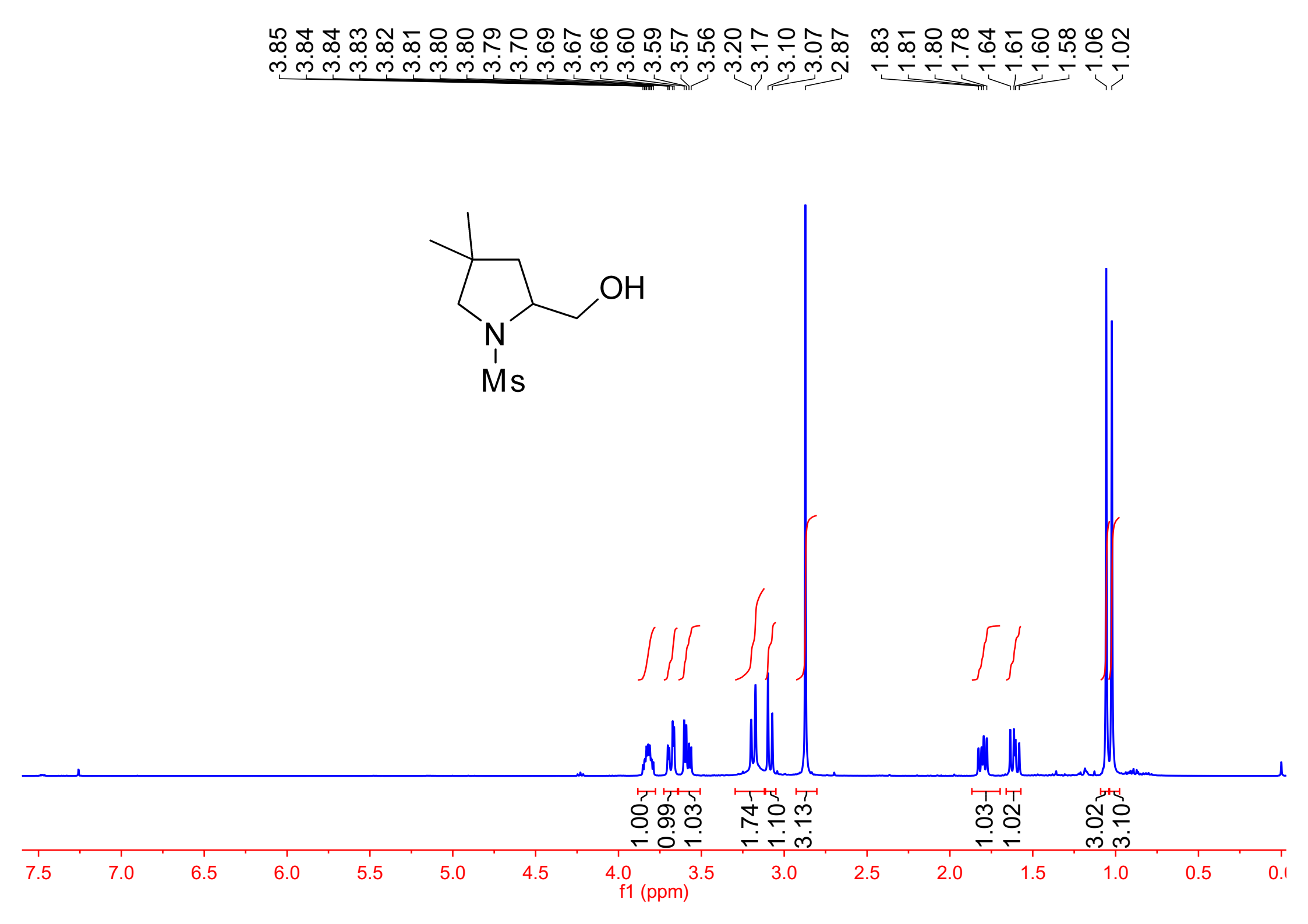
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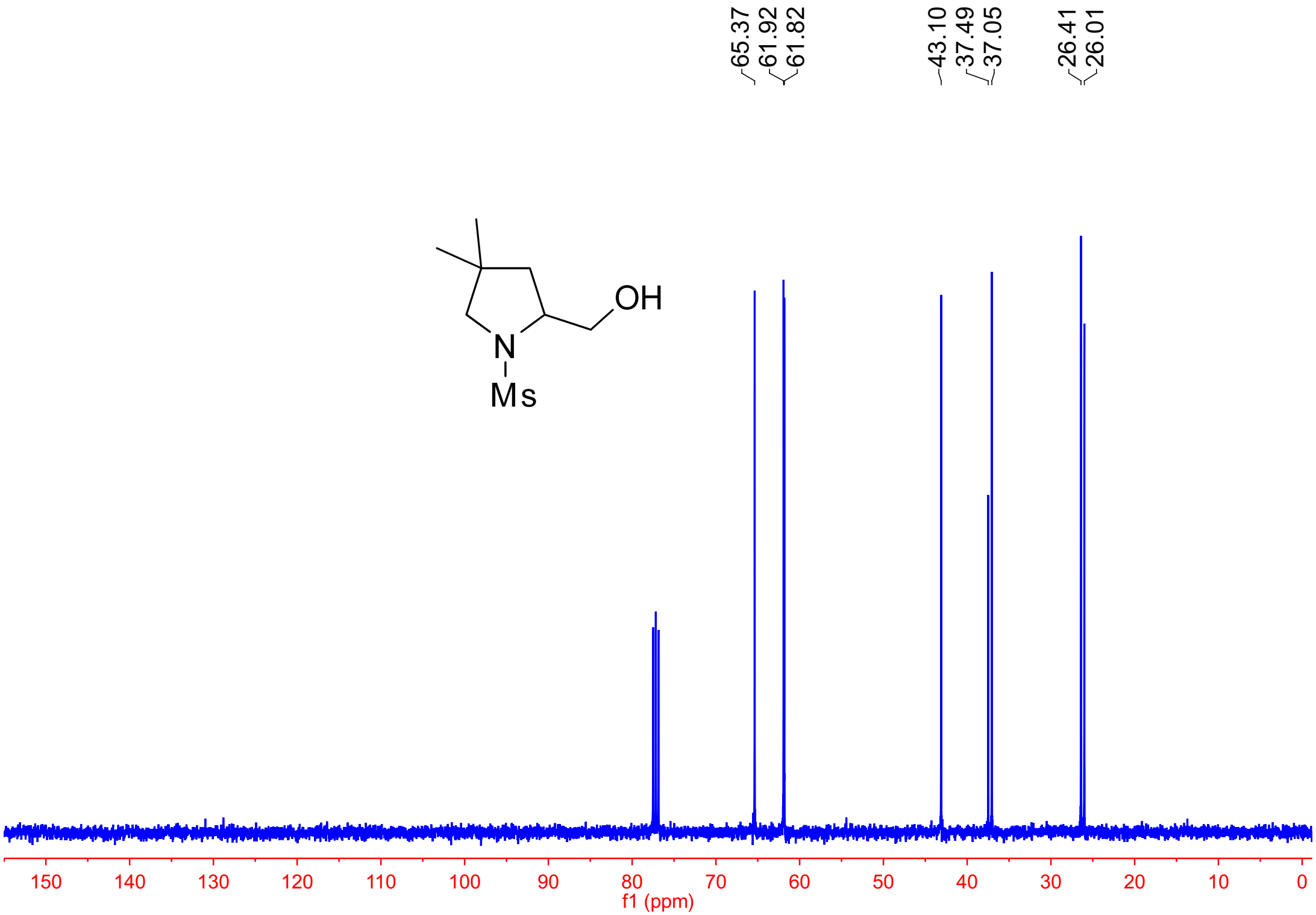
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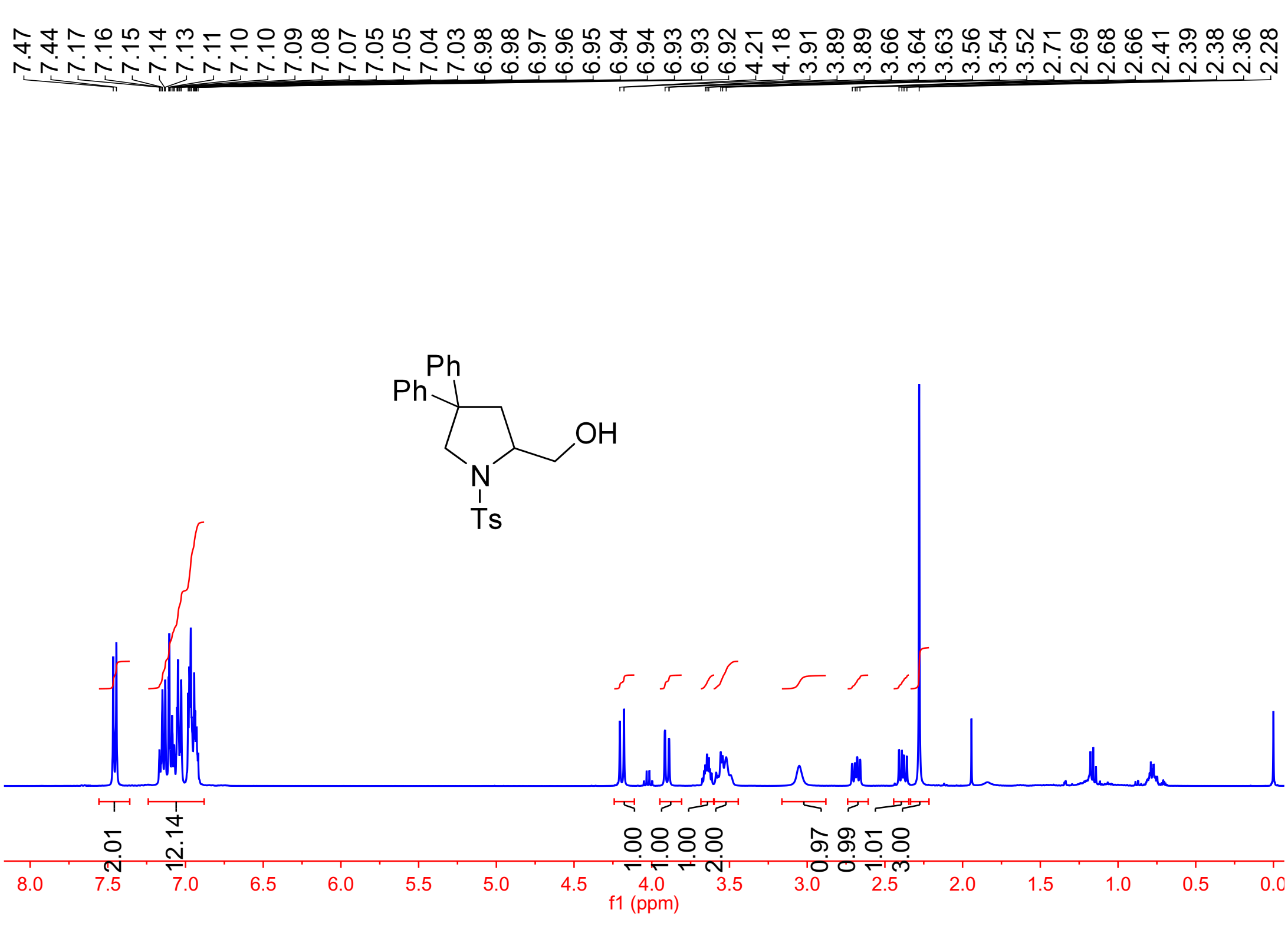
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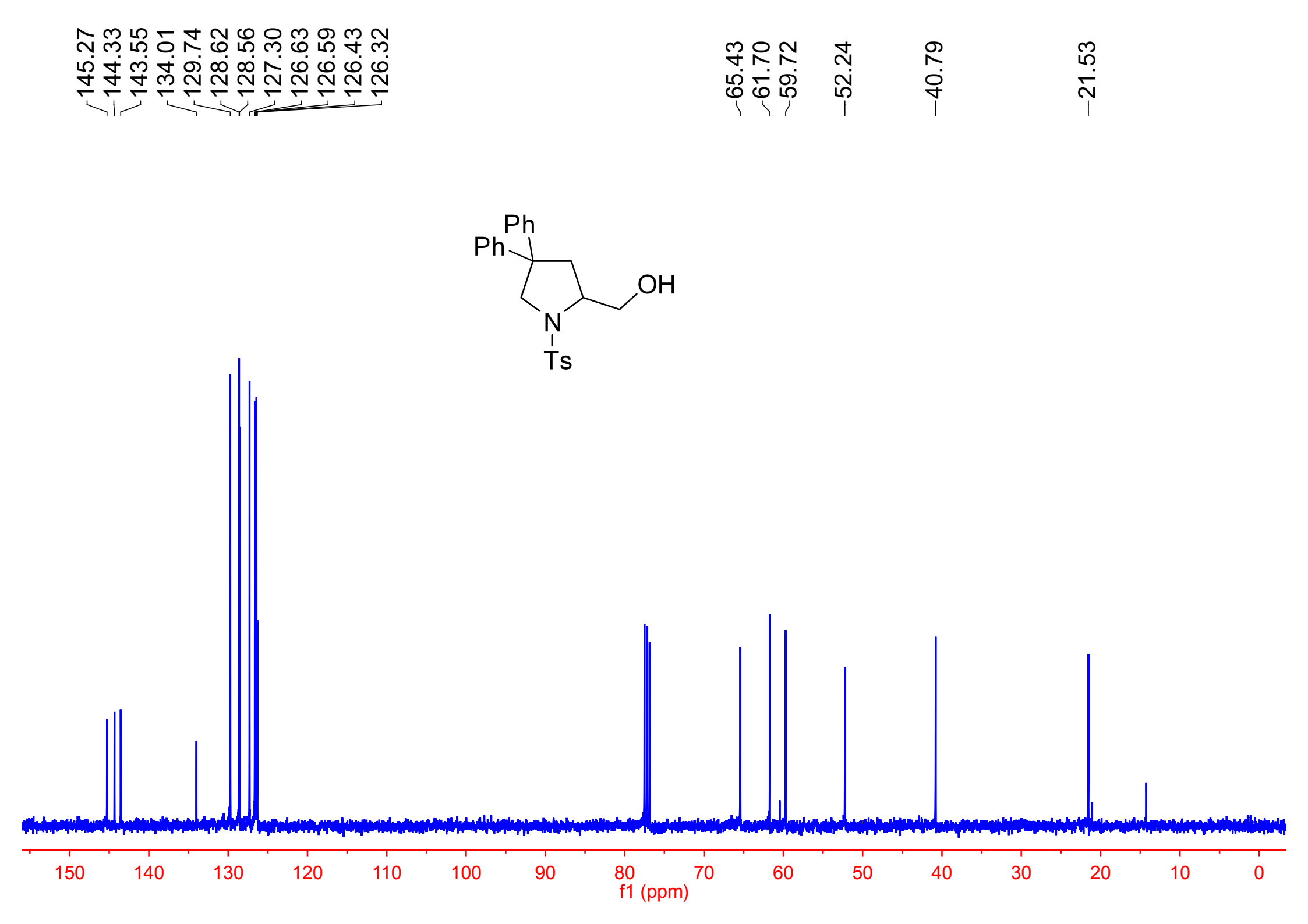
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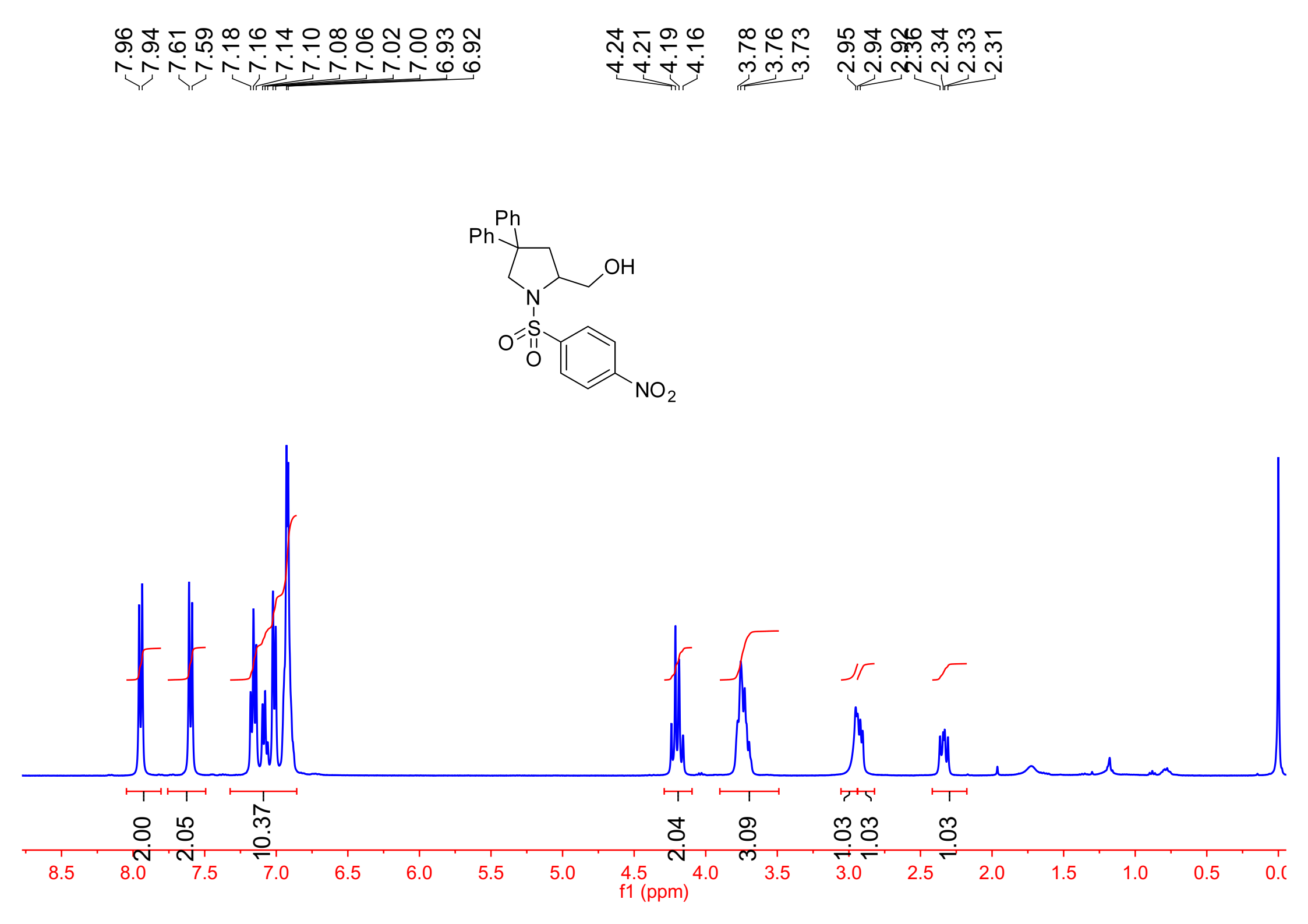
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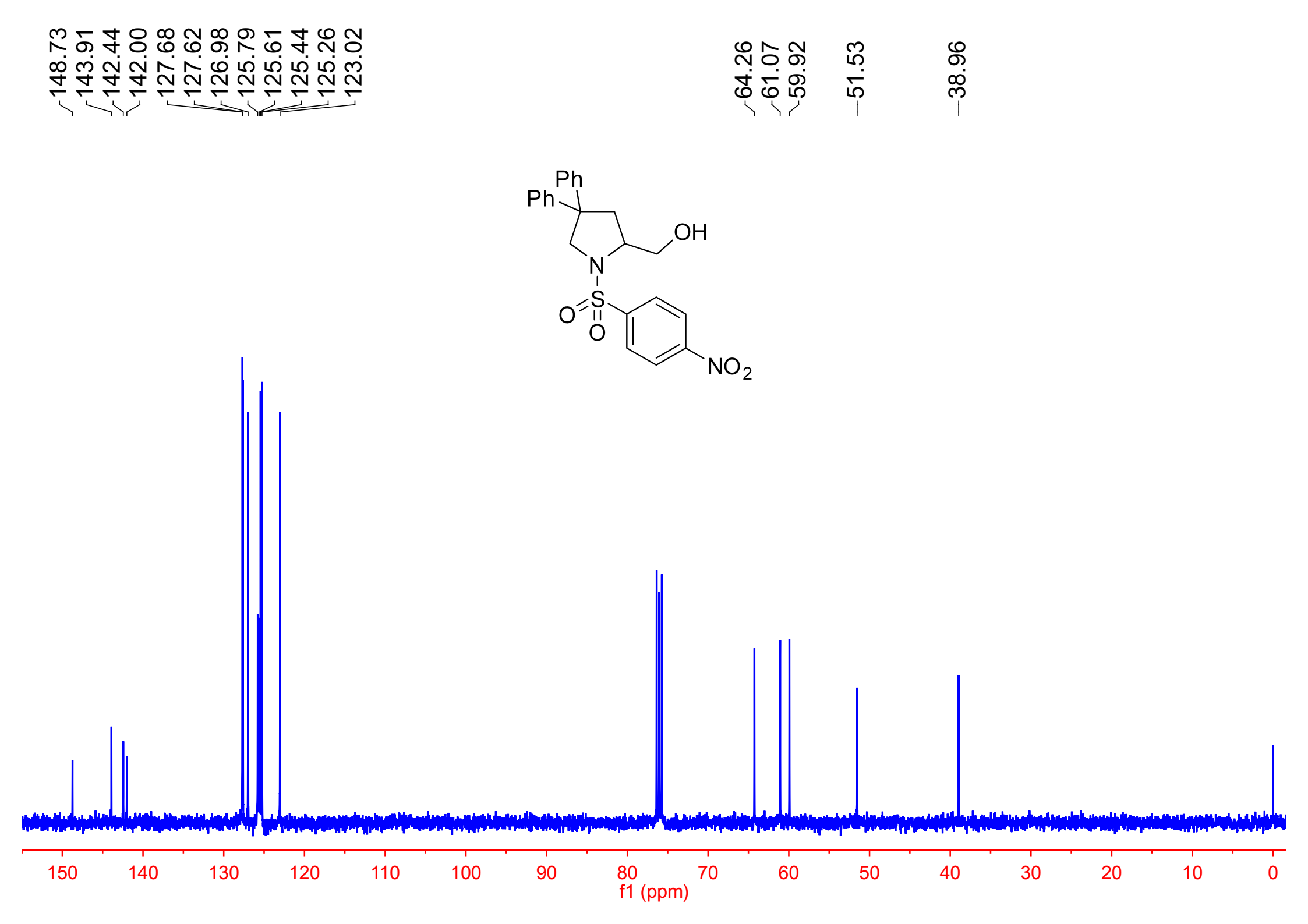
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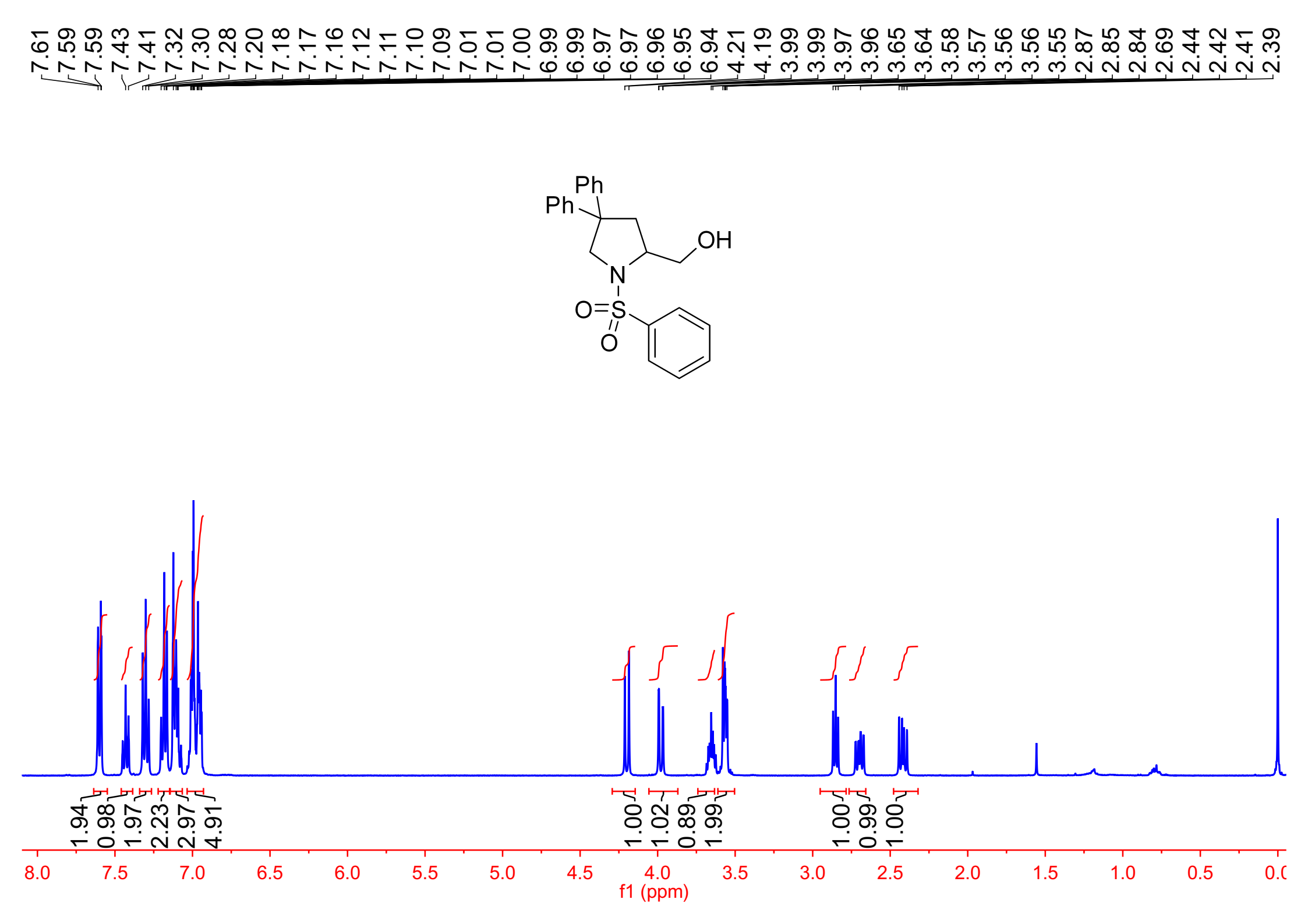
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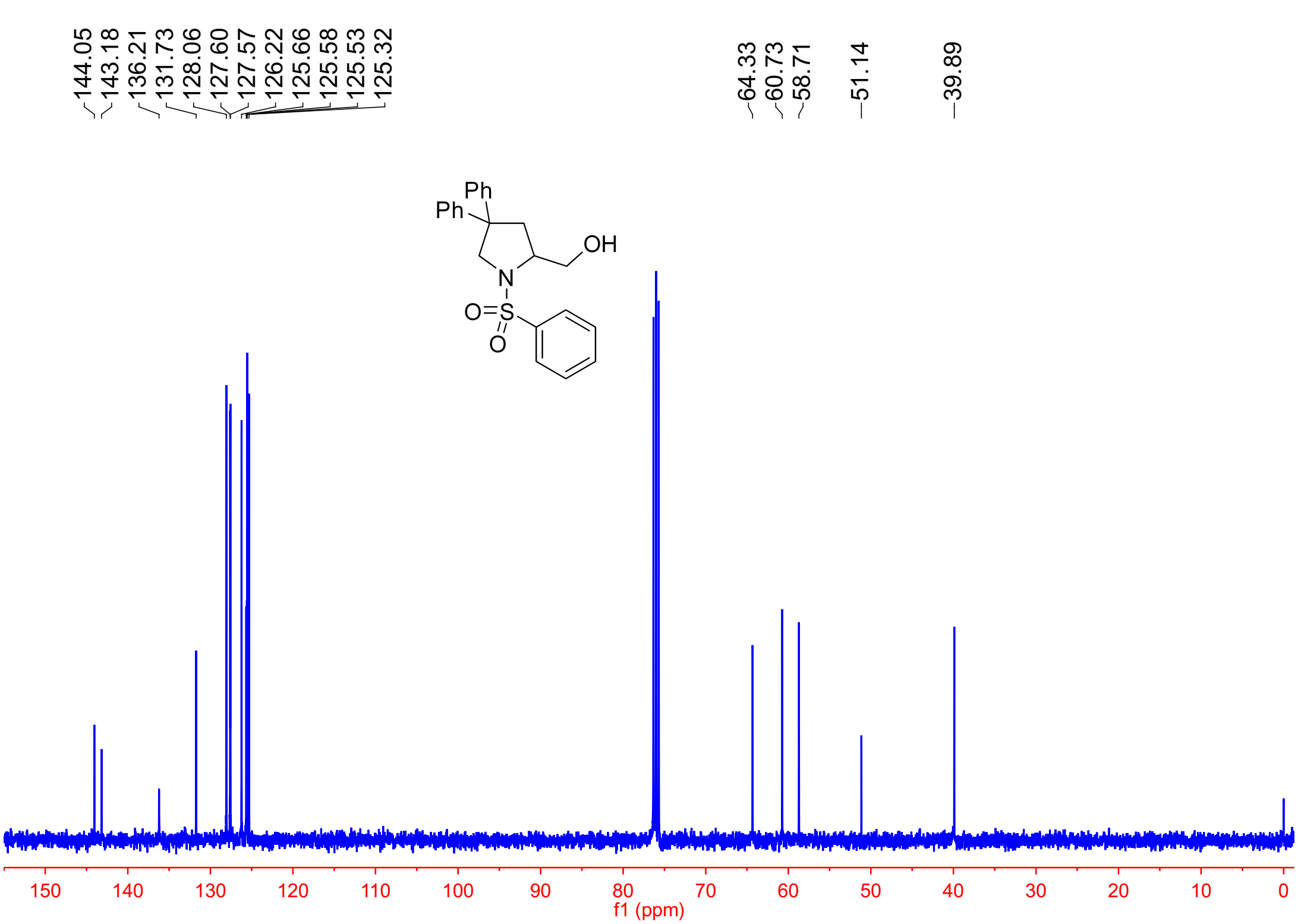
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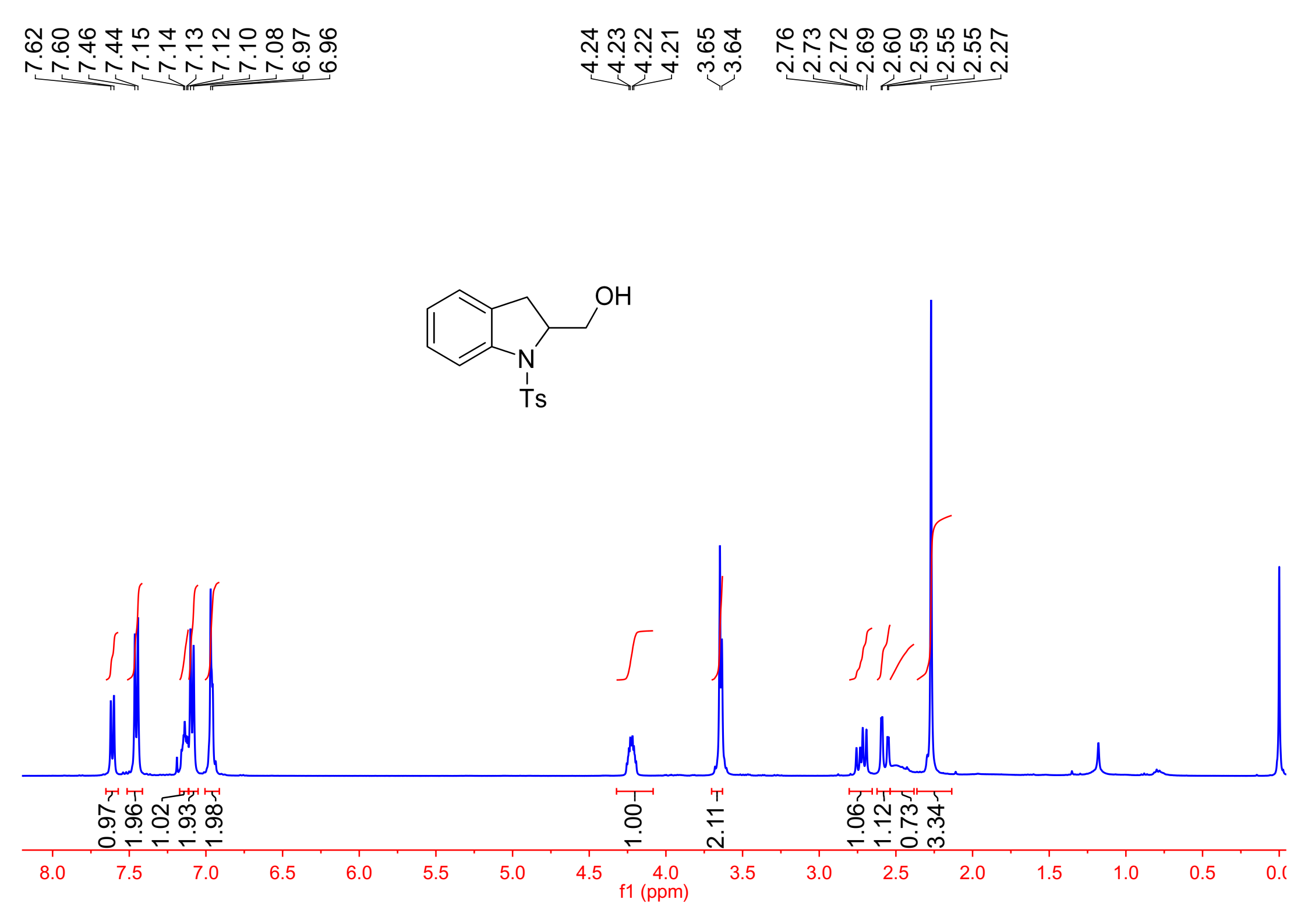
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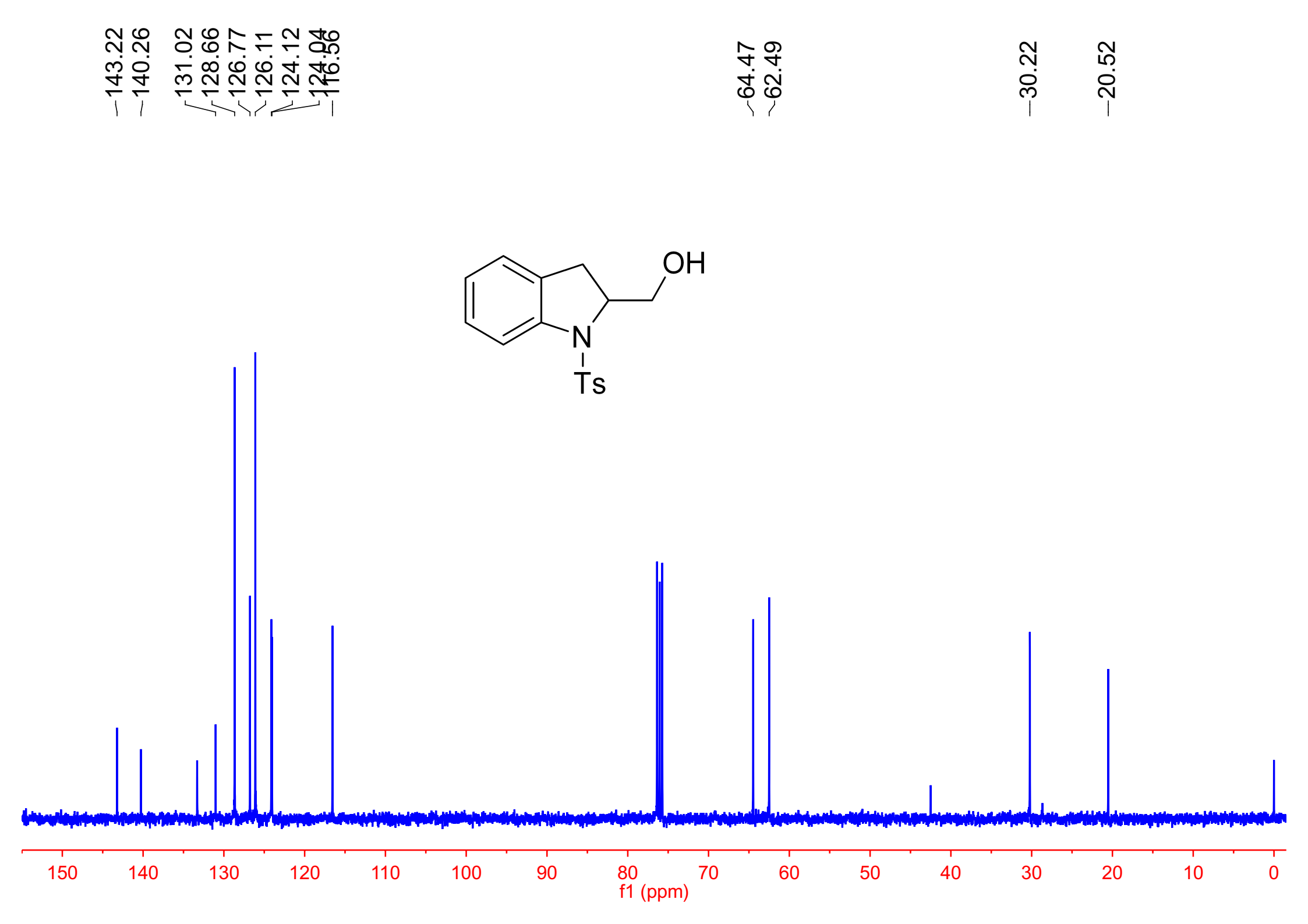
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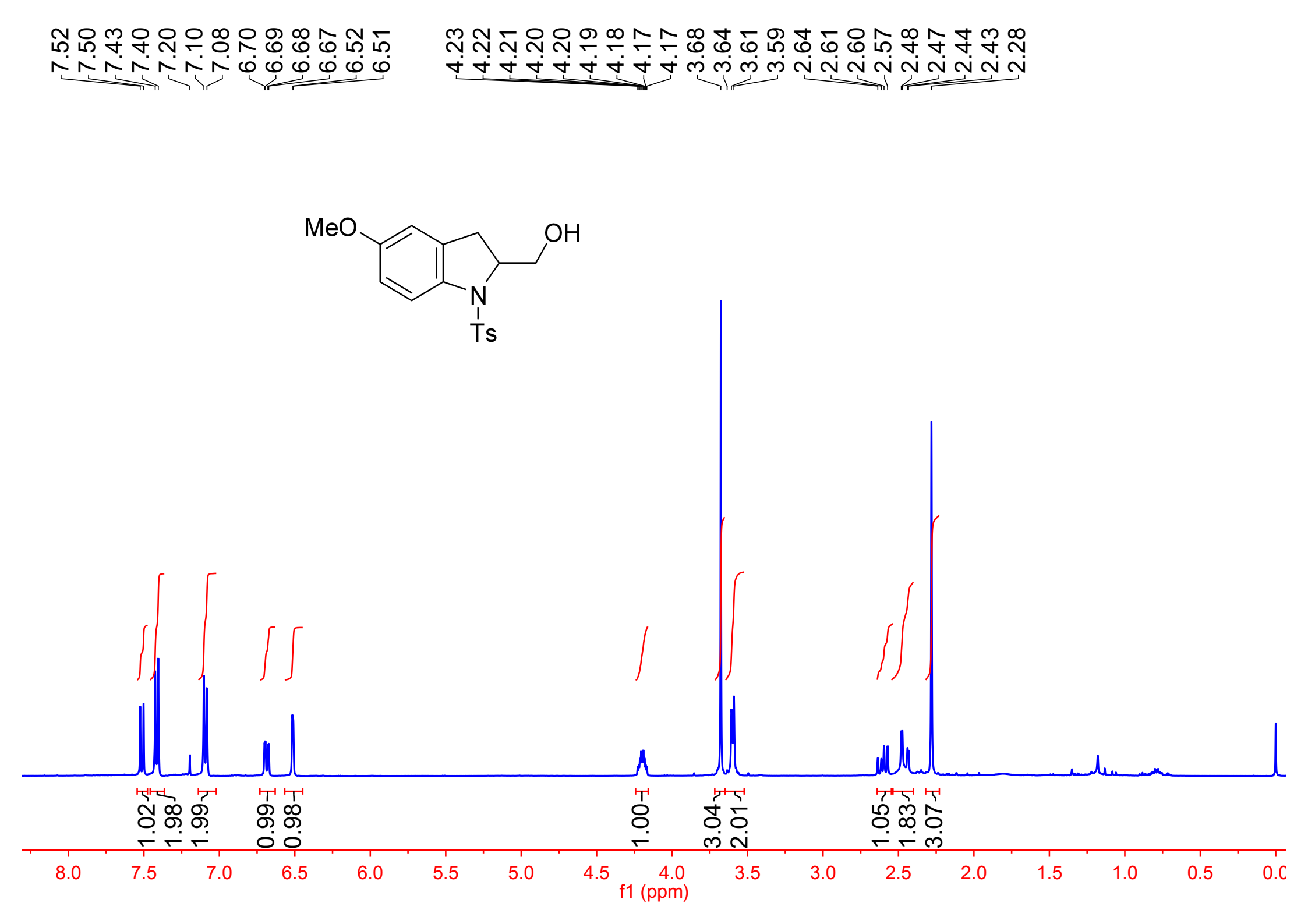
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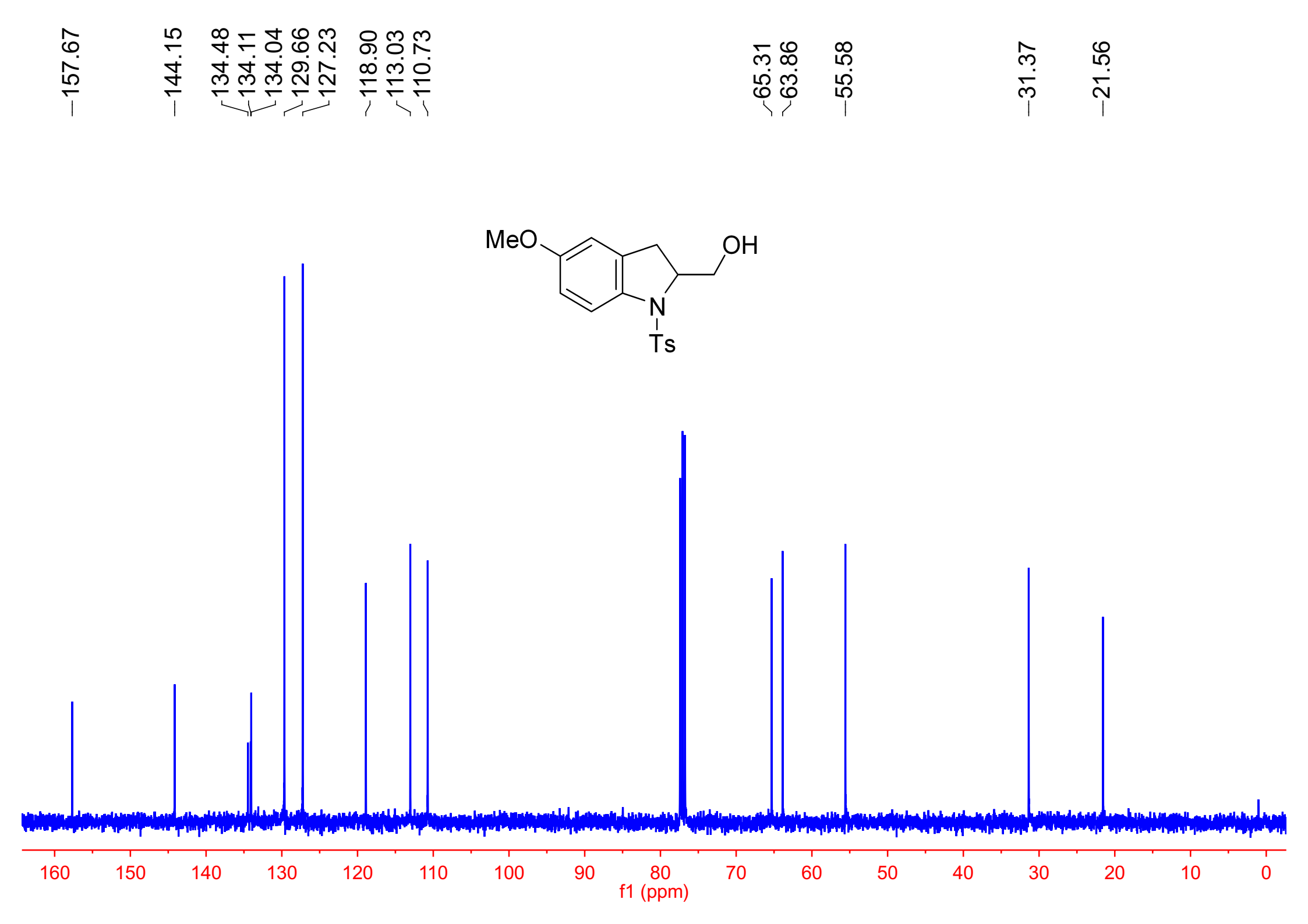
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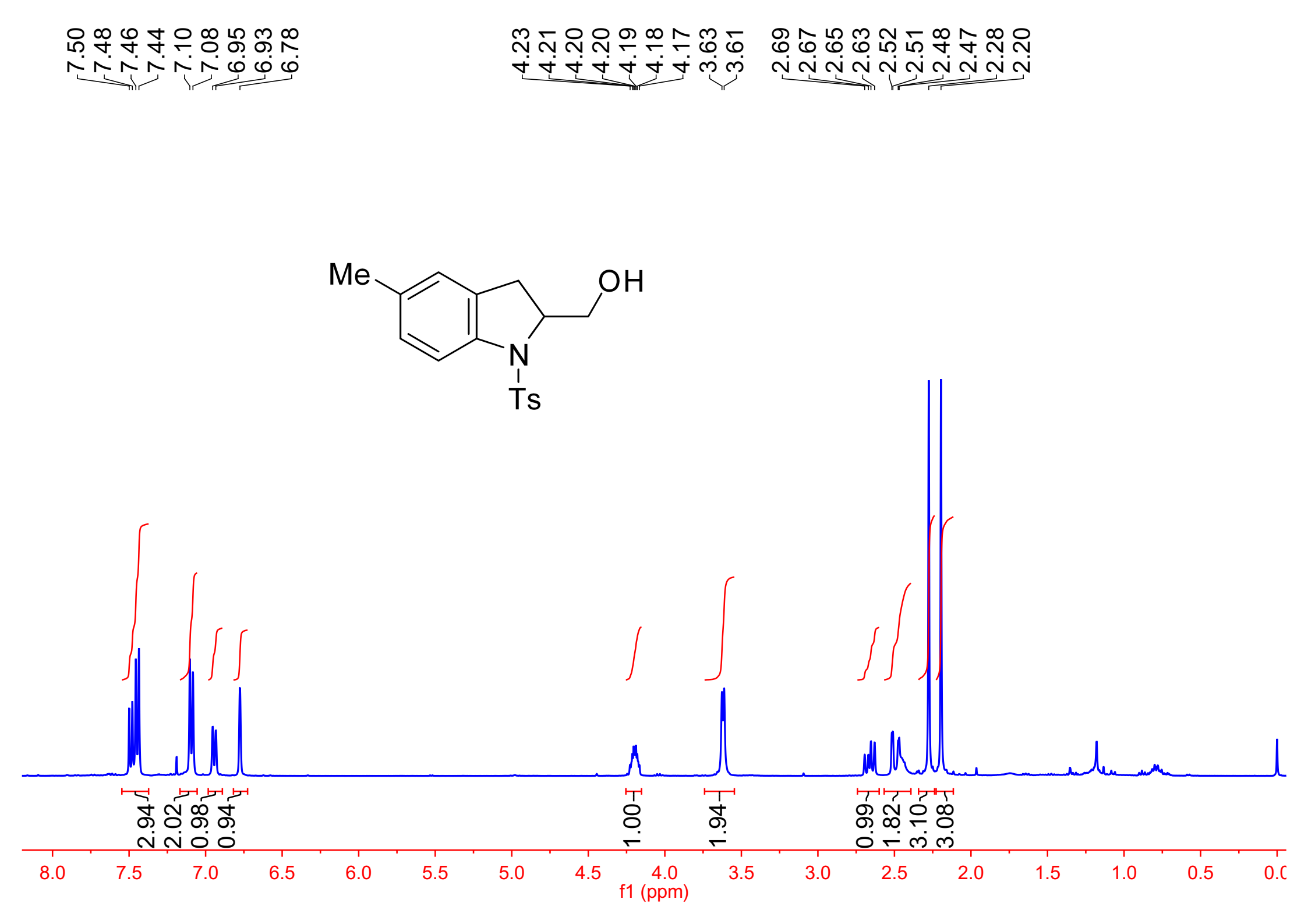
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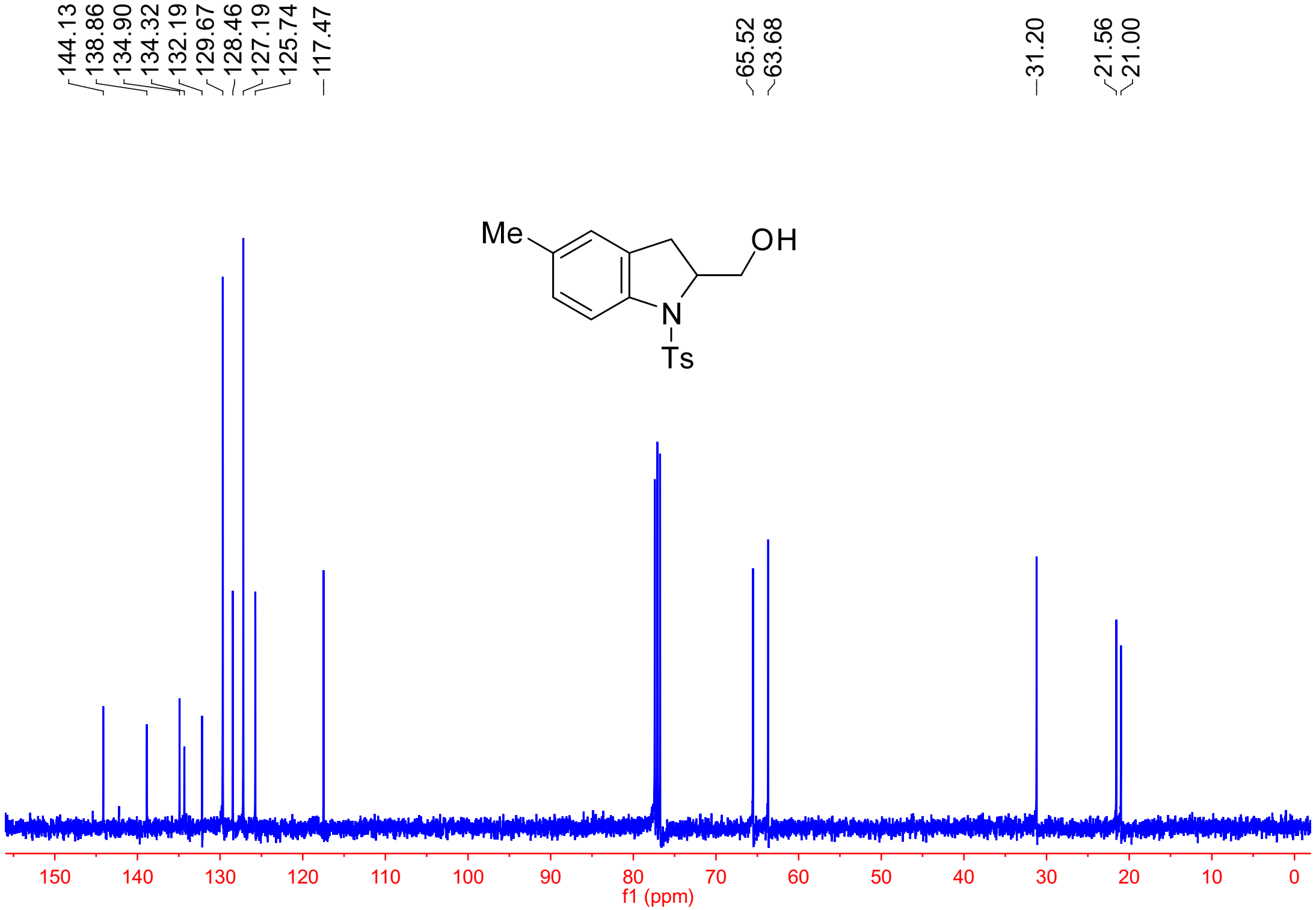
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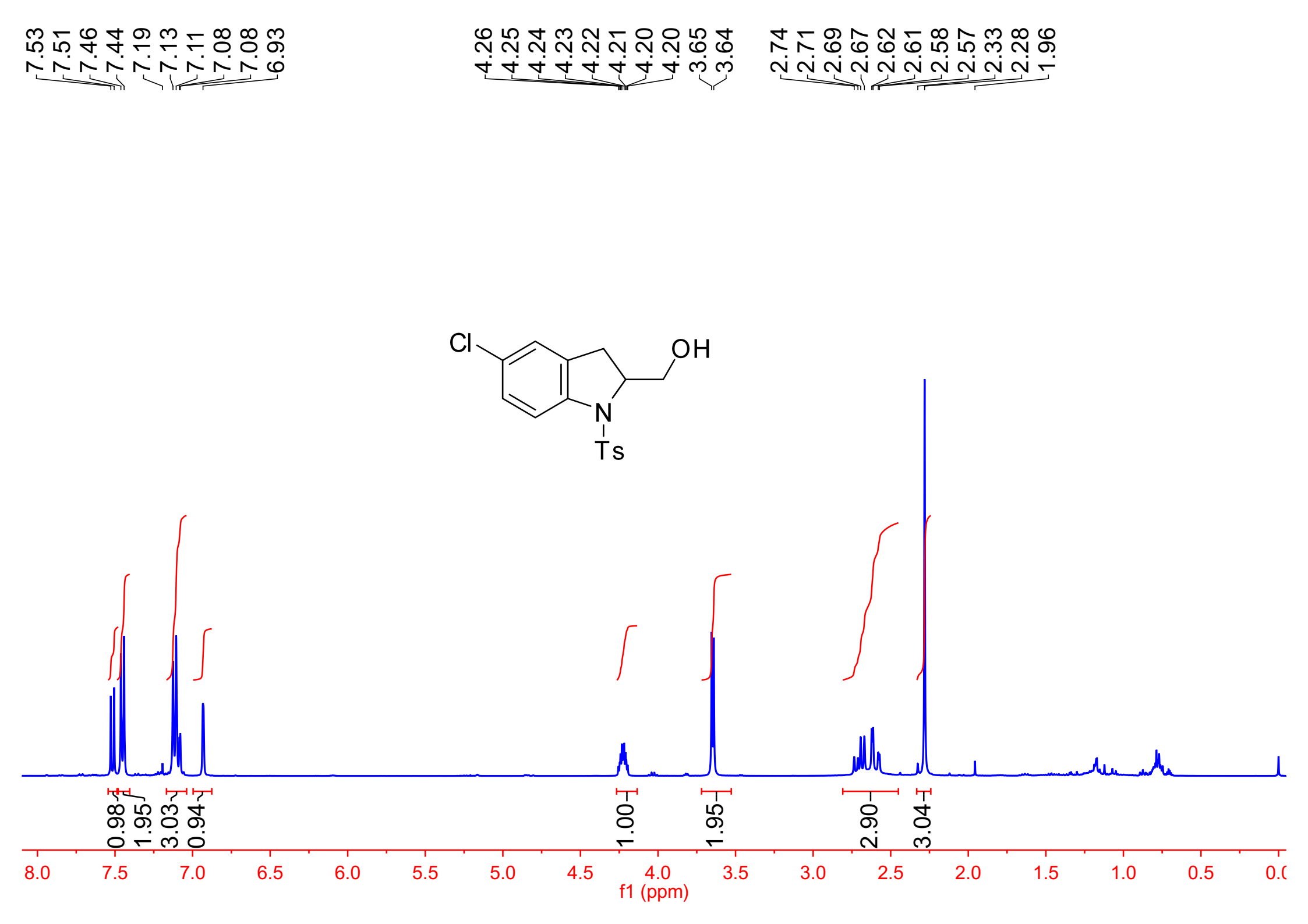
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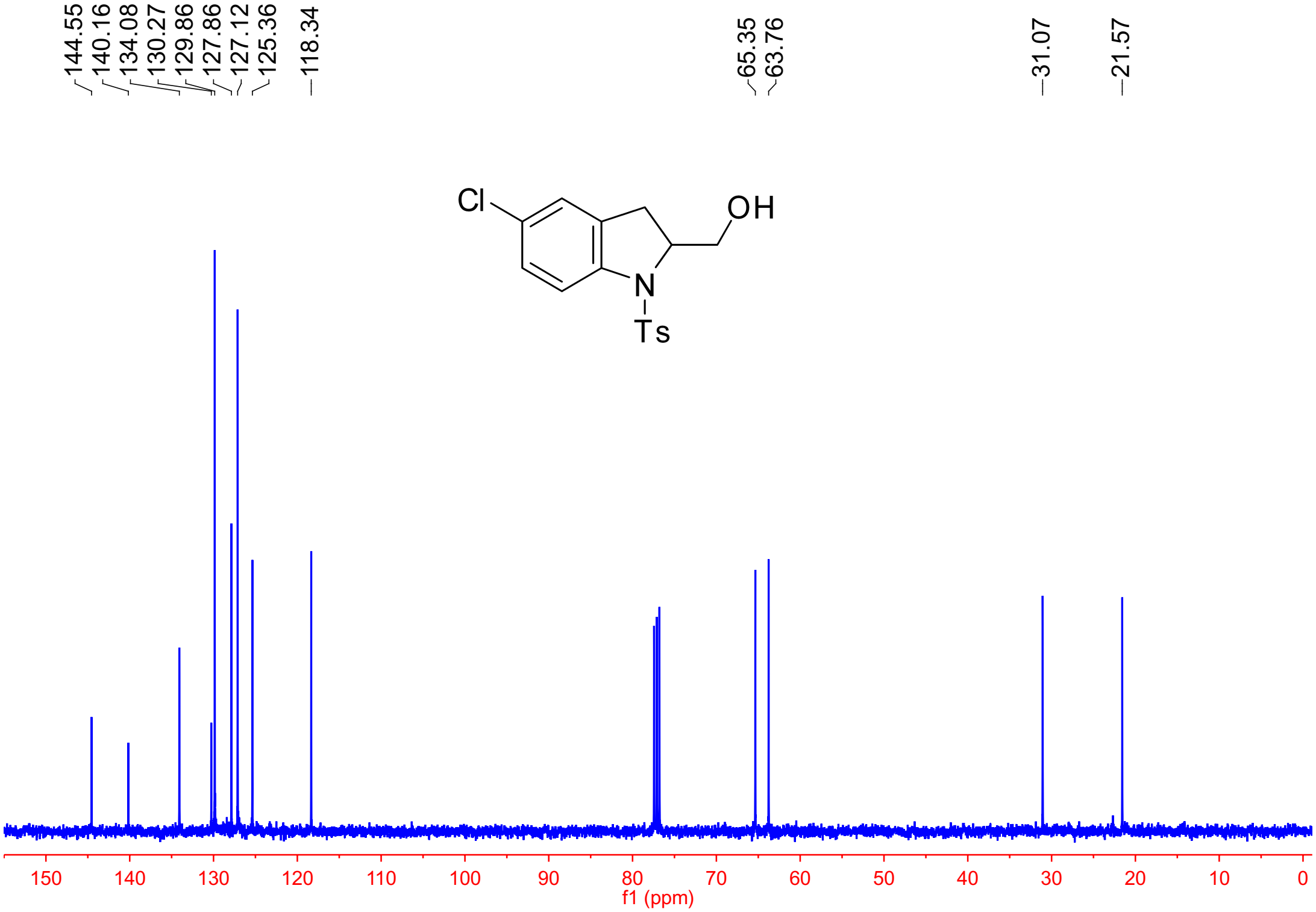
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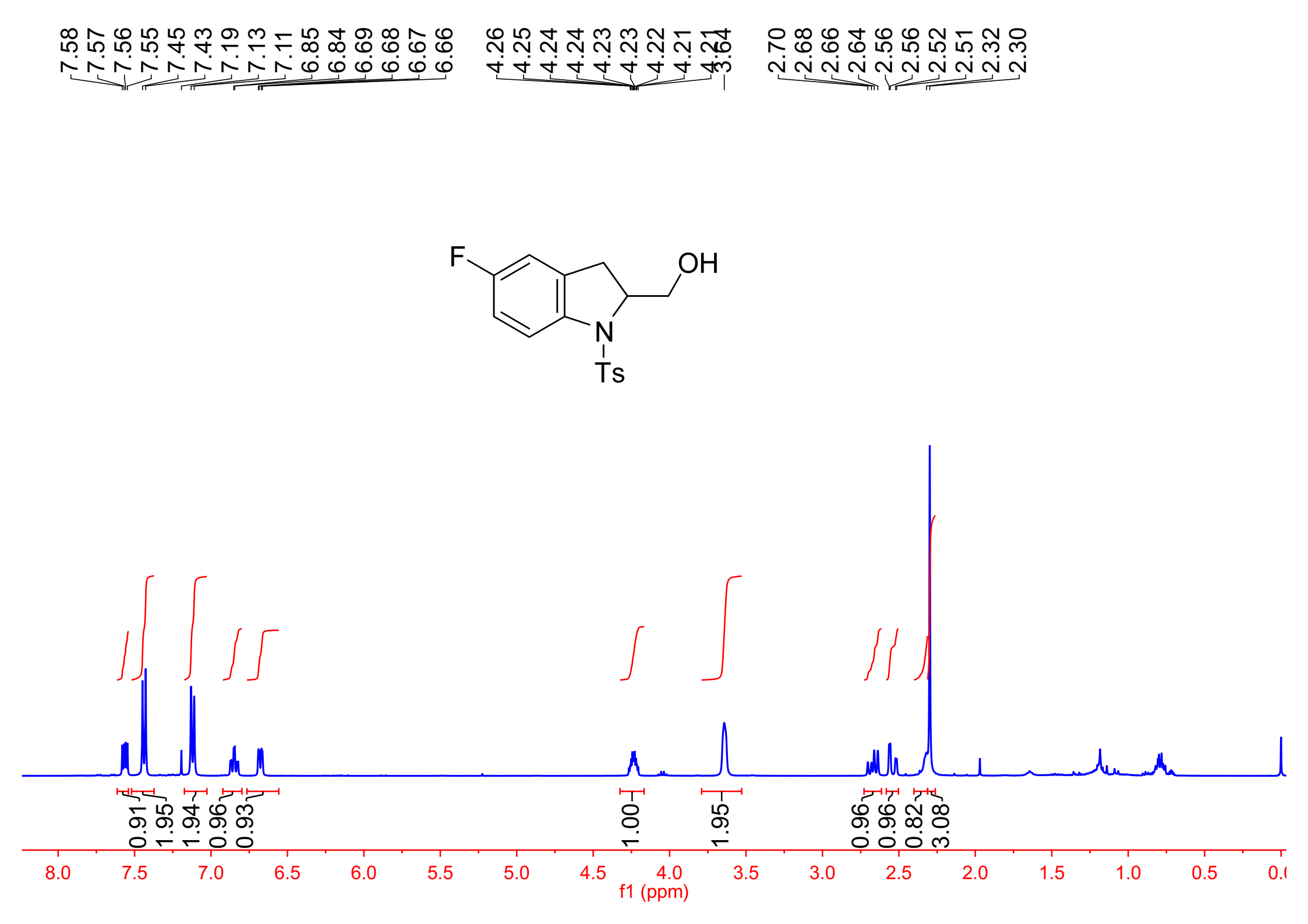
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7.13
7.11
6.85
6.84
6.69
6.68
6.67
6.66
4.26
4.25
4.24
4.24
4.23
4.23
4.22
4.21
4.21
3.64
2.70
2.68
2.66
2.64
2.56
2.56
2.52
2.51
2.32
2.30
F
OH
N
Ts
0.91
1.95
1.94
0.96
0.93
1.00
1.95
0.96
0.96
0.82
3.08
8.0
7.5
7.0
6.5
6.0
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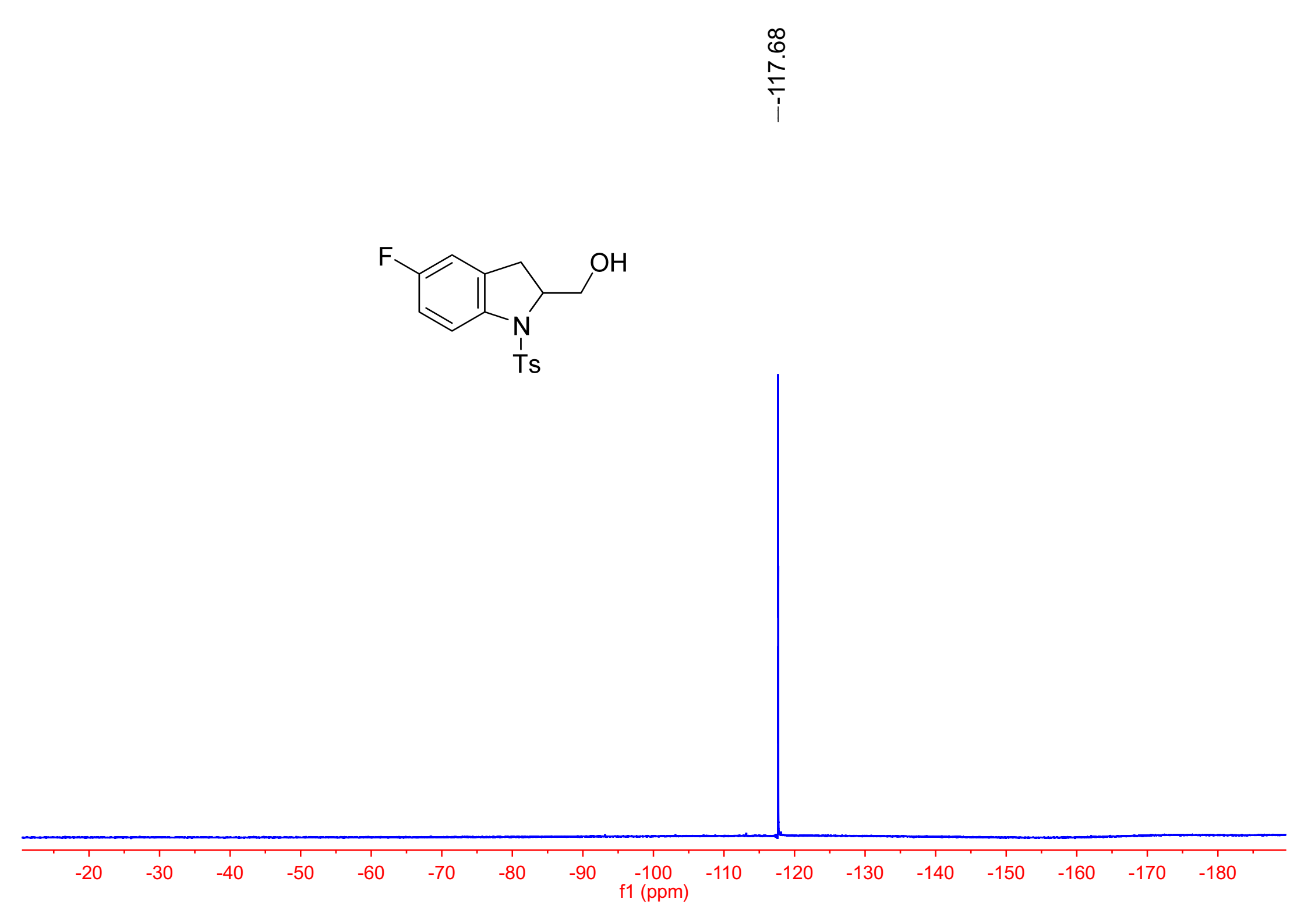
f1 (ppm)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-40
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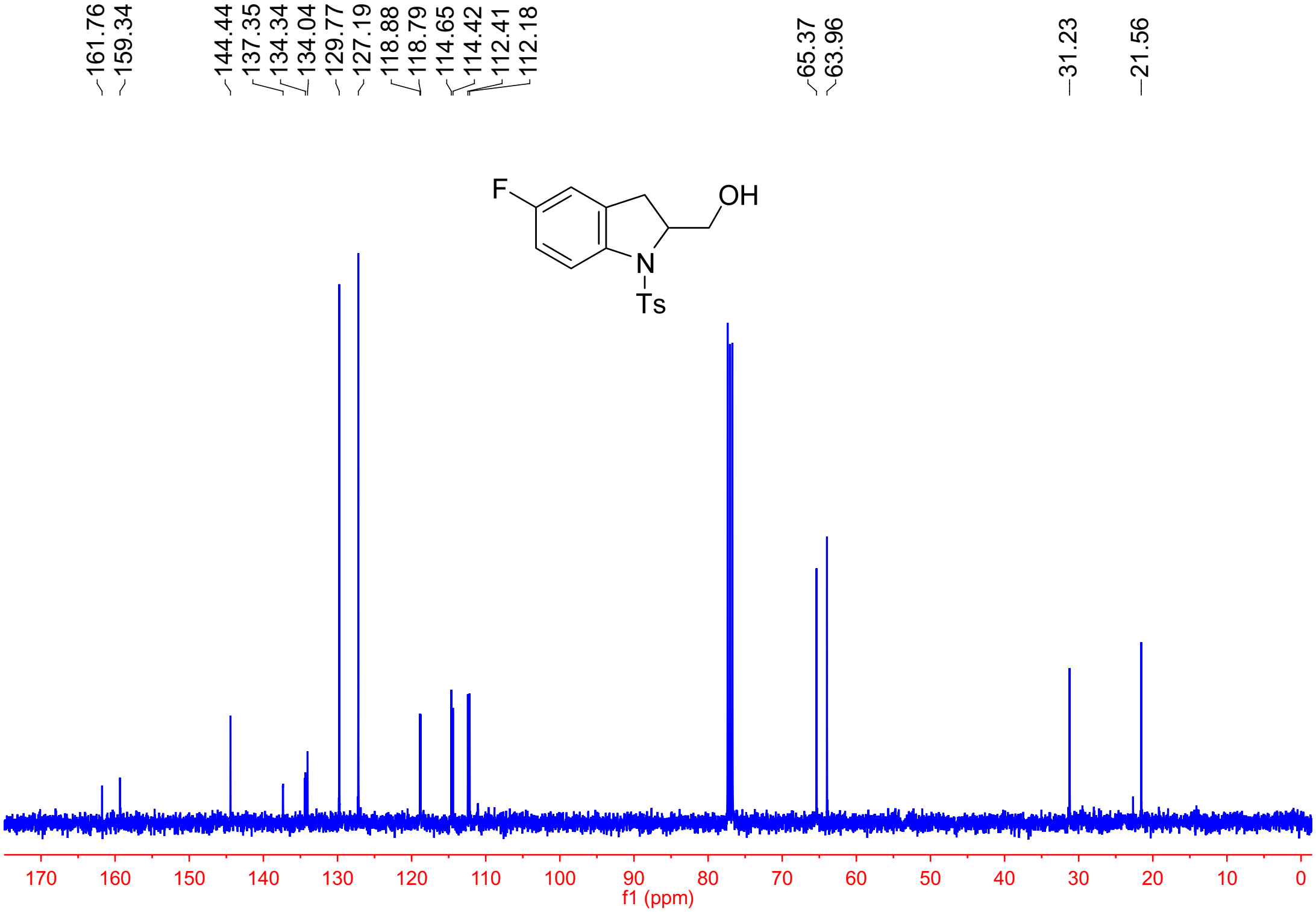
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
f1 (ppm)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114.42
112.41
112.18
65.37
63.96
31.23
21.56
F
OH
N
Ts
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90
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50
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10
