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Photoinduced synthesis of (*E*)-vinyl sulfones through the insertion of sulfur dioxide

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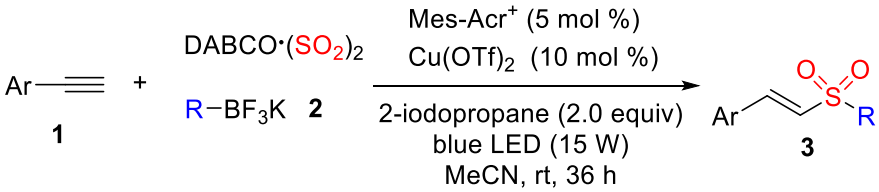
Supporting Information

1. General experimental method (S2-S3).
2. General experimental procedure and characterization data (S4-S9).
3. ^{1}H and ^{13}C NMR spectra of compounds **3** (S10-S43).

General experimental methods:

Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 25–35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

*General experimental procedure for the reaction of potassium alkyltrifluoroborates **1**, DABCO•$(SO_2)_2$, and alkynes **2**:*

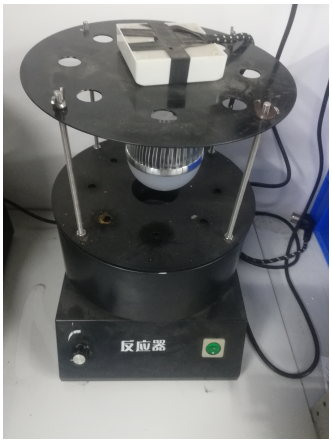


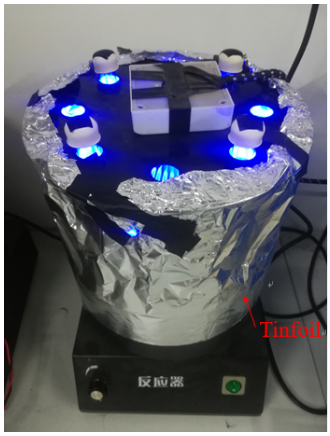
Potassium alkyltrifluoroborate **2** (0.2 mmol) and DABCO•$(SO_2)_2$ (0.16 mmol) were combined with $Cu(OTf)_2$ (10 mol %) and Mes-Acr^+ (5 mol %) in a tube. The tube was evacuated and backfilled with N_2 three times before the addition of alkyne **1** (0.4 mmol) and 2-iodopropane (0.4 mmol) in MeCN (3.0 mL). The mixture was then placed around a blue LED (15 W) with a distance of 10 centimeters, and was stirred under blue light irradiation for 36 hours at room temperature. After completion of reaction as indicated by TLC, the mixture was purified directly by flash column chromatography (EtOAc/*n*-hexane, 1:6) to provide the desired product **3**.

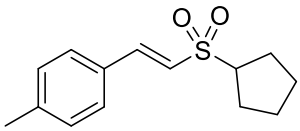
*A typical experimental procedure for the synthesis of compound **3a***:

Potassium cyclopentyltrifluoroborate **2a** (0.2 mmol) and DABCO•$(SO_2)_2$ (0.16 mmol) were combined with $Cu(OTf)_2$ (10 mol %) and Mes-Acr^+ (5 mol %) in a tube. The tube was evacuated and backfilled with N_2 three times before the addition of 1-ethynyl-4-methylbenzene **1a** (0.4 mmol) and 2-iodopropane (0.4 mmol) in MeCN (3.0 mL). The mixture was then placed around a blue LED (15 W) with a distance of 10 centimeters, and was stirred under blue light irradiation for 36 hours at room temperature. After completion of reaction as indicated by TLC, the mixture was purified directly by flash column chromatography (EtOAc/*n*-hexane, 1:6) to provide the desired product **3a** as white solid (36.5 mg, 73% yield).

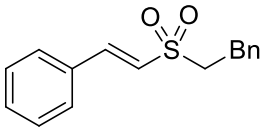
Experimental setup:



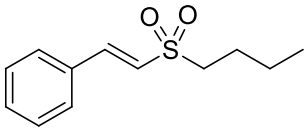




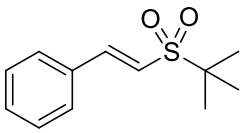
(*E*)-1-(2-(Cyclopentylsulfonyl)vinyl)-4-methylbenzene **(3a)**: White solid, 36.5 mg (73% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 15.5 Hz, 1H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 6.76 (d, *J* = 15.5 Hz, 1H), 3.65 – 3.20 (m, 1H), 2.40 (s, 3H), 2.16 – 1.97 (m, 4H), 1.88 – 1.76 (m, 2H), 1.73 – 1.62 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.76, 141.82, 129.79, 129.65, 128.47, 122.58, 63.04, 27.07, 25.98, 21.48; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{14}H_{19}O_2S^+$: 251.1100; found: 251.1103.



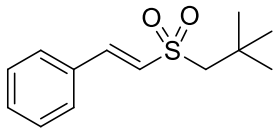
(*E*)-(2-(Phenethylsulfonyl)viny)benzene **(3b)**: White solid, 32.6 mg (60% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 15.5 Hz, 1H), 7.51 – 7.40 (m, 5H), 7.31 (t, *J* = 7.3 Hz, 2H), 7.26 – 7.19 (m, 3H), 6.72 (d, *J* = 15.5 Hz, 1H), 3.37 (t, *J* = 8.12 Hz, 2H), 3.16 (t, *J* = 8.12 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.98, 137.49, 132.09, 131.36, 129.07, 128.84, 128.54, 128.39, 126.95, 124.62, 56.48, 28.77; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{16}H_{17}O_2S^+$: 273.0944; found: 273.0979.



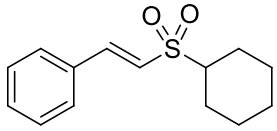
(*E*)-(2-(Butylsulfonyl)vinyl)benzene **(3c)**: White solid, 34.9 mg (78% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 15.5 Hz, 1H), 7.56 – 7.49 (m, 2H), 7.47 – 7.40 (m, 3H), 6.83 (d, *J* = 15.5 Hz, 1H), 3.41 – 2.80 (m, 2H), 1.85 – 1.78 (m, 2H), 1.56 – 1.37 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.76, 132.22, 131.31, 129.12, 128.53, 124.64, 54.92, 24.51, 21.62, 13.53; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{12}H_{17}O_2S^+$: 225.0944; found: 225.0940.



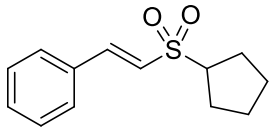
(*E*)-(2-(*tert*-Butylsulfonyl)vinyl)benzene [1] **(3d)**: White solid, 32.7 mg (73% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 15.6 Hz, 1H), 7.54 – 7.52 (m, 2H), 7.48 – 7.38 (m, 3H), 6.83 (d, *J* = 15.5 Hz, 1H), 1.42 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.38, 132.43, 131.20, 129.07, 128.48, 120.57, 58.86, 23.35.



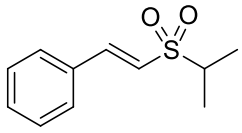
(*E*)-(2-(Neopentylsulfonyl)vinyl)benzene **(3e)**: White solid, 26.7 mg (56% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 15.5 Hz, 1H), 7.53 – 7.48 (m, 2H), 7.47 – 7.37 (m, 3H), 6.87 (d, *J* = 15.4 Hz, 1H), 3.04 (s, 2H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.18, 132.31, 131.16, 129.09, 128.44, 127.49, 66.67, 32.15, 29.87; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{13}H_{19}O_2S^+$: 239.1100; found: 239.1098.



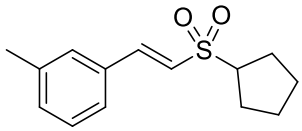
(*E*)-(2-(Cyclohexylsulfonyl)vinyl)benzene **(3f)**: White solid, 44.0 mg (88% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.48 (m, 3H), 7.47 – 7.37 (m, 3H), 6.78 (d, *J* = 15.5 Hz, 1H), 2.91 – 2.83 (m, 1H), 2.21 (d, *J* = 11.7 Hz, 2H), 1.90 (d, *J* = 13.2 Hz, 2H), 1.54 – 1.44 (m, 2H), 1.37 – 1.12 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.44, 132.26, 131.12, 128.98, 128.40, 122.66, 62.44, 25.32, 25.27, 24.96; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{14}H_{19}O_2S^+$: 251.1100; found: 251.1142.



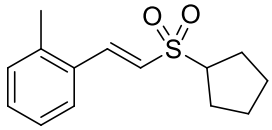
(*E*)-(2-(Cyclopentylsulfonyl)vinyl)benzene **(3g)**: White solid, 35.4 mg (75% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 15.5 Hz, 1H), 7.53 – 7.49 (m, 2H), 7.48 – 7.35 (m, 3H), 6.82 (d, *J* = 15.5 Hz, 1H), 3.60 – 3.24 (m, 1H), 2.18 – 1.92 (m, 4H), 1.88 – 1.75 (m, 2H), 1.73 – 1.56 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.72, 132.37, 131.17, 129.06, 128.45, 123.83, 62.97, 27.03, 25.96; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{13}H_{17}O_2S^+$: 237.0944; found: 237.0947.



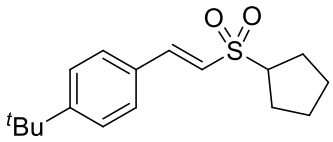
(*E*)-(2-(Isopropylsulfonyl)vinyl)benzene **(3h)**: White solid, 32.3 mg (77% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 15.5 Hz, 1H), 7.56 – 7.50 (m, 2H), 7.48 – 7.40 (m, 3H), 6.80 (d, *J* = 15.5 Hz, 1H), 3.18 – 3.12 (m, 1H), 1.40 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.81, 132.32, 131.26, 129.08, 128.49, 122.24, 54.62, 15.54; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{11}H_{15}O_2S^+$: 211.0787; found: 211.0787



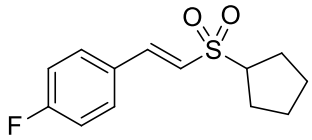
(*E*)-1-(2-(Cyclopentylsulfonyl)vinyl)-3-methylbenzene **(3i)**: White solid, 33.5 mg (67% yield); ^{1}H NMR (400 MHz, $CDCl_3$) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 15.5 Hz, 1H), 7.37 – 7.22 (m, 4H), 6.80 (d, *J* = 15.5 Hz, 1H), 3.62 – 3.05 (m, 1H), 2.38 (s, 3H), 2.14 – 1.96 (m, 4H), 1.87 – 1.74 (m, 2H), 1.72 – 1.60 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.91, 138.81, 132.31, 131.98, 129.03, 128.92, 125.66, 123.53, 62.97, 27.03, 25.96, 21.21; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{14}H_{19}O_2S^+$: 251.1100; found: 251.1100.



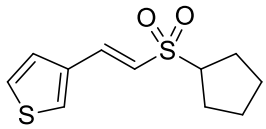
(*E*)-1-(2-(Cyclopentylsulfonyl)vinyl)-2-methylbenzene **(3j)**: White solid, 29.0 mg (58% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 15.4 Hz, 1H), 7.50 (d, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 7.4 Hz, 1H), 7.28 – 7.20 (m, 2H), 6.73 (d, *J* = 15.4 Hz, 1H), 3.65 – 3.24 (m, 1H), 2.45 (s, 3H), 2.16 – 1.98 (m, 4H), 1.88 – 1.77 (m, 2H), 1.73 – 1.63 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.58, 138.07, 131.43, 131.02, 130.90, 126.80, 126.49, 124.88, 62.93, 27.09, 26.01, 19.74; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{14}H_{19}O_2S^+$: 251.1100; found: 251.1103.



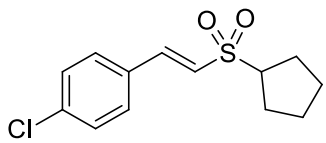
(*E*)-1-(*tert*-Butyl)-4-(2-(cyclopentylsulfonyl)vinyl)benzene **(3k)**: White solid, 42.6 mg (73% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 15.5 Hz, 1H), 7.50 – 7.42 (m, 4H), 6.78 (d, *J* = 15.5 Hz, 1H), 3.49 – 3.37 (m, 1H), 2.19 – 1.94 (m, 4H), 1.78 - 1.82 (m, 2H), 1.72 – 1.57 (m, 2H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.90, 144.66, 129.63, 128.33, 126.04, 122.78, 63.06, 34.92, 31.04, 27.06, 25.97; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{17}H_{25}O_2S^+$: 293.1570; found: 293.1569.



(*E*)-1-(2-(Cyclopentylsulfonyl)vinyl)-4-fluorobenzene **(3l)**: White solid, 39.6 mg (78% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 – 7.46 (m, 3H), 7.10 (t, *J* = 8.6 Hz, 2H), 6.75 (d, *J* = 15.5 Hz, 1H), 3.50 – 3.35 (m, 1H), 2.16 – 1.95 (m, 4H), 1.88 – 1.74 (m, 2H), 1.73 – 1.56 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.30 (d, J_{CF-1} = 252.9 Hz), 143.37, 130.49 (d, J_{CF-3} = 8.7 Hz), 128.64, 123.62, 116.28 (d, J_{CF-2} = 22.1 Hz), 62.96, 27.02, 25.95; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{13}H_{16}FO_2S^+$: 255.0850; found: 255.0841.

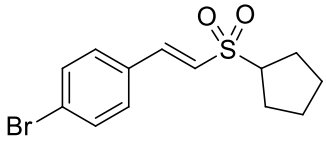


(*E*)-3-(2-(Cyclopentylsulfonyl)vinyl)thiophene **(3m)**: White solid, 31.0 mg (64% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.51 (m, 2H), 7.39 – 7.37 (m, 1H), 7.27 (d, *J* = 4.9 Hz, 1H), 6.64 (d, *J* = 15.4 Hz, 1H), 3.62 – 3.05 (m, 1H), 2.12 – 1.96 (m, 4H), 1.88 – 1.73 (m, 2H), 1.71 – 1.57 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.13, 135.25, 129.89, 127.50, 125.05, 123.16, 63.03, 27.04, 25.95; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{11}H_{15}O_2S_2^+$: 243,0508; found: 243,0505.

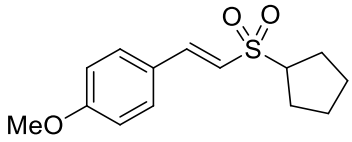


(*E*)-1-Chloro-4-(2-(cyclopentylsulfonyl)vinyl)benzene **(3n)**: White solid, 33.1mg (61% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 15.5 Hz, 1H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 6.80 (d, *J* = 15.5 Hz, 1H), 3.60 – 3.18 (m, 1H), 2.14 – 1.95 (m, 4H), 1.88 – 1.73 (m, 2H), 1.71 – 1.59 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.23,

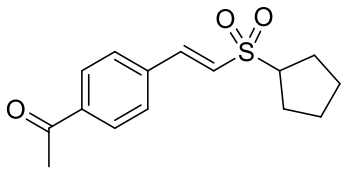
137.19, 130.84, 129.64, 129.36, 124.47, 62.92, 27.00, 25.95; HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{13}H_{16}ClO_2S^+$: 271.0554; found: 271.0560.



(*E*)-1-Bromo-4-(2-(cyclopentylsulfonyl)vinyl)benzene **(3o)**: White solid, 42.1 mg (67% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.51 (m, 3H), 7.38 (d, *J* = 8.2 Hz, 2H), 6.83 (d, *J* = 15.5 Hz, 1H), 3.54 – 3.18 (m, 1H), 2.14 – 1.98 (m, 4H), 1.87 – 1.76 (m, 2H), 1.72 – 1.61 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.31, 132.35, 131.30, 129.81, 125.61, 124.64, 62.96, 27.02, 25.96; HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{13}H_{16}BrO_2S^+$: 315.0049; found: 315.0046.



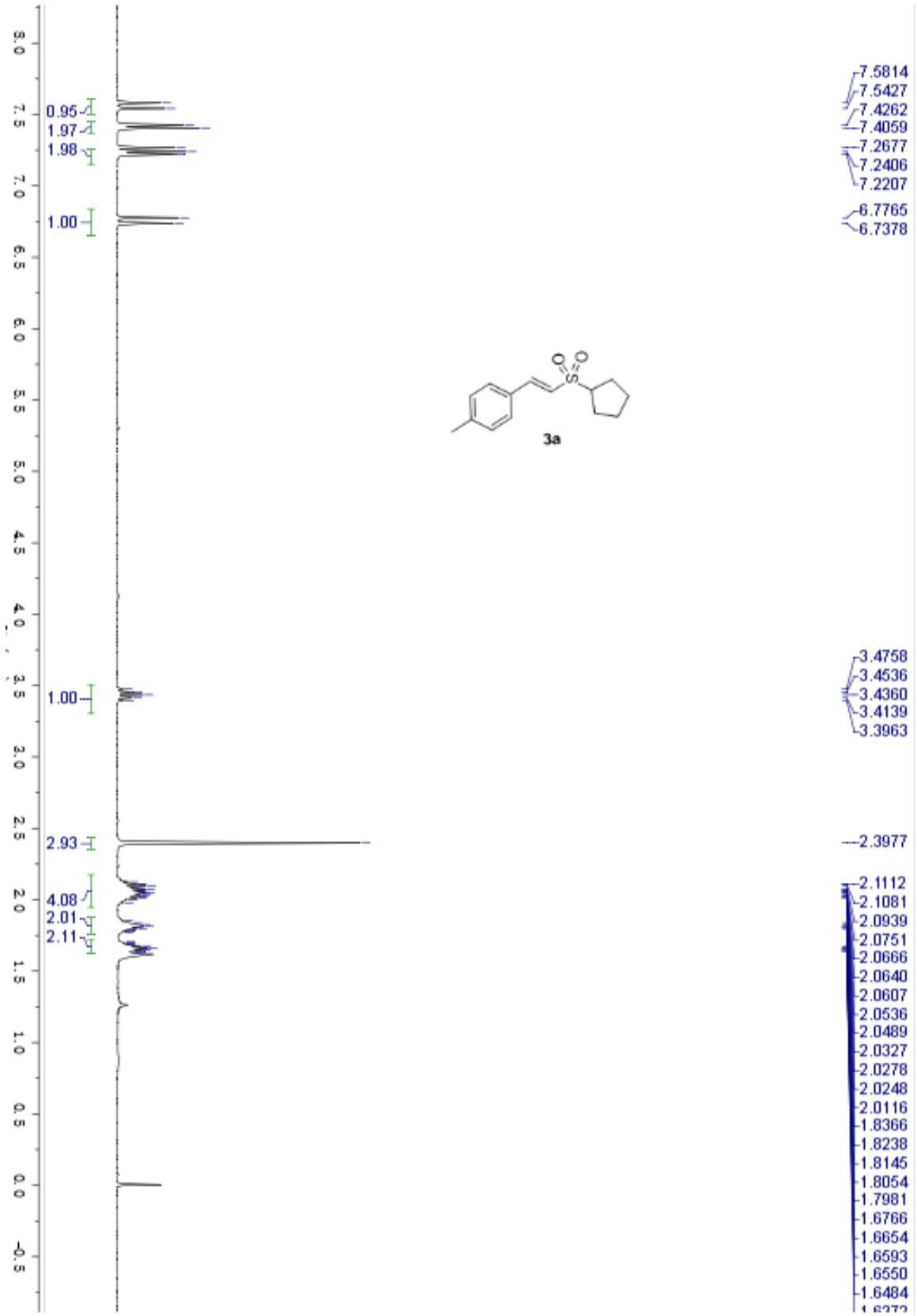
(*E*)-1-(2-(Cyclopentylsulfonyl)vinyl)-4-methoxybenzene **(3p)**: White solid, 26.6 mg (50% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 15.5 Hz, 1H), 7.46 (d, *J* = 8.7 Hz, 2H), 6.93 (d, *J* = 8.7 Hz, 2H), 6.66 (d, *J* = 15.4 Hz, 1H), 3.85 (s, 3H), 3.49 – 3.37 (m, 1H), 2.14 – 1.95 (m, 4H), 1.88 – 1.74 (m, 2H), 1.70 – 1.57 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.01, 144.43, 130.25, 125.02, 120.94, 114.48, 63.12, 55.41, 27.10, 25.98; HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{14}H_{19}O_3S^+$: 267.1049; found: 267.1049.



