Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

for

Palladium-Catalyzed Cascade Cyclization for the Construction of *spiro*-N,O-Acetals†

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General Procedures and New Compounds Characterization

Content:

1. General Considerations.

All commercially available compounds were used as received. ^{1}H and ^{13}C spectroscopy were recorded on an Agilent-400 MHz (400 MHz for ^{1}H; 376 MHz for ^{19}F; 100 MHz for ^{13}C). $CDCl_3$ was purchased from J&K Company. The chemical shifts (δ) are given in parts per million relative to internal standard TMS (0 ppm for ^{1}H), $CDCl_3$ (77.0 ppm for ^{13}C). Flash column chromatography was performed on silica gel 60 (particle size 300-400 mesh ASTM, purchased from Yantai, China) and eluted with petroleum ether/ethyl acetate. All solvents were dried and purified according to the procedure from 'Purification of Laboratory Chemicals book'. AgF was purchased from *Aldrich*. Compounds **2a** phenylglyoxylic acid was purchased from Adamas Company, **2b** from TCI, and **2c-d** from Aldrich.

2. General Procedure for Palladium-Catalyzed Cascade Cyclization

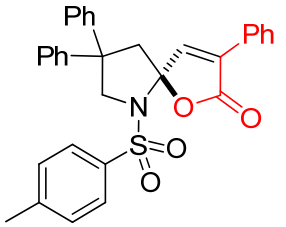
2.1. Optimization of reaction conditions:

To a dried glass tube, $Pd(OAc)_2$ (1.1 mg, 0.005 mmol), oxidant (0.2 mmol), alkene **1a** (0.1 mmol), and phenylglyoxylic acid **2a** (30 mg, 0.2 mmol) were dissolved in dry CH_3CN (0.5 mL). The reaction tube was sealed and stirred for 16 h at 70 ºC. After the reaction completed, EtOAc (4.0 mL) was added to the mixture. Then the solution was filtrated through a short pad of silica gel, and the filtrate was concentrated. The crude product was analyzed by ^{1}H NMR with 2,2,2-trifluoro-*N*,*N*-dimethylacetamide (CF_3-DMA) as an internal standard. The results were summarized in Table 1.

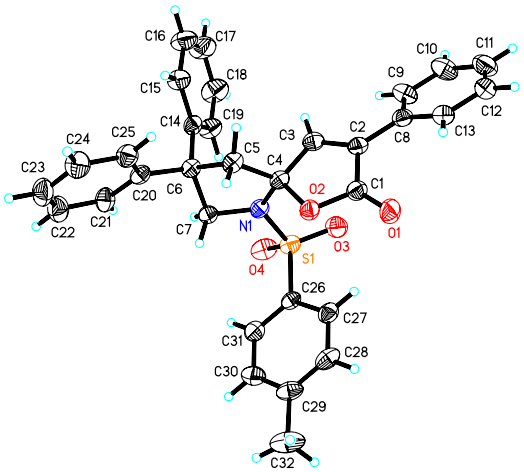
2.2 General Procedure for Palladium-Catalyzed Cascade Cyclization.

To a dried glass tube, $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), BQ (21.6 mg, 0.4 mmol), alkene **1** (0.2 mmol), and glyoxylic acid **2** (0.4 mmol) were dissolved in dry CH_3CN (1.0 mL). The reaction mixture was stirred on a preheated oil-bath at 70 ºC for 16 h, The reaction tube was sealed and stirred for 16 h at 70 ºC. After the reaction completed, EtOAc (8.0 mL) was added to the mixture. Then the solution was filtrated through a short pad of silica gel, and the filtrate was concentrated. The residue was

purified by column chromatography on silica gel with a gradient eluent of petroleum ether and ethyl acetate to afford the products **3**. The results were summarized in Table 2 and 3. The compound 3e was characterized by X-ray (see below).



3e



3 Time Course Experiments

3.2 ^{1}H NMR experiment at 30 ºC

To an oven-dried the NMR tube, the substrate**1a** (15.4 mg, 0.05 mmol), $Pd(OAc)_2$ (0.55 mg, 0.0025 mmol) and BQ (5.4 mg, 0.05 mmol), PhCOCOOH (15 mg, 0.1 mmol) were dissolved in CD_3CN (0.5 mL). The reaction was monitored by ^{1}H NMR at 30 ºC. We found that a intermediate **4a** was initial formed, and gradually converted to final *spiro* product **3a** (see Figure S1).

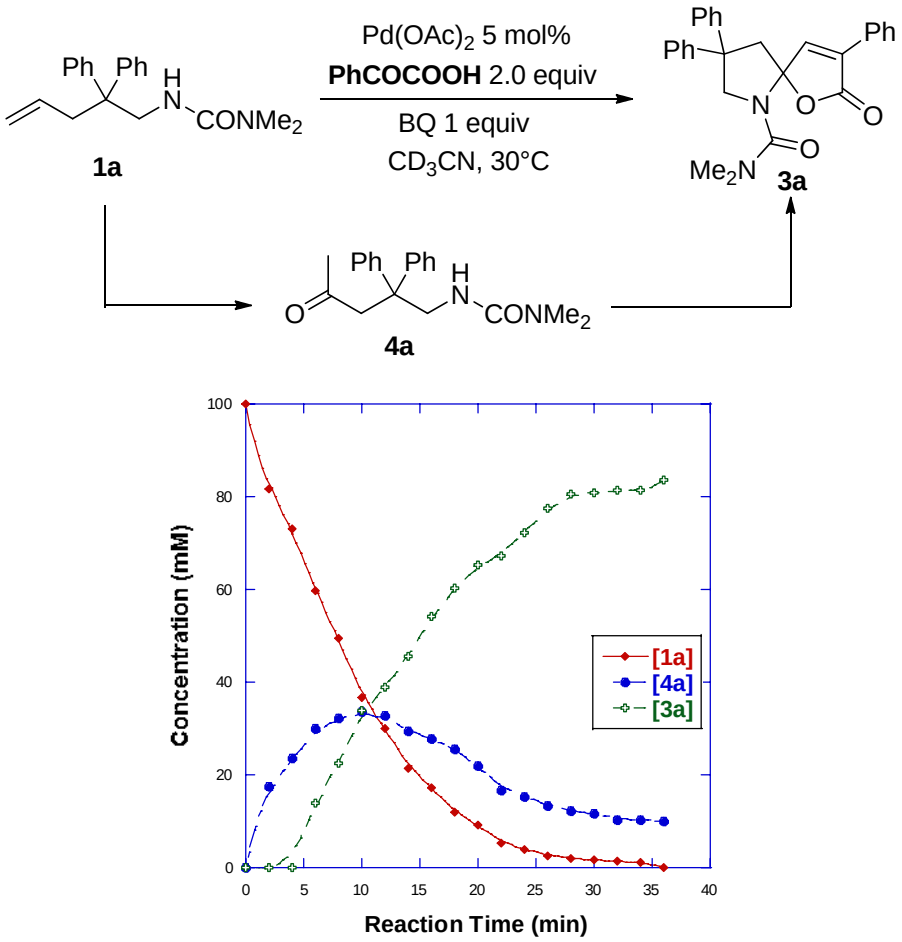
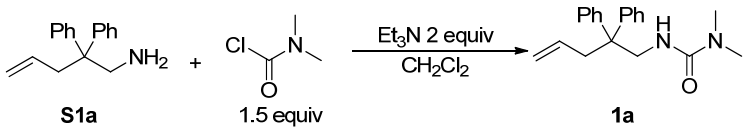


Figure S1. Time Course of Reaction of **1a**.

4 Procedure of Substrate Synthesis

4.1 Typical procedures for the synthesis of substrate 1a:

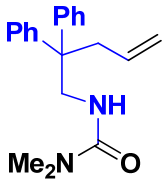


Substrate S**1a** was synthesized according to the previous literature.[S1] In a three-necked 100 ml flask, substrate S**1a** (4.8 g, 20 mmol) and Et_3N (4.1 ml, 36 mmol, 1.8 equiv) were dissolved in dry CH_2Cl_2 (50 mL). A solution of *N*,*N*-dimethylcarbamic chloride (2.8 ml, 36 mmol, 1.8 equiv) in CH_2Cl_2 (10 mL) was added dropwisely at 0 ^oC. The mixture was allowed to warm to room temperature and stirred over night. After reaction completed, water (20 mL) was added. The mixtrue

(S1) C. F. Bender, R. A. Widenhoefer, *J. Am. Chem. Soc.* 2005, **127**, 1070.

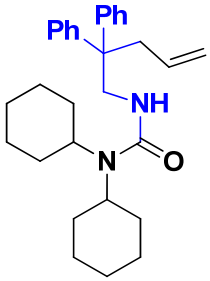
was extracted with CH_2Cl_2. The organic layer was combined and dried over $MgSO_4$. After removal of the solvent, the residue was purified by column chromatography on silical gel (PE/EA = 3:1) to afford the desired product **1a** (5.54g, 90%).

Substrates **1b-1p** were synthesized with the same produce with **1a**[S2], Substrate **1e**[S3], **1g**[S4], **1j**[S2], **1l**[S2], **1m**[S2] were known products.



1a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.29 (m, 4H), 7.24-7.19 (m, 6H), 5.52-5.42 (m, 1H), 4.99-4.93 (m, 2H), 3.92 (s, 3H), 2.86 (d, *J* = 7.6 Hz, 2H), 2.70 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.07, 145.53, 133.88, 128.13, 128.03, 126.33， 118.31, 50.43, 47.47, 41.96, 35.84. HRMS: m/z (ESI) calculated $[M+H]^+$: 309.1967, measured: 309.1956.



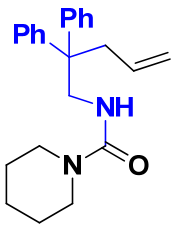
1b

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.26 (m, 4H), 7.20-7.17 (m, 6H), 5.48-5.41 (m, 1H), 4.96-4.92 (m, 2H), 3.96 (d, *J* = 5.2 Hz, 2H), 3.79 (t, *J* = 10.4 Hz, 1H), 3.25 (t, *J* = 12.0 Hz, 2H), 2.85 (d, *J* = 7.2 Hz, 2H), 1.75-1.47 (m, 10H), 1.38-1.30 (m, 4H), 1.22-1.12 (m, 4H), 0.95-0.86 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.38, 145.84, 133.87, 128.24, 128.08, 126.31， 118.40, 54.56, 50.17, 47.58, 42.26, 31.54, 26.31, 25.29. HRMS: m/z (ESI) calculated $[M+H]^+$: 445.3219, measured: 445.3209.

(S2) T. Wu, J. Cheng, P. Chen, G. Liu, *Chem.comm*. 2013, **49**, 8707.

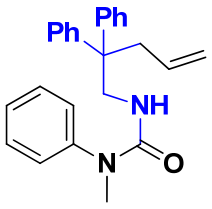
(S3) T. Wu, G. Yin, G. Liu, *J. Am. Chem. Soc*. 2009, **131**, 16354.

(S4) X. Han, R. A. Widenhoefer, *Angew. Chem. Int. Ed*. 2006, **45**, 1747.



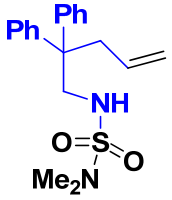
1c

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.29 (m, 4H), 7.24-7.19 (m, 6H), 5.52-5.42 (m, 1H), 4.99-4.93 (m, 2H), 3.98-3.97 (m, 1H), 3.93 (d, *J* = 5.2 Hz, 2H), 3.11 (t, *J* = 5.6 Hz, 4H), 2.87 (d, *J* = 7.2 Hz, 2H), 1.56-1.49 (m, 2H), 1.45-1.39 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.41, 145.63, 133.97, 128.16, 128.09, 126.36, 118.31, 50.54, 47.43, 44.88, 42.12, 25.47, 24.38. HRMS: m/z (ESI) calculated $[M+H]^+$: 349.2280, measured: 439.2270.



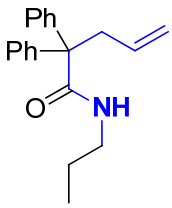
1d

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.09 (m, 9H), 7.01-6.93 (m, 6H), 5.45-5.35 (m, 1H), 4.93-4.87 (m, 2H), 3.94 (t, *J* = 5.6 Hz, 1H), 3.86 (d, *J* = 5.2 Hz, 2H), 3.18 (s, 3H), 2.72 (d, *J* = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.68, 145.02, 142.56, 133.54, 129.54, 127.79, 127.51, 126.83, 126.79, 125.88, 118.09, 49.97, 47.14, 41.72, 36.59. HRMS: m/z (ESI) calculated $[M+H]^+$: 371.2123, measured: 371.2110.



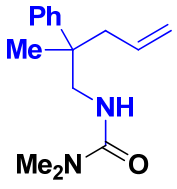
1f

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.21 (m, 6H), 7.17-7.14 (m, 4H), 5.39-5.31 (m, 1H), 5.09-4.99 (m, 2H), 3.68-3.64 (m, 3H), 2.94 (d, *J* = 6.8 Hz, 2H), 2.67 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.65, 133.35, 128.39, 127.76, 126.73, 118.88, 49.59, 49.41, 41.23, 37.83. HRMS: m/z (ESI) calculated $[M+H]^+$: 345.1637, measured: 345.1619.



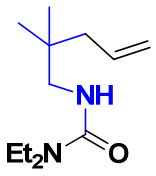
1h

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.22 (m, 10H), 5.81-5.71 (m, 1H), 5.54 (br, 1H), 5.00-4.91 (m, 2H), 3.23-3.16 (m, 4H), 1.41 (m, *J* = 7.2 Hz, 2H), 0.77 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.86, 143.06, 135.33, 128.89, 128.14, 126.82, 117.53, 60.46, 43.29, 41.43, 22.47, 11.15. HRMS: m/z (ESI) calculated $[M+H]^+$: 294.1858, measured: 294.1858.



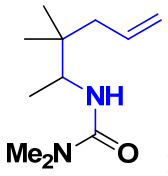
1i

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.31 (m, 4H), 7.23-7.18 (m, 1H), 5.61-5.51 (m, 1H), 5.03-4.93 (m, 2H), 4.18 (t, br, *J* = 4.8 Hz, 1H), 3.55-3.49 (ddd, *J* = 13.2 Hz, 6.8 Hz, 2.0 Hz, 1H), 3.43-3.38 (ddd, *J* = 13.2 Hz, 4.8 Hz, 2.0 Hz, 1H), 2.73 (s, 6H), 2.59-2.54 (m, 1H), 2.36-2.31 (m, 1H), 1.31 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.03, 144.65, 134.05, 128.08, 126.08, 125.85, 117.20, 50.72, 44.04, 41.73, 35.61, 22.12. HRMS: m/z (ESI) calculated $[M+H]^+$: 247.1810, measured: 247.1811.



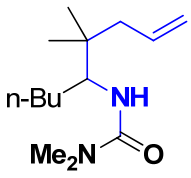
1k

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.92-5.82 (m, 1H), 5.06-5.01 (m, 2H), 4.58 (s, 1H), 3.27 (q, *J* = 7.2 Hz, 4H), 3.09 (d, *J* = 6.0 Hz, 2H), 1.99 (d, *J* = 7.6 Hz, 2H), 1.14 (t, *J* = 7.2 Hz, 6H), 0.89 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.24, 135.29, 116.85, 50.05, 44.69, 41.08, 34.73, 24.87, 13.75. HRMS: m/z (ESI) calculated $[M+H]^+$: 213.1967, measured: 213.1967.



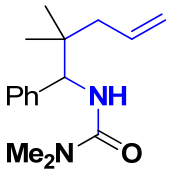
1n

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.94-5.83 (m, 1H), 5.05-5.01 (m, 2H), 4.41 (d, *J* = 9.2 Hz, 1H), 3.84-3.77 (m, 1H), 2.89 (s, 6H), 2.10-2.05 (dd, *J* = 13.6, 7.6 Hz, 1H), 2.01-1.96 (dd, *J* = 13.6, 7.6 Hz, 1H), 1.08 (d, *J* = 6.8 Hz, 3H), 0.88 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.64, 135.18, 116.70, 52.59, 43.32, 36.81, 35.76, 23.61, 23.15, 16.24. HRMS: m/z (ESI) calculated $[M+H]^+$: 199.1810, measured: 199.1807.



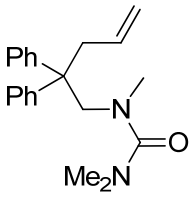
1o

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.95-5.85 (m, 1H), 5.05-5.01 (m, 2H), 4.14 (d, *J* = 10.0 Hz, 1H), 3.67 (dt, *J* = 10.0 Hz, 2.4 Hz, 1H), 2.91 (s, 6H), 2.10-2.04 (dd, *J* = 13.6 Hz, 7.6 Hz, 1H), 2.01-1.96 (dd, *J* = 13.6 Hz, 6.4 Hz, 1H), 1.69-1.62 (m, 1H), 1.39-1.25 (m, 4H), 1.16-1.07 (m, 1H), 0.88 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.43, 135.78, 116.84, 57.62, 43.68, 37.69, 36.15, 30.38, 28.97, 24.35, 23.63, 22.68, 13.95. HRMS: m/z (ESI) calculated $[M+H]^+$: 241.2280, measured: 241.2269.



1p

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.28 (m, 2H), 7.22-7.19 (m, 3H), 5.98-5.89 (m, 1H), 5.11-5.05 (m, 3H), 4.75 (d, *J* = 8.8Hz, 1H), 2.90 (s, 6H), 2.11-2.05 (dd, *J* = 13.6 Hz, 7.6 Hz, 1H), 2.02-1.97 (dd, *J* = 14.0 Hz, 7.2 Hz, 1H), 0.91 (s, 3H), 0.90 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.54, 140.94, 135.36, 128.16, 127.66, 126.69, 117.51, 62.28, 43.81, 37.71, 36.09, 24.44, 24.22. HRMS: m/z (ESI) calculated $[M+H]^+$: 261.1967, measured: 261.1959.

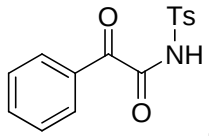


N-Me-**1a**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.14 (m, 10H), 5.61-5.54 (m, 1H), 4.86-4.81 (m, 2H), 4.15 (s, 2H), 2.82 (d, *J* = 6.8Hz, 2H), 2.67 (s, 6H), 2.16 (s, 3 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.52, 146.49, 134.27, 128.09, 127.52, 125.73, 117.59, 57.52, 51.16,

41.57, 41.35, 37.97. HRMS: m/z (ESI) calculated $[M+H]^+$: 323.2123, measured: 323.2121.

4.2 Typical procedures for the synthesis of substrate 2f:

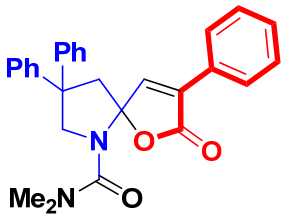


2f

To an oven-dried three-necked flask, **2a** (7.5g, 50 mmol) was dissolved in THF (100 mL). Then Et_3N (10.75 mL, 150 mmol) and TsNCO (11.5 ml, 75 mmol) were sequentially added. The mixture was stirred for 1 h at 60 ºC. After that, the mixture was cooled to room temperature and stirred for an extra 10 h. The solvent was removed under vacuum, and the residue was purified by column chromatography on silica gel (EA/PE= 1:1) to give the product **2f** in 25% yield.

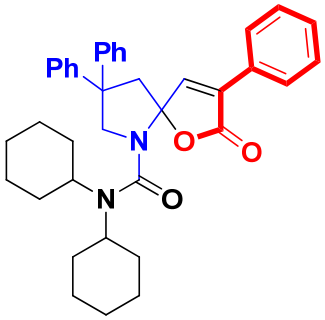
2f: ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.59 (br, 1H), 8.26 (d, J = 7.6 Hz, 2H), 8.03 (d, J = 8.0 Hz, 2H), 7.64 (t, J = 7.2 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 7.36 (d, J = 8.0 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.07, 158.52, 145.46, 135.08, 134.68, 131.56, 131.06, 129.51, 128.48, 128.37, 21.45. HRMS: m/z (ESI) calculated [M+NH4]+: 321.0909, measured: 321.0908

5 New product characterization



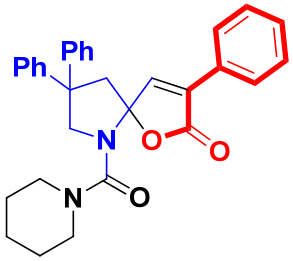
3a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69-7.67 (m, 2H), 7.37-7.22 (m, 13H), 6.83 (s, 1H), 4.42 (dd, J = 10.8, 1.6 Hz, 1H), 4.12 (d, J = 10.8 Hz, 1H), 3.31 (d, J = 13.2 Hz, 1H), 3.01 (dd, J = 13.2, 1.6 Hz, 1H), 2.85 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.71, 160.47, 144.23, 148.49, 143.94, 129.29, 128.97, 128.91, 128.63, 128.40, 127.13, 127.01, 126.88, 126.57, 126.48, 99.07, 59.17, 52.43, 49.06, 38.22. HRMS: m/z (ESI) calculated $[M+H]^+$: 439.2022, measured: 439.2013.



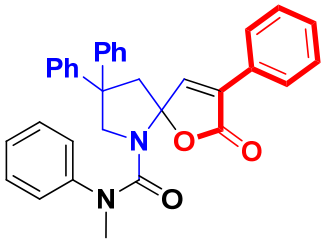
3b

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73-7.70 (m, 2H), 7.36-7.21 (m, 13H), 7.05 (s, 1H), 4.29 (d, *J* = 10.0 Hz, 1H), 3.98 (d, *J* = 10.0 Hz, 1H), 3.30 (m, 2H), 3.25 (d, *J* = 13.6 Hz, 1H), 2.99 (d, *J* = 13.6 Hz, 1H), 1.94-1.49 (m, 14H), 1.27-1.07 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.77, 158.90, 144.80, 148.68, 144.46, 129.39, 128.91, 128.69, 128.50, 128.36, 128.30, 127.22, 126.83, 126.75, 126.64, 126.56, 99.99, 59.73, 57.25, 52.35, 49.39, 31.54, 31.29, 26.29, 26.07, 25.23. HRMS: m/z (ESI) calculated $[M+H]^+$: 575.3274, measured: 575.3273.



3c

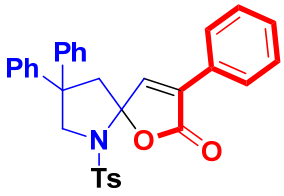
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.68 (m, 2H), 7.76-7.22 (m, 13H), 6.83 (s, 1H), 4.40 (d, *J* = 10.4 Hz, 1H), 4.10 (d, *J* = 10.8 Hz, 1H), 3.28 (d, *J* = 13.2 Hz, 1H), 3.24 (m, 4H), 3.01 (d, *J* = 12.8 Hz, 1H), 1.56 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.69, 160.08, 144.30, 148.45, 143.95, 129.31, 128.93, 128.86, 128.59, 128.38, 127.11, 126.96, 126.83, 126.57, 126.48, 98.98, 59.27, 52.41, 49.24, 47.27, 25.64, 24.30. HRMS: m/z (ESI) calculated $[M+H]^+$: 479.2335, measured: 479.2336.



3d

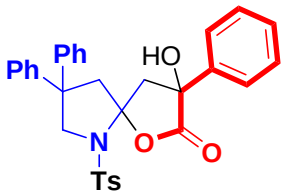
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72-7.70 (m, 2H), 7.39-7.13 (m, 16H), 6.96 (d, *J* = 6.8 Hz, 2H), 6.70 (s, 1H), 4.08 (dd, *J* = 11.2, 1.6 Hz, 1H), 3.24 (s, 3H), 3.16 (t, *J* = 10.8 Hz, 2H), 2.91 (dd, *J* = 13.2, 1.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.91,

157.82, 144.95, 143.96, 148.96, 143.77, 129.87, 129.44, 128.98, 128.95, 128.73, 128.61, 128.39, 127.23, 127.06, 126.81, 126.39, 126.25, 125.99, 125.38, 98.54, 58.38, 52.13, 48.68, 40.03. **HRMS**: m/z (ESI) calculated $[M+H]^+$: 501.2178, measured: 501.2175.

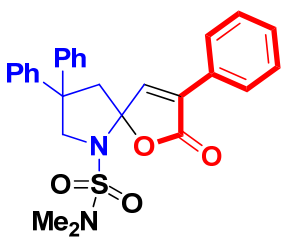


3e

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76-7.70 (m, 4H), 7.41-7.09 (m, 13H), 7.11-7.09 (m, 2H), 6.68 (s, 1H), 4.69 (dd, *J* = 10.0, 1.6 Hz, 1H), 3.73 (d, *J* = 9.6 Hz, 1H), 3.27 (d, *J* = 13.2 Hz, 1H), 3.04 (d, *J* = 13.2, 1.2 Hz, 1H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.11, 146.11, 144.15, 143.93, 143.40, 135.46, 131.04, 129.69, 129.57, 128.92, 128.69, 128.67, 128.55, 127.81, 127.38, 127.11, 127.02, 126.69, 126.35, 98.26, 57.46, 51.28, 50.02, 21.53. HRMS: m/z (ESI) calculated $[M+H]^+$: 522.1739, measured: 522.1736.

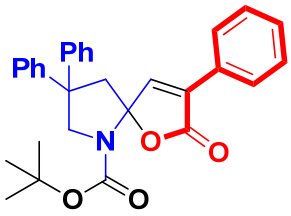


^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 7.35-7.14 (m, 13H), 7.08 (d, *J* = 7.6 Hz, 2H), 4.61 (d, *J* = 9.6 Hz, 1H), 3.33-3.24 (m, 3H), 3.18 (d, *J* = 14.8 Hz, 1H), 3.07 (d, *J* = 12.8 Hz, 1H), 2.41 (s, 3H), 1.63 (d, *J* = 14.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ174.92, 144.21, 143.90, 143.15, 138.83, 135.30, 129.71, 128.81, 128.76, 128.60, 128.43, 128.35, 127.07, 126.88, 126.34, 125.44 100.92, 79.46, 56.51, 52.52, 51.25, 50.91, 21.63. HRMS: m/z (ESI) calculated $[M+H]^+$: 540.1845, measured: 540.1846.



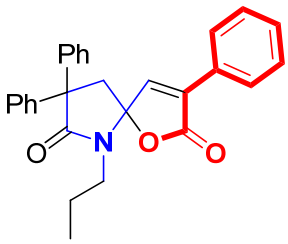
3f

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.69 (m, 2H), 7.34-7.23 (m, 13H), 6.84 (s, 1H), 4.53 (d, *J* = 9.6 Hz, 1H), 4.21 (d, *J* = 10.0 Hz, 1H), 3.35 (d, *J* = 13.6 Hz, 1H), 3.11 (d, *J* = 13.6 Hz, 1H), 2.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.09, 145.70, 144.06, 143.48, 131.37, 129.54, 128.90, 128.72, 128.68, 128.56, 127.33, 127.06, 127.01, 126.48, 126.47, 97.83, 58.63, 51.52, 49.78, 37.62. HRMS: m/z (ESI) calculated $[M+H]^+$: 475.1692, measured: 475.1675.



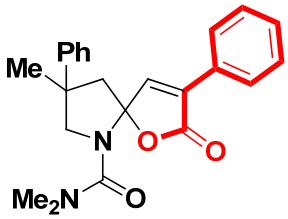
3g

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (m, 2H), 7.38-7.20 (m, 13H), 6.46 (s, 1H), 4.64 (d, *J* = 11.6 Hz, 1H), 4.13 (d, *J* = 10.8 Hz, 1H), 3.34 (d, *J* = 13.2 Hz, 1H)，3.07 (d, *J* = 13.2 Hz, 1H), 1.29 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.76, 152.70, 145.76, 144.39, 143.95, 131.59, 129.49, 128.96, 128.74, 127.14, 127.07, 126.96, 126.59, 126.46, 96.34, 81.58, 55.96, 50.57, 49.69, 28.11. HRMS: m/z (ESI) calculated $[M+H]^+$: 468.2175, measured: 468.2176.



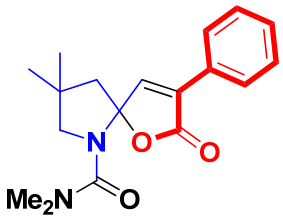
3h

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.83 (m, 2H), 7.46-7.24 (m, 13H), 7.11 (s, 1H), 3.32 (d, *J* = 14.4 Hz, 1H), 3.24-3.09 (m, 2H)，3.11 (d, *J* = 14.8 Hz, 1H), 1.66-1.50 (m, 2H), 0.87 (t, *J* = 7.6Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.33, 168.69, 144.14, 144.03, 141.68, 130.37, 134.56, 128.93, 128.73, 128.47, 128.23, 127.94, 127.59, 127.50, 127.30, 127.22, 96.49, 56.54, 45.20, 41.98, 22.62, 11.45. HRMS: m/z (ESI) calculated $[M+H]^+$: 424.1913, measured: 424.1892.



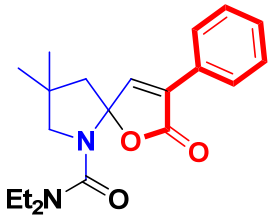
3i

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.83 (m, 2.74H), 7.67 (m, 0.37H), 7.39-7.19 (m, 11.96H), 4.05 (d, *J* = 9.6 Hz, 1H), 3.96 (d, *J* = 10.0Hz, 0.37H), 3.74 (d, *J* = 10.0 Hz, 1.37H), 2.87-2.78 (m, 8.22H), 2.68 (d, *J* = 13.6 Hz, 1.00H)，2.55 (d, *J* = 13.6 Hz, 0.37H), 2.43 (d, *J* = 13.6 Hz, 1.0H), 1.69 (s, 3H), 1.53 (s, 1.11H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.72, 169.66, 160.67, 160.49, 148.02, 146.87, 145.79, 145.24, 129.32, 129.19, 131.56, 129.00, 128.91, 128.66, 128.56, 128.51, 128.41, 128.33, 127.14, 127.12, 126.58, 126.46, 125.49, 125.09, 99.72, 99.71, 61.66, 61.33, 49.75, 49.64, 44.86, 43.68, 38.11, 37.90, 28.73, 28.70. HRMS: m/z (ESI) calculated $[M+H]^+$: 377.1865, measured: 377.1853.



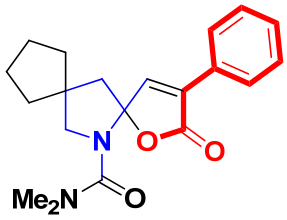
3j

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.84 (d, *J* = 7.2 Hz, 2H), 7.39-7.29 (m, 4H), 3.47 (d, *J* = 9.6 Hz, 1H), 3.36 (d, *J* = 10.4 Hz, 1H), 2.78 (s, 6H), 2.18 (d, *J* = 14.0, 1H), 2.04 (d, *J* = 13.2 Hz, 1H), 1.35 (s, 3H), 1.20 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.82, 160.75, 146.43, 130.84, 129.26, 129.04, 128.38, 127.17, 100.33, 62.93, 51.13, 38.03, 36.69, 27.49, 26.55. HRMS: m/z (ESI) calculated $[M+H]^+$: 315.1709, measured: 315.1698.



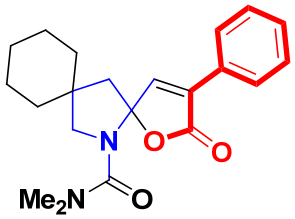
3k

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.85 (m, 2H), 7.40-7.34 (m, 4H), 3.42 (d, *J* = 9.6, 1H), 3.35-3.27 (m, 3H), 3.15-3.06 (m, *J* = 7.2 Hz, 2H), 2.18 (d, *J* = 13.6 Hz, 1H), 2.06 (d, *J* = 13.6, 1H), 1.35 (s, 3H), 1.20 (s, 3H), 1.06 (t, *J* = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.89, 160.27, 146.89, 130.34, 129.31, 129.02, 128.39, 127.17, 100.62, 63.17, 51.10, 41.54, 36.86, 27.50, 26.61, 13.15. HRMS: m/z (ESI) calculated $[M+H]^+$: 343.2022, measured: 343.2021.



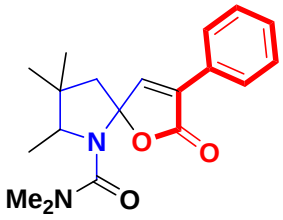
3l

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 6.8 Hz, 2H), 7.38-7.28 (m, 4H), 3.54 (d, *J* = 9.6 Hz, 1H), 3.41 (d, *J* = 9.6 Hz, 1H), 2.78 (s, 6H), 2.30 (d, *J* = 13.2Hz, 1H), 2.08 (d, *J* = 13.2 Hz, 1H), 1.90-1.88 (m, 2H)，1.75-1.66 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.89, 160.82, 146.06, 131.42, 129.28, 129.08, 128.39, 127.18, 100.11, 61.70, 49.31, 47.47, 38.06, 37.29, 37.23, 24.56, 24.16. HRMS: m/z (ESI) calculated $[M+H]^+$: 341.1865, measured: 341.1848.



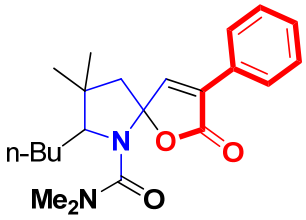
3m

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.82 (m, 2H), 7.40-7.33 (m, 3H), 7.32 (s, 1H), 3.46 (dd, *J* = 14.4, 9.6 Hz, 2H), 2.80 (s, 6H), 2.12 (dd, *J* = 15.6, 14 Hz, 2H), 2.16-2.09 (m, 2H), 1.57-1.46 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.00, 161.06, 146.31, 131.29, 129.36, 129.19, 128.52, 127.30, 100.13, 40.61, 38.19, 37.45, 35.15, 25.71, 23.71, 23.07. HRMS: m/z (ESI) calculated $[M+H]^+$: 355.2022, measured: 355.2011.



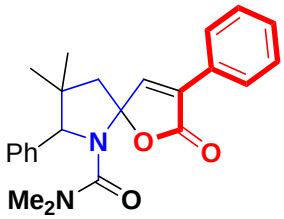
3n

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.77 (m, 2H), 7.43-7.38 (m, 3H), 7.16 (s, 1H), 3.84 (q, *J* = 6.4 Hz, 1H), 2.79 (s, 6H), 2.17 (d, *J* = 13.2 Hz, 1H), 1.98 (d, *J* = 13.2 Hz, 1H), 1.23 (s, 3H), 1.11 (s, 3H), 1.08 (d, *J* = 6.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.89, 161.61, 145.34, 132.31, 129.63, 128.85, 128.74, 127.20, 100.71, 65.17, 52.60, 38.36, 37.82, 26.39, 21.62, 13.79. HRMS: m/z (ESI) calculated $[M+H]^+$: 329.1865, measured: 329.1857.



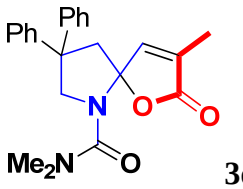
3o

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.77 (m, 2H), 7.43-7.39 (m, 3H), 7.15 (s, 1H), 3.86 (t, *J* = 6.8 Hz, 1H), 2.79 (s, 6H), 2.19 (d, *J* = 13.2 Hz, 1H), 1.98 (d, *J* = 13.2 Hz, 1H), 1.55-1.49 (m, 2H), 1.33-1.30 (m, 4H), 1.28 (s, 3H), 1.19 (s, 3H), 0.90 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.64, 161.79, 145.17, 132.04, 129.54, 128.83, 128.68, 127.13, 100.85, 68.81, 53.26, 38.81, 37.60, 30.80, 28.88, 28.06, 23.02, 22.07, 13.92. HRMS: m/z (ESI) calculated $[M+H]^+$: 371.2335, measured: 371.2322.



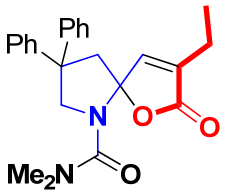
3p

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.82 (m, 2H), 7.44-7.25 (m, 9H), 4.96 (s, 1H), 2.79 (s, 6H), 2.38 (d, *J* = 17.2 Hz, 1H), 2.13 (d, *J* = 18.0 Hz, 1H), 1.19 (s, 3H), 0.99 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.58, 160.84, 145.06, 137.99, 132.61, 129.71, 128.75, 128.06, 127.39, 127.20, 126.82, 100.57, 74.04, 52.62, 39.66, 37.55, 26.90, 23.01. HRMS: m/z (ESI) calculated $[M+H]^+$: 391.2022, measured: 391.2000.



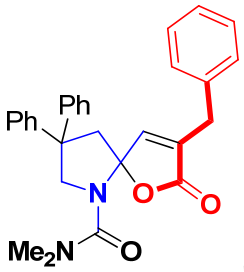
3q

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.18 (m, 10H), 6.36 (s, 1H), 4.34 (d, *J* = 10.4 Hz, 1H), 4.09 (d, *J* = 10.4 Hz, 1H), 3.17 (d, *J* = 13.2 Hz, 1H), 2.90 (d, *J* = 13.2 Hz, 1H), 2.81 (s, 6H), 1.79 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.19, 160.42, 149.11, 144.46, 144.17, 128.85, 128.61, 127.56, 126.92, 126.83, 126.62, 126.52, 99.82, 59.17, 52.19, 48.86, 38.18, 10.25. HRMS: m/z (ESI) calculated $[M+H]^+$: 377.1865, measured: 377.1851.



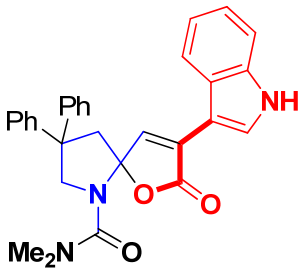
3r

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.19 (m, 10H), 6.37 (s, 1H), 4.32 (d, *J* = 10.4 Hz, 1H), 4.12 (d, *J* = 10.4 Hz, 1H), 3.14 (d, *J* = 13.2 Hz, 1H), 2.92 (d, *J* = 13.2 Hz, 1H), 2.78 (s, 6H), 2.24-2.17 (m, 2H), 1.04 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.45, 160.42, 147.09, 144.50, 143.99, 133.88, 128.60, 128.36, 126.67, 126.57, 126.49, 126.30, 99.88, 58.96, 51.81, 48.91, 37.99, 18.06, 11.45. HRMS: m/z (ESI) calculated $[M+H]^+$: 391.2022, measured: 391.2007.



3s

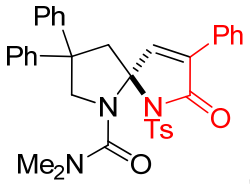
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.13 (m, 15H), 6.32 (t, *J* = 1.6 Hz, 1H), 4.26 (d, *J* = 10.4Hz, 1H), 4.12 (d, *J* = 10.4 Hz, 1H), 3.51 (s, 2H), 3.07 (d, *J* = 13.2 Hz, 1H), 2.87 (d, *J* = 13.2 Hz, 1H), 2.71 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.11, 160.62, 148.88, 144.71, 143.99, 137.25, 132.56, 128.67, 128.66, 128.54, 128.44, 126.76, 126.67, 126.56, 126.32, 100.19, 59.06, 51.72, 49.23, 38.09, 31.18. HRMS: m/z (ESI) calculated $[M+H]^+$: 453.2178, measured: 453.2177.



3t

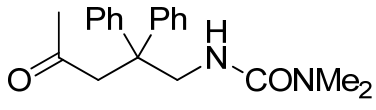
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.39 (s, 1H), 7.88 (d, *J* = 1.6 Hz, 1H), 7.37-7.16 (m, 12H), 7.07-6.99 (m, 2H), 6.78 (s, 1H), 4.38 (d, *J* = 10.4 Hz, 1H), 4.22 (d, *J* = 10.4 Hz, 1H), 3.14 (d, *J* = 13.2 Hz, 1H), 3.04 (d, *J* = 12.8 Hz, 1H), 2.77 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.19, 160.84, 145.06, 144.22, 139.25, 136.29, 128.91, 128.57, 127.00, 126.92, 126.82, 126.76, 126.67, 125.69, 125.40, 122.25, 120.40, 119.17,

111.95, 104.93, 100.71, 59.13, 51.86, 49.73, 38.27. HRMS: m/z (ESI) calculated $[M+H]^+$: 478.2131, measured: 478.2112.



3u

3u: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.27 (m, 4H), 7.23-7.17 (m, 6H), 4.79 (t, *J* = 4.8 Hz, 1H), 4.04 (d, *J* = 5.6 Hz, 2H), 3.37 (s, 2H), 2.73 (s, 6H), 1.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.13, 159.79, 149.63, 144.71, 144.00, 143.66, 136.55, 129.79, 129.34, 129.16, 128.74, 128.44, 128.13, 128.09, 127.81, 127.24, 127.03, 126.98, 126.78, 126.58, 85.71, 58.96, 53.15, 48.15, 38.21, 21.56. HRMS: m/z (ESI) calculated $[M+H]^+$: 592.2270, measured: 592.2250.

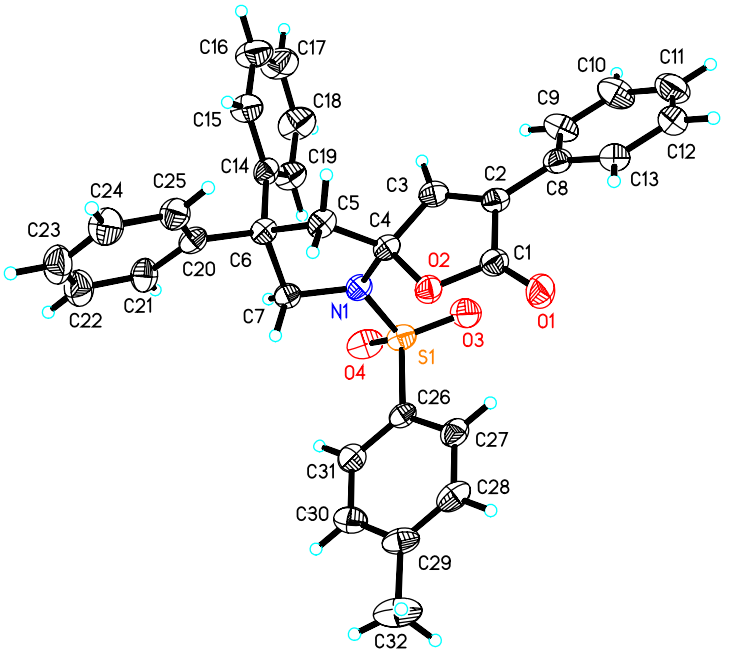


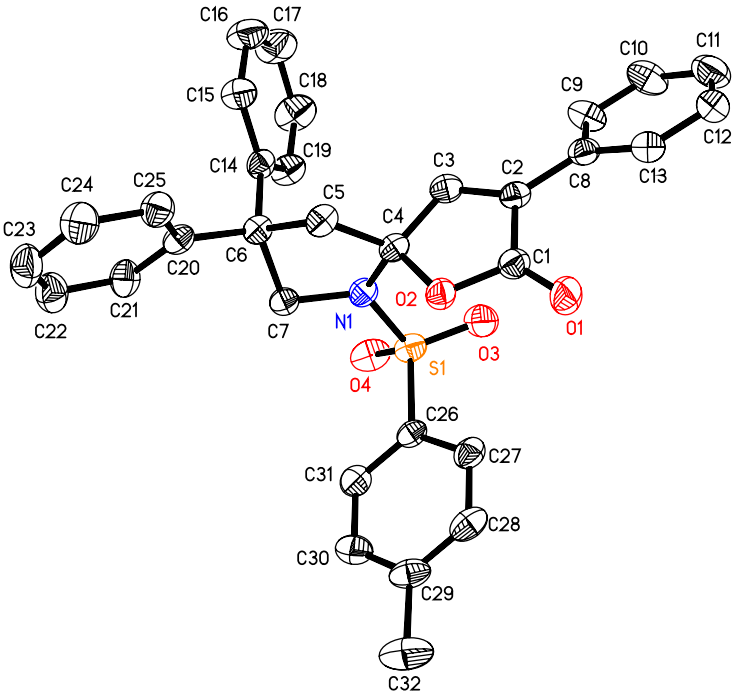
4a

4a: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.27 (m, 4H), 7.23-7.17 (m, 6H), 4.79 (t, *J* = 4.8 Hz, 1H), 4.04 (d, *J* = 5.6 Hz, 2H), 3.37 (s, 2H), 2.73 (s, 6H), 1.87 (s, 3H). ^{1}H NMR (400 MHz, CD_3CN) δ 7.30-7.27 (m, 4H), 7.22-7.17 (m, 6H), 4.69 (br, 1H), 4.01 (d, *J* = 5.2 Hz, 2H), 3.42 (s, 2H), 2.66 (s, 6H), 1.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.70, 158.16, 144.99, 128.13, 127.61, 126.48, 51.13, 49.75, 48.94, 35.82, 31.89. HRMS: m/z (ESI) calculated $[M+H]^+$: 325.1916, measured: 325.1911.

6 X-ray data

6.1 The X-ray of compound 3e


C16
C17
C18
C15
C19
C14
C24
C25
C23
C20
C6
C22
C21
C7
C5
C4
C3
C2
C8
C9
C10
C11
C12
C13
O2
C1
N1
O1
O3
S1
O4
C26
C27
C31
C28
C30
C29
C32


C16
C17
C18
C15
C19
C14
C24
C25
C23
C20
C6
C22
C21
C7
C5
C4
C3
C2
C8
C9
C10
C11
C12
C13
O2
C1
N1
O1
O3
S1
O4
C26
C27
C31
C28
C30
C29
C32

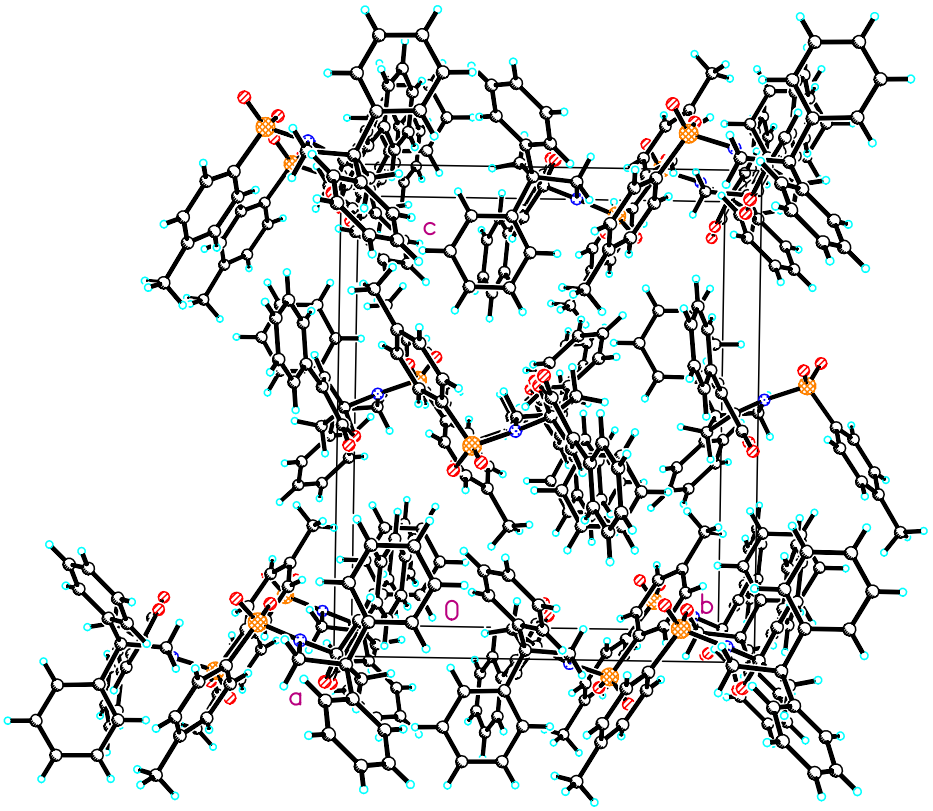


Table 1. Crystal data and structure refinement for **3e**.

