Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.


Supporting Information

for

# Pd-Catalyzed Intermolecular Enantioselective Hydroamination of Styenes Coupled with Alochol Oxidation

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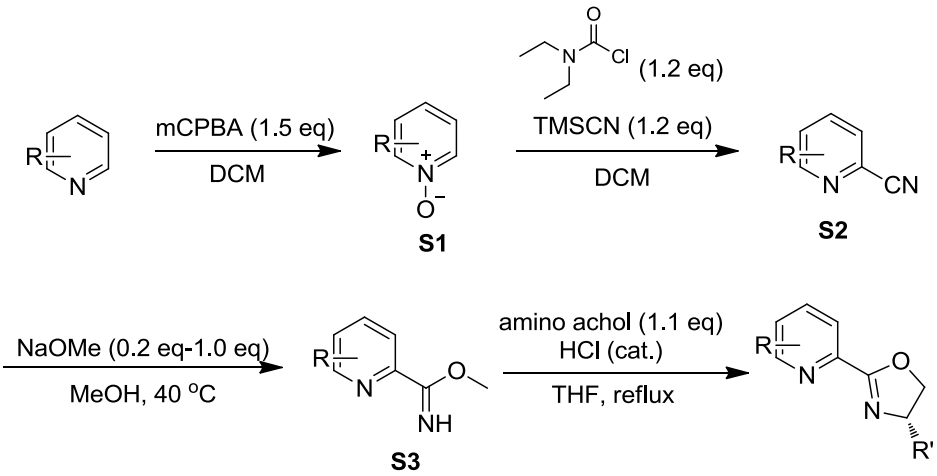
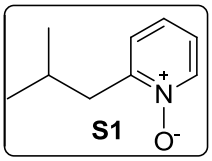
# 1. General

All reactions were run under $N_2$ in oven-dried Schlenk tubes. Palladium acetate was purchased from Strem Chemical. Other commercial reagents with high purity were purchased and used without further purification, unless otherwise noted. Reactions were monitored by thin-layer chromatography (TLC) carried out on 25 mm silica gel plates using UV light as a visualizing agent. $^{1}H$ and $^{13}C$ spectra were recorded on a Varian Mercury-400 MHz or an Agilent-400 MHz spectrometer. $CDCl_3$ was purchased from J&K. 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 $^{13}C$). High performance liquid chromatography was performed on Waters 2487-600E Series HPLC using DAICEL Chiralpak AD-H chiral column eluted with a mixture of hexane and isopropyl alcohol. Optical rotation was measured on a Rudolph-Autopol I. Flash column chromatography was performed on silica gel 60 (particle size 200-400 mesh ASTM, purchased from Yantai, China) and eluted with petroleum ether/ethyl acetate (PE/EA). Ethyl acetate was purified according to the procedure from "Purification of Laboratory Chemicals book".

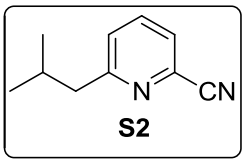
# 2. Synthesis of chiral pyridine-oxazoline ligands

Chiral pyridine-oxazoline ligands were synthesized according to the literatures and we take **L12** as an example for the operation in detail.[S1-3] Some new ligands with different substituents were characterized as below.

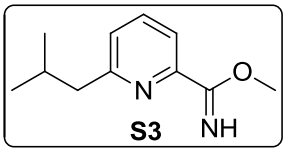





To a stirred solution of 2-isobutylpyridine (5.0 g, 37 mmol) in $CH_2Cl_2$ (120 mL), 85% m-CPBA (11.2 g, 63 mmol, 1.7 equiv) was added portion-wise at 0 ℃. The resulting mixture was stirred at room temperature for 12 h. After the reaction was completed, the reaction mixture was diluted with $CH_2Cl_2$, and solid $Na_2CO_3$ (4.0 equiv) was added. The mixture was stirred for 10 min, and filtered. The filtrate was dried over $Na_2SO_4$ and concentrated under vacuum. The crude residue was purified by silica gel chromatography to afford the pure product **S1** as a white solid (5.0 g, 90% yield). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 6.4 Hz, 1H), 7.22 – 7.08 (m, 3H), 2.78 (d, *J* = 6.8 Hz, 2H), 2.35 – 2.19 (m, 1H), 0.96 (d, *J* = 6.8 Hz, 6H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 151.7, 139.8, 126.6, 125.4, 123.4, 39.7, 25.3, 22.5; HRMS (ESI) calcd for $C_9H_{13}NO$ $[M+H]^+$: 152.1075, found: 152.1071.