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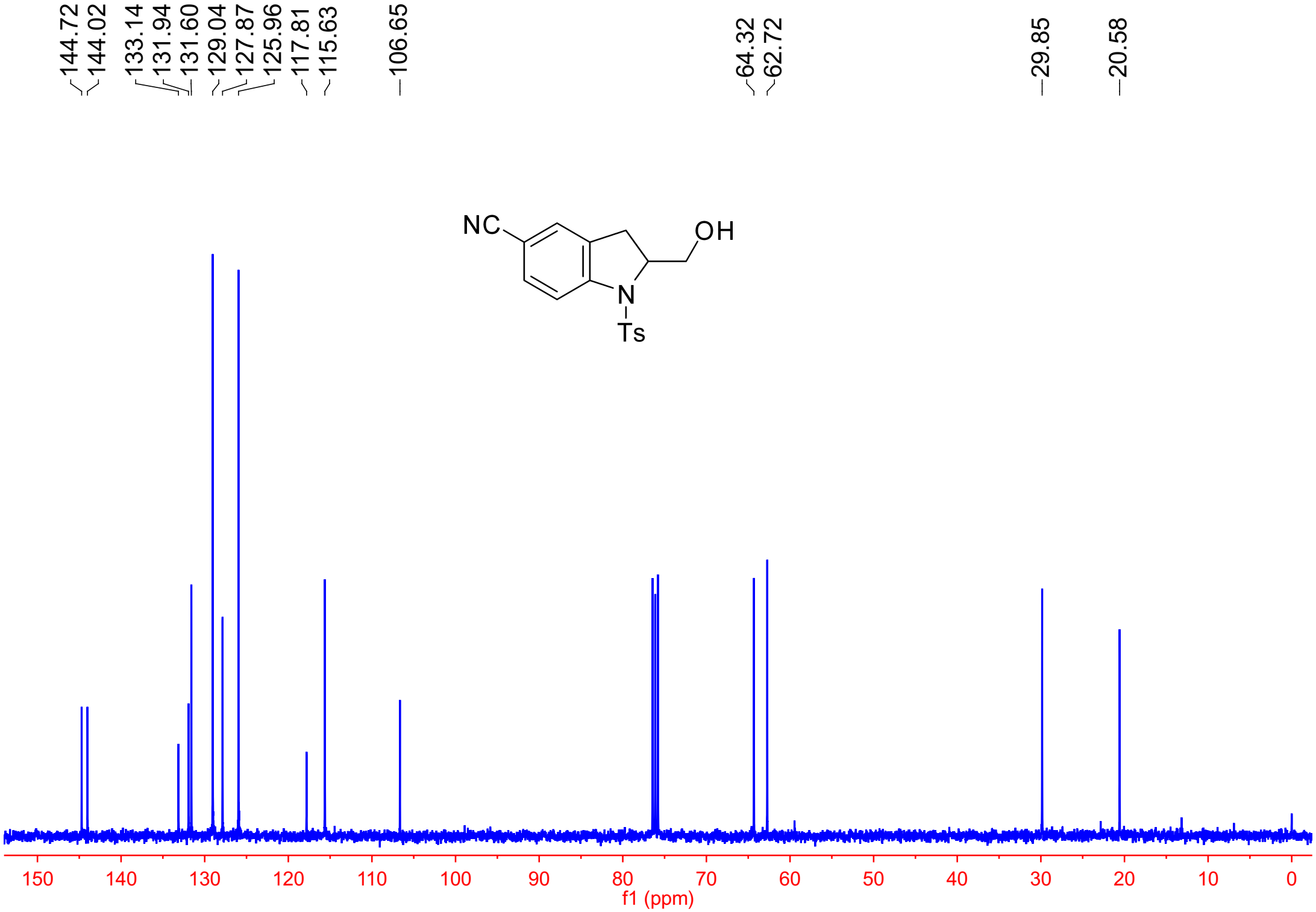
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144.02
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115.63
106.65
64.32
62.72
29.85
20.58
NC
OH
N
Ts
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130
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110
100
90
80
70
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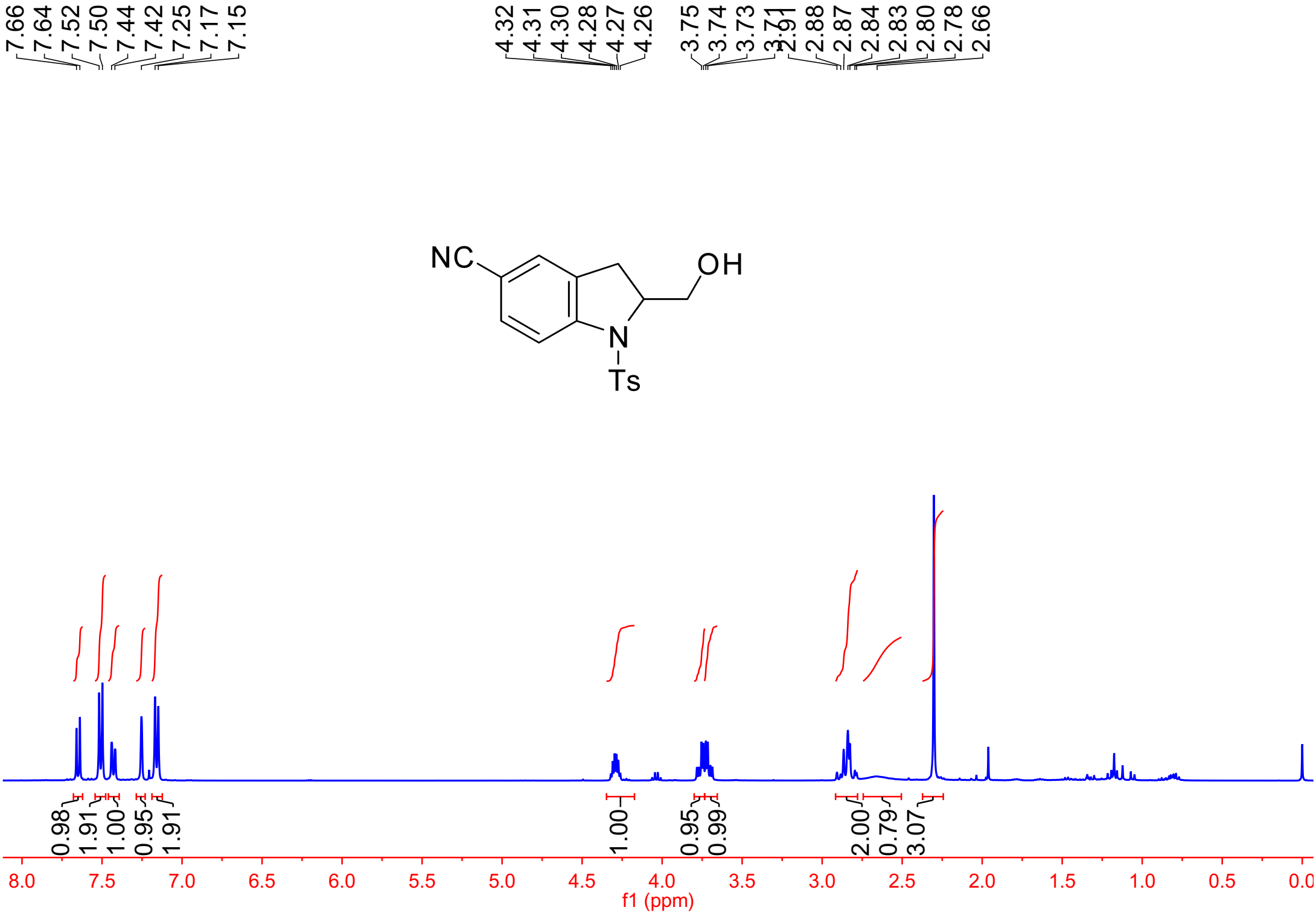
50
40
30
20
10
0
f1 (ppm)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7.17
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4.28
4.27
4.26
3.75
3.74
3.73
3.71
2.91
2.88
2.87
2.84
2.83
2.80
2.78
2.66
NC
OH
N
Ts
0.98
1.91
1.00
0.95
1.91
1.00
0.95
0.99
2.00
0.79
3.07
8.0
7.5
7.0
6.5
6.0
5.5
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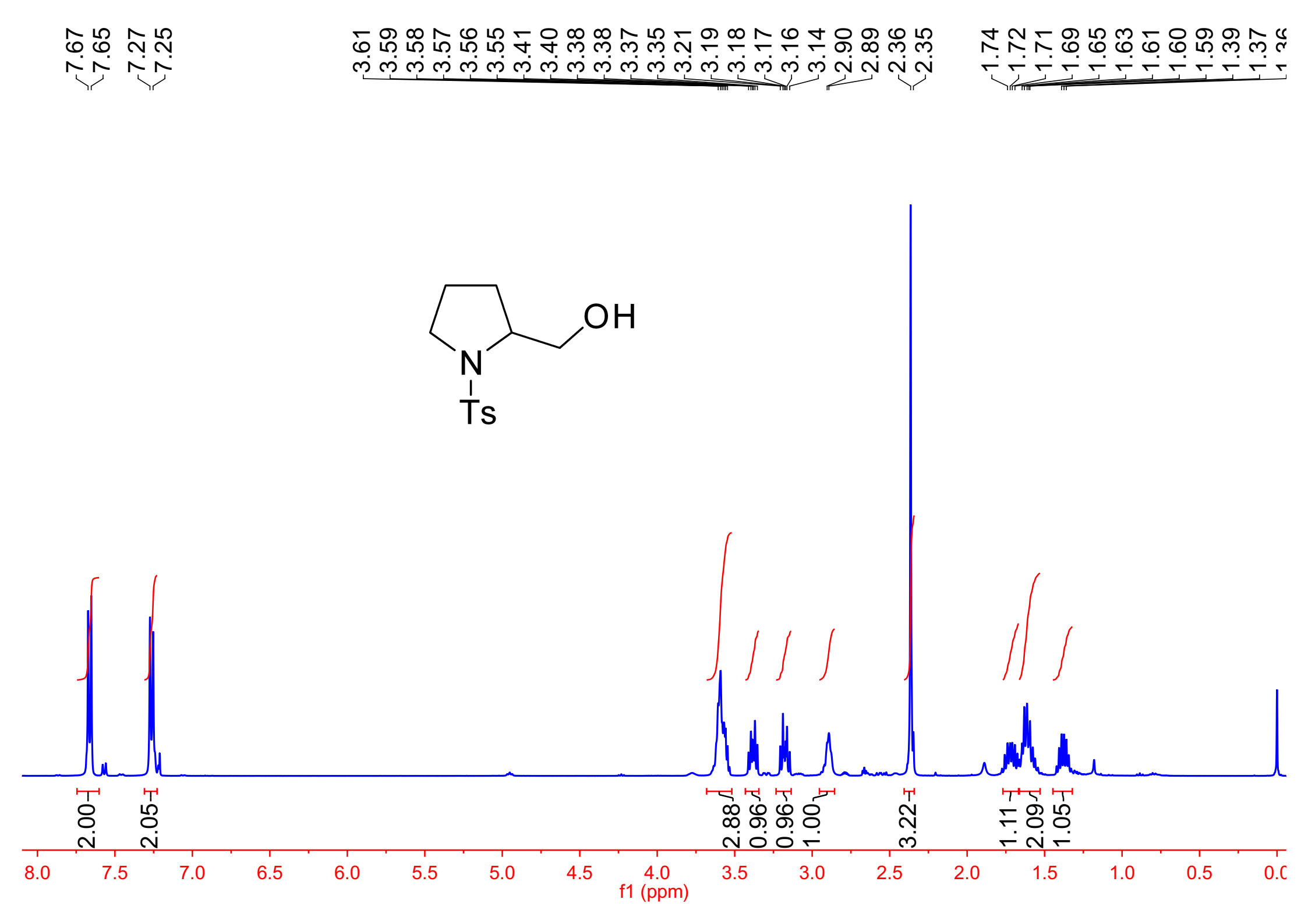
f1 (ppm)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3.38
3.38
3.37
3.35
3.21
3.19
3.18
3.17
3.16
3.14
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2.36
2.35
1.74
1.72
1.71
1.69
1.65
1.63
1.61
1.60
1.59
1.39
1.37
OH
N
Ts
2.00
2.05
2.88
0.96
0.96
1.00
3.22
1.11
2.09
1.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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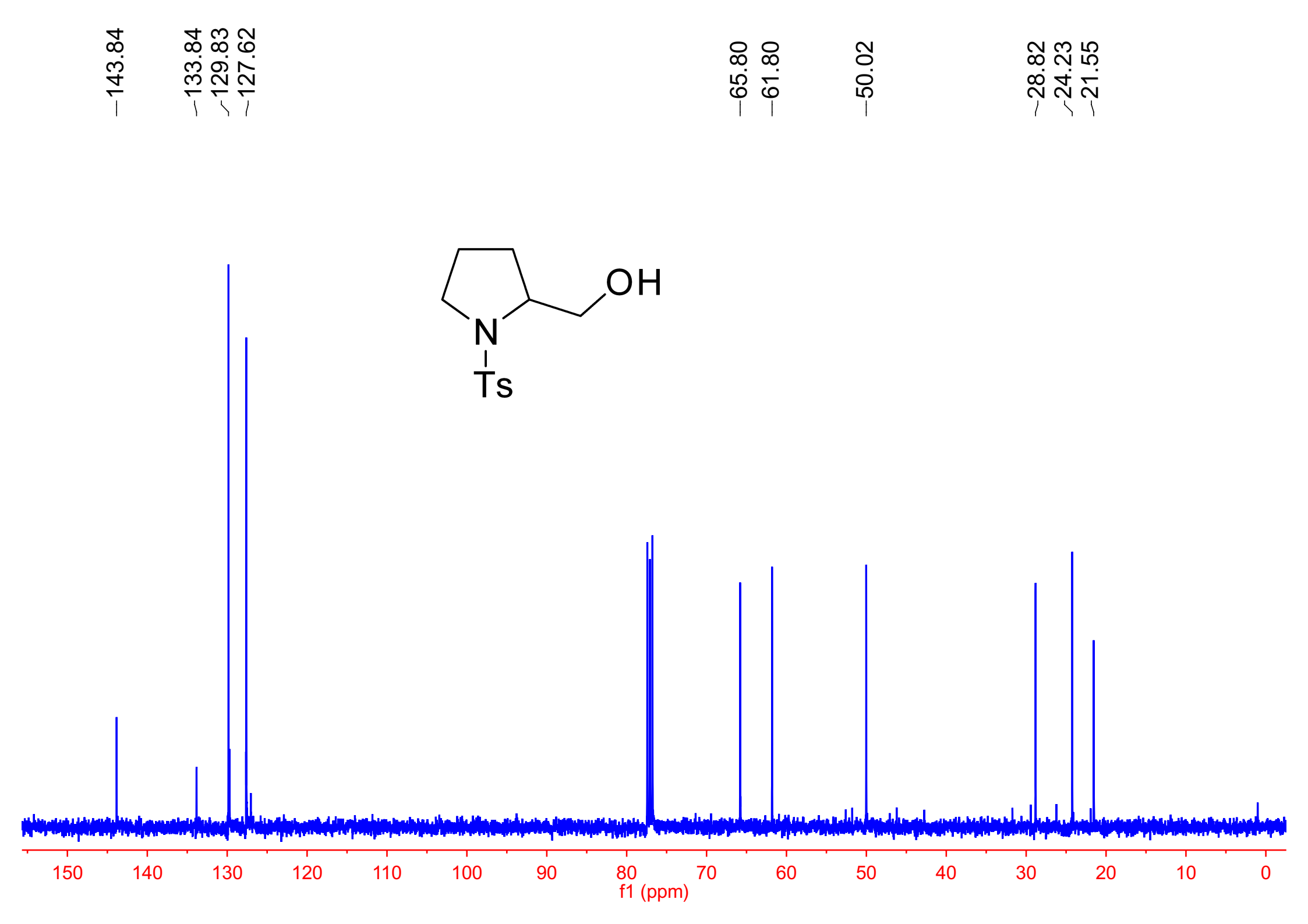
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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133.84
129.83
127.62
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61.80
50.02
28.82
24.23
21.55
OH
N
Ts
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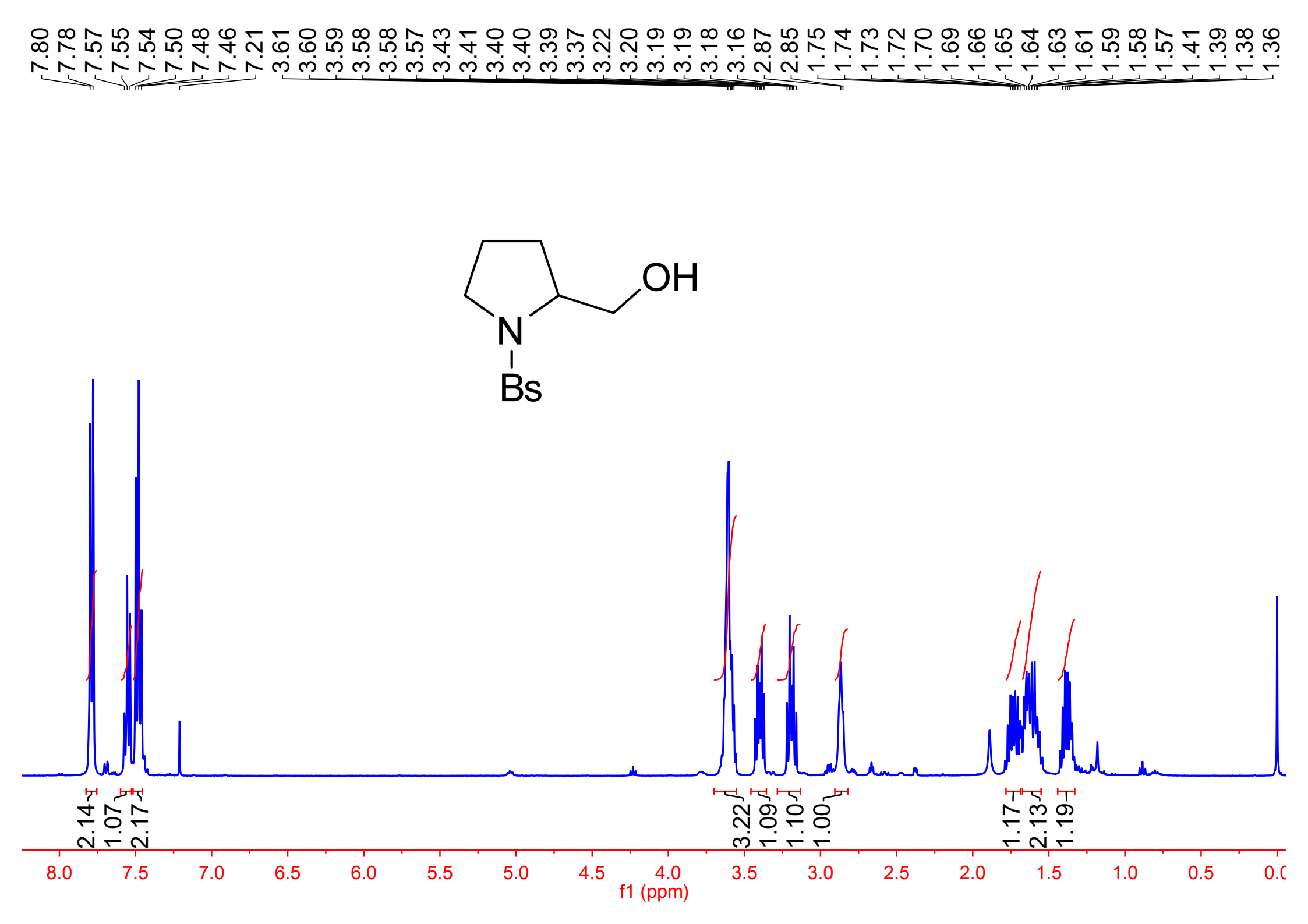
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.78
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7.50
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7.46
7.21
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3.60
3.59
3.58
3.58
3.57
3.43
3.41
3.40
3.40
3.39
3.37
3.22
3.20
3.19
3.19
3.18
3.16
2.87
2.85
1.75
1.74
1.73
1.72
1.70
1.69
1.66
1.65
1.64
1.63
1.61
1.59
1.58
1.57
1.41
1.39
1.38
1.36
OH
N
Bs
2.14
1.07
2.17
3.22
1.09
1.10
1.00
1.17
2.13
1.19
8.0
7.5
7.0
6.5
6.0
5.5
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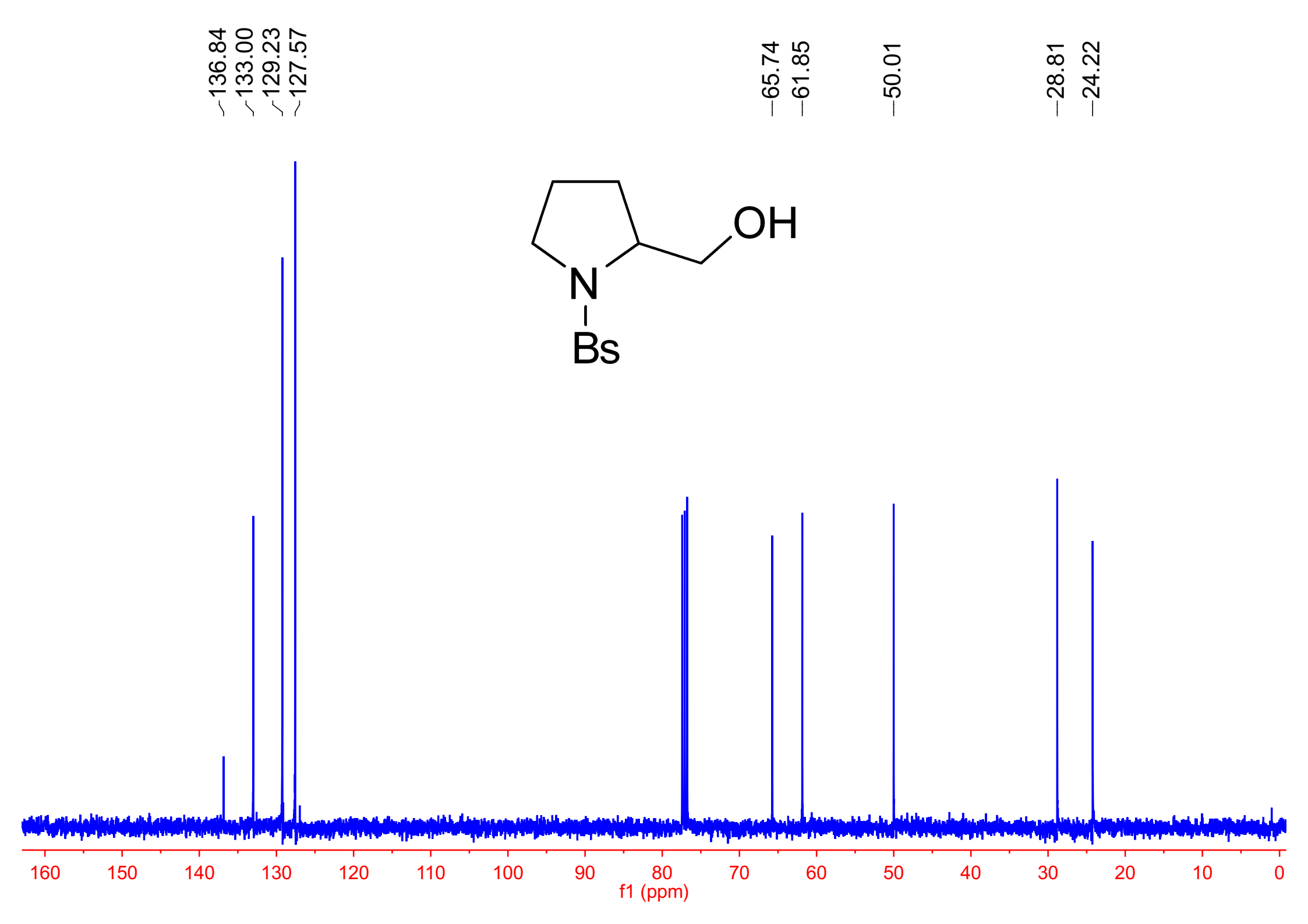
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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65.74
61.85
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28.81
24.22
OH
N
Bs
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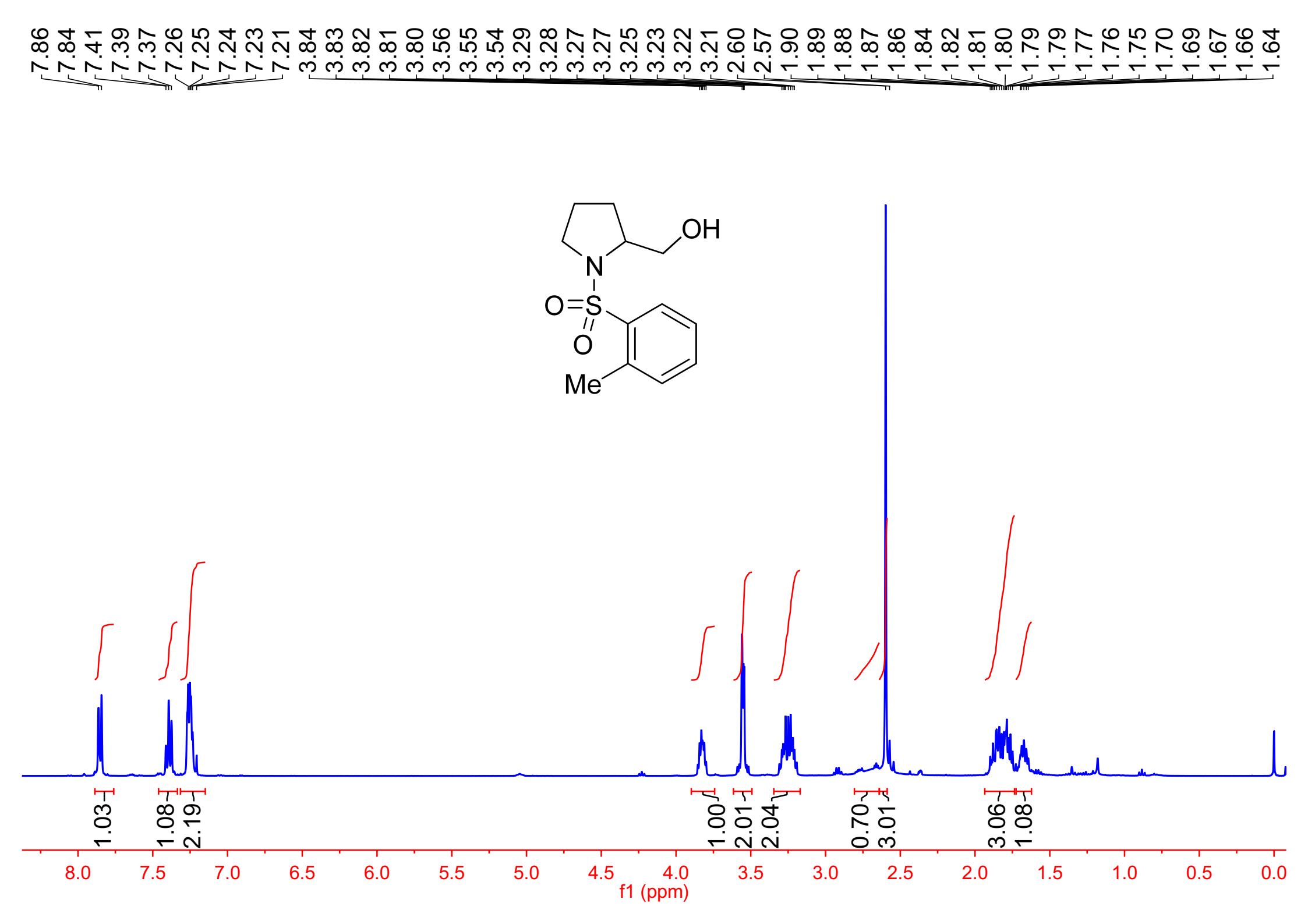