Identification code	cd213171
Empirical formula	C32 H27 N O4 S
Formula weight	521.61
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 12.2848(7) Å alpha = 90°. b = 13.6708(8) Å beta = 90°. c = 15.9987(9) Å gamma = 90°.
Volume	2686.9(3) Å^3

Z, Calculated density	4, 1.289 Mg/m^3
Absorption coefficient	0.159 mm^{-1}
F(000)	1096
Crystal size	0.212 x 0.187 x 0.112 mm^3
Theta range for data collection	1.96 to 26.00°.
Limiting indices	-14<=h<=15, -16<=k<=15, -19<=l<=19
Reflections collected / unique	16356 / 5264 [R(int) = 0.0442]
Completeness to theta = 26.00	100.0 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.70277
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5264 / 0 / 344
Goodness-of-fit on F^2	1.071
Final R indices [I>2sigma(I)]	R1 = 0.0531, wR2 = 0.1158
R indices (all data)	R1 = 0.0694, wR2 = 0.1233
Absolute structure parameter	0.41(9)
Largest diff. peak and hole	0.239 and -0.187 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd213171.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S(1)	5440(1)	6790(1)	9310(1)	52(1)
N(1)	5969(2)	5749(2)	9626(1)	47(1)
O(1)	2754(2)	5162(2)	10734(1)	76(1)
O(2)	4548(2)	5228(1)	10560(1)	51(1)
O(3)	4386(2)	6573(2)	8990(1)	70(1)
O(4)	6252(2)	7234(2)	8793(1)	69(1)
C(1)	3527(3)	4956(2)	10313(2)	51(1)
C(2)	3607(2)	4435(2)	9504(2)	47(1)
C(3)	4653(2)	4430(2)	9295(2)	51(1)
C(4)	5332(2)	4921(2)	9938(2)	46(1)
C(5)	6201(2)	4294(2)	10361(2)	49(1)
C(6)	7306(2)	4605(2)	9993(2)	44(1)
C(7)	7092(2)	5707(2)	9911(2)	45(1)
C(8)	2655(3)	4077(2)	9036(2)	50(1)
C(9)	2717(3)	3988(2)	8168(2)	67(1)
C(10)	1846(4)	3673(3)	7719(2)	80(1)
C(11)	880(4)	3434(3)	8124(3)	84(1)
C(12)	809(3)	3524(2)	8978(3)	71(1)
C(13)	1692(3)	3843(2)	9433(2)	60(1)
C(14)	7456(2)	4101(2)	9141(2)	47(1)
C(15)	7645(3)	3101(2)	9128(2)	63(1)
C(16)	7684(4)	2592(3)	8377(2)	83(1)
C(17)	7547(4)	3076(3)	7641(2)	93(1)
C(18)	7377(4)	4073(3)	7638(2)	86(1)
C(19)	7338(3)	4568(2)	8386(2)	62(1)
C(20)	8251(2)	4407(2)	10585(2)	47(1)
C(21)	9179(2)	4977(3)	10548(2)	58(1)
C(22)	10026(3)	4850(3)	11102(2)	73(1)
C(23)	9970(3)	4128(3)	11700(2)	76(1)
C(24)	9063(3)	3542(3)	11736(2)	74(1)
C(25)	8203(3)	3670(2)	11184(2)	59(1)
C(26)	5291(2)	7531(2)	10208(2)	46(1)
C(27)	4330(3)	7509(2)	10652(2)	55(1)

C(28)	4255(3)	8064(2)	11370(2)	60(1)
C(29)	5104(3)	8618(2)	11656(2)	62(1)
C(30)	6054(3)	8637(2)	11190(2)	65(1)
C(31)	6154(3)	8097(2)	10468(2)	56(1)
C(32)	5026(5)	9179(3)	12471(2)	98(1)

Table 3. Bond lengths [A] and angles [deg] for cd213171.

S(1)-O(3)	1.423(2)
S(1)-O(4)	1.431(2)
S(1)-N(1)	1.644(2)
S(1)-C(26)	1.767(3)
N(1)-C(7)	1.455(4)
N(1)-C(4)	1.464(3)
O(1)-C(1)	1.198(3)
O(2)-C(1)	1.366(3)
O(2)-C(4)	1.448(3)
C(1)-C(2)	1.481(4)
C(2)-C(3)	1.327(4)
C(2)-C(8)	1.472(4)
C(3)-C(4)	1.484(4)
C(3)-H(3)	0.9300
C(4)-C(5)	1.527(4)
C(5)-C(6)	1.540(4)
C(5)-H(5A)	0.9700
C(5)-H(5B)	0.9700
C(6)-C(20)	1.522(4)
C(6)-C(7)	1.535(4)
C(6)-C(14)	1.539(4)
C(7)-H(7A)	0.9700
C(7)-H(7B)	0.9700
C(8)-C(13)	1.380(4)
C(8)-C(9)	1.396(4)
C(9)-C(10)	1.359(5)
C(9)-H(9)	0.9300
C(10)-C(11)	1.391(6)
C(10)-H(10)	0.9300
C(11)-C(12)	1.375(5)
C(11)-H(11)	0.9300
C(12)-C(13)	1.377(5)
C(12)-H(12)	0.9300
C(13)-H(13)	0.9300
C(14)-C(19)	1.374(4)
C(14)-C(15)	1.387(4)
C(15)-C(16)	1.389(5)
C(15)-H(15)	0.9300
C(16)-C(17)	1.360(5)
C(16)-H(16)	0.9300
C(17)-C(18)	1.378(6)

C(17)-H(17)	0.9300
C(18)-C(19)	1.376(4)
C(18)-H(18)	0.9300
C(19)-H(19)	0.9300
C(20)-C(21)	1.383(4)
C(20)-C(25)	1.392(4)
C(21)-C(22)	1.378(4)
C(21)-H(21)	0.9300
C(22)-C(23)	1.376(5)
C(22)-H(22)	0.9300
C(23)-C(24)	1.373(5)
C(23)-H(23)	0.9300
C(24)-C(25)	1.387(5)
C(24)-H(24)	0.9300
C(25)-H(25)	0.9300
C(26)-C(31)	1.377(4)
C(26)-C(27)	1.378(4)
C(27)-C(28)	1.380(4)
C(27)-H(27)	0.9300
C(28)-C(29)	1.368(5)
C(28)-H(28)	0.9300
C(29)-C(30)	1.385(5)
C(29)-C(32)	1.515(4)
C(30)-C(31)	1.377(4)
C(30)-H(30)	0.9300
C(31)-H(31)	0.9300
C(32)-H(32A)	0.9600
C(32)-H(32B)	0.9600
C(32)-H(32C)	0.9600

O(3)-S(1)-O(4)	120.96(14)
O(3)-S(1)-N(1)	106.84(13)
O(4)-S(1)-N(1)	105.63(13)
O(3)-S(1)-C(26)	108.53(14)
O(4)-S(1)-C(26)	107.34(14)
N(1)-S(1)-C(26)	106.73(12)
C(7)-N(1)-C(4)	111.7(2)
C(7)-N(1)-S(1)	120.34(19)
C(4)-N(1)-S(1)	124.23(19)
C(1)-O(2)-C(4)	109.4(2)
O(1)-C(1)-O(2)	120.1(3)
O(1)-C(1)-C(2)	131.1(3)
O(2)-C(1)-C(2)	108.8(2)
C(3)-C(2)-C(8)	129.7(3)

C(3)-C(2)-C(1)	106.7(3)
C(8)-C(2)-C(1)	123.5(3)
C(2)-C(3)-C(4)	111.5(3)
C(2)-C(3)-H(3)	124.2
C(4)-C(3)-H(3)	124.2
O(2)-C(4)-N(1)	111.5(2)
O(2)-C(4)-C(3)	103.5(2)
N(1)-C(4)-C(3)	114.4(2)
O(2)-C(4)-C(5)	108.9(2)
N(1)-C(4)-C(5)	102.2(2)
C(3)-C(4)-C(5)	116.5(2)
C(4)-C(5)-C(6)	107.0(2)
C(4)-C(5)-H(5A)	110.3
C(6)-C(5)-H(5A)	110.3
C(4)-C(5)-H(5B)	110.3
C(6)-C(5)-H(5B)	110.3
H(5A)-C(5)-H(5B)	108.6
C(20)-C(6)-C(7)	111.0(2)
C(20)-C(6)-C(14)	112.3(2)
C(7)-C(6)-C(14)	112.6(2)
C(20)-C(6)-C(5)	112.7(2)
C(7)-C(6)-C(5)	98.8(2)
C(14)-C(6)-C(5)	108.6(2)
N(1)-C(7)-C(6)	103.2(2)
N(1)-C(7)-H(7A)	111.1
C(6)-C(7)-H(7A)	111.1
N(1)-C(7)-H(7B)	111.1
C(6)-C(7)-H(7B)	111.1
H(7A)-C(7)-H(7B)	109.1
C(13)-C(8)-C(9)	118.9(3)
C(13)-C(8)-C(2)	121.6(3)
C(9)-C(8)-C(2)	119.5(3)
C(10)-C(9)-C(8)	120.8(4)
C(10)-C(9)-H(9)	119.6
C(8)-C(9)-H(9)	119.6
C(9)-C(10)-C(11)	120.0(3)
C(9)-C(10)-H(10)	120.0
C(11)-C(10)-H(10)	120.0
C(12)-C(11)-C(10)	119.7(4)
C(12)-C(11)-H(11)	120.1
C(10)-C(11)-H(11)	120.1
C(11)-C(12)-C(13)	120.2(4)
C(11)-C(12)-H(12)	119.9
C(13)-C(12)-H(12)	119.9

C(12)-C(13)-C(8)	120.4(3)
C(12)-C(13)-H(13)	119.8
C(8)-C(13)-H(13)	119.8
C(19)-C(14)-C(15)	117.5(3)
C(19)-C(14)-C(6)	124.0(3)
C(15)-C(14)-C(6)	118.3(2)
C(14)-C(15)-C(16)	120.8(3)
C(14)-C(15)-H(15)	119.6
C(16)-C(15)-H(15)	119.6
C(17)-C(16)-C(15)	120.0(4)
C(17)-C(16)-H(16)	120.0
C(15)-C(16)-H(16)	120.0
C(16)-C(17)-C(18)	120.2(3)
C(16)-C(17)-H(17)	119.9
C(18)-C(17)-H(17)	119.9
C(19)-C(18)-C(17)	119.2(4)
C(19)-C(18)-H(18)	120.4
C(17)-C(18)-H(18)	120.4
C(14)-C(19)-C(18)	122.2(3)
C(14)-C(19)-H(19)	118.9
C(18)-C(19)-H(19)	118.9
C(21)-C(20)-C(25)	118.2(3)
C(21)-C(20)-C(6)	120.1(2)
C(25)-C(20)-C(6)	121.7(3)
C(22)-C(21)-C(20)	121.5(3)
C(22)-C(21)-H(21)	119.2
C(20)-C(21)-H(21)	119.2
C(23)-C(22)-C(21)	120.0(3)
C(23)-C(22)-H(22)	120.0
C(21)-C(22)-H(22)	120.0
C(24)-C(23)-C(22)	119.2(3)
C(24)-C(23)-H(23)	120.4
C(22)-C(23)-H(23)	120.4
C(23)-C(24)-C(25)	121.1(3)
C(23)-C(24)-H(24)	119.4
C(25)-C(24)-H(24)	119.4
C(24)-C(25)-C(20)	119.9(3)
C(24)-C(25)-H(25)	120.1
C(20)-C(25)-H(25)	120.1
C(31)-C(26)-C(27)	121.1(3)
C(31)-C(26)-S(1)	119.2(2)
C(27)-C(26)-S(1)	119.7(2)
C(26)-C(27)-C(28)	118.3(3)
C(26)-C(27)-H(27)	120.8

C(28)-C(27)-H(27)	120.8
C(29)-C(28)-C(27)	122.2(3)
C(29)-C(28)-H(28)	118.9
C(27)-C(28)-H(28)	118.9
C(28)-C(29)-C(30)	118.2(3)
C(28)-C(29)-C(32)	121.4(4)
C(30)-C(29)-C(32)	120.4(4)
C(31)-C(30)-C(29)	121.2(3)
C(31)-C(30)-H(30)	119.4
C(29)-C(30)-H(30)	119.4
C(30)-C(31)-C(26)	119.1(3)
C(30)-C(31)-H(31)	120.5
C(26)-C(31)-H(31)	120.5
C(29)-C(32)-H(32A)	109.5
C(29)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(29)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd213171. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
S(1)	68(1)	47(1)	41(1)	5(1)	0(1)	10(1)
N(1)	49(1)	43(1)	47(1)	5(1)	0(1)	5(1)
O(1)	47(1)	107(2)	75(1)	-33(2)	14(1)	1(1)
O(2)	46(1)	62(1)	44(1)	-9(1)	2(1)	4(1)
O(3)	82(2)	65(2)	64(1)	-3(1)	-27(1)	12(1)
O(4)	102(2)	60(1)	47(1)	12(1)	17(1)	7(1)
C(1)	52(2)	50(2)	52(2)	-3(1)	4(2)	2(1)
C(2)	53(2)	37(2)	51(2)	-1(1)	3(2)	3(1)
C(3)	59(2)	49(2)	46(1)	-3(1)	5(2)	3(1)
C(4)	48(2)	45(2)	45(1)	-1(1)	6(1)	6(1)
C(5)	51(2)	48(2)	48(2)	8(1)	10(1)	4(1)
C(6)	46(2)	42(2)	44(1)	6(1)	7(1)	1(1)
C(7)	49(2)	45(2)	41(1)	0(1)	4(1)	2(1)
C(8)	56(2)	39(2)	56(2)	-2(1)	-1(2)	4(1)
C(9)	84(3)	57(2)	61(2)	-3(2)	-7(2)	-17(2)
C(10)	99(3)	78(3)	63(2)	-8(2)	-11(2)	-21(2)
C(11)	88(3)	66(3)	97(3)	-13(2)	-31(3)	-10(2)
C(12)	57(2)	59(2)	96(3)	-13(2)	-3(2)	-3(2)
C(13)	60(2)	52(2)	68(2)	-8(2)	-1(2)	0(2)
C(14)	41(2)	51(2)	49(2)	0(1)	10(1)	2(1)
C(15)	74(2)	55(2)	59(2)	-2(2)	1(2)	4(2)
C(16)	104(3)	65(2)	81(2)	-20(2)	4(2)	13(2)
C(17)	117(4)	99(3)	64(2)	-33(2)	-1(2)	23(3)
C(18)	117(4)	95(3)	46(2)	-4(2)	6(2)	21(3)
C(19)	77(2)	61(2)	46(2)	2(1)	7(2)	10(2)
C(20)	51(2)	48(2)	41(1)	0(1)	9(1)	9(1)
C(21)	46(2)	75(2)	55(2)	8(2)	8(2)	2(2)
C(22)	52(2)	92(3)	74(2)	-2(2)	3(2)	0(2)
C(23)	64(2)	98(3)	67(2)	3(2)	-15(2)	14(2)
C(24)	87(3)	72(2)	62(2)	16(2)	-12(2)	7(2)
C(25)	61(2)	57(2)	58(2)	7(2)	-2(2)	4(2)
C(26)	53(2)	38(1)	47(2)	5(1)	3(1)	10(1)
C(27)	55(2)	44(2)	66(2)	6(2)	3(2)	8(1)
C(28)	72(2)	48(2)	61(2)	13(2)	22(2)	14(2)
C(29)	96(3)	40(2)	51(2)	4(1)	6(2)	11(2)

C(30)	82(2)	48(2)	65(2)	−5(2)	−2(2)	−4(2)
C(31)	54(2)	49(2)	64(2)	5(2)	9(2)	−1(1)
C(32)	157(4)	71(3)	67(2)	−14(2)	9(2)	14(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd213171.

	x	y	z	U(eq)
H(3)	4926	4155	8807	61
H(5A)	6068	3606	10252	59
H(5B)	6191	4399	10960	59
H(7A)	7177	6036	10445	54
H(7B)	7580	6003	9507	54
H(9)	3361	4145	7895	81
H(10)	1896	3618	7141	96
H(11)	284	3215	7818	100
H(12)	162	3369	9250	85
H(13)	1639	3901	10011	72
H(15)	7748	2767	9628	75
H(16)	7802	1921	8377	100
H(17)	7569	2734	7139	112
H(18)	7288	4406	7136	103
H(19)	7229	5241	8380	74
H(21)	9234	5458	10139	70
H(22)	10635	5252	11073	87
H(23)	10540	4037	12075	92
H(24)	9025	3050	12137	88
H(25)	7597	3265	11215	70
H(27)	3747	7129	10472	66
H(28)	3606	8062	11669	72
H(30)	6634	9020	11369	78
H(31)	6795	8115	10159	67
H(32A)	4818	8741	12912	148
H(32B)	4492	9687	12418	148
H(32C)	5721	9464	12599	148

Table 6. Torsion angles [deg] for cd213171.

O(3)-S(1)-N(1)-C(7)	173.9(2)
O(4)-S(1)-N(1)-C(7)	43.9(2)
C(26)-S(1)-N(1)-C(7)	-70.2(2)
O(3)-S(1)-N(1)-C(4)	-29.7(2)
O(4)-S(1)-N(1)-C(4)	-159.7(2)
C(26)-S(1)-N(1)-C(4)	86.3(2)
C(4)-O(2)-C(1)-O(1)	-178.3(3)
C(4)-O(2)-C(1)-C(2)	0.8(3)
O(1)-C(1)-C(2)-C(3)	177.9(3)
O(2)-C(1)-C(2)-C(3)	-1.0(3)
O(1)-C(1)-C(2)-C(8)	1.8(5)
O(2)-C(1)-C(2)-C(8)	-177.1(3)
C(8)-C(2)-C(3)-C(4)	176.6(3)
C(1)-C(2)-C(3)-C(4)	0.9(3)
C(1)-O(2)-C(4)-N(1)	123.2(2)
C(1)-O(2)-C(4)-C(3)	-0.2(3)
C(1)-O(2)-C(4)-C(5)	-124.8(3)
C(7)-N(1)-C(4)-O(2)	109.7(2)
S(1)-N(1)-C(4)-O(2)	-48.6(3)
C(7)-N(1)-C(4)-C(3)	-133.3(2)
S(1)-N(1)-C(4)-C(3)	68.5(3)
C(7)-N(1)-C(4)-C(5)	-6.5(3)
S(1)-N(1)-C(4)-C(5)	-164.70(19)
C(2)-C(3)-C(4)-O(2)	-0.4(3)
C(2)-C(3)-C(4)-N(1)	-122.0(3)
C(2)-C(3)-C(4)-C(5)	119.0(3)
O(2)-C(4)-C(5)-C(6)	-138.2(2)
N(1)-C(4)-C(5)-C(6)	-20.2(3)
C(3)-C(4)-C(5)-C(6)	105.3(3)
C(4)-C(5)-C(6)-C(20)	154.5(2)
C(4)-C(5)-C(6)-C(7)	37.2(3)
C(4)-C(5)-C(6)-C(14)	-80.4(3)
C(4)-N(1)-C(7)-C(6)	30.4(3)
S(1)-N(1)-C(7)-C(6)	-170.44(18)
C(20)-C(6)-C(7)-N(1)	-158.2(2)
C(14)-C(6)-C(7)-N(1)	74.8(3)
C(5)-C(6)-C(7)-N(1)	-39.7(3)
C(3)-C(2)-C(8)-C(13)	158.5(3)
C(1)-C(2)-C(8)-C(13)	-26.4(4)
C(3)-C(2)-C(8)-C(9)	-22.7(5)
C(1)-C(2)-C(8)-C(9)	152.5(3)

C(13)-C(8)-C(9)-C(10)	-0.1(5)
C(2)-C(8)-C(9)-C(10)	-179.0(3)
C(8)-C(9)-C(10)-C(11)	-0.2(6)
C(9)-C(10)-C(11)-C(12)	0.5(6)
C(10)-C(11)-C(12)-C(13)	-0.5(6)
C(11)-C(12)-C(13)-C(8)	0.2(5)
C(9)-C(8)-C(13)-C(12)	0.1(5)
C(2)-C(8)-C(13)-C(12)	179.0(3)
C(20)-C(6)-C(14)-C(19)	-130.4(3)
C(7)-C(6)-C(14)-C(19)	-4.2(4)
C(5)-C(6)-C(14)-C(19)	104.2(3)
C(20)-C(6)-C(14)-C(15)	54.3(4)
C(7)-C(6)-C(14)-C(15)	-179.4(3)
C(5)-C(6)-C(14)-C(15)	-71.1(3)
C(19)-C(14)-C(15)-C(16)	-1.7(5)
C(6)-C(14)-C(15)-C(16)	173.9(3)
C(14)-C(15)-C(16)-C(17)	0.8(6)
C(15)-C(16)-C(17)-C(18)	0.4(7)
C(16)-C(17)-C(18)-C(19)	-0.5(7)
C(15)-C(14)-C(19)-C(18)	1.6(5)
C(6)-C(14)-C(19)-C(18)	-173.7(4)
C(17)-C(18)-C(19)-C(14)	-0.5(7)
C(7)-C(6)-C(20)-C(21)	-42.5(4)
C(14)-C(6)-C(20)-C(21)	84.6(3)
C(5)-C(6)-C(20)-C(21)	-152.2(3)
C(7)-C(6)-C(20)-C(25)	136.2(3)
C(14)-C(6)-C(20)-C(25)	-96.7(3)
C(5)-C(6)-C(20)-C(25)	26.4(4)
C(25)-C(20)-C(21)-C(22)	-2.1(4)
C(6)-C(20)-C(21)-C(22)	176.6(3)
C(20)-C(21)-C(22)-C(23)	1.4(5)
C(21)-C(22)-C(23)-C(24)	-0.2(5)
C(22)-C(23)-C(24)-C(25)	-0.4(6)
C(23)-C(24)-C(25)-C(20)	-0.3(5)
C(21)-C(20)-C(25)-C(24)	1.5(5)
C(6)-C(20)-C(25)-C(24)	-177.1(3)
O(3)-S(1)-C(26)-C(31)	-158.7(2)
O(4)-S(1)-C(26)-C(31)	-26.4(3)
N(1)-S(1)-C(26)-C(31)	86.5(2)
O(3)-S(1)-C(26)-C(27)	23.5(3)
O(4)-S(1)-C(26)-C(27)	155.8(2)
N(1)-S(1)-C(26)-C(27)	-91.3(2)
C(31)-C(26)-C(27)-C(28)	-0.5(4)
S(1)-C(26)-C(27)-C(28)	177.3(2)

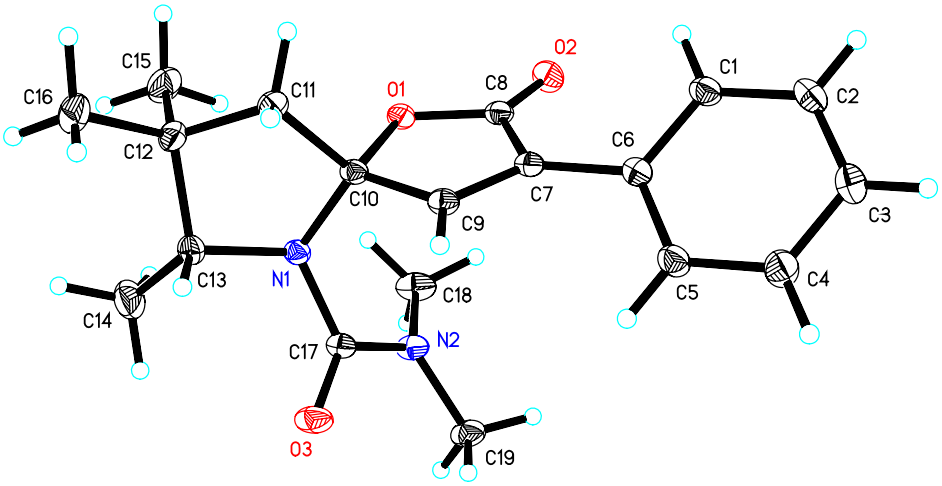
C(26)-C(27)-C(28)-C(29)	-1.0(4)
C(27)-C(28)-C(29)-C(30)	1.9(5)
C(27)-C(28)-C(29)-C(32)	-176.6(3)
C(28)-C(29)-C(30)-C(31)	-1.4(5)
C(32)-C(29)-C(30)-C(31)	177.1(3)
C(29)-C(30)-C(31)-C(26)	0.0(4)
C(27)-C(26)-C(31)-C(30)	1.0(4)
S(1)-C(26)-C(31)-C(30)	-176.8(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd213171 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

6.2 The X-ray of compound 3n



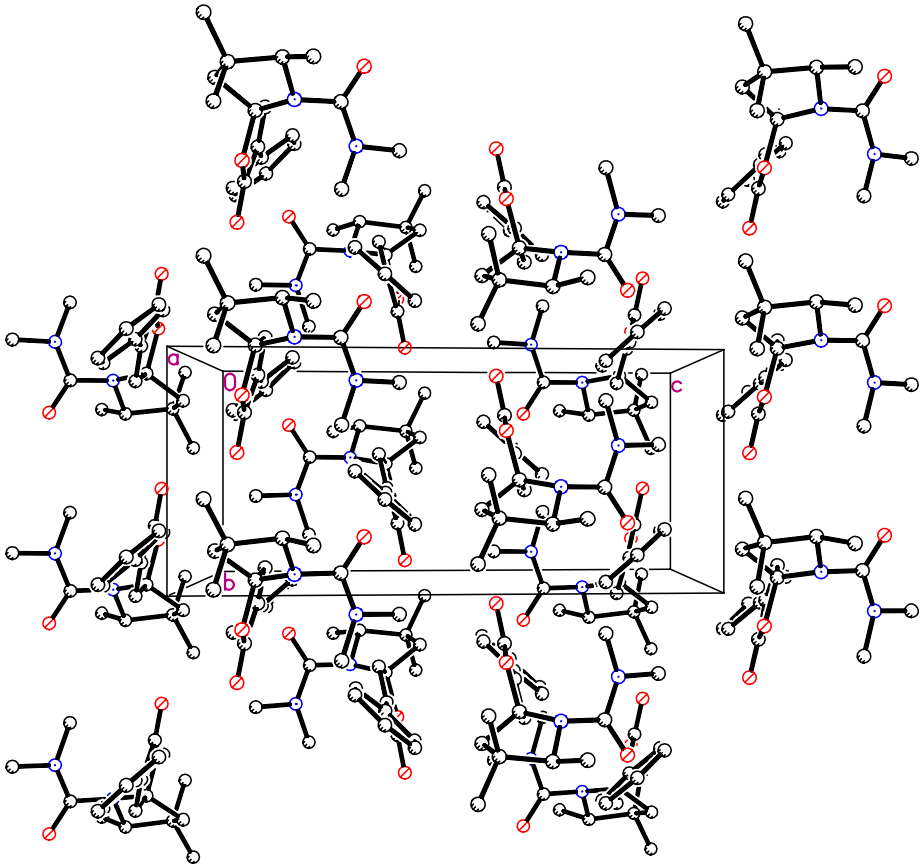


Table 1. Crystal data and structure refinement for dm14072.

Identification code	dm14072	
Empirical formula	C19 H24 N2 O3	
Formula weight	328.40	
Temperature	140(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 17.8501(14) Å	α= 90°.
	b = 6.6794(5) Å	β= 97.213(2)°.
	c = 15.1970(12) Å	γ = 90°.
Volume	1797.6(2) Å^3	
Z	4	
Density (calculated)	1.213 Mg/m^3	

Absorption coefficient	0.082 mm^{-1}
F(000)	704
Crystal size	0.260 x 0.100 x 0.080 mm^3
Theta range for data collection	1.150 to 30.538°.
Index ranges	-25<=h<=25, -9<=k<=8, -20<=l<=21
Reflections collected	17524
Independent reflections	5472 [R(int) = 0.0370]
Completeness to theta = 25.242°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7461 and 0.6703
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5472 / 0 / 222
Goodness-of-fit on F^2	1.036
Final R indices [I>2sigma(I)]	R1 = 0.0468, wR2 = 0.1240
R indices (all data)	R1 = 0.0735, wR2 = 0.1458
Extinction coefficient	n/a
Largest diff. peak and hole	0.380 and -0.206 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for dm14072. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	1888(1)	4338(1)	2884(1)	22(1)
N(2)	2362(1)	6115(2)	1757(1)	28(1)
O(1)	2475(1)	6758(1)	3904(1)	25(1)
O(2)	3343(1)	9184(1)	4079(1)	33(1)
O(3)	2138(1)	2786(1)	1598(1)	36(1)
C(1)	4932(1)	7254(2)	4305(1)	29(1)
C(2)	5708(1)	7369(2)	4322(1)	33(1)
C(3)	6070(1)	6249(2)	3742(1)	34(1)
C(4)	5652(1)	4997(2)	3138(1)	36(1)
C(5)	4877(1)	4867(2)	3122(1)	31(1)
C(6)	4508(1)	5989(2)	3706(1)	24(1)
C(7)	3690(1)	5785(2)	3709(1)	22(1)
C(8)	3190(1)	7453(2)	3922(1)	24(1)
C(9)	3248(1)	4191(2)	3535(1)	24(1)
C(10)	2446(1)	4632(2)	3655(1)	22(1)
C(11)	2149(1)	3367(2)	4377(1)	28(1)
C(12)	1311(1)	3031(2)	4062(1)	29(1)
C(13)	1343(1)	2782(2)	3065(1)	27(1)
C(14)	610(1)	3030(3)	2456(1)	48(1)
C(15)	842(1)	4848(3)	4270(1)	43(1)
C(16)	1012(1)	1140(3)	4465(1)	44(1)
C(17)	2132(1)	4320(2)	2035(1)	23(1)
C(18)	2074(1)	8032(2)	2016(1)	39(1)
C(19)	2720(1)	6168(2)	949(1)	36(1)

Table 3. Bond lengths [Å] and angles [°] for dm14072.

N(1)-C(17)	1.4124(16)
N(1)-C(10)	1.4537(16)
N(1)-C(13)	1.4725(16)
N(2)-C(17)	1.3523(15)
N(2)-C(18)	1.4521(17)
N(2)-C(19)	1.4533(18)
O(1)-C(8)	1.3558(15)
O(1)-C(10)	1.4683(14)
O(2)-C(8)	1.2047(15)
O(3)-C(17)	1.2214(14)
C(1)-C(2)	1.385(2)
C(1)-C(6)	1.3933(18)
C(1)-H(1)	0.9500
C(2)-C(3)	1.378(2)
C(2)-H(2)	0.9500
C(3)-C(4)	1.388(2)
C(3)-H(3)	0.9500
C(4)-C(5)	1.382(2)
C(4)-H(4)	0.9500
C(5)-C(6)	1.3894(19)
C(5)-H(5)	0.9500
C(6)-C(7)	1.4681(18)
C(7)-C(9)	1.3314(17)
C(7)-C(8)	1.4876(17)
C(9)-C(10)	1.4948(18)
C(9)-H(9)	0.9500
C(10)-C(11)	1.5315(17)
C(11)-C(12)	1.5295(19)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-C(16)	1.529(2)
C(12)-C(15)	1.530(2)
C(12)-C(13)	1.5333(19)
C(13)-C(14)	1.515(2)
C(13)-H(13)	1.0000
C(14)-H(14A)	0.9800

C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800
C(19)-H(19C)	0.9800

C(17)-N(1)-C(10)	118.61(10)
C(17)-N(1)-C(13)	116.81(10)
C(10)-N(1)-C(13)	110.25(10)
C(17)-N(2)-C(18)	124.45(12)
C(17)-N(2)-C(19)	117.93(11)
C(18)-N(2)-C(19)	114.56(11)
C(8)-O(1)-C(10)	109.81(9)
C(2)-C(1)-C(6)	120.38(13)
C(2)-C(1)-H(1)	119.8
C(6)-C(1)-H(1)	119.8
C(3)-C(2)-C(1)	120.44(13)
C(3)-C(2)-H(2)	119.8
C(1)-C(2)-H(2)	119.8
C(2)-C(3)-C(4)	119.54(13)
C(2)-C(3)-H(3)	120.2
C(4)-C(3)-H(3)	120.2
C(5)-C(4)-C(3)	120.26(14)
C(5)-C(4)-H(4)	119.9
C(3)-C(4)-H(4)	119.9
C(4)-C(5)-C(6)	120.54(12)
C(4)-C(5)-H(5)	119.7
C(6)-C(5)-H(5)	119.7
C(5)-C(6)-C(1)	118.84(12)

C(5)-C(6)-C(7)	120.25(11)
C(1)-C(6)-C(7)	120.87(12)
C(9)-C(7)-C(6)	129.68(11)
C(9)-C(7)-C(8)	106.61(11)
C(6)-C(7)-C(8)	123.71(10)
O(2)-C(8)-O(1)	121.48(12)
O(2)-C(8)-C(7)	129.39(12)
O(1)-C(8)-C(7)	109.12(10)
C(7)-C(9)-C(10)	111.74(11)
C(7)-C(9)-H(9)	124.1
C(10)-C(9)-H(9)	124.1
N(1)-C(10)-O(1)	109.63(9)
N(1)-C(10)-C(9)	116.43(10)
O(1)-C(10)-C(9)	102.64(9)
N(1)-C(10)-C(11)	103.87(10)
O(1)-C(10)-C(11)	110.69(10)
C(9)-C(10)-C(11)	113.67(10)
C(12)-C(11)-C(10)	105.63(10)
C(12)-C(11)-H(11A)	110.6
C(10)-C(11)-H(11A)	110.6
C(12)-C(11)-H(11B)	110.6
C(10)-C(11)-H(11B)	110.6
H(11A)-C(11)-H(11B)	108.7
C(16)-C(12)-C(11)	111.85(12)
C(16)-C(12)-C(15)	110.22(13)
C(11)-C(12)-C(15)	110.99(12)
C(16)-C(12)-C(13)	111.38(12)
C(11)-C(12)-C(13)	99.70(10)
C(15)-C(12)-C(13)	112.34(12)
N(1)-C(13)-C(14)	110.41(11)
N(1)-C(13)-C(12)	102.46(10)
C(14)-C(13)-C(12)	117.04(12)
N(1)-C(13)-H(13)	108.9
C(14)-C(13)-H(13)	108.9
C(12)-C(13)-H(13)	108.9
C(13)-C(14)-H(14A)	109.5
C(13)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5

C(13)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(12)-C(15)-H(15A)	109.5
C(12)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(12)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(12)-C(16)-H(16A)	109.5
C(12)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(12)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
O(3)-C(17)-N(2)	123.39(12)
O(3)-C(17)-N(1)	121.88(11)
N(2)-C(17)-N(1)	114.73(10)
N(2)-C(18)-H(18A)	109.5
N(2)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
N(2)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
N(2)-C(19)-H(19A)	109.5
N(2)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
N(2)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for dm14072. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	27(1)	20(1)	19(1)	0(1)	3(1)	-2(1)
N(2)	44(1)	18(1)	22(1)	1(1)	8(1)	0(1)
O(1)	29(1)	21(1)	25(1)	-6(1)	6(1)	-2(1)
O(2)	39(1)	22(1)	40(1)	-9(1)	7(1)	-4(1)
O(3)	58(1)	21(1)	29(1)	-6(1)	12(1)	-2(1)
C(1)	34(1)	27(1)	26(1)	-4(1)	2(1)	-4(1)
C(2)	34(1)	31(1)	33(1)	-2(1)	-6(1)	-6(1)
C(3)	27(1)	31(1)	42(1)	2(1)	-2(1)	-1(1)
C(4)	30(1)	34(1)	44(1)	-8(1)	5(1)	2(1)
C(5)	30(1)	29(1)	33(1)	-8(1)	2(1)	-1(1)
C(6)	28(1)	21(1)	23(1)	2(1)	1(1)	-1(1)
C(7)	28(1)	21(1)	18(1)	-1(1)	2(1)	-1(1)
C(8)	32(1)	22(1)	19(1)	-3(1)	5(1)	-3(1)
C(9)	29(1)	20(1)	22(1)	-1(1)	3(1)	2(1)
C(10)	28(1)	18(1)	20(1)	-1(1)	3(1)	-1(1)
C(11)	34(1)	29(1)	22(1)	4(1)	4(1)	-4(1)
C(12)	32(1)	28(1)	28(1)	1(1)	9(1)	-5(1)
C(13)	31(1)	23(1)	27(1)	0(1)	4(1)	-6(1)
C(14)	38(1)	60(1)	43(1)	5(1)	-5(1)	-17(1)
C(15)	38(1)	49(1)	47(1)	-5(1)	19(1)	2(1)
C(16)	48(1)	46(1)	40(1)	9(1)	10(1)	-16(1)
C(17)	30(1)	19(1)	20(1)	0(1)	2(1)	2(1)
C(18)	67(1)	18(1)	31(1)	1(1)	8(1)	5(1)
C(19)	55(1)	32(1)	24(1)	2(1)	13(1)	-7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for dm14072.

	x	y	z	U(eq)
H(1)	4687	8041	4704	35
H(2)	5993	8225	4737	40
H(3)	6603	6334	3754	41
H(4)	5898	4227	2735	43
H(5)	4595	4002	2709	37
H(9)	3421	2928	3357	28
H(11A)	2420	2073	4449	34
H(11B)	2216	4082	4953	34
H(13)	1562	1438	2957	32
H(14A)	450	4434	2457	72
H(14B)	220	2183	2665	72
H(14C)	684	2635	1852	72
H(15A)	309	4602	4058	65
H(15B)	1015	6033	3973	65
H(15C)	902	5073	4913	65
H(16A)	1063	1275	5112	67
H(16B)	1304	-21	4308	67
H(16C)	479	953	4234	67
H(18A)	1787	8670	1499	58
H(18B)	2498	8896	2247	58
H(18C)	1744	7824	2477	58
H(19A)	2937	4853	850	55
H(19B)	3121	7180	1008	55
H(19C)	2343	6507	446	55

Table 6. Torsion angles [°] for dm14072.

C(6)-C(1)-C(2)-C(3)	0.6(2)
C(1)-C(2)-C(3)-C(4)	-0.1(2)
C(2)-C(3)-C(4)-C(5)	-0.3(2)
C(3)-C(4)-C(5)-C(6)	0.2(2)
C(4)-C(5)-C(6)-C(1)	0.2(2)
C(4)-C(5)-C(6)-C(7)	-177.59(13)
C(2)-C(1)-C(6)-C(5)	-0.7(2)
C(2)-C(1)-C(6)-C(7)	177.16(12)
C(5)-C(6)-C(7)-C(9)	32.0(2)
C(1)-C(6)-C(7)-C(9)	-145.78(14)
C(5)-C(6)-C(7)-C(8)	-147.86(13)
C(1)-C(6)-C(7)-C(8)	34.36(18)
C(10)-O(1)-C(8)-O(2)	177.30(12)
C(10)-O(1)-C(8)-C(7)	-1.70(13)
C(9)-C(7)-C(8)-O(2)	-176.22(14)
C(6)-C(7)-C(8)-O(2)	3.7(2)
C(9)-C(7)-C(8)-O(1)	2.68(14)
C(6)-C(7)-C(8)-O(1)	-177.44(11)
C(6)-C(7)-C(9)-C(10)	177.56(12)
C(8)-C(7)-C(9)-C(10)	-2.56(14)
C(17)-N(1)-C(10)-O(1)	95.38(12)
C(13)-N(1)-C(10)-O(1)	-126.13(10)
C(17)-N(1)-C(10)-C(9)	-20.52(15)
C(13)-N(1)-C(10)-C(9)	117.97(11)
C(17)-N(1)-C(10)-C(11)	-146.29(10)
C(13)-N(1)-C(10)-C(11)	-7.80(13)
C(8)-O(1)-C(10)-N(1)	-124.16(11)
C(8)-O(1)-C(10)-C(9)	0.20(12)
C(8)-O(1)-C(10)-C(11)	121.84(11)
C(7)-C(9)-C(10)-N(1)	121.31(12)
C(7)-C(9)-C(10)-O(1)	1.57(13)
C(7)-C(9)-C(10)-C(11)	-118.01(12)
N(1)-C(10)-C(11)-C(12)	-19.42(13)
O(1)-C(10)-C(11)-C(12)	98.18(12)
C(9)-C(10)-C(11)-C(12)	-146.92(11)
C(10)-C(11)-C(12)-C(16)	155.32(12)

C(10)-C(11)-C(12)-C(15)	-81.12(14)
C(10)-C(11)-C(12)-C(13)	37.48(12)
C(17)-N(1)-C(13)-C(14)	-63.62(15)
C(10)-N(1)-C(13)-C(14)	157.06(12)
C(17)-N(1)-C(13)-C(12)	171.01(10)
C(10)-N(1)-C(13)-C(12)	31.69(13)
C(16)-C(12)-C(13)-N(1)	-159.41(12)
C(11)-C(12)-C(13)-N(1)	-41.22(12)
C(15)-C(12)-C(13)-N(1)	76.38(14)
C(16)-C(12)-C(13)-C(14)	79.68(16)
C(11)-C(12)-C(13)-C(14)	-162.13(12)
C(15)-C(12)-C(13)-C(14)	-44.53(16)
C(18)-N(2)-C(17)-O(3)	151.18(14)
C(19)-N(2)-C(17)-O(3)	-7.9(2)
C(18)-N(2)-C(17)-N(1)	-29.46(18)
C(19)-N(2)-C(17)-N(1)	171.48(12)
C(10)-N(1)-C(17)-O(3)	109.31(14)
C(13)-N(1)-C(17)-O(3)	-26.53(18)
C(10)-N(1)-C(17)-N(2)	-70.06(14)
C(13)-N(1)-C(17)-N(2)	154.10(11)

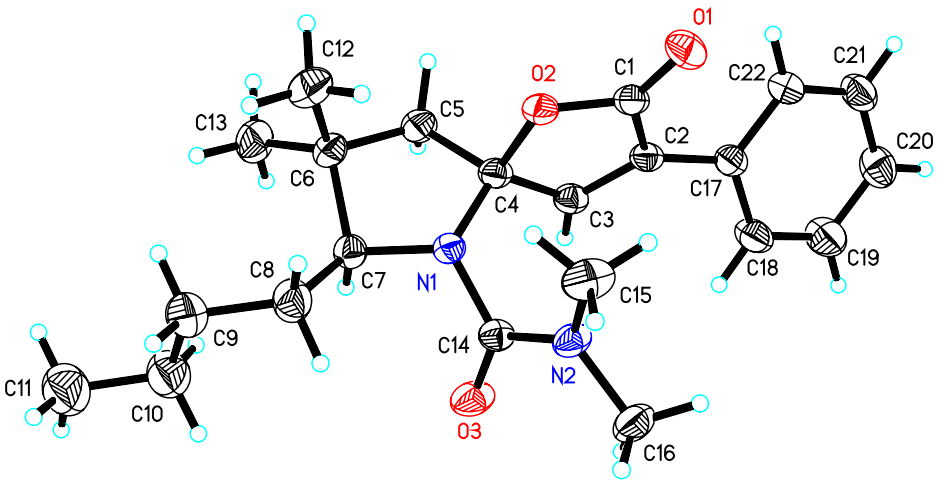
Symmetry transformations used to generate equivalent atoms:

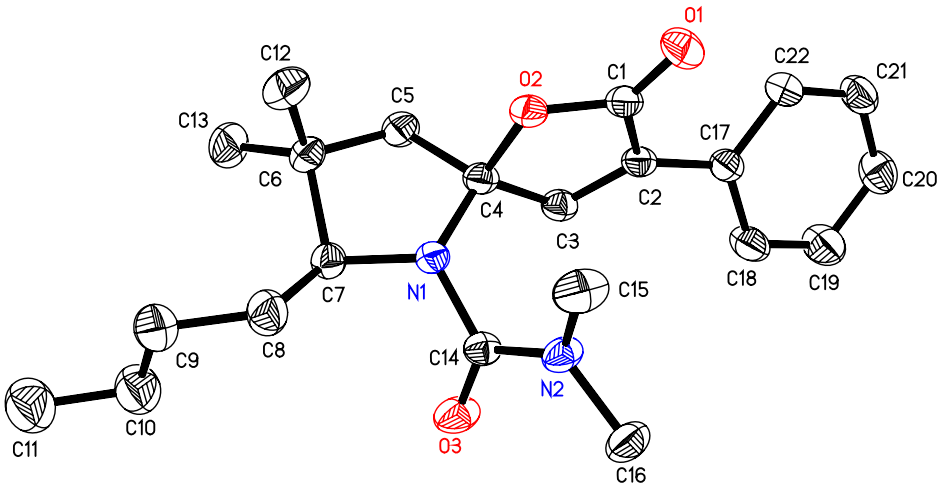
Table 7. Hydrogen bonds for dm14072 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(11)-H(11A)...O(2)#1	0.99	2.64	3.5772(17)	157.4
C(18)-H(18C)...O(1)	0.98	2.49	2.9915(18)	111.5
C(19)-H(19C)...O(1)#2	0.98	2.65	3.3816(17)	131.5

Symmetry transformations used to generate equivalent atoms:
#1 x,y-1,z #2 x,-y+3/2,z-1/2

6.3 The X-ray of compound 3o




O1
C12
C1
C22
C21
O2
C5
C13
C17
C6
C4
C2
C20
C3
C18
C19
C7
N1
C15
C9
C8
C14
N2
C10
O3
C11
C16

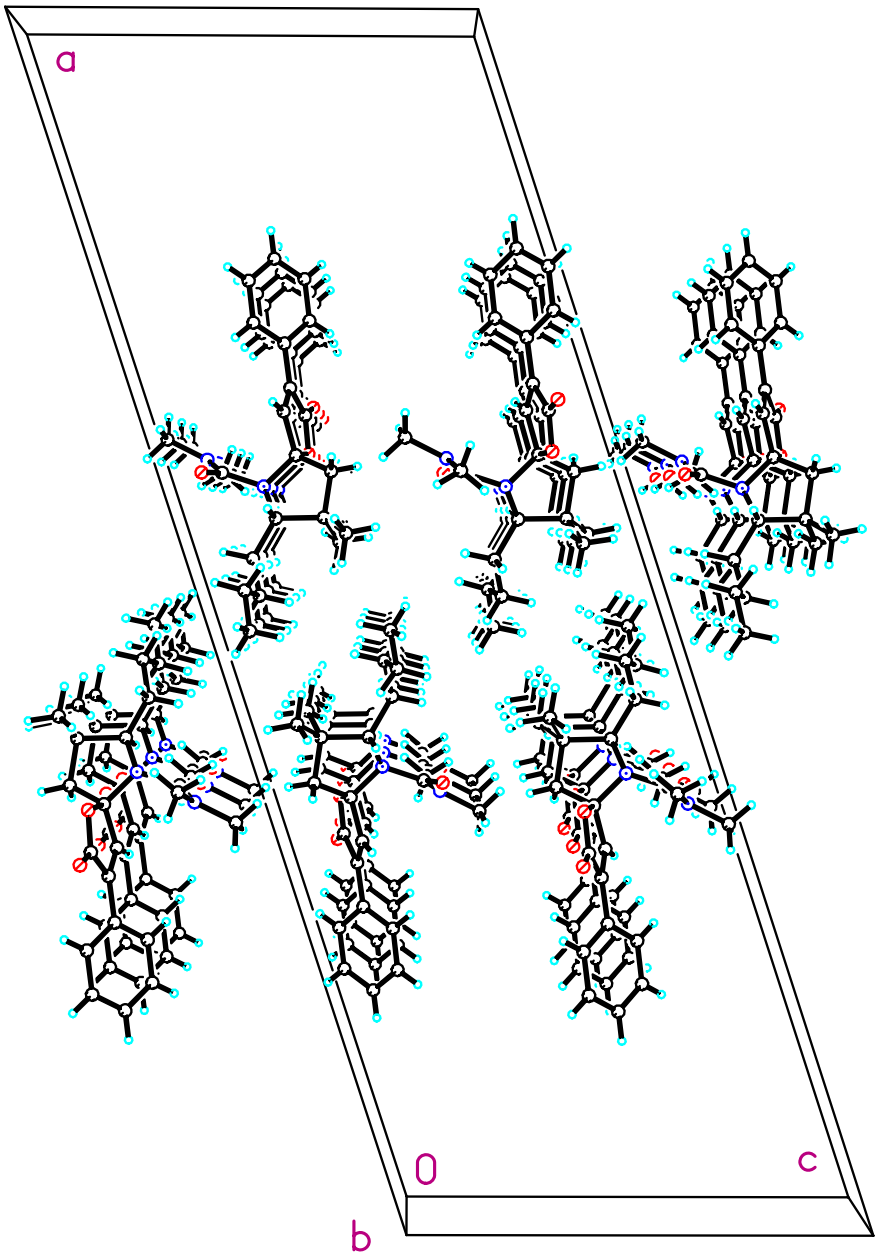

a
0
c
b

Table 1. Crystal data and structure refinement for cd21467.