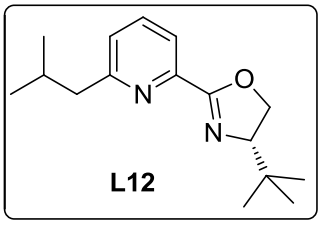



To a solution of **S1** (5.6 g, 37 mmol) in $CH_2Cl_2$ (100 mL) was added trimethylsilyl cyanide (TMSCN, 4.58 g, 46.3 mmol, 1.25 equiv). Then a solution of diethylcarbamide chloride (6.79 g, 46.3 mmol, 1.25 equiv) in $CH_2Cl_2$ (50 mL) was slowly added in 30 min, and the reaction mixture was stirred at room temperature for 24 h. After $K_2CO_3$ (100 mL, 10 % aq.) was added, the mixture was extracted several times with $CH_2Cl_2$. The combined organic phases was washed with water and then dried over $MgSO_4$. The organic phase was concentrated and purified by silica gel chromatography to afford the product **S2** as a white solid (5.16 g, 87% yield). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.72 (t, *J* = 7.6 Hz, 1H), 7.52 (d, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 7.6 Hz, 1H), 2.68 (d, *J* = 7.2 Hz, 2H), 2.18 – 2.03 (m, 1H), 0.91 (d, *J* = 6.4 Hz, 6H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 163.9, 136.9, 133.3, 127.0, 125.9, 117.5, 47.2, 29.1, 22.3; HRMS (ESI) calcd for $C_{10}H_{13}N_2$ $[M+H]^+$: 161.1073, found: 161.1075.




**S3**

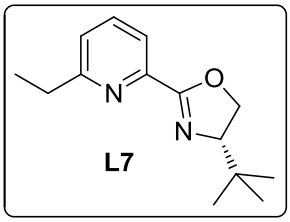
To a stirred solution of **S2** (4.8 g, 30 mmol) in MeOH (50 mL), NaOMe (540 mg, 10 mol) was added portion-wise under $N_2$. The mixture was stirred at 40 $^oC$ for 12 h. The solvent was removed under vacuum, and the residue was then dissolved in 50 mL of EtOAc and then washed with water and brine. The organic phase was separated and dried over $MgSO_4$. After removal of the solvent, the crude product **S3** was used in next step without further purification. Colorless oil, yield: 5.50 g (95%). $^1H$ NMR (400 MHz, $CDCl_3$) δ 9.19 (s, 1H), 7.69 – 7.56 (m, 2H), 7.15 (d, *J* = 6.4 Hz, 1H), 3.98 (s, 3H), 2.66 (d, *J* = 7.2 Hz, 2H), 2.14 (td, *J* = 13.6, 6.4 Hz, 1H), 0.91 (d, *J* = 6.4 Hz, 6H), $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 167.3, 161.2, 146.6, 137.1, 125.2, 118.0, 53.8, 47.2, 28.9, 22.4; HRMS (ESI) calcd for $C_{11}H_{17}N_2O$ $[M+H]^+$:193.1335, found: 193.1337.




**L12**

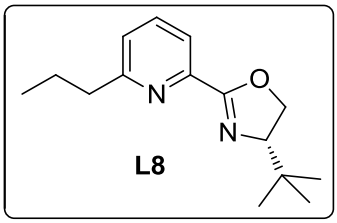
To a mixture of **S3** (1.47 g, 7.7 mmol, 1.0 equiv) and (S)-2-amino-1-*tert*-butylethanol (1.1 g, 9.2 mmol, 1.2 equiv) in dry THF (20 mL), HCl (1 drop, 37% aq.) was added, and the mixture was refluxed under argon atmosphere until all the starting materials were consumed with monitored by TLC. After the reaction was complete (12 h), the mixture was diluted with saturated $NaHCO_3$ (aq., 60 mL) and extracted with EtOAc (3 x 50 mL). After the removal of solvent under reduced pressure, the residue was purified by silica gel chromatography (1:1 hexanes/EtOAc) to afford product **L12** as a white solid (1.45 g 72% yield). $[\alpha]_D^{27.7}$ -55.90 (*c* 0.2, MeOH); $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.17 (d, *J* = 8.0 Hz, 1H), 4.42 (dd, J = 10.4, 8.8 Hz, 1H), 4.28 (t, *J* = 8.4 Hz, 1H), 4.07 (dd, *J* = 10.4, 8.8 Hz, 1H), 2.73 (dd, *J* = 13.6, 7.6 Hz, 1H), 2.69 (dd, *J* = 13.6, 7.6 Hz, 1H), 2.17 – 2.02 (m,

1H), 0.94 (s, 9H), 0.91 (d, *J* = 7.2 Hz, 3H), 0.89 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.9, 161.7, 146.6, 136.3, 125.20, 121.5, 76.2, 69.4, 47.4, 34.0, 29.0, 25.9, 22.34, 22.30; HRMS (ESI) calcd for $C_{16}H_{25}N_2O$ $[M+H]^+$: 261.1961, found: 261.1965.



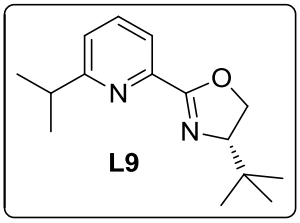
**L7**

Colrless oil, $[\alpha]_D^{27.5}$ -42.16 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.0 Hz, 1H), 7.67 (t, *J* = 7.6 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 1H), 4.45 (dd, *J* = 10.4, 8.8 Hz, 1H), 4.31 (t, *J* = 8.8 Hz, 1H), 4.09 (dd, *J* = 10.4, 8.0 Hz, 1H), 2.91 (q, *J* = 7.6 Hz, 2H), 1.31 (t, *J* = 7.6 Hz, 3H), 0.96 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 163.8, 162.8, 146.4, 136.8, 123.8, 121.5, 76.2, 69.4, 34.0, 31.5, 25.9, 14.2; HRMS (ESI) calcd for $C1_4H_{21}N_2O$ $[M+H]^+$: 233.1648, found: 233.1655.