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.26
7.25
7.24
7.23
7.21
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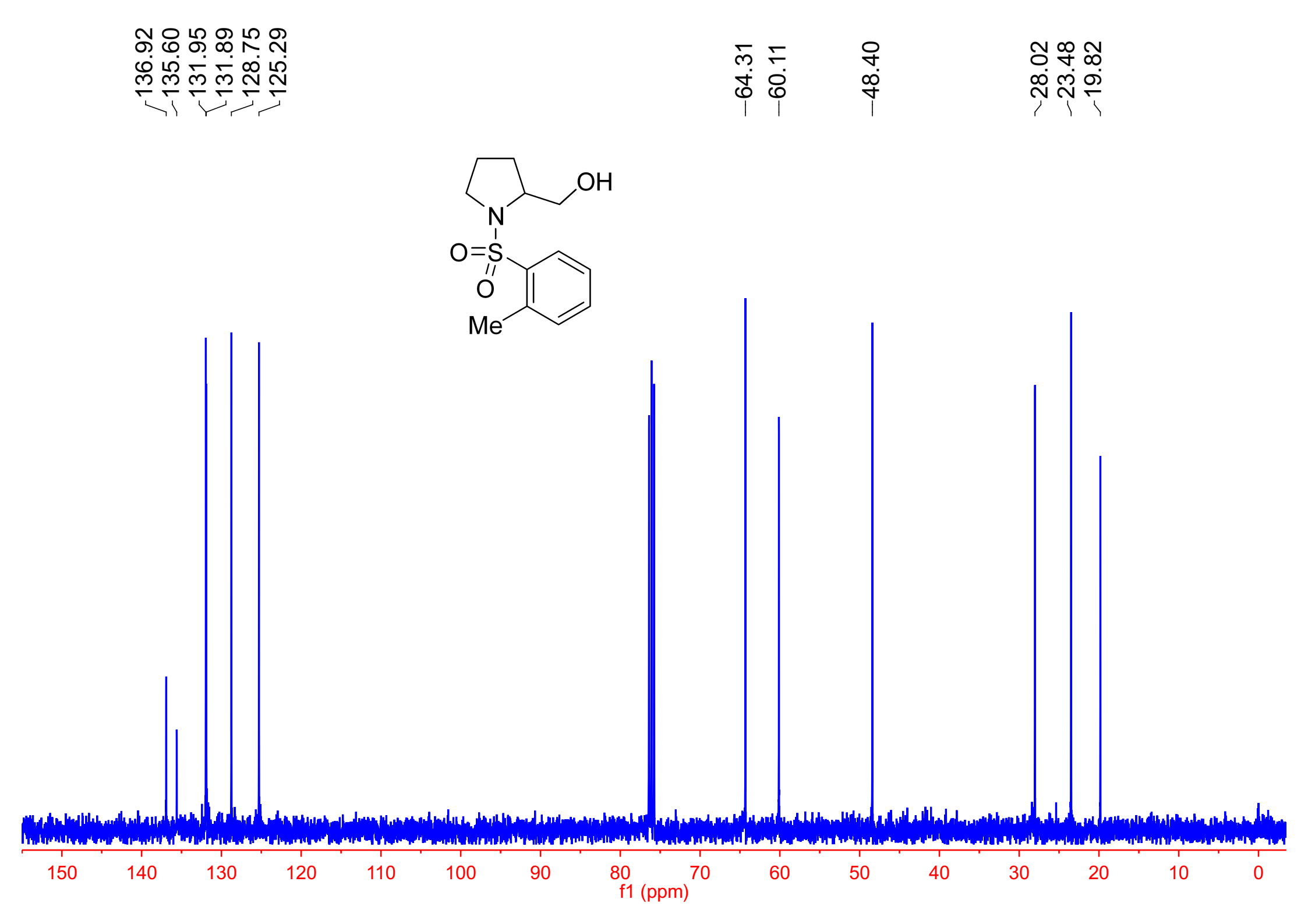
3.83
3.82
3.81
3.80
3.56
3.55
3.54
3.29
3.28
3.27
3.27
3.25
3.23
3.22
3.21
2.60
2.57
1.90
1.89
1.88
1.87
1.86
1.84
1.82
1.81
1.80
1.79
1.79
1.77
1.76
1.75
1.70
1.69
1.67
1.66
1.64
OH
N
O=S
O
Me
1.03
1.08
2.19
1.00
2.01
2.04
0.70
3.01
3.06
1.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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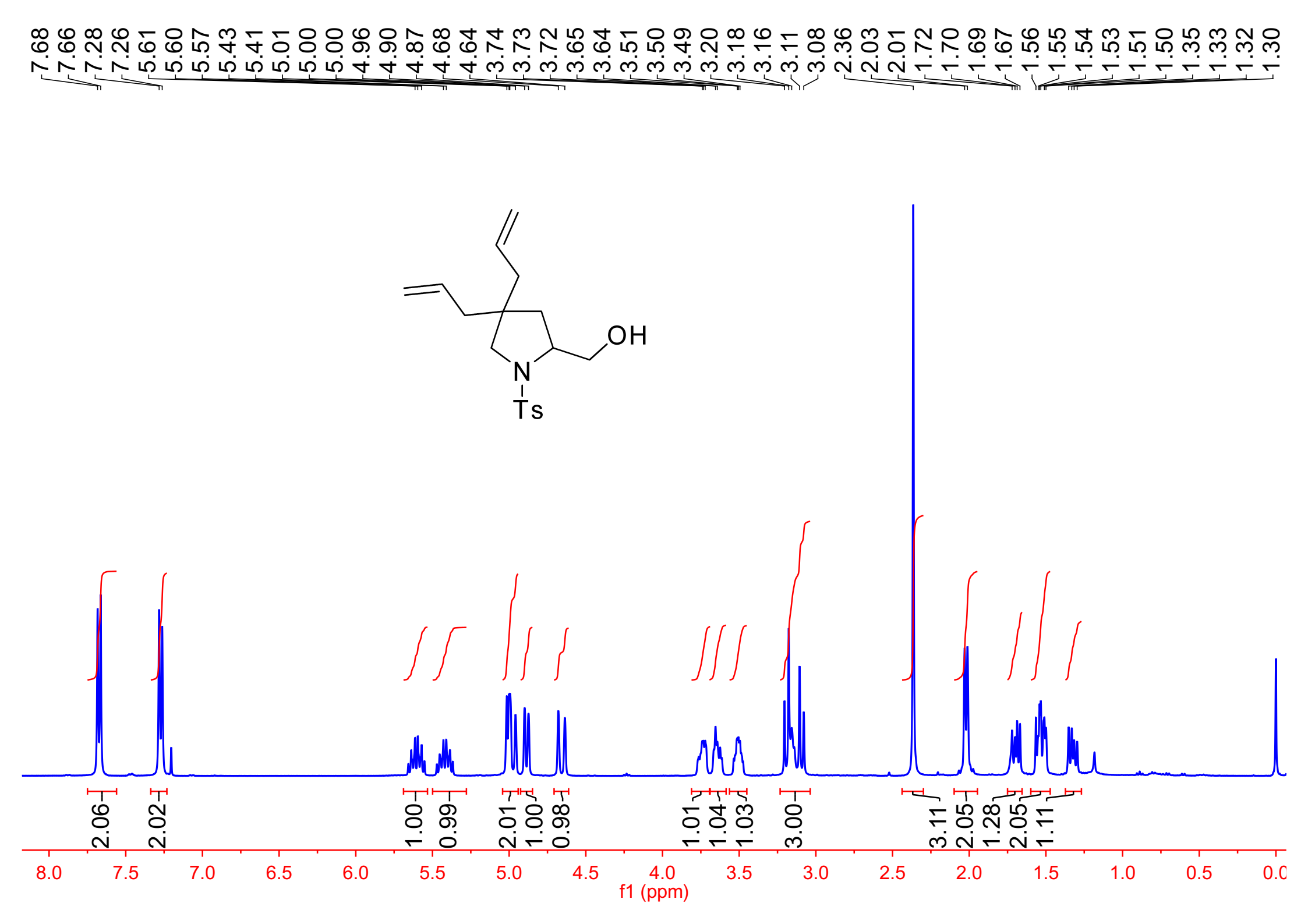
125.29
64.31
60.11
48.40
28.02
23.48
19.82
OH
N
O=S
O
Me
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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5.41
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5.00
5.00
4.96
4.90
4.87
4.68
4.64
3.74
3.73
3.72
3.65
3.64
3.51
3.50
3.49
3.20
3.18
3.16
3.11
3.08
2.36
2.03
2.01
1.72
1.70
1.69
1.67
1.56
1.55
1.54
1.53
1.51
1.50
1.35
1.33
1.32
1.30
OH
N
Ts
2.06
2.02
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2.01
1.00
0.98
1.01
1.04
1.03
3.00
3.11
2.05
1.28
2.05
1.11
8.0
7.5
7.0
6.5
6.0
5.5
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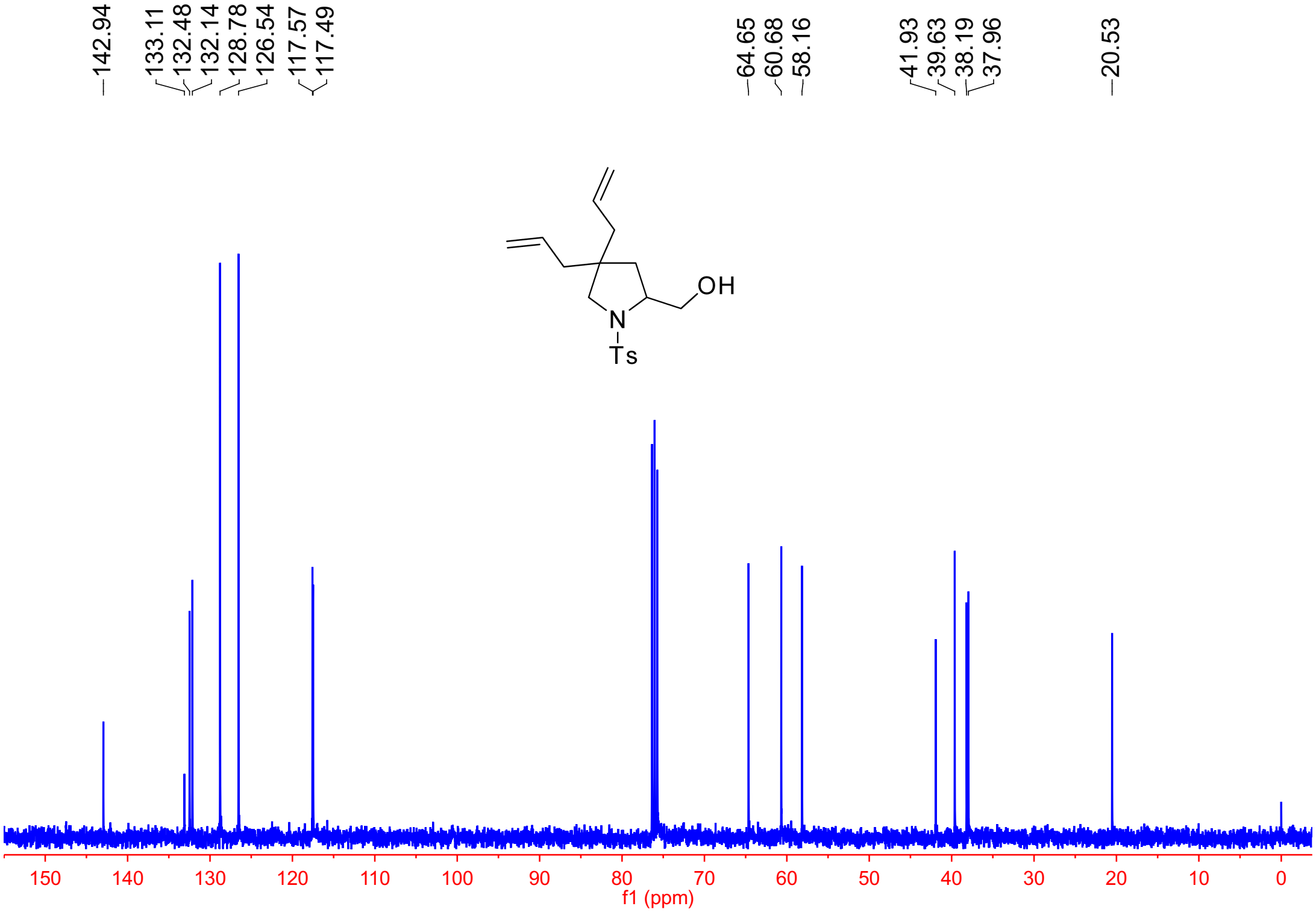
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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41.93
39.63
38.19
37.96
20.53
OH
N
Ts
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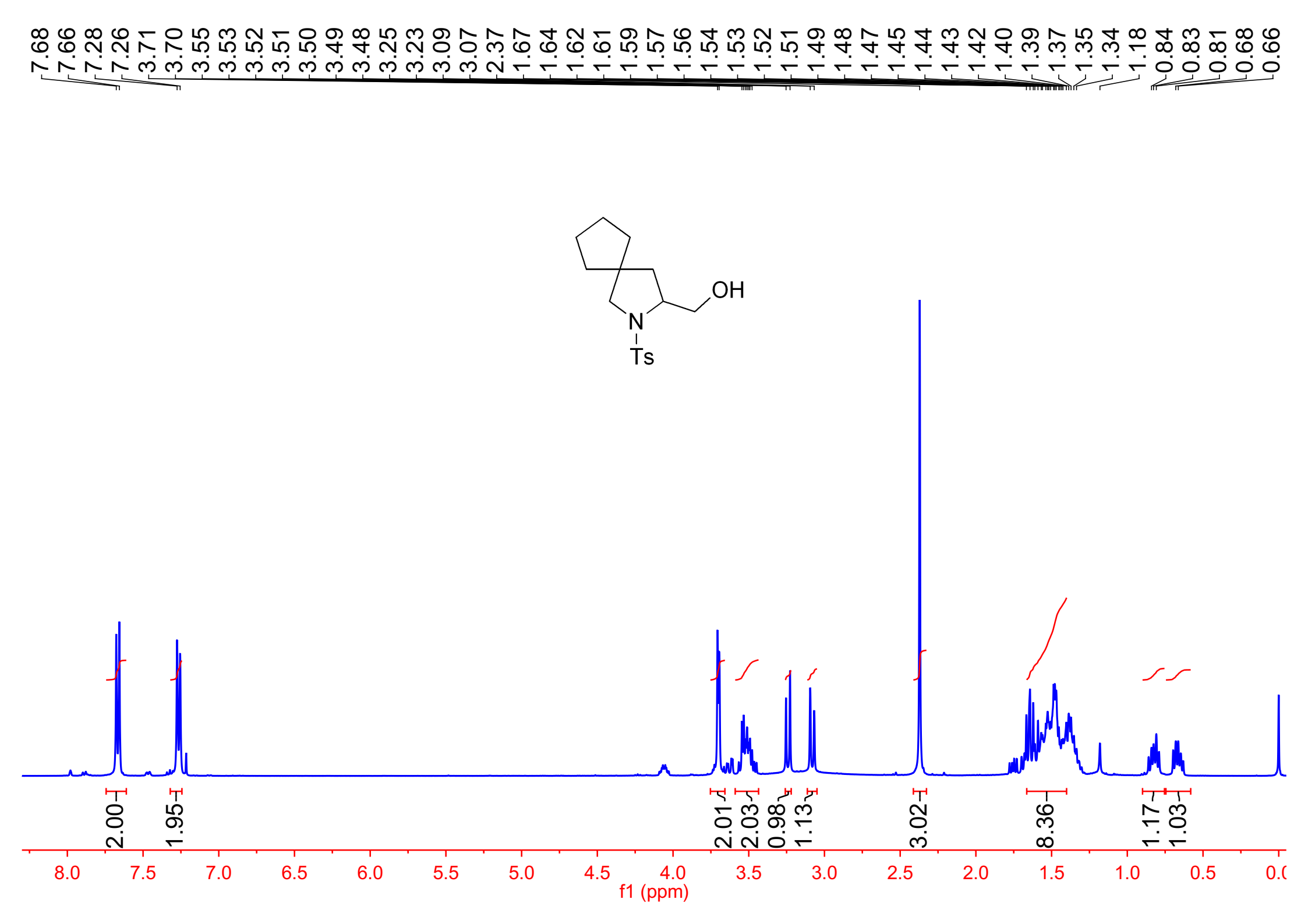
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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3.51
3.50
3.49
3.48
3.25
3.23
3.09
3.07
2.37
1.67
1.64
1.62
1.61
1.59
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1.56
1.54
1.53
1.52
1.51
1.49
1.48
1.47
1.45
1.44
1.43
1.42
1.40
1.39
1.37
1.35
1.34
1.18
0.84
0.83
0.81
0.68
0.66
OH
N
Ts
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1.95
2.01
2.03
0.98
1.13
3.02
8.36
1.17
1.03
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7.0
6.5
6.0
5.5
5.0
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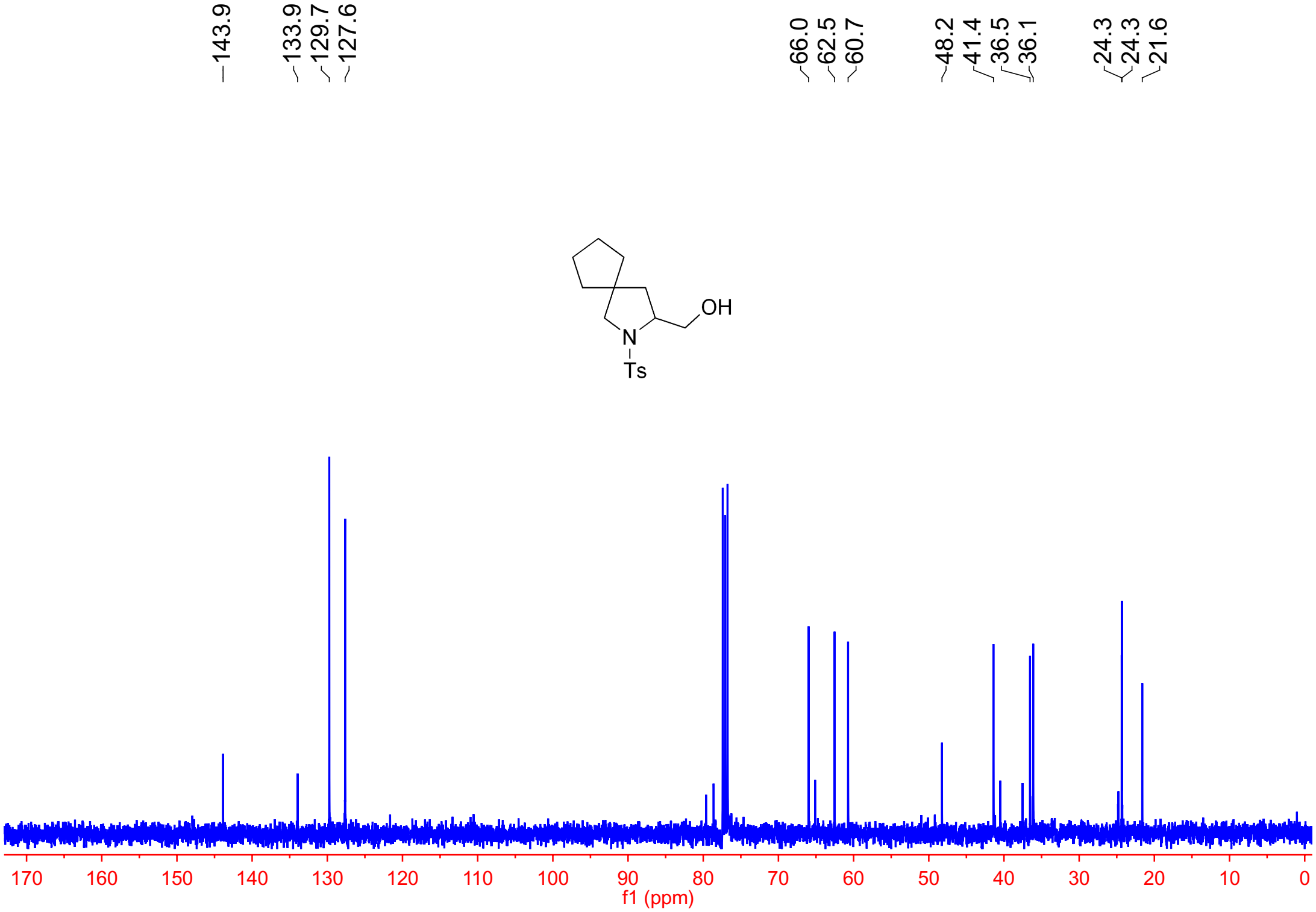
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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24.3
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21.6
OH
N
Ts
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100
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80
70
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50
40