(*E*)-1-(4-(2-(Cyclopentylsulfonyl)vinyl)phenyl)ethanone **(3q)**: White solid, 33.4 mg (60% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 15.5 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 7.9 Hz, 2H), 6.76 (d, *J* = 15.5 Hz, 1H), 3.64 – 3.19 (m, 1H), 2.38 (s, 3H), 2.29 – 1.93 (m, 4H), 1.91 – 1.73 (m, 2H), 1.74 – 1.48 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.72, 141.78, 129.76, 129.62, 128.45, 122.57, 63.01, 27.04, 25.95, 21.45; HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{15}H_{19}O_3S^+$: 279.1049; found: 279.1051.

Refence:

1. Z. Liu, X. Chen, J. Chen, L. Qu, Y. Xia, H. Wu, H. Ma, S. Zhu, Y. Zhao, *RSC Adv.* **2015**, *5*, 71215.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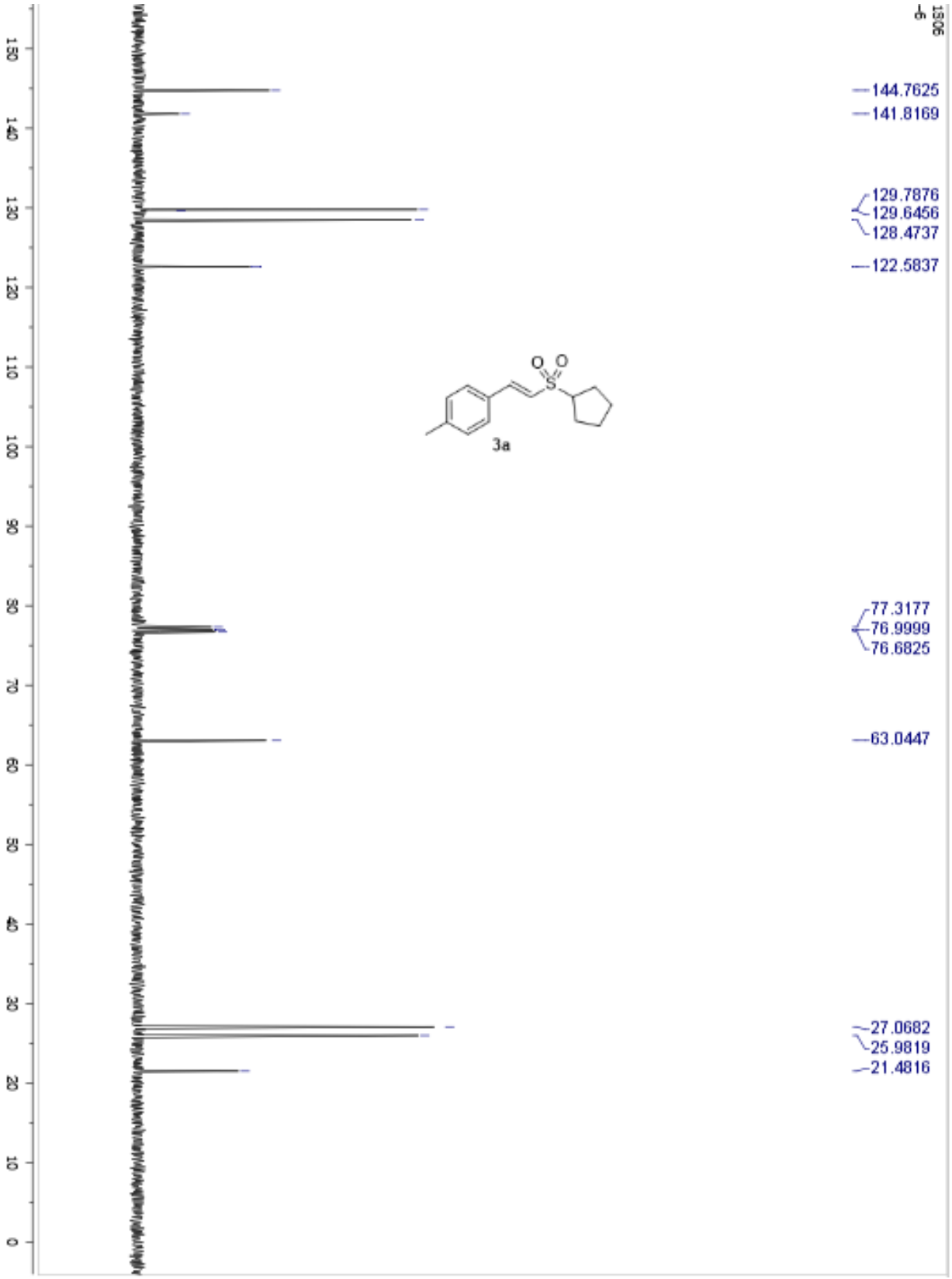
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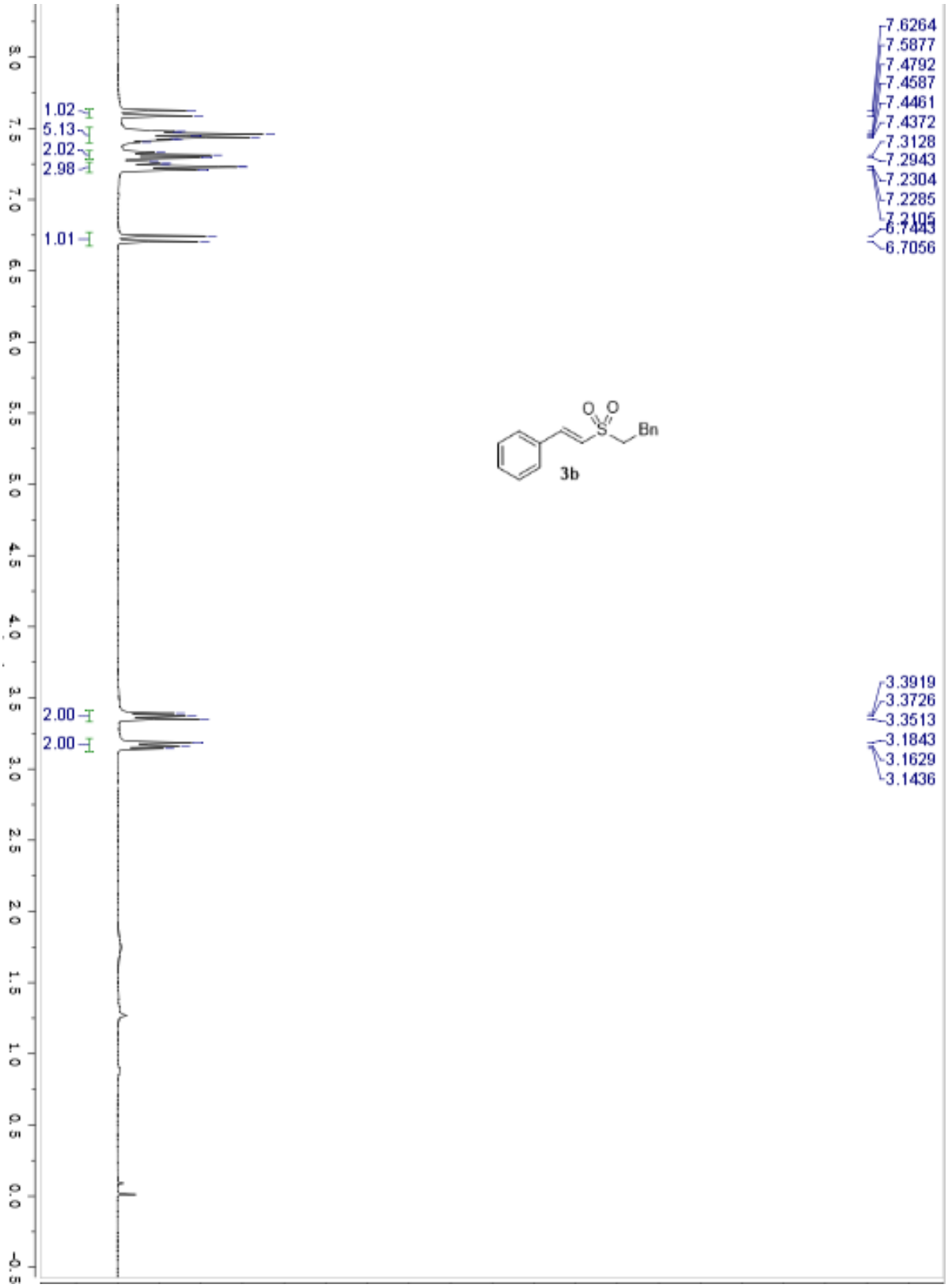
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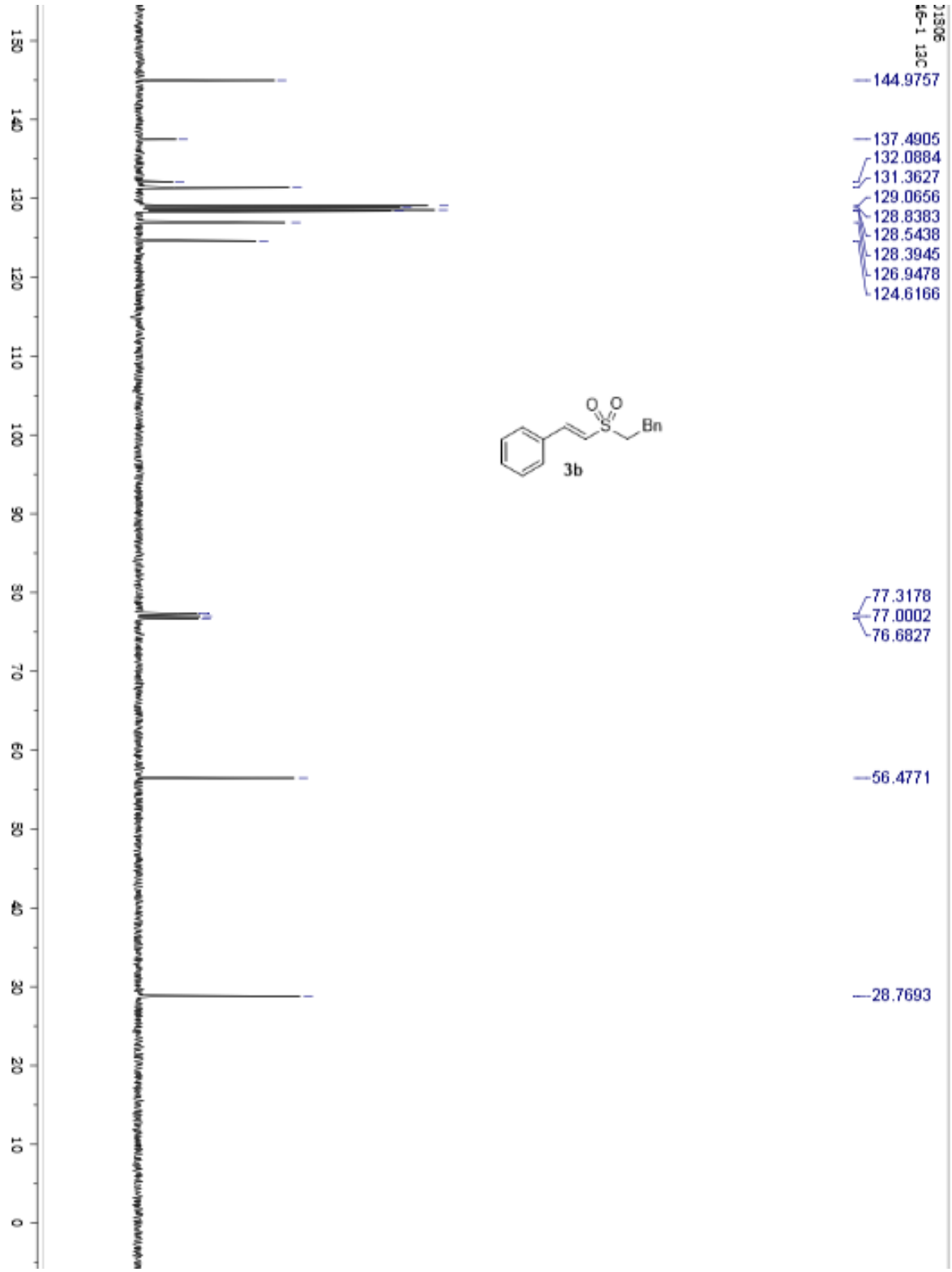
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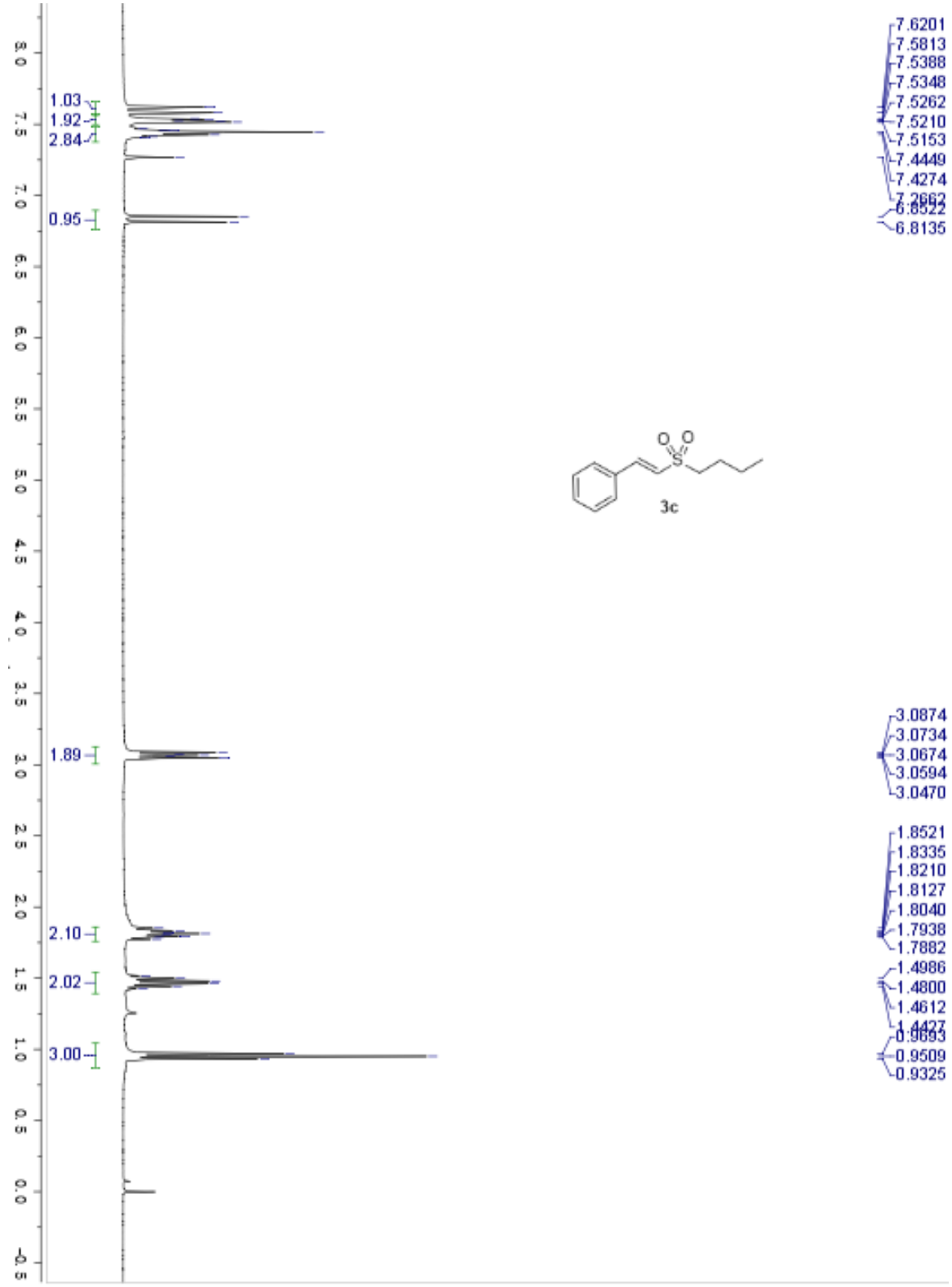
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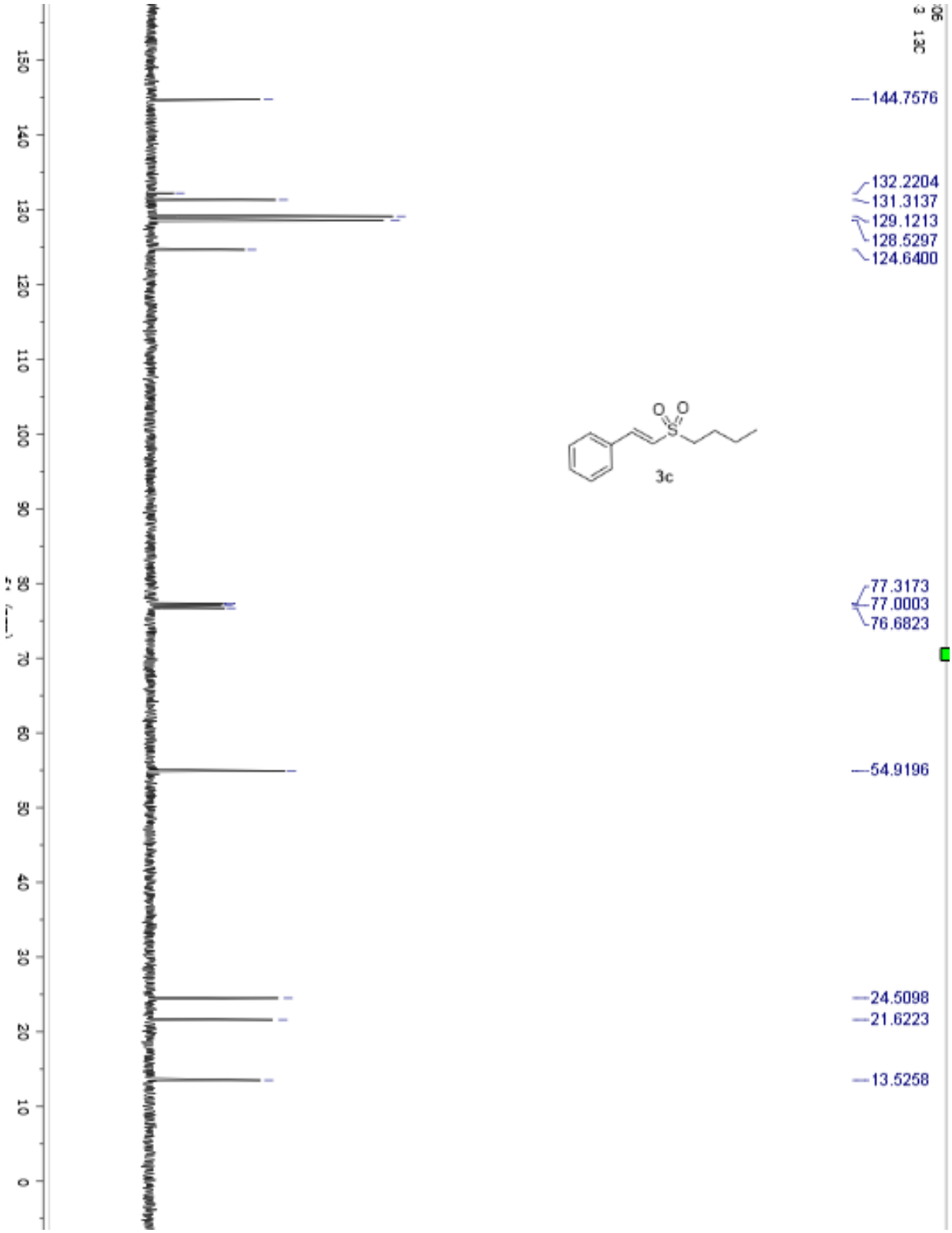