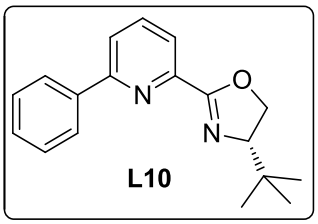


**L8**

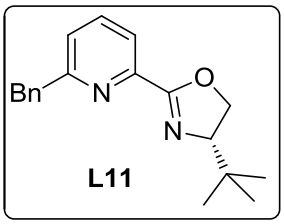
White solid, $[\alpha]_D^{27.5}$ -55.49 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.8 Hz, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 7.8 Hz, 1H), 4.45 (dd, *J* = 10.0, 8.8 Hz, 1H), 4.31 (t, *J* = 8.4 Hz, 1H), 4.09 (dd, *J* = 10.0, 8.4 Hz, 1H), 2.84 (dd, J = 8.8 Hz, 6.8 Hz, 2H), 1.75 (dq, *J* = 14.8, 7.2 Hz, 2H), 0.97 (t, *J* = 7.2 Hz, 3H), 0.96 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.8, 162.5, 146.5, 136.6, 124.5, 121.5, 76.2, 69.4, 40.4, 34.0, 25.9, 23.4, 13.9; HRMS (ESI) calcd for $C_{15}H_{23}N_2O$ $[M+H]^+$: 247.1805, found: 247.1808.




White solid, $[\alpha]_D^{27.6}$ -55.29 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.8 Hz, 1H), 7.69 (t, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 7.8 Hz, 1H), 4.45 (dd, *J* = 10.4, 8.8 Hz, 1H), 4.31 (t, *J* = 8.4 Hz, 1H), 4.09 (dd, *J* = 10.4, 8.4 Hz, 1H), 3.27 – 3.14 (m, 1H), 1.30 (d, *J* = 6.8 Hz, 6H), 0.96 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.8, 162.9, 146.1, 136.9, 121.8, 121.7, 76.2, 69.4, 36.6, 34.0, 25.9, 22.9, 22.8; HRMS (ESI) calcd for $C_{15}H_{23}N_2O$ $[M+H]^+$: 247.1805, found: 247.1807.

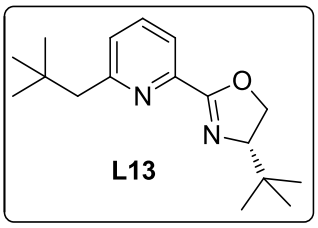



White solid, $[\alpha]_D^{27.6}$ -33.65 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 8.16 – 7.99 (m, 3H), 7.88 – 7.76 (m, 2H), 7.55 – 7.35 (m, 3H), 4.48 (dd, *J* = 10.4, 8.8 Hz, 1H), 4.35 (t, *J* = 8.4 Hz, 1H), 4.14 (dd, *J* = 10.4, 8.4 Hz, 1H), 0.99 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.9, 157.4, 147.0, 138.8, 137.2, 129.2, 128.7, 127.2, 122.5, 122.3, 76.3, 69.4, 34.0, 26.0; HRMS (ESI) calcd for $C_{18}H_{21}N_2O$ $[M+H]^+$: 281.1648, found: 281.1649.



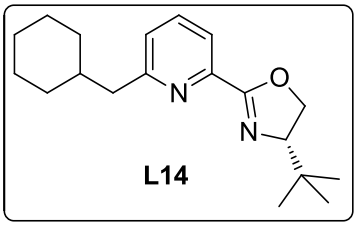

White soild, $[\alpha]_D^{27.7}$ -30.95 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.6 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.36 – 7.17 (m, 5H), 7.08 (d, *J* = 7.6 Hz, 1H), 4.46 (t, *J* = 9.6 Hz, 1H), 4.33 (t, *J* = 8.4 Hz, 1H), 4.27 (s, 2H), 4.11 (dd, *J* = 10.4, 8.4 Hz, 1H), 0.97 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.7, 161.3, 146.4, 139.0,

136.9, 129.3, 128.6, 126.5, 125.0, 121.8, 76.3, 69.4, 44.6, 34.0, 25.9; HRMS (ESI) calcd for $C_{19}H_{23}N_2O$ $[M+H]^+$: 295.1805, found: 295.1810.



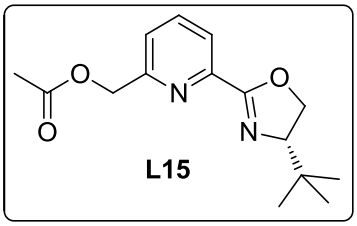

**L13**

White solid, $[\alpha]_D^{27.8}$ -55.79 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.6 Hz, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 7.6 Hz, 1H), 4.43 (dd, *J* = 10.4, 8.8 Hz, 1H), 4.29 (t, *J* = 8.0 Hz, 1H), 4.07 (dd, *J* = 10.4, 8.0 Hz, 1H), 2.80 (d, *J* = 12.4 Hz, 1H), 2.74 (d, *J* = 12.4 Hz, 1H), 0.95 (s, 9H), 0.94 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 163.1, 160.2, 146.3, 135.8, 126.3, 121.5, 76.1, 69.4, 51.6, 34.0, 32.1, 29.5, 25.9; HRMS (ESI) calcd for $C_{17}H_{27}N_2O$ $[M+H]^+$: 275.2118, found: 275.2125.