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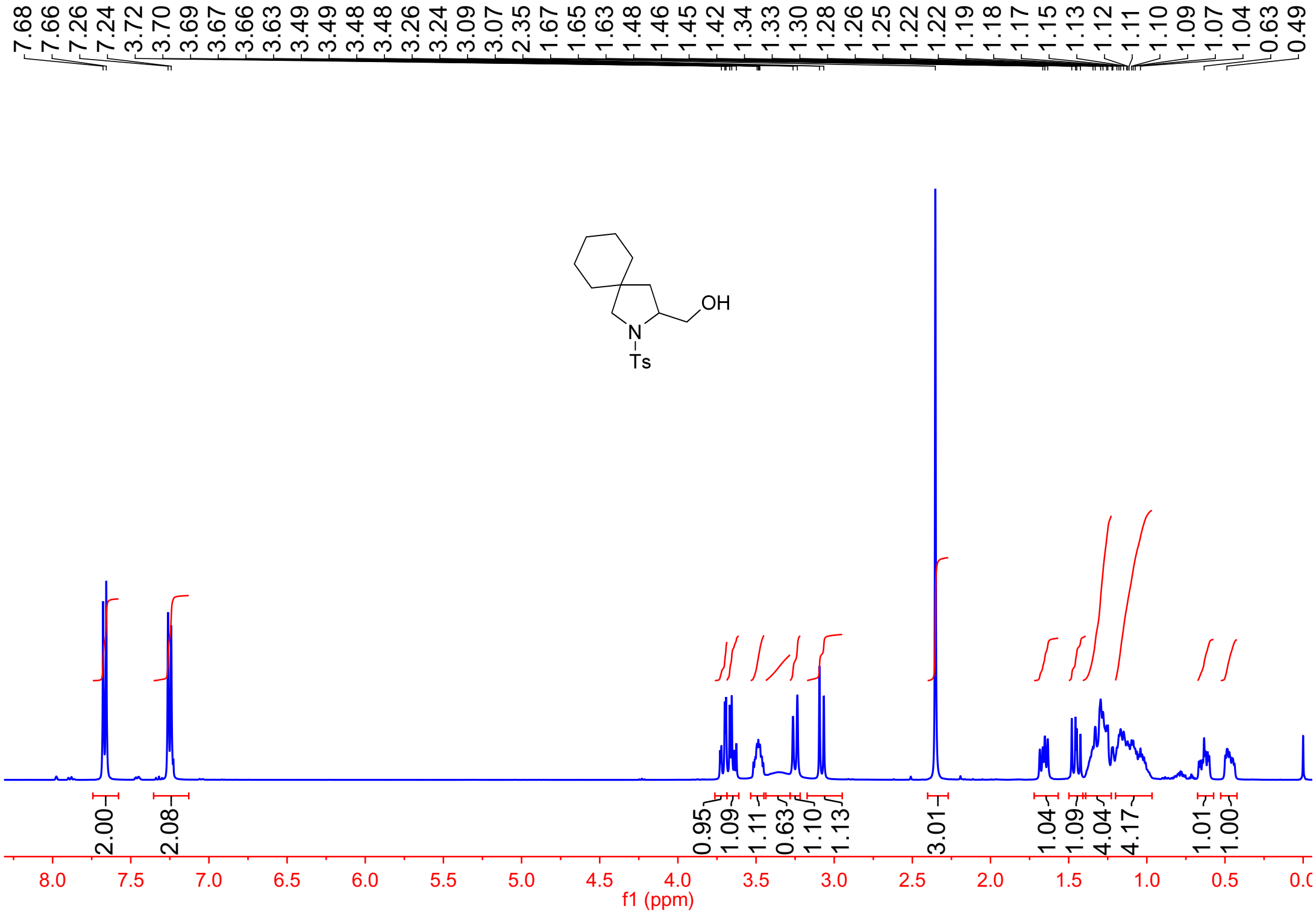
10
0
f1 (ppm)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3.49
3.49
3.48
3.48
3.26
3.24
3.09
3.07
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1.67
1.65
1.63
1.48
1.46
1.45
1.42
1.34
1.33
1.30
1.28
1.26
1.25
1.22
1.22
1.19
1.18
1.17
1.15
1.13
1.12
1.11
1.10
1.09
1.07
1.04
0.63
0.49
OH
N
Ts
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2.08
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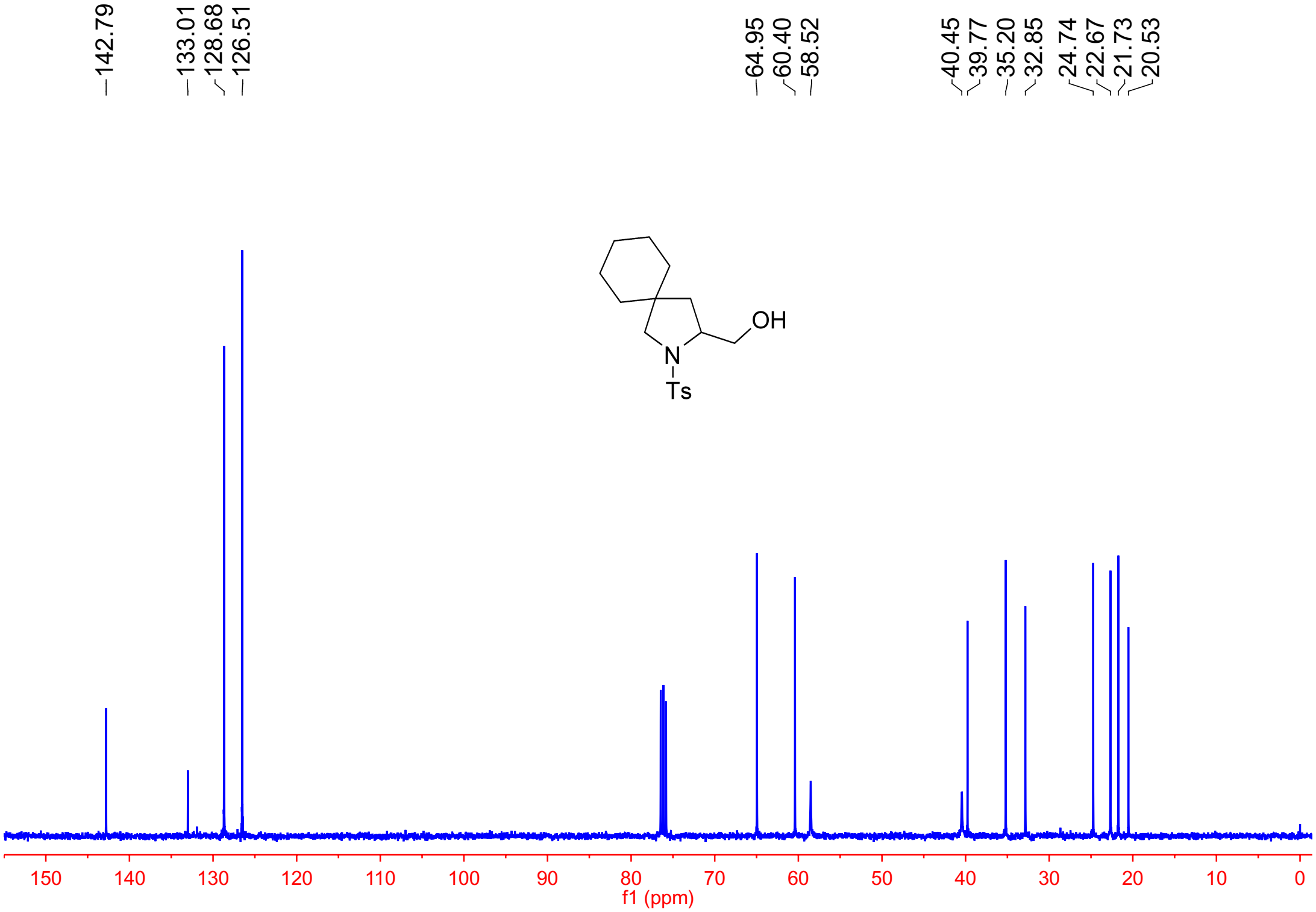
1.09
1.11
0.63
1.10
1.13
3.01
1.04
1.09
4.04
4.17
1.01
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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32.85
24.74
22.67
21.73
20.53
OH
N
Ts
150
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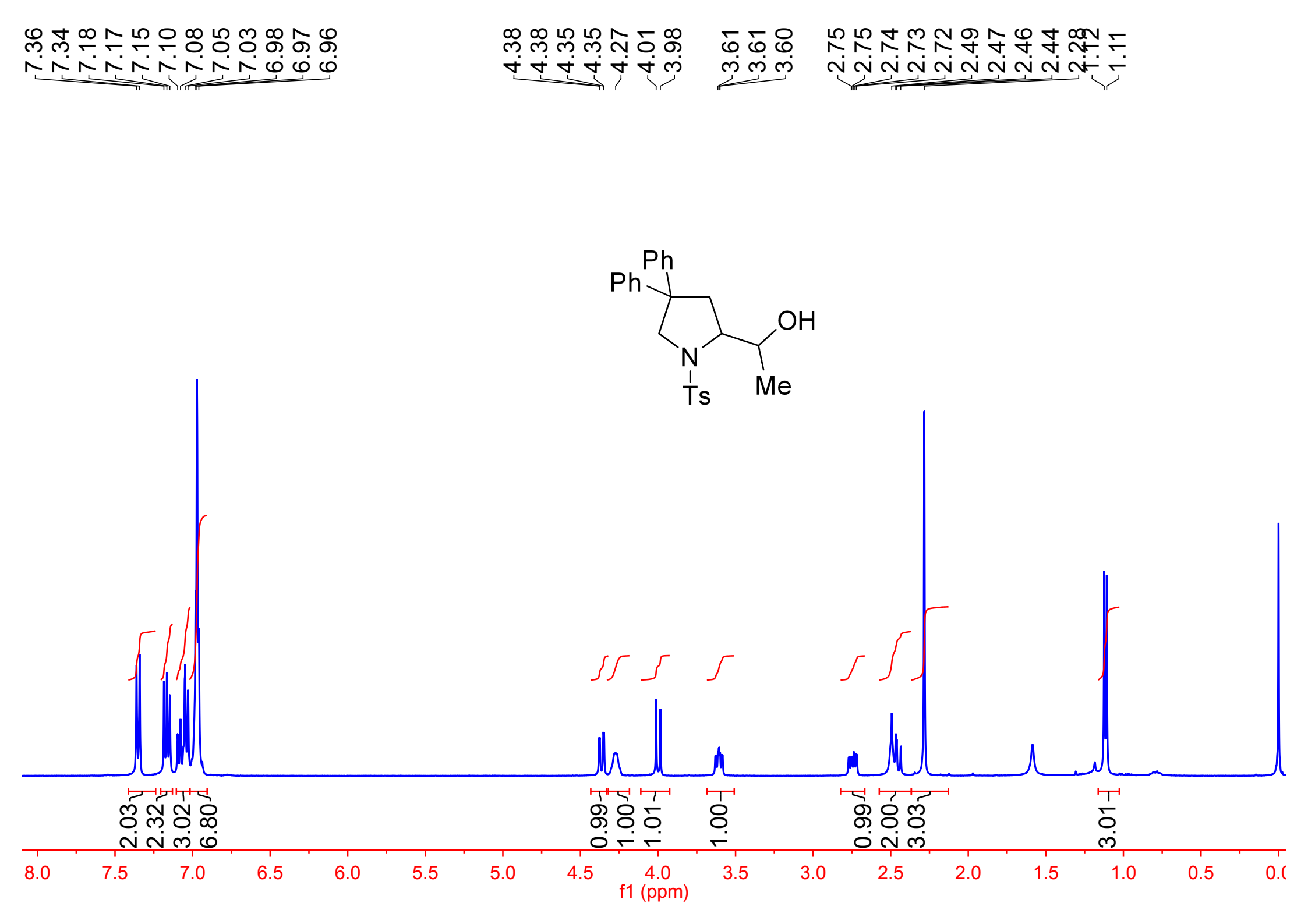
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.97
6.96
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4.38
4.35
4.35
4.27
4.01
3.98
3.61
3.61
3.60
2.75
2.75
2.74
2.73
2.72
2.49
2.47
2.46
2.44
2.28
1.12
1.11
Ph
Ph
OH
N
Ts
Me
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2.32
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1.01
1.00
0.99
2.00
3.03
3.01
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7.5
7.0
6.5
6.0
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5.0
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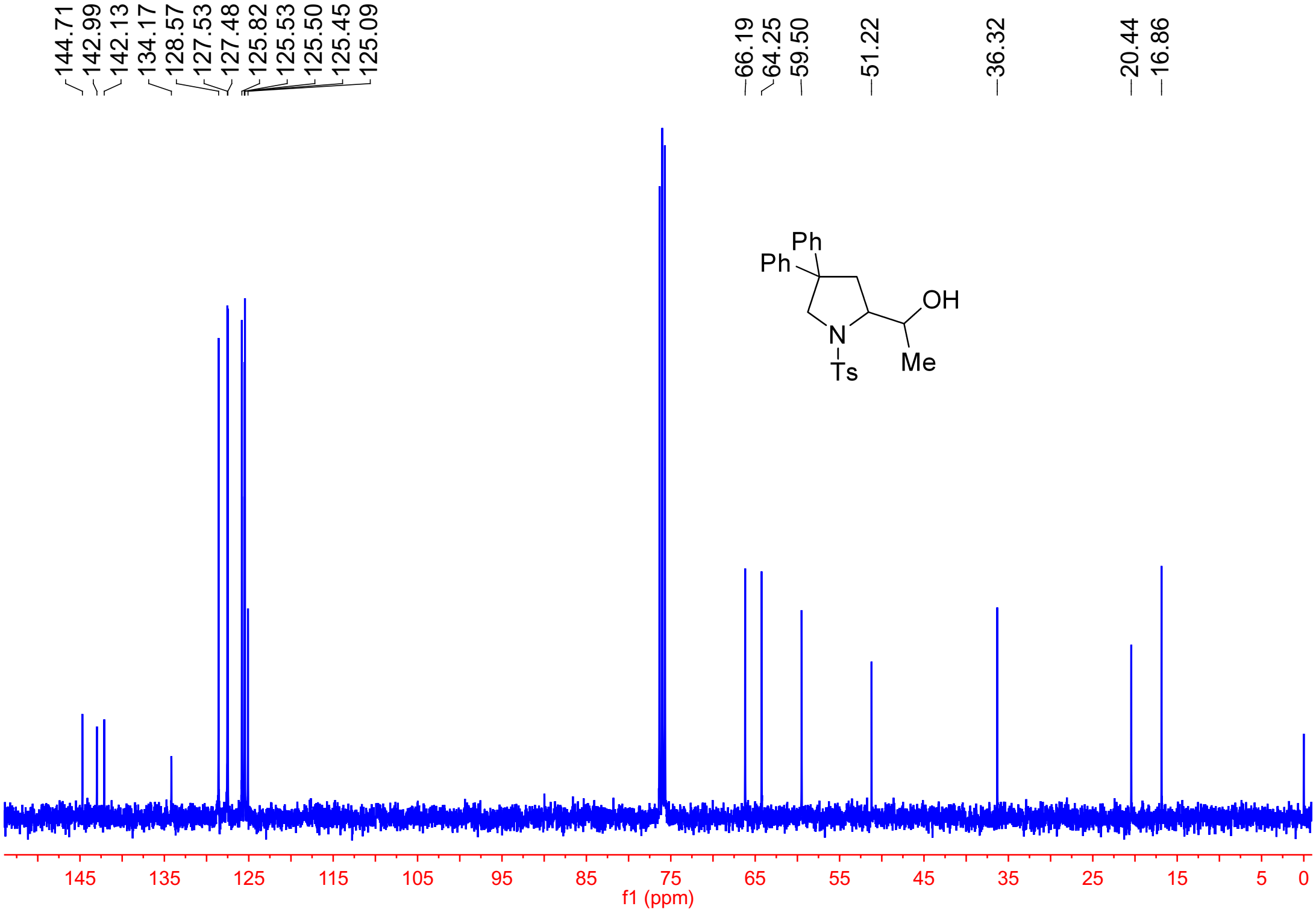
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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125.50
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64.25
59.50
51.22
36.32
20.44
16.86
Ph
Ph
OH
N
Ts
Me
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125
115
105
95
85
75
65
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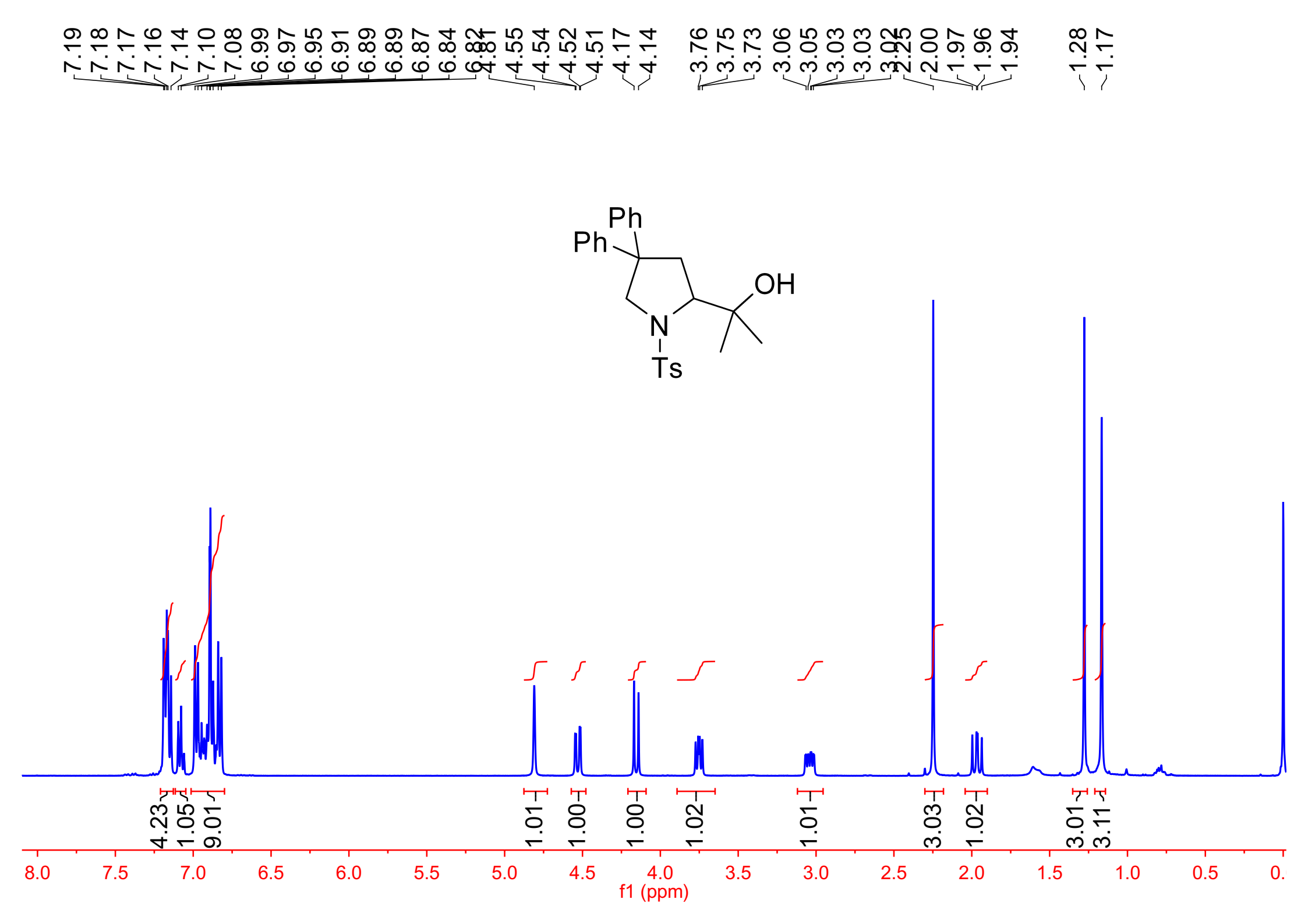
45
35
25
15
5
0
f1 (ppm)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6.84
6.82
6.81
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4.54
4.52
4.51
4.17
4.14
3.76
3.75
3.73
3.06
3.05
3.03
3.03
3.02
2.25
2.00
1.97
1.96
1.94
1.28
1.17
Ph
Ph
OH
N
Ts
4.23
1.05
9.01
1.01
1.00
1.00
1.02
1.01
3.03
1.02
3.01
3.11
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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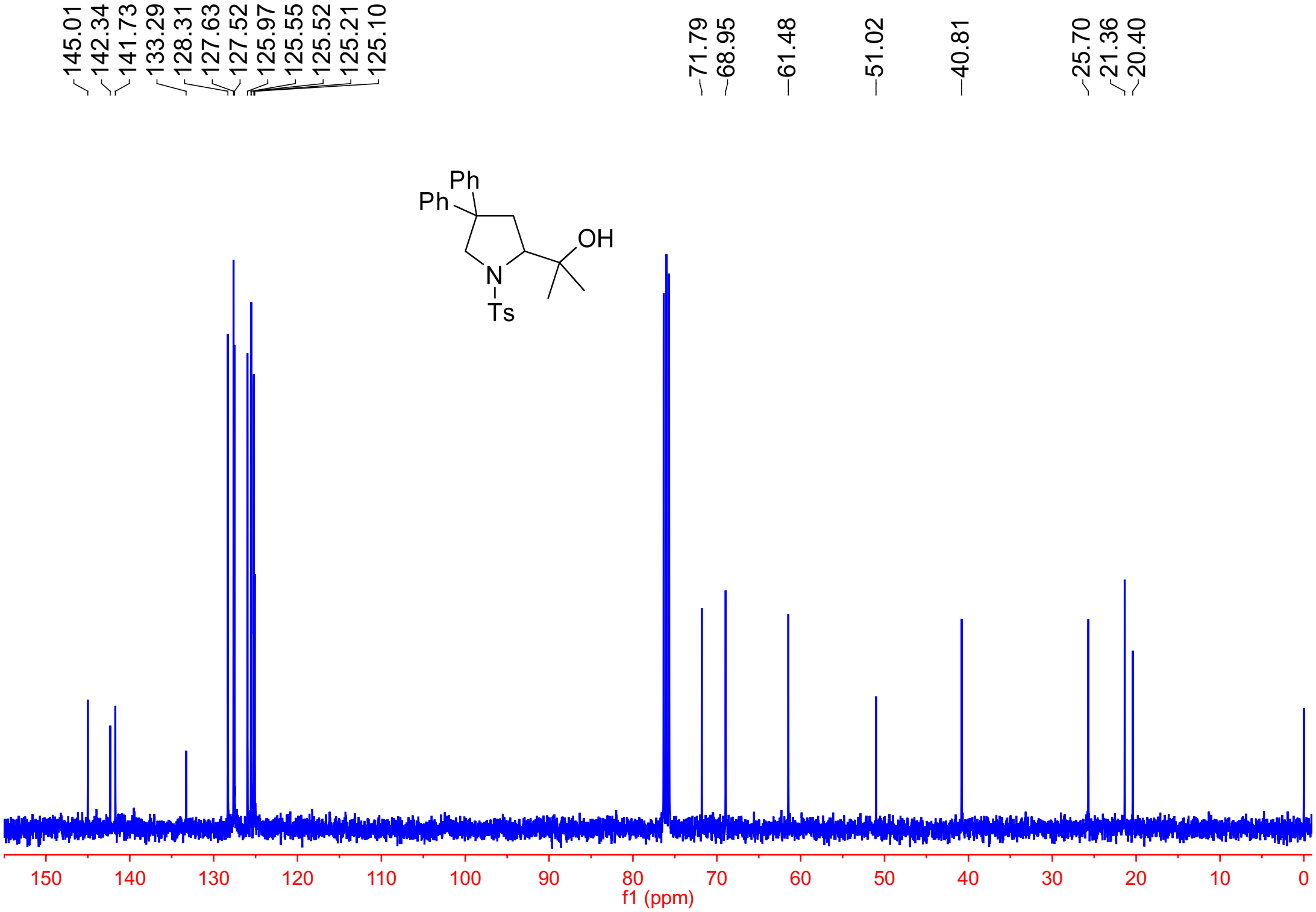
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)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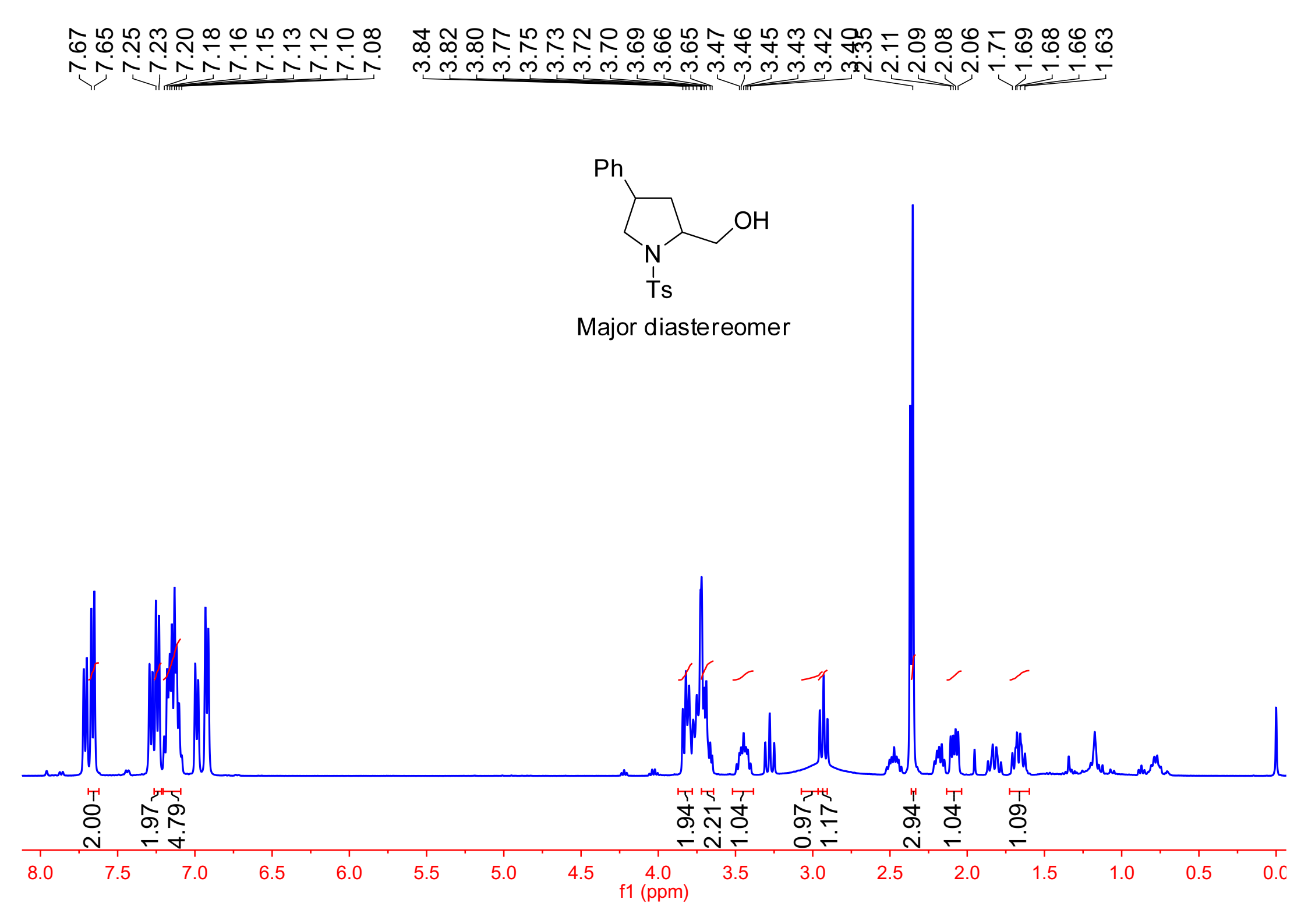
125.52
125.21
125.10
71.79
68.95
61.48
51.02