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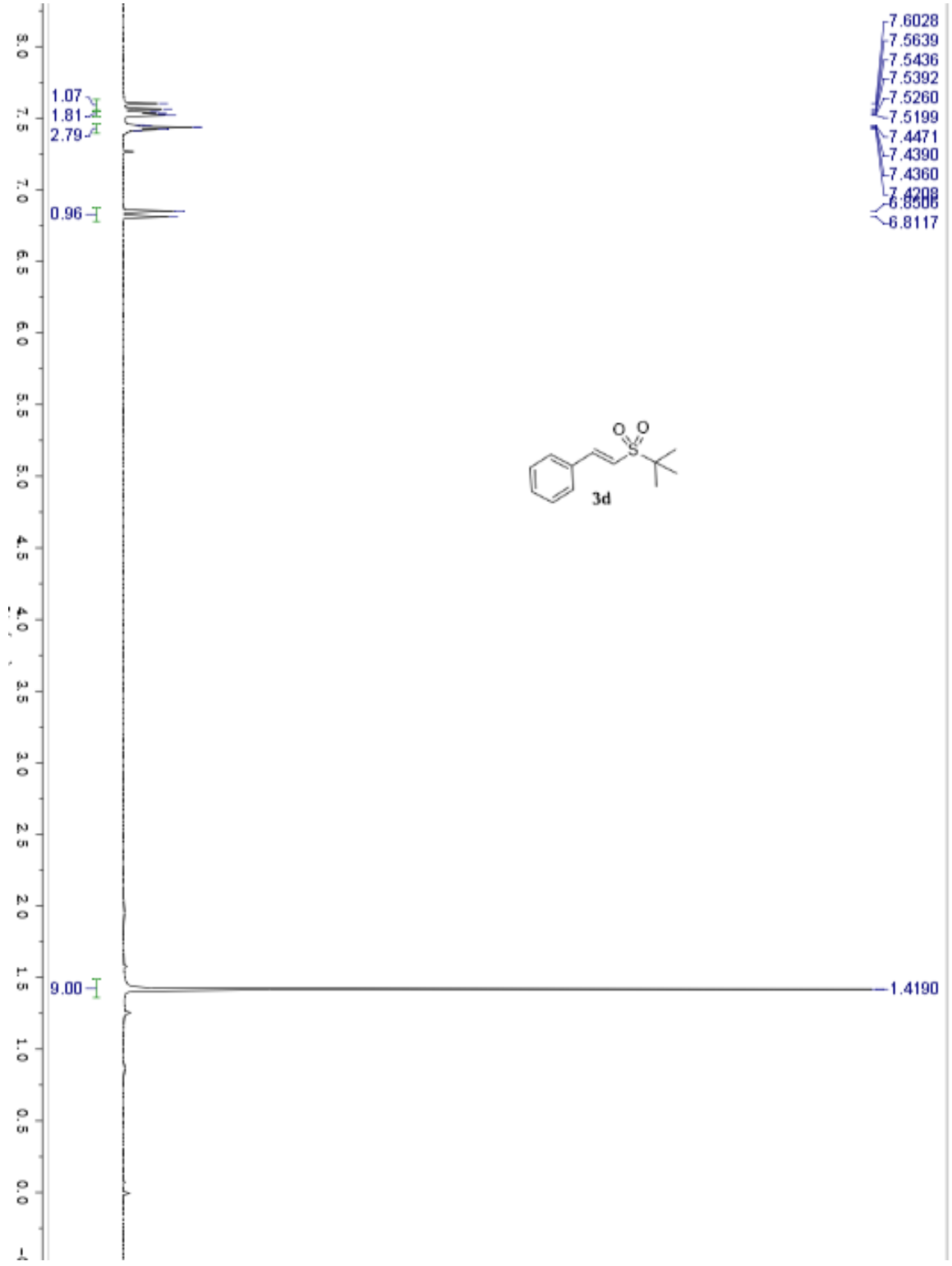
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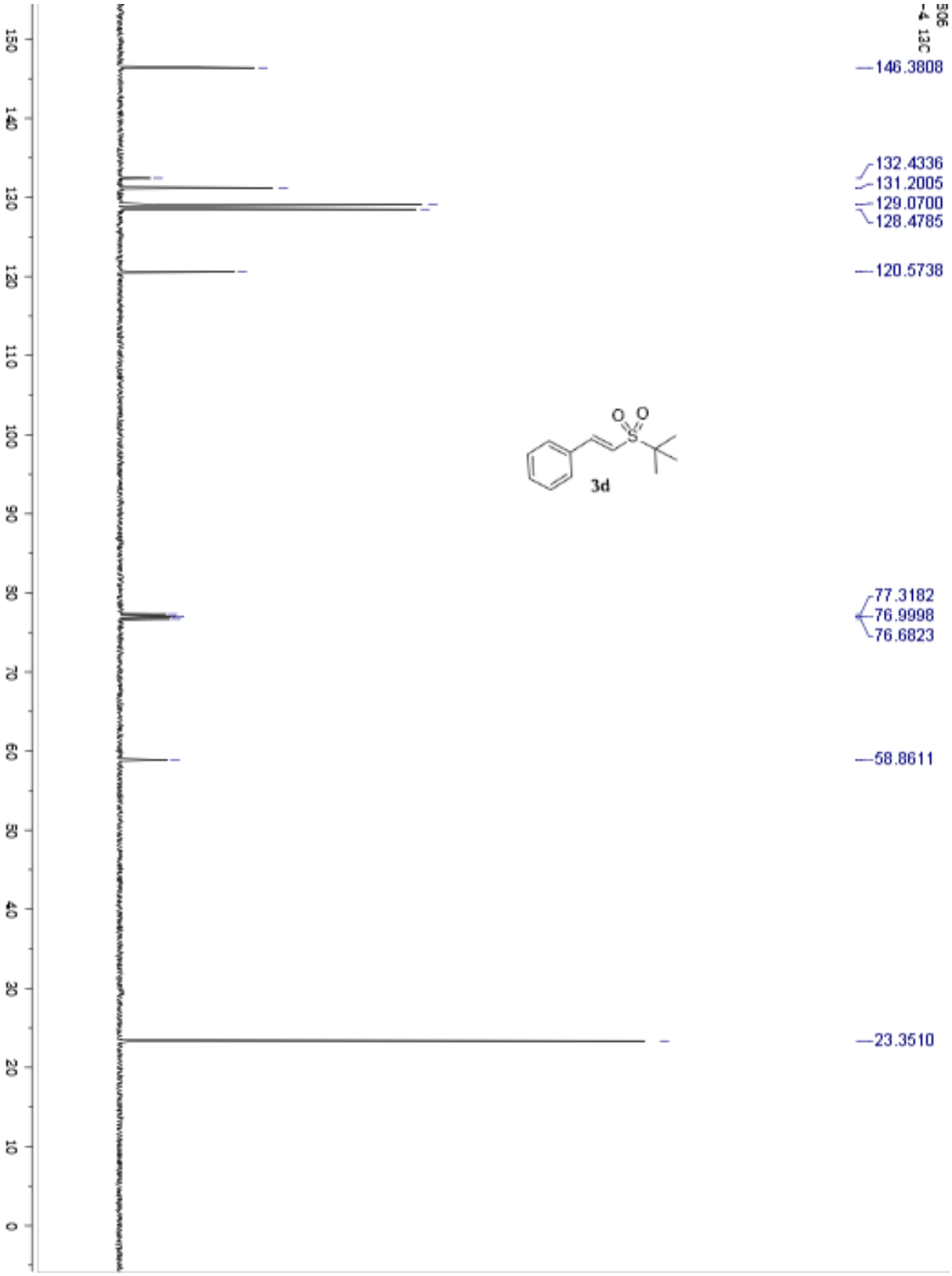
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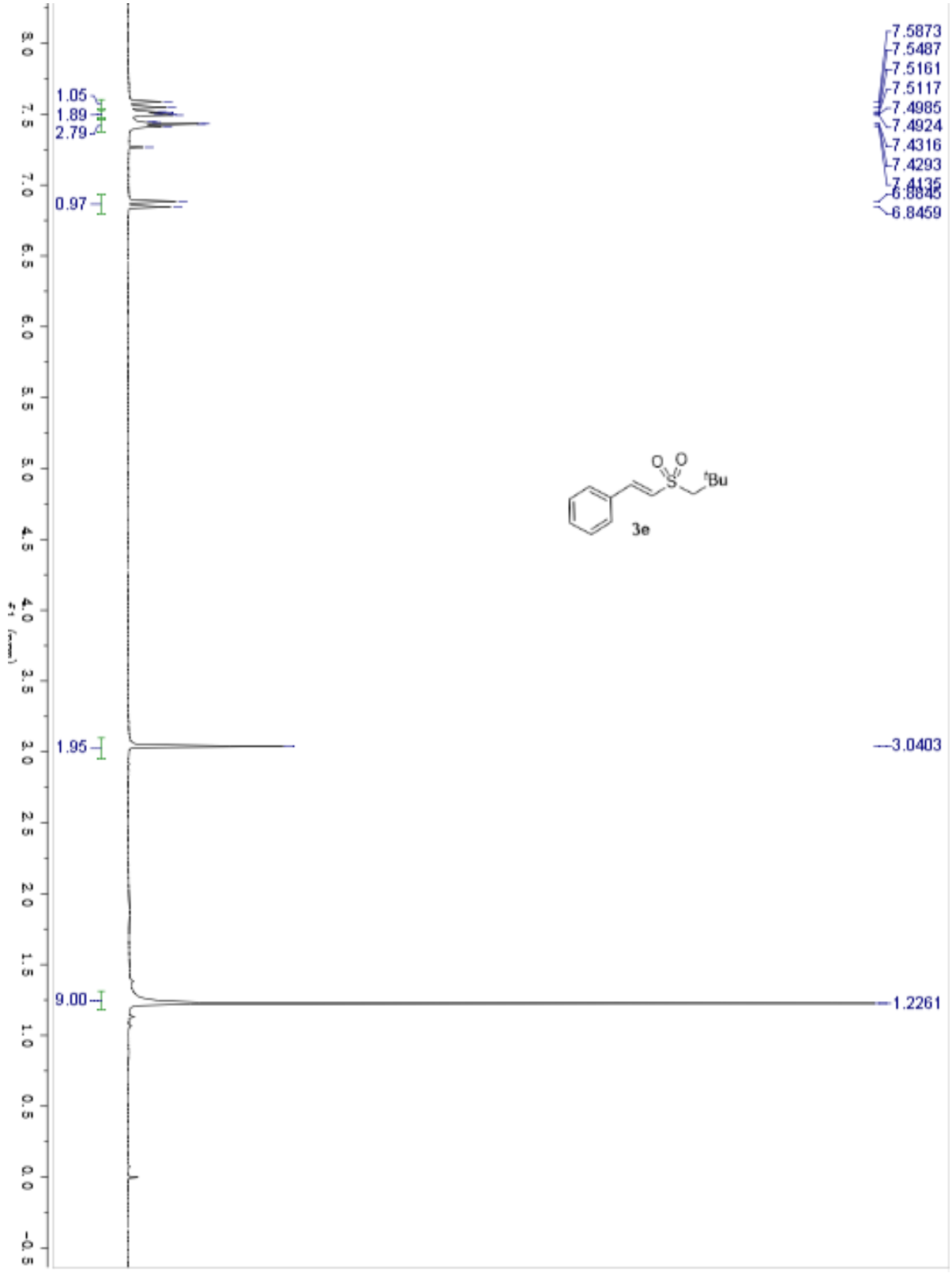
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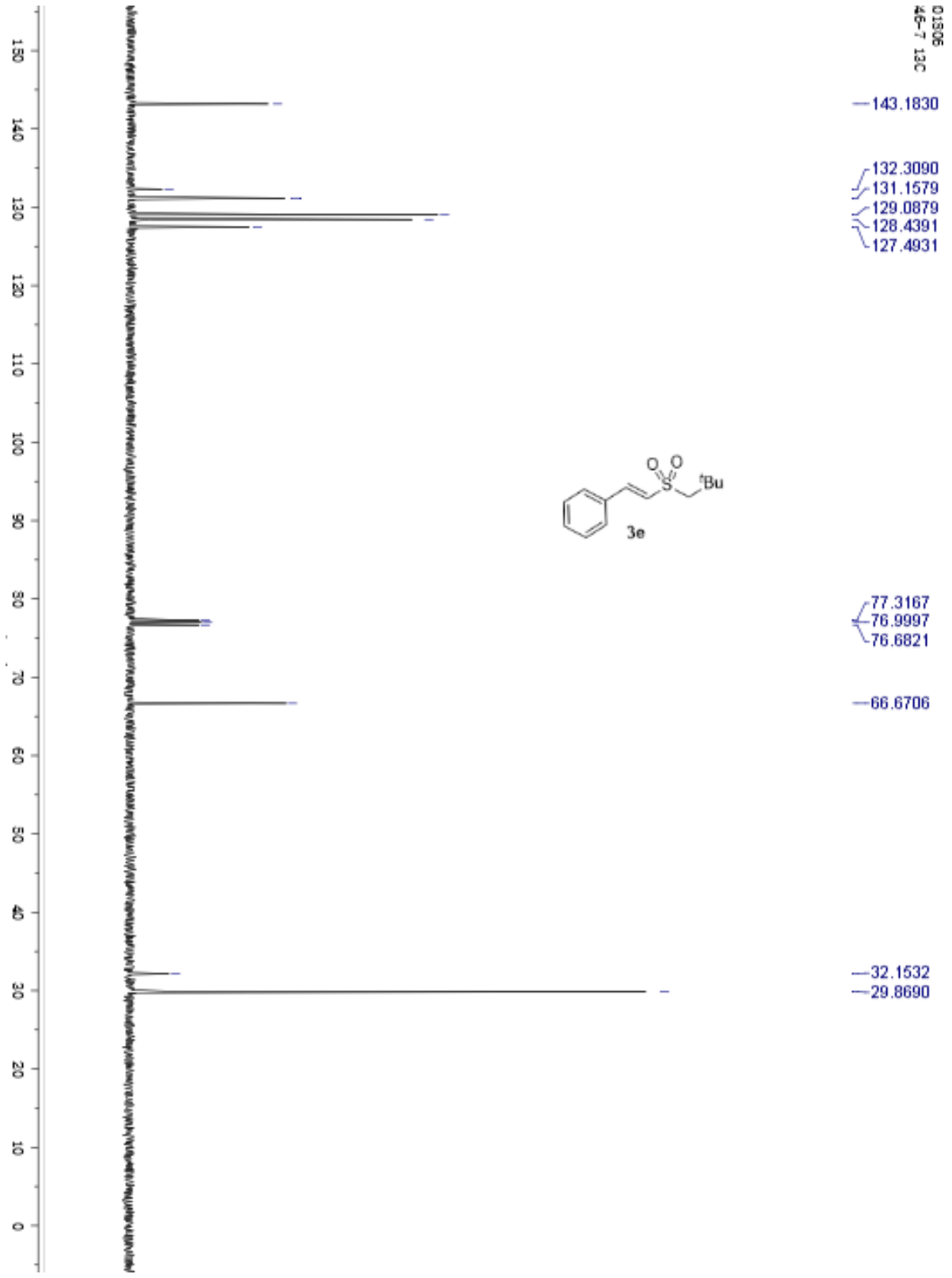
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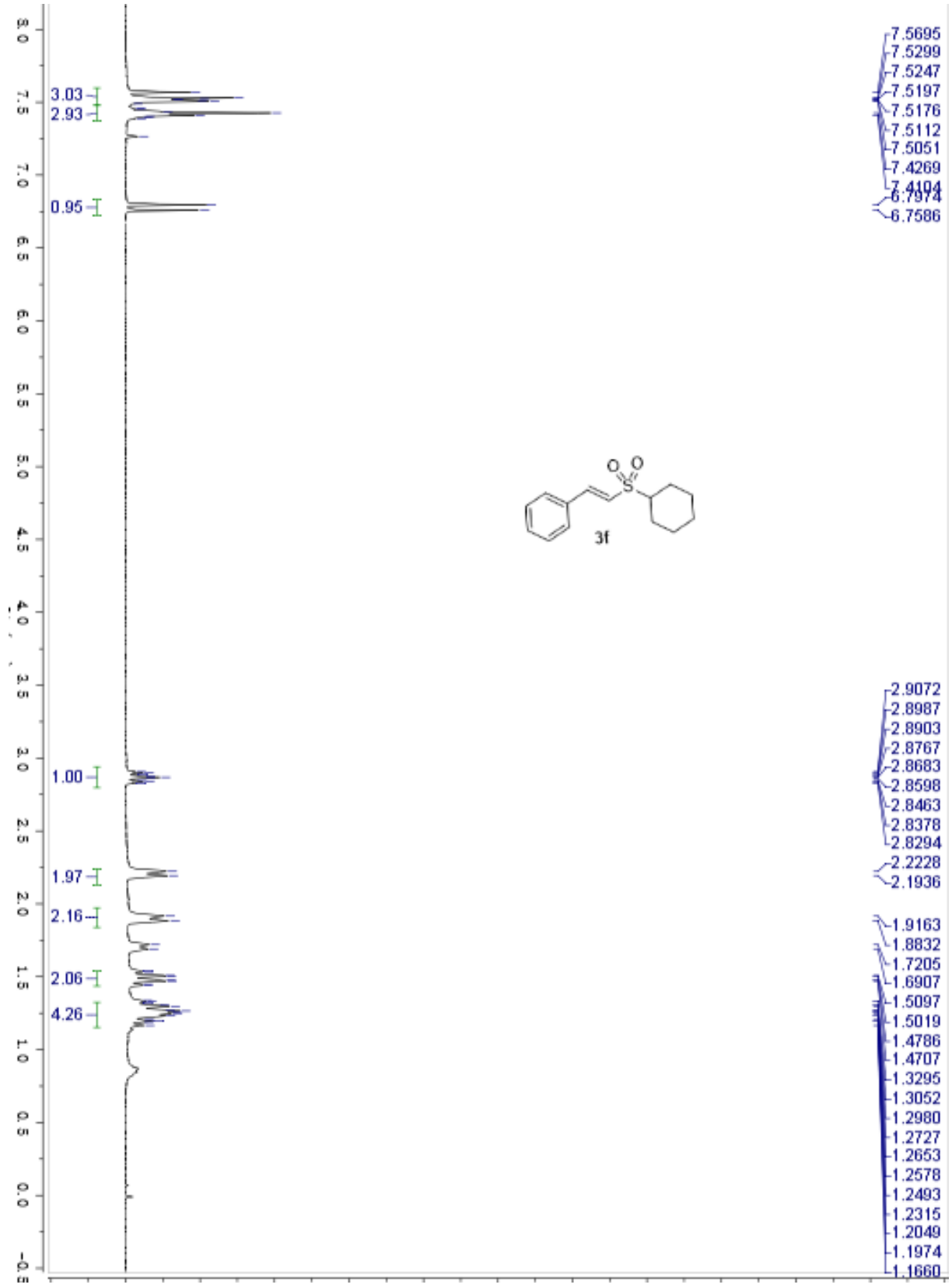
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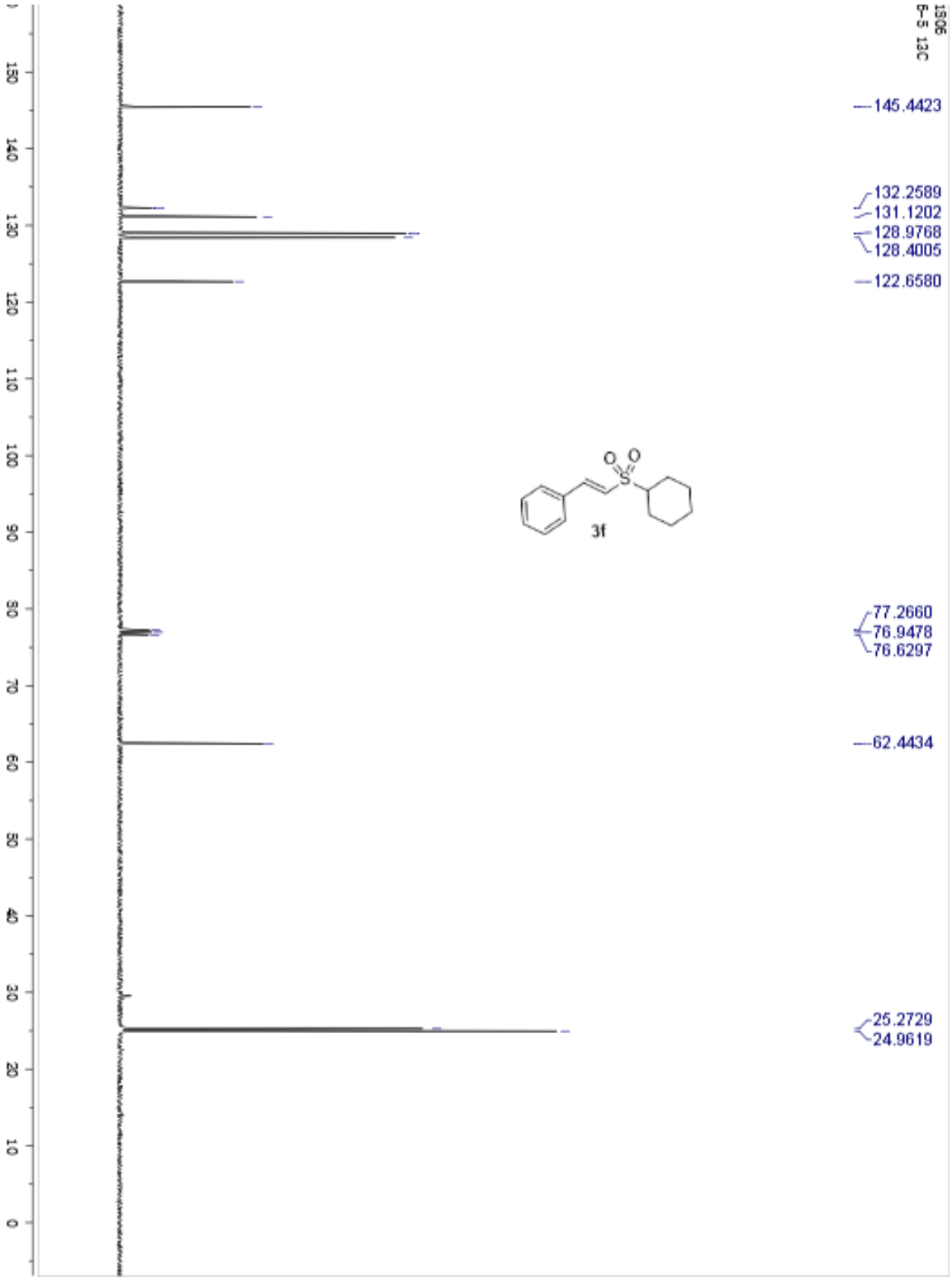