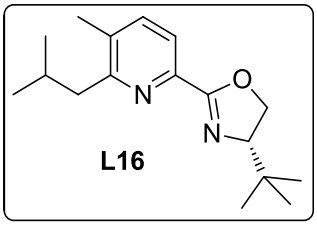



**L14**

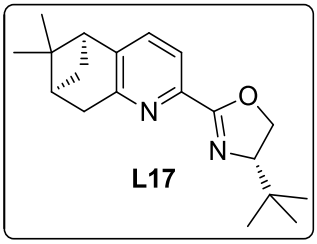
White solid, $[\alpha]_D^{27.8}$ -33.97 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.6 Hz, 1H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.18 (d, *J* = 7.6 Hz, 1H), 4.46 (dd, *J* = 10.4, 8.8 Hz, 1H), 4.32 (t, *J* = 8.8 Hz, 1H), 4.09 (dd, *J* = 10.4, 8.4 Hz, 1H), 2.75 (dd, *J* = 12.8, 7.6 Hz, 1H), 2.72 (dd, *J* = 13.2, 7.2 Hz, 1H), 1.82 – 1.72 (m, 1H), 1.71 – 1.59 (m, 5H), 1.25 – 1.11 (m, 3H), 1.05 – 0.94 (m, 2H), 0.97 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.9, 161.4, 146.6, 136.1, 125.4, 121.5, 76.2, 69.4, 46.2, 38.3, 34.0, 33.0, 26.5, 26.1, 25.9; HRMS (ESI) calcd for $C_{19}H_{29}N_2O$ $[M+H]^+$: 301.2274, found: 301.2278.




Yellow solid, $[\alpha]_D^{27.8}$ -33.31 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 7.6 Hz, 1H), 7.79 (t, *J* = 7.6 Hz, 1H), 7.45 (d, *J* = 7.6 Hz, 1H), 5.28 (s, 2H), 4.45 (dd, *J* = 10.4, 8.8 Hz, 1H), 4.31 (t, *J* = 8.8 Hz, 1H), 4.11 (dd, *J* = 10.4, 8.4 Hz, 1H), 2.16 (s, 3H), 0.96 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 170.5, 162.2, 156.0, 146.6, 137.3, 123.4, 123.3, 76.3, 69.4, 66.7, 34.0, 25.9, 20.9; HRMS (ESI) calcd for $C_{15}H_{21}N_2O_3$ $[M+H]^+$: 277.1547, found: 277.1548.

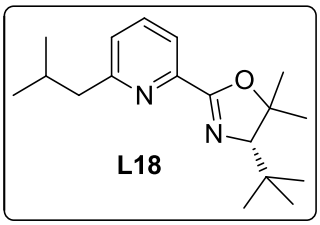



Colorless oil, $[\alpha]_D^{27.8}$ -47.46 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.0 Hz, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 4.41 (dd, *J* = 10.4, 8.8 Hz, 1H), 4.28 (t, *J* = 8.4 Hz, 1H), 4.05 (dd, *J* = 10.4, 8.4 Hz, 1H), 2.72 (d, *J* = 7.2 Hz, 2H), 2.33 (s, 3H), 2.14-2.11 (m, 1H), 0.94 (s, 9H), 0.92 (d, *J* = 7.2 Hz, 3H), 0.90 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 163.1, 160.2, 144.1, 137.8, 133.8, 121.6, 76.1, 69.3, 43.8, 34.0, 28.9, 25.9, 22.4, 19.4; HRMS (ESI) calcd for $C_{17}H_{27}N_2O$ $[M+H]^+$: 275.2118, found: 275.2122.




Colorless oil, $[\alpha]_D^{28.0}$ -133.22 (*c* 0.2, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 1H), 4.41 (dd, *J* = 10.0, 8.8 Hz, 1H), 4.28 (t, *J* = 8.4 Hz, 1H), 4.07 (dd, *J* = 10.0, 8.4 Hz, 1H), 3.18 (d, *J* = 2.8 Hz, 2H), 2.78 (t, *J* = 5.6

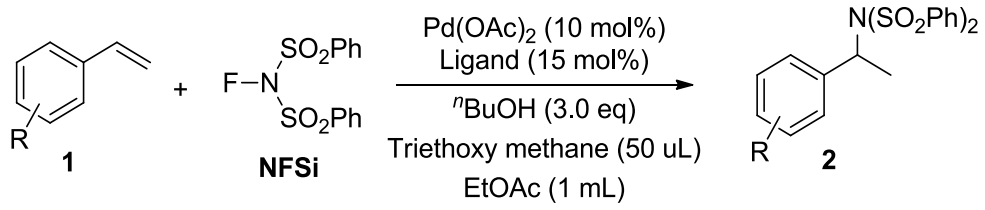
Hz, 1H), 2.71 – 2.62 (m, 1H), 2.37 – 2.33 (m, 1H), 1.38 (s, 3H), 1.23 (d, *J* = 9.6 Hz, 1H), 0.94 (s, 9H), 0.61 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.8, 157.1, 144.5, 144.0, 133.3, 121.2, 76.3, 69.2, 46.6, 40.0, 39.4, 36.6, 34.0, 31.6, 25.9, 21.2; HRMS (ESI) calcd for $C_{19}H_{27}N_2O$ $[M+H]^+$: 299.2118, found: 299.2120.