40.81
25.70
21.36
20.40
Ph
Ph
OH
N
Ts
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.08
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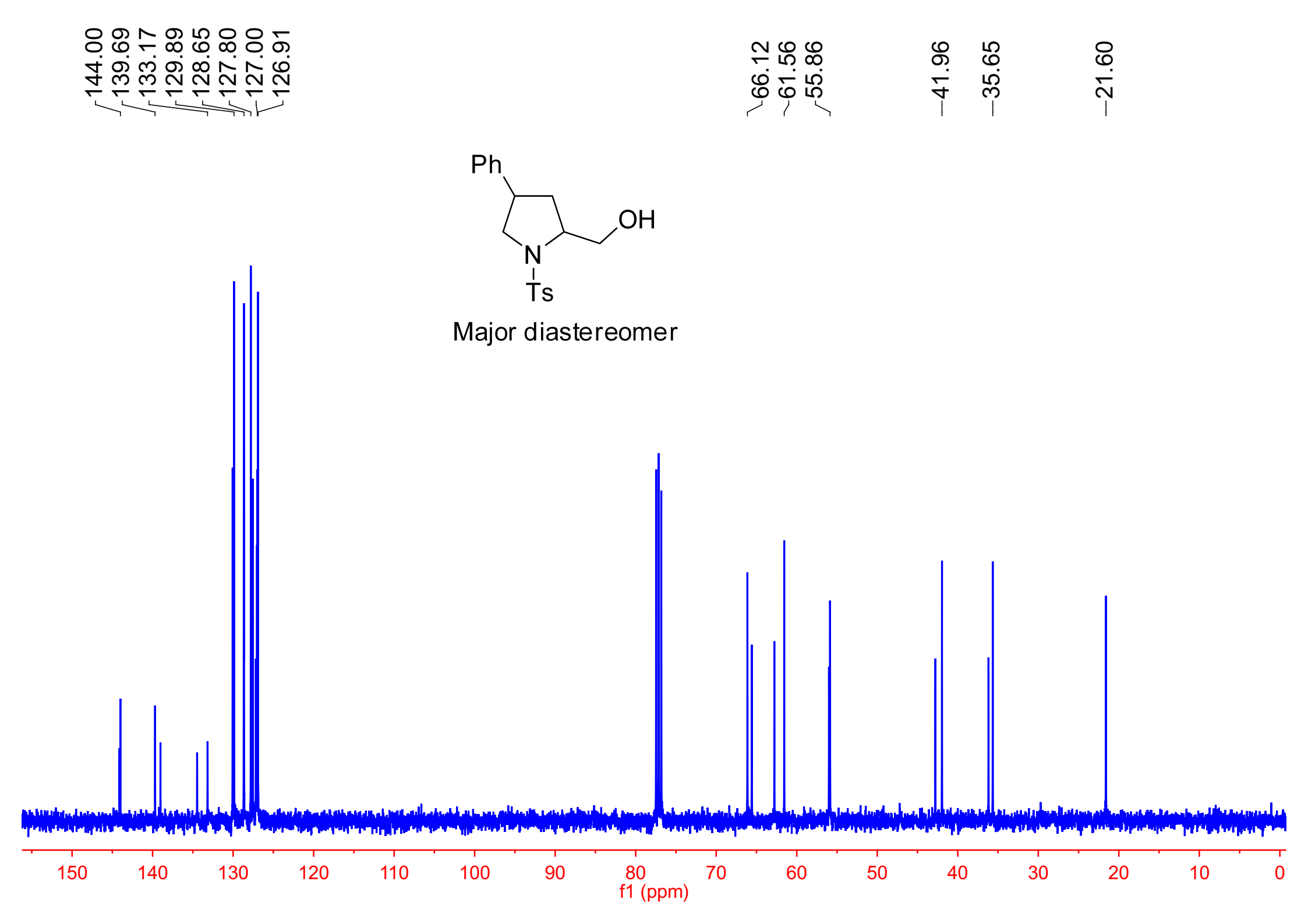
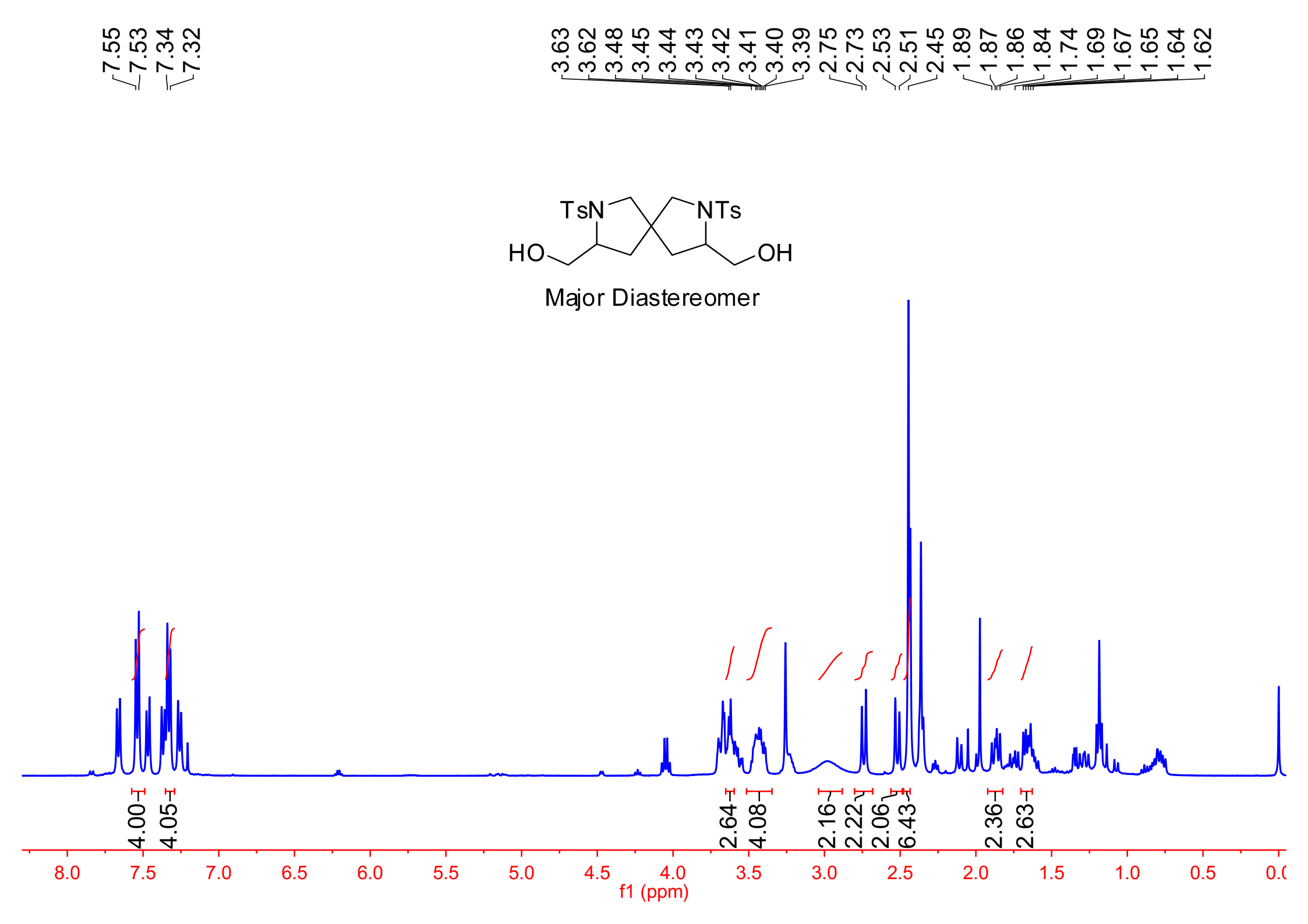
3.82
3.80
3.77
3.75
3.73
3.72
3.70
3.69
3.66
3.65
3.47
3.46
3.45
3.43
3.42
3.40
2.35
2.11
2.09
2.08
2.06
1.71
1.69
1.68
1.66
1.63
Ph
OH
N
Ts
Major diastereomer
2.00
1.97
4.79
1.94
2.21
1.04
0.97
1.17
2.94
1.04
1.09
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


144.00
139.69
133.17
129.89
128.65
127.80
127.00
126.91
66.12
61.56
55.86
41.96
35.65
21.60
Ph
OH
N
Ts
Major diastereomer
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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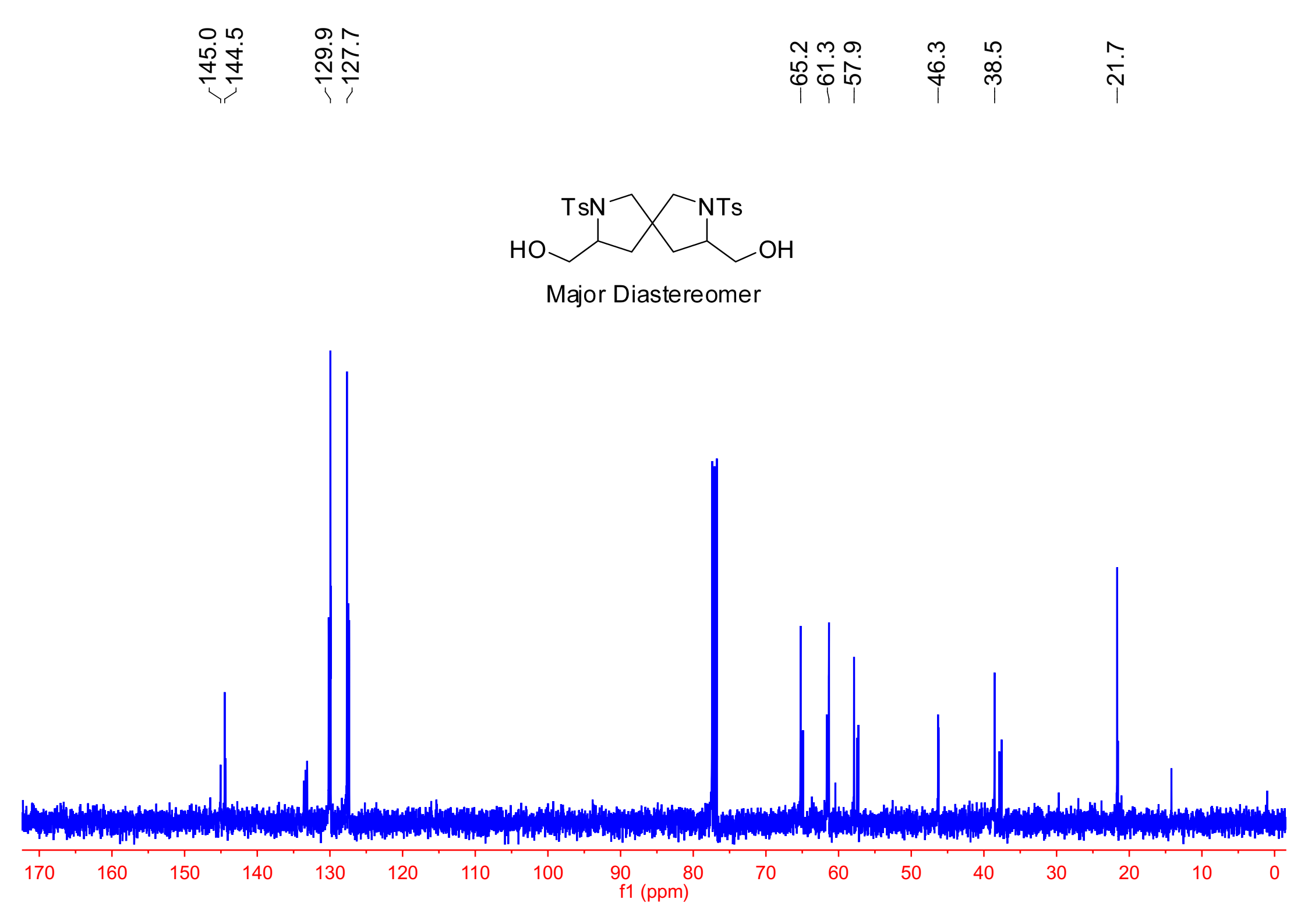
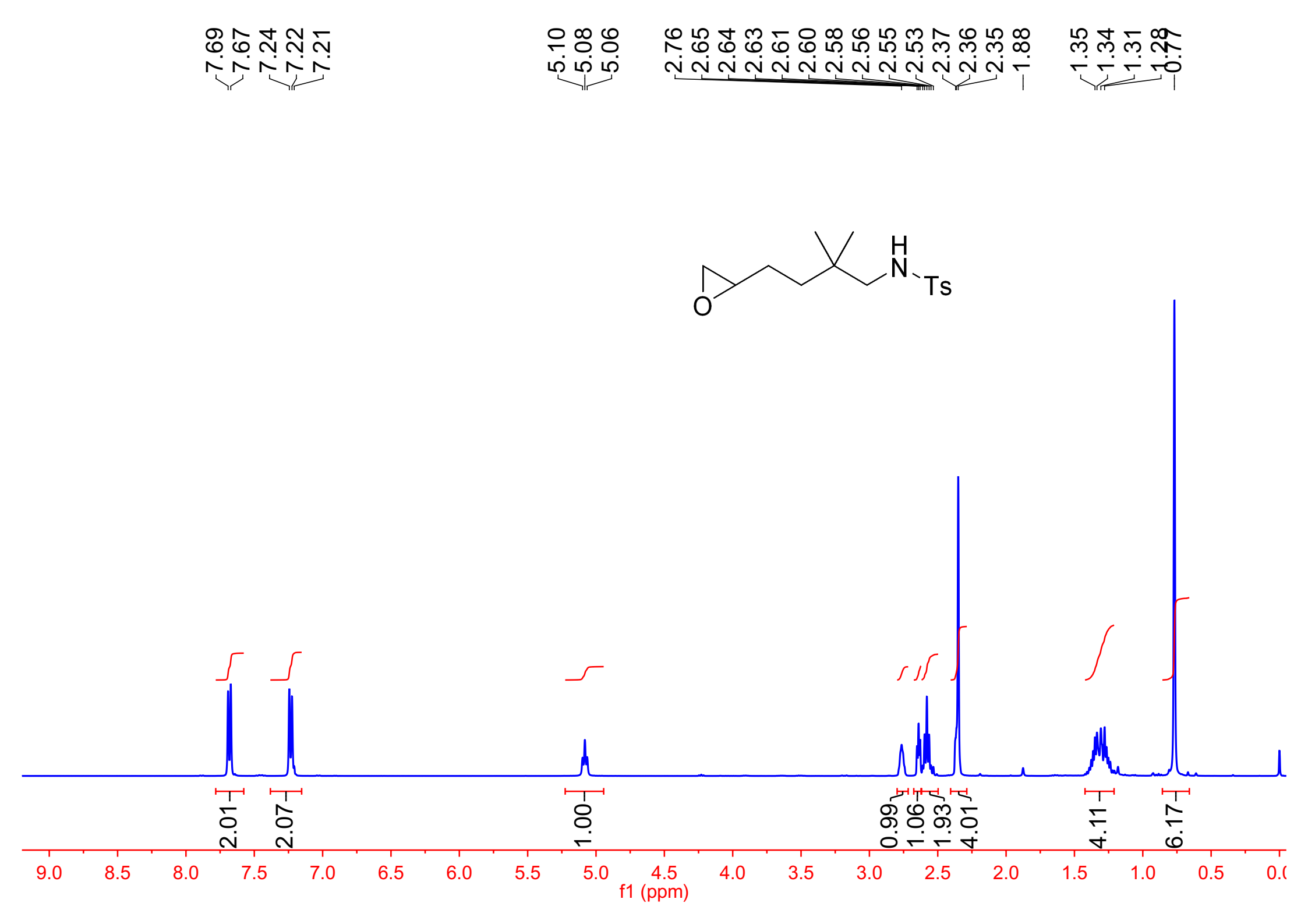
7.53
7.34
7.32
3.63
3.62
3.48
3.45
3.44
3.43
3.42
3.41
3.40
3.39
2.75
2.73
2.53
2.51
2.45
1.89
1.87
1.86
1.84
1.74
1.69
1.67
1.65
1.64
1.62
TsN
NTs
HO
OH
Major Diastereomer
4.00
4.05
2.64
4.08
2.16
2.22
2.06
6.43
2.36
2.63
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


145.0
144.5
129.9
127.7
65.2
61.3
57.9
46.3
38.5
21.7
TsN
NTs
HO
OH
Major Diastereomer
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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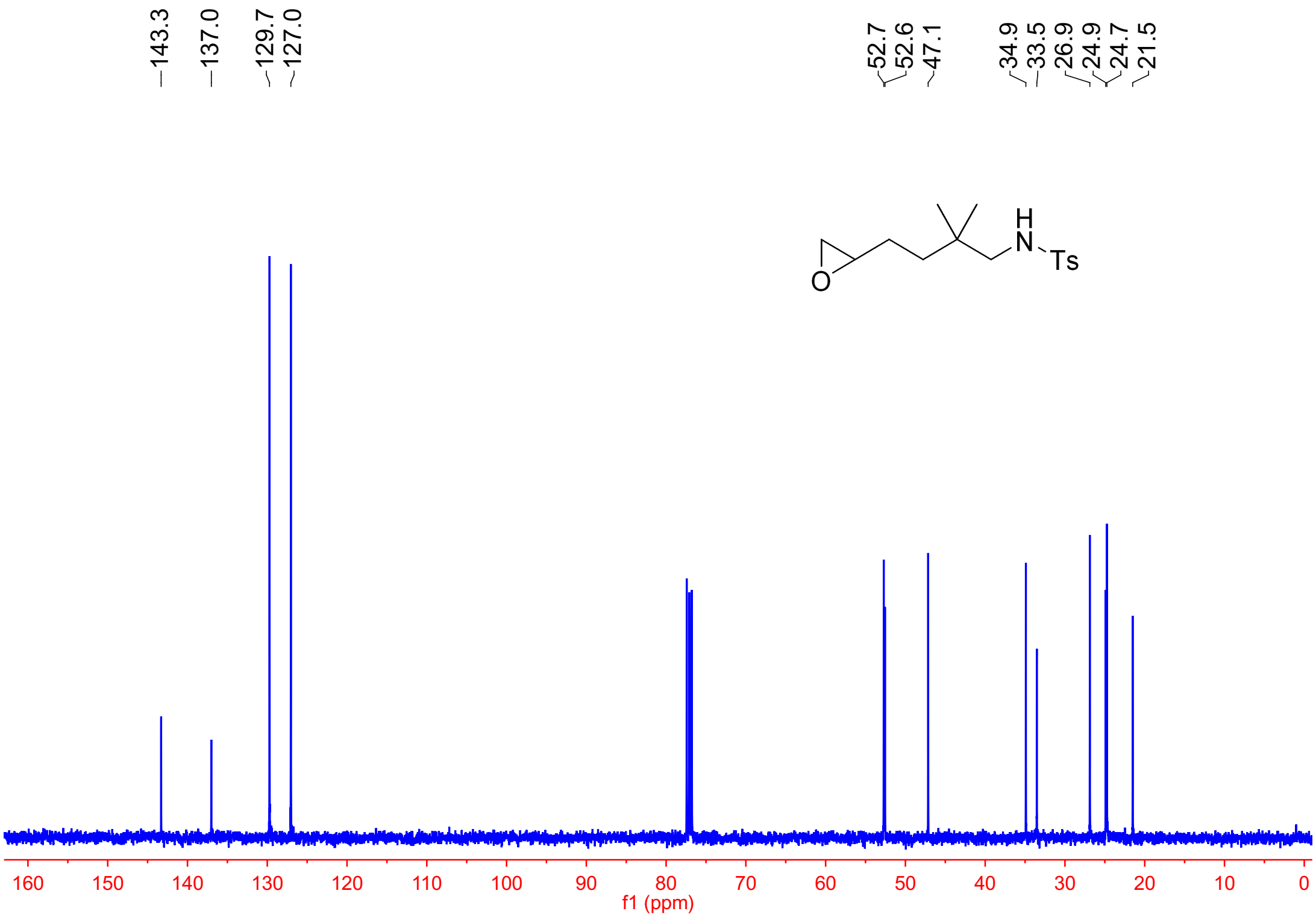
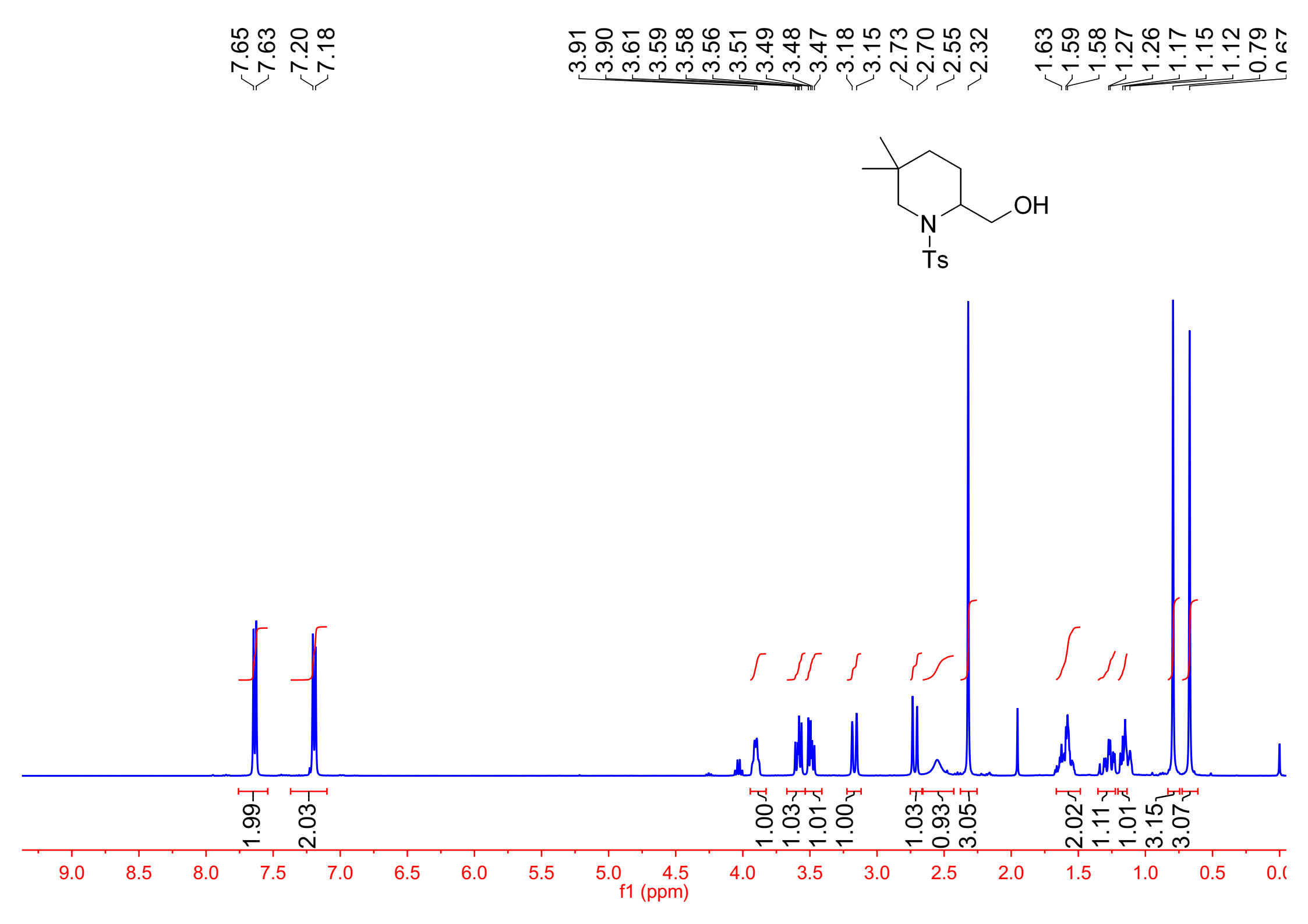
7.67
7.24
7.22
7.21
5.10
5.08
5.06
2.76
2.65
2.64
2.63
2.61
2.60
2.58
2.56
2.55
2.53
2.37
2.36
2.35
1.88
1.35
1.34
1.31
1.28
0.77
O
H
N
Ts
2.01
2.07
1.00
0.99
1.06
1.93
4.01
4.11
6.17
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


143.3
137.0
129.7
127.0
52.7
52.6
47.1
34.9
33.5
26.9
24.9
24.7
21.5
O
H
N
Ts
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.65
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7.20
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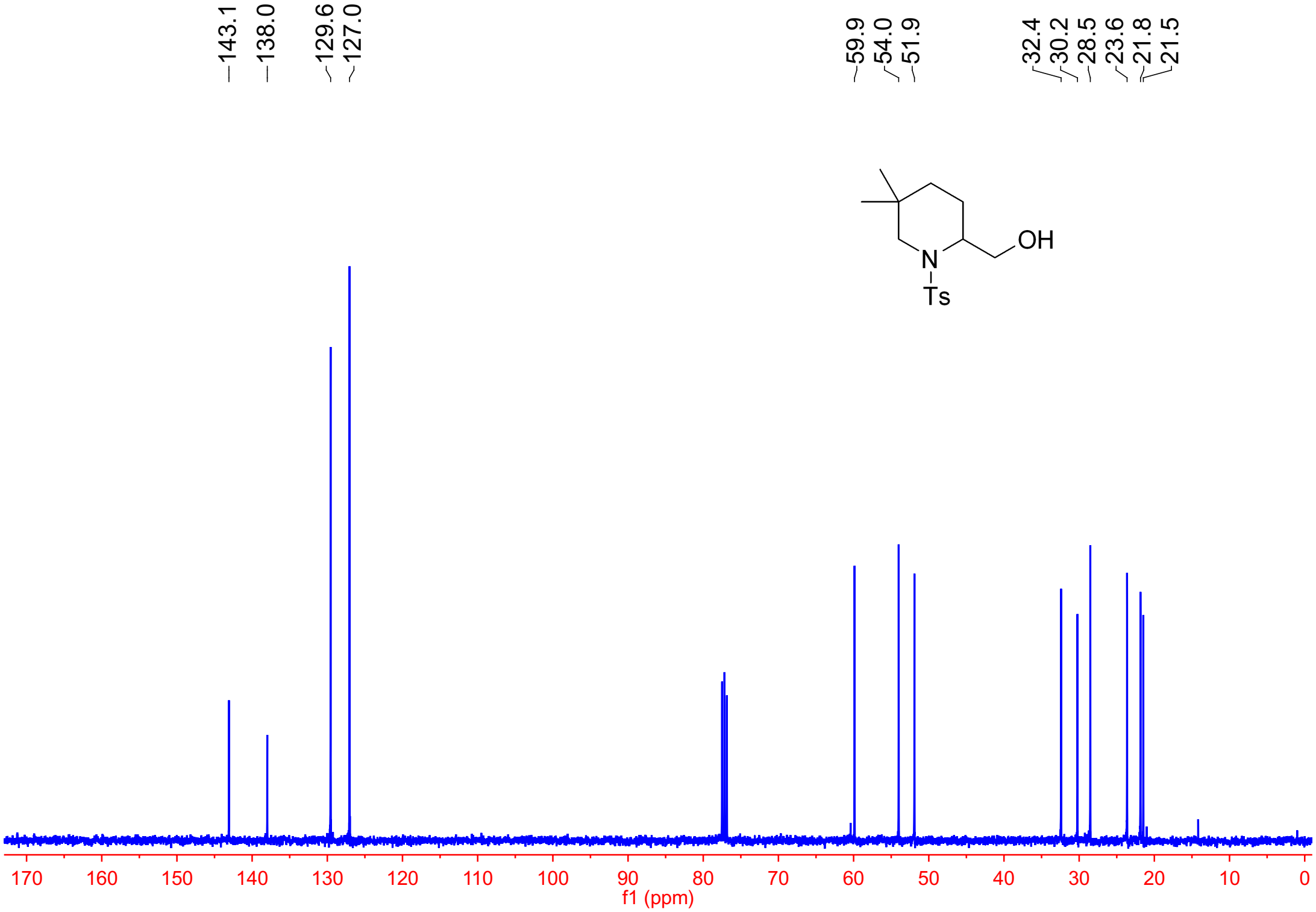
3.90
3.61
3.59
3.58
3.56
3.51
3.49
3.48
3.47
3.18
3.15
2.73
2.70
2.55
2.32
1.63
1.59
1.58
1.27
1.26
1.17
1.15
1.12
0.79
OH
N
Ts
1.99
2.03
1.00
1.03
1.01
1.00
1.03
0.93
3.05
2.02
1.11
1.01
3.15
3.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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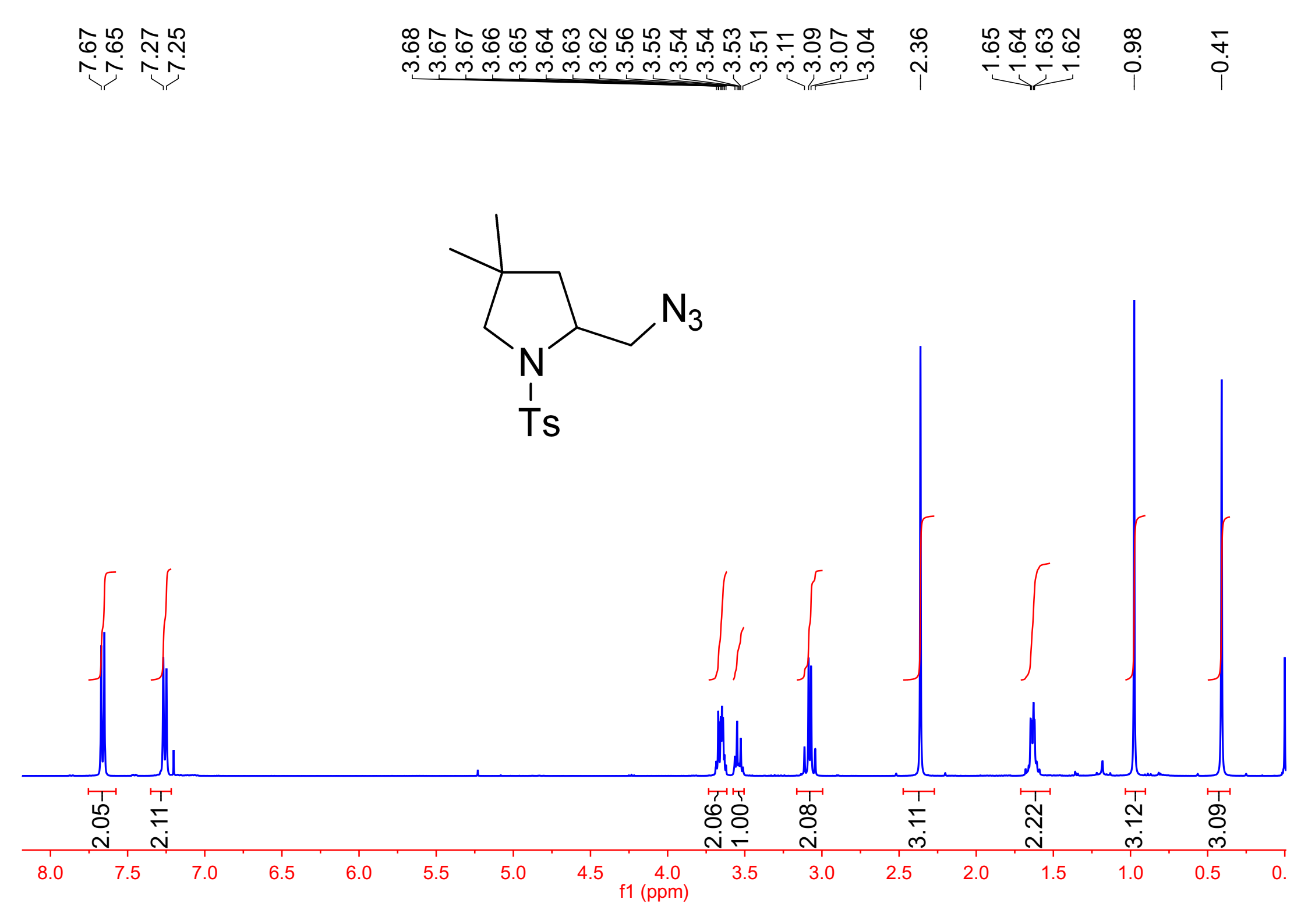
138.0
129.6
127.0
59.9
54.0
51.9
32.4
30.2
28.5
23.6
21.8
21.5
OH
N
Ts
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.67
7.65
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3.68
3.67
3.67
3.66
3.65
3.64
3.63