Identification code	cd21467	
Empirical formula	C22 H30 N2 O3	
Formula weight	370.48	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 42.588(8) Å	α= 90°.
	b = 6.6428(12) Å	β= 108.203(4)°.
	c = 15.520(3) Å	γ = 90°.
Volume	4171.0(13) $Å^3$	
Z	8	
Density (calculated)	1.180 Mg/m^3	
Absorption coefficient	0.078 mm^{-1}	
F(000)	1600	
Crystal size	0.211 x 0.165 x 0.123 mm^3	
Theta range for data collection	2.014 to 25.999°.	
Index ranges	-43<=h<=52, -8<=k<=7, -19<=l<=19	
Reflections collected	11845	
Independent reflections	4092 [R(int) = 0.0444]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7456 and 0.5916	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4092 / 0 / 249	
Goodness-of-fit on F^2	1.053	
Final R indices [I>2sigma(I)]	R1 = 0.0633, wR2 = 0.1694	
R indices (all data)	R1 = 0.0863, wR2 = 0.1856	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.407 and -0.205 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for cd21467. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	1121(1)	8000(3)	2215(1)	38(1)
N(2)	1336(1)	6415(3)	1183(1)	46(1)
O(1)	1777(1)	3206(2)	3750(1)	61(1)
O(2)	1390(1)	5586(2)	3348(1)	44(1)
O(3)	1227(1)	9728(3)	1050(1)	62(1)
C(1)	1705(1)	4926(3)	3538(1)	43(1)
C(2)	1917(1)	6609(3)	3423(1)	40(1)
C(3)	1720(1)	8177(3)	3142(1)	42(1)
C(4)	1372(1)	7715(3)	3092(1)	40(1)
C(5)	1243(1)	8966(4)	3732(2)	49(1)
C(6)	874(1)	9240(4)	3273(2)	48(1)
C(7)	867(1)	9463(3)	2275(2)	44(1)
C(8)	548(1)	9027(5)	1521(2)	62(1)
C(9)	255(1)	10410(5)	1432(2)	72(1)
C(10)	302(1)	12547(5)	1207(2)	81(1)
C(11)	-5(1)	13826(6)	1075(3)	101(1)
C(12)	681(1)	7393(5)	3413(2)	72(1)
C(13)	754(1)	11116(5)	3642(2)	71(1)
C(14)	1229(1)	8140(3)	1439(1)	39(1)
C(15)	1237(1)	4423(4)	1377(2)	66(1)
C(16)	1497(1)	6482(4)	485(2)	63(1)
C(17)	2276(1)	6450(3)	3602(1)	43(1)
C(18)	2431(1)	7588(4)	3105(2)	60(1)
C(19)	2768(1)	7516(5)	3292(2)	72(1)
C(20)	2958(1)	6295(4)	3969(2)	64(1)
C(21)	2808(1)	5166(4)	4464(2)	61(1)
C(22)	2471(1)	5223(4)	4284(2)	54(1)

Table 3. Bond lengths [Å] and angles [°] for cd21467.

N(1)-C(14)	1.420(3)
N(1)-C(4)	1.457(3)
N(1)-C(7)	1.477(3)
N(2)-C(14)	1.339(3)
N(2)-C(15)	1.450(3)
N(2)-C(16)	1.452(3)
O(1)-C(1)	1.202(3)
O(2)-C(1)	1.355(3)
O(2)-C(4)	1.465(3)
O(3)-C(14)	1.214(3)
C(1)-C(2)	1.480(3)
C(2)-C(3)	1.323(3)
C(2)-C(17)	1.470(3)
C(3)-C(4)	1.492(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.522(3)
C(5)-C(6)	1.520(3)
C(5)-H(5A)	0.9700
C(5)-H(5B)	0.9700
C(6)-C(13)	1.526(3)
C(6)-C(12)	1.529(4)
C(6)-C(7)	1.548(3)
C(7)-C(8)	1.519(3)
C(7)-H(7)	0.9800
C(8)-C(9)	1.519(4)
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(9)-C(10)	1.490(4)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-C(11)	1.518(4)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(11)-H(11A)	0.9600
C(11)-H(11B)	0.9600
C(11)-H(11C)	0.9600

C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-H(16A)	0.9600
C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600
C(17)-C(18)	1.387(3)
C(17)-C(22)	1.387(3)
C(18)-C(19)	1.370(4)
C(18)-H(18)	0.9300
C(19)-C(20)	1.374(4)
C(19)-H(19)	0.9300
C(20)-C(21)	1.365(4)
C(20)-H(20)	0.9300
C(21)-C(22)	1.376(3)
C(21)-H(21)	0.9300
C(22)-H(22)	0.9300

C(14)-N(1)-C(4)	117.59(16)
C(14)-N(1)-C(7)	116.61(17)
C(4)-N(1)-C(7)	111.34(16)
C(14)-N(2)-C(15)	124.8(2)
C(14)-N(2)-C(16)	118.62(19)
C(15)-N(2)-C(16)	114.82(19)
C(1)-O(2)-C(4)	109.63(16)
O(1)-C(1)-O(2)	121.1(2)
O(1)-C(1)-C(2)	129.7(2)
O(2)-C(1)-C(2)	109.17(18)
C(3)-C(2)-C(17)	129.3(2)
C(3)-C(2)-C(1)	106.7(2)
C(17)-C(2)-C(1)	123.97(19)
C(2)-C(3)-C(4)	111.9(2)

C(2)-C(3)-H(3)	124.1
C(4)-C(3)-H(3)	124.1
N(1)-C(4)-O(2)	109.90(17)
N(1)-C(4)-C(3)	116.65(17)
O(2)-C(4)-C(3)	102.53(16)
N(1)-C(4)-C(5)	103.32(17)
O(2)-C(4)-C(5)	110.45(17)
C(3)-C(4)-C(5)	114.10(19)
C(6)-C(5)-C(4)	106.86(18)
C(6)-C(5)-H(5A)	110.4
C(4)-C(5)-H(5A)	110.4
C(6)-C(5)-H(5B)	110.4
C(4)-C(5)-H(5B)	110.4
H(5A)-C(5)-H(5B)	108.6
C(5)-C(6)-C(13)	110.0(2)
C(5)-C(6)-C(12)	111.1(2)
C(13)-C(6)-C(12)	110.2(2)
C(5)-C(6)-C(7)	100.01(16)
C(13)-C(6)-C(7)	113.2(2)
C(12)-C(6)-C(7)	112.1(2)
N(1)-C(7)-C(8)	108.82(19)
N(1)-C(7)-C(6)	102.39(17)
C(8)-C(7)-C(6)	119.0(2)
N(1)-C(7)-H(7)	108.7
C(8)-C(7)-H(7)	108.7
C(6)-C(7)-H(7)	108.7
C(7)-C(8)-C(9)	117.8(2)
C(7)-C(8)-H(8A)	107.9
C(9)-C(8)-H(8A)	107.9
C(7)-C(8)-H(8B)	107.9
C(9)-C(8)-H(8B)	107.9
H(8A)-C(8)-H(8B)	107.2
C(10)-C(9)-C(8)	115.5(2)
C(10)-C(9)-H(9A)	108.4
C(8)-C(9)-H(9A)	108.4
C(10)-C(9)-H(9B)	108.4
C(8)-C(9)-H(9B)	108.4
H(9A)-C(9)-H(9B)	107.5

C(9)-C(10)-C(11)	113.0(3)
C(9)-C(10)-H(10A)	109.0
C(11)-C(10)-H(10A)	109.0
C(9)-C(10)-H(10B)	109.0
C(11)-C(10)-H(10B)	109.0
H(10A)-C(10)-H(10B)	107.8
C(10)-C(11)-H(11A)	109.5
C(10)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(10)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(6)-C(12)-H(12A)	109.5
C(6)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(6)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(6)-C(13)-H(13A)	109.5
C(6)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(6)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
O(3)-C(14)-N(2)	123.0(2)
O(3)-C(14)-N(1)	121.71(19)
N(2)-C(14)-N(1)	115.25(18)
N(2)-C(15)-H(15A)	109.5
N(2)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
N(2)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
N(2)-C(16)-H(16A)	109.5
N(2)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
N(2)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5

H(16B)-C(16)-H(16C)	109.5
C(18)-C(17)-C(22)	117.9(2)
C(18)-C(17)-C(2)	120.3(2)
C(22)-C(17)-C(2)	121.7(2)
C(19)-C(18)-C(17)	120.8(2)
C(19)-C(18)-H(18)	119.6
C(17)-C(18)-H(18)	119.6
C(18)-C(19)-C(20)	120.7(3)
C(18)-C(19)-H(19)	119.7
C(20)-C(19)-H(19)	119.7
C(21)-C(20)-C(19)	119.2(3)
C(21)-C(20)-H(20)	120.4
C(19)-C(20)-H(20)	120.4
C(20)-C(21)-C(22)	120.8(2)
C(20)-C(21)-H(21)	119.6
C(22)-C(21)-H(21)	119.6
C(21)-C(22)-C(17)	120.6(2)
C(21)-C(22)-H(22)	119.7
C(17)-C(22)-H(22)	119.7

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for cd21467. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	43(1)	40(1)	34(1)	1(1)	14(1)	4(1)
N(2)	67(1)	35(1)	44(1)	-2(1)	28(1)	0(1)
O(1)	72(1)	40(1)	74(1)	17(1)	26(1)	8(1)
O(2)	53(1)	40(1)	45(1)	10(1)	22(1)	2(1)
O(3)	100(1)	39(1)	62(1)	10(1)	45(1)	6(1)
C(1)	55(1)	41(1)	36(1)	5(1)	18(1)	4(1)
C(2)	51(1)	37(1)	32(1)	2(1)	14(1)	3(1)
C(3)	51(1)	36(1)	40(1)	3(1)	15(1)	-1(1)
C(4)	50(1)	36(1)	36(1)	4(1)	15(1)	2(1)
C(5)	58(2)	53(2)	38(1)	-4(1)	18(1)	6(1)
C(6)	53(1)	51(1)	45(1)	-1(1)	23(1)	6(1)
C(7)	46(1)	42(1)	46(1)	1(1)	18(1)	4(1)
C(8)	48(2)	78(2)	58(2)	-4(1)	12(1)	3(1)
C(9)	58(2)	85(2)	70(2)	6(2)	17(1)	2(1)
C(10)	71(2)	78(2)	94(2)	15(2)	27(2)	4(2)
C(11)	87(2)	97(3)	119(3)	26(2)	33(2)	9(2)
C(12)	73(2)	80(2)	77(2)	14(2)	43(2)	-1(2)
C(13)	75(2)	83(2)	59(2)	-13(2)	26(2)	20(2)
C(14)	48(1)	33(1)	38(1)	0(1)	14(1)	-3(1)
C(15)	109(2)	38(1)	61(2)	-8(1)	39(2)	-10(1)
C(16)	88(2)	58(2)	57(2)	-1(1)	44(2)	9(1)
C(17)	49(1)	39(1)	38(1)	0(1)	11(1)	4(1)
C(18)	53(2)	62(2)	62(2)	23(1)	14(1)	4(1)
C(19)	54(2)	76(2)	88(2)	26(2)	22(2)	-3(1)
C(20)	46(1)	66(2)	75(2)	3(1)	10(1)	1(1)
C(21)	53(2)	65(2)	55(2)	9(1)	5(1)	12(1)
C(22)	59(2)	56(2)	47(1)	12(1)	16(1)	9(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for cd21467.

	x	y	z	U(eq)
H(3)	1790	9418	2994	50
H(5A)	1283	8278	4308	59
H(5B)	1353	10263	3842	59
H(7)	942	10821	2188	52
H(8A)	480	7666	1604	75
H(8B)	596	9053	950	75
H(9A)	65	9875	964	86
H(9B)	204	10383	1999	86
H(10A)	364	12586	655	97
H(10B)	483	13122	1691	97
H(11A)	-188	13222	622	151
H(11B)	33	15152	883	151
H(11C)	-54	13912	1637	151
H(12A)	757	6225	3170	109
H(12B)	450	7590	3107	109
H(12C)	717	7203	4049	109
H(13A)	520	11264	3358	106
H(13B)	865	12279	3514	106
H(13C)	800	10986	4286	106
H(15A)	1086	4538	1722	100
H(15B)	1428	3678	1720	100
H(15C)	1130	3733	818	100
H(16A)	1343	6084	-86	95
H(16B)	1682	5580	642	95
H(16C)	1572	7827	437	95
H(18)	2306	8410	2640	72
H(19)	2868	8300	2956	87
H(20)	3186	6239	4090	77
H(21)	2936	4347	4927	73
H(22)	2373	4432	4623	65

Table 6. Torsion angles [°] for cd21467.

C(4)-O(2)-C(1)-O(1)	-178.7(2)
C(4)-O(2)-C(1)-C(2)	0.4(2)
O(1)-C(1)-C(2)-C(3)	177.0(2)
O(2)-C(1)-C(2)-C(3)	-2.0(2)
O(1)-C(1)-C(2)-C(17)	-3.1(4)
O(2)-C(1)-C(2)-C(17)	177.86(18)
C(17)-C(2)-C(3)-C(4)	-177.1(2)
C(1)-C(2)-C(3)-C(4)	2.8(2)
C(14)-N(1)-C(4)-O(2)	-99.1(2)
C(7)-N(1)-C(4)-O(2)	122.59(18)
C(14)-N(1)-C(4)-C(3)	17.0(3)
C(7)-N(1)-C(4)-C(3)	-121.3(2)
C(14)-N(1)-C(4)-C(5)	143.02(19)
C(7)-N(1)-C(4)-C(5)	4.7(2)
C(1)-O(2)-C(4)-N(1)	125.85(17)
C(1)-O(2)-C(4)-C(3)	1.2(2)
C(1)-O(2)-C(4)-C(5)	-120.79(19)
C(2)-C(3)-C(4)-N(1)	-122.7(2)
C(2)-C(3)-C(4)-O(2)	-2.5(2)
C(2)-C(3)-C(4)-C(5)	116.9(2)
N(1)-C(4)-C(5)-C(6)	21.1(2)
O(2)-C(4)-C(5)-C(6)	-96.4(2)
C(3)-C(4)-C(5)-C(6)	148.70(19)
C(4)-C(5)-C(6)-C(13)	-156.3(2)
C(4)-C(5)-C(6)-C(12)	81.5(2)
C(4)-C(5)-C(6)-C(7)	-36.9(2)
C(14)-N(1)-C(7)-C(8)	66.7(2)
C(4)-N(1)-C(7)-C(8)	-154.56(19)
C(14)-N(1)-C(7)-C(6)	-166.48(17)
C(4)-N(1)-C(7)-C(6)	-27.7(2)
C(5)-C(6)-C(7)-N(1)	38.2(2)
C(13)-C(6)-C(7)-N(1)	155.1(2)
C(12)-C(6)-C(7)-N(1)	-79.5(2)
C(5)-C(6)-C(7)-C(8)	158.2(2)
C(13)-C(6)-C(7)-C(8)	-84.9(3)
C(12)-C(6)-C(7)-C(8)	40.4(3)

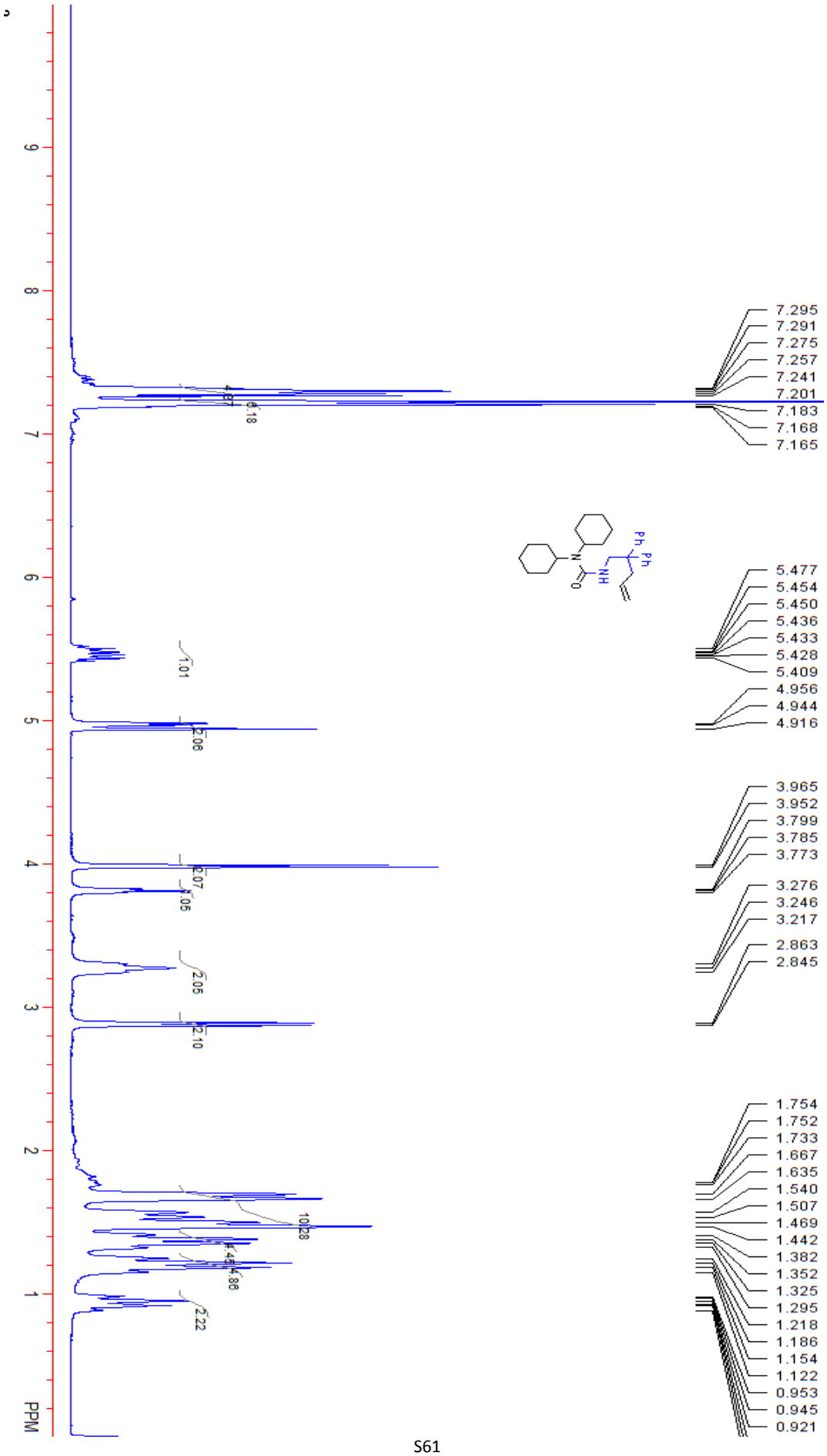
N(1)-C(7)-C(8)-C(9)	-177.7(2)
C(6)-C(7)-C(8)-C(9)	65.7(3)
C(7)-C(8)-C(9)-C(10)	63.6(4)
C(8)-C(9)-C(10)-C(11)	176.8(3)
C(15)-N(2)-C(14)-O(3)	-155.9(3)
C(16)-N(2)-C(14)-O(3)	8.3(4)
C(15)-N(2)-C(14)-N(1)	24.9(3)
C(16)-N(2)-C(14)-N(1)	-170.8(2)
C(4)-N(1)-C(14)-O(3)	-107.3(2)
C(7)-N(1)-C(14)-O(3)	28.9(3)
C(4)-N(1)-C(14)-N(2)	71.9(2)
C(7)-N(1)-C(14)-N(2)	-152.0(2)
C(3)-C(2)-C(17)-C(18)	-31.6(4)
C(1)-C(2)-C(17)-C(18)	148.5(2)
C(3)-C(2)-C(17)-C(22)	146.3(3)
C(1)-C(2)-C(17)-C(22)	-33.5(3)
C(22)-C(17)-C(18)-C(19)	-0.8(4)
C(2)-C(17)-C(18)-C(19)	177.3(3)
C(17)-C(18)-C(19)-C(20)	0.7(5)
C(18)-C(19)-C(20)-C(21)	-0.7(5)
C(19)-C(20)-C(21)-C(22)	0.7(5)
C(20)-C(21)-C(22)-C(17)	-0.7(4)
C(18)-C(17)-C(22)-C(21)	0.8(4)
C(2)-C(17)-C(22)-C(21)	-177.2(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd21467 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(15)-H(15A)...O(2)	0.96	2.55	3.022(3)	110.6