**L18**

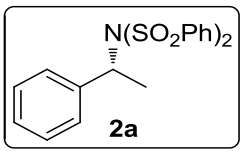
Colorless oil, $[\alpha]_D^{27.9}$ -8.80 (*c* 0.2, MeOH); $^1$H NMR (400 MHz, $CDCl_3$) δ 7.6 (d, *J* = 7.8 Hz, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.14 (d, *J* = 8.0 Hz, 1H), 3.59 (s, 1H), 2.73 (dd, *J* = 13.2, 7.6 Hz, 1H), 2.69 (dd, *J* = 13.2, 7.6 Hz, 1H), 2.14 – 2.04 (m, 1H), 1.54 (s, 3H), 1.50 (s, 3H), 1.09 (s, 9H), 0.89 (d, *J* = 7.2 Hz, 3H), 0.87 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 161.7, 147.4, 136.1, 124.9, 121.3, 88.5, 82.9, 47.3, 34.1, 30.7, 28.9, 27.8, 23.2, 22.4, 22.3; HRMS (ESI) calcd for $C_{18}H_{29}N_2O$ $[M+H]^+$: 289.2274, found: 289.2282.

## 3. General Procedure for Pd-catalyzed hydroamination of styrenes.

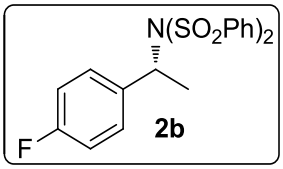



To an oven-dried Schlenk tube, $Pd(OAc)_2$ (4.4 mg, 0.01 mmol), liagnd L13 (7.8 mg，0.015 mmol), and NFSi (175 mg, 0.6 mmol) were dissolved in EtOAc(1 mL) under $N_2$ atmosphere. Then styrene (0.2 mmol), n-butyl alcohol (44.5 mg, 0.6 mmol) and triethyl orthoformate (50 μL, 3 mmol) were added subsequently. The resulting mixture was stirred at 30 $^o$C, and monitored by TLC. After the styrene was consumed, the solvent was removed and the residue was purified by silca gel chromatography to

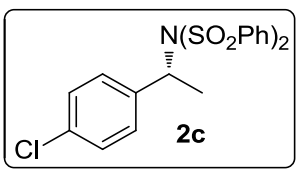
give the product (See Table 1).




White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.71 (bs, 4H), 7.56 (t, *J* = 7.6 Hz, 2H), 7.46 – 7.40 (m, 6H), 7.30 – 7.27 (m, 3H), 5.63 (q, *J* = 7.6 Hz, 1H), 1.65 (d, *J* = 7.6 Hz, 3H); $^{13}$C NMR (100MHz, $CDCl_3$) δ 140.4, 137.3, 133.5, 128.8, 128.7, 128.2, 128.1, 128.0, 60.3, 18.0; IR cm$^{-1}$: 1449, 1349, 1170, 1083, 959, 878, 757, 583; HRMS (ESI) calcd for $C_{10}H_{19}NO_4S_2Na$ $[M+Na]^+$: 424.0648, found: 424.0648; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 9/1, flow 0.7 mL/min, detection at 214 nm) retention time = 14.0 min (minor) and 19.8 min (major).

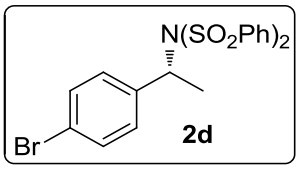



White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.75 (bs, 4H), 7.60 (t, *J* = 7.2 Hz, 2H), 7.48-7.41 (m, 6H), 6.96 (t, *J* = 8.8 Hz, 2H), 5.59 (q, *J* = 7.2 Hz, 1H), 1.64 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 163.4 (d, *J* = 246.2 Hz), 140.4, 133.6, 133.3 (d, *J* = 3.8 Hz), 130.6 (d, *J* = 8.2 Hz), 128.8, 128.1, 114.9 (d, *J* = 21.6 Hz), 59.7, 18.2; HRMS (EI) calcd for $C_{19}H_{15}FNO_4S_2$ $[M-CH_3]$: 404.0427, found: 404.0422; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 9/1, flow 0.7 mL/min, detection at 214 nm) retention time = 12.9 min (minor) and 16.5 min (major).

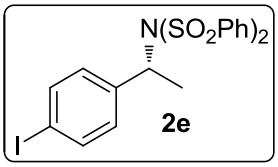



White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.75 (bs, 4H), 7.60 (t, *J* = 7.6 Hz, 2H), 7.5 (t, *J* = 7.6 Hz, 4H), 7.4 (d, *J* = 8.4 Hz, 2H), 7.2 (d, *J* = 8.4 Hz, 2H), 5.58 (q, *J* = 7.2 Hz,

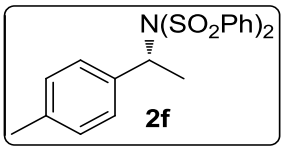
1H), 1.63 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.3, 136.0, 134.0, 133.6, 130.2, 128.8, 128.3, 128.1, 59.5, 18.0; HRMS (EI) calcd for $C_{19}H_{15}ClNO_4S_2$ [M-$CH_3$]: 420.0131, found: 420.0130; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 7/3, flow 0.7 mL/min, detection at 214 nm) retention time = 7.4 min (minor) and 9.9 min (major).