3.62
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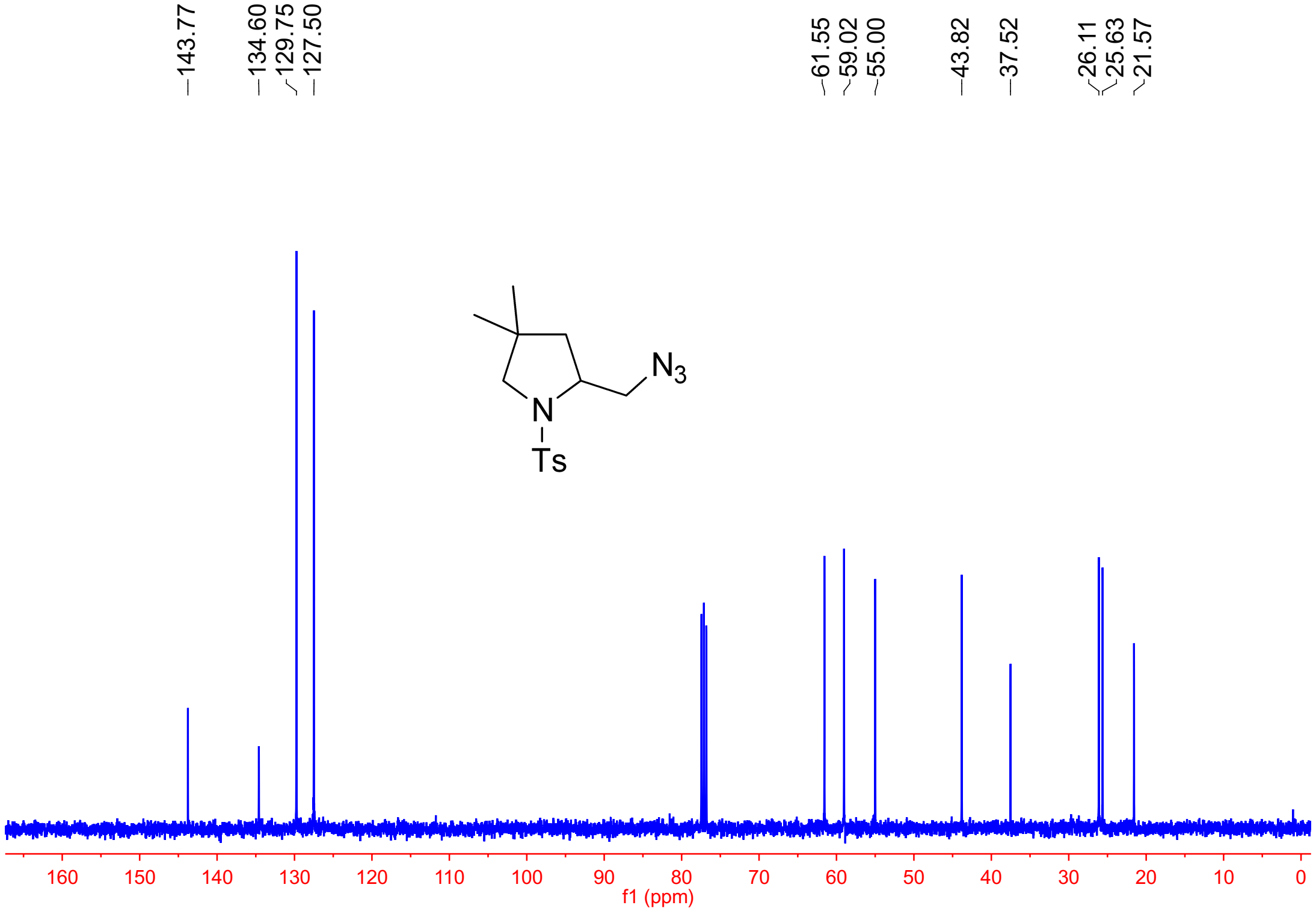
3.55
3.54
3.54
3.53
3.51
3.11
3.09
3.07
3.04
2.36
1.65
1.64
1.63
1.62
0.98
0.41
N3
N
Ts
2.05
2.11
2.06
1.00
2.08
3.11
2.22
3.12
3.09
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)


143.77
134.60
129.75
127.50
61.55
59.02
55.00
43.82
37.52
26.11
25.63
21.57
N3
N
Ts
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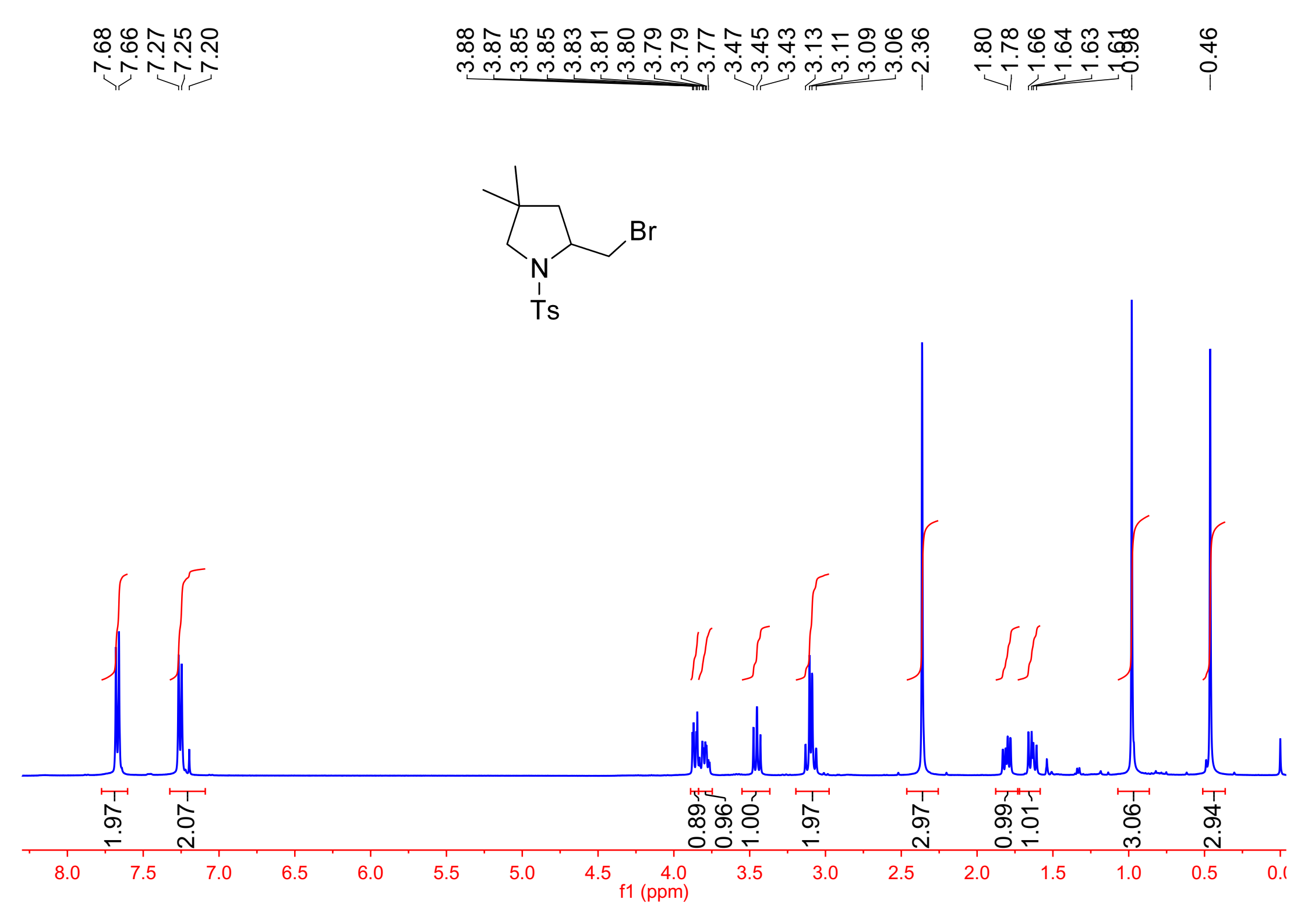
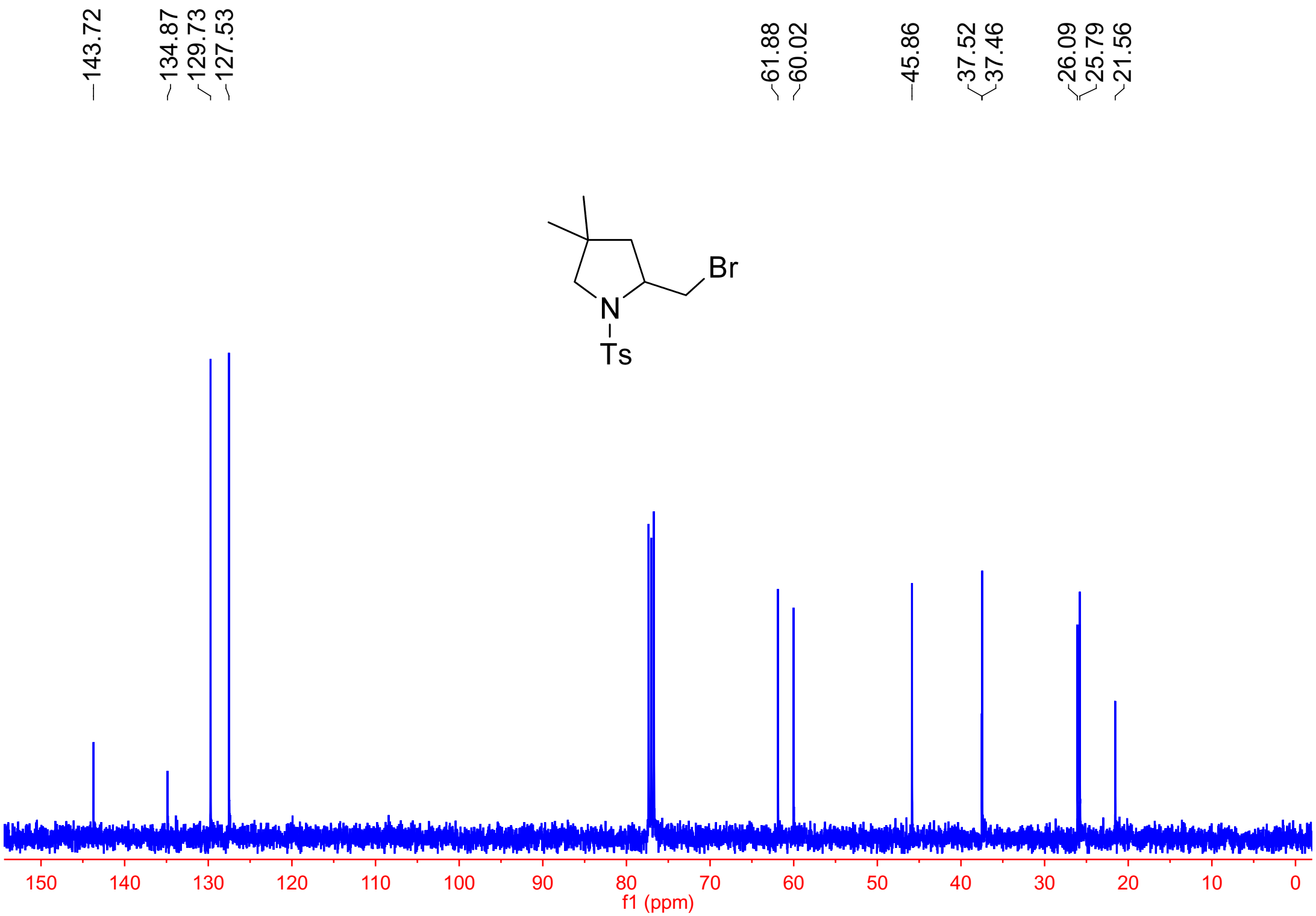
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.68
7.66
7.27
7.25
7.20
3.88
3.87
3.85
3.85
3.83
3.81
3.80
3.79
3.79
3.77
3.47
3.45
3.43
3.13
3.11
3.09
3.06
2.36
1.80
1.78
1.66
1.64
1.63
1.61
0.98
0.46
Br
N
Ts
1.97
2.07
0.89
0.96
1.00
1.97
2.97
0.99
1.01
3.06
2.94
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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134.87
129.73
127.53
61.88
60.02
45.86
37.52
37.46
26.09
25.79
21.56
Br
N
Ts
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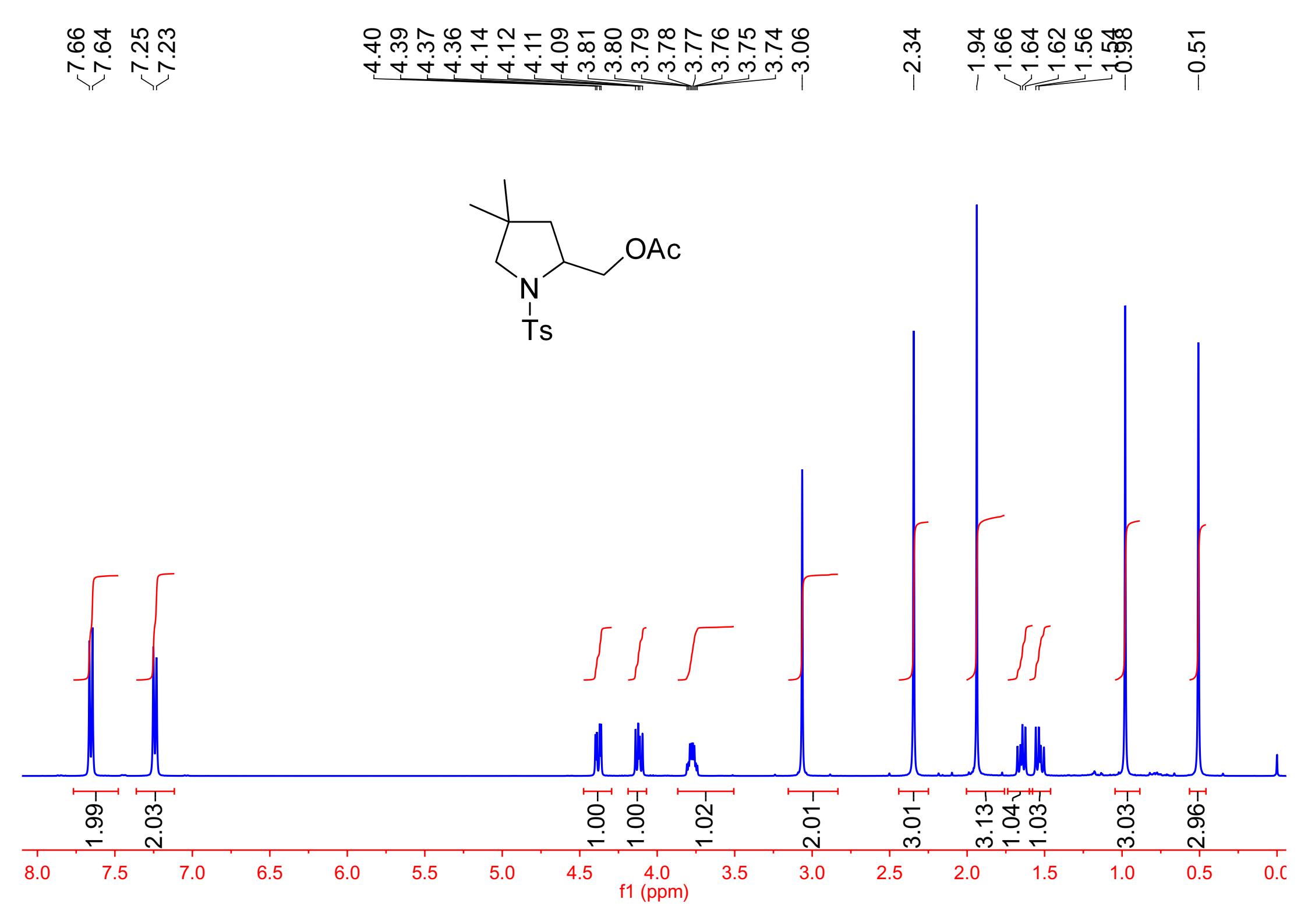
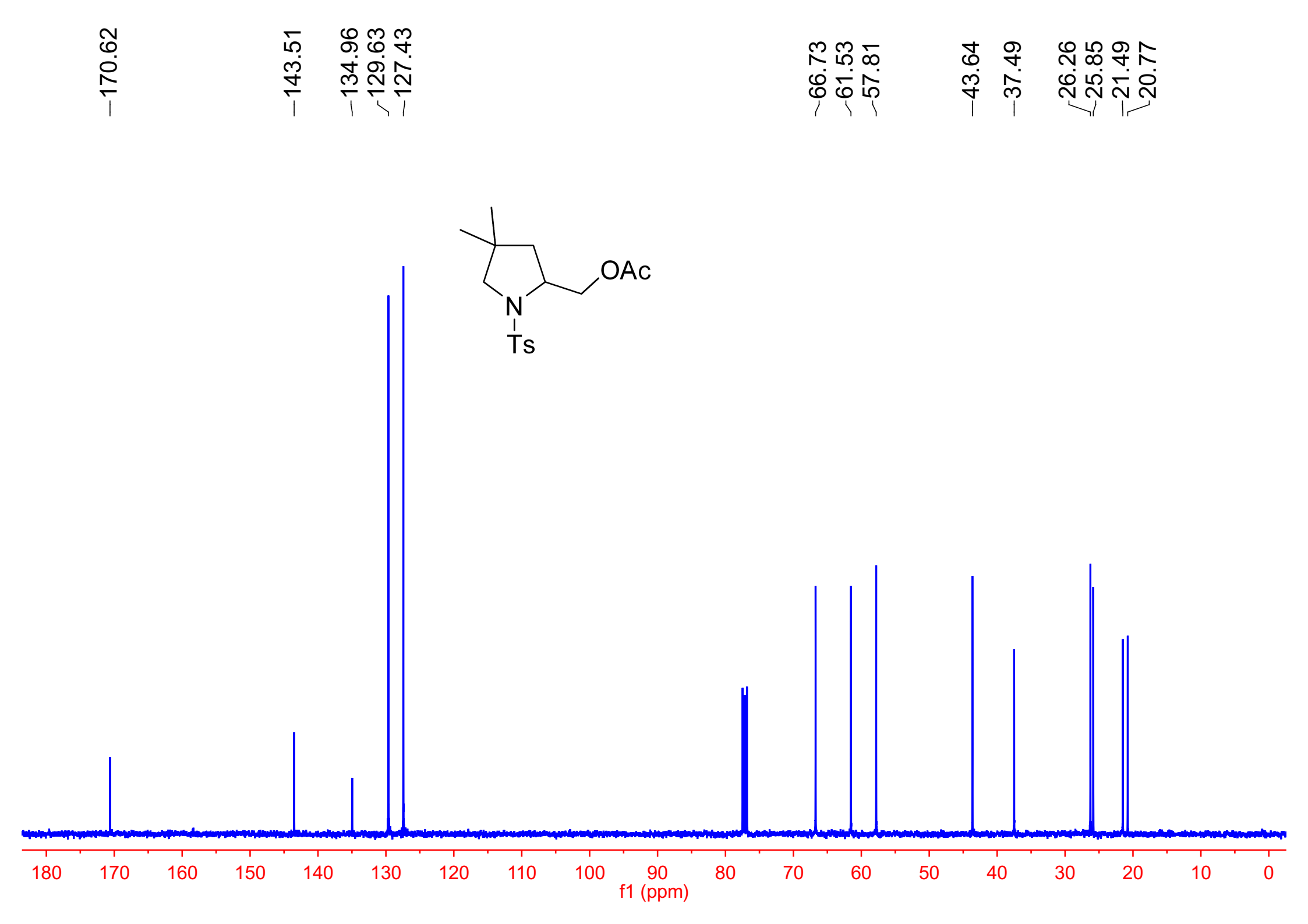
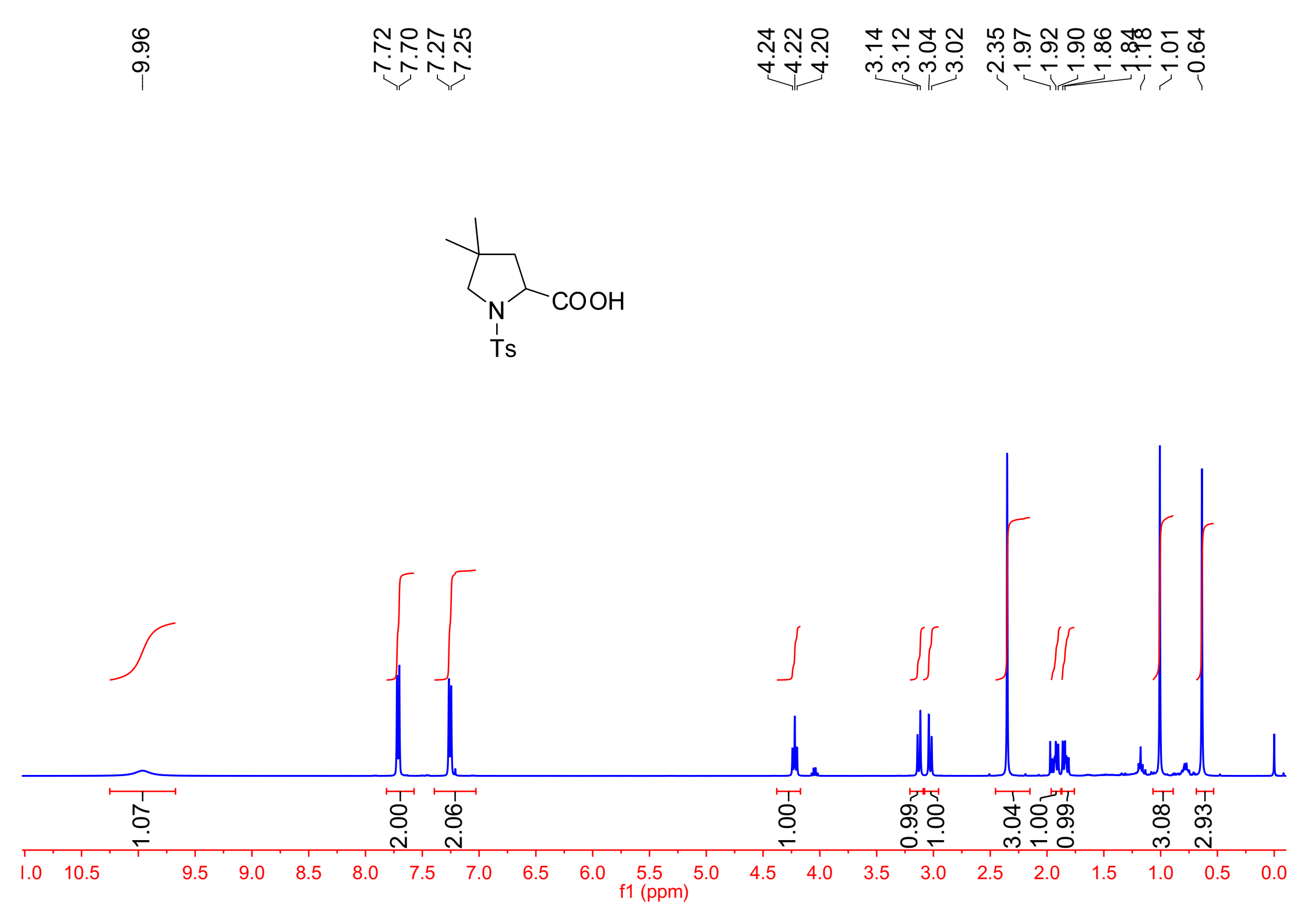
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.66
7.64
7.25
7.23
4.40
4.39
4.37
4.36
4.14
4.12
4.11
4.09
3.81
3.80
3.79
3.78
3.77
3.76
3.75
3.74
3.06
2.34
1.94
1.66
1.64
1.62
1.56
1.54
0.98
0.51
OAc
N
Ts
1.99
2.03
1.00
1.00
1.02
2.01
3.01
3.13
1.04
1.03
3.03
2.96
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.62
143.51
134.96
129.63
127.43
66.73
61.53
57.81
43.64
37.49
26.26
25.85
21.49
20.77
OAc
N
Ts
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.96
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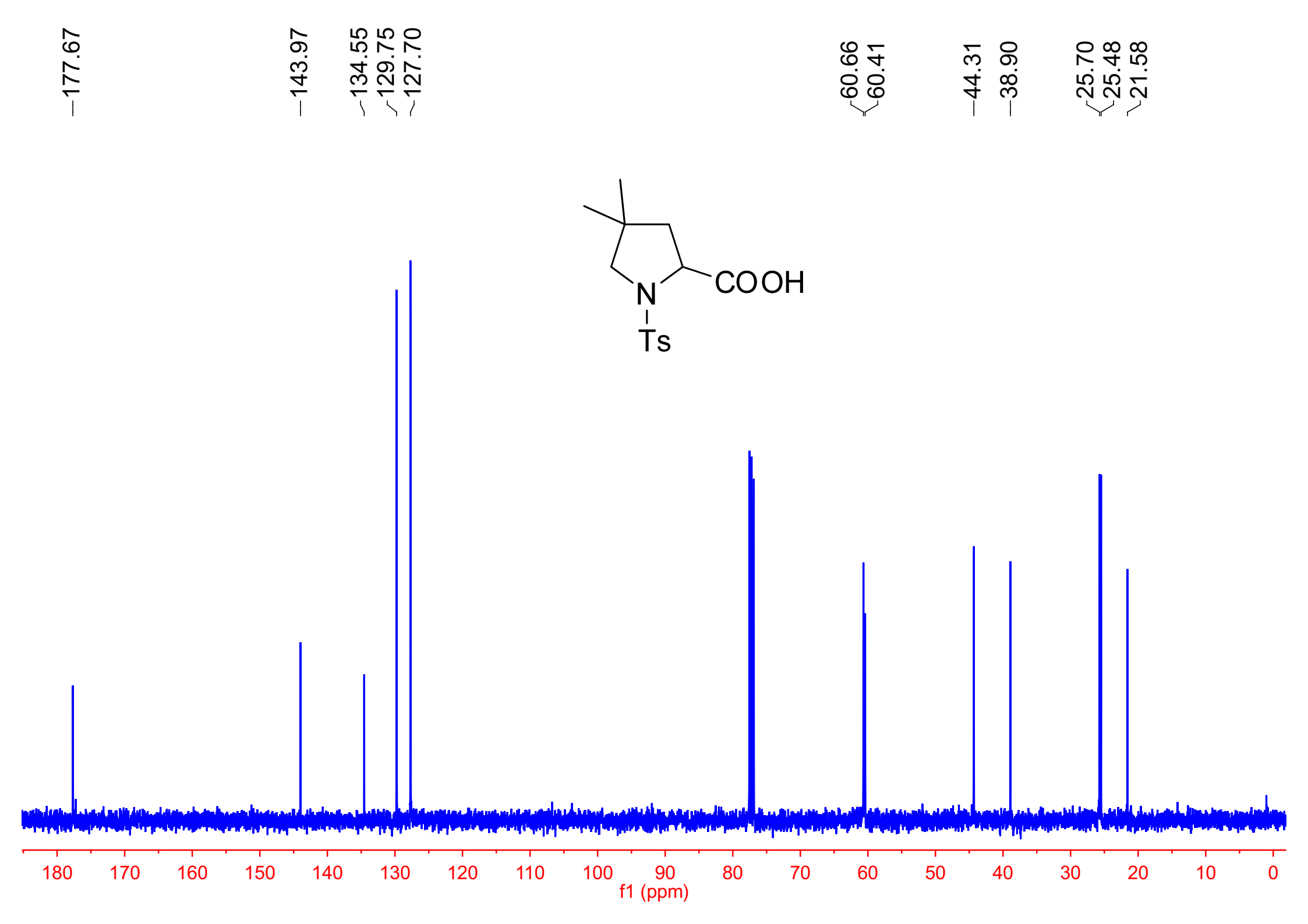
7.70
7.27
7.25
4.24
4.22
4.20
3.14
3.12
3.04
3.02
2.35
1.97
1.92
1.90
1.86
1.84
1.18
1.01
0.64
N
Ts
COOH
1.07
2.00
2.06
1.00
0.99
1.00
3.04
1.00
0.99
3.08
2.93
f1 (ppm)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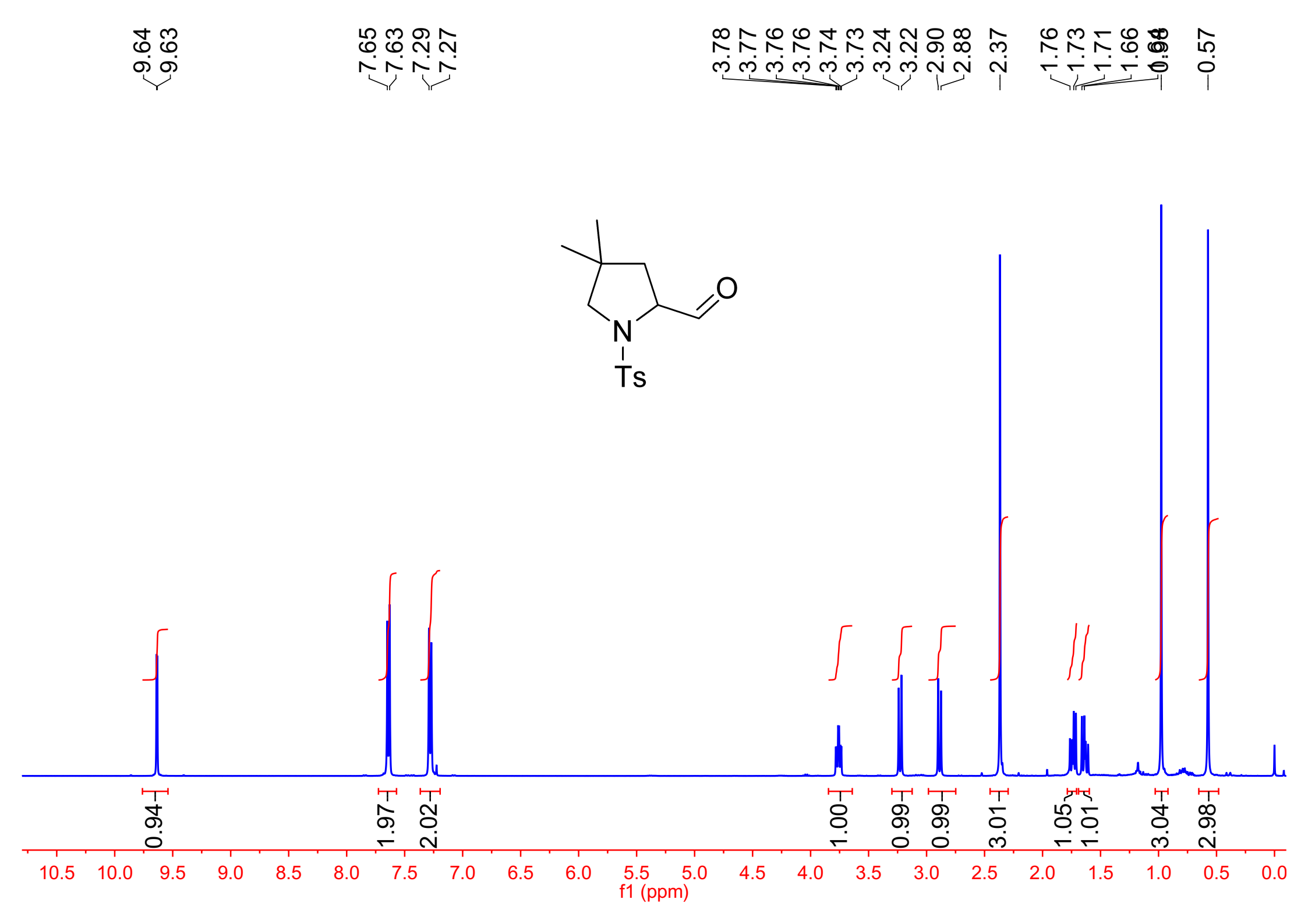
60.41
44.31
38.90
25.70
25.48
21.58
COOH
N
Ts
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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3.77
3.76
3.76
3.74
3.73
3.24
3.22
2.90
2.88
2.37
1.76
1.73
1.71
1.66
0.94
0.57
O
N
Ts
0.94
1.97
2.02
1.00
0.99
0.99
3.01
1.05
1.01
3.04
2.98
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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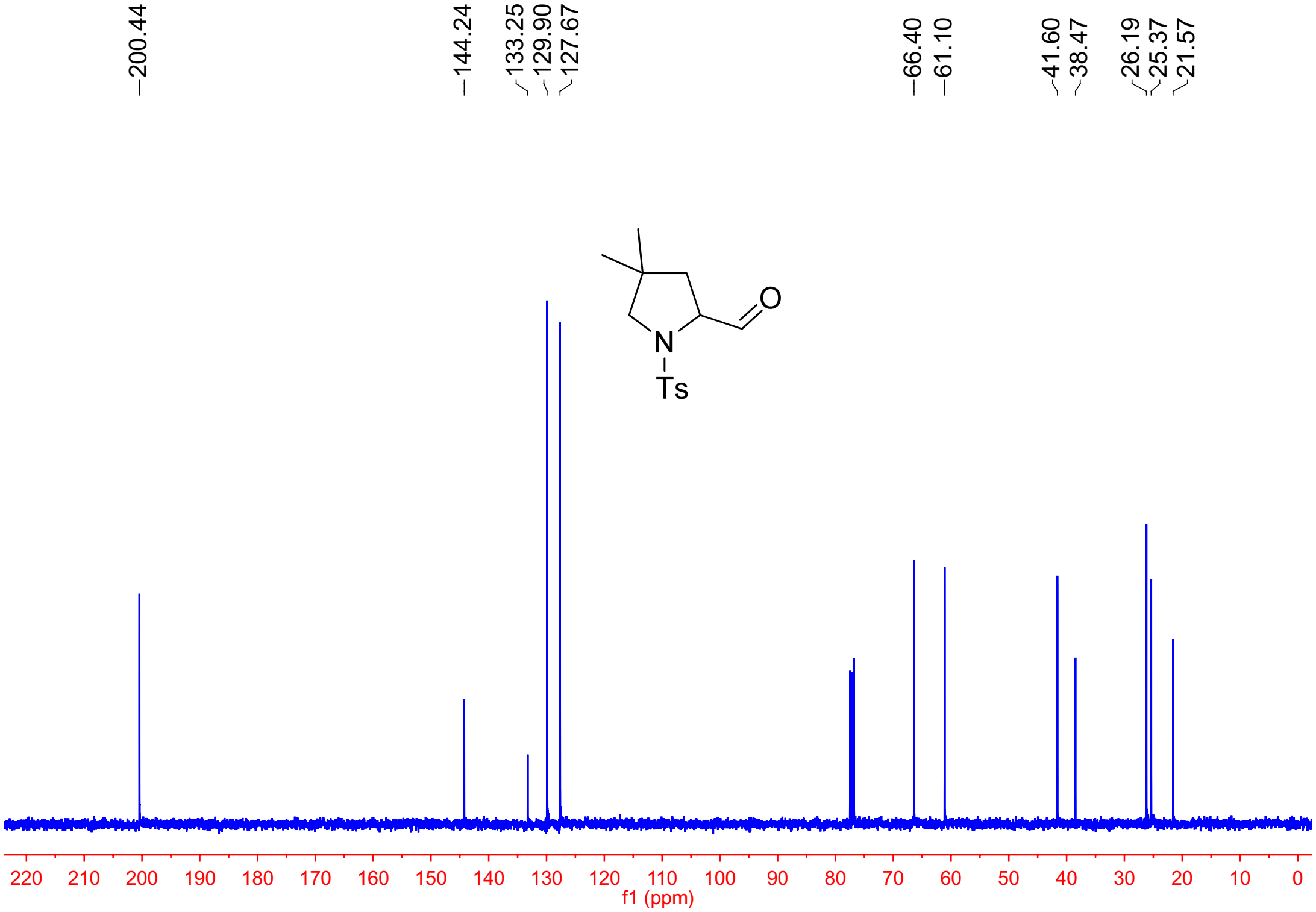