Symmetry transformations used to generate equivalent atoms: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3.785
3.773
3.276
3.246
3.217
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1.752
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N
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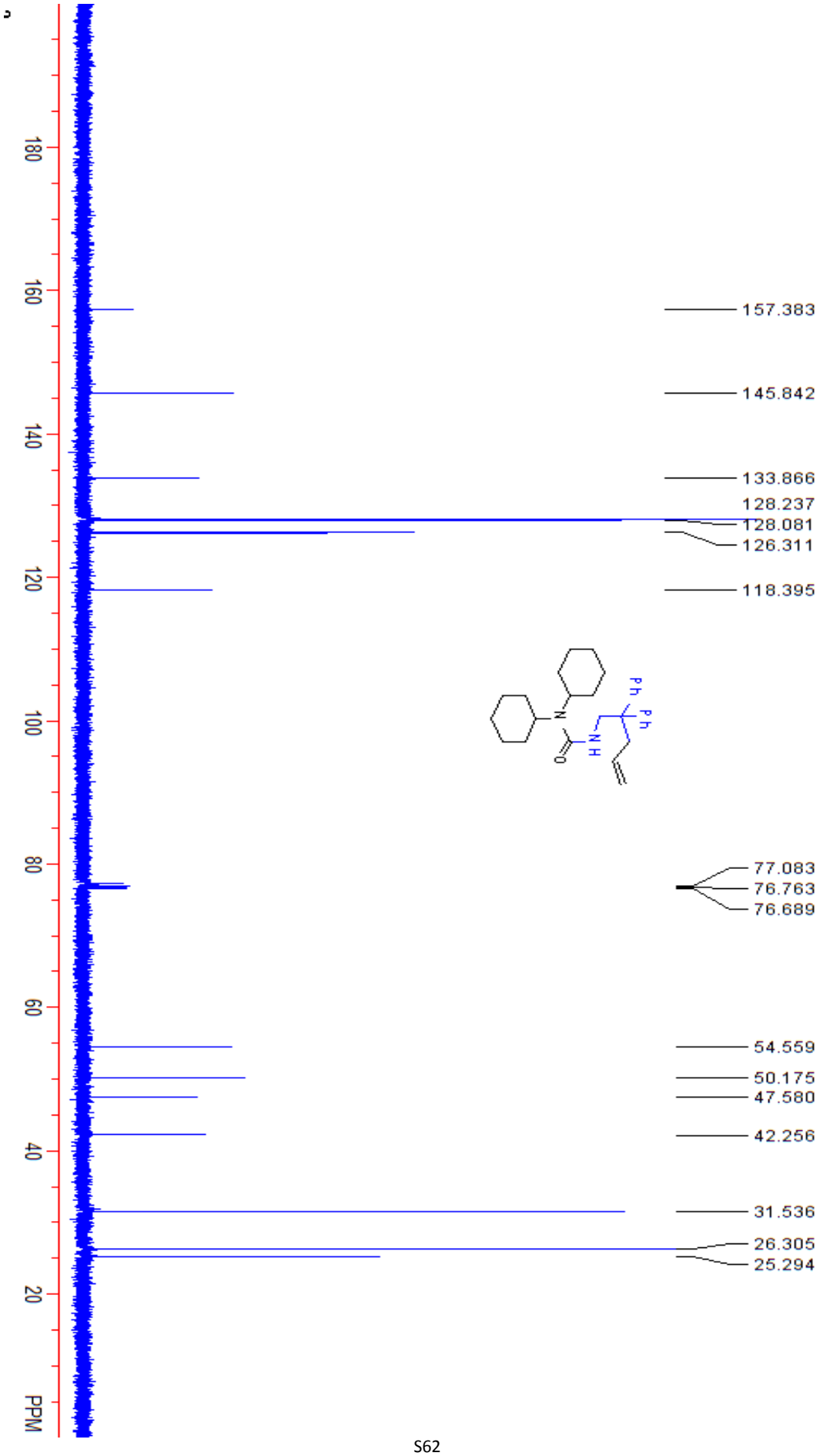
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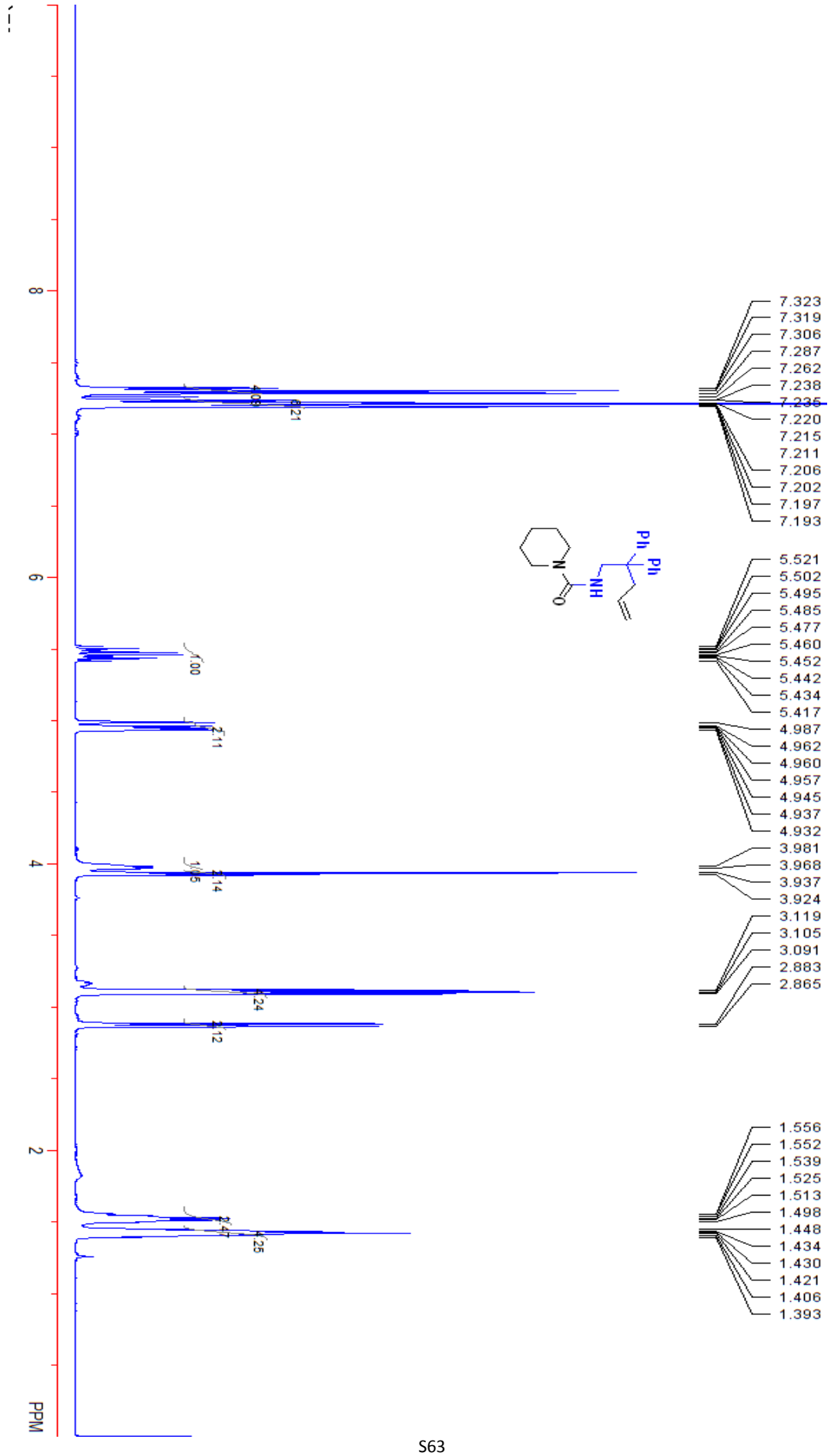
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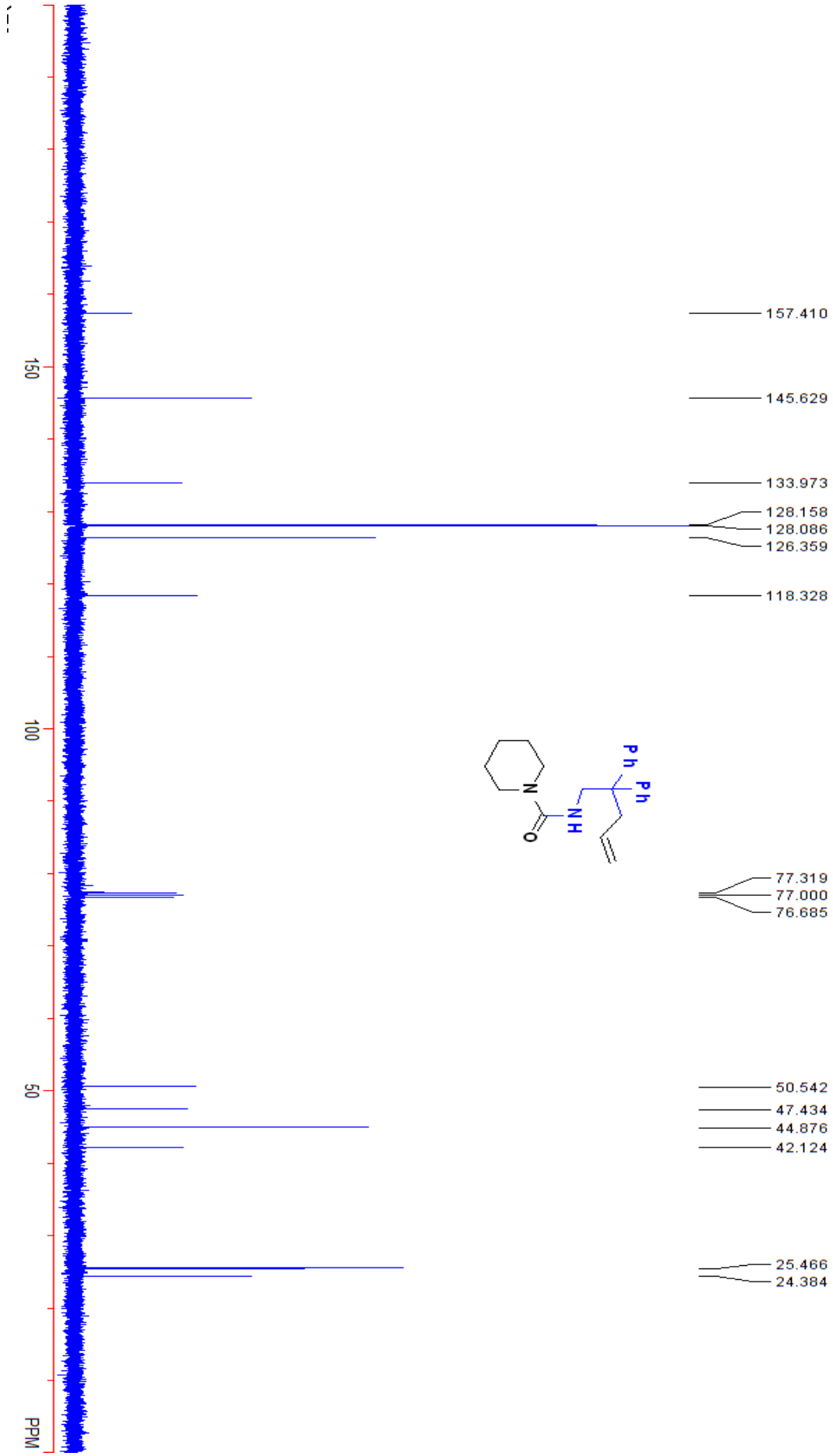
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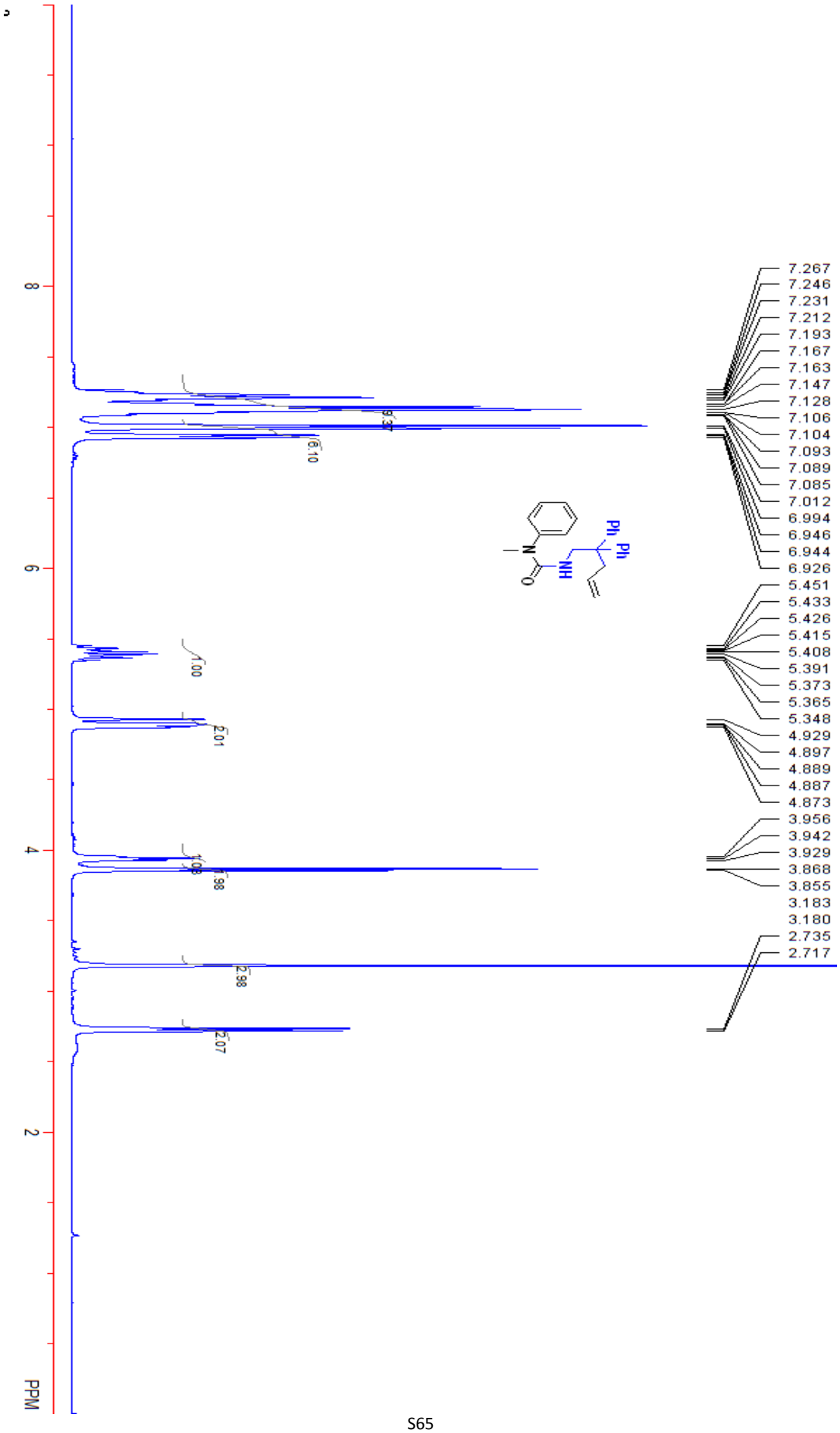
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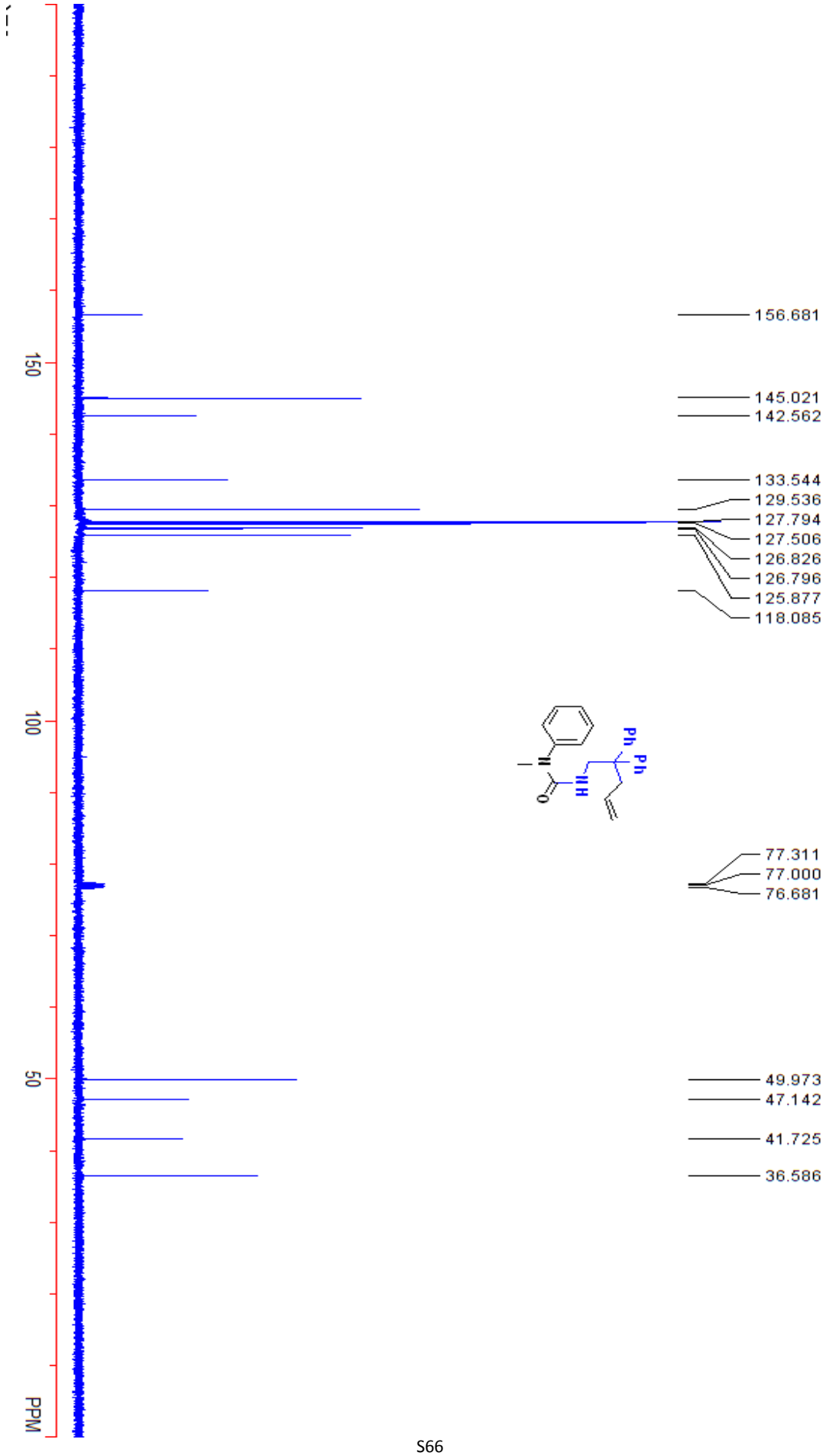
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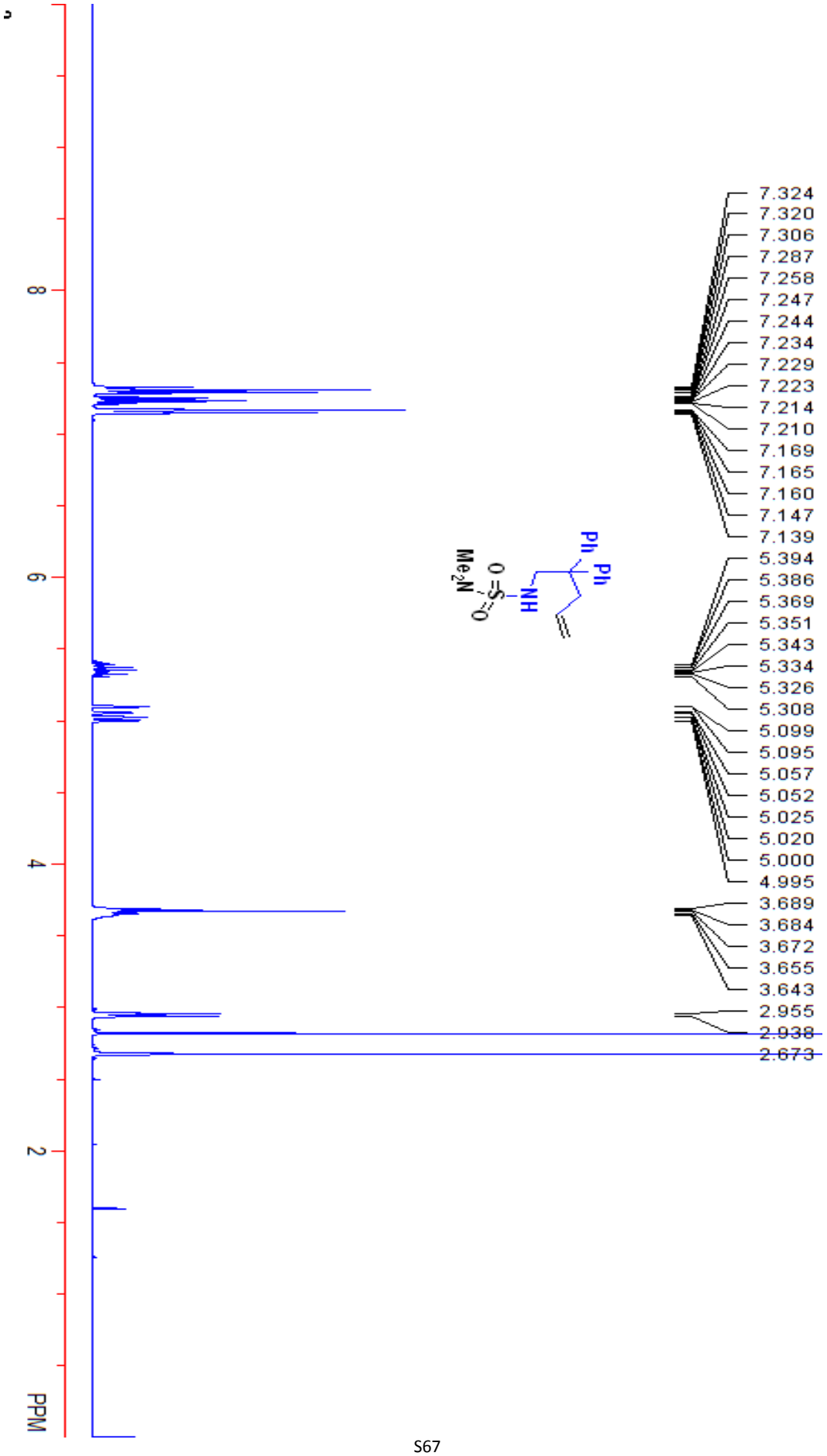
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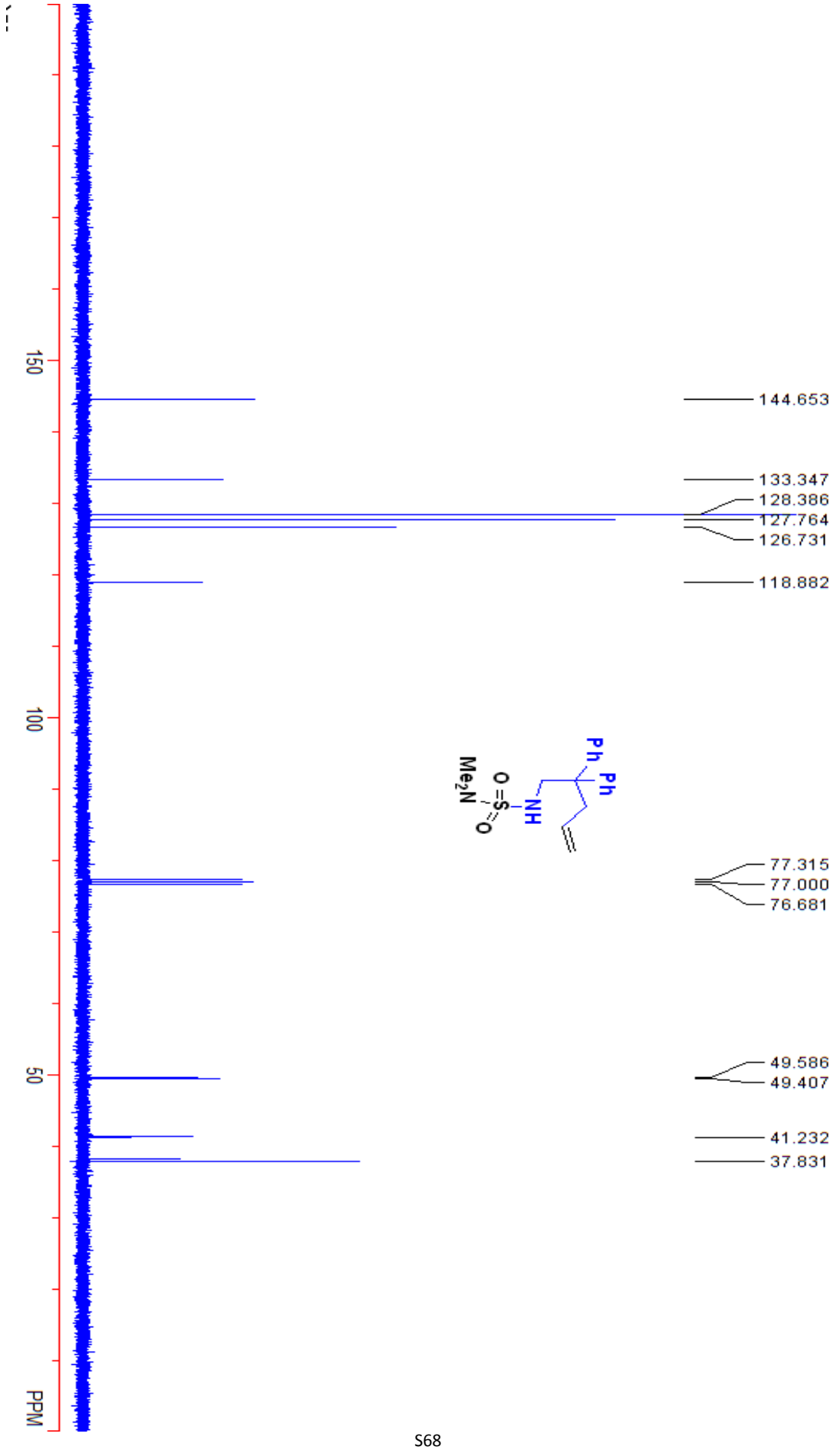
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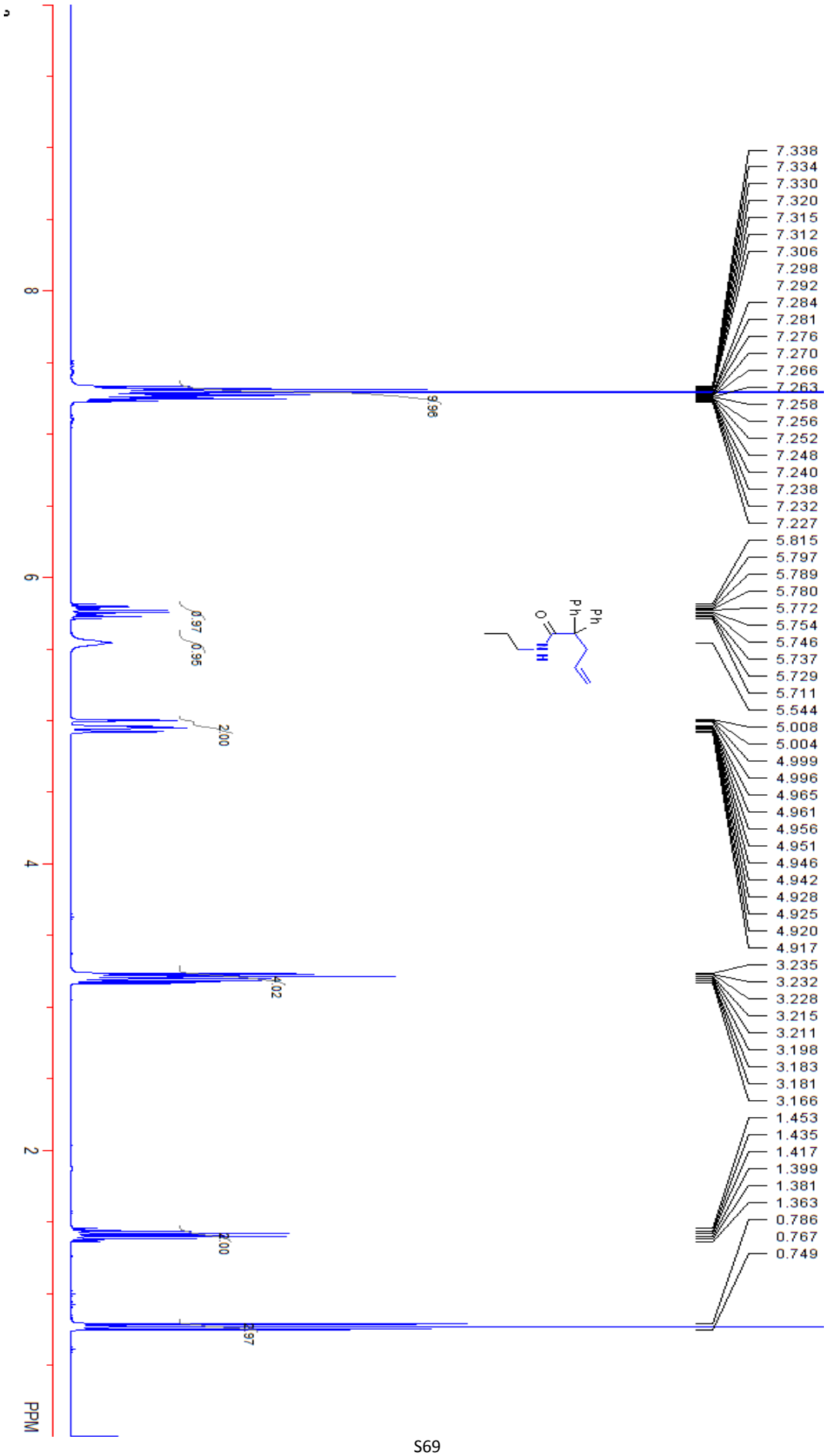
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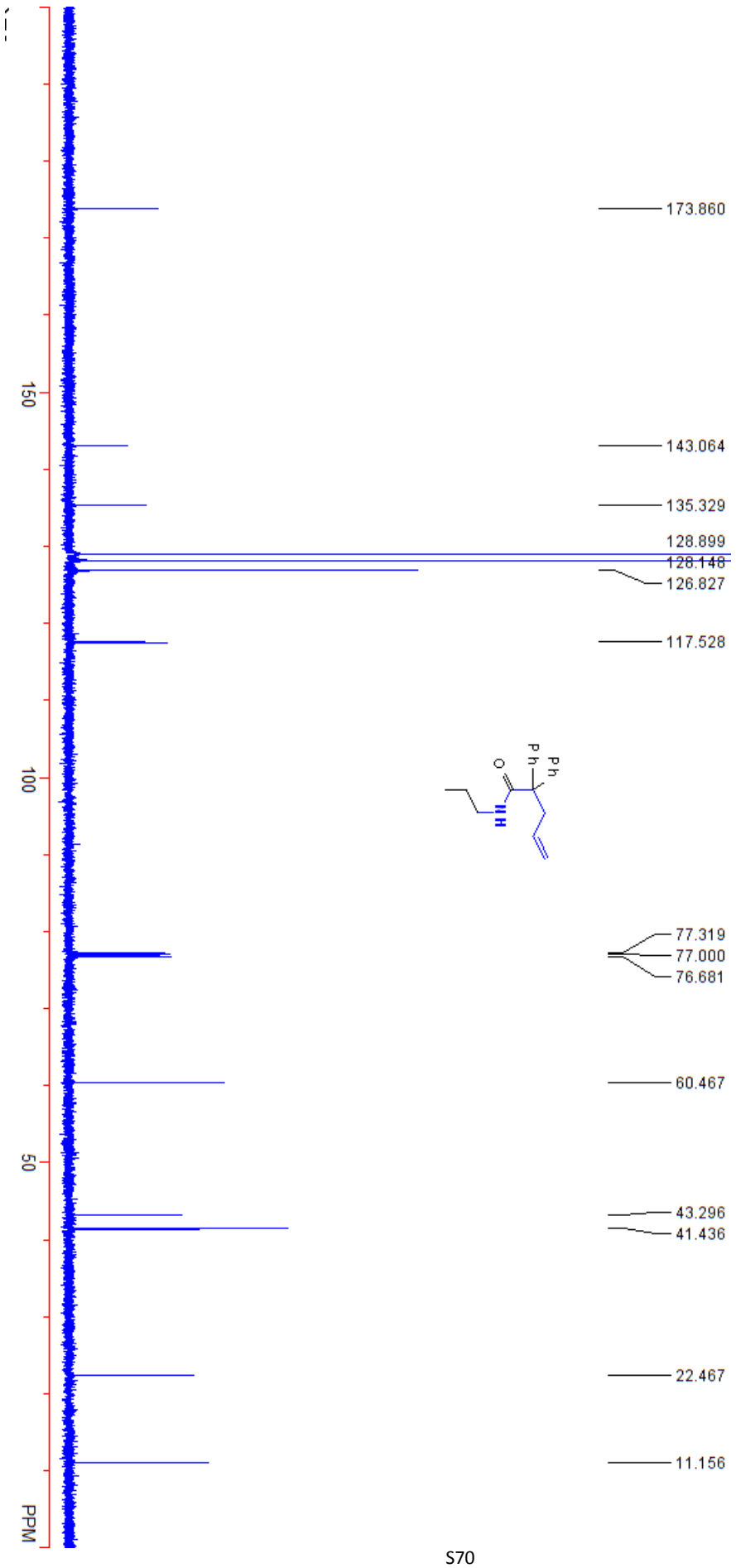
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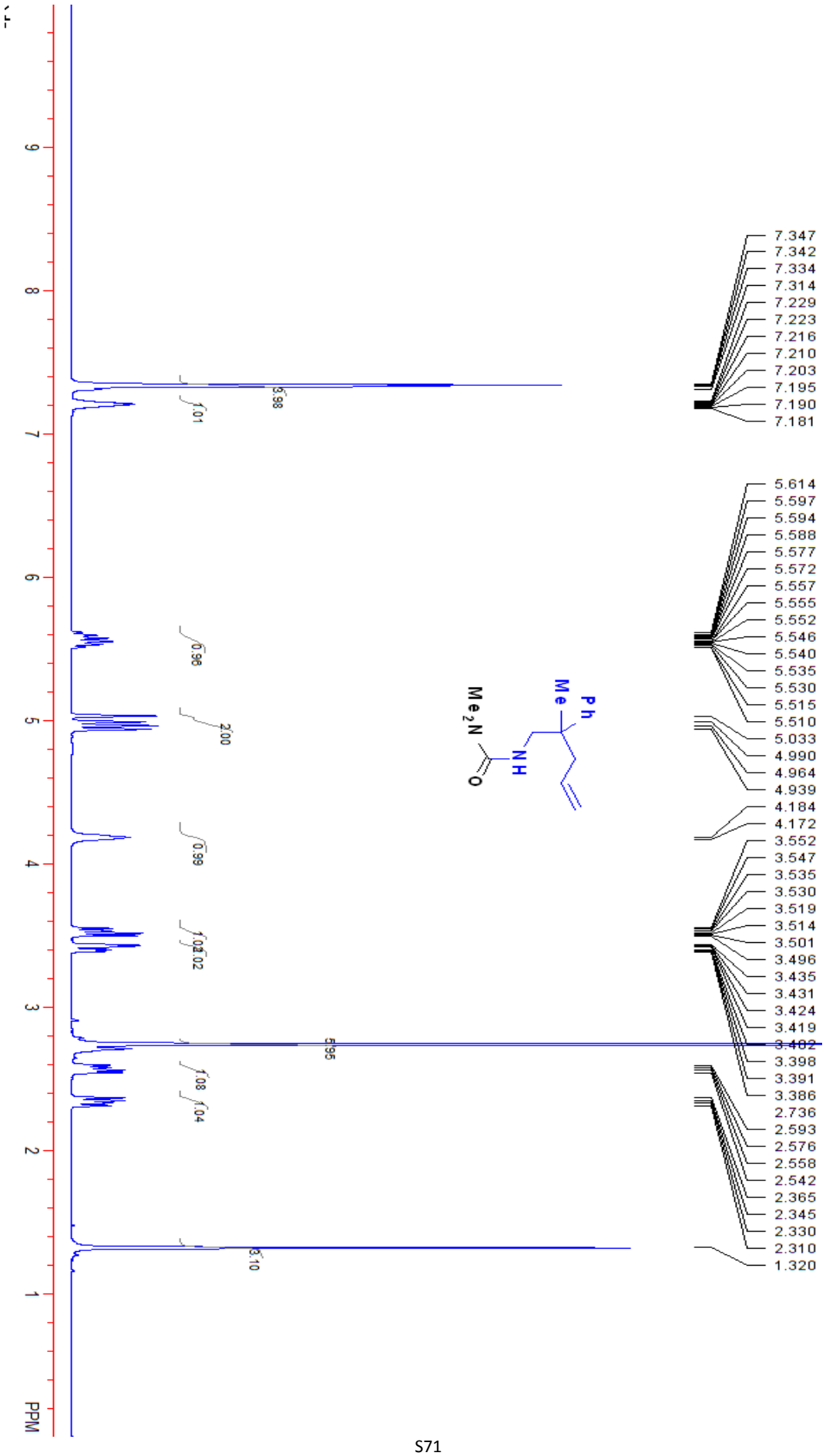
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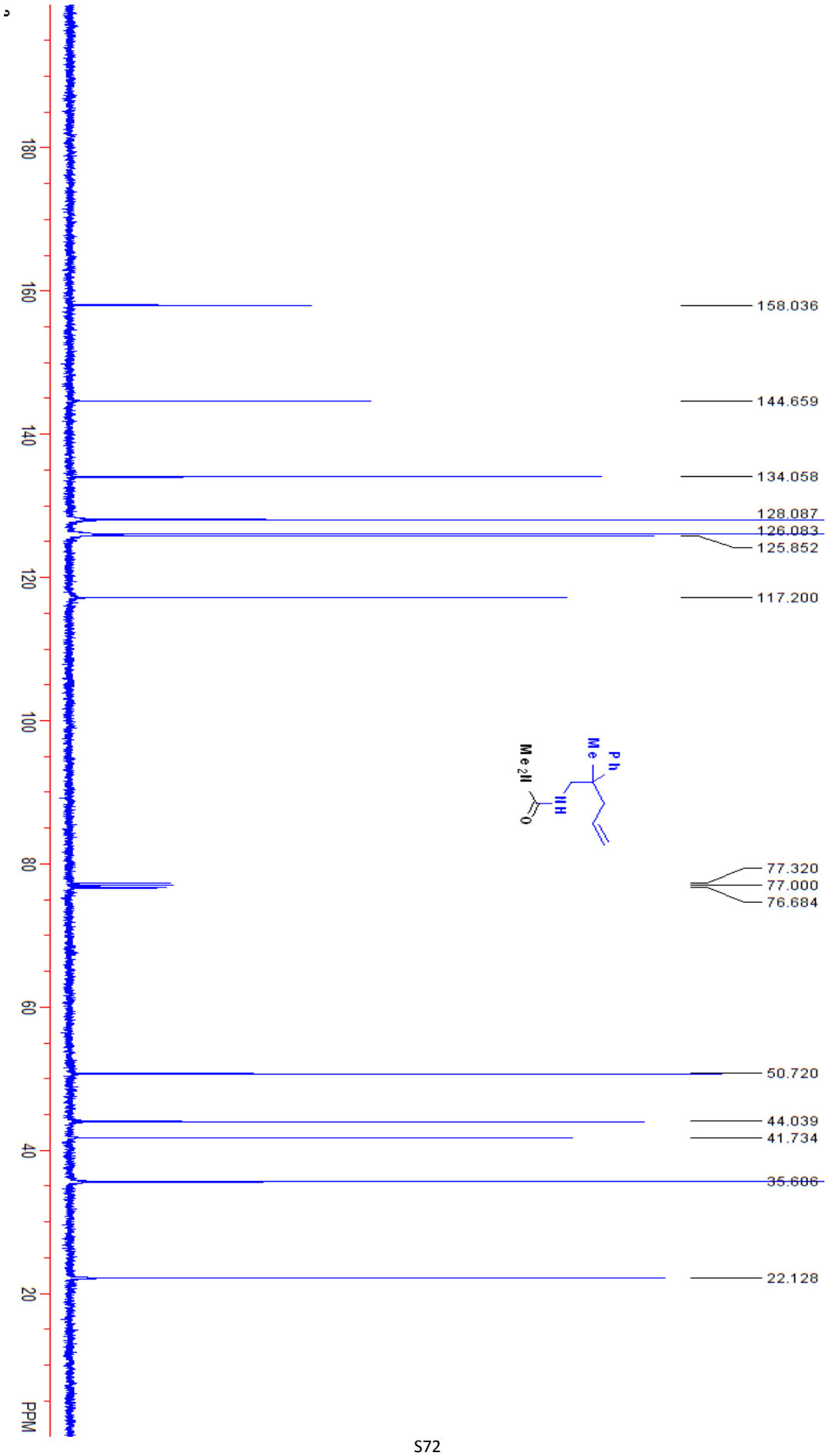
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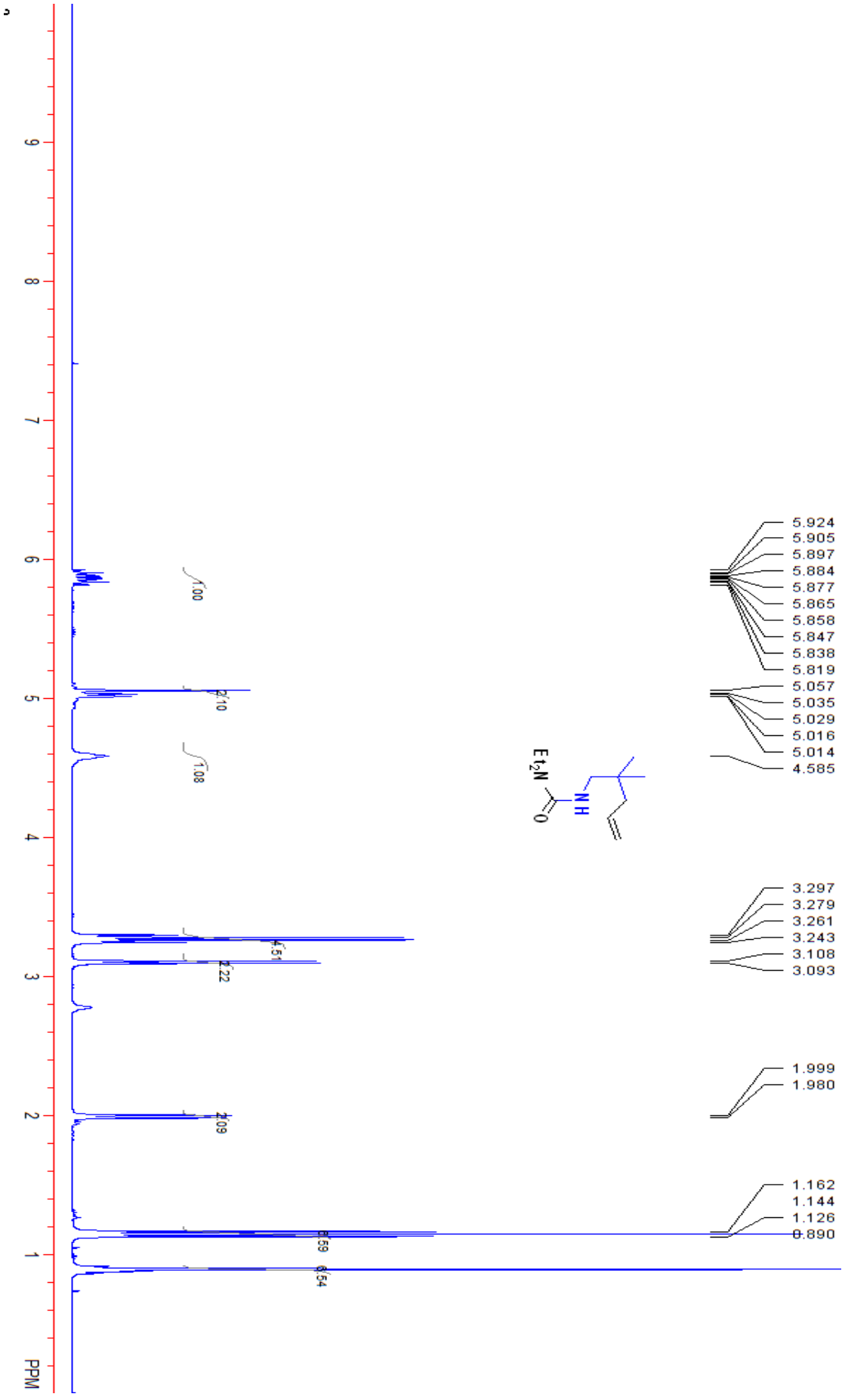