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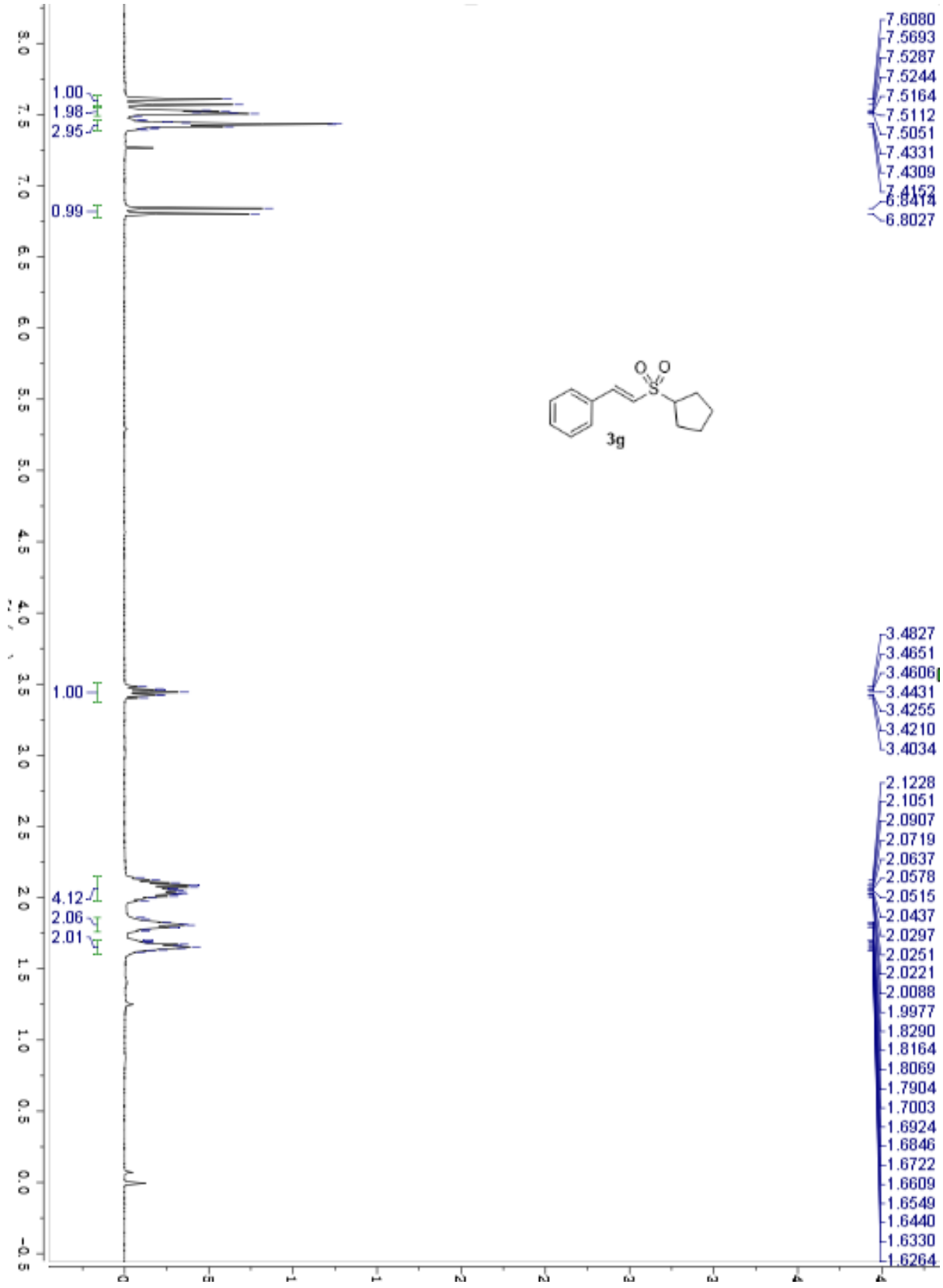
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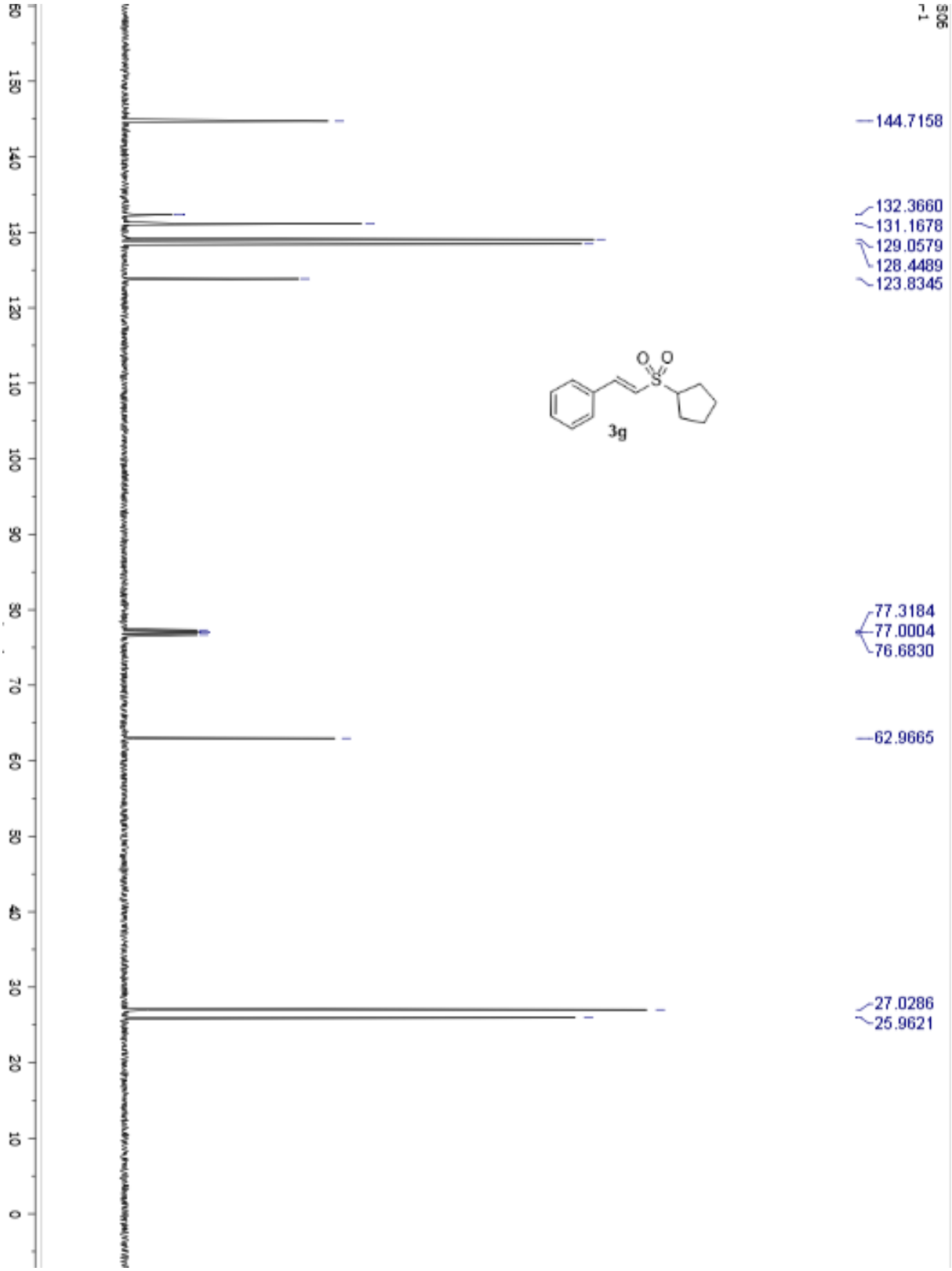
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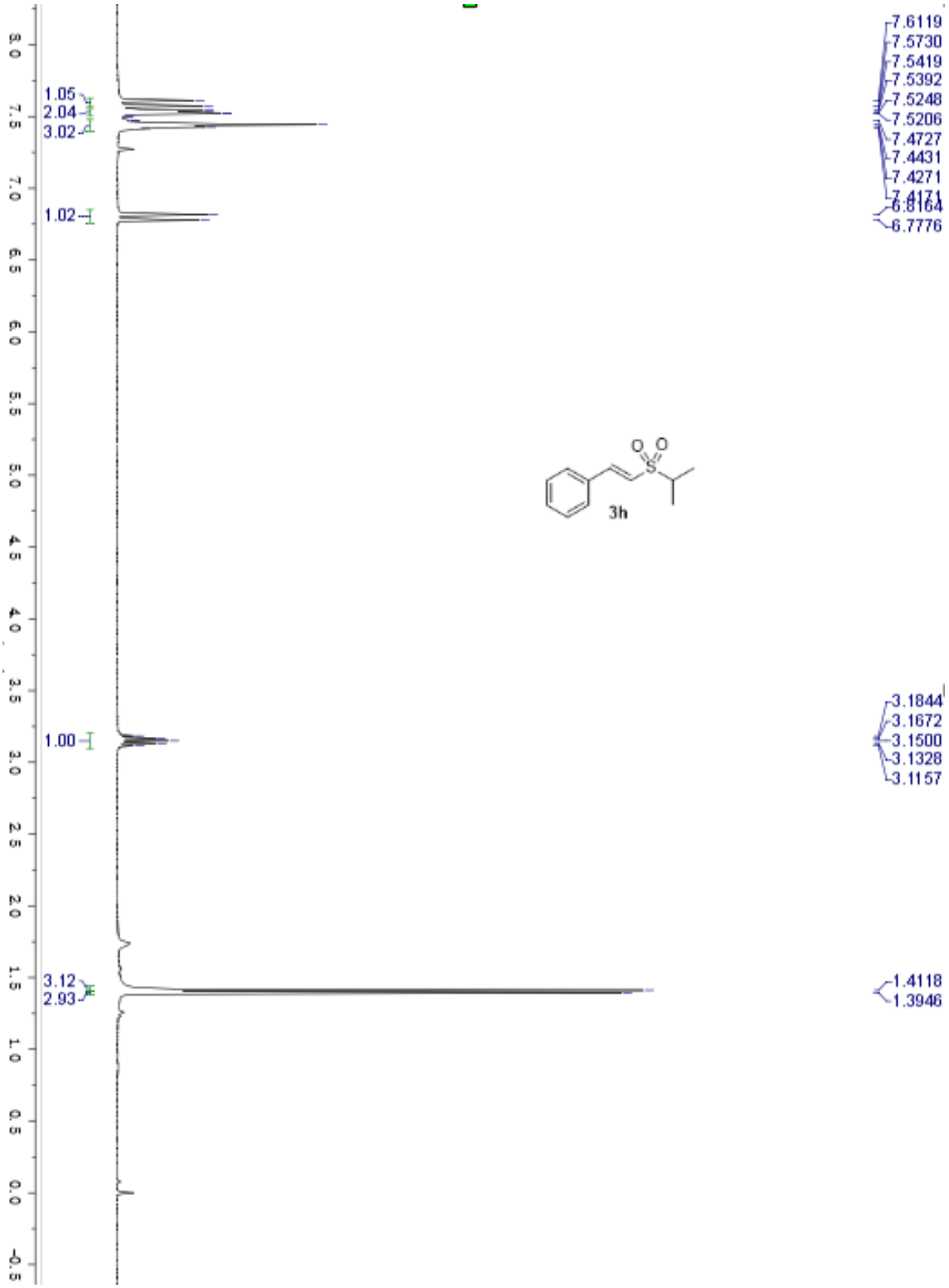
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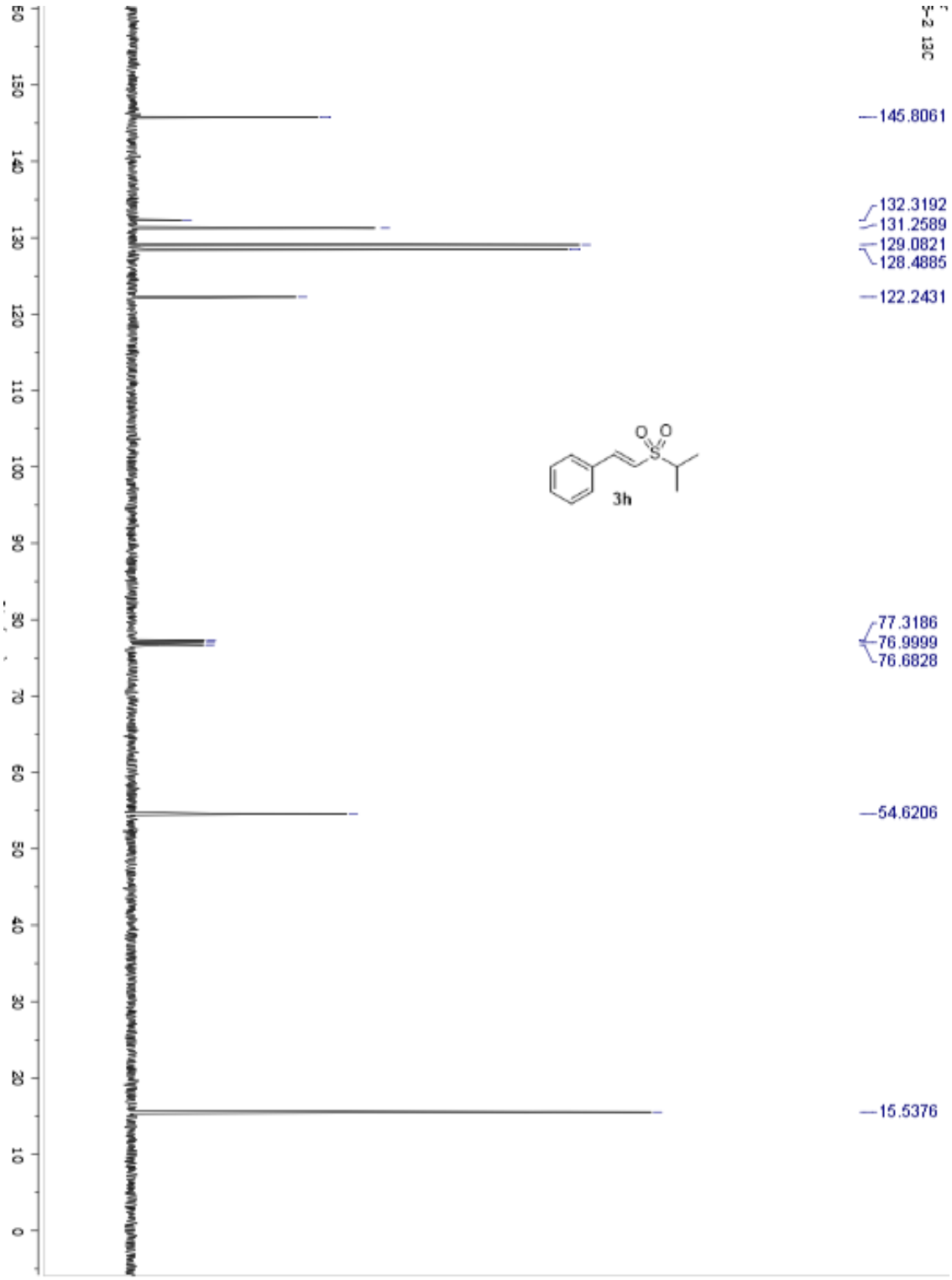
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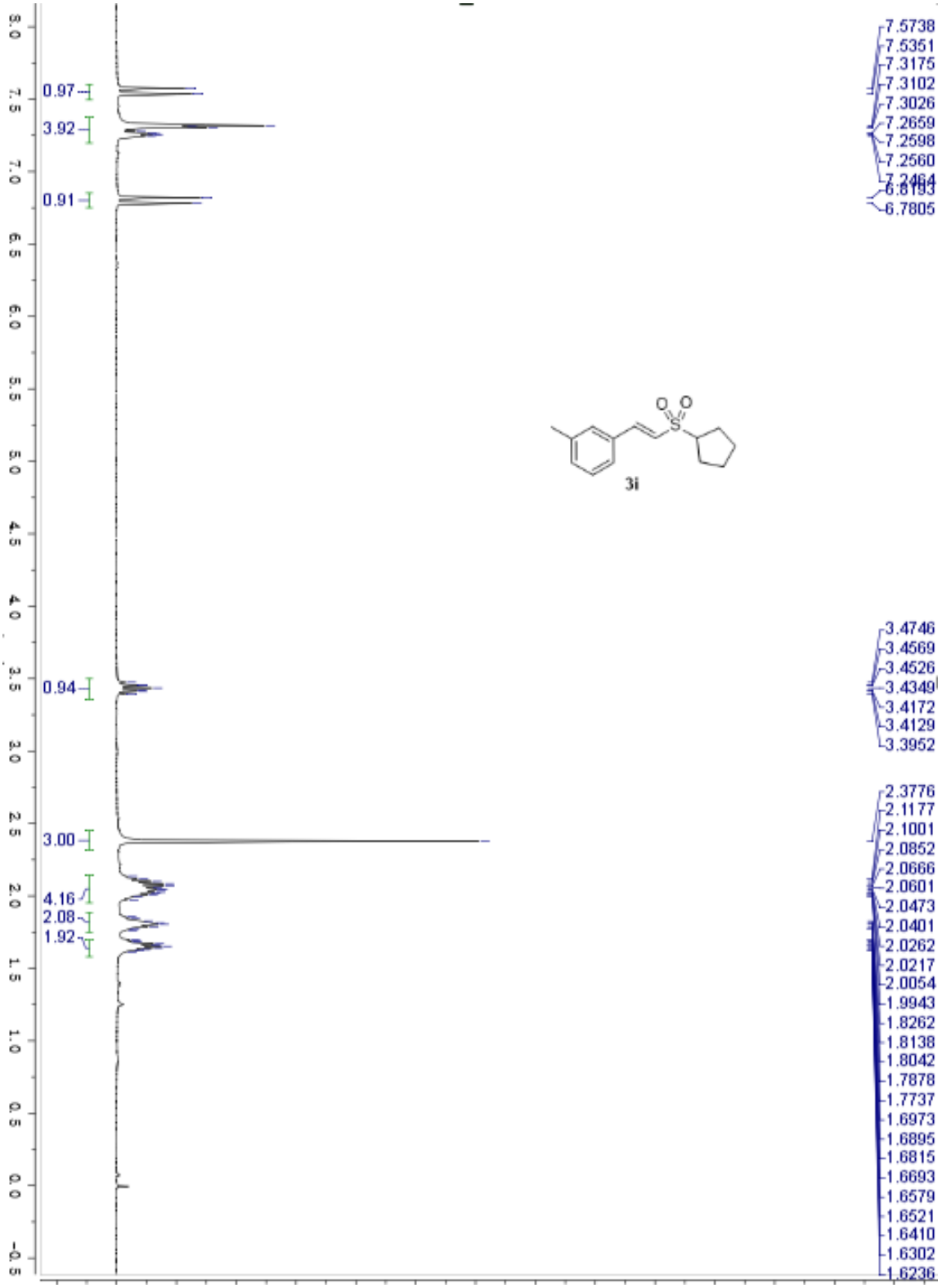
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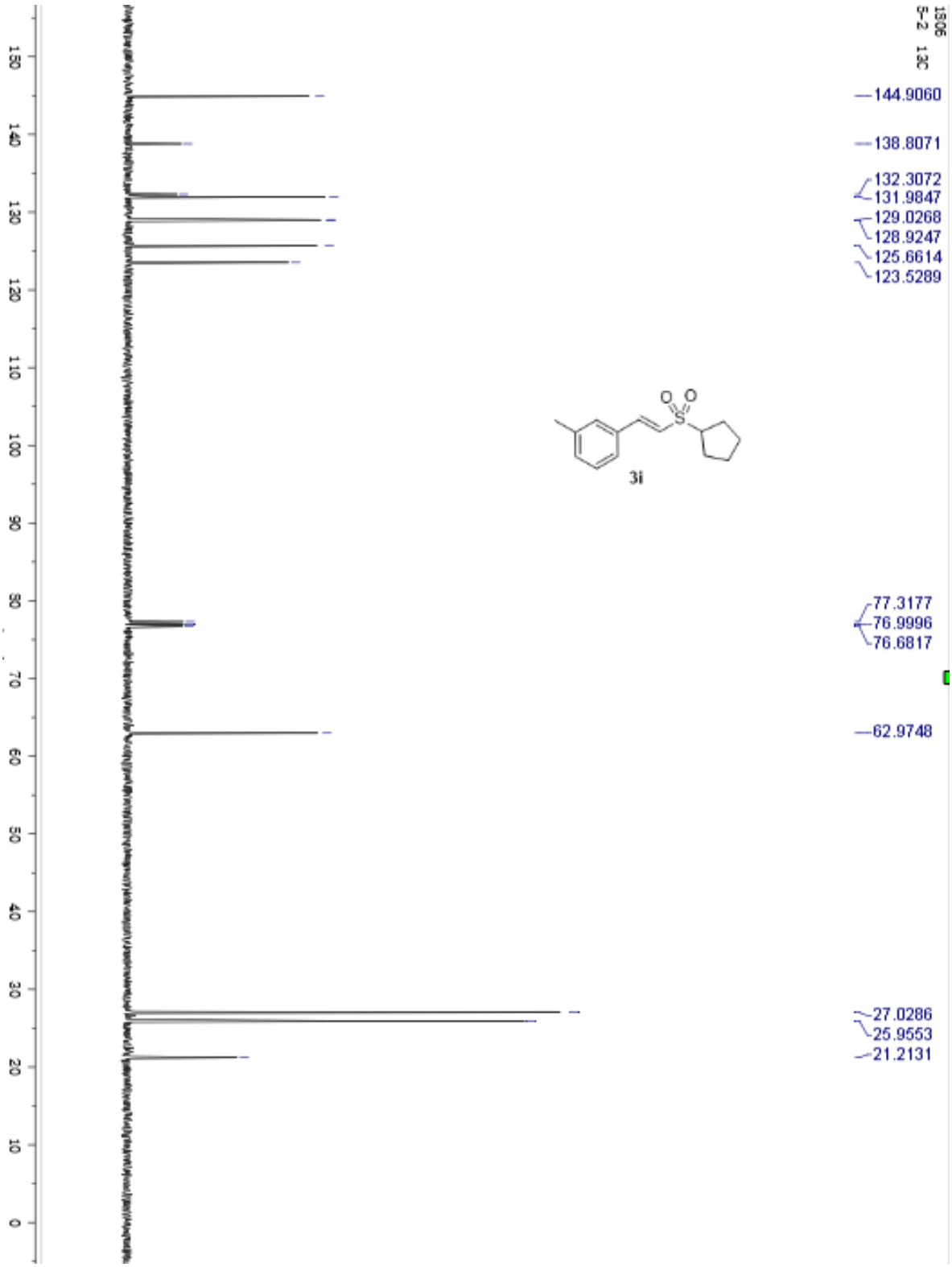