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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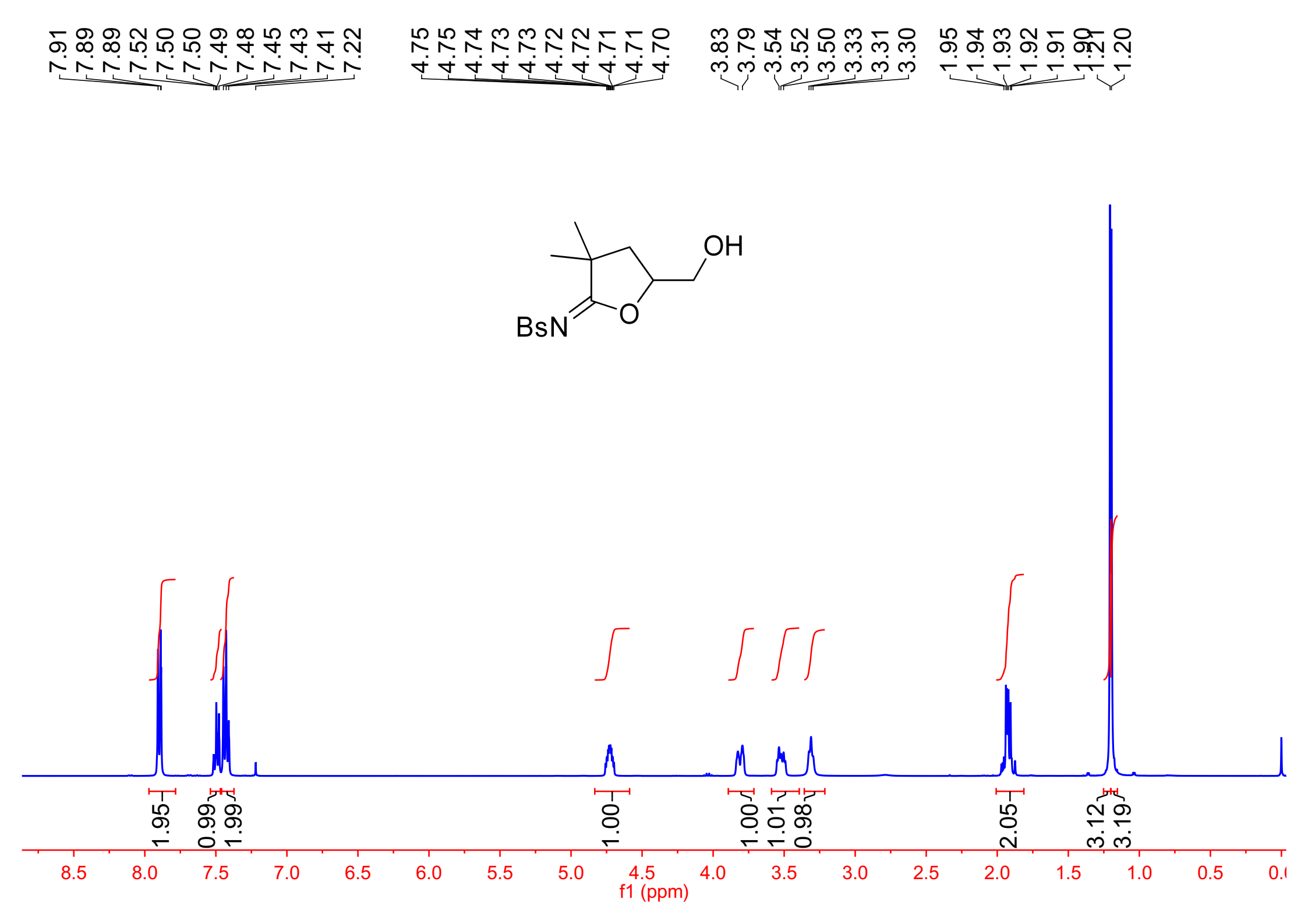
66.40
61.10
41.60
38.47
26.19
25.37
21.57
O
N
Ts
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.50
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7.48
7.45
7.43
7.41
7.22
4.75
4.75
4.74
4.73
4.73
4.72
4.72
4.71
4.71
4.70
3.83
3.79
3.54
3.52
3.50
3.33
3.31
3.30
1.95
1.94
1.93
1.92
1.91
1.90
1.21
1.20
OH
BsN
O
1.95
0.99
1.99
1.00
1.00
1.01
0.98
2.05
3.12
3.19
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.0
3.5
3.0
2.5
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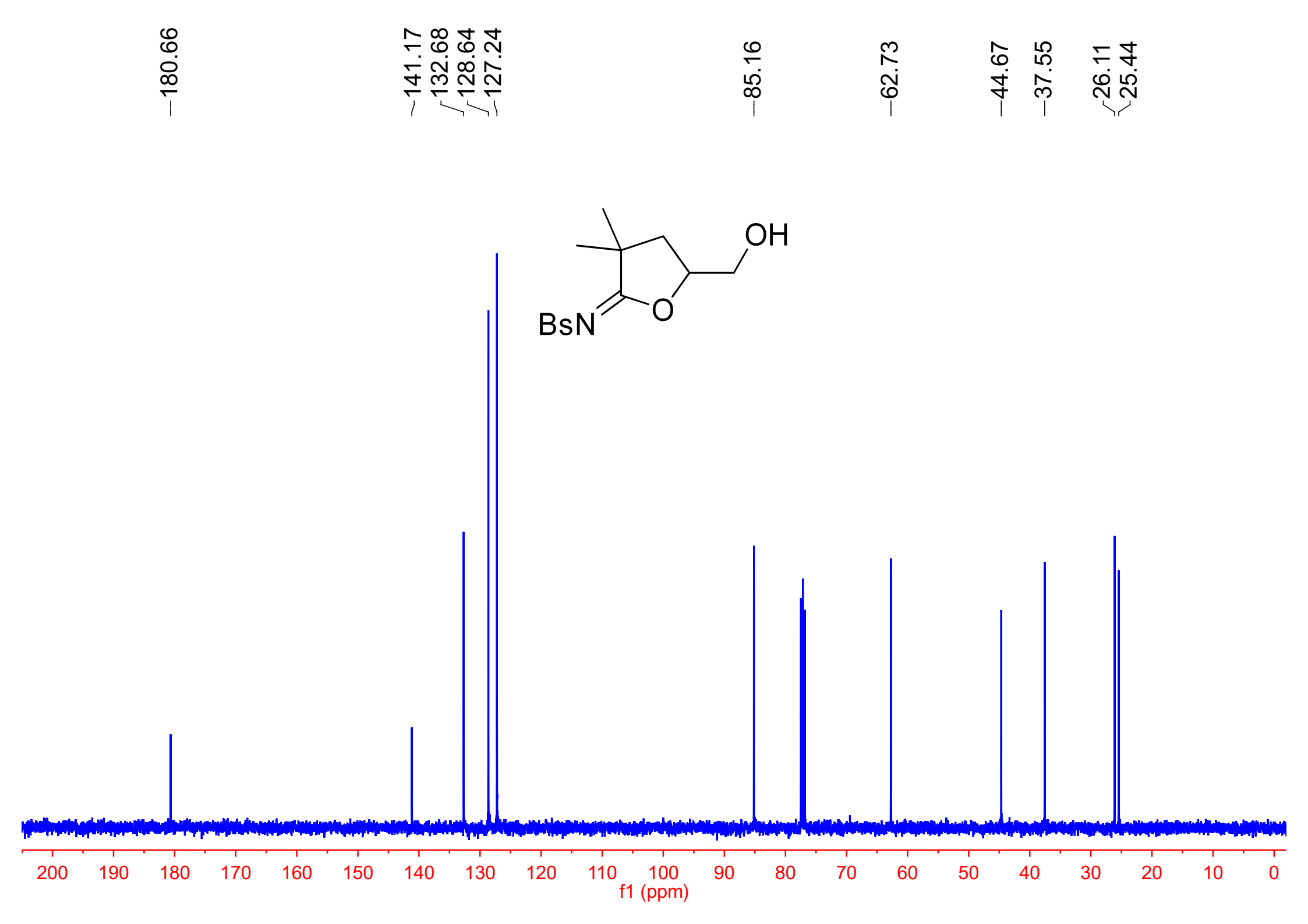
1.5
1.0
0.5
f1 (ppm)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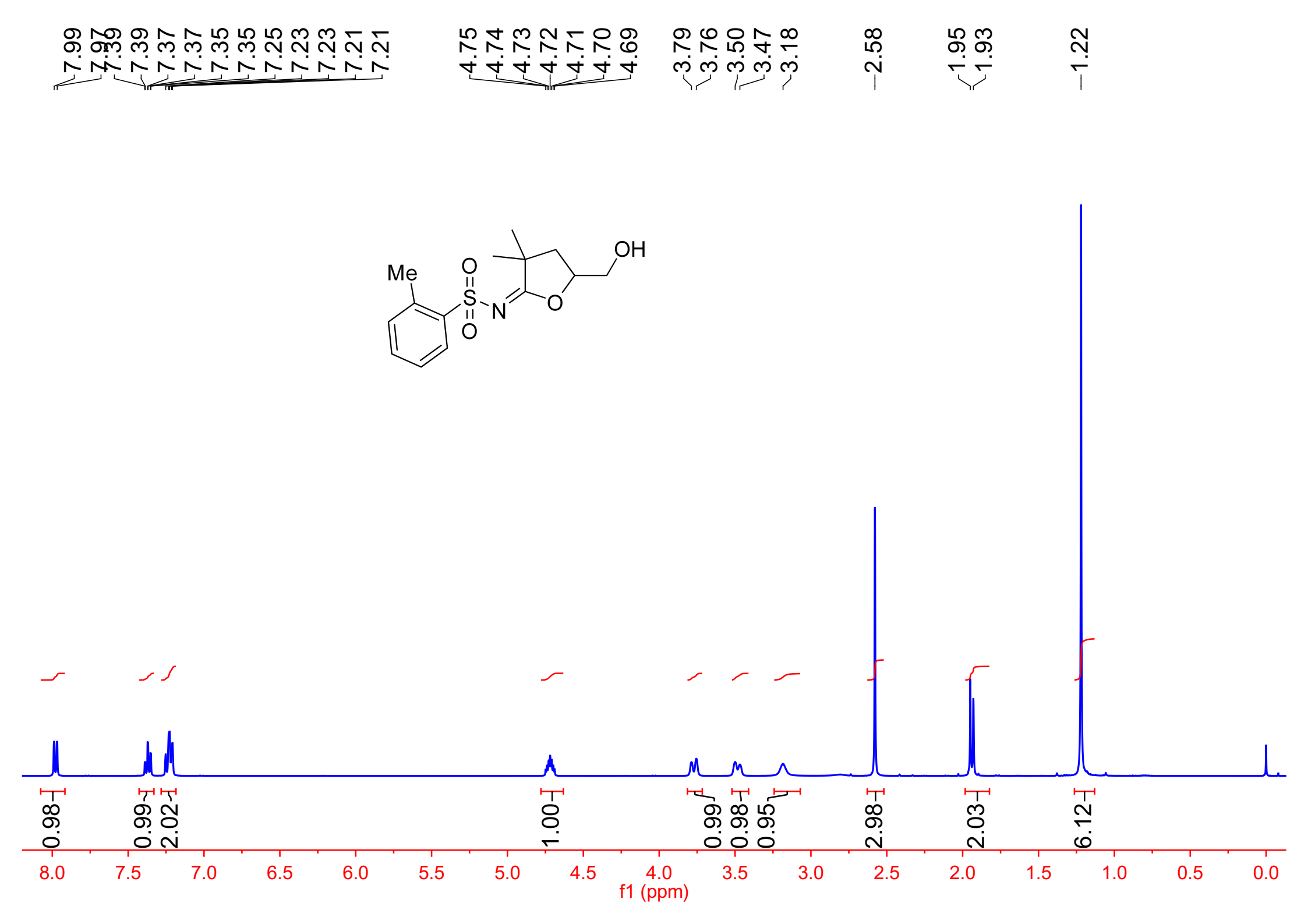
127.24
85.16
62.73
44.67
37.55
26.11
25.44
OH
O
BsN
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.35
7.35
7.25
7.23
7.23
7.21
7.21
4.75
4.74
4.73
4.72
4.71
4.70
4.69
3.79
3.76
3.50
3.47
3.18
2.58
1.95
1.93
1.22
Me
S
O
O
N
O
OH
0.98
0.99
2.02
1.00
0.99
0.98
0.95
2.98
2.03
6.12
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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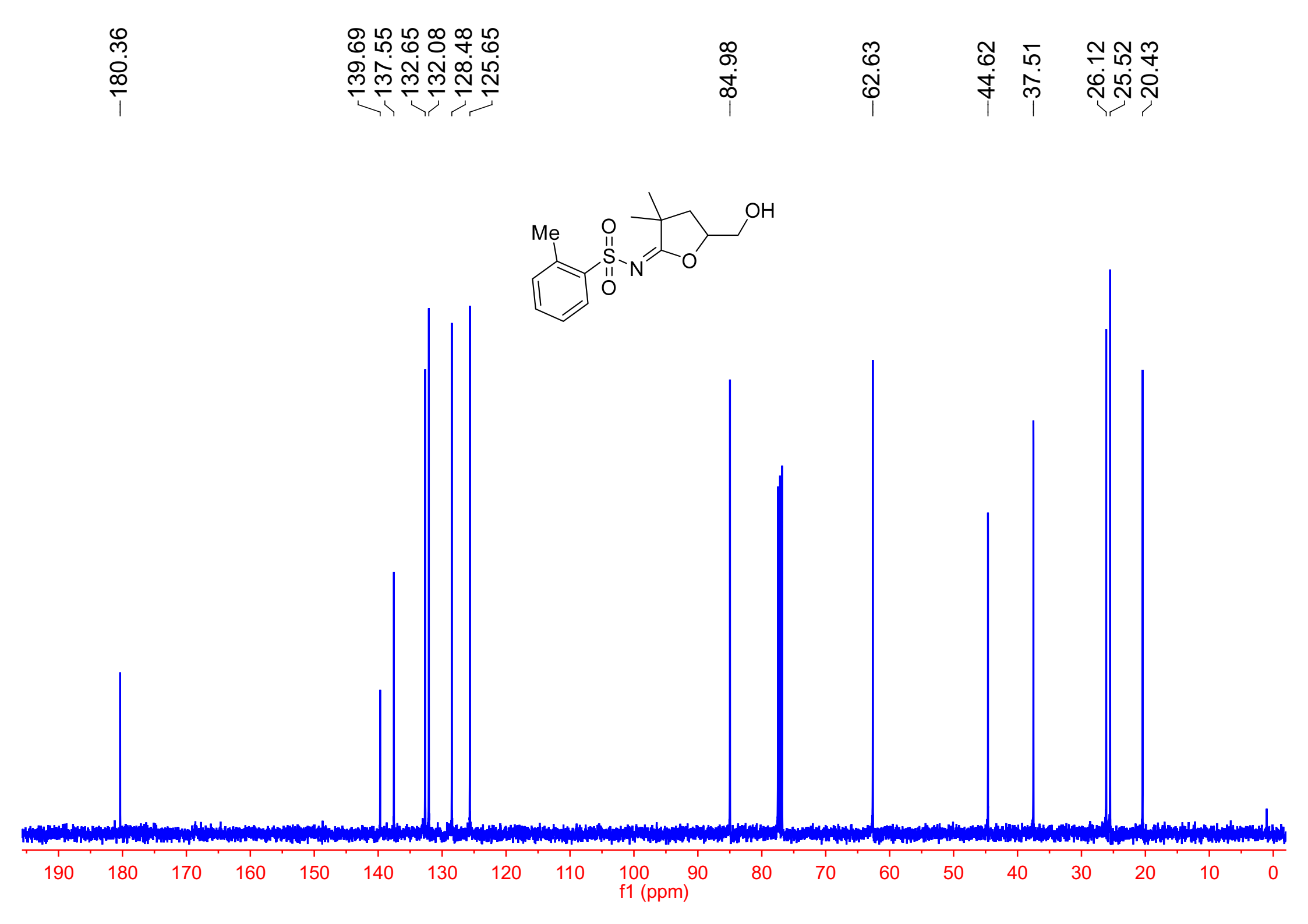
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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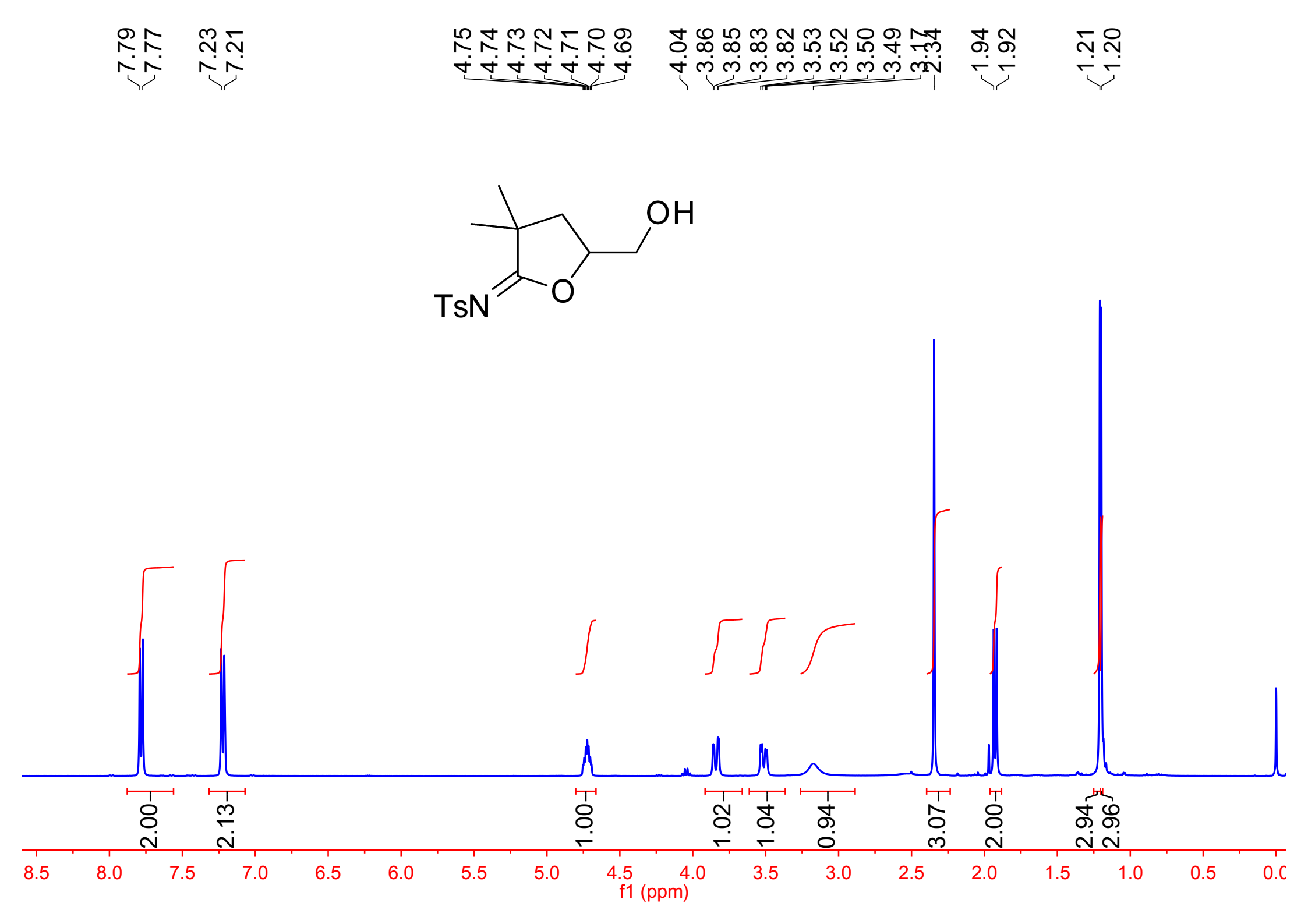
132.08
128.48
125.65
84.98
62.63
44.62
37.51
26.12
25.52
20.43
Me
S
O
O
N
O
OH
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.23
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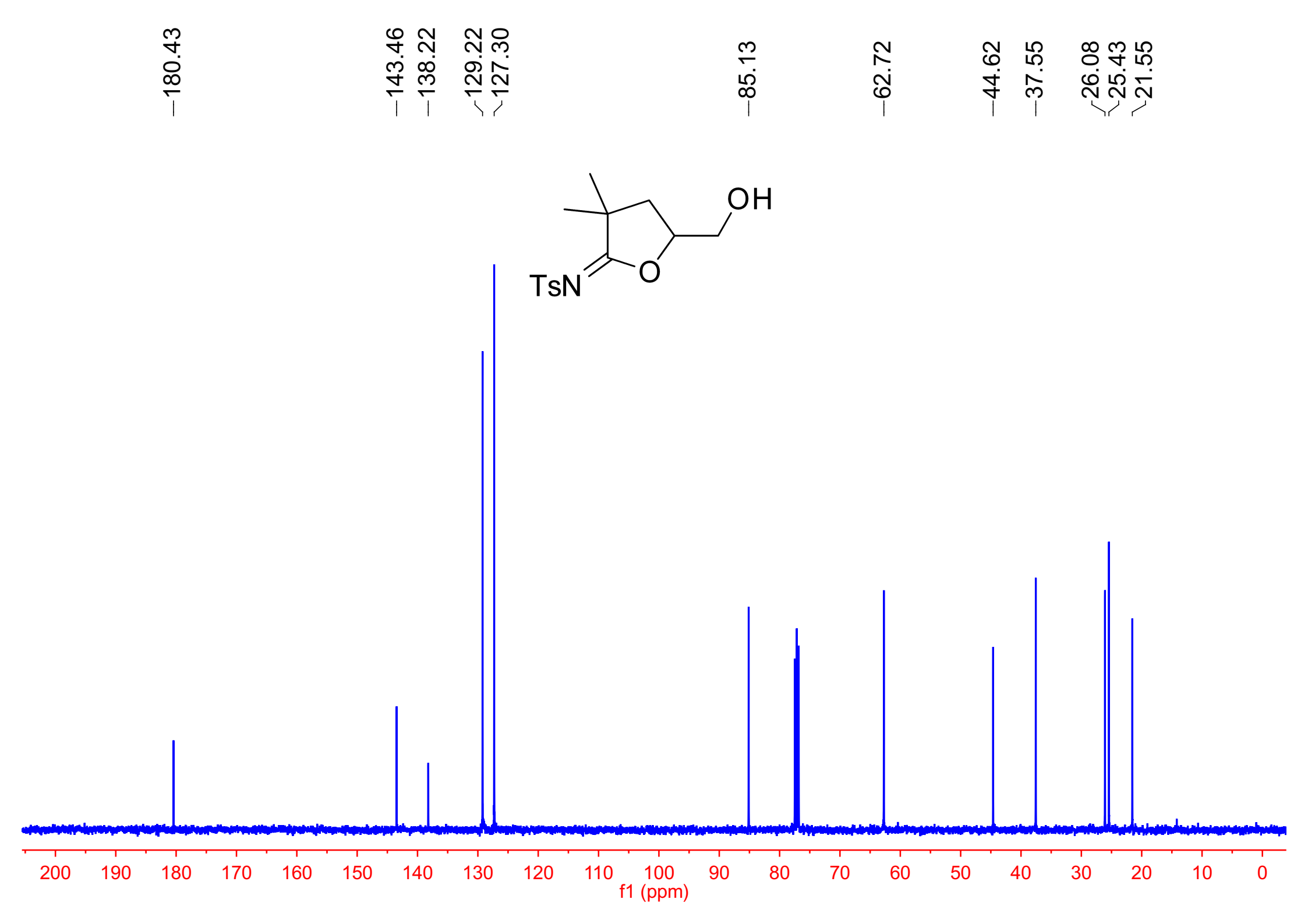
4.74
4.73
4.72
4.71
4.70
4.69
4.04
3.86
3.85
3.83
3.82
3.53
3.52
3.50
3.49
3.17
2.34
1.94
1.92
1.21
1.20
OH
O
TsN
2.00
2.13
1.00
1.02
1.04
0.94
3.07
2.00
2.94
2.96
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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85.13
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44.62
37.55
26.08
25.43
21.55
OH
TsN
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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50
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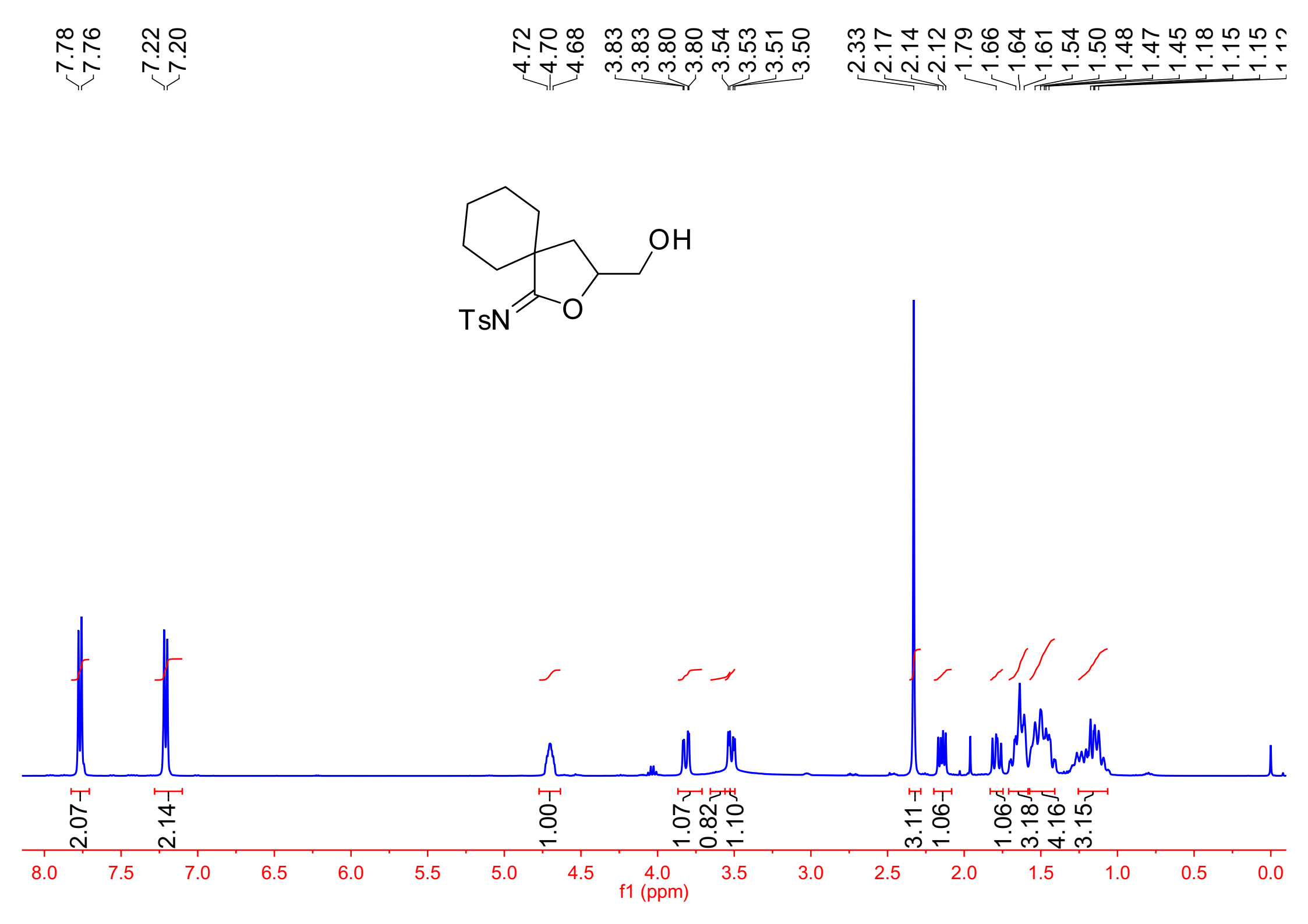
f1 (ppm)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7.76
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7.20
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4.68
3.83
3.83
3.80
3.80
3.54
3.53
3.51
3.50
2.33
2.17
2.14
2.12
1.79
1.66
1.64
1.61
1.54
1.50
1.48
1.47
1.45
1.18
1.15
1.15
OH
TsN
O
2.07
2.14
1.00