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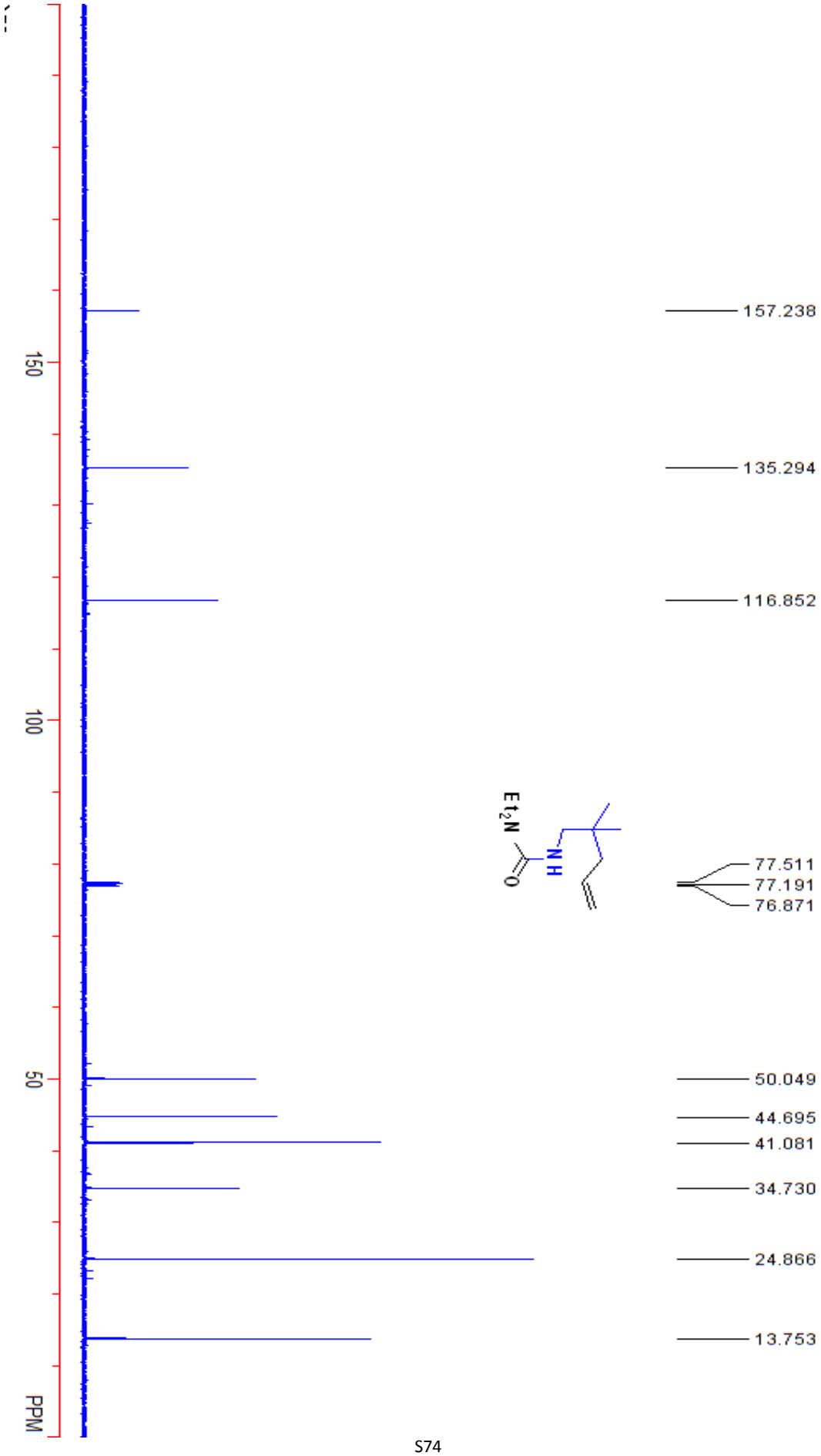
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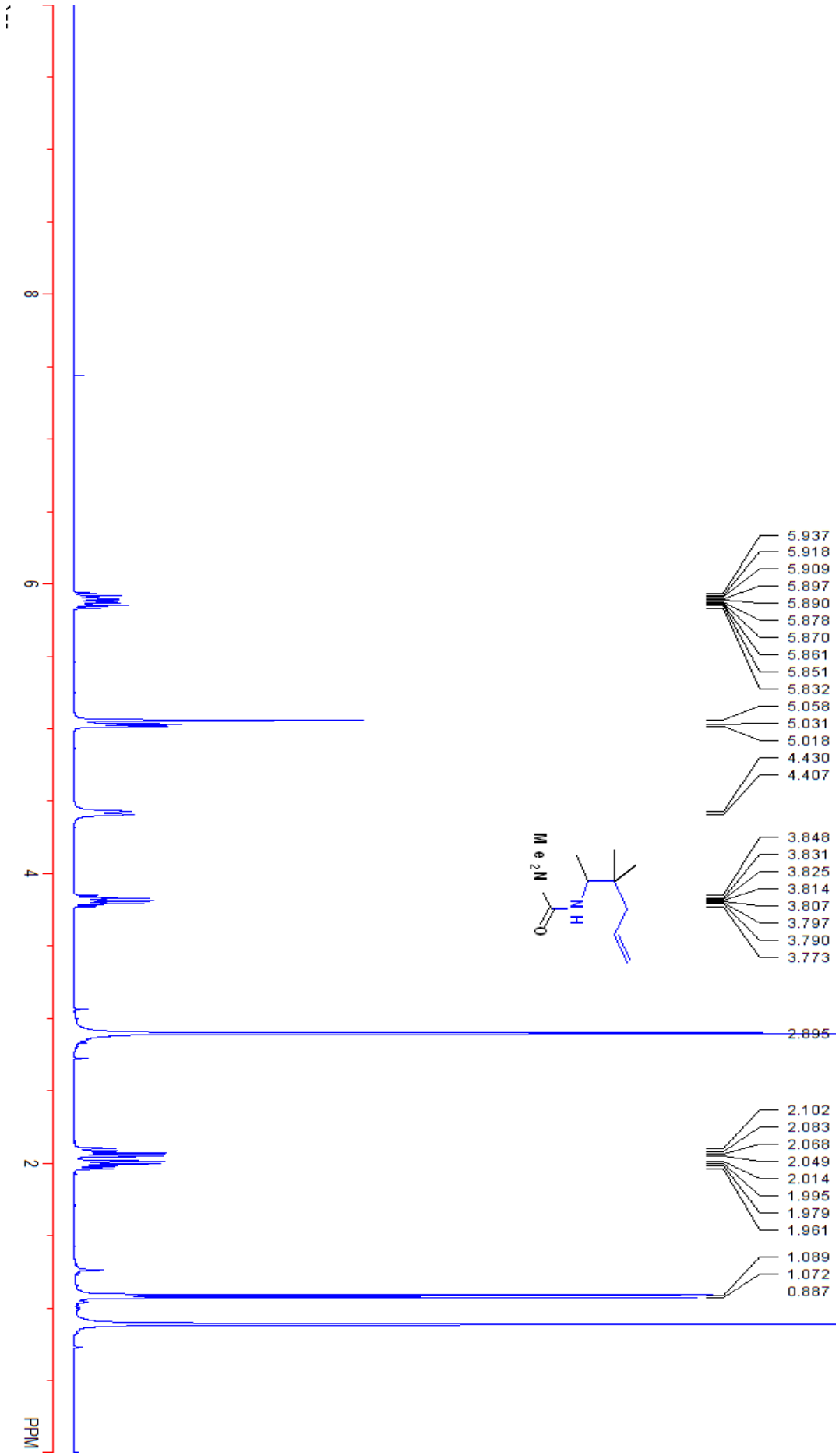
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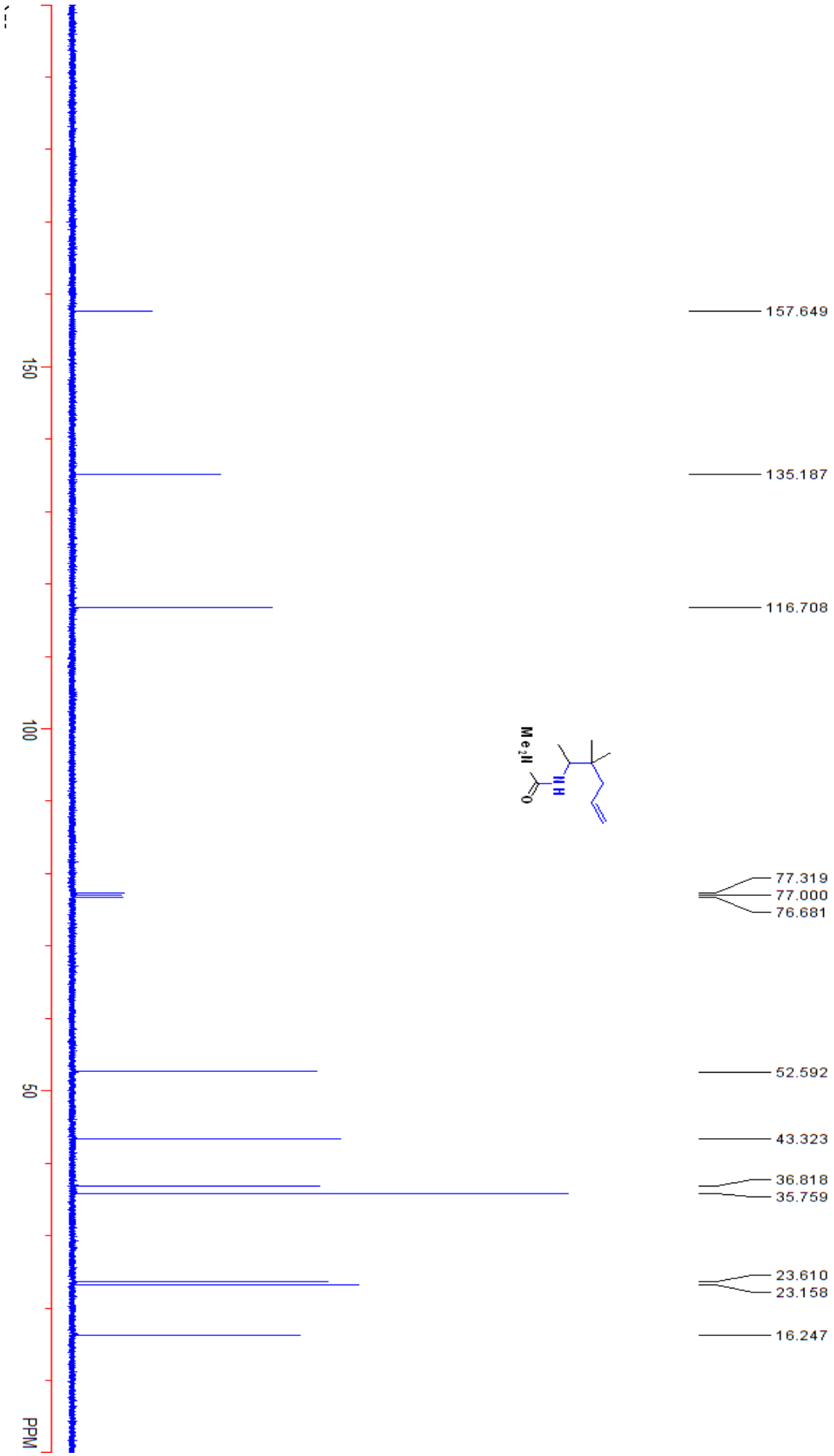
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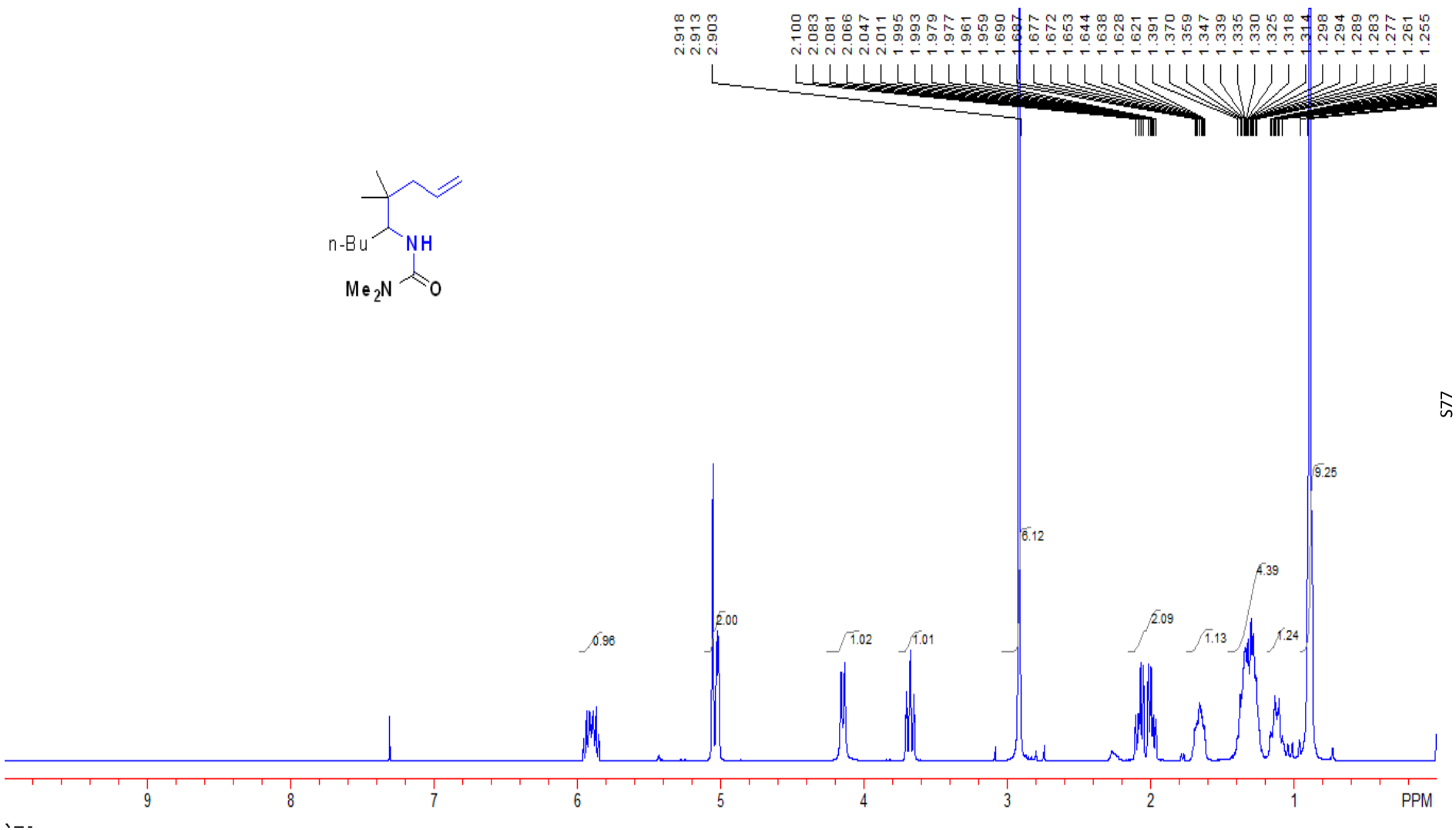
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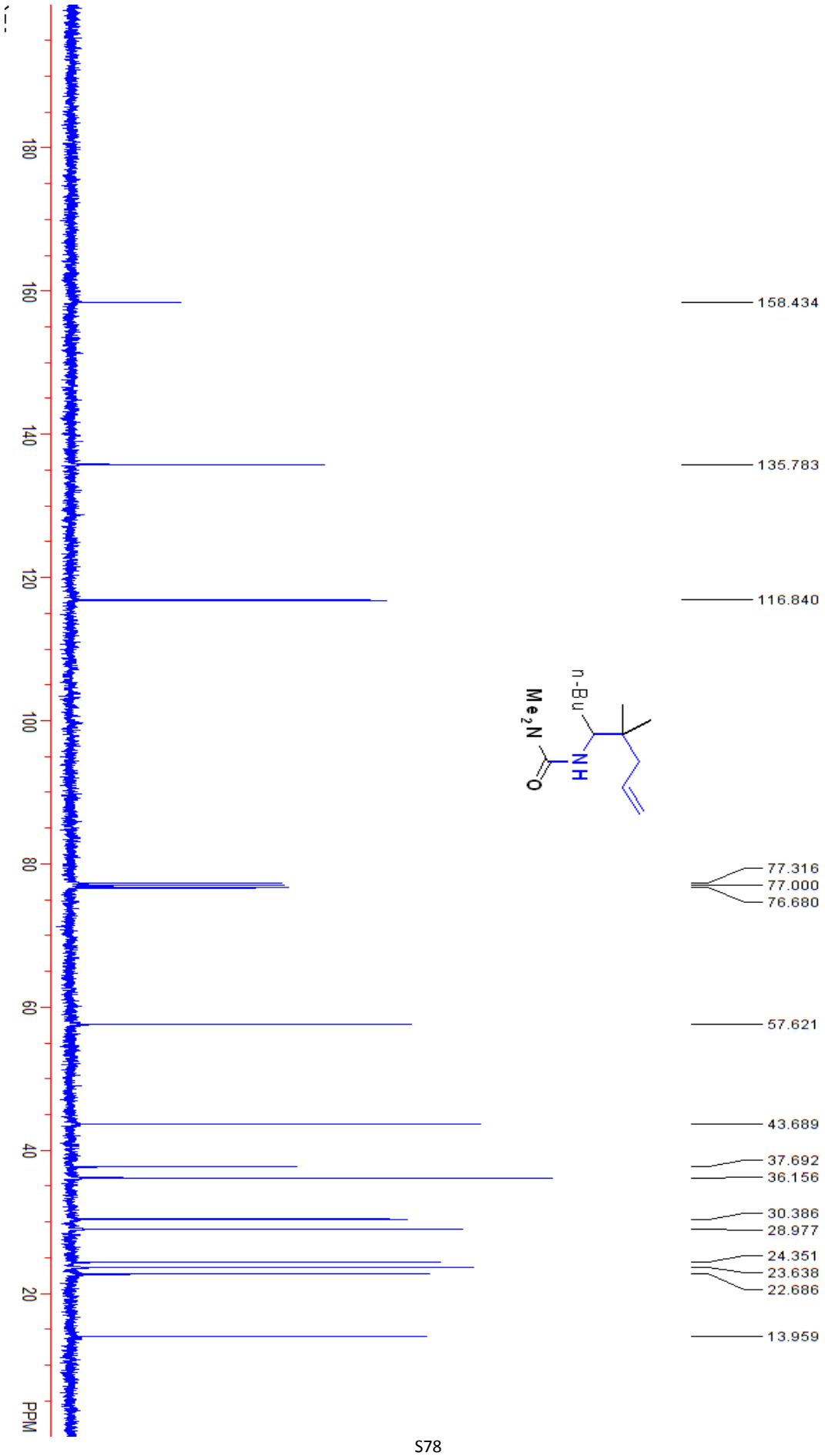
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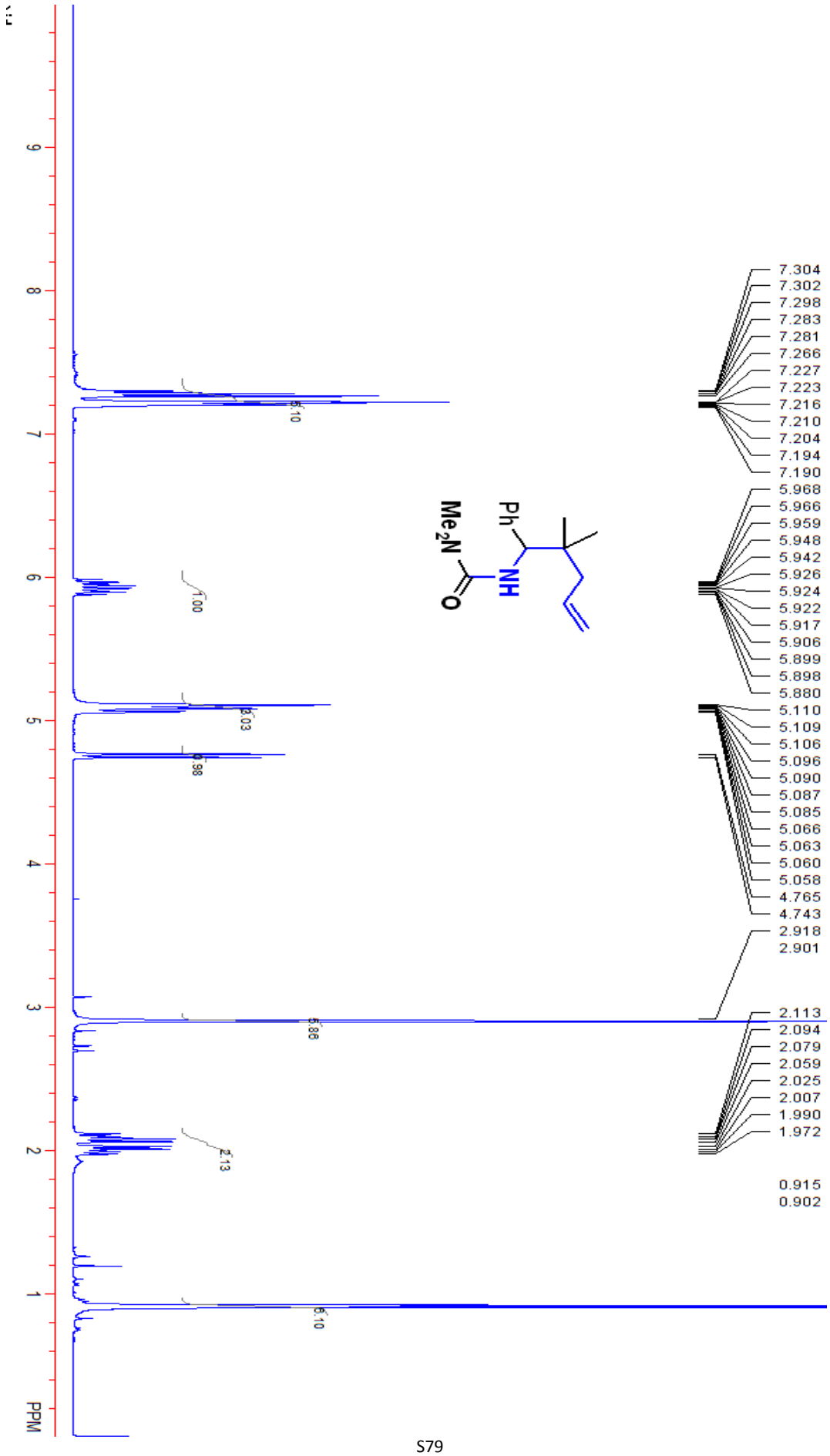
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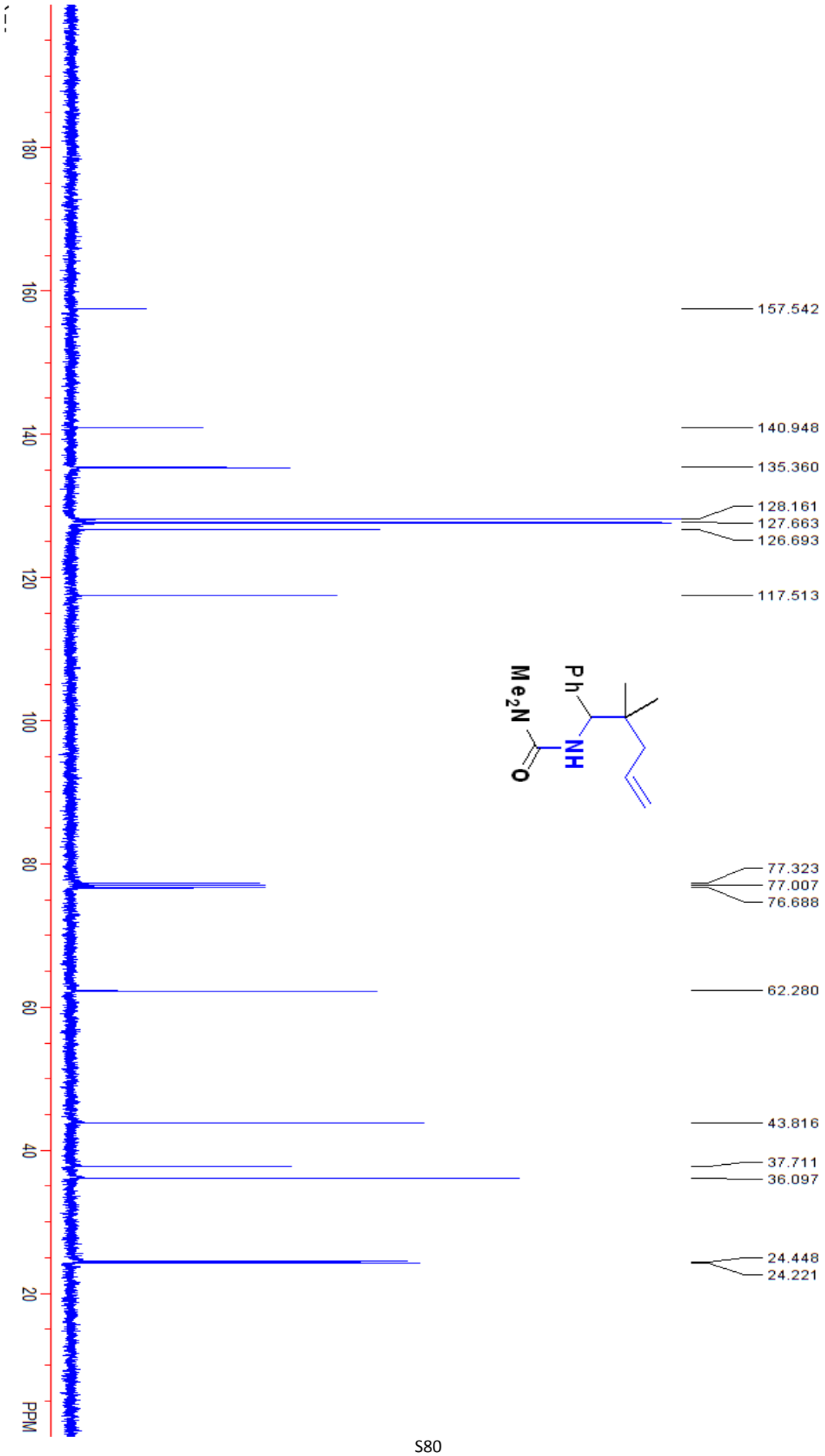
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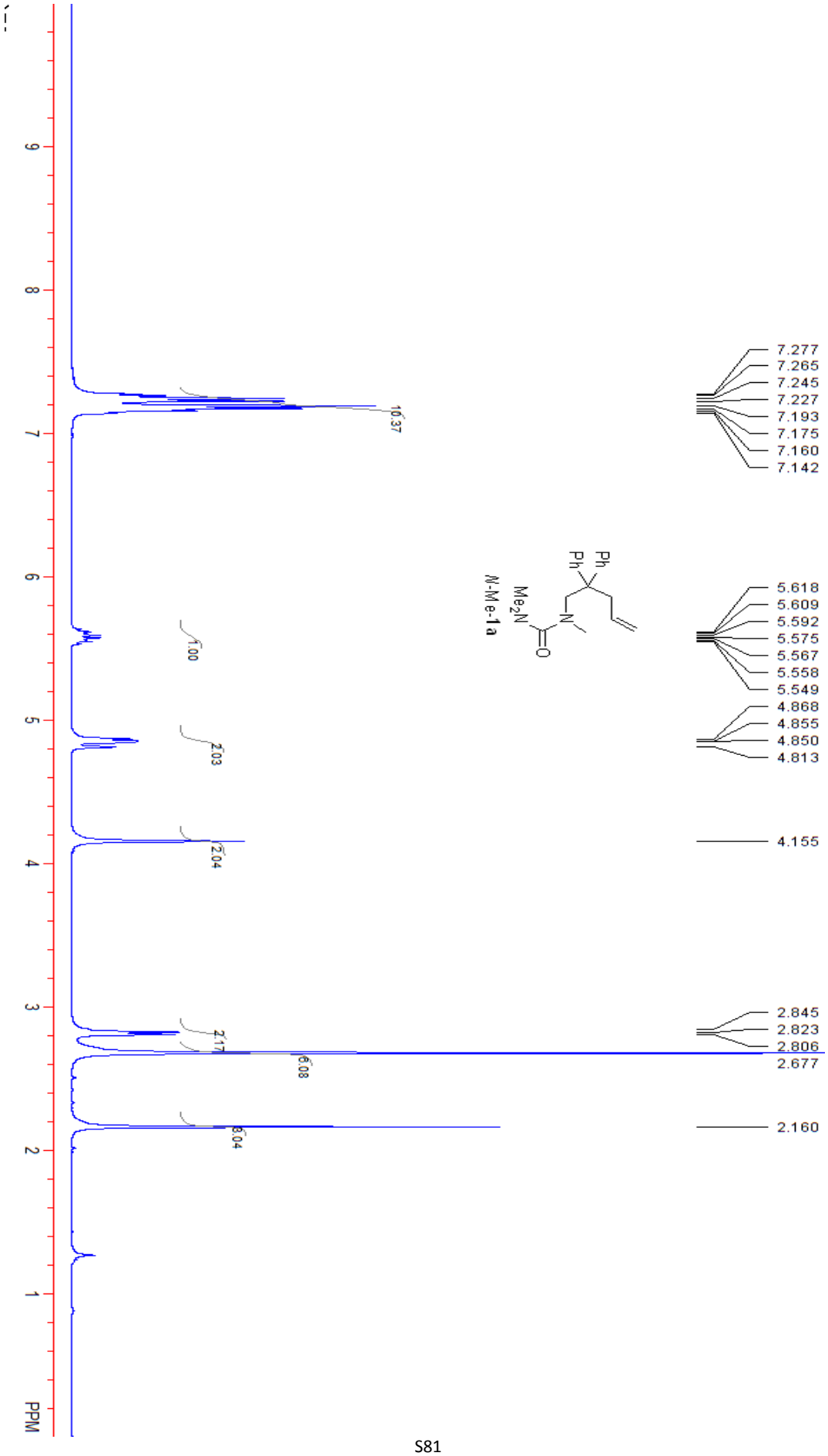
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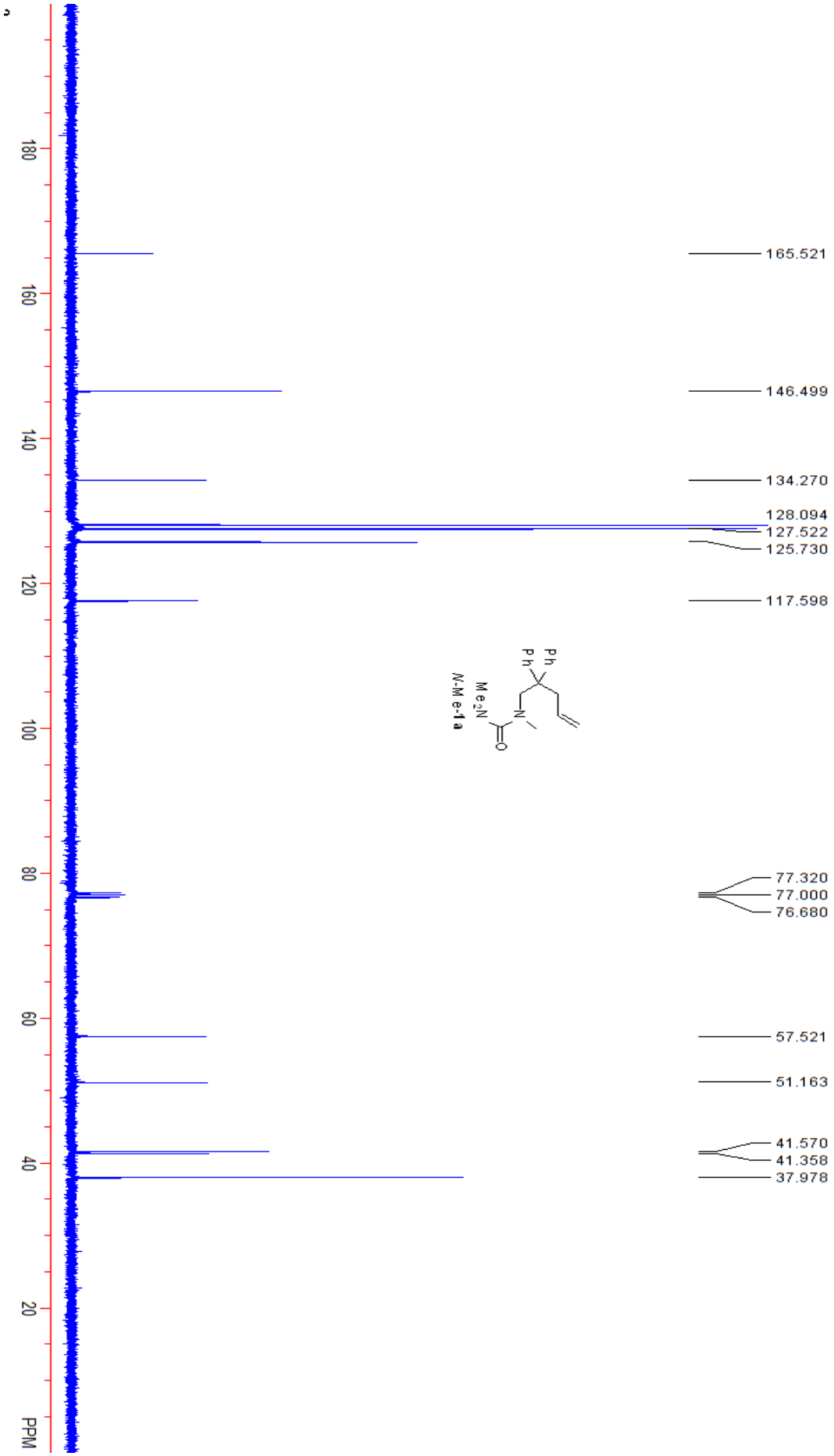
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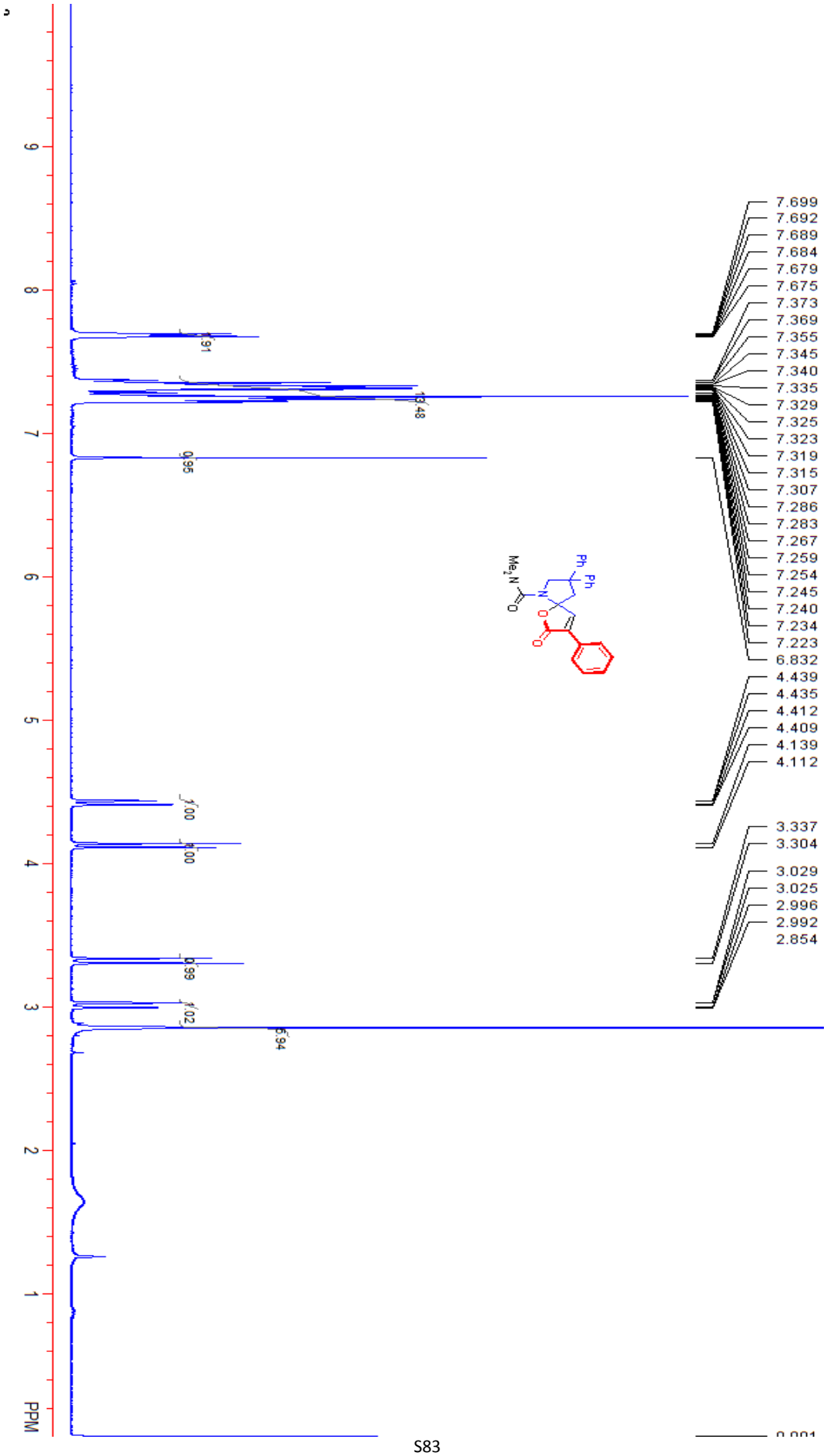
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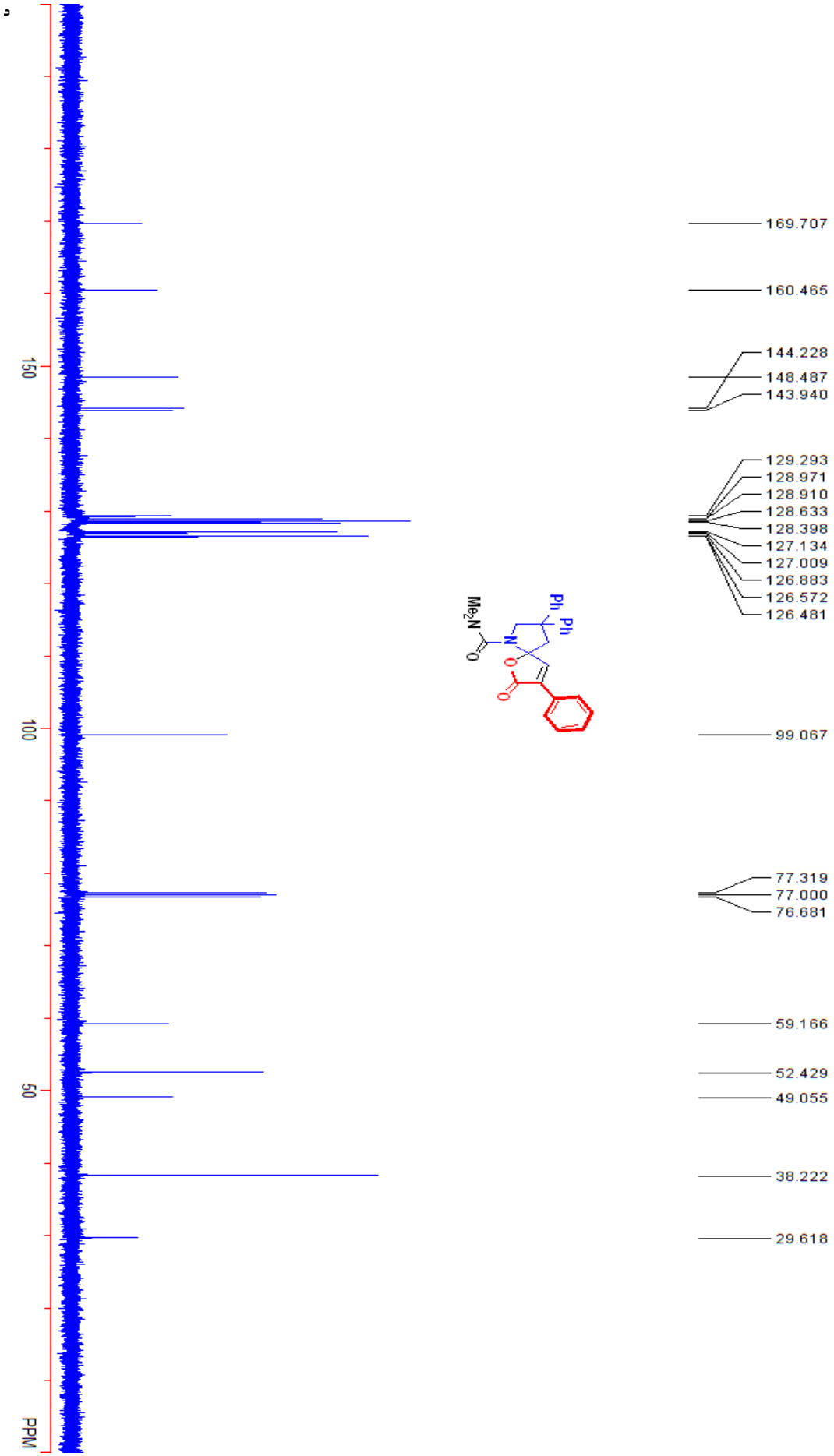
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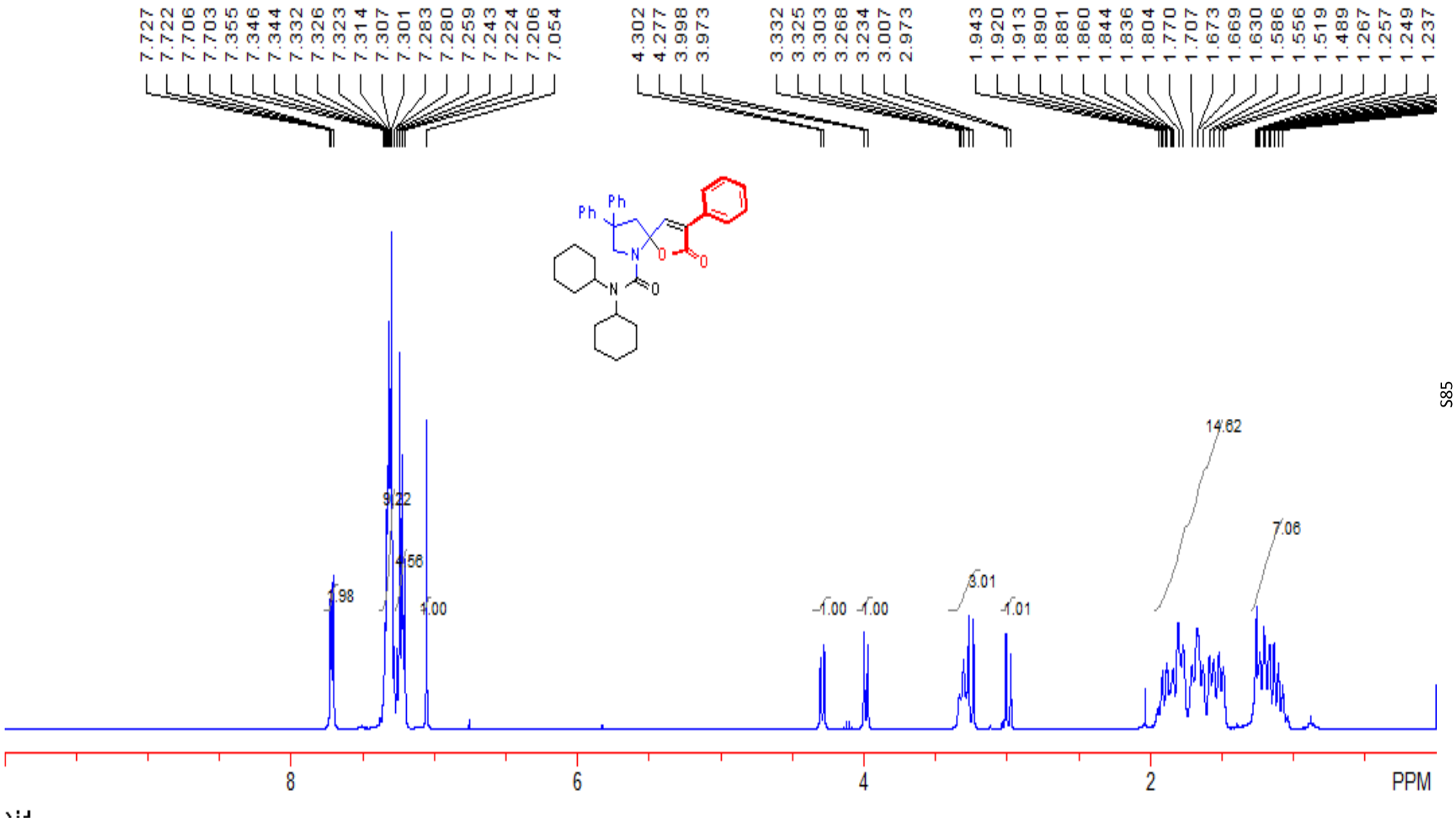
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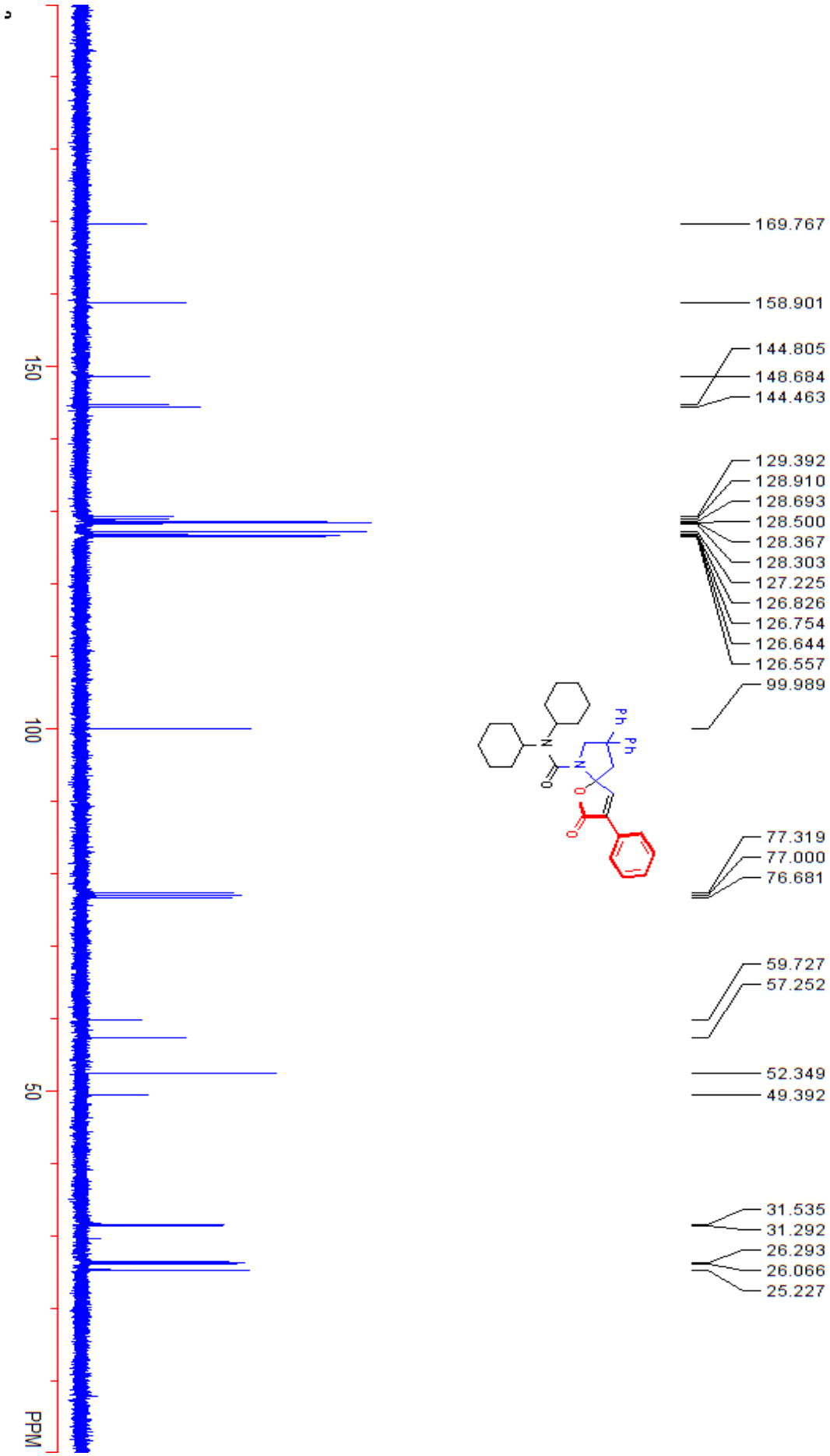
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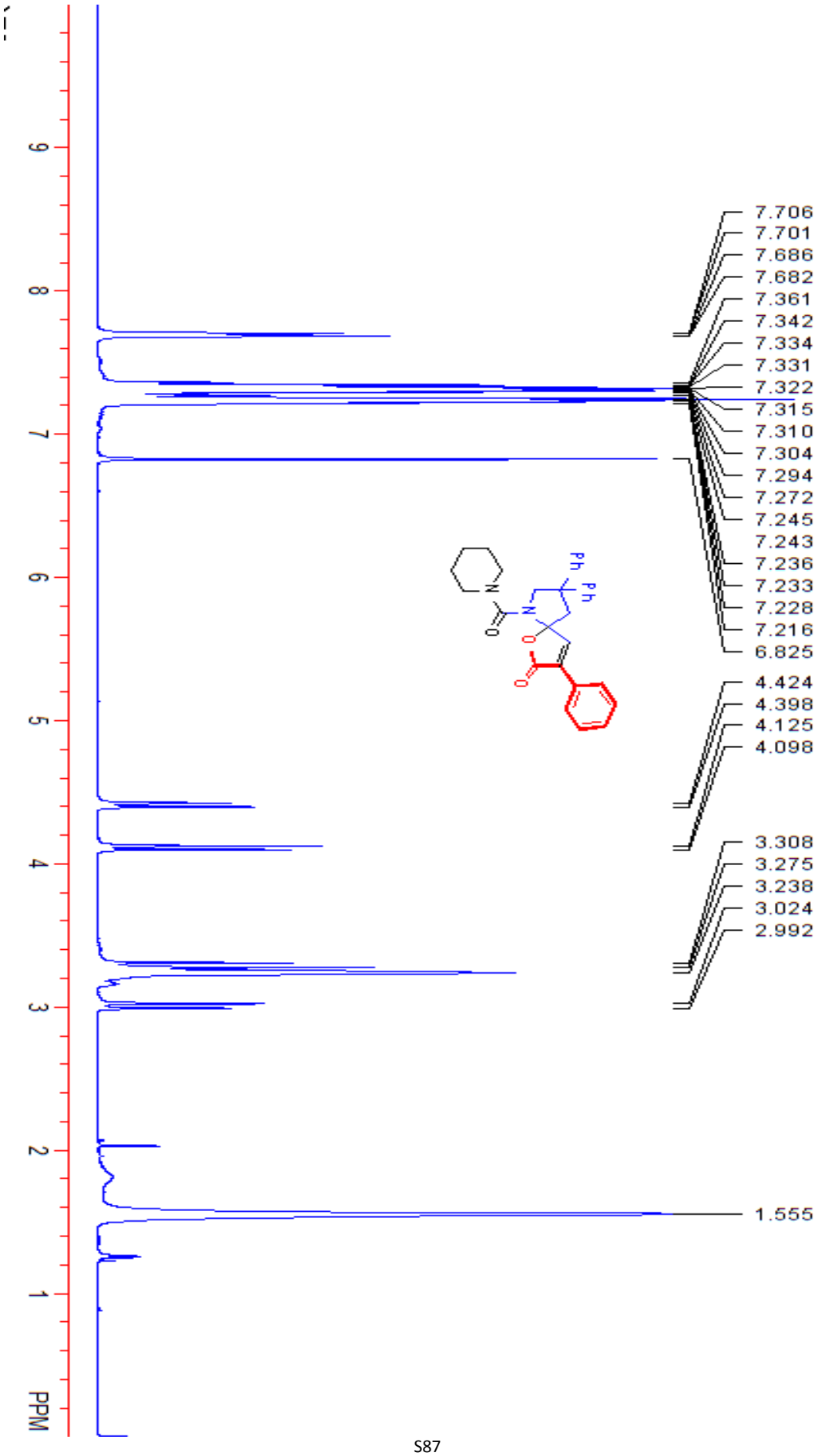
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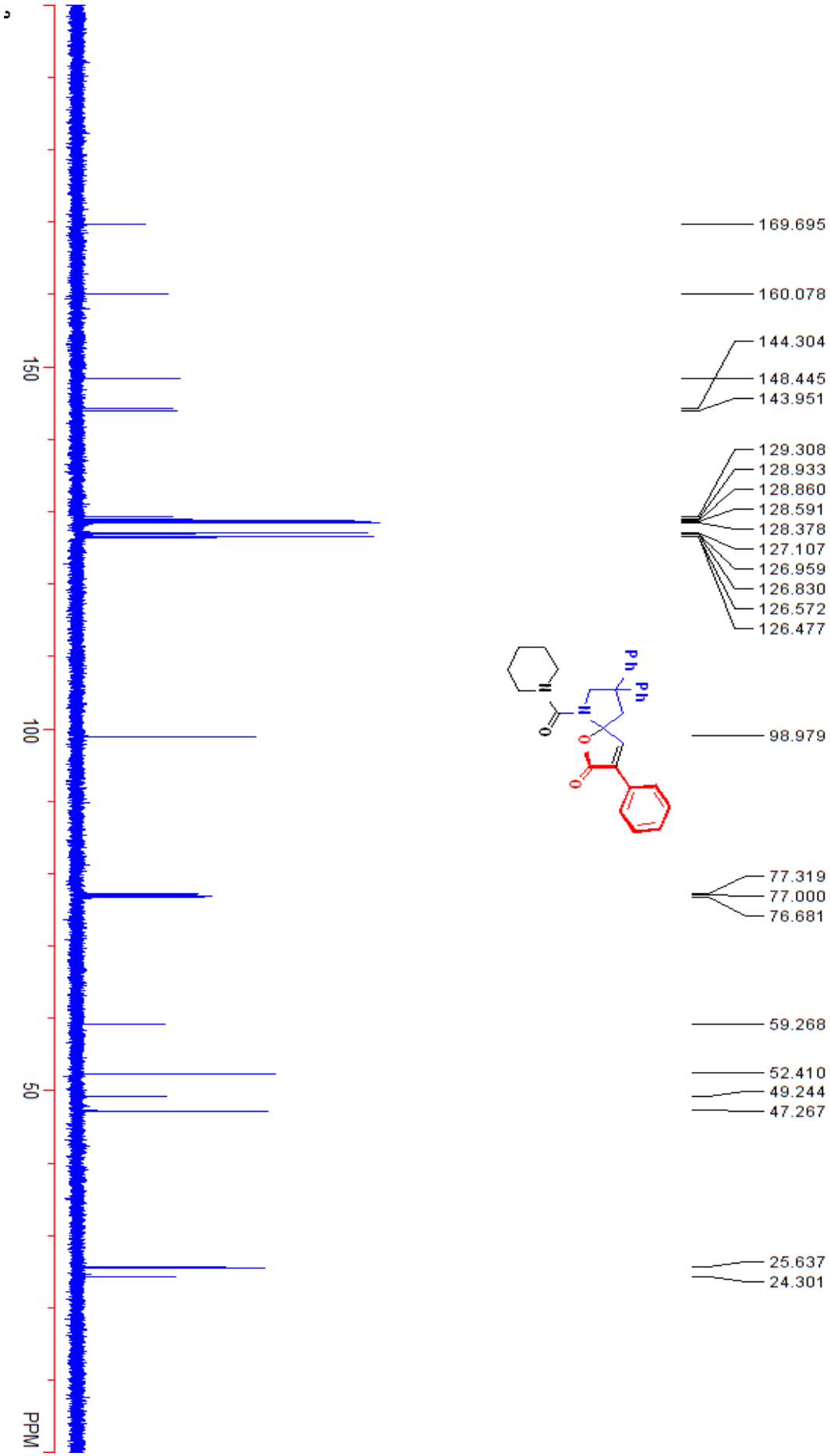