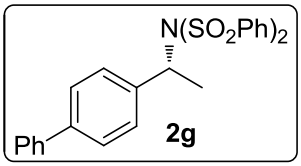

White solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.61 (bs, 4H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 4H), 7.24 (d, *J* = 8.8 Hz, 2H), 7.15 (d, *J* = 8.8 Hz, 2H), 5.56 (q, *J* = 7.2 Hz, 1H), 1.62 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.3, 136.5, 133.6, 131.1, 130.5, 128.8, 128.1, 122.1, 59.6, 17.9; HRMS (EI) calcd for $C_{20}H_{18}BrNO_4S_2$ [M]: 478.9861, found: 478.9868; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 8/2, flow 0.7 mL/min, detection at 214 nm) retention time = 9.5 min (minor) and 14.2 min (major).



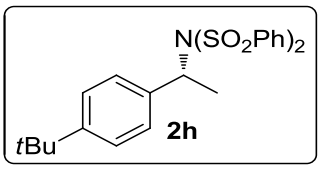

White solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.72 (bs, 4H), 7.60 – 7.55 (m, 4H), 7.44 (t, *J* = 7.6 Hz, 4H), 7.14 (d, *J* = 8.2 Hz, 2H), 5.54 (q, *J* = 7.2 Hz, 1H), 1.60 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.3, 137.2, 137.1, 133.7, 130.7, 128.9, 128.1, 93.9, 59.7, 17.9; HRMS (ESI) calcd for $C_{20}H_{22}IN_2O_4S_2$ $[M+NH_4]^+$: 545.0060, found: 545.0066; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 7/3, flow 0.7 mL/min, detection at 214 nm) retention time = 8.1 min (minor) and 11.3 min (major).




White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.73 (bs, 4H), 7.57 (t, *J* = 7.2 Hz, 2H), 7.43 (t, *J* = 7.6 Hz, 4H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 5.61 (q, *J* = 7.2 Hz, 1H), 2.36 (s, 3H), 1.65 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.6, 137.8, 134.4, 133.4, 128.8, 128.7, 128.5, 128.2, 60.3, 21.1, 18.1; HRMS (ESI) calcd for $C_{20}H_{21}NO_4S_2Na$ $[M+Na]^+$: 438.0804, found: 438.0803; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 9/1, flow 0.7 mL/min, detection at 214 nm) retention time = 12.4 min (minor) and 21.1 min (major).

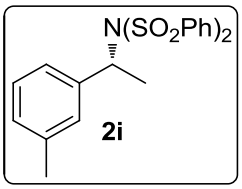



White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.78 (s, 4H), 7.66 – 7.54 (m, 4H), 7.54 – 7.35 (m, 11H), 5.72 (q, *J* = 7.2 Hz, 1H), 1.72 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.9, 140.5, 136.4, 133.5, 129.3, 128.9, 128.8, 128.2, 127.6, 127.1, 126.7, 60.2, 18.2; HRMS (ESI) calcd for $C_{26}H_{27}N_2O_4S_2$ $[M+NH_4]^+$: 495.1407, found: 495.1408; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 7/3, flow 0.7 mL/min, detection at 214 nm) retention time = 8.3 min (minor) and 10.1 min (major).

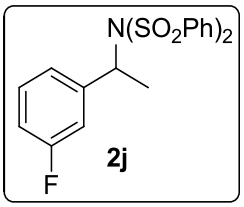



Colorless oil; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.68 (bs, 4H), 7.56 (t, *J* = 11.2 Hz, 2H), 7.43 – 7.38 (m, 6H), 7.31 (d, *J* = 8.4 Hz, 2H), 5.66 (q, *J* = 7.2 Hz, 1H), 1.67 (d, *J* = 7.2 Hz, 3H), 1.36 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 151.2, 140.6, 134.2, 133.5, 128.7, 128.2, 125.0, 60.3, 34.6, 31.4, 18.2; HRMS (EI) calcd for $C_{24}H_{27}NO_4S_2$ [M]: 457.1381, found: 457.1380; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol =

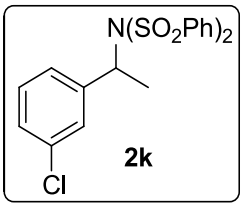
9/1, flow 0.7 mL/min, detection at 214 nm) retention time = 9.6 min (minor) and 10.7 min (major).



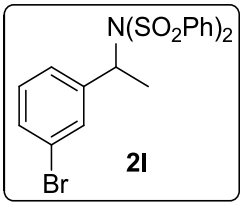

White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.73 (bs, 4H), 7.57 (t, *J* = 7.2 Hz, 2H), 7.43 (t, *J* = 7.2 Hz, 4H), 7.26 (d, *J* = 7.2 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.15 (s, 1H), 7.09 (d, *J* = 7.2 Hz, 1H), 5.62 (q, *J* = 7.2 Hz, 1H), 2.23 (s, 3H), 1.65 (d, *J* = 7.2 Hz, 3H). $^{13}$C NMR (100MHz, $CDCl_3$) δ 140.5, 137.6, 137.2, 133.4, 129.6, 128.6, 128.3, 128.2, 128.0, 125.6, 60.3, 21.3, 18.1; HRMS (ESI) calcd for $C_{21}H_{21}NO_4S_2Na$ $[M+Na]^+$: 438.0804, found: 438.0823; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 7/3, flow 0.7 mL/min, detection at 214 nm) retention time = 7.1 min (minor) and 9.0 min (major).