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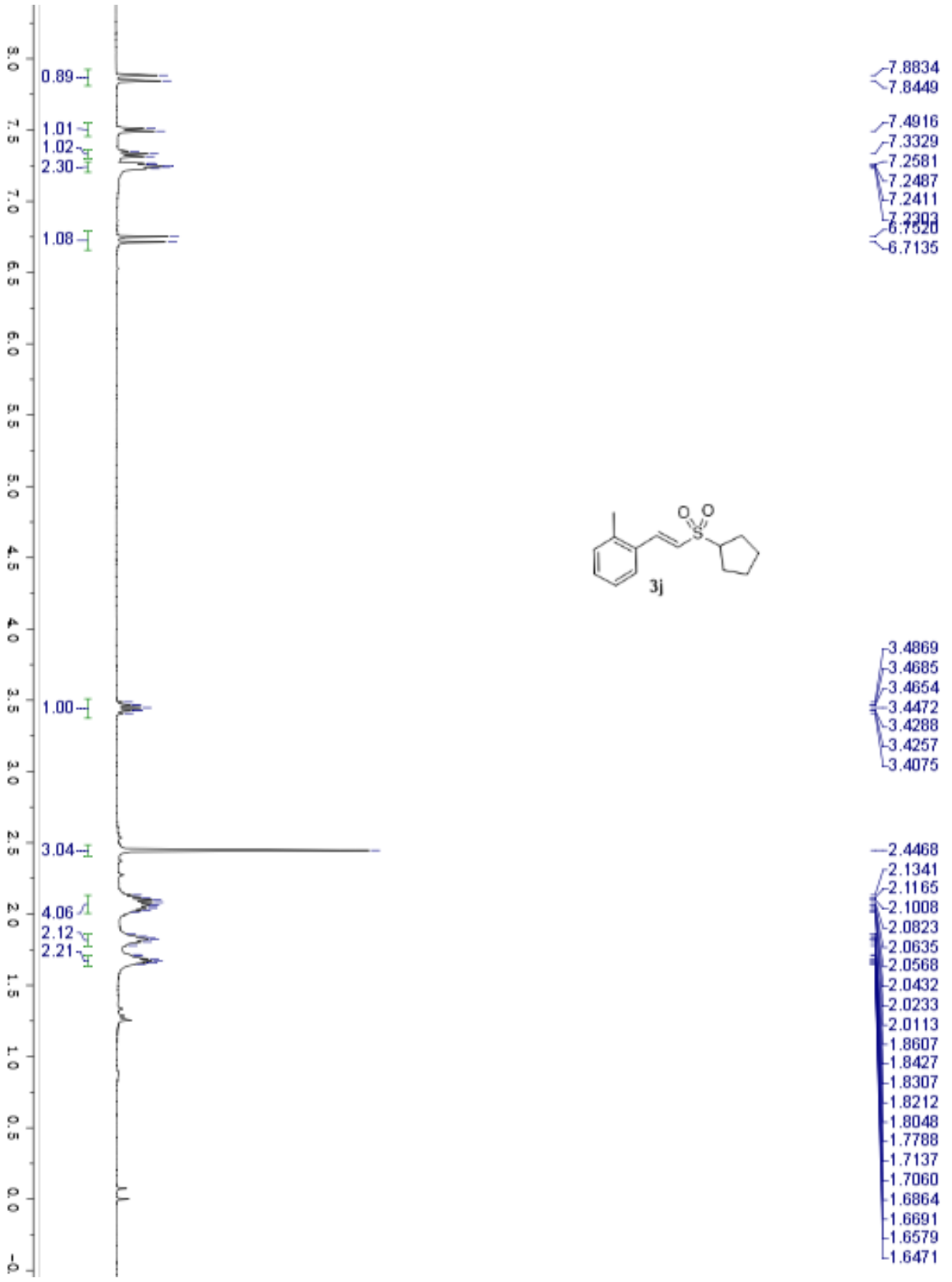
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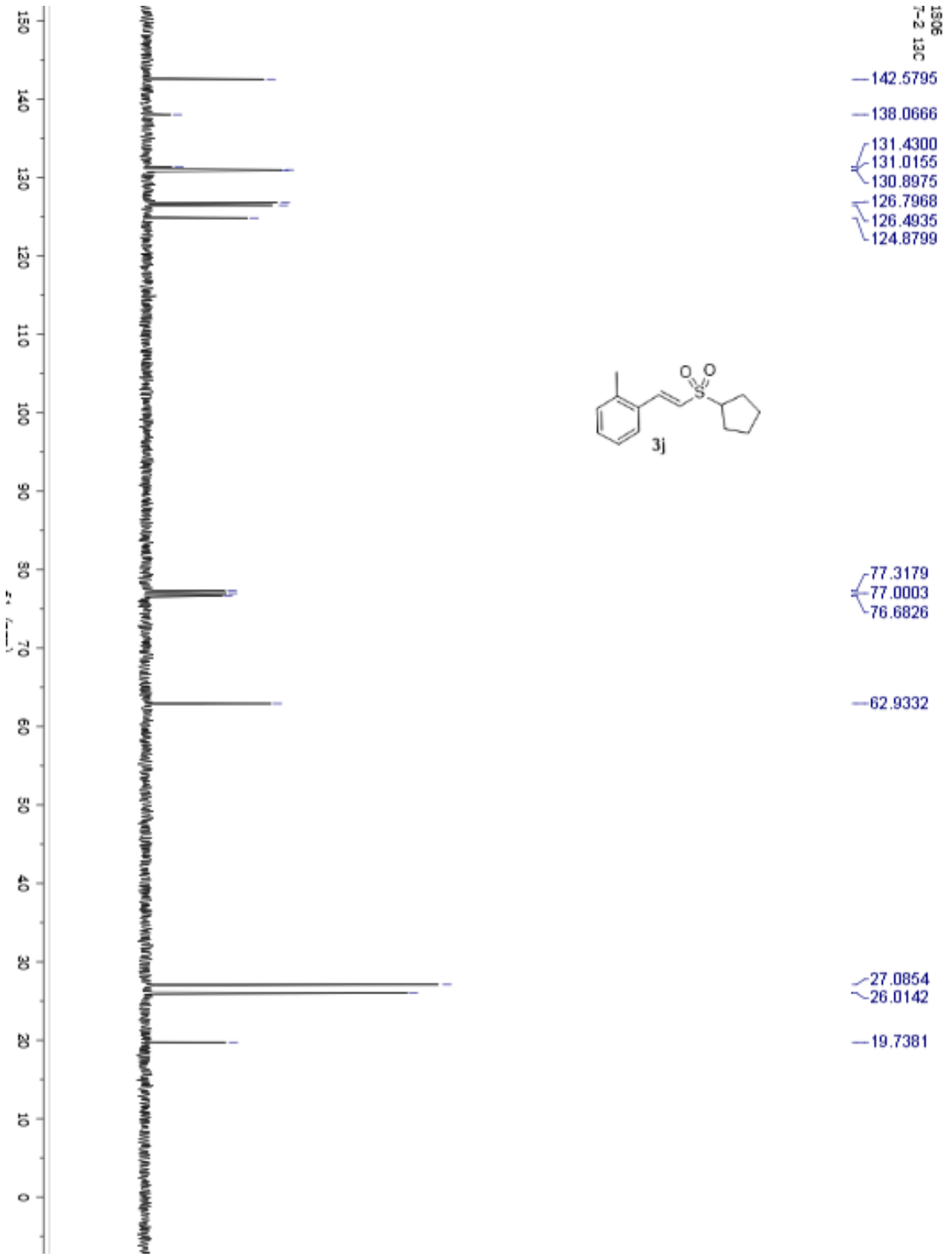
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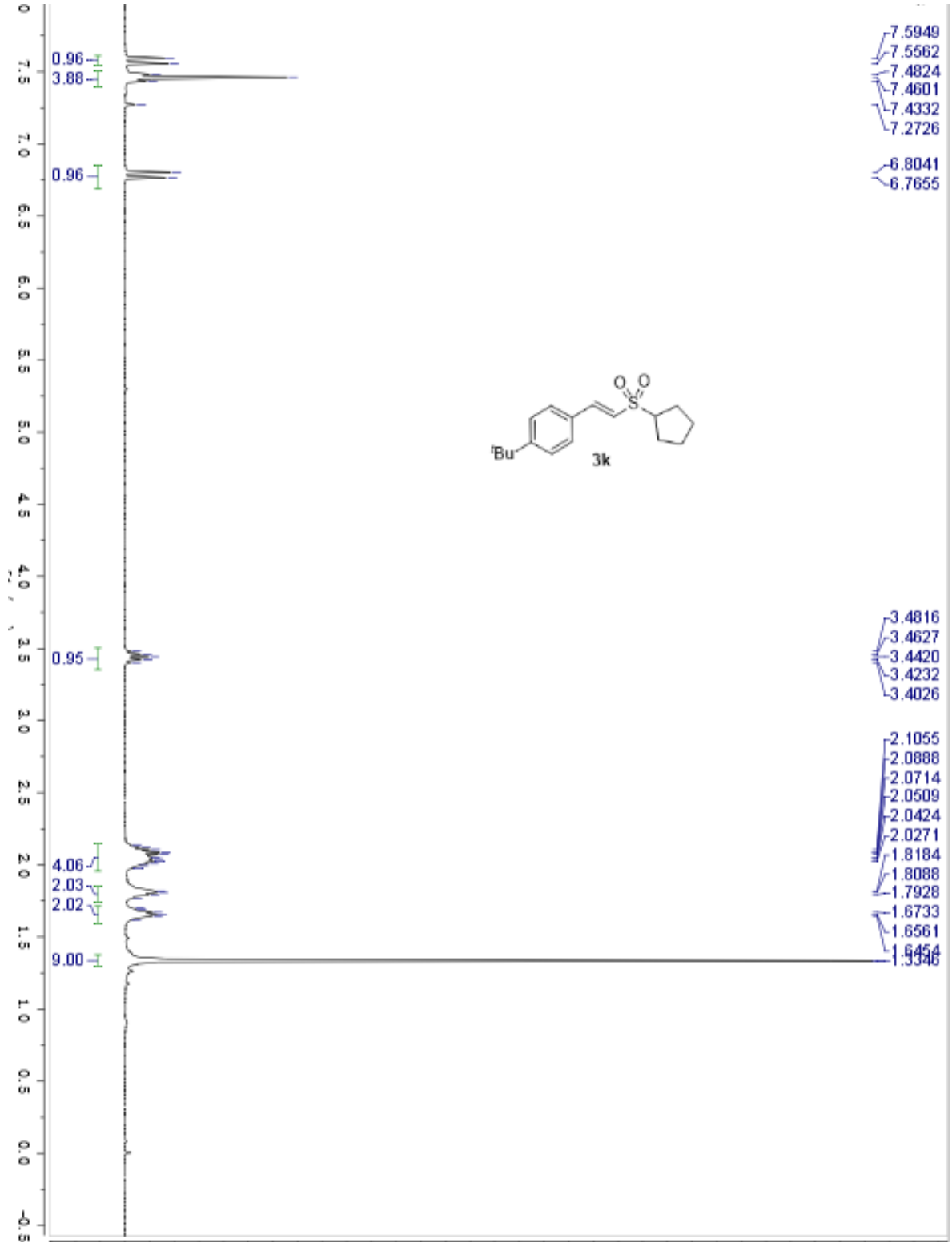
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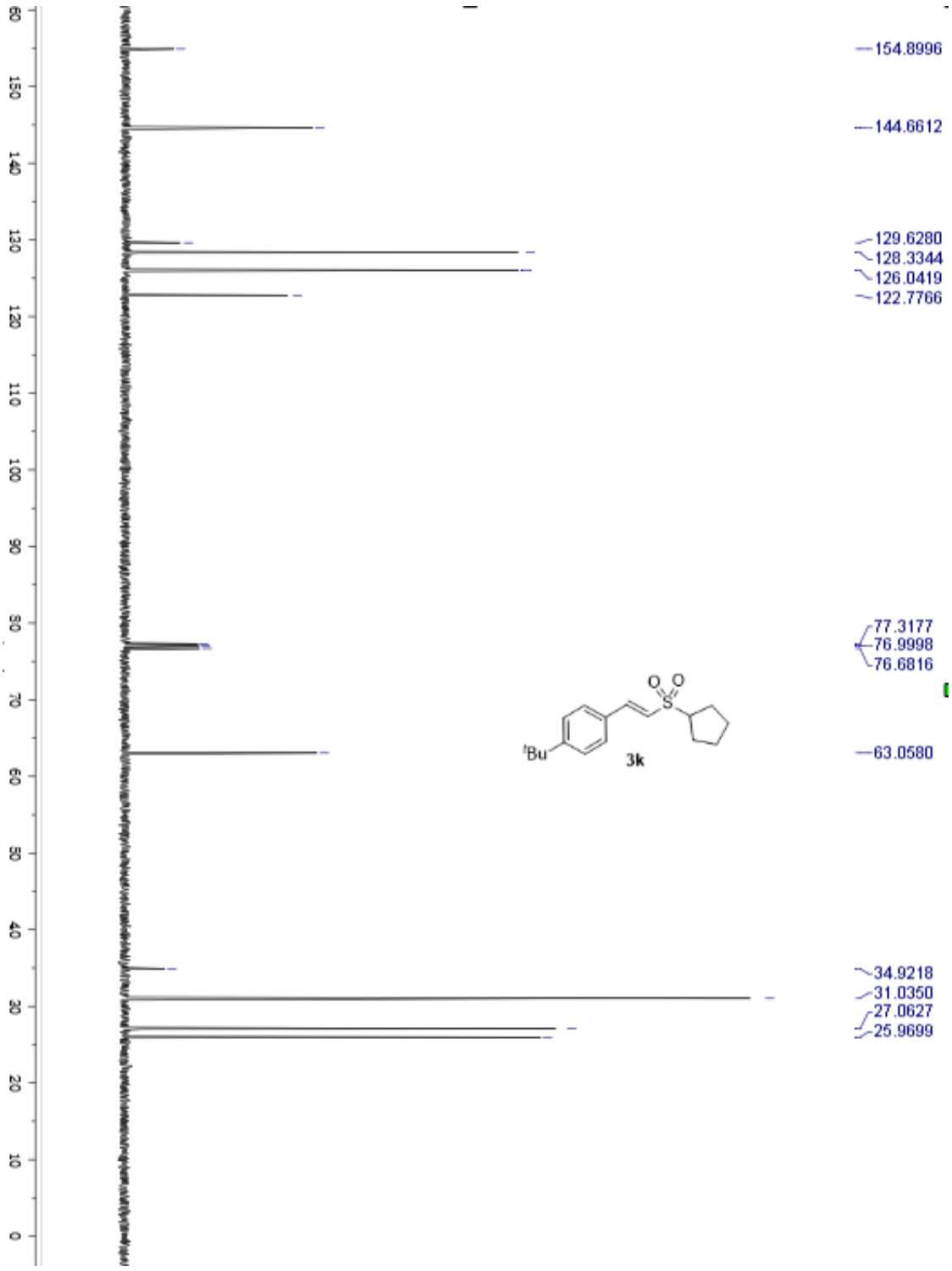
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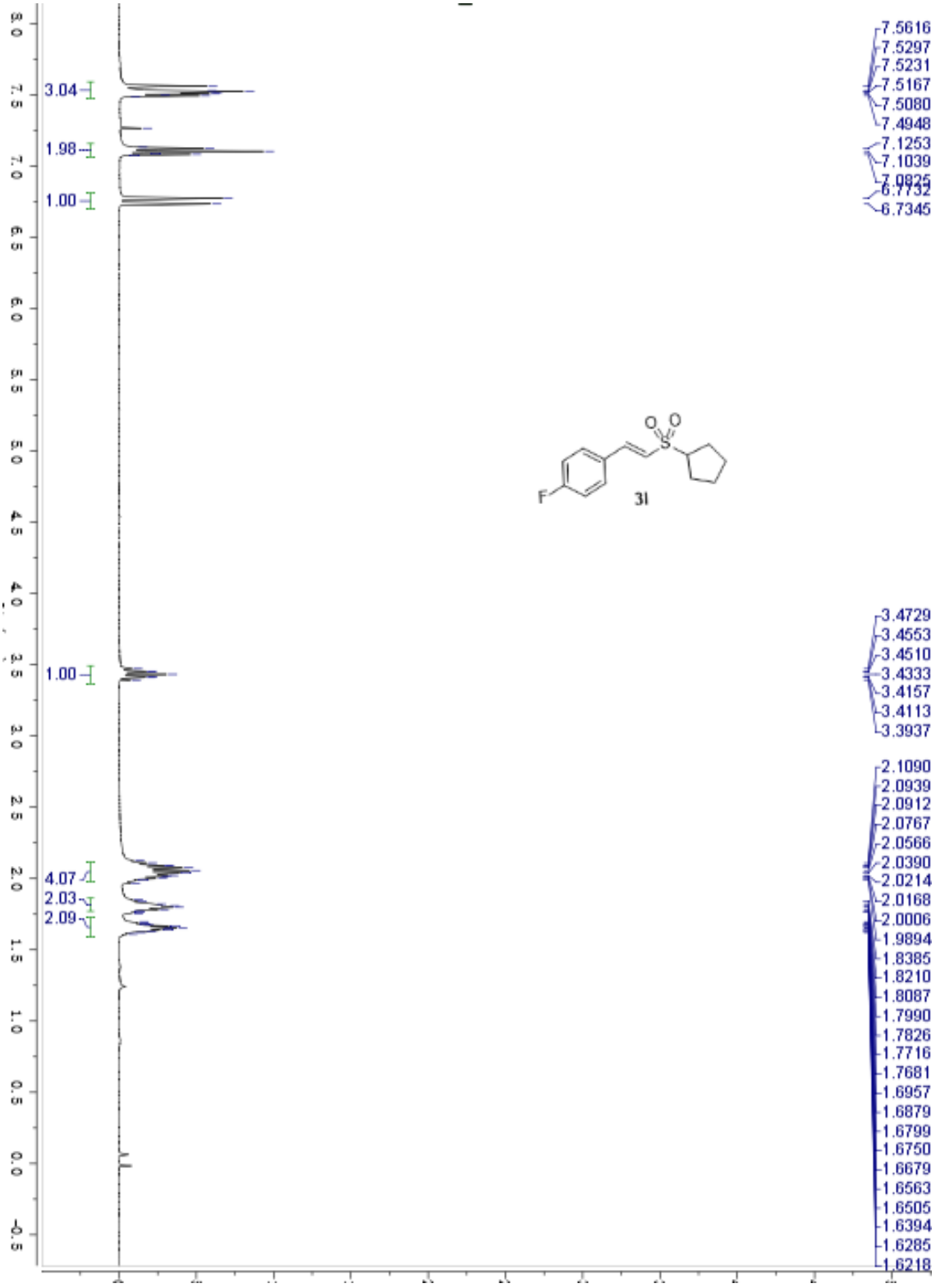
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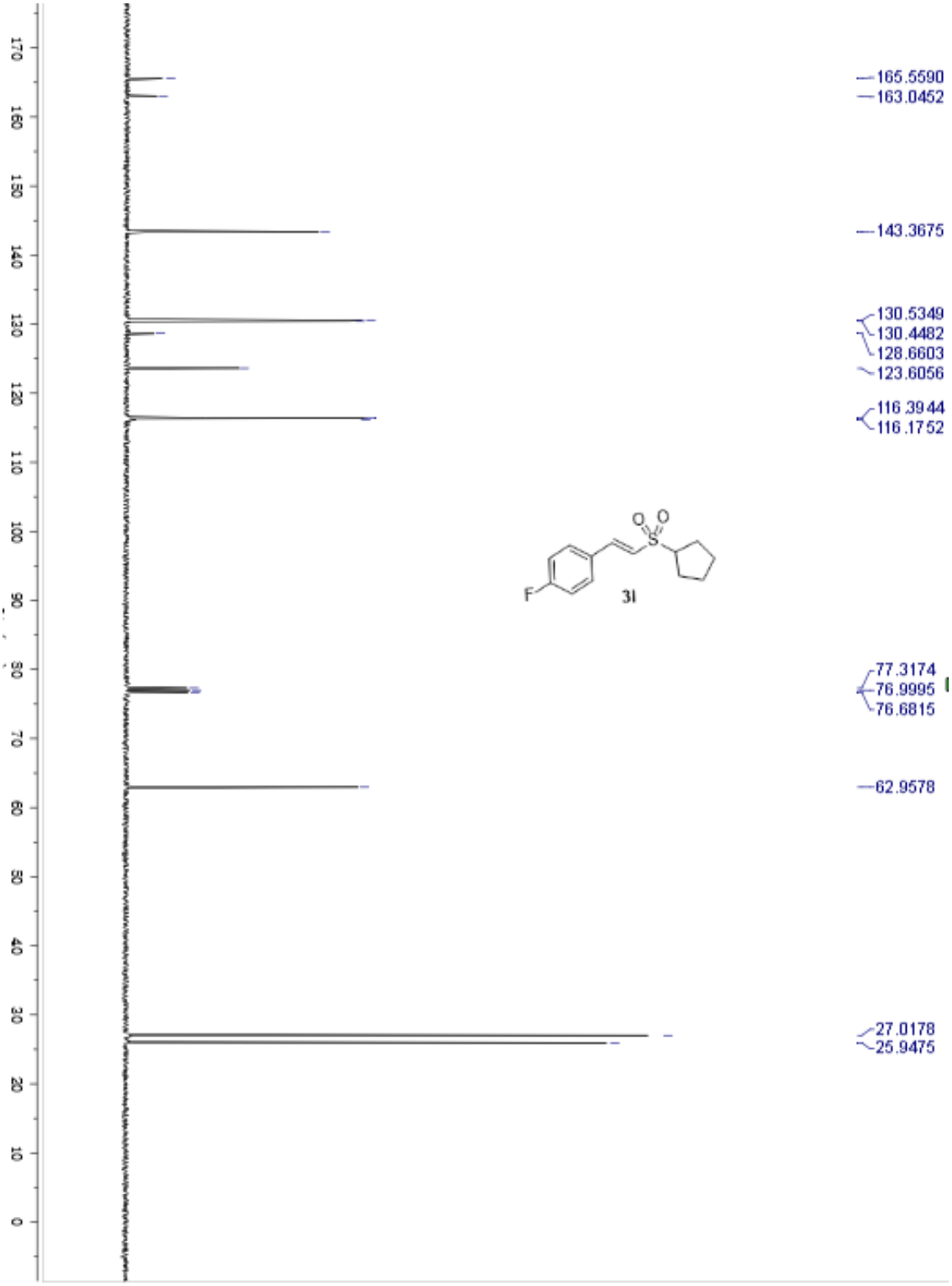