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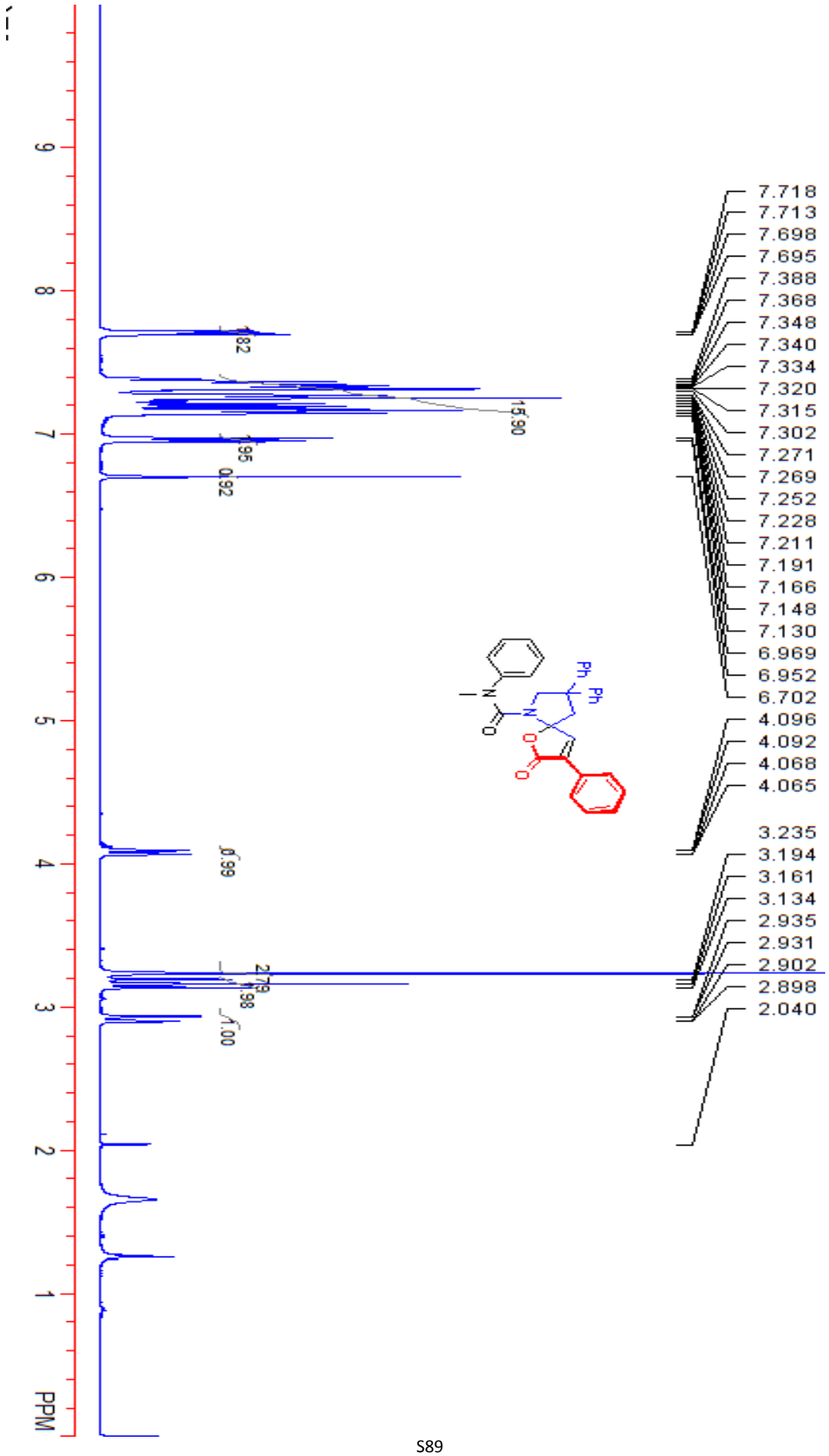
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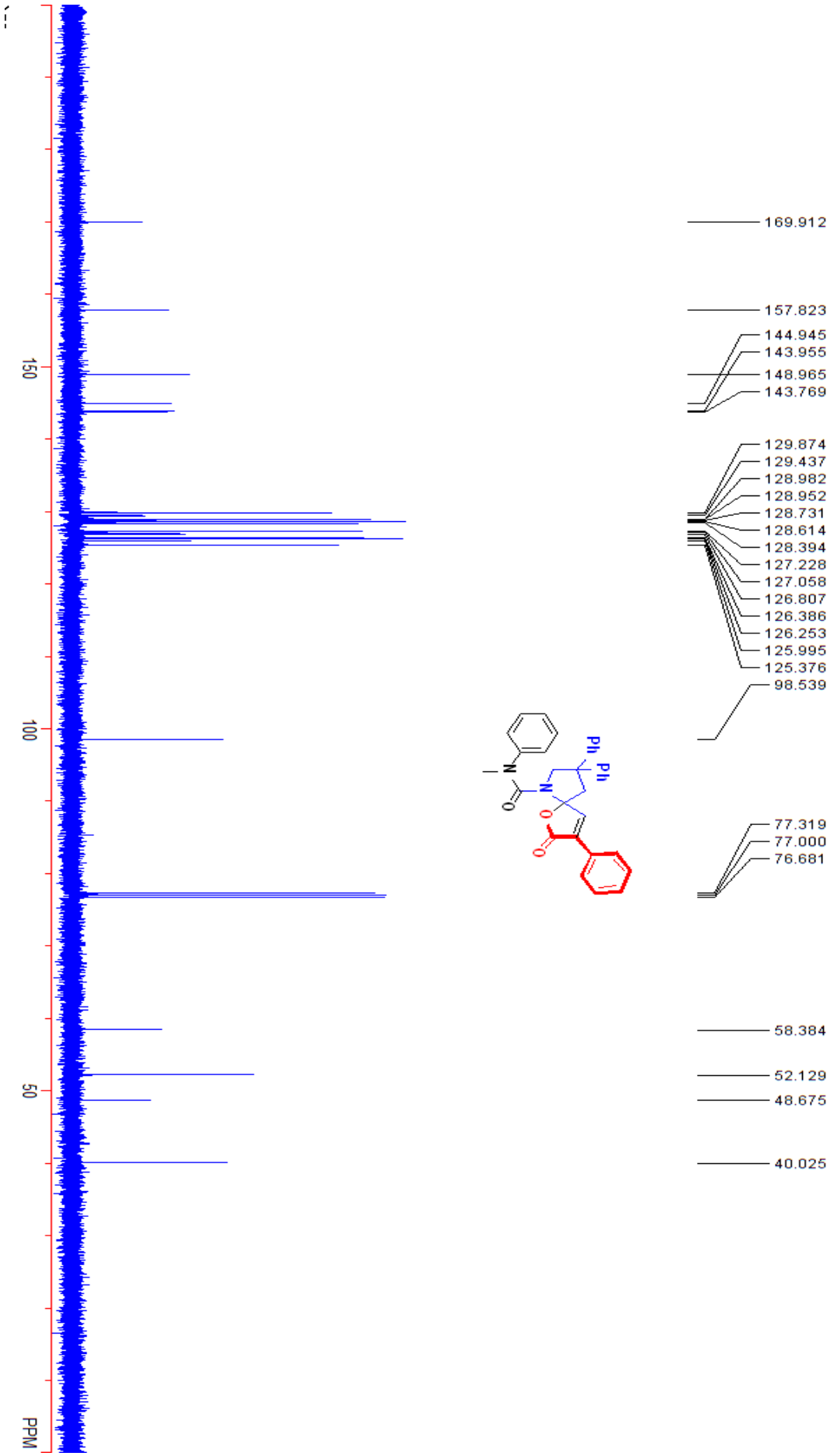
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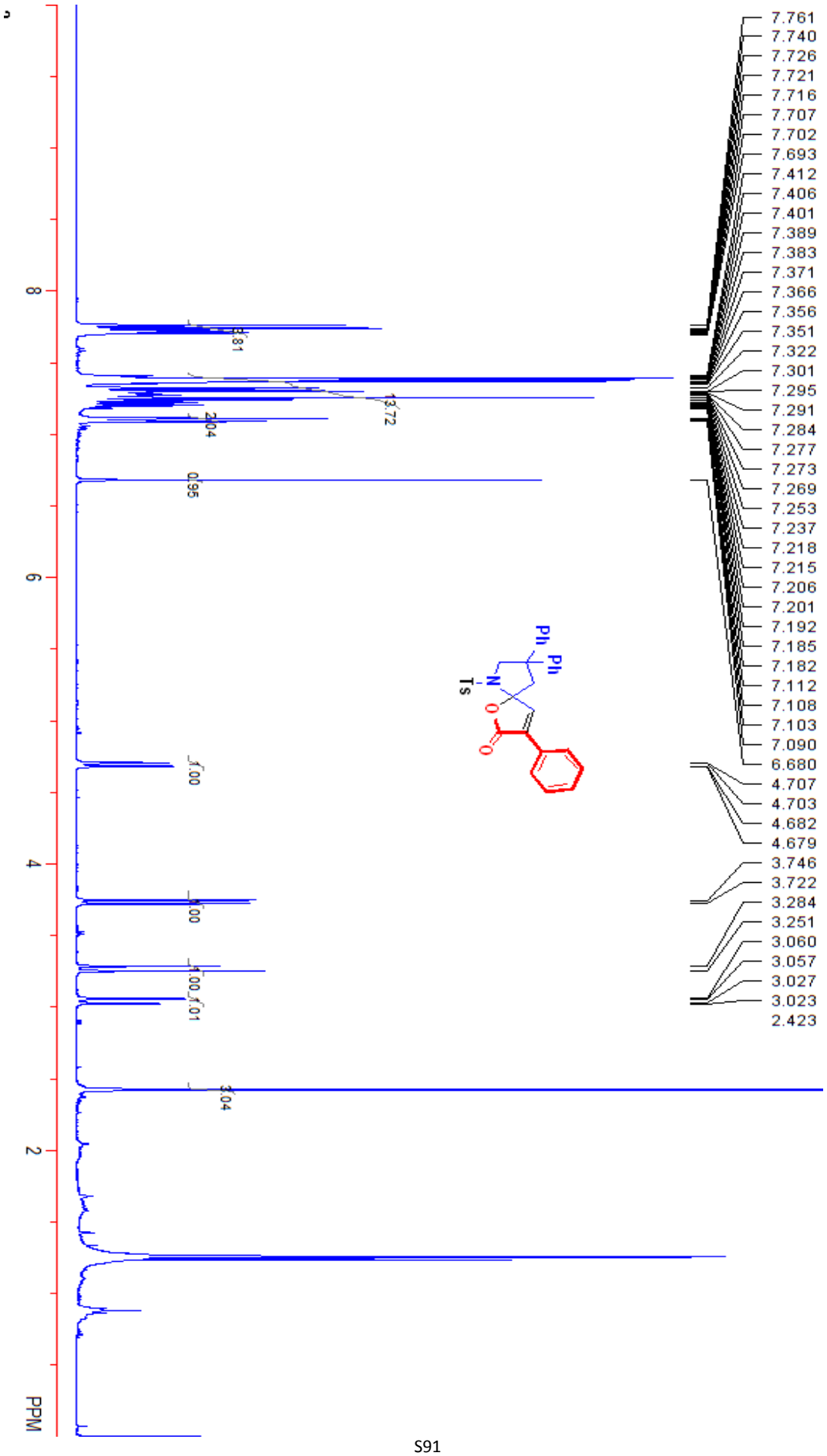
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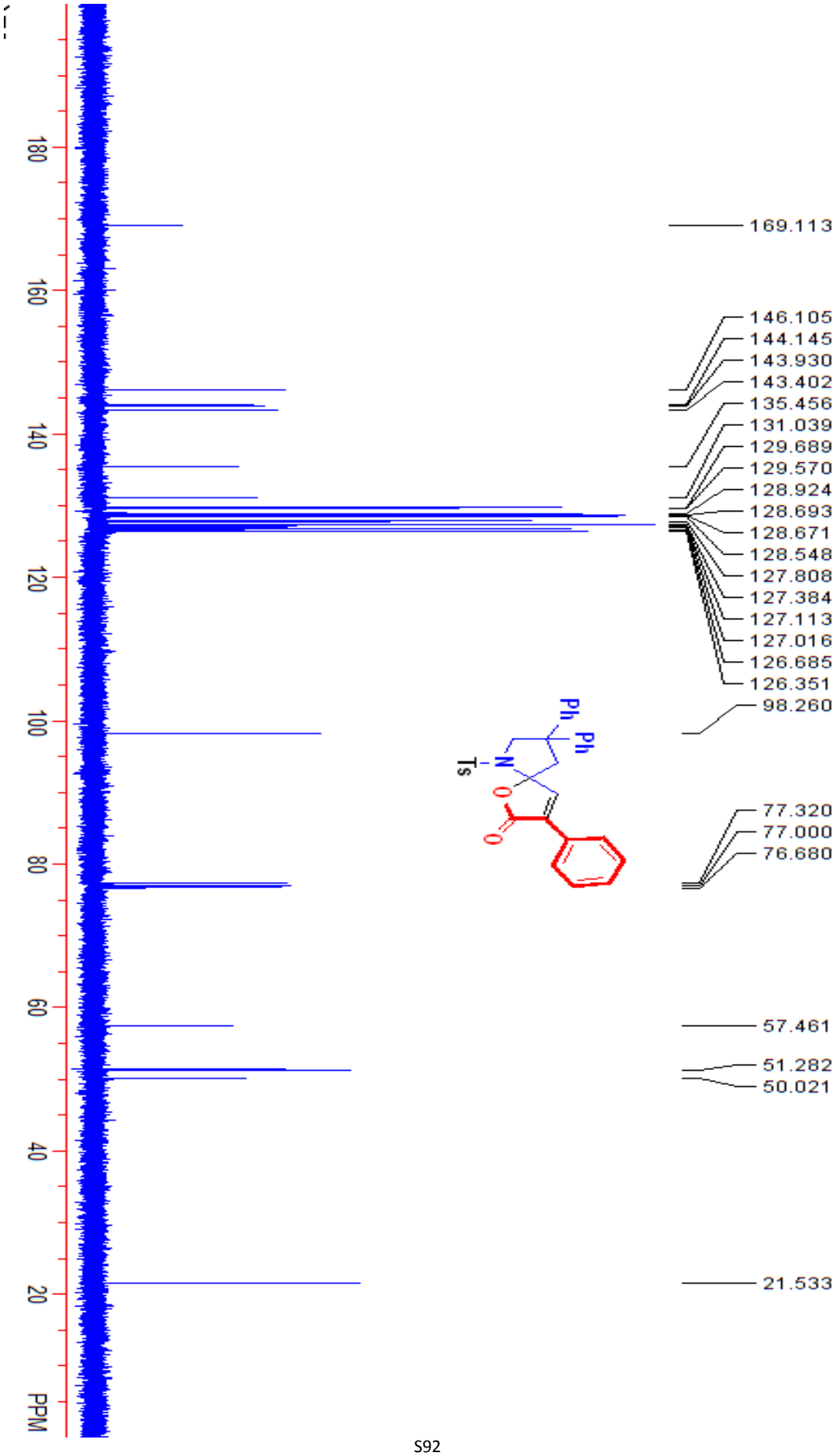
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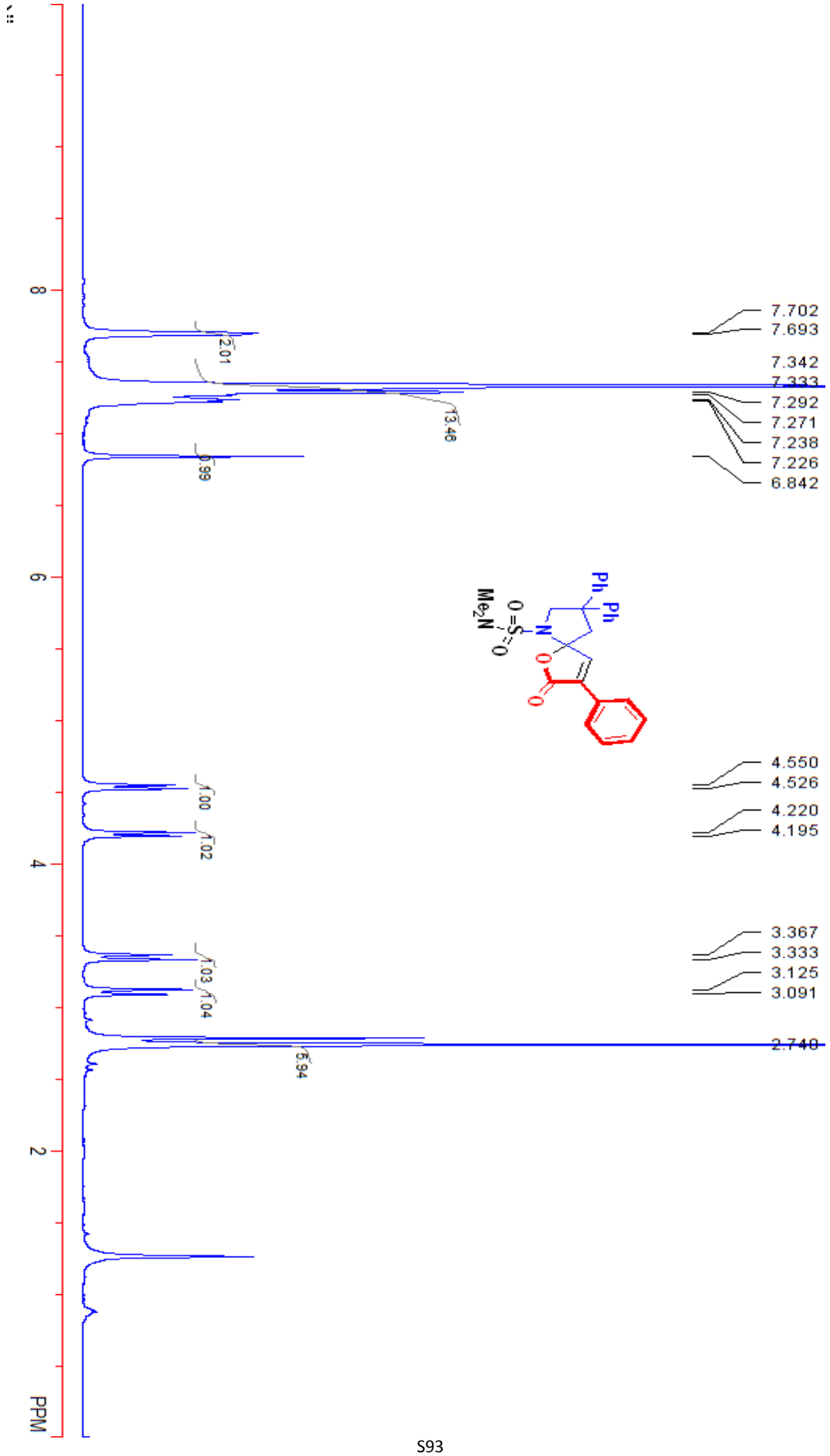
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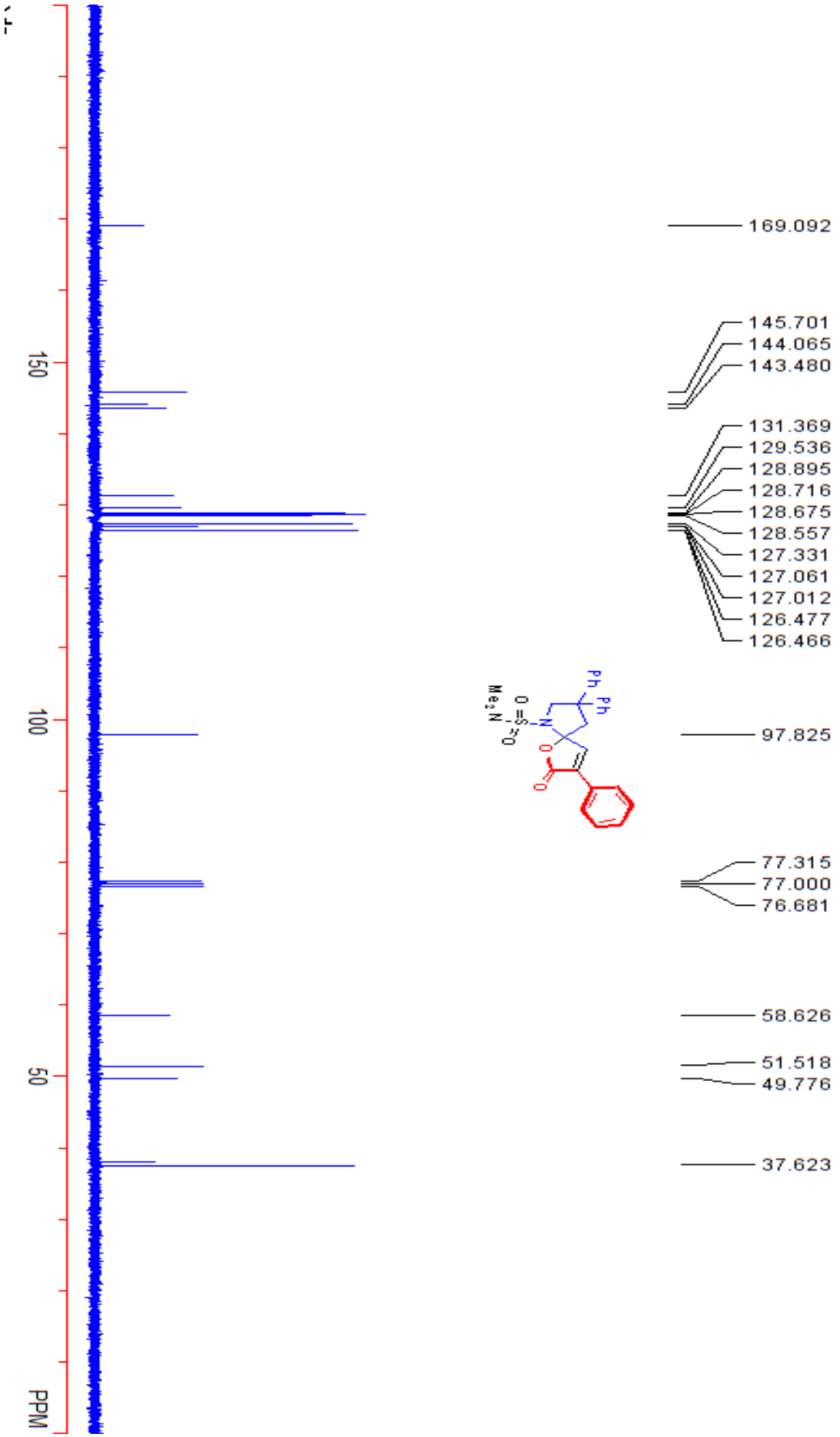
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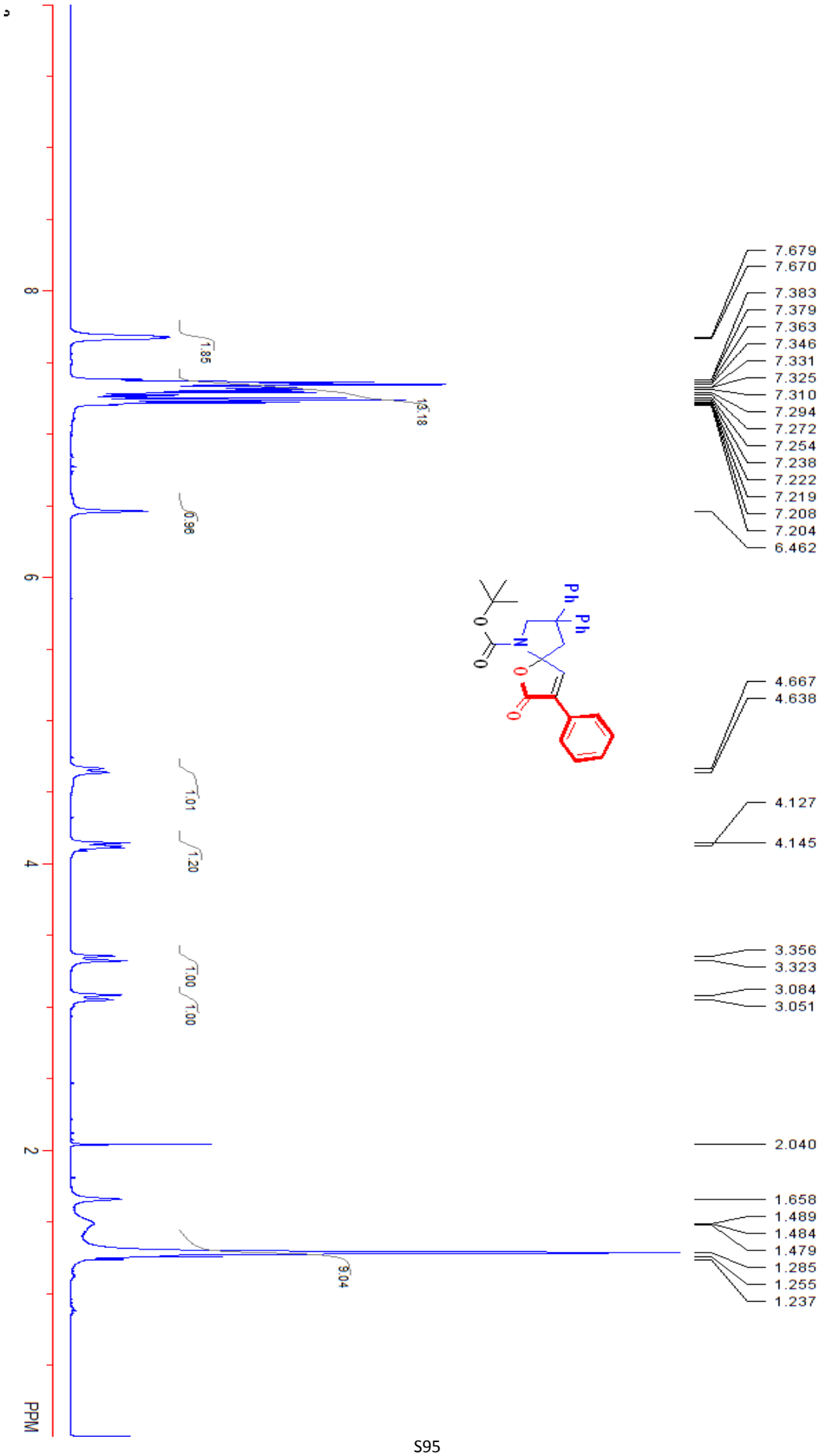
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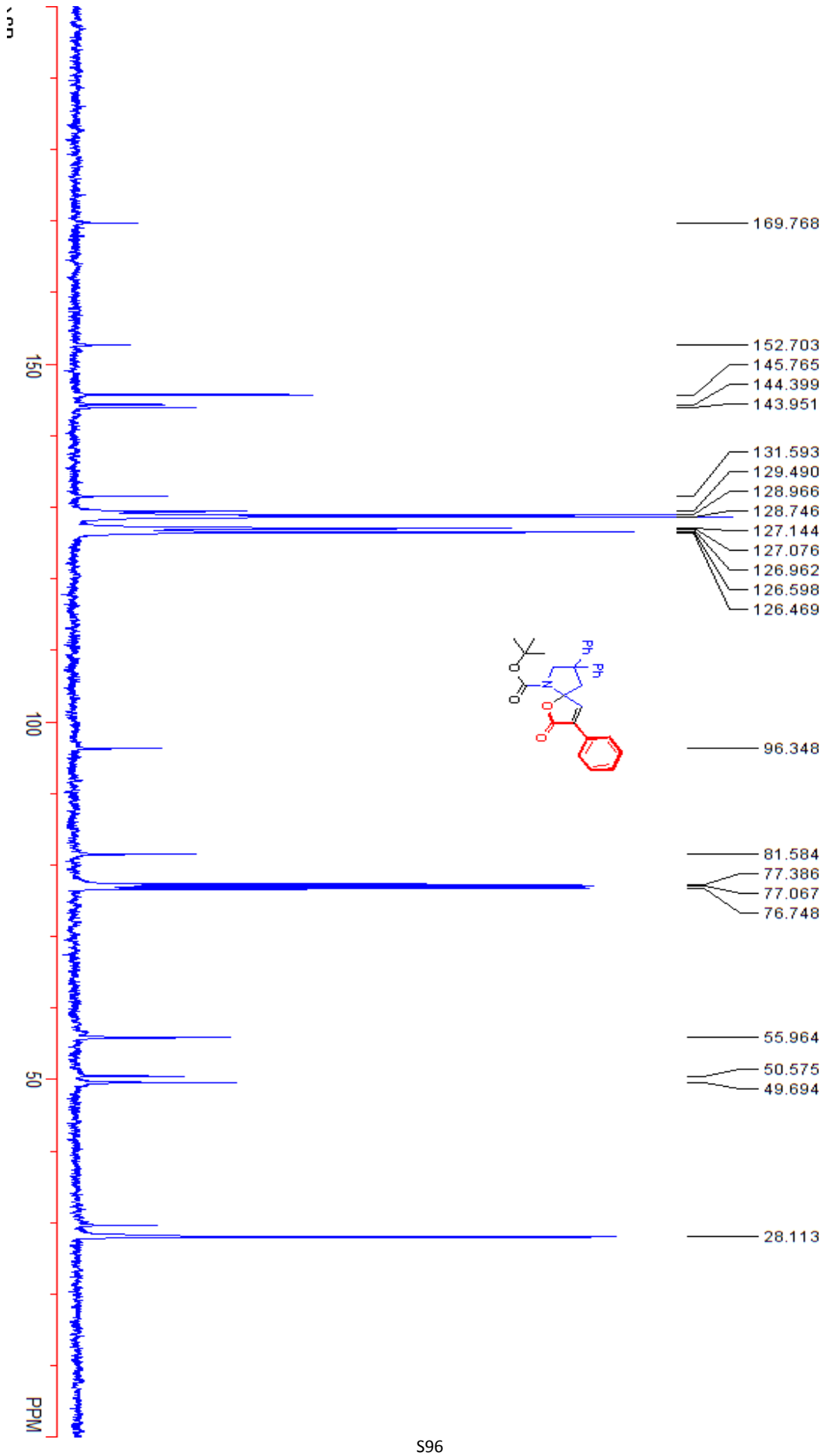
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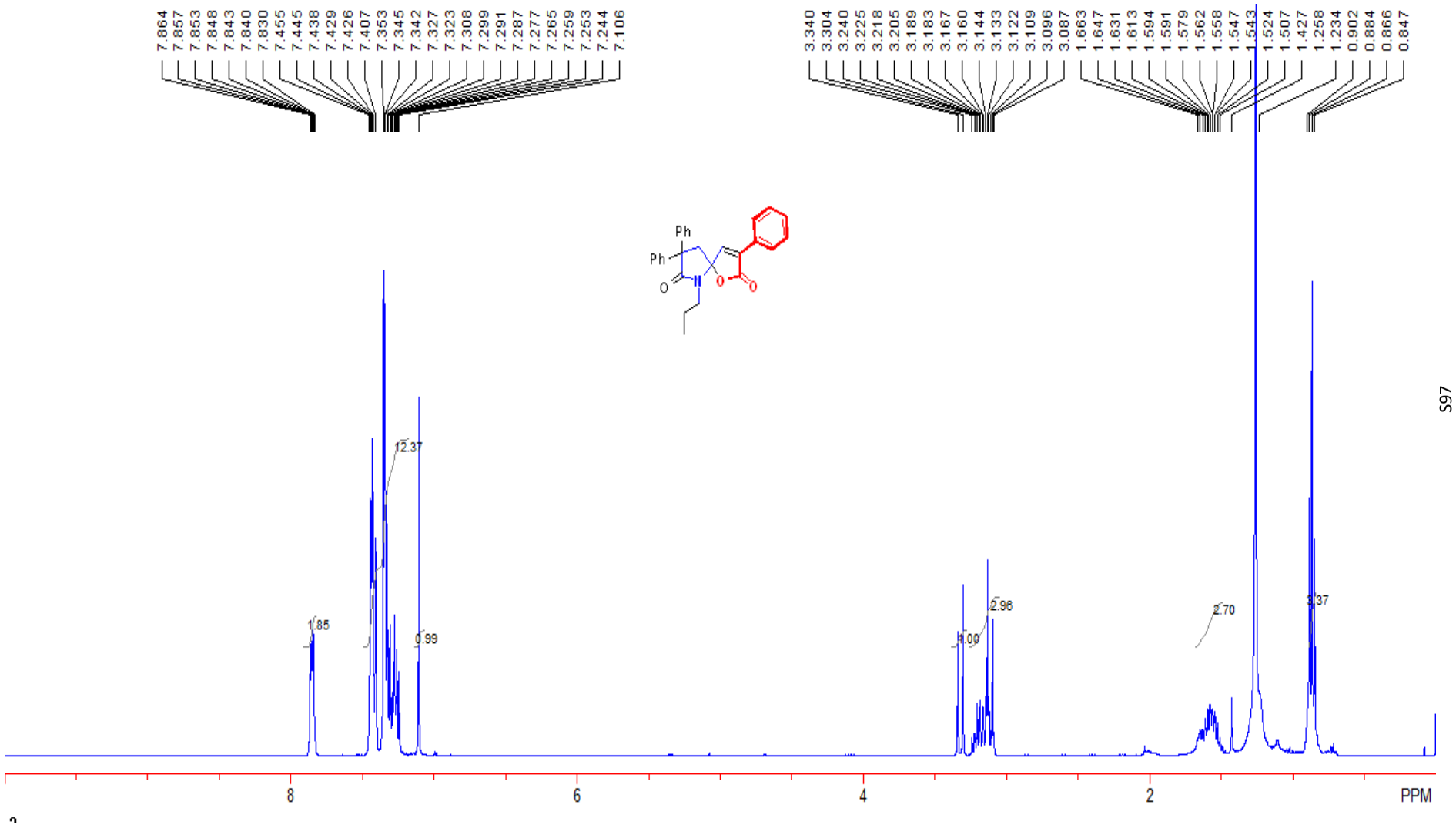
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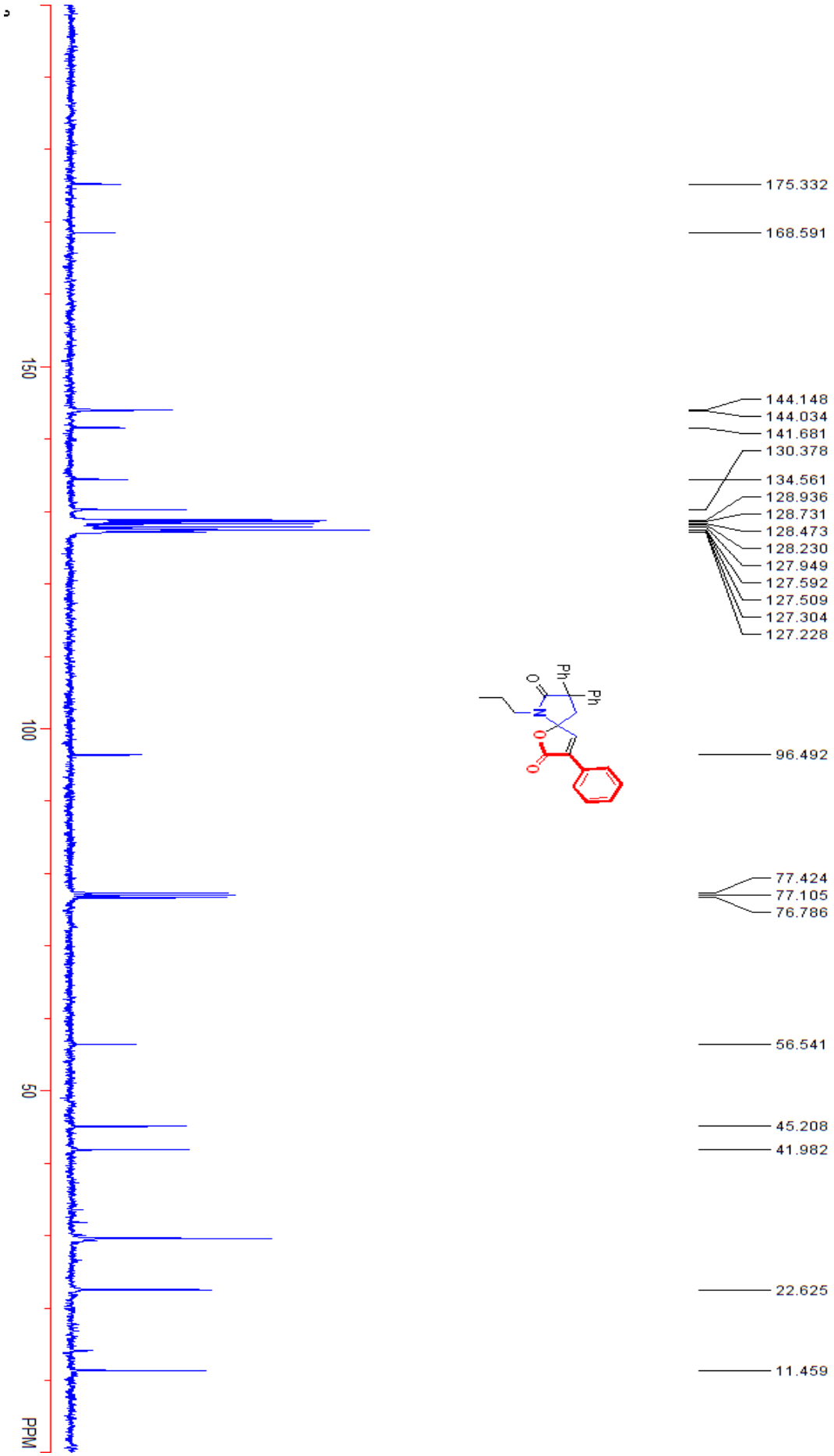
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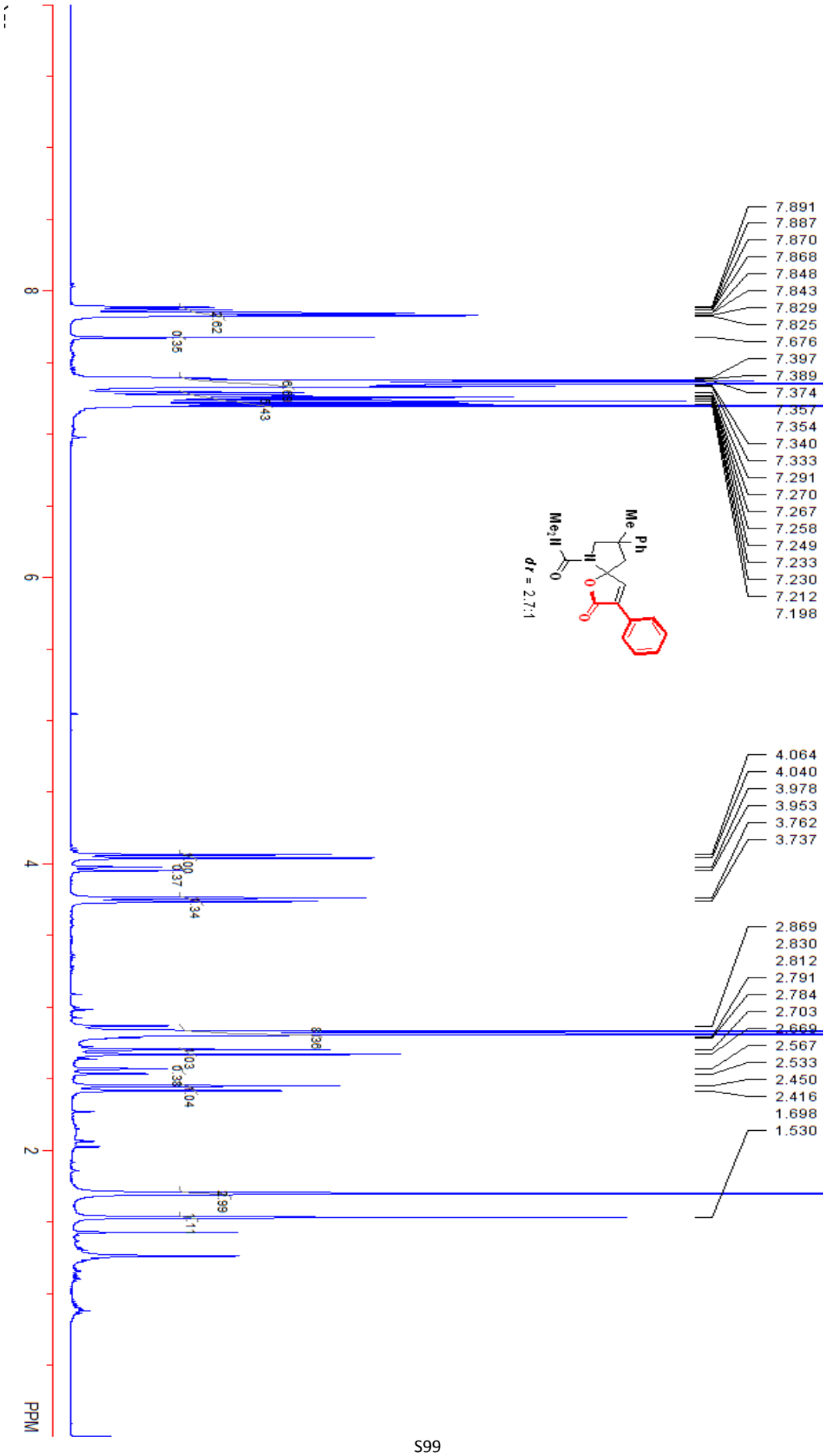
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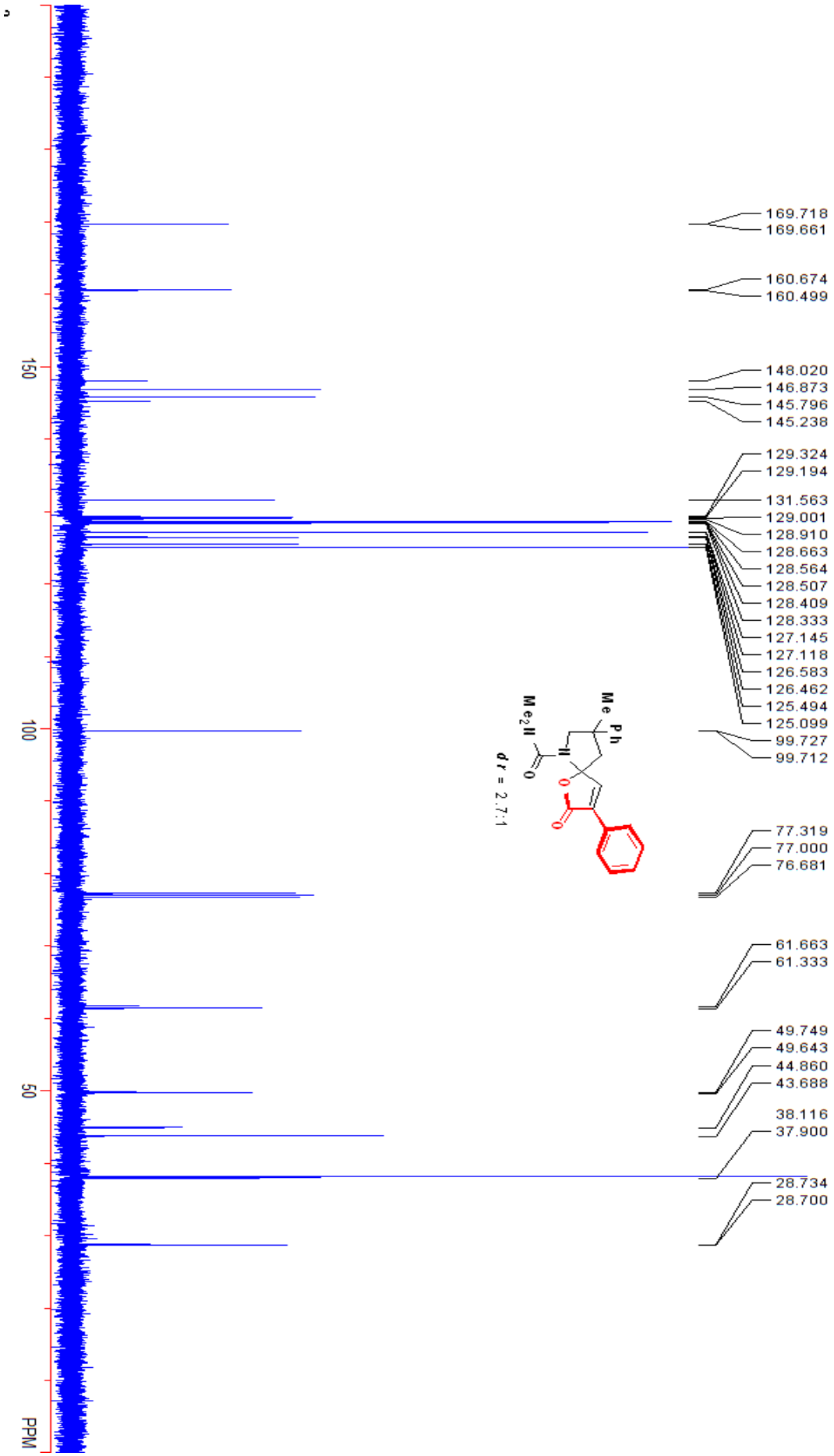
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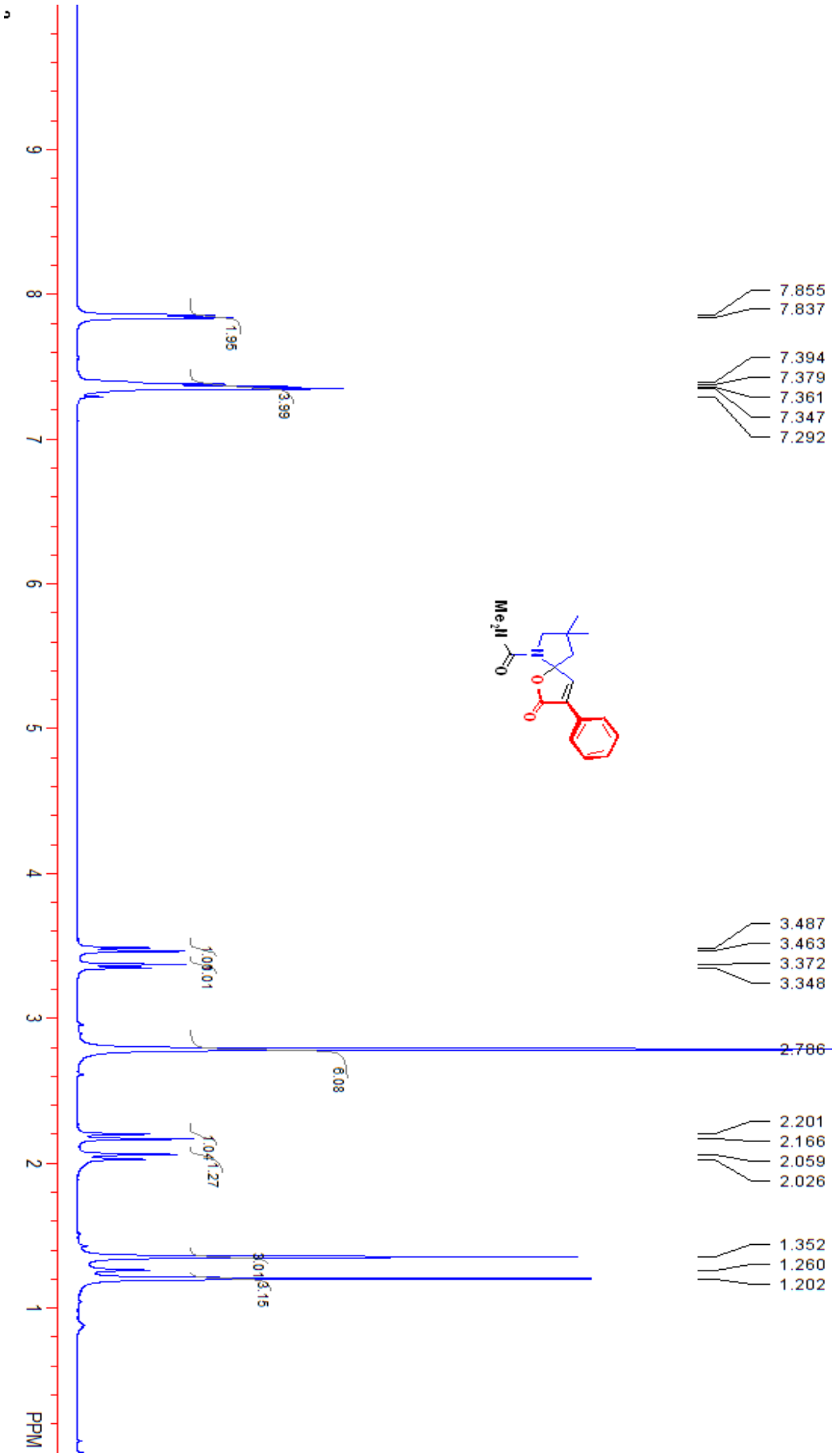
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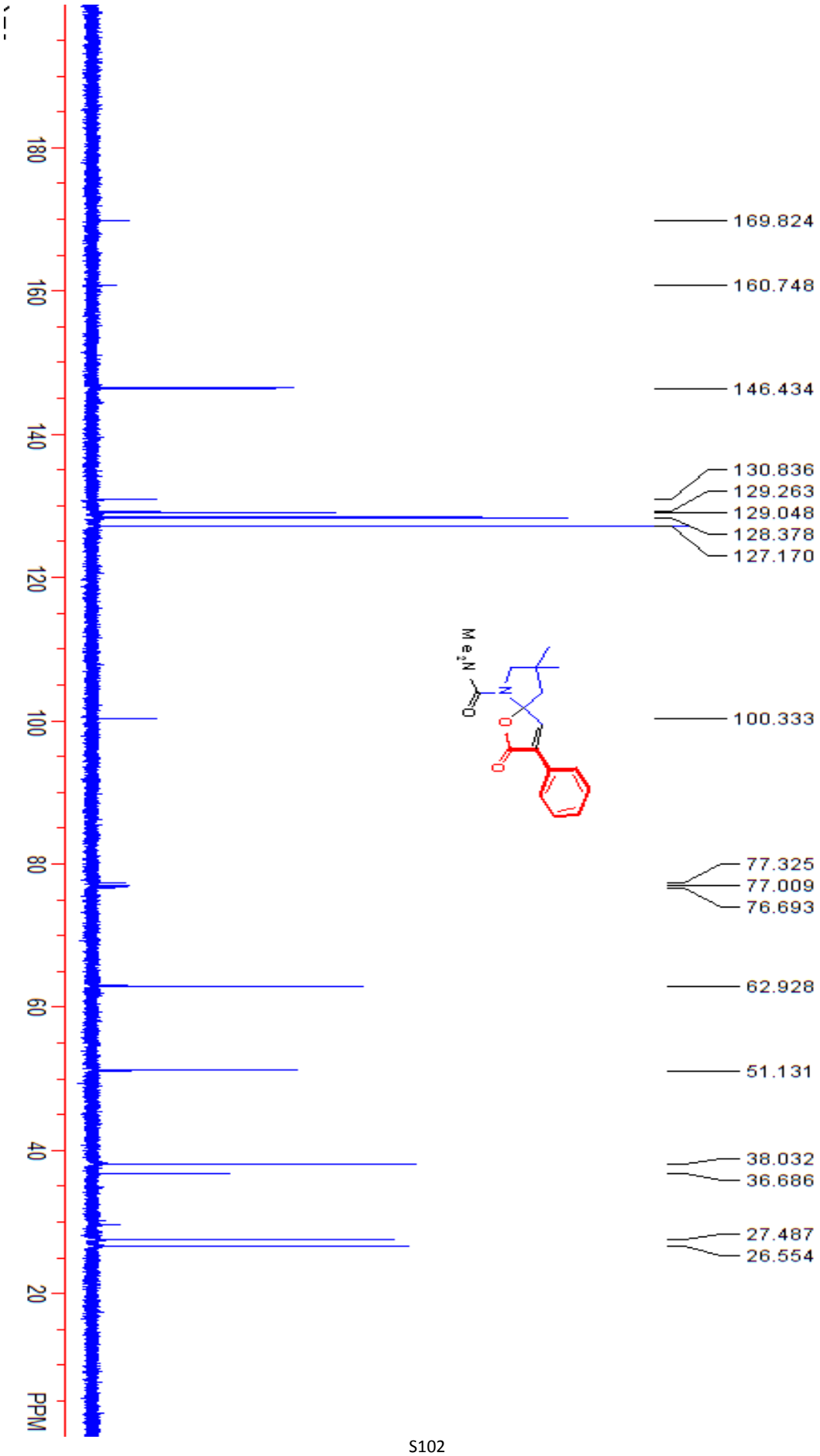
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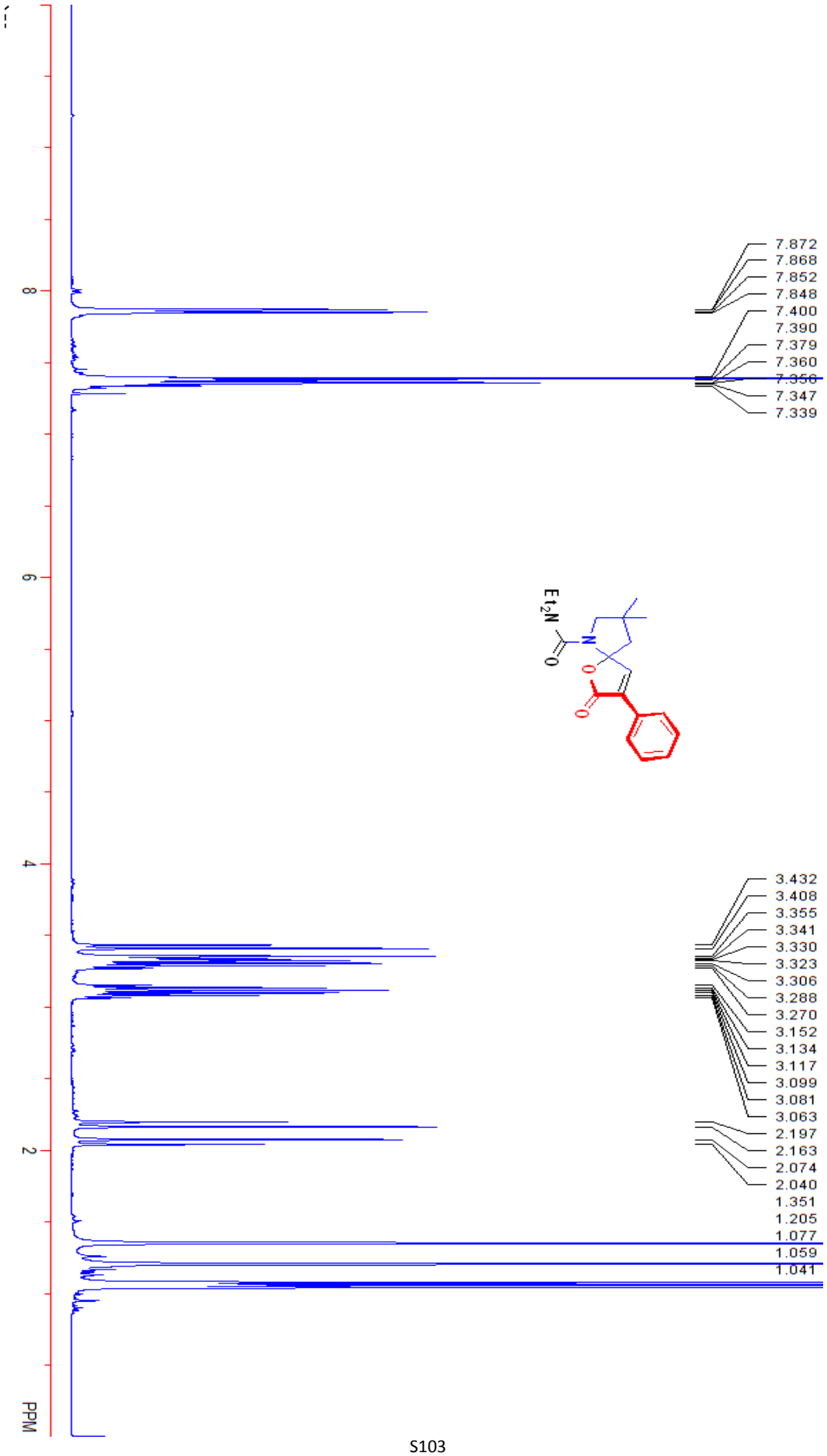
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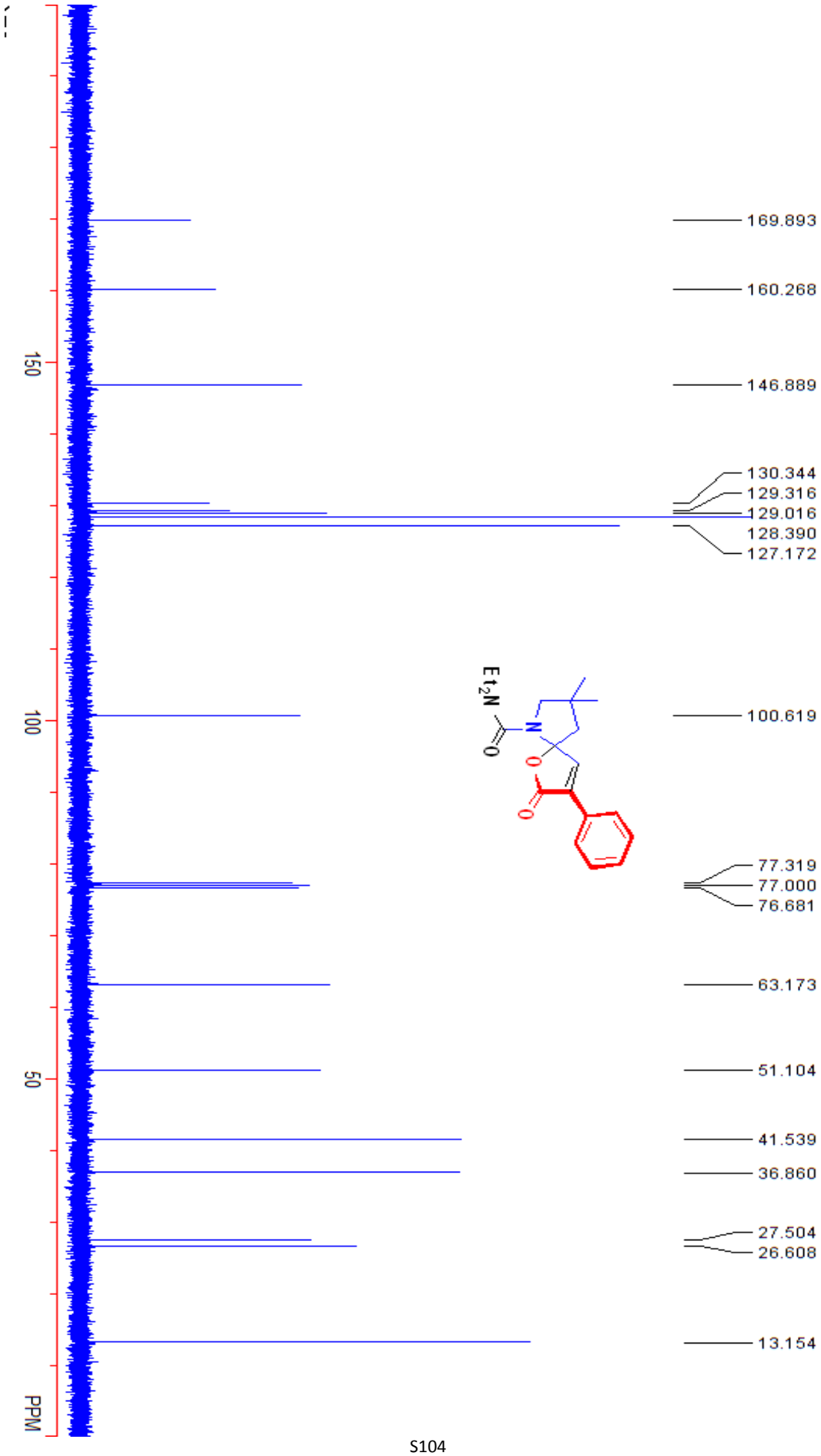