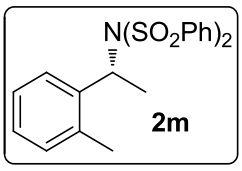

White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.76 (s, 4H), 7.60 (t, *J* = 7.6 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 4H), 7.28 – 7.20 (m, 2H), 7.12 (d, *J* = 10.4 Hz, 1H), 7.06 – 6.90 (m, 1H), 5.57 (q, *J* = 7.2 Hz, 1H), 1.64 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.6 (d, *J* = 245.2 Hz), 140.3, 140.1 (d, *J* = 7.6 Hz), 133.7, 129.5 (d, *J* = 8.5 Hz), 128.8, 128.2, 124.3 (d, *J* = 3.2 Hz), 115.8 (d, *J* = 22.0 Hz), 114.9 (d, *J* = 20.4 Hz), 59.6, 59.6, 18.0; HRMS (ESI) calcd for $C2_0H_{22}FN_2O_4S_2$ $[M+NH_4]^+$: 437.1000, found: 437.1002; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 7/3, flow 0.7 mL/min, detection at 214 nm) retention time = 7.8 min (minor) and 9.6 min (major).




White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.76 (bs, 4H), 7.61 (t, *J* = 7.6 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 4H), 7.35 – 7.26 (m, 3H), 7.24 – 7.16 (m, 1H), 5.56 (q, *J* = 7.2 Hz, 1H), 1.64 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 139.6, 134.1, 133.8, 129.4, 129.0, 128.9, 128.2, 128.1, 126.7, 59.6, 18.0; HRMS (ESI) calcd for $C_{20}H_{22}ClN_2O_4S_2$ $[M+NH_4]^+$: 453.0704, found: 453.0703; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 7/3, flow 0.7 mL/min, detection at 214 nm) retention time = 7.7 min (minor) and 9.9 min (major).

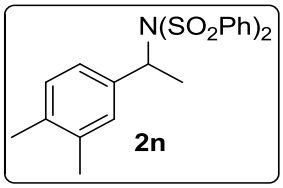



White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.77 (bs, 4H), 7.61 (t, *J* = 7.6 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 5H), 7.44 – 7.34 (m, 2H), 7.15 (t, *J* = 8.0 Hz, 1H), 5.55 (q, *J* = 7.2 Hz, 1H), 1.63 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 139.8, 133.8, 131.8, 131.1, 129.7, 128.9, 128.2, 127.1, 122.3, 59.5, 18.0; HRMS (ESI) calcd for $C_{20}H_{22}BrN_2O_4S_2$ $[M+NH_4]^+$: 497.0199, found: 497.0195; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 7/3, flow 0.7 mL/min, detection at 214 nm) retention time = 7.6 min (minor) and 10.1 min (major).




White solid; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.73 (bs, 4H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.36 (t, *J* = 7.6 Hz, 4H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.12 (t, *J* =

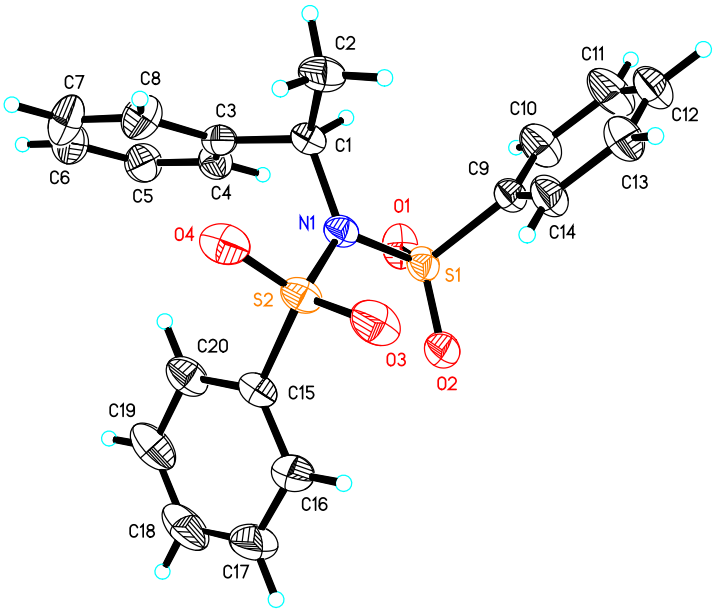
7.2 Hz, 1H), 6.66 (d, *J* = 8.0 Hz, 1H), 5.98 (q, *J* = 7.2 Hz, 1H), 1.99 (s, 3H), 1.89 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.7, 139.0, 133.7, 133.1, 130.2, 130.0, 128.5, 128.4, 128.1, 125.7, 58.2, 19.9, 19.4; HRMS (ESI) calcd for $C_{21}H_{21}NO_4S_2Na$ $[M+Na]^+$: 438.0804, found: 438.0811; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 9/1, flow 0.7 mL/min, detection at 214 nm) retention time = 24.9 min (minor) and 33.8 min (major).