1.07
0.82
1.10
3.11
1.06
1.06
3.18
4.16
3.15
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
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2.5
2.0
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1.0
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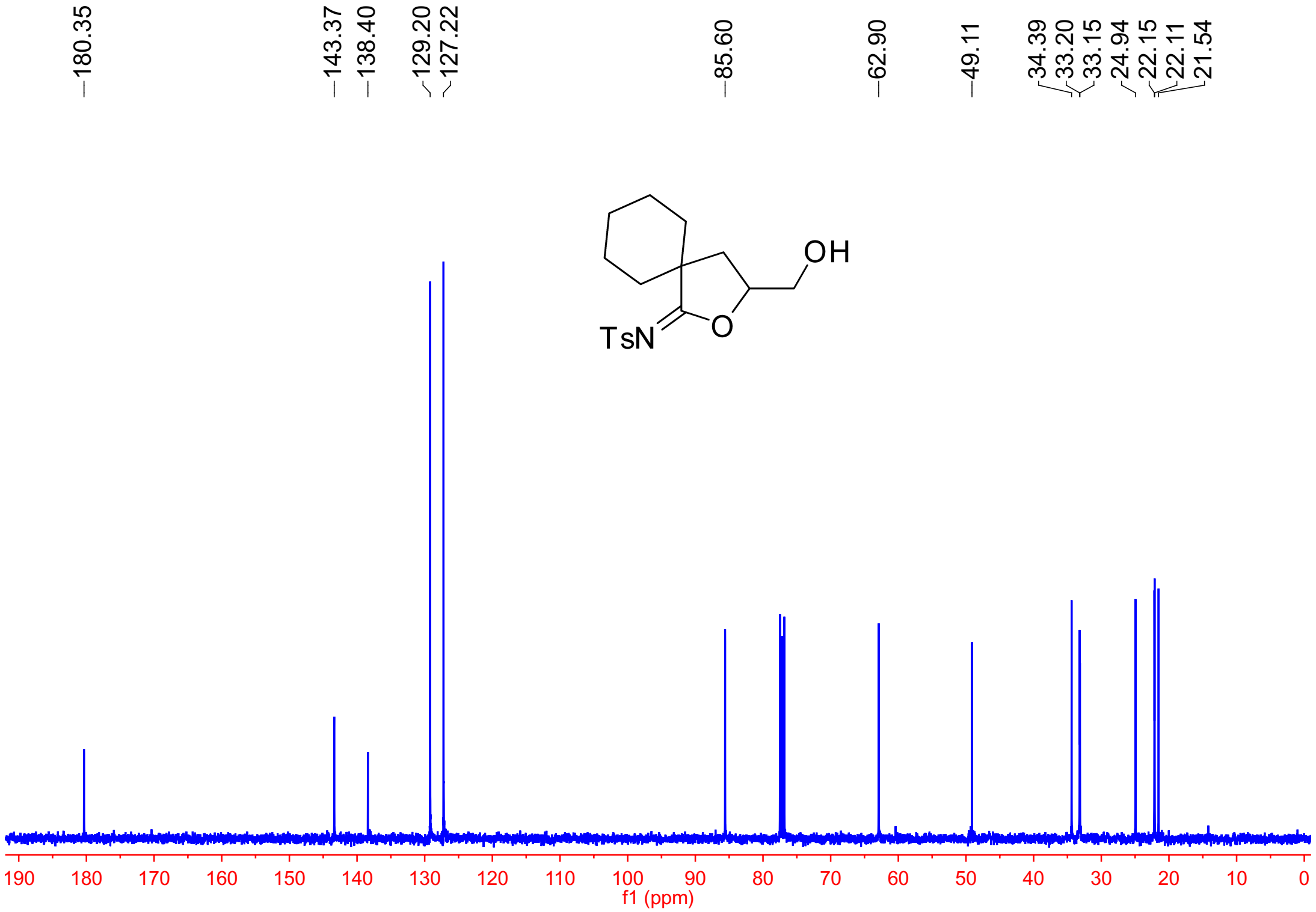
0.0
f1 (ppm)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143.37
138.40
129.20
127.22
85.60
62.90
49.11
34.39
33.20
33.15
24.94
22.15
22.11
21.54
OH
TsN
O
190
180
170
160
150
140
130
120
110
100
90
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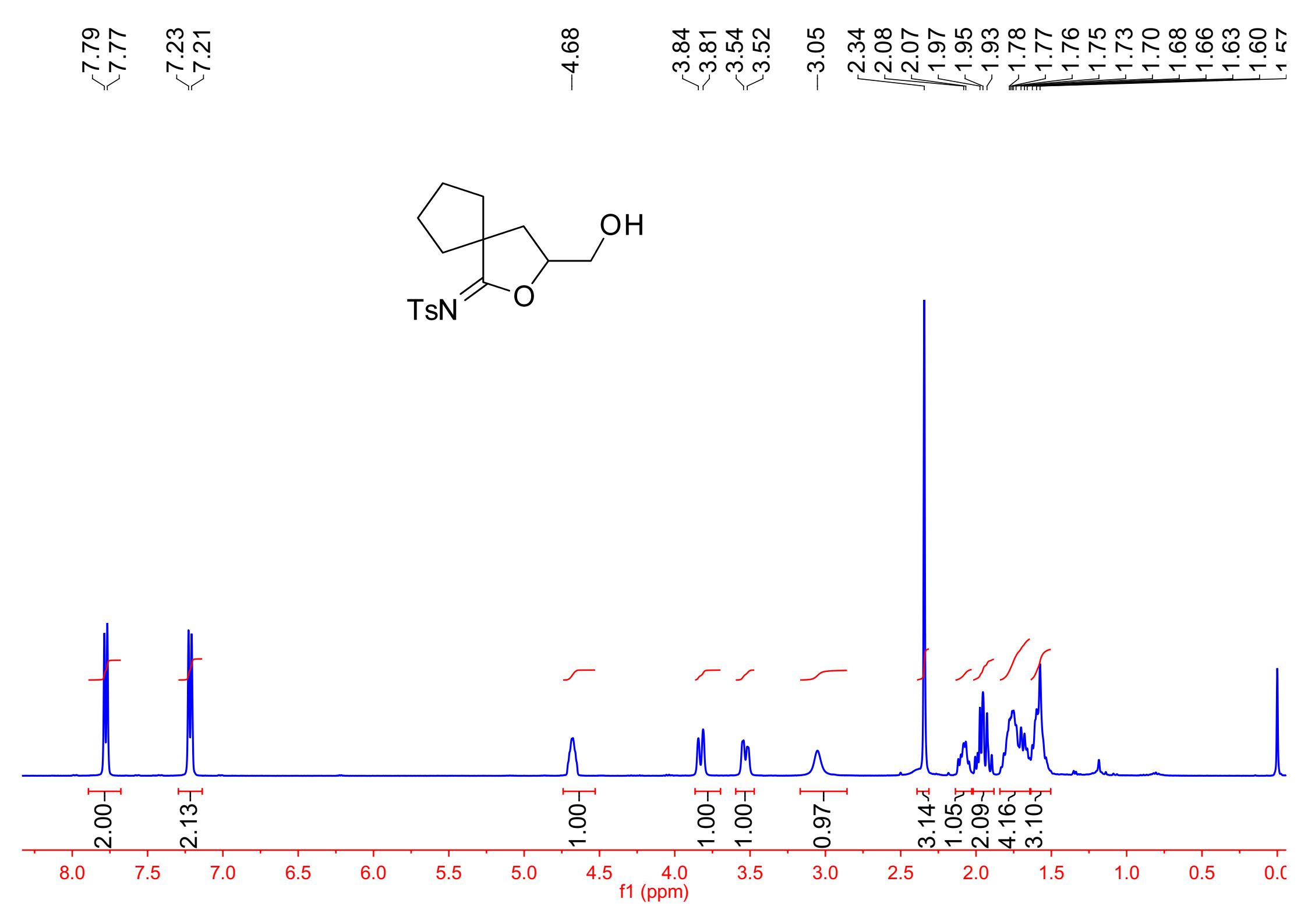
70
60
50
40
30
20
10
0
f1 (ppm)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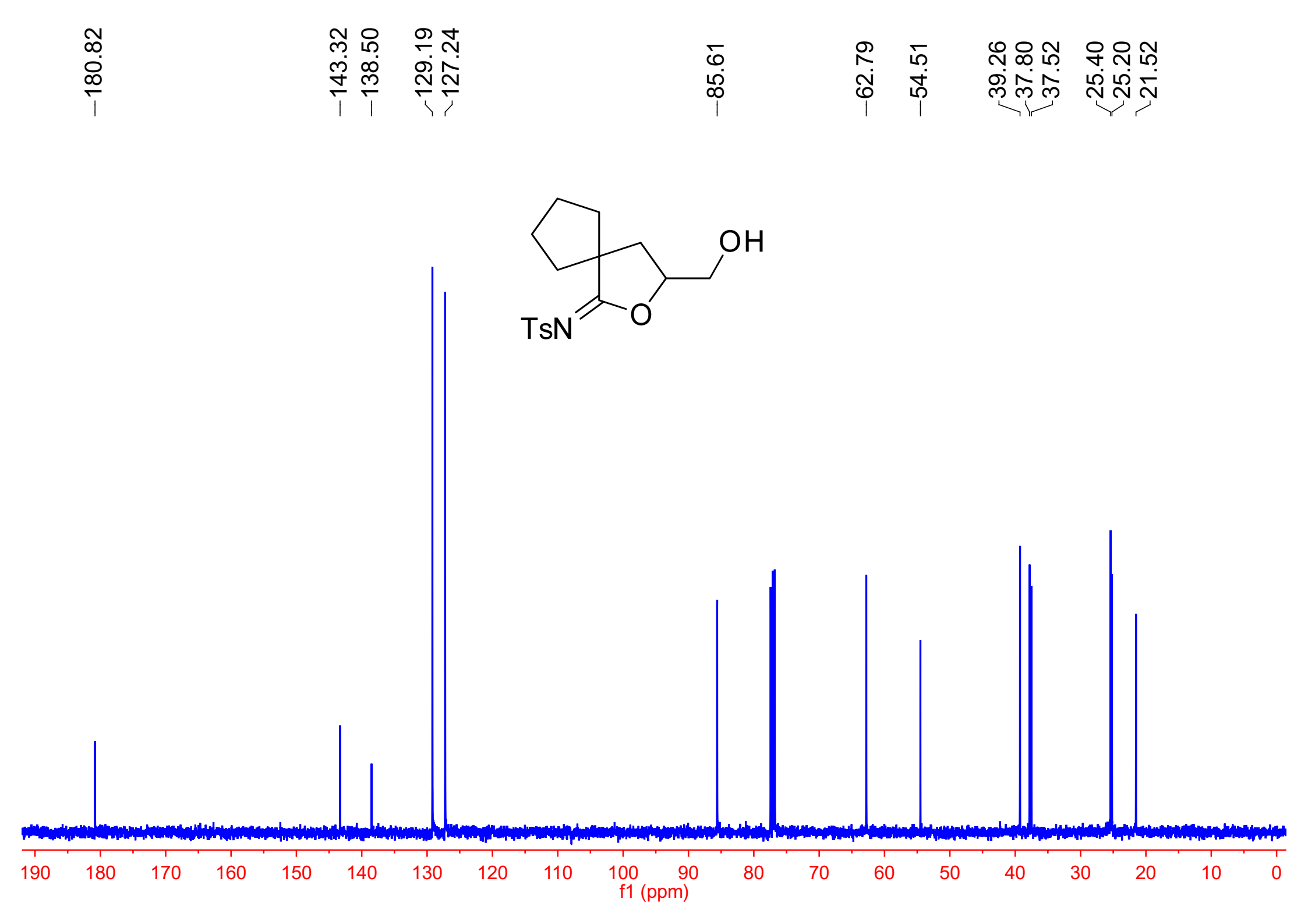
7.23
7.21
4.68
3.84
3.81
3.54
3.52
3.05
2.34
2.08
2.07
1.97
1.95
1.93
1.78
1.77
1.76
1.75
1.73
1.70
1.68
1.66
1.63
1.60
OH
O
TsN
2.00
2.13
1.00
1.00
1.00
0.97
3.14
1.05
2.09
4.16
3.10
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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138.50
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127.24
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54.51
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37.80
37.52
25.40
25.20
21.52
OH
TsN
O
190
180
170
160
150
140
130
120
110
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90
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70
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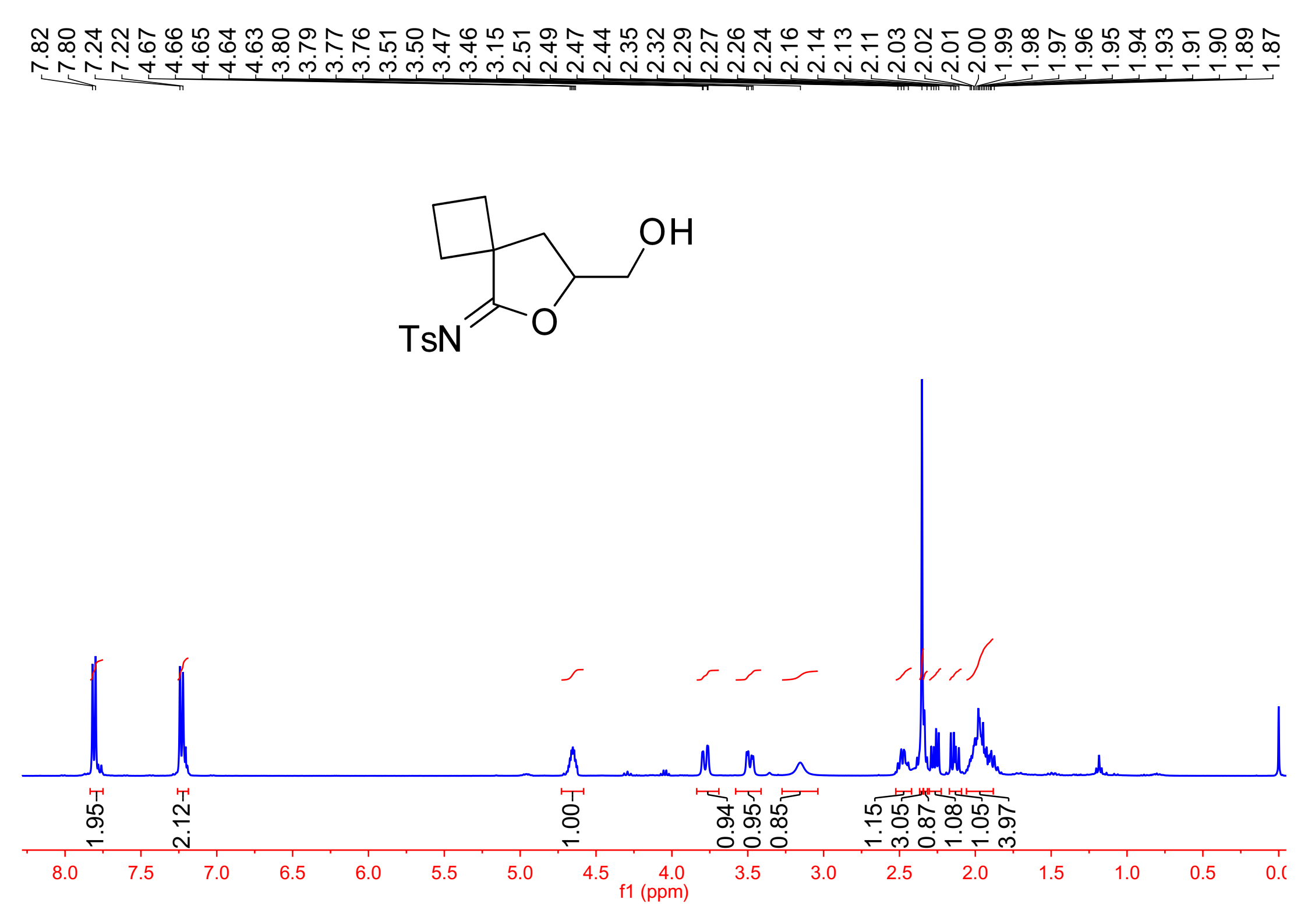
50
40
30
20
10
0
f1 (ppm)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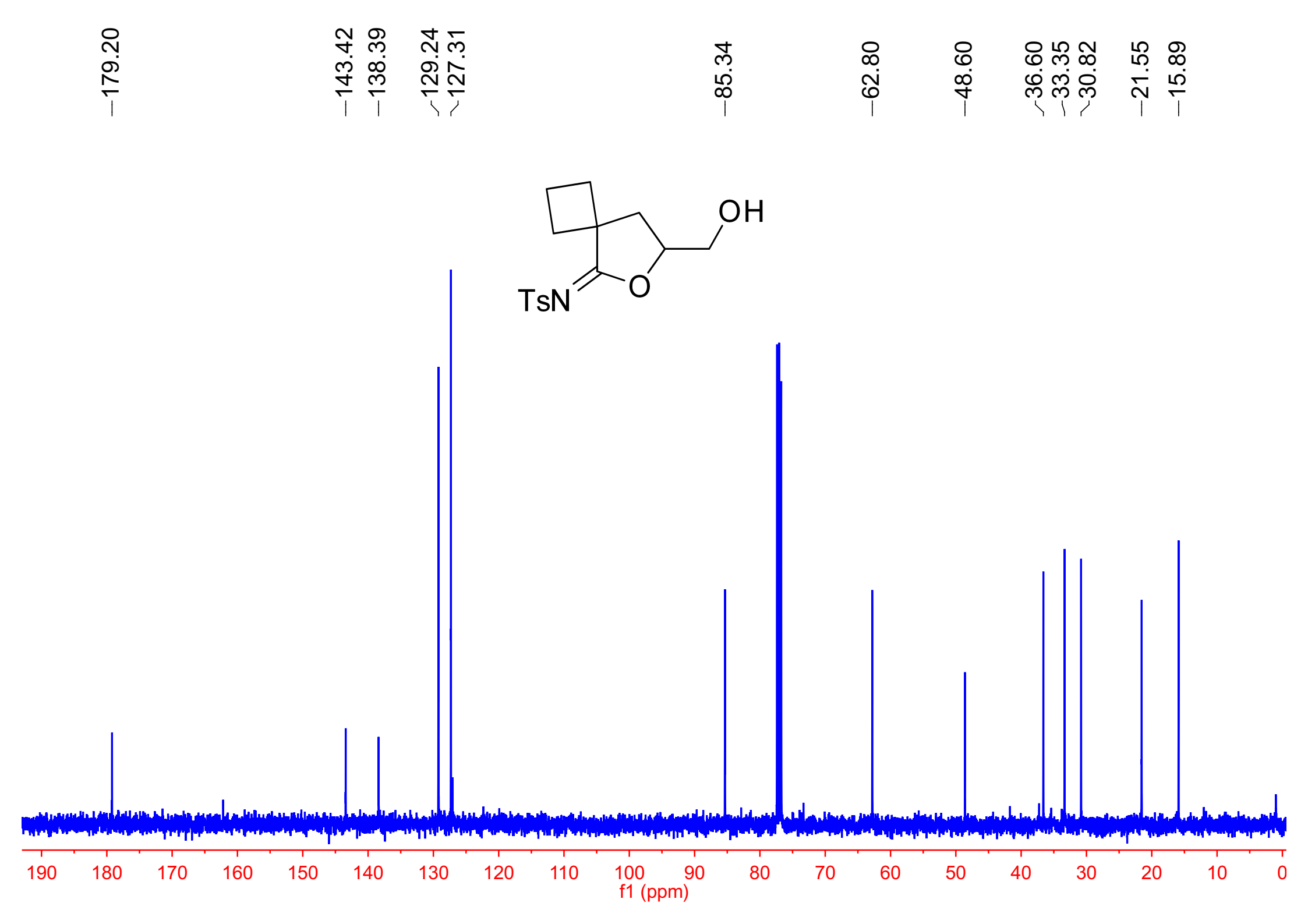
OH
O
TsN
1.95
2.12
1.00
0.94
0.95
0.85
1.15
3.05
0.87
1.08
1.05
3.97
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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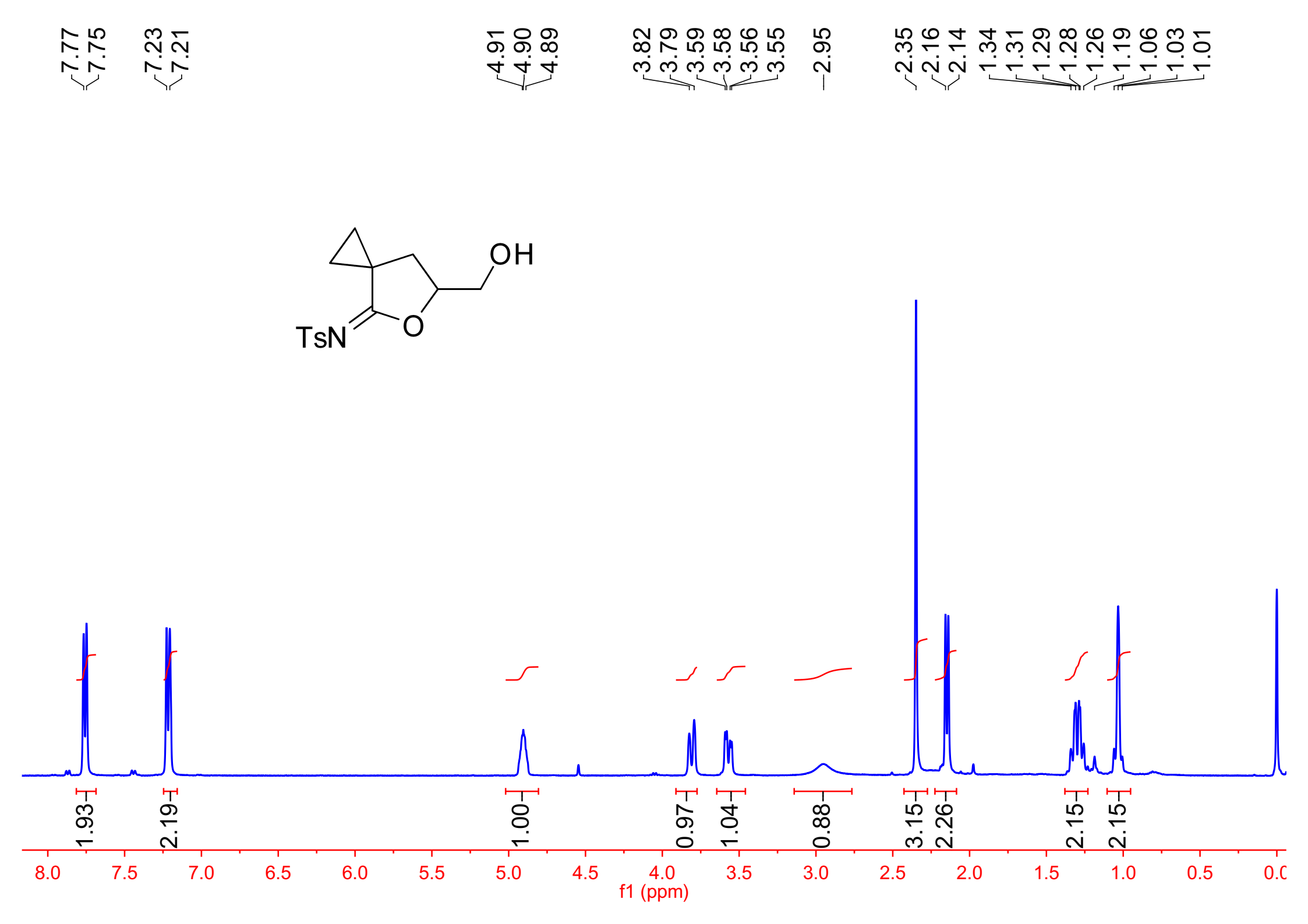
129.24
127.31
85.34
62.80
48.60
36.60
33.35
30.82
21.55
15.89
OH
TsN
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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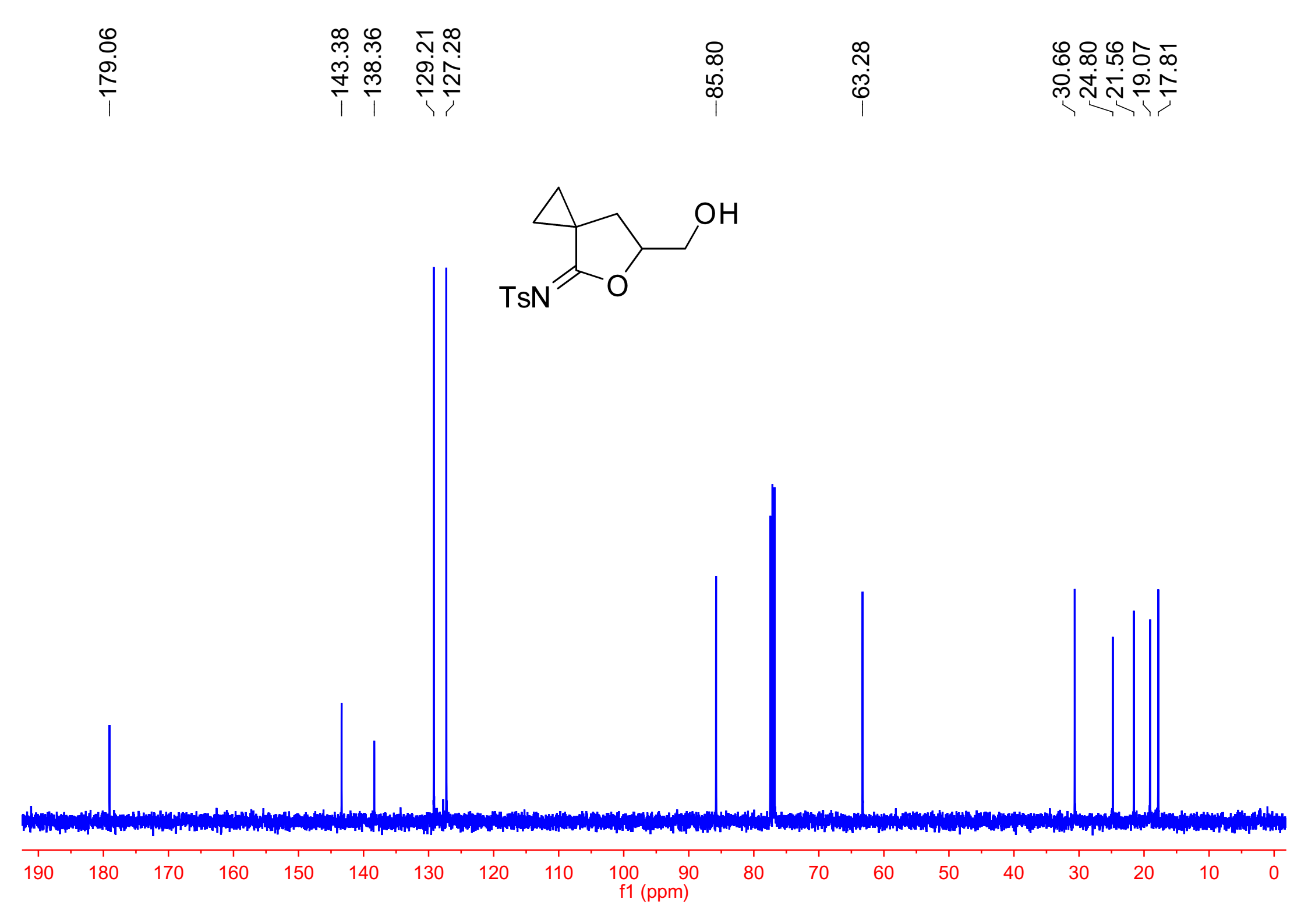
7.75
7.23
7.21
4.91
4.90
4.89
3.82
3.79
3.59
3.58
3.56
3.55
2.95
2.35
2.16
2.14
1.34
1.31
1.29
1.28
1.26
1.19
1.06
1.03
1.01
OH
TsN
O
1.93
2.19
1.00
0.97
1.04
0.88
3.15
2.26
2.15
2.15
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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143.38
138.36
129.21
127.28
85.80
63.28
30.66
24.80
21.56
19.07
17.81
OH
TsN
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)