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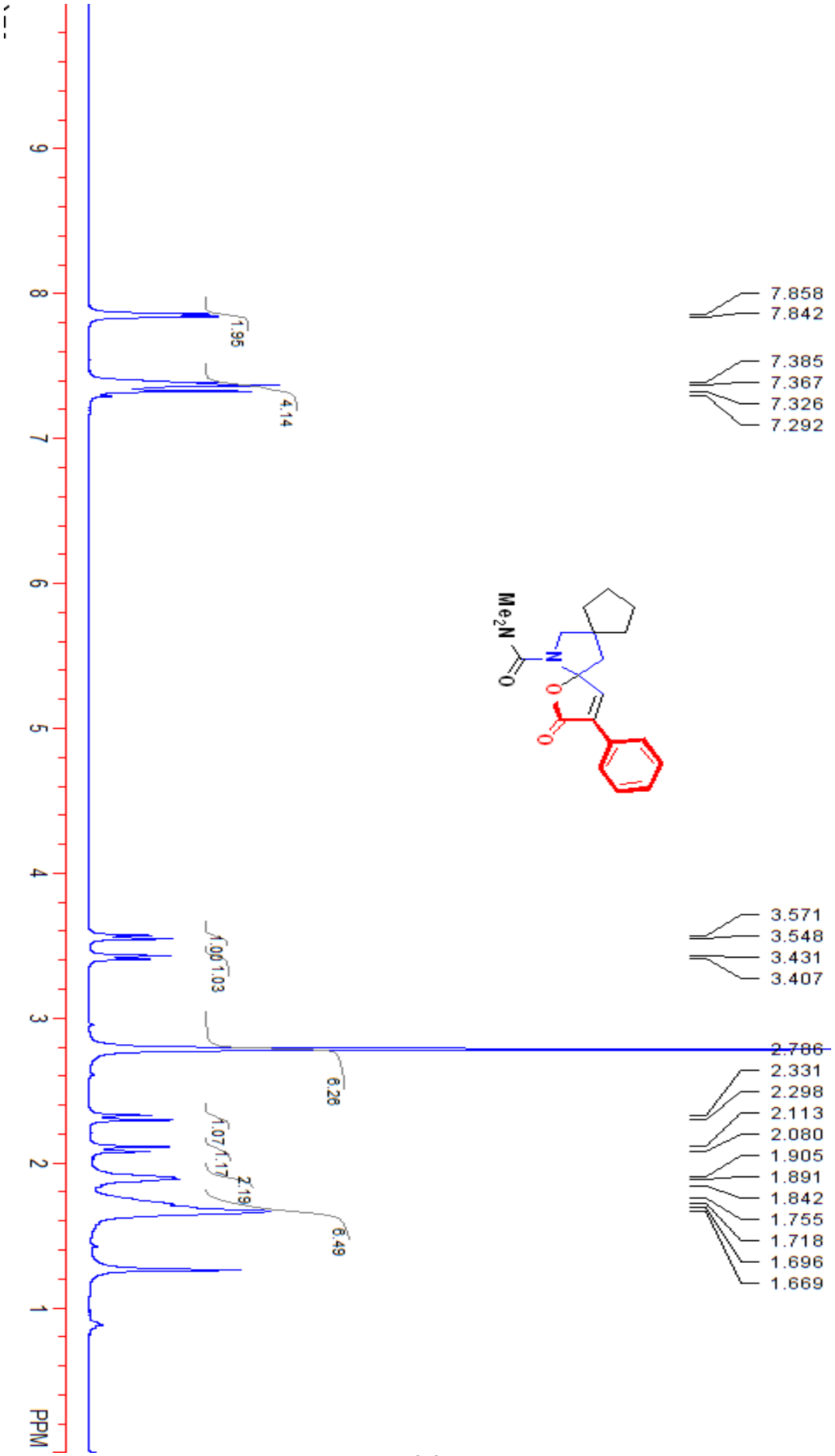
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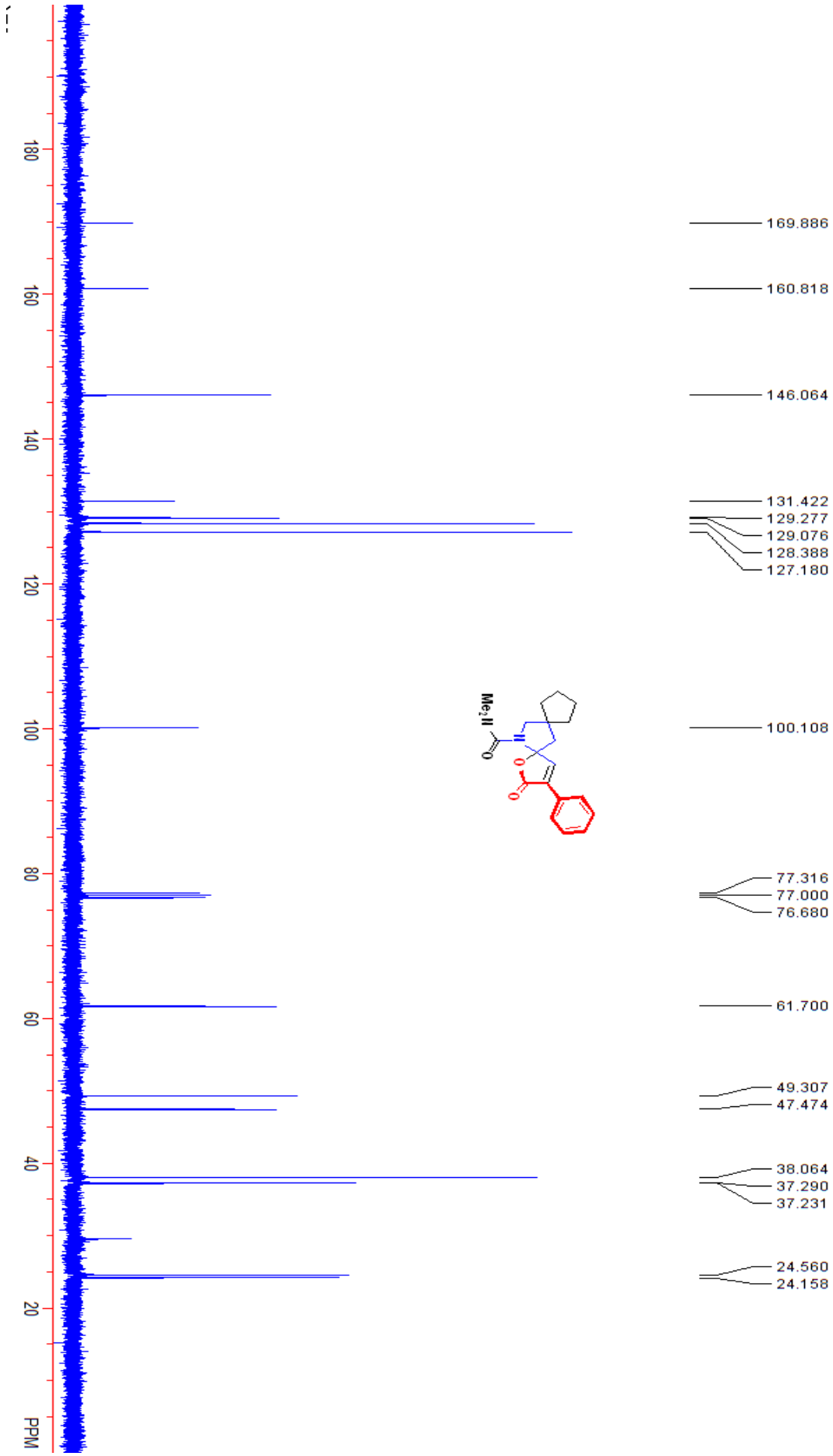
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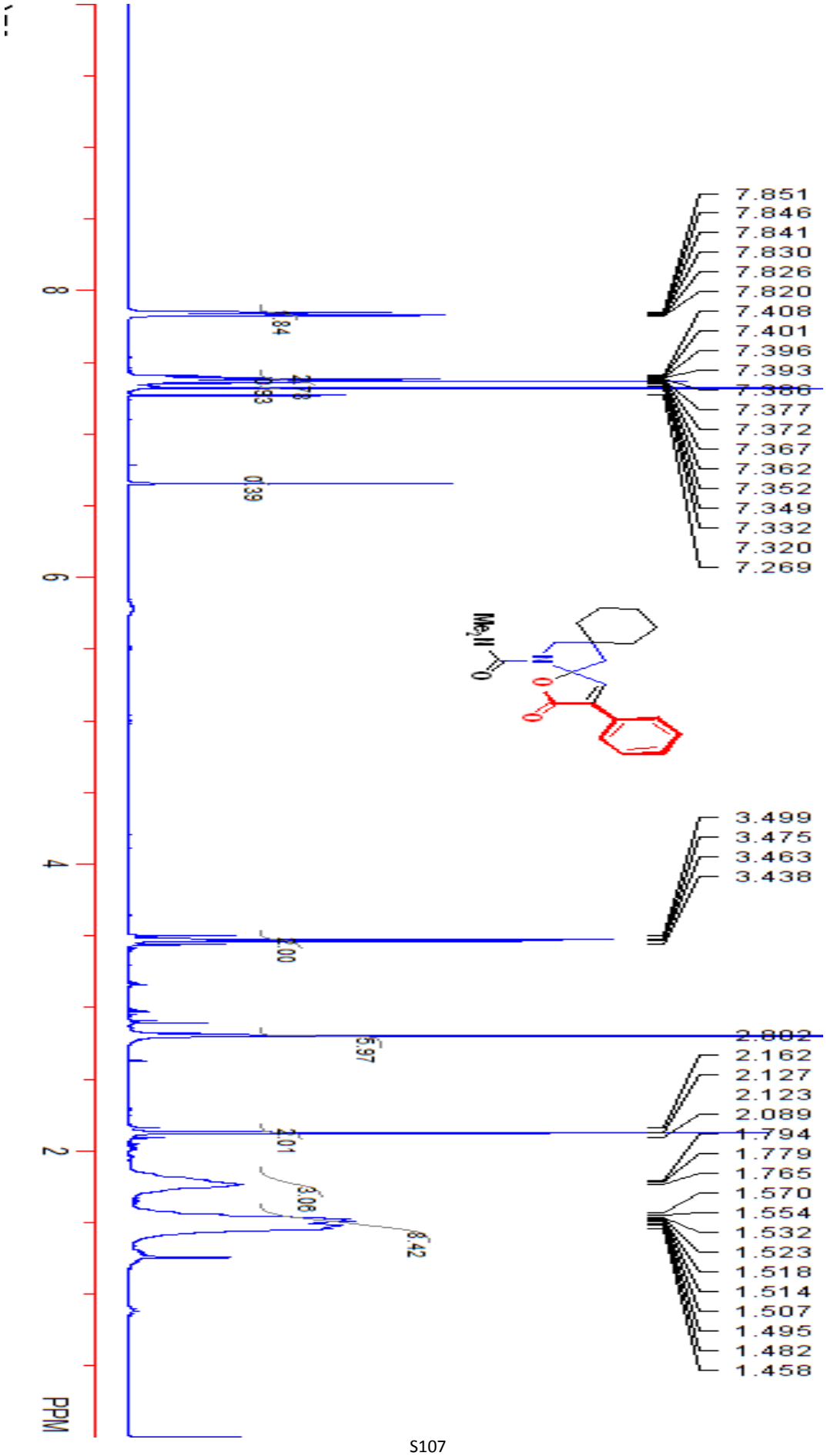
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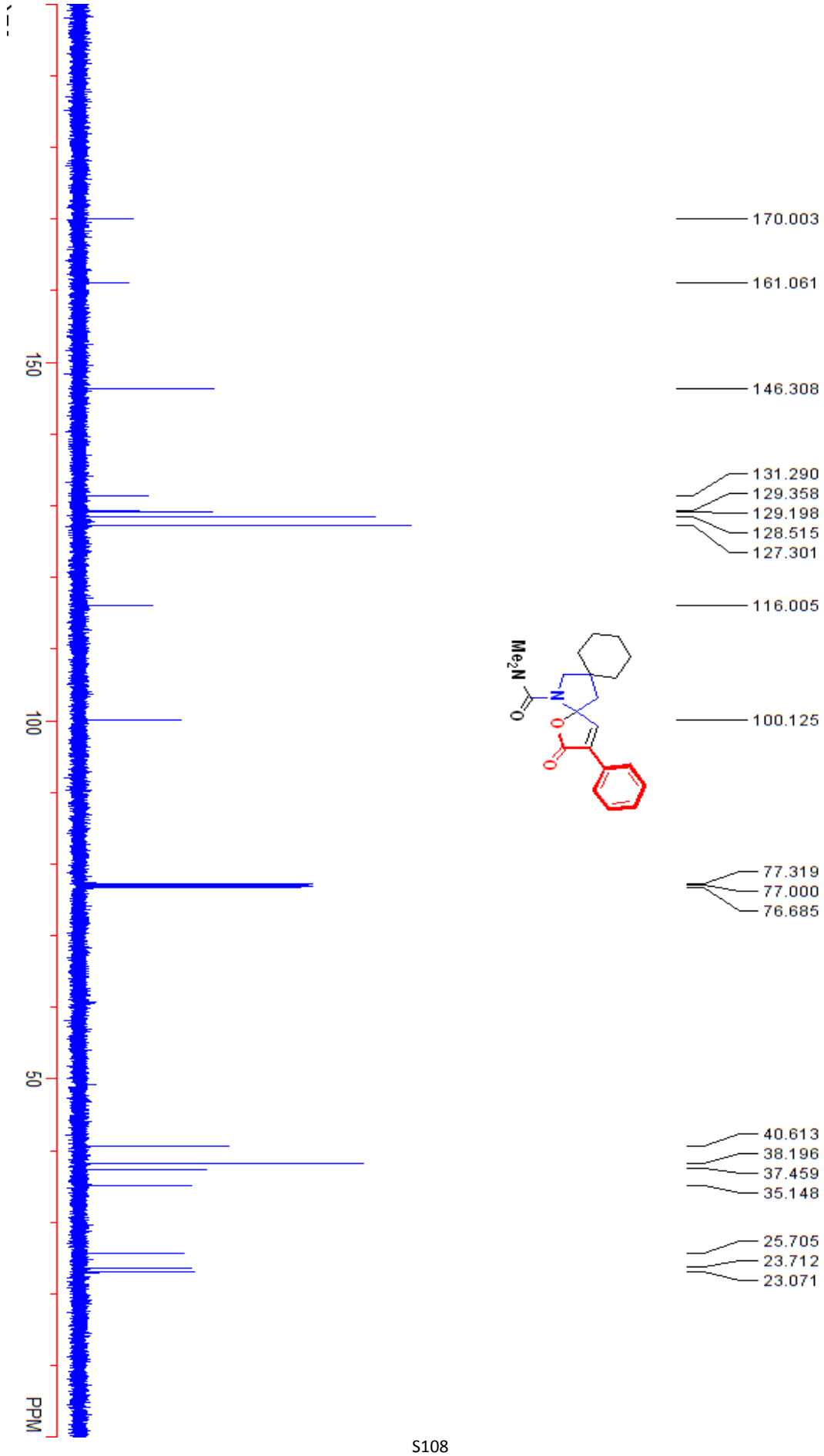
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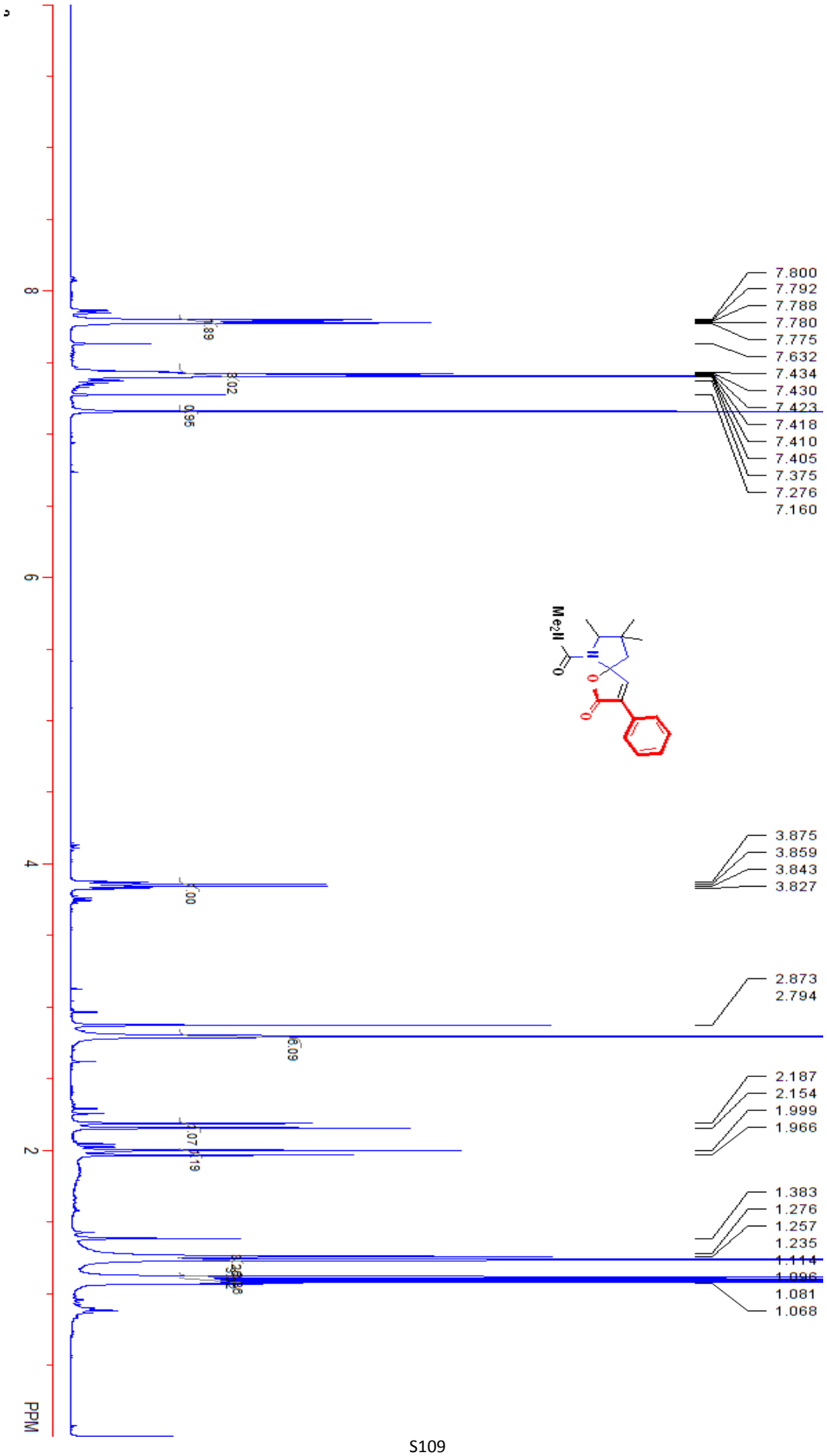
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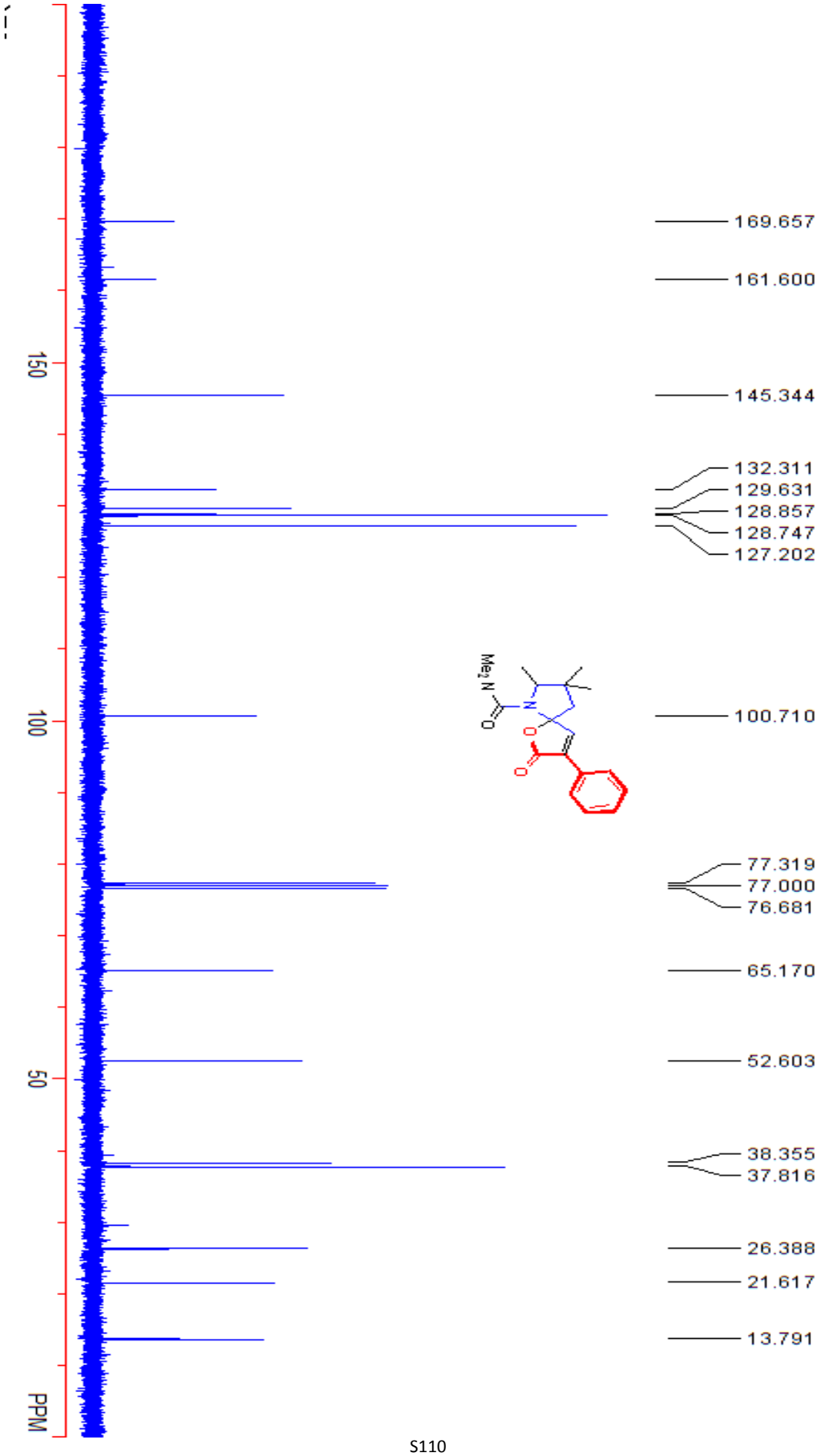
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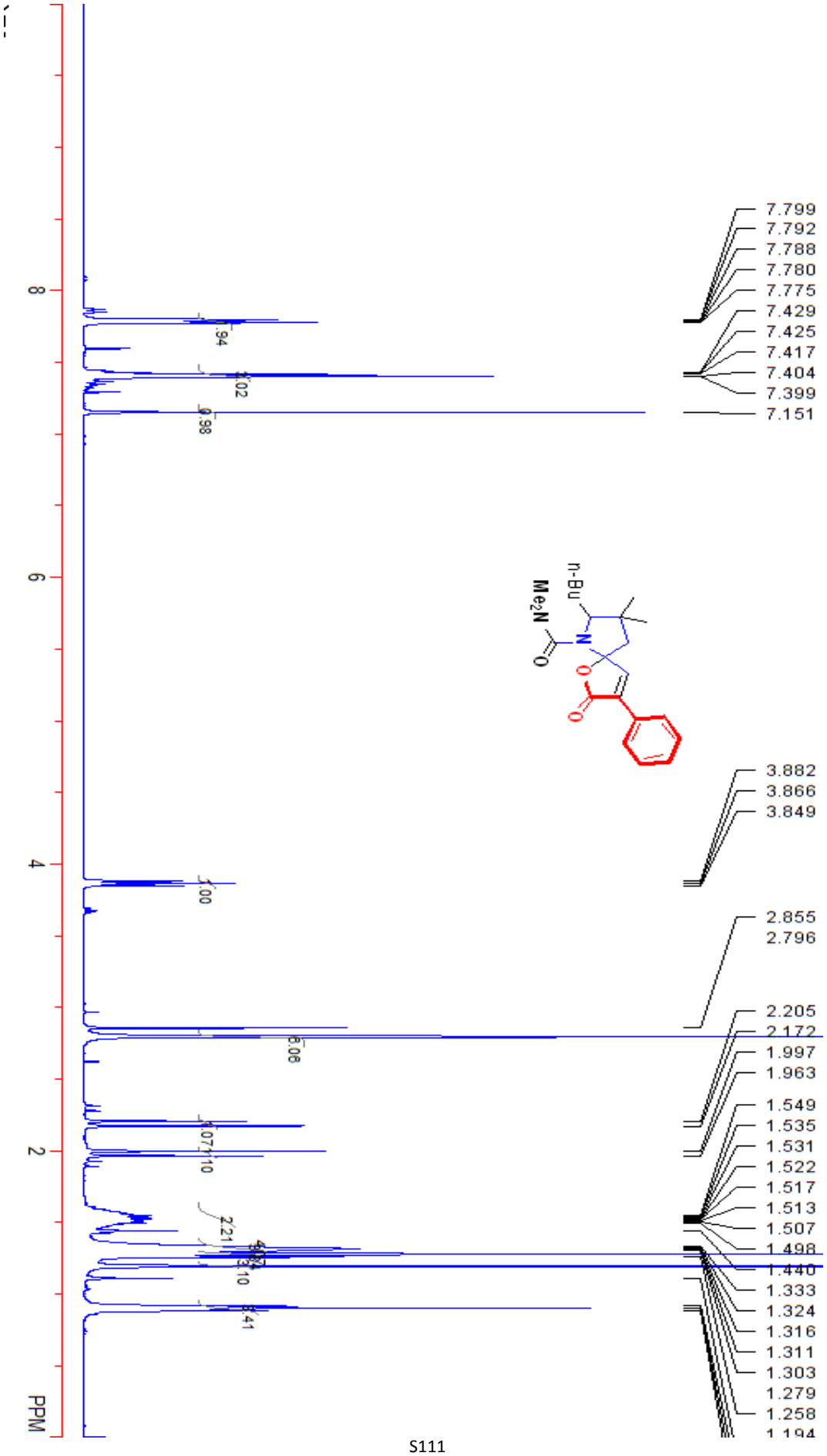
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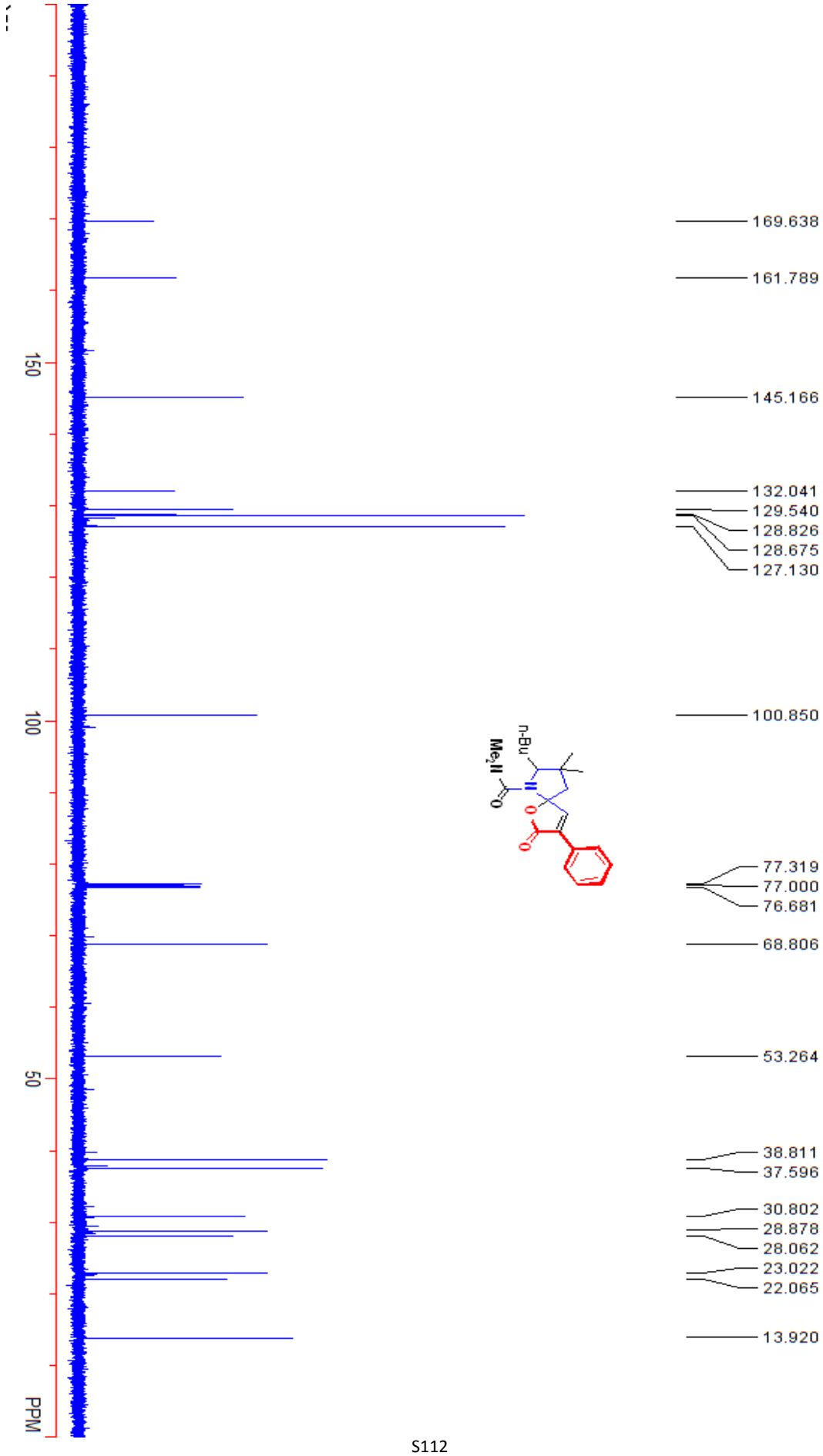
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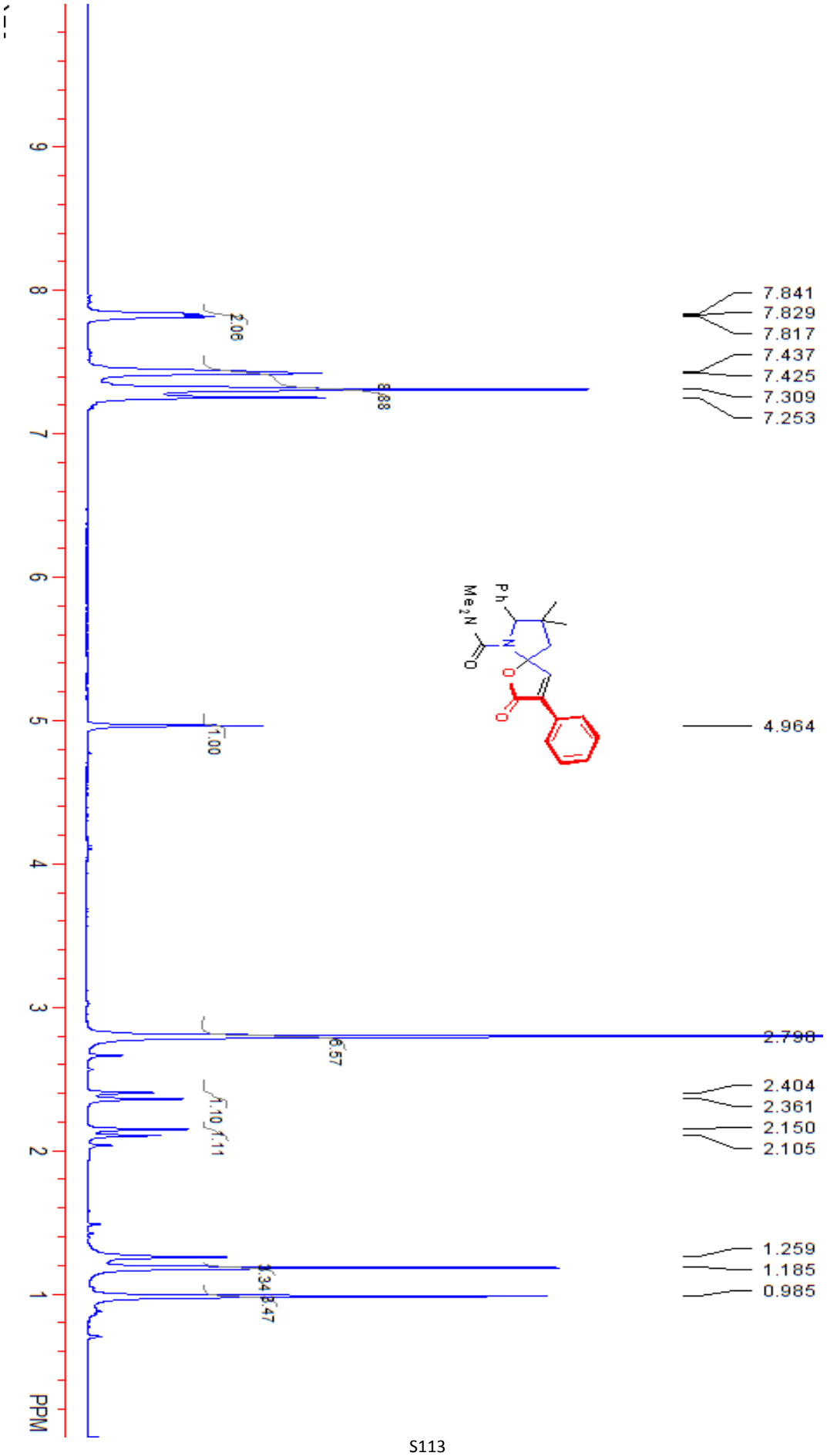
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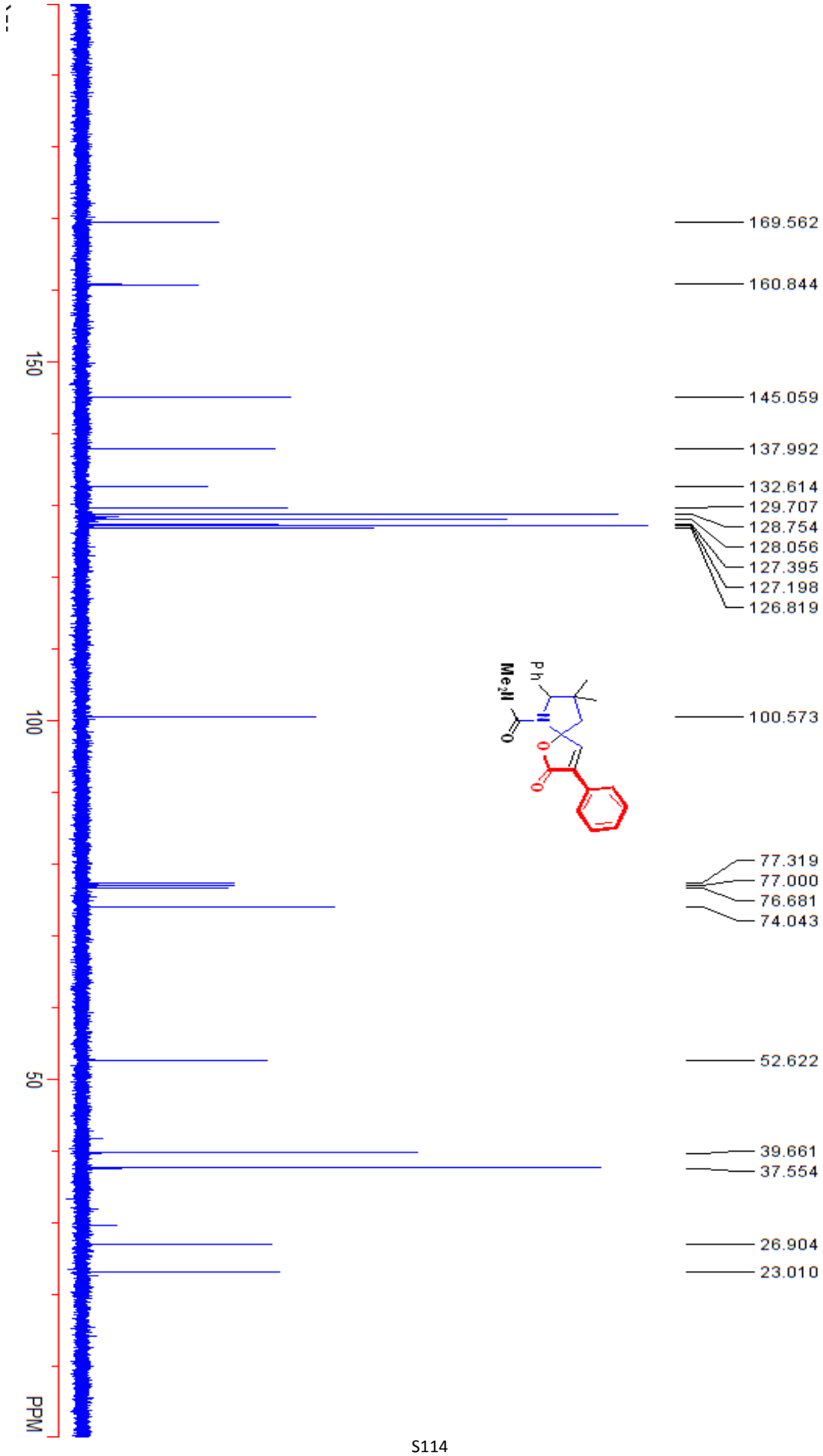
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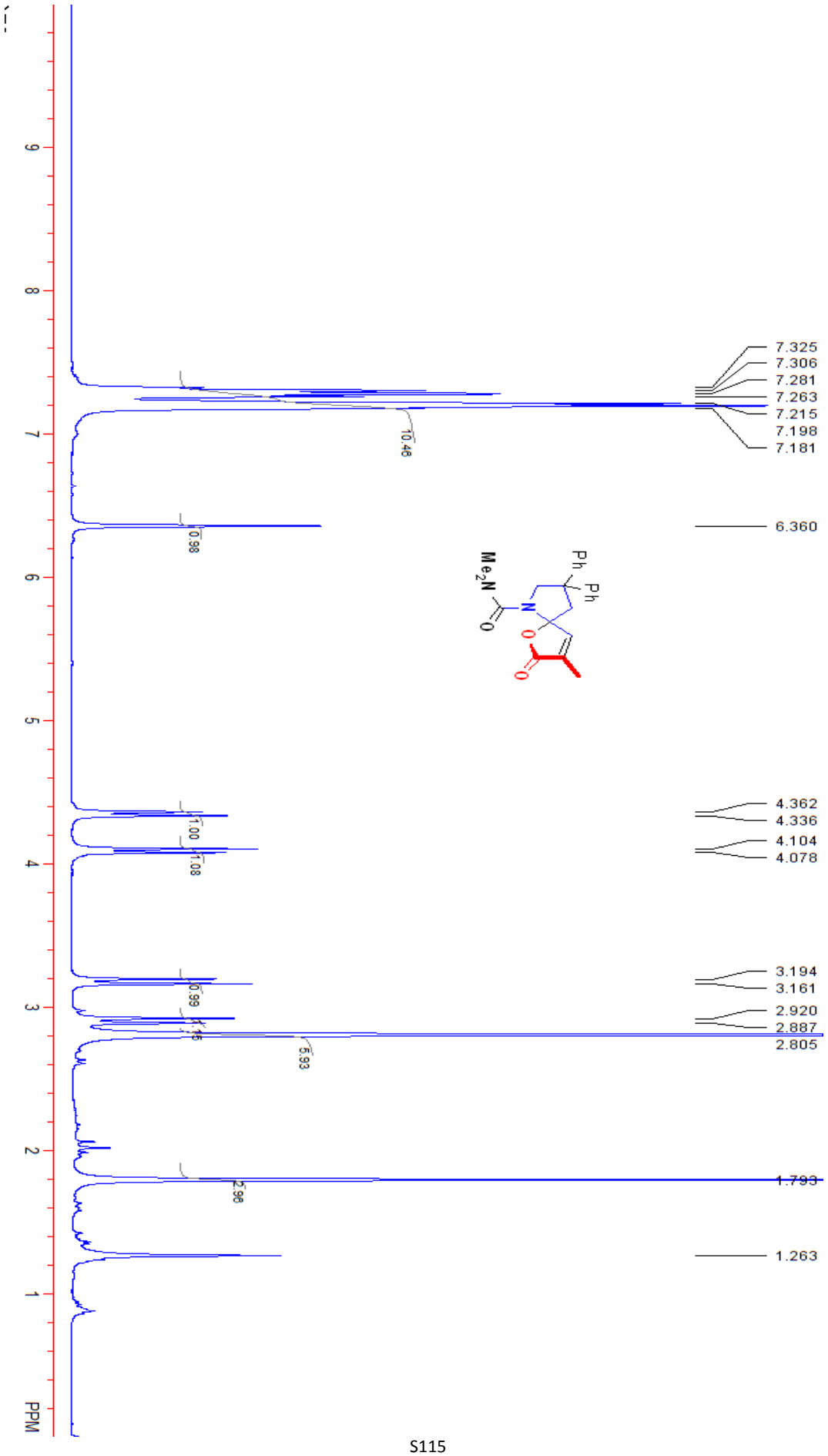
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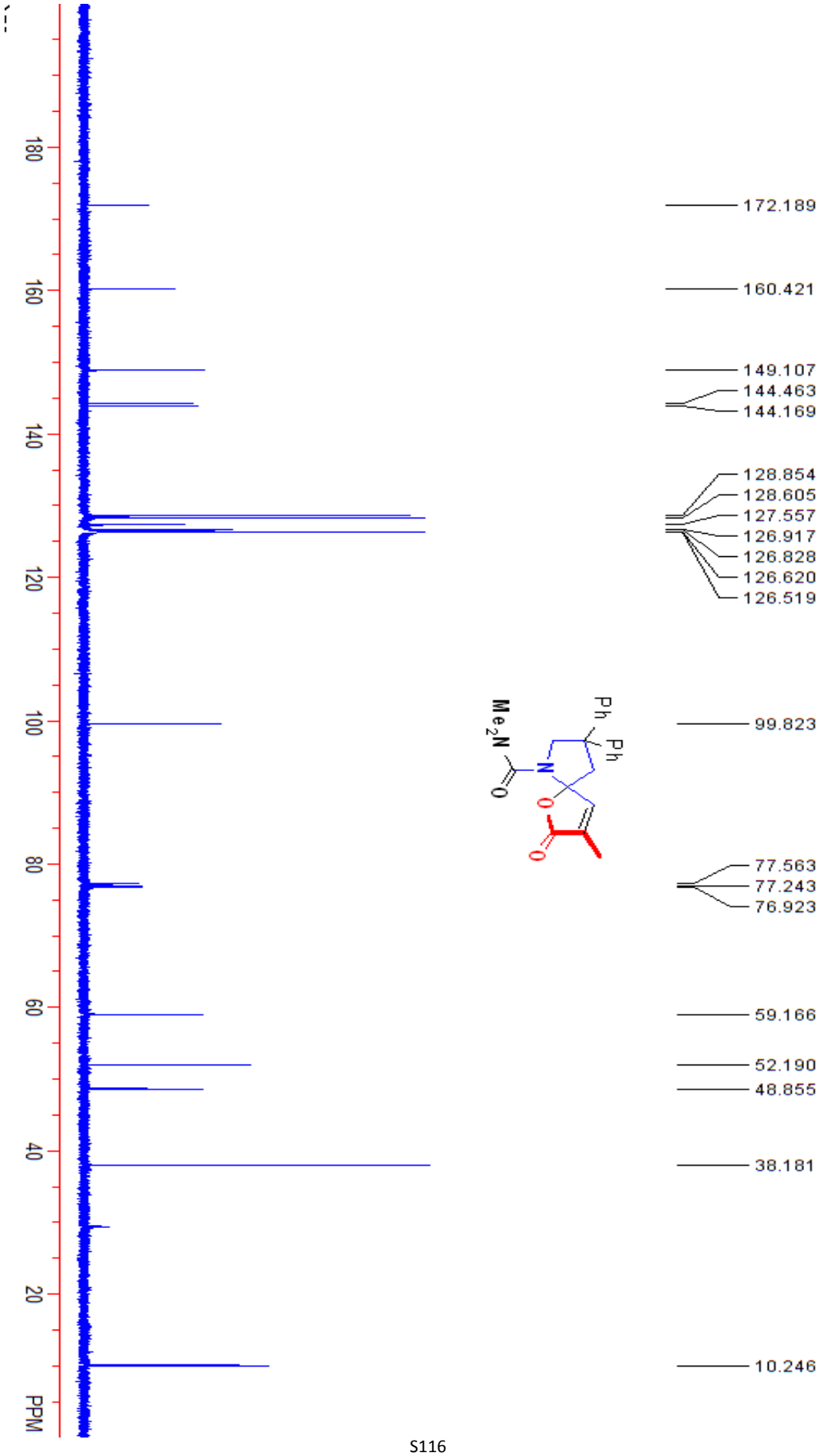
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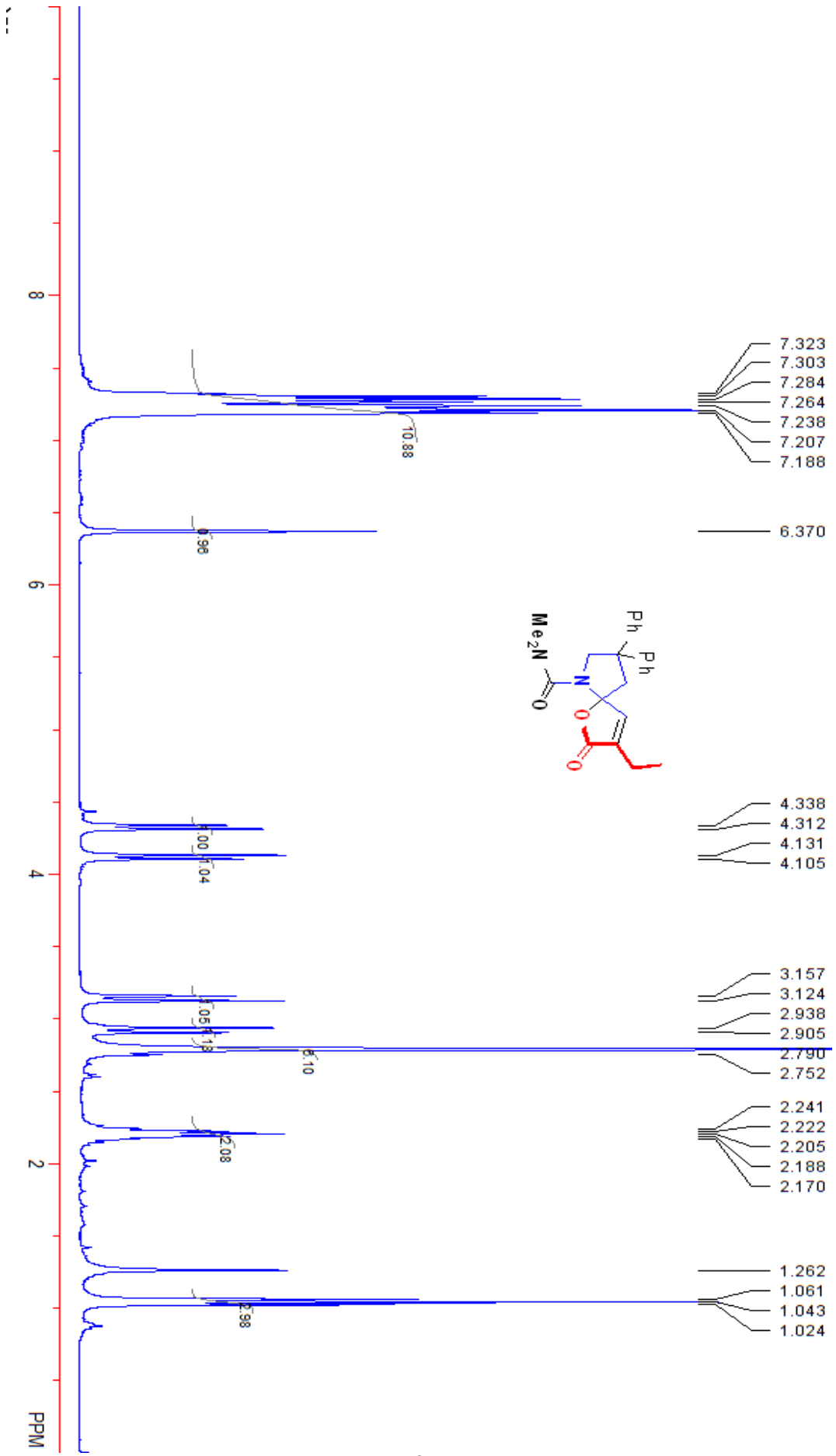
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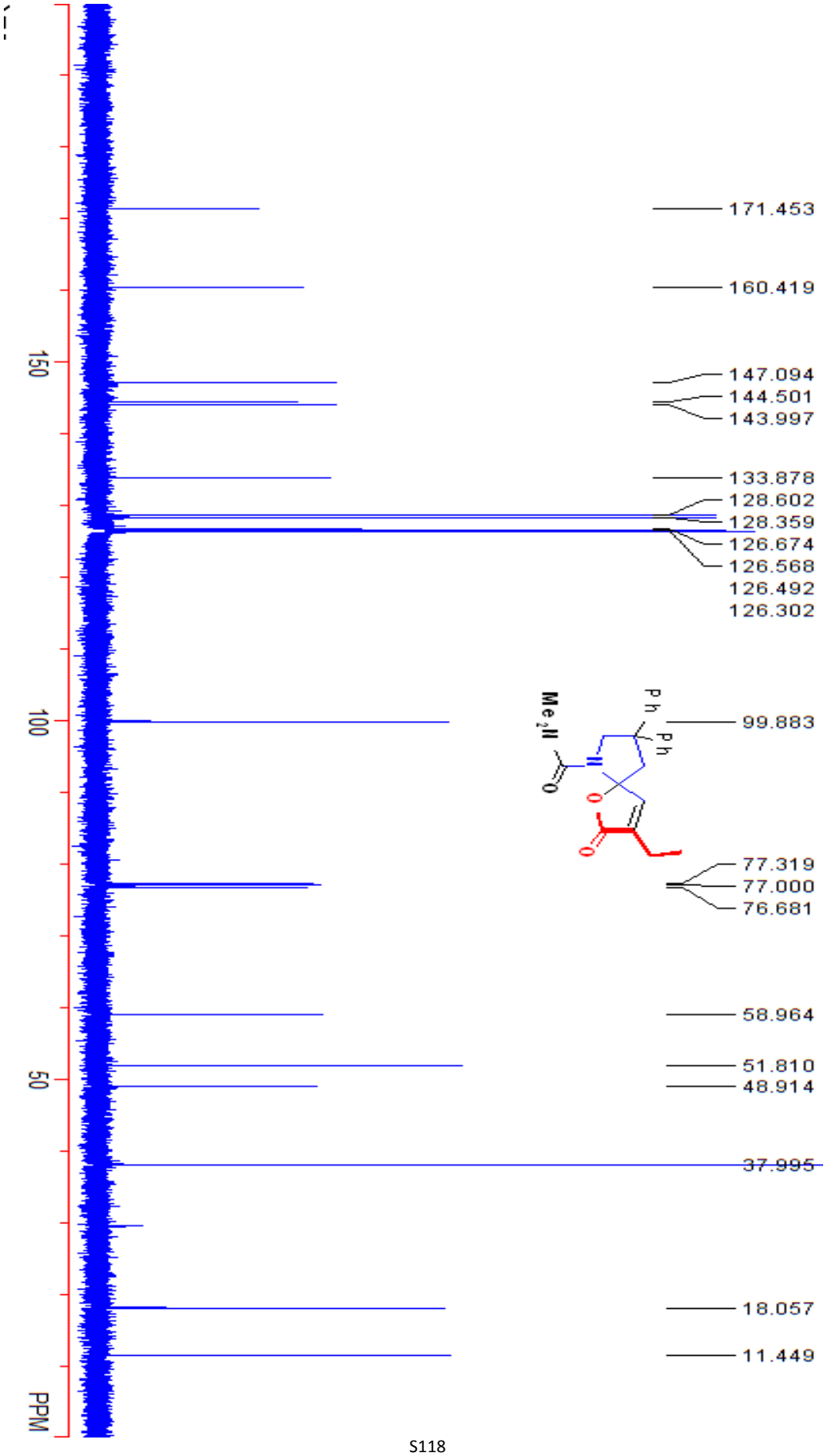
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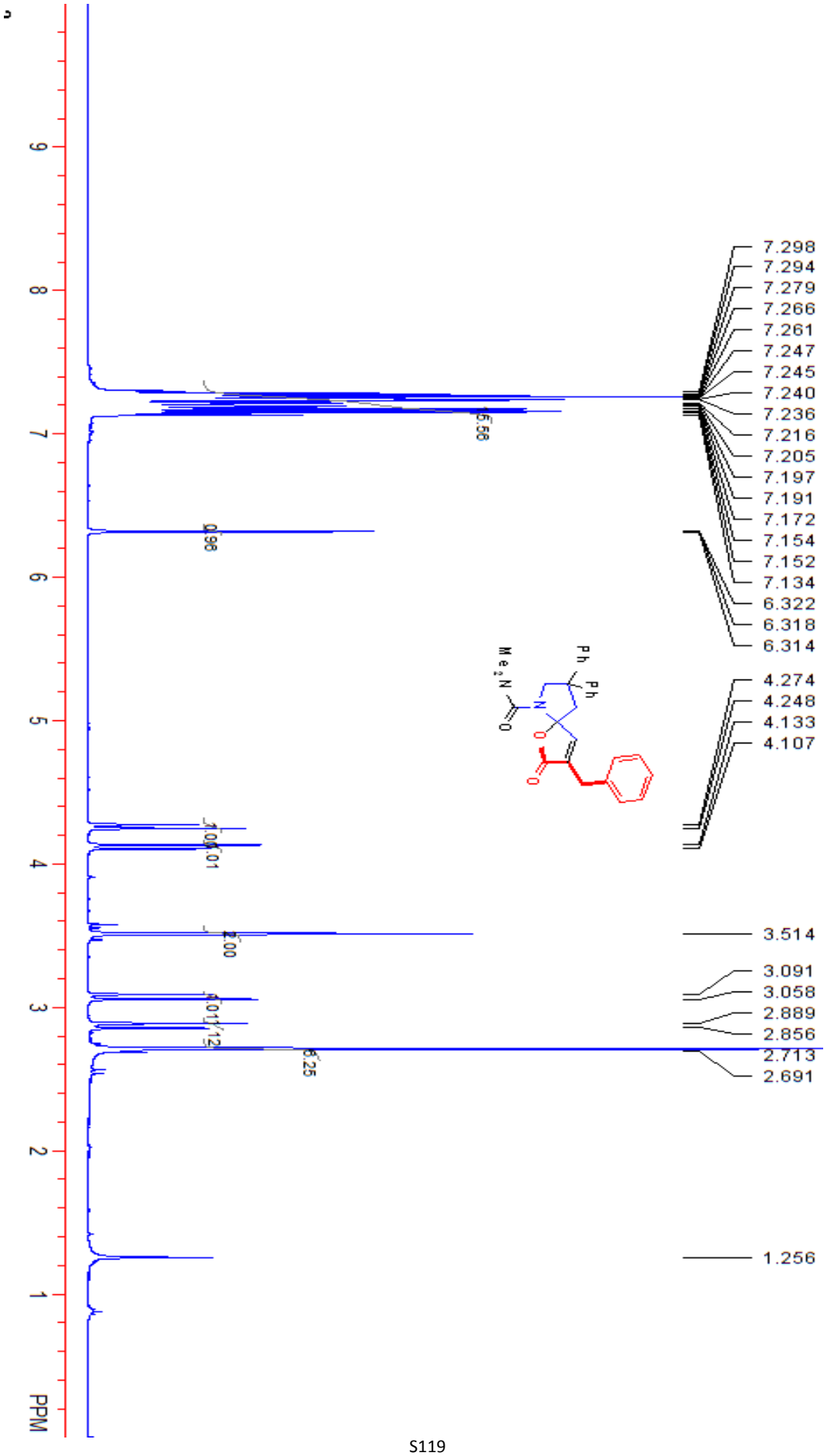