**2n**

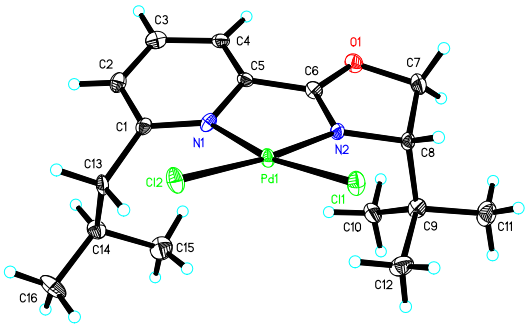
White solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.75 (br, 4H), 7.58 (t, *J* = 7.6 Hz, 2H), 7.42 (t, *J* = 7.6 Hz, 4H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.08 – 7.05 (m, 2H), 5.63 (q, *J* = 7.2 Hz, 1H), 2.26 (s, 3H), 2.12 (s, 3H), 1.67 (d, *J* = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.6, 136.4, 136.1, 134.7, 133.4, 130.2, 129.2, 128.7, 128.2, 125.9, 60.4, 19.7, 19.5, 18.2; HRMS (ESI) calcd for $C_{22}H_{27}N_2O_4S_2$ $[M+NH_4]^+$: 447.1407, found: 447.1409; HPLC (DAICEL Chiralpak AD-H, hexane/ isopropanol = 9/1, flow 0.7 mL/min, detection at 214 nm) retention time = 10.5 min (minor) and 15.0 min (major).

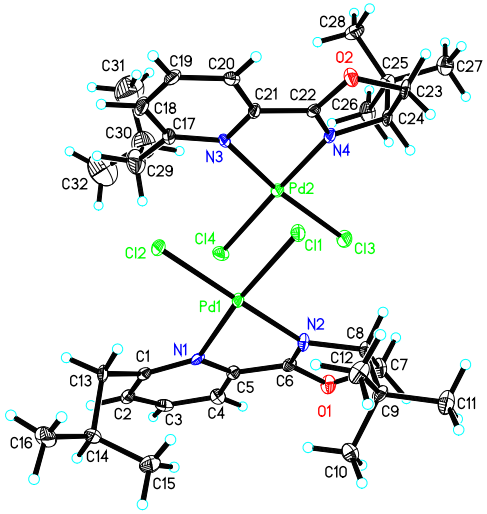
**X-Ray structure of enantiopure 2a and (L12)$PdCl_2$**




| Crystal data and structure refinement for **2a** | |
|---|---|
| Identification code | cd213489 |
| Empirical formula | C20 H19 N O4 S2 |
| Formula weight | 401.48 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| space group | P2(1) |
| Unit cell dimensions | a = 8.4228(10) Å α = 90 ° |
| | b = 13.7083(17) Å β= 104.508(2) ° |
| | c = 8.6714(11) Å γ = 90° |
| Volume | 969.3(2) Å $^3$ |
| Z | 2 |
| Calculated density | 1.376 Mg/m$^3$ |
| Absorption coefficient | 0.300 mm$^{-1}$ |
| F(000) | 420 |

| | |
|---|---|
| Crystal size | 0.212 x 0.175 x 0.114 $mm^3$ |
| Theta range for data collection | 2.43 to 26.00 ° |
| Limiting indices | -10≤h≤10, -15≤k≤16, -10≤l≤10 |
| Reflections collected | 5776 |
| unique | 3147 [R(int) = 0.0429] |
| Completeness to theta = 26.00 ° | 99.9 % |
| Absorption correction | Empirical |
| Max. and min. transmission | 1.00000 and 0.46327 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 3147 / 1 / 246 |
| Goodness-of-fit on $F^2$ | 1.059 |
| Final R indices [I>2sigma(I)] | $R_1$ = 0.0426, $wR_2$ = 0.1018 |
| R indices (all data) | $R_1$ = 0.0450, $wR_2$ = 0.1040 |
| Absolute structure parameter | 0.05(7) |
| Extinction coefficient | 0.017(3) |
| Largest diff. peak and hole | 0.434 and -0.328 e. $Å^{-3}$ |



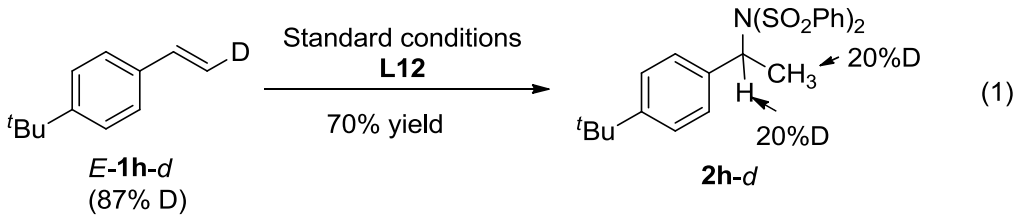



**X-ray of (L12)$PdCl_2$**

| Crystal data and structure refinement for mo_dm14620_0m. | | |
|---|---|---|
| Identification code | (**L12**)$PdCl_2$ | |
| Empirical formula | C16 