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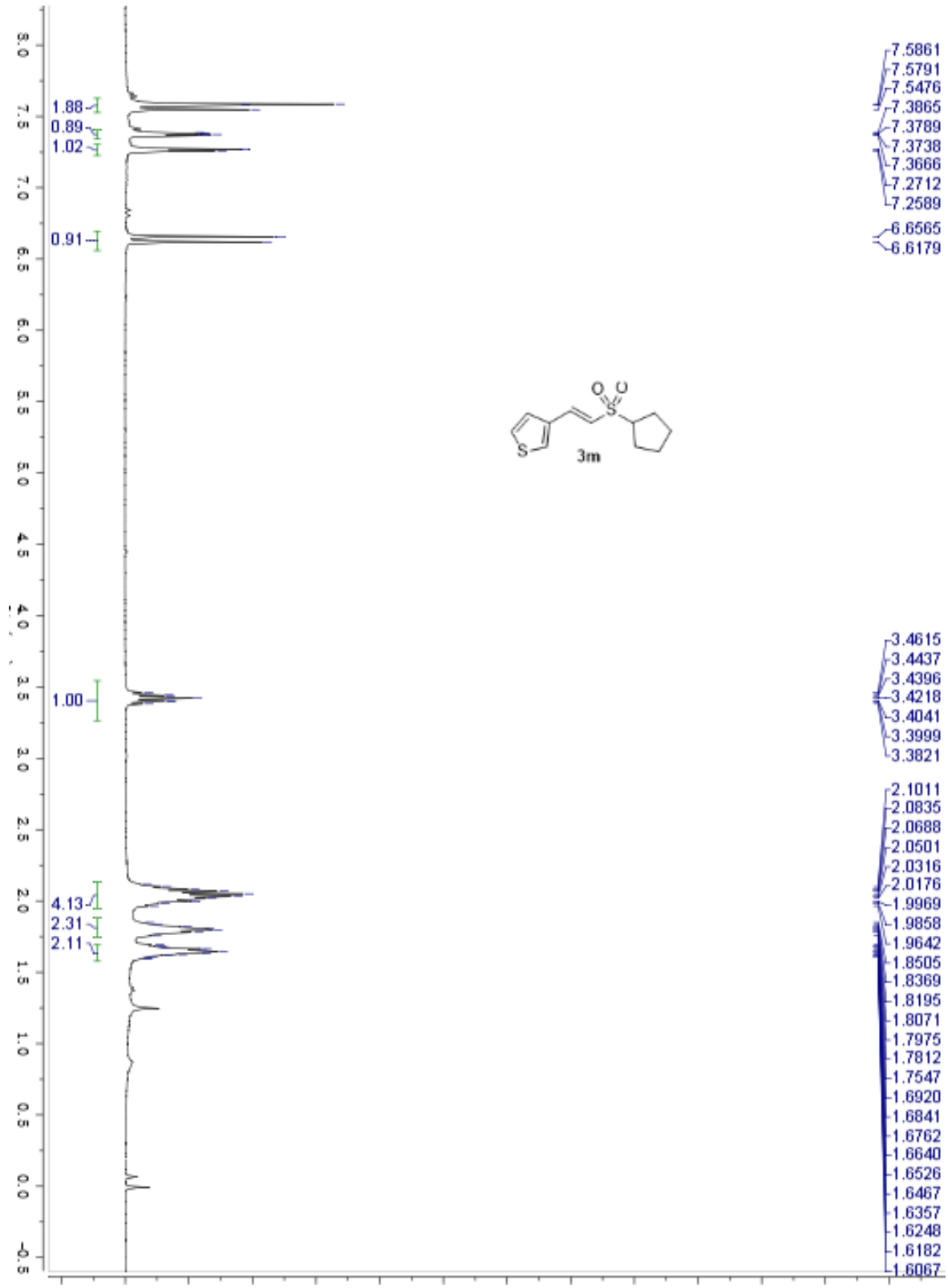
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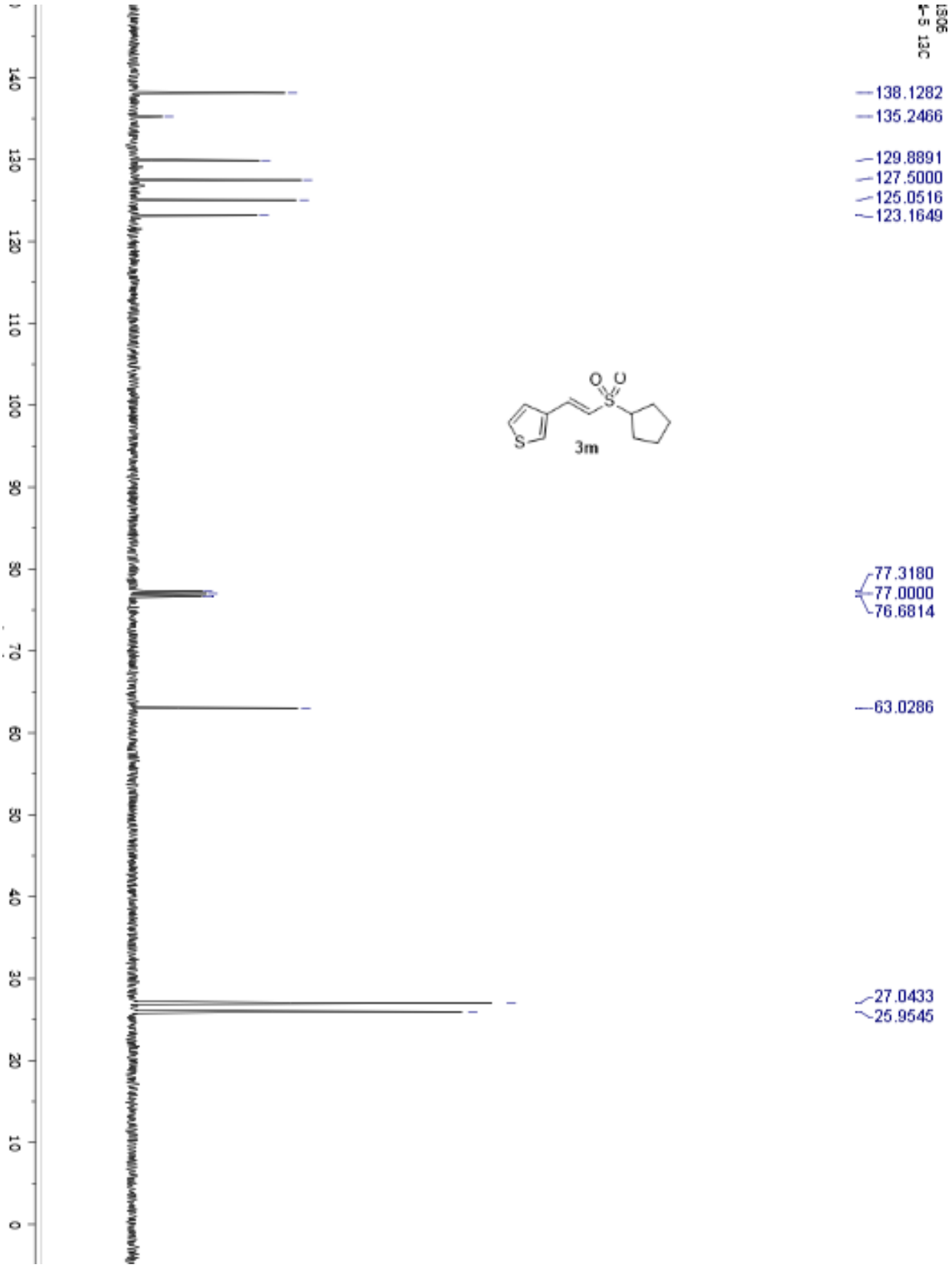
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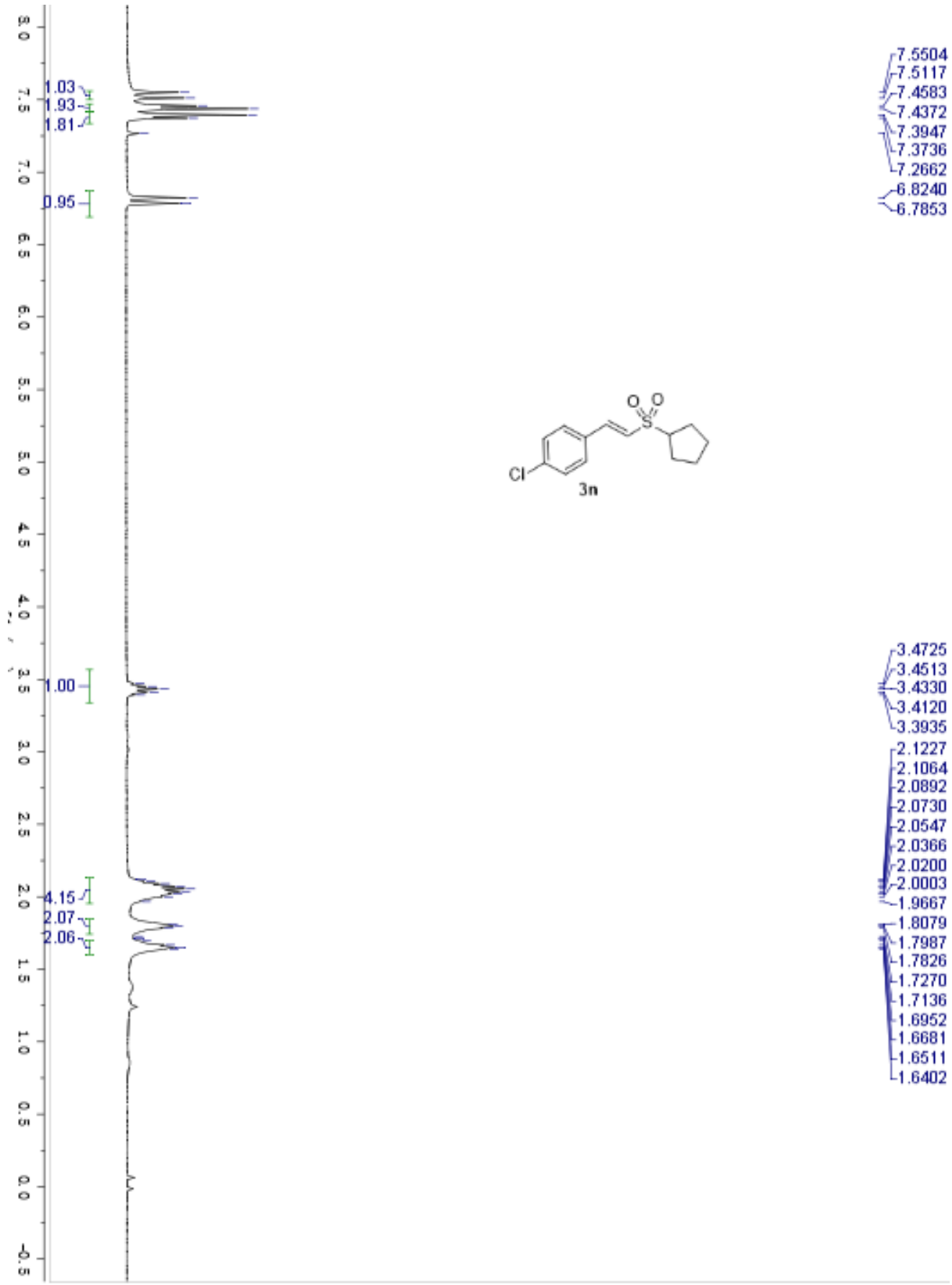
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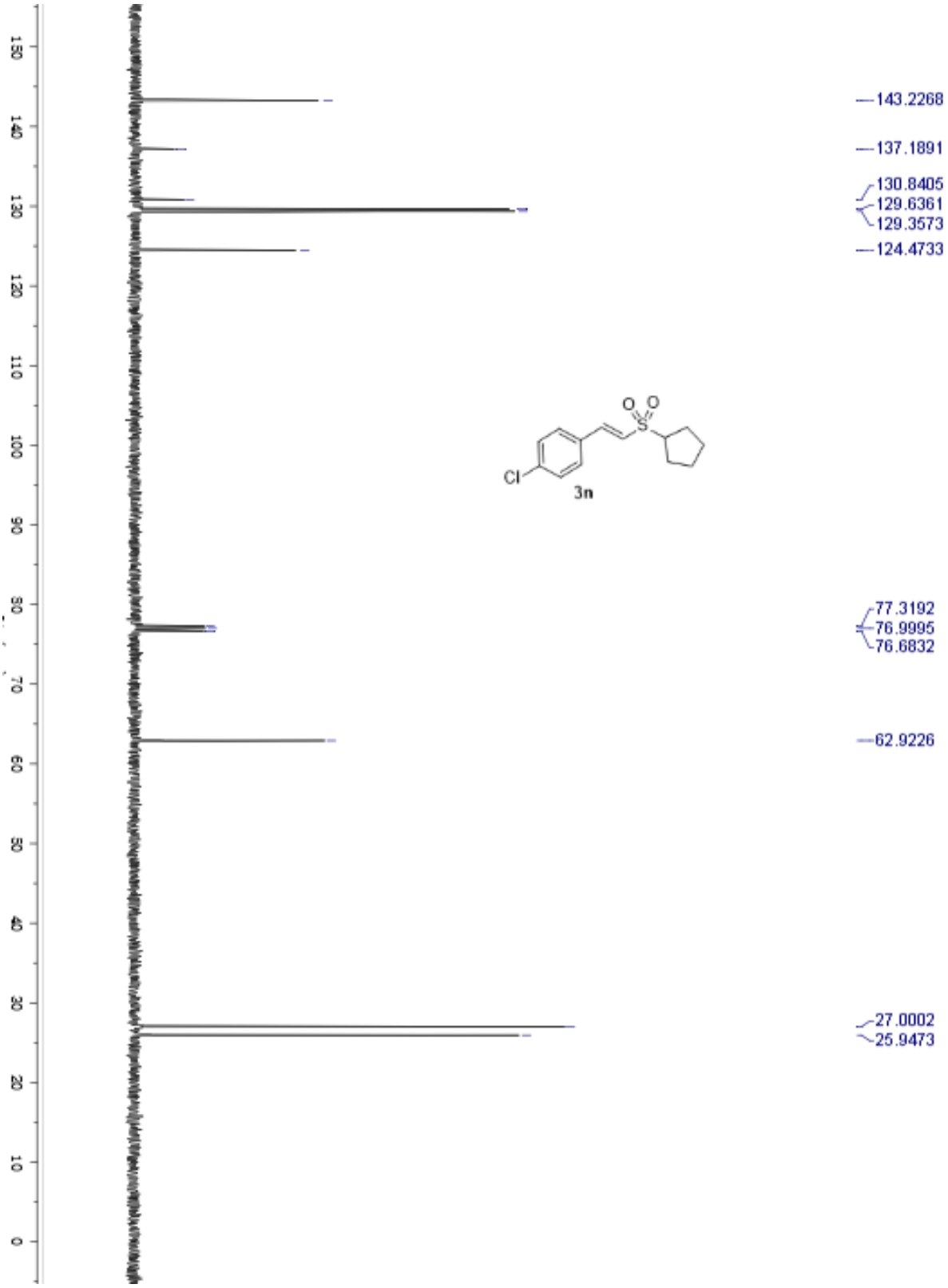
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Cl
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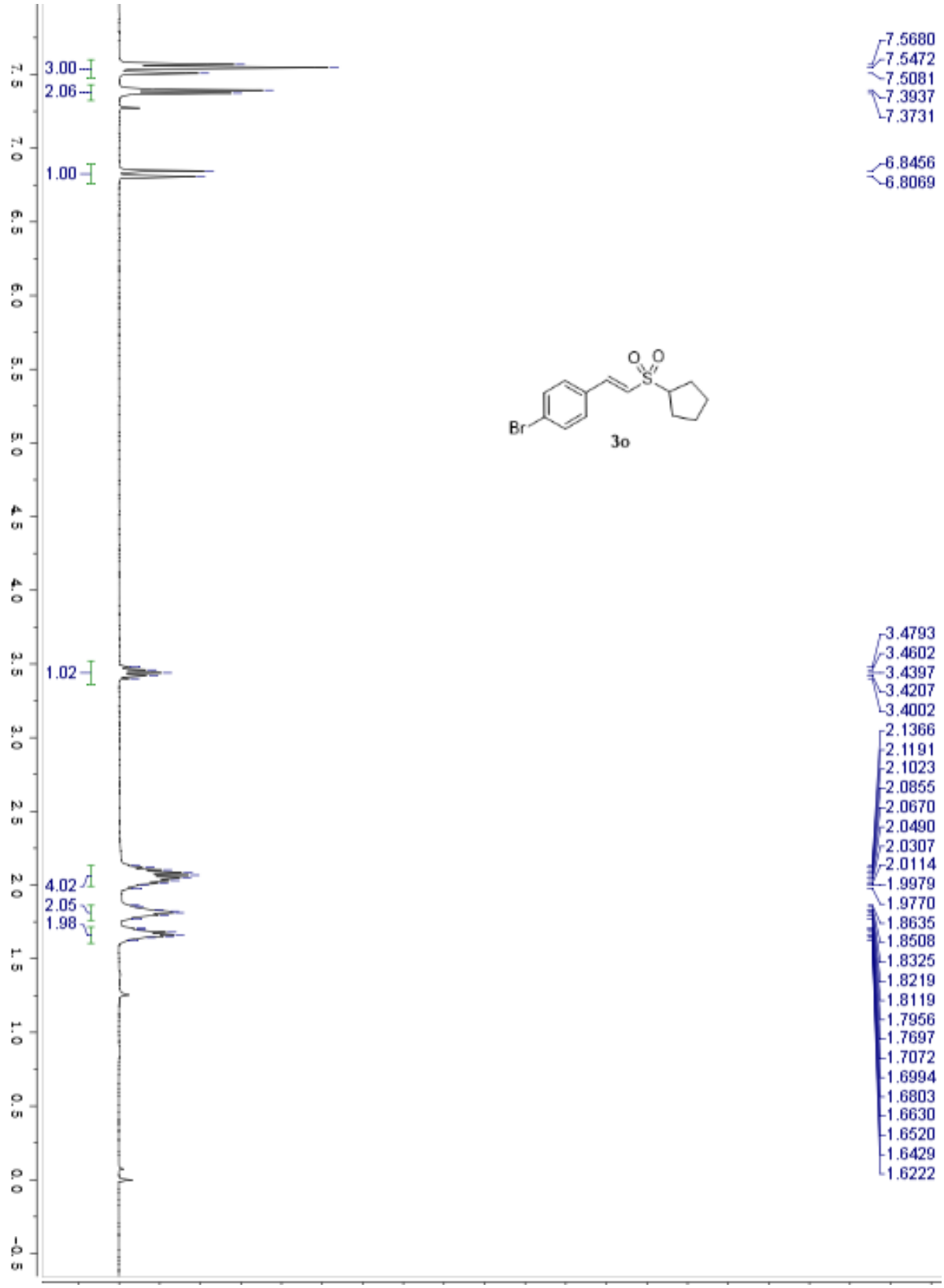