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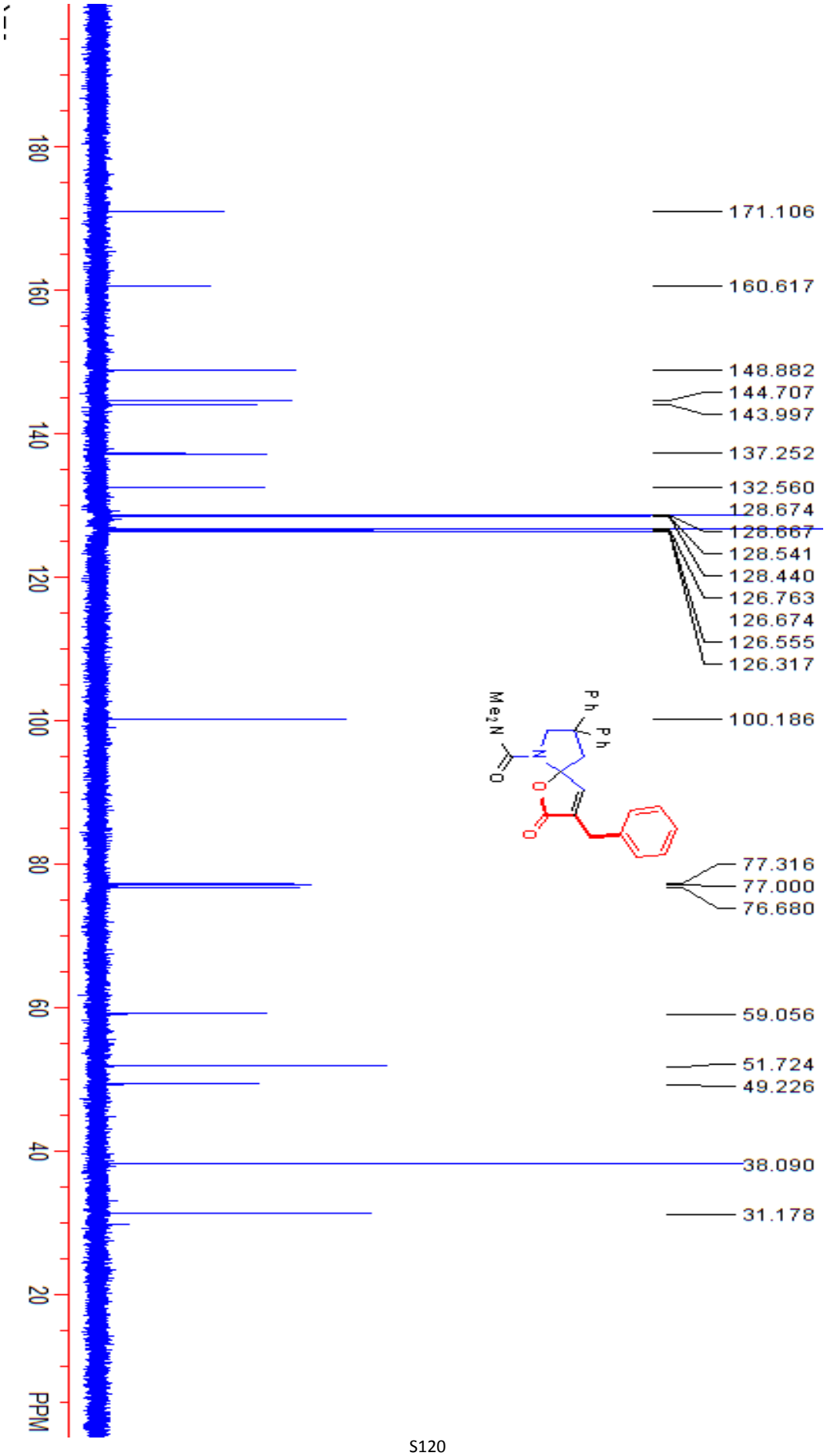
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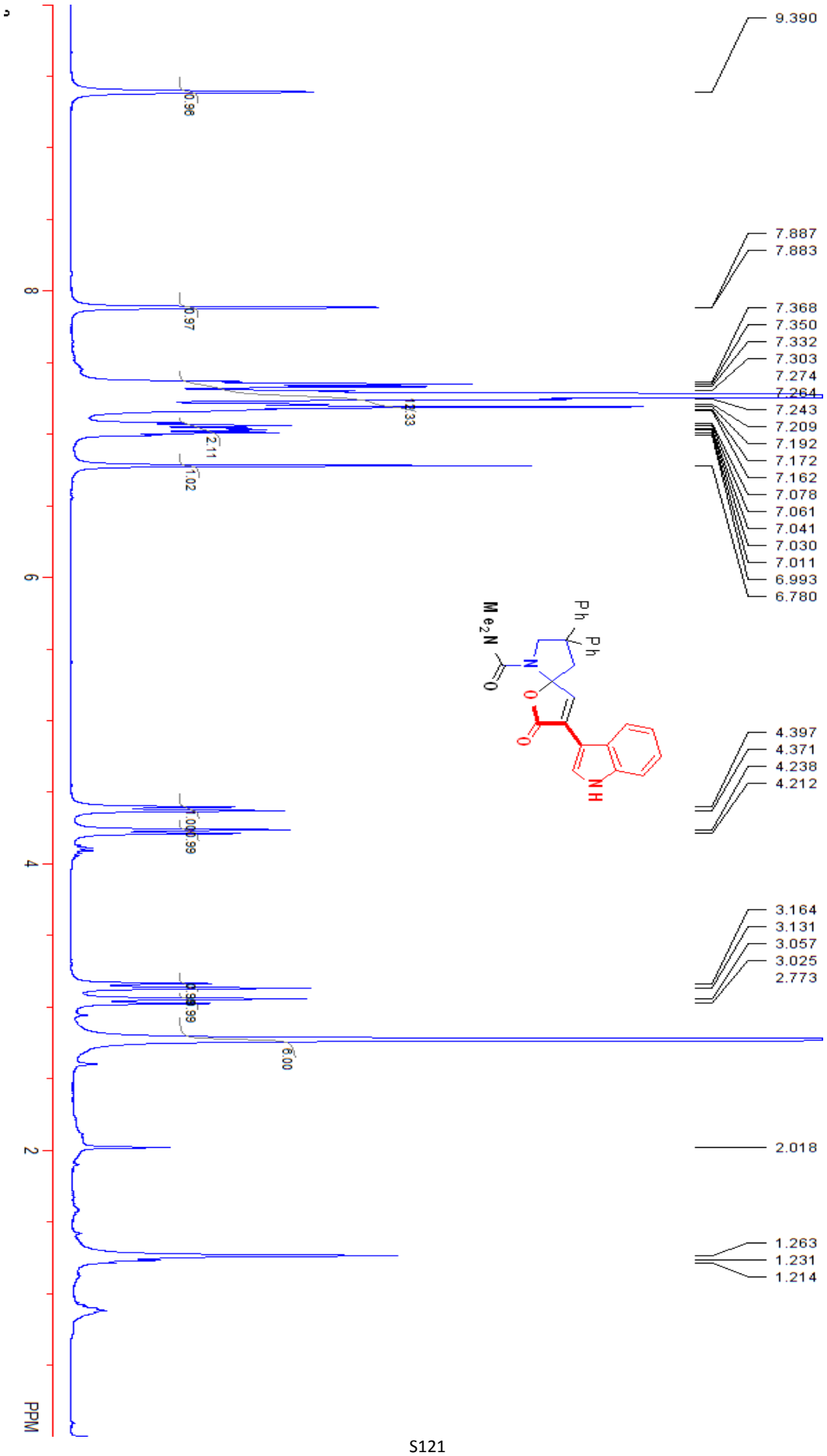
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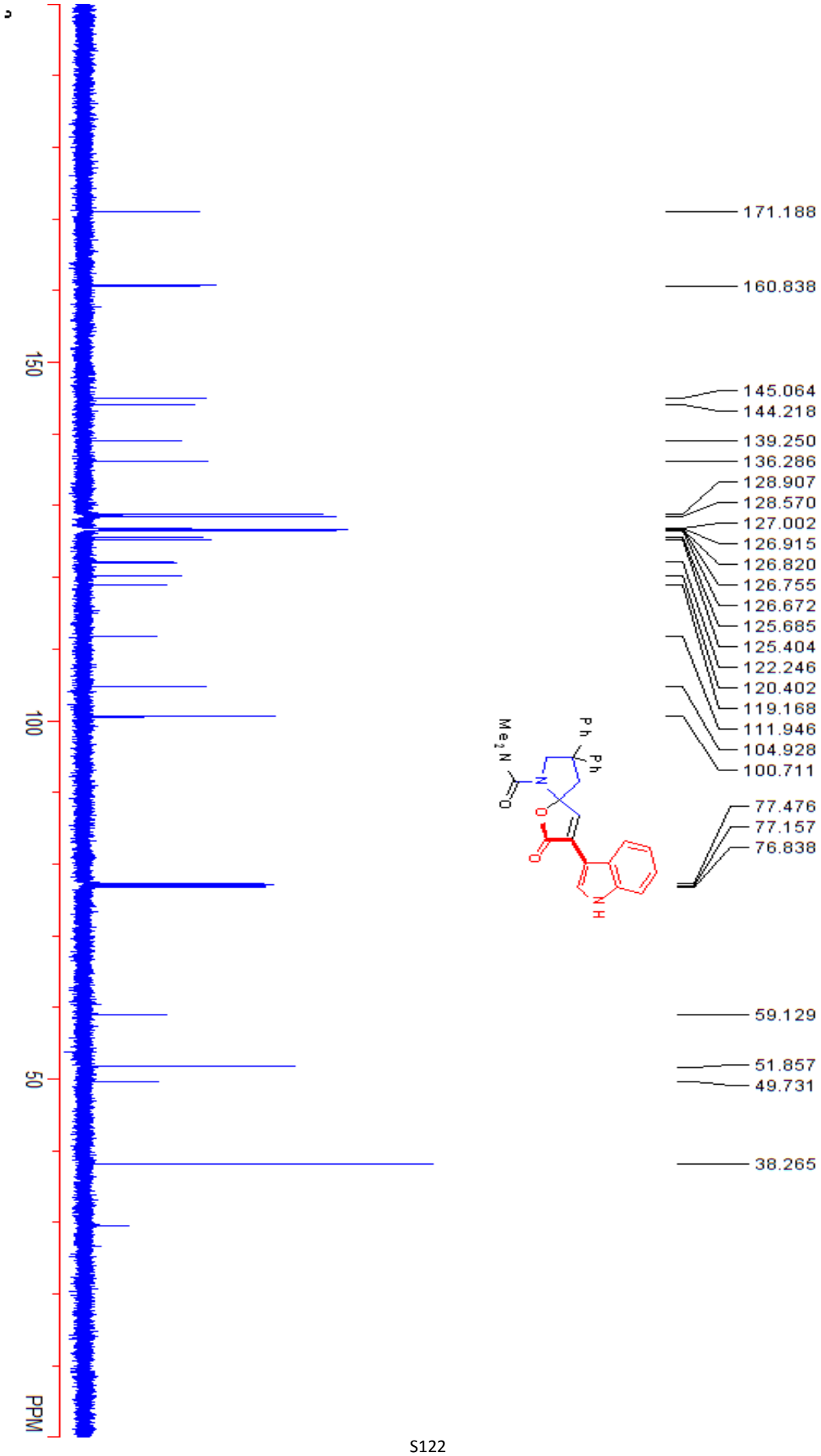
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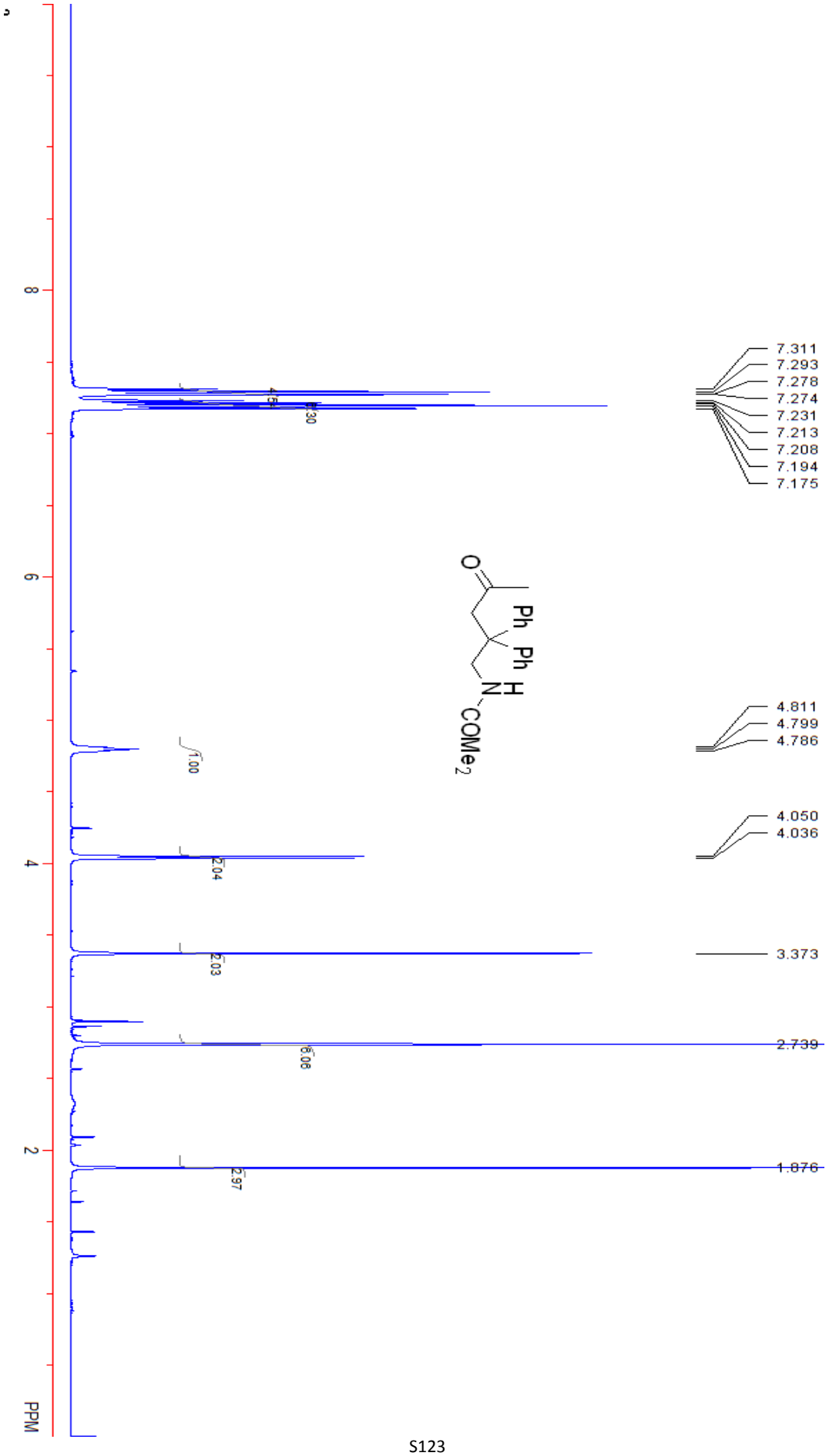
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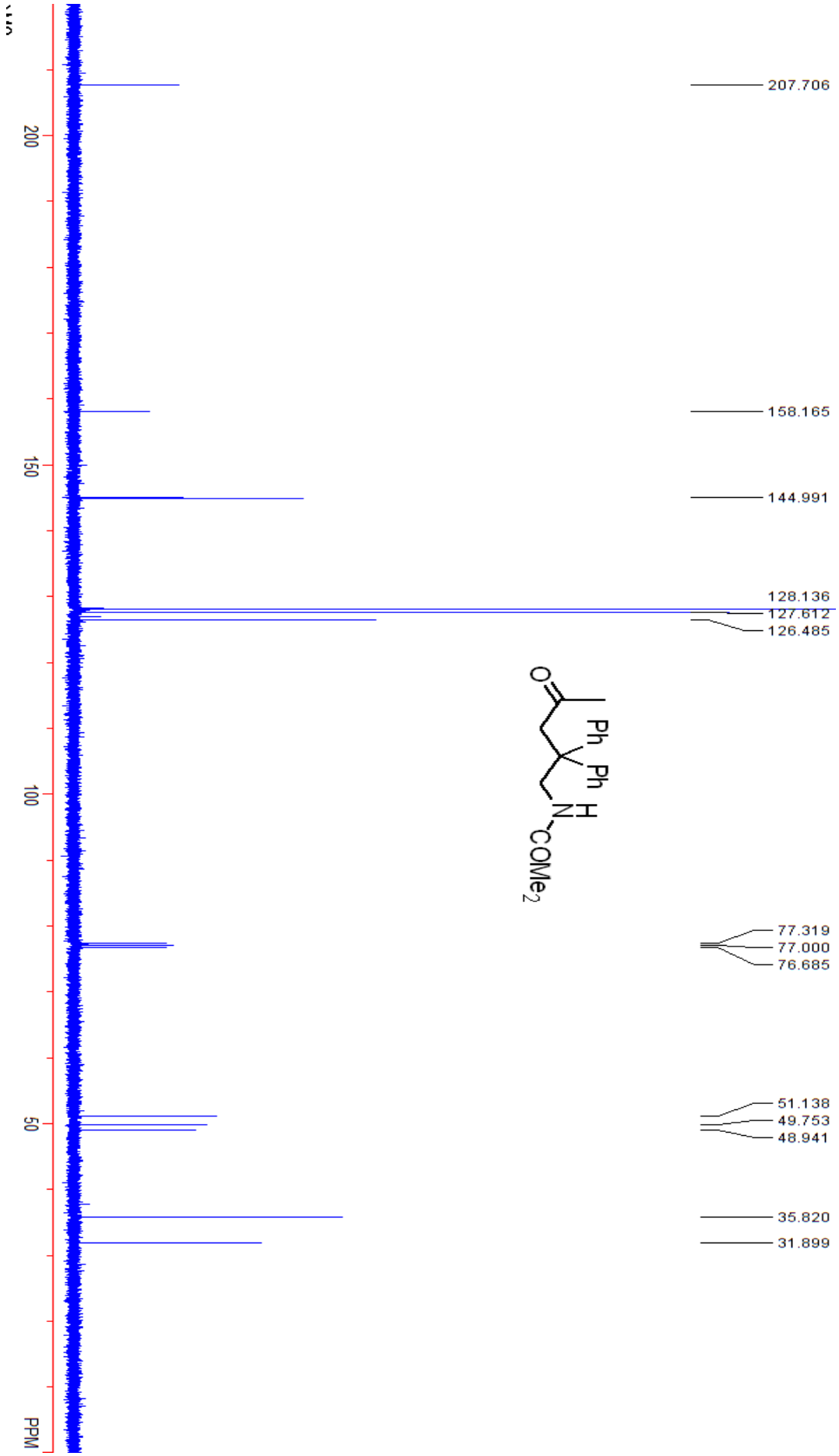
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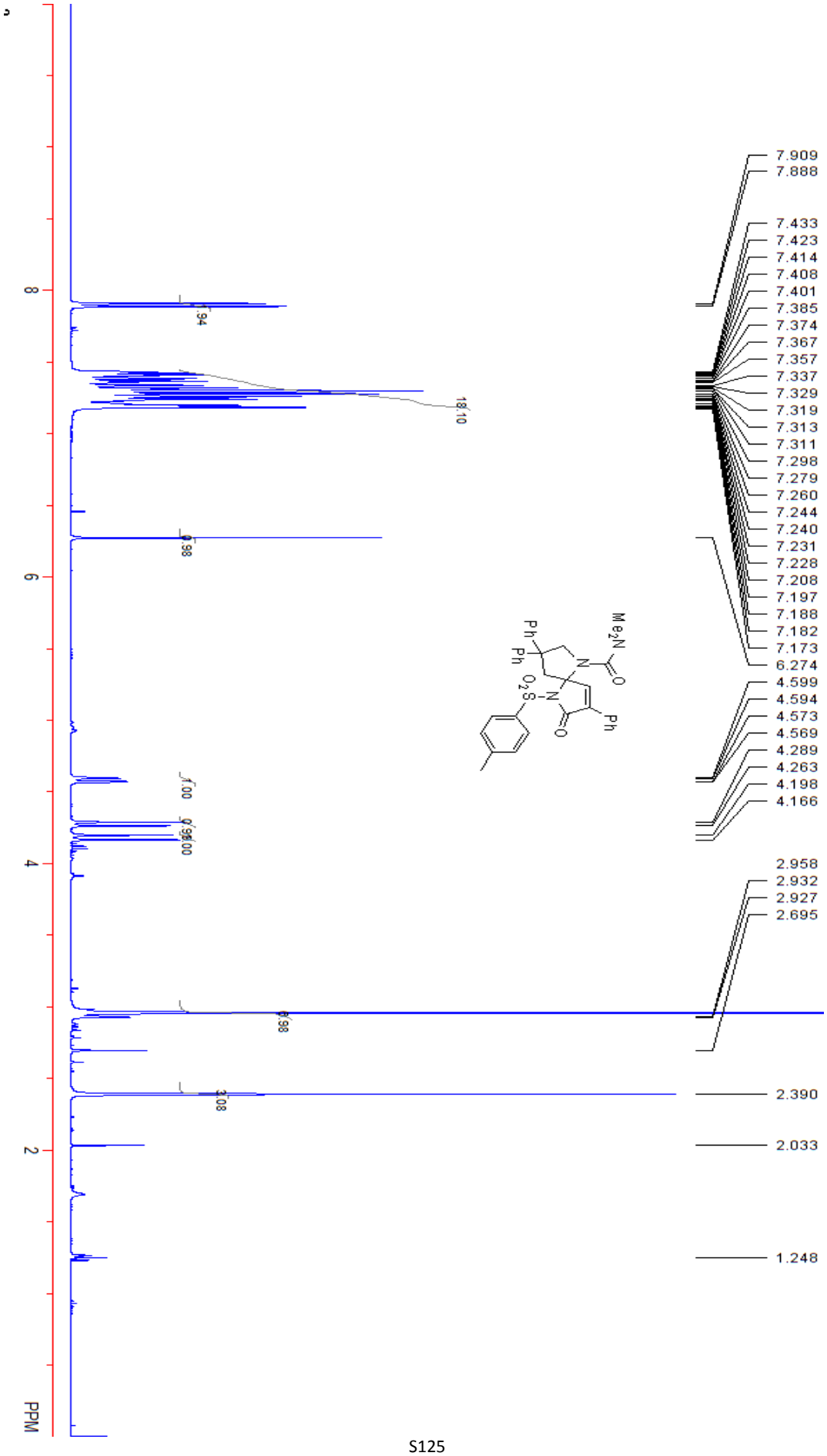
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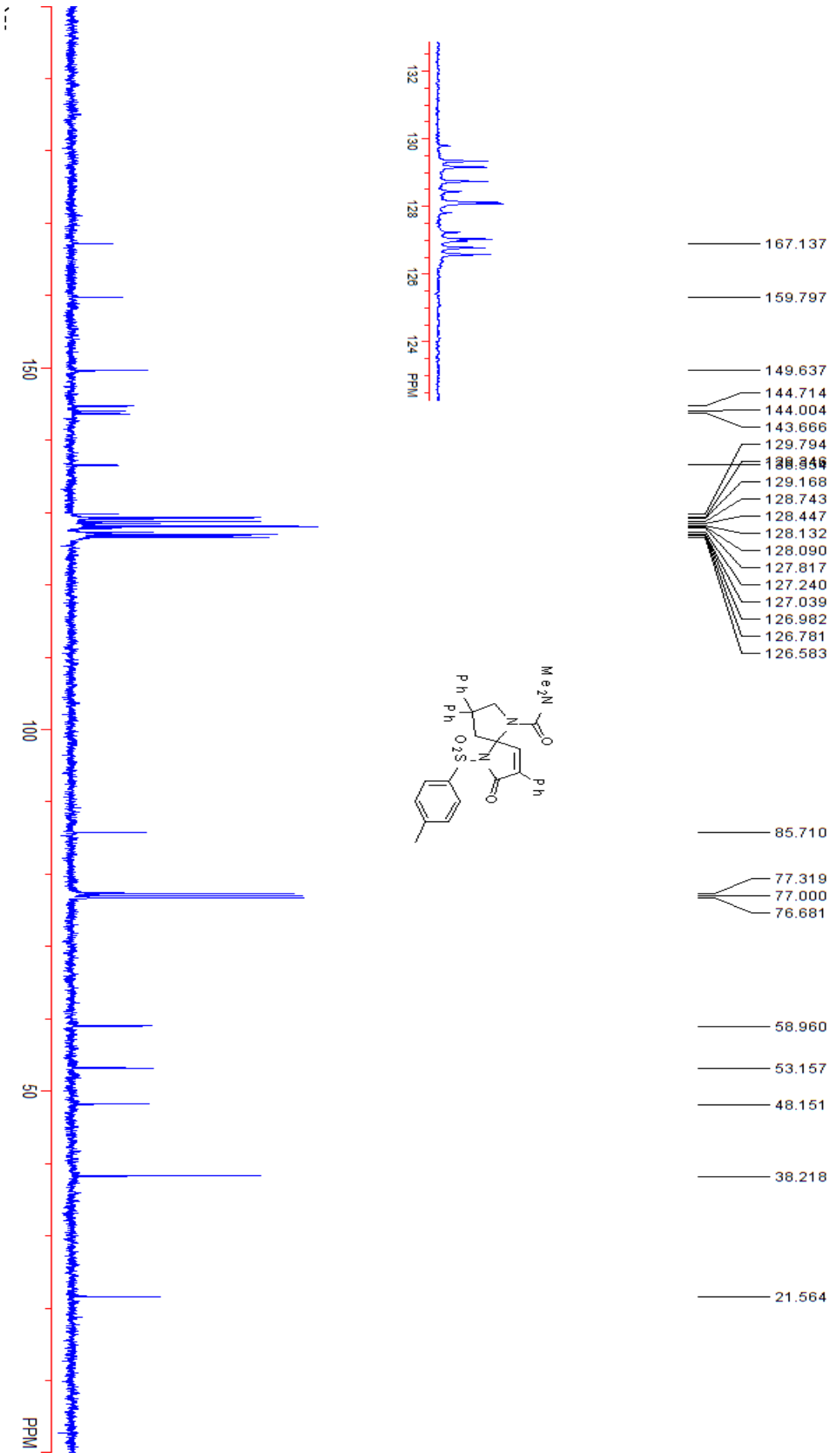
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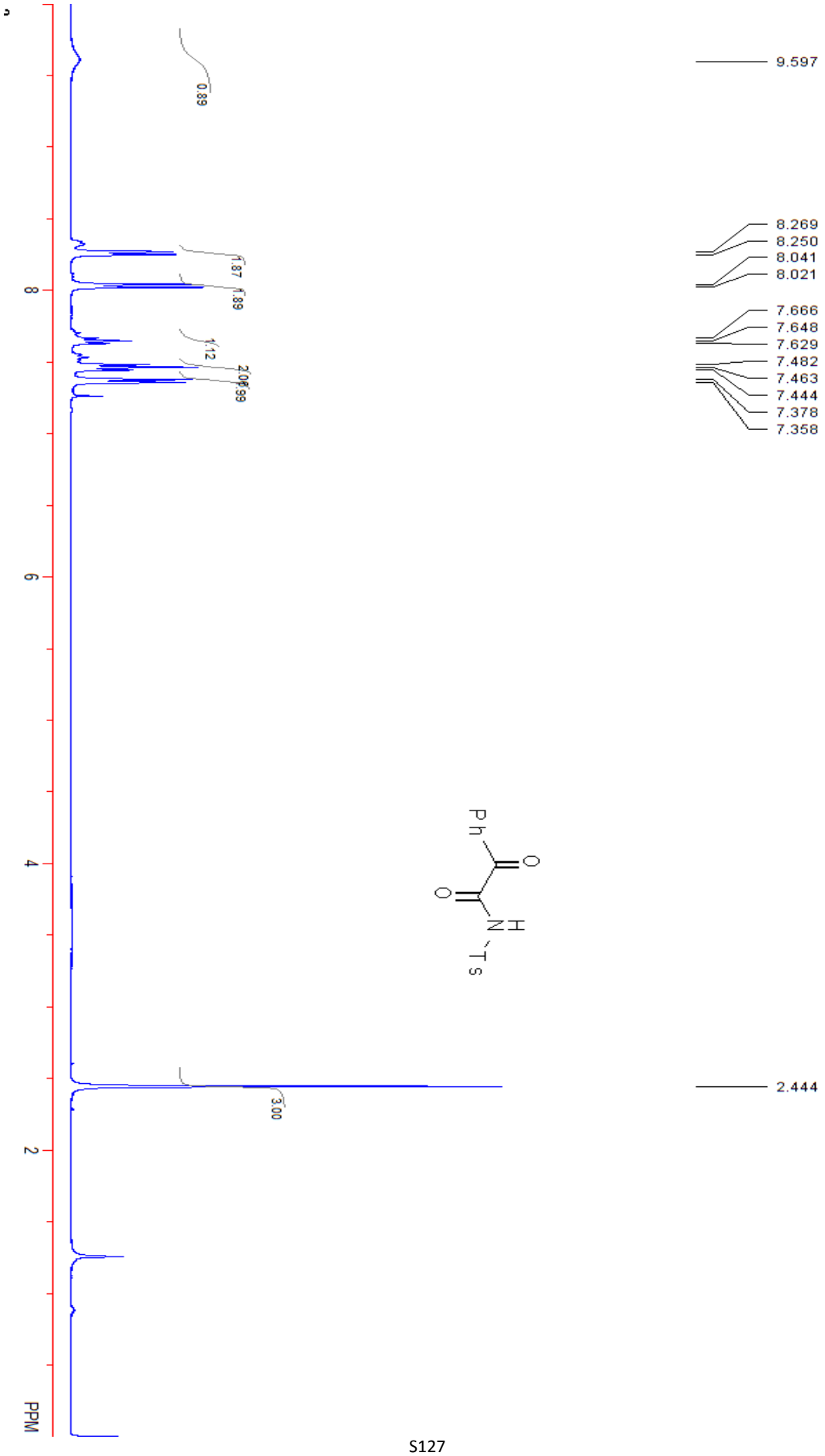
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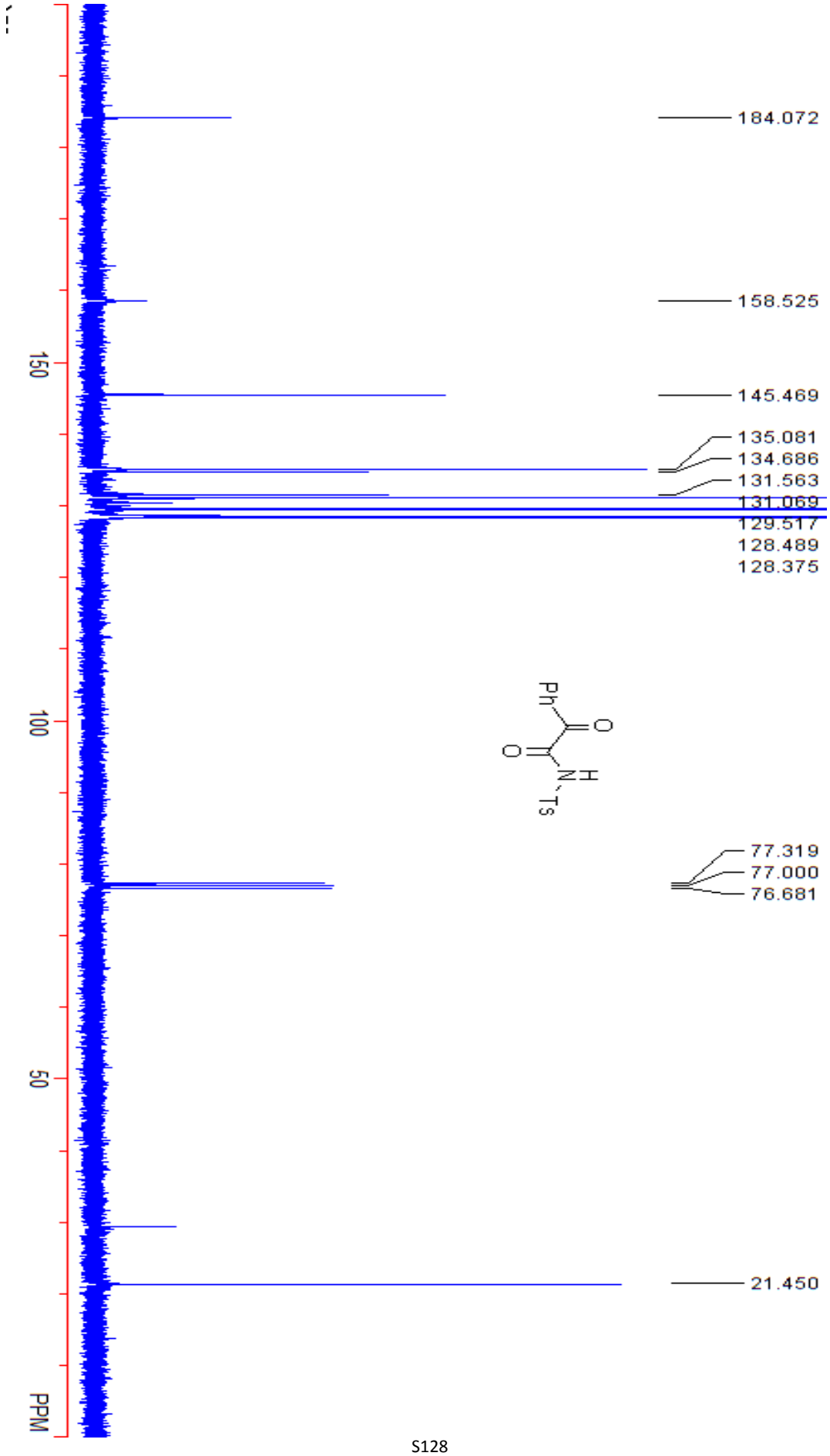
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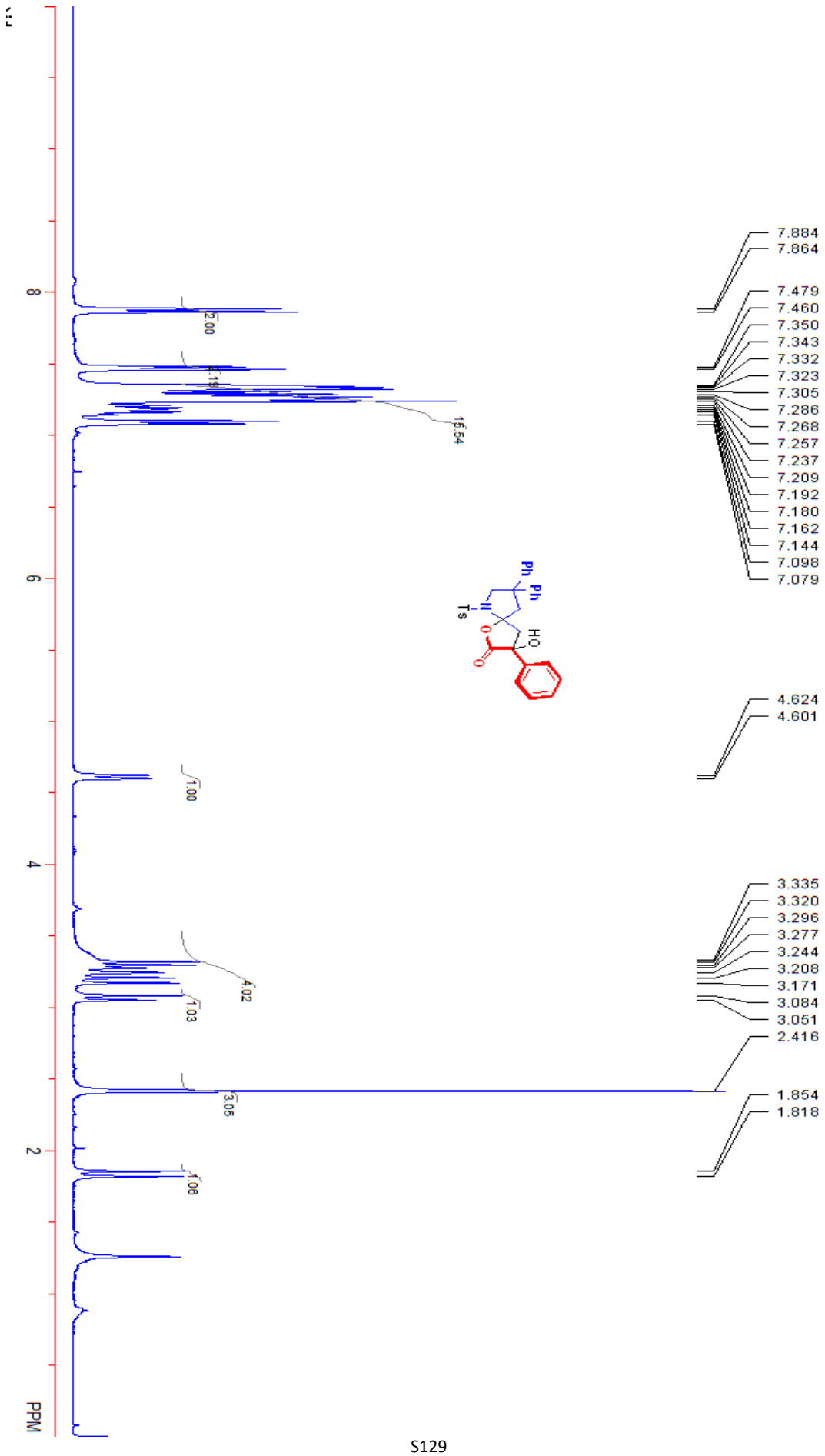
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3.171
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1.854
1.818
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2.18
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Ph
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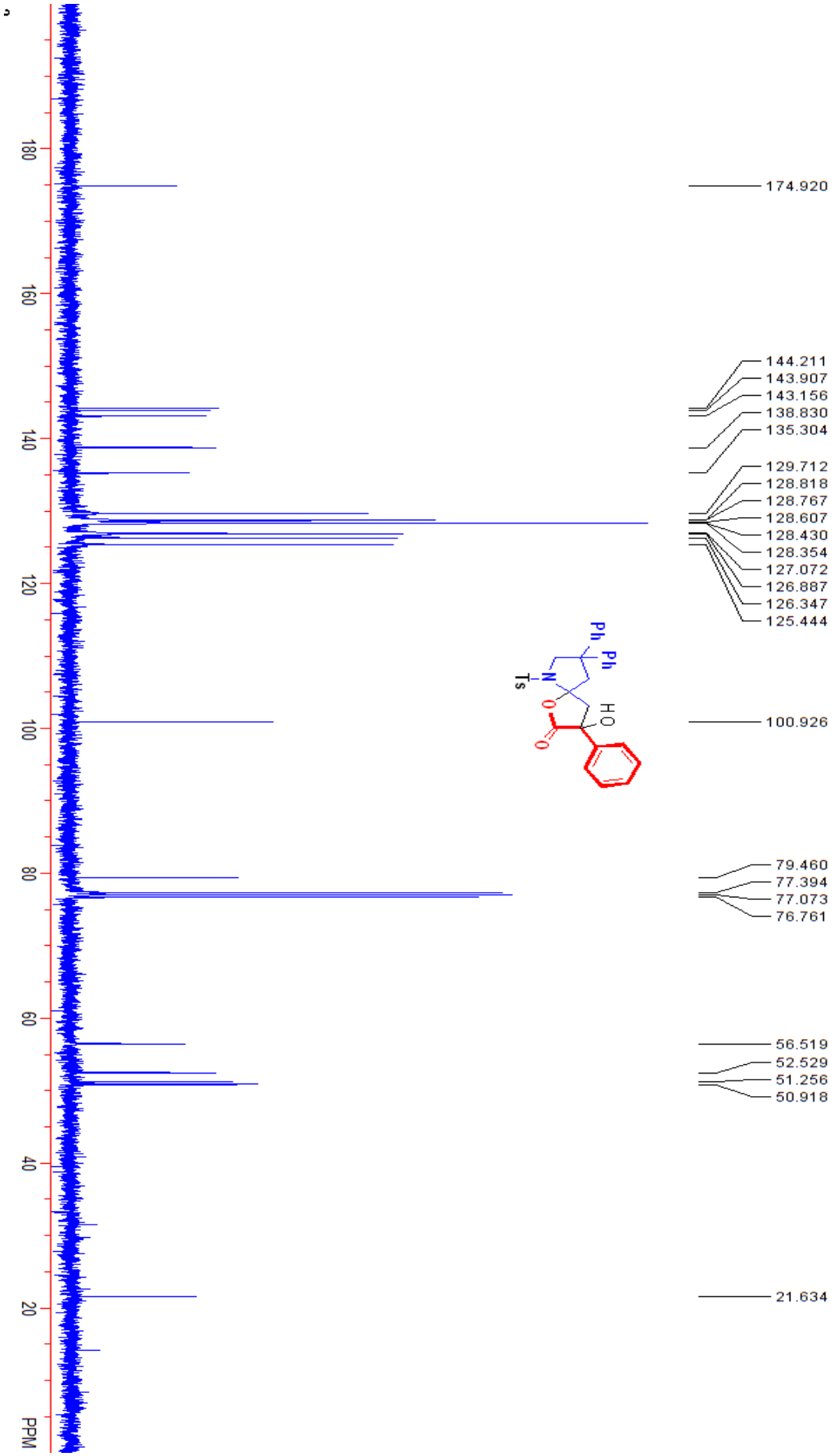
2
PPM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PPM
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