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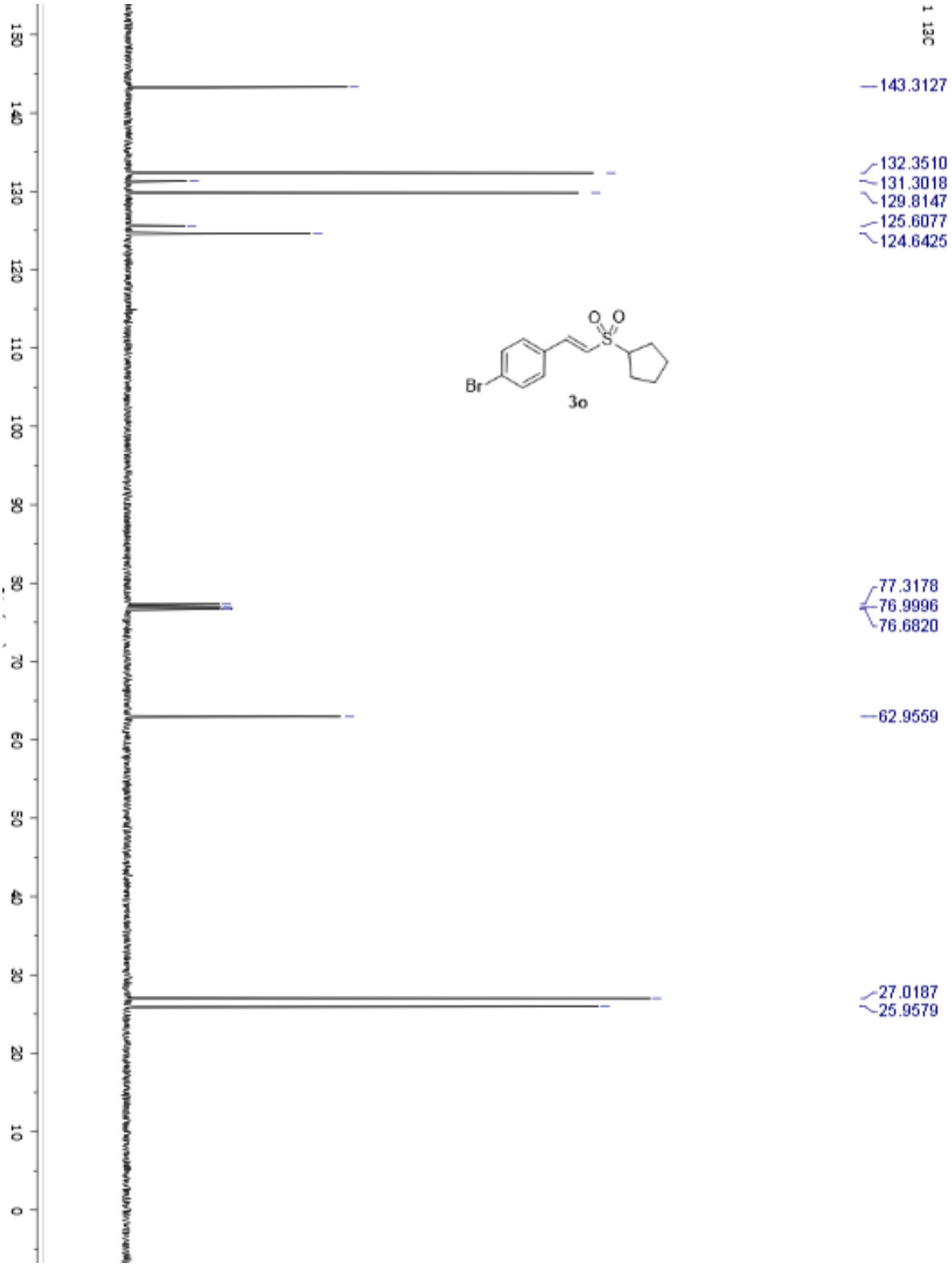
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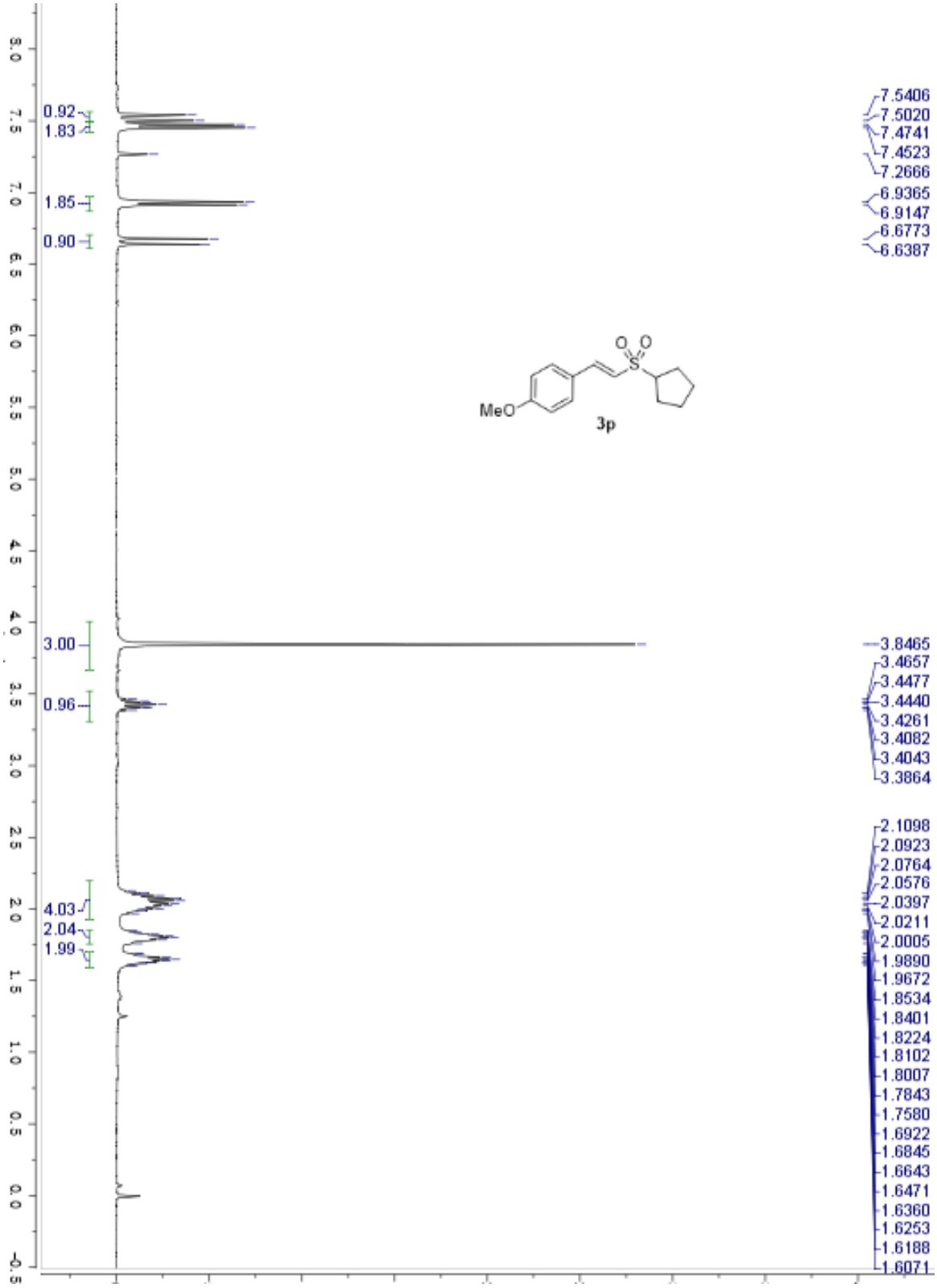
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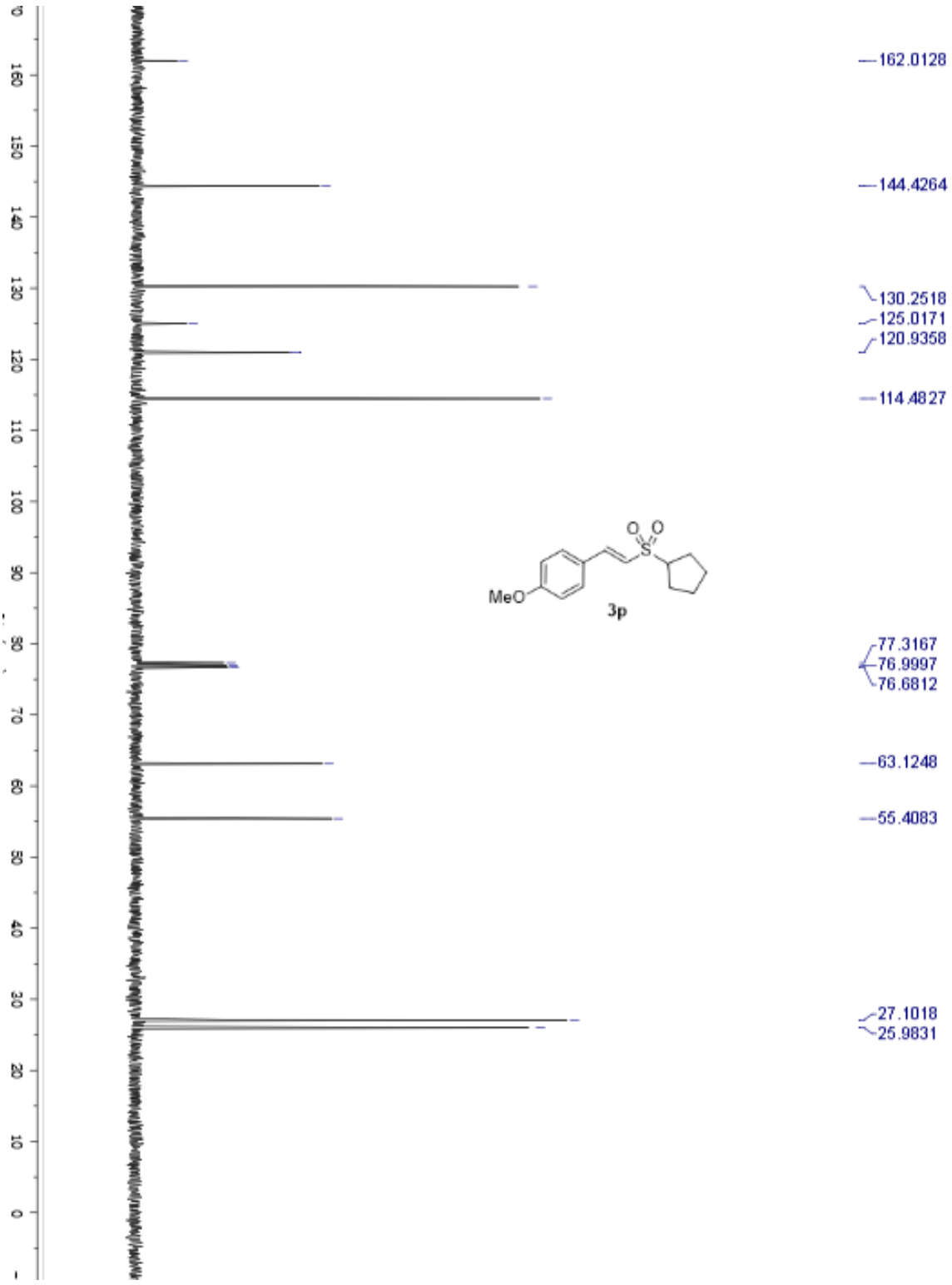
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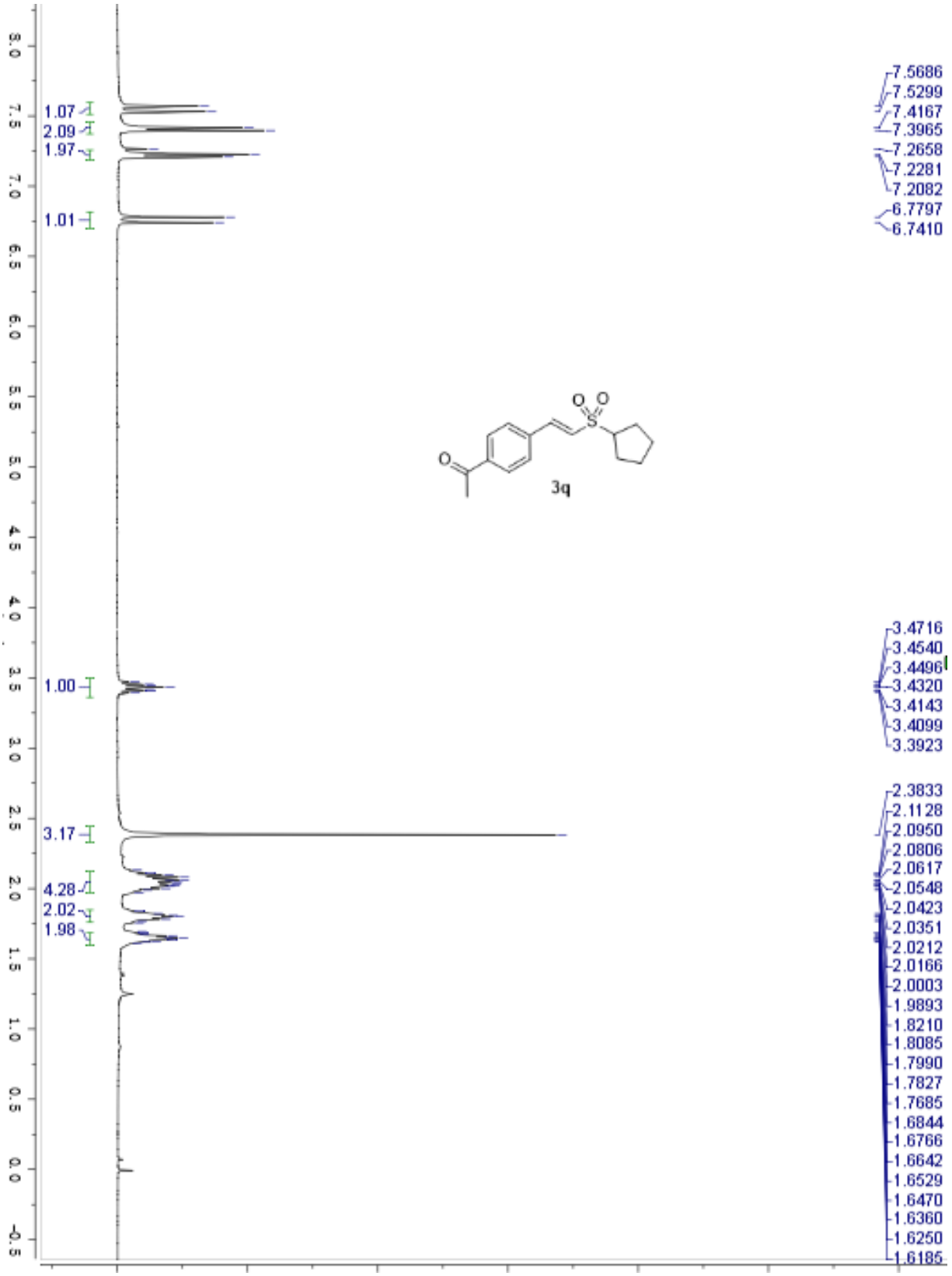
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O
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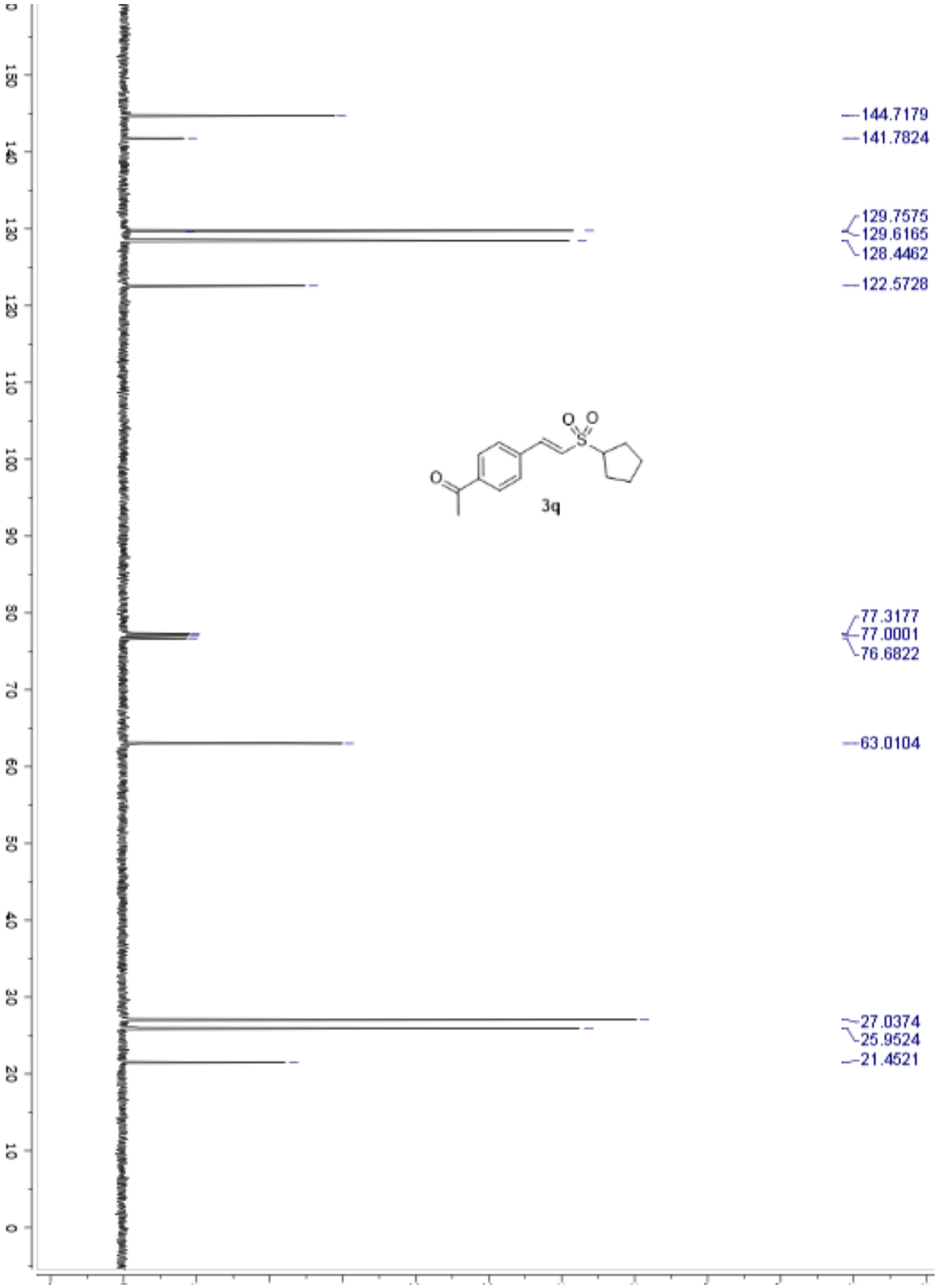
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O
O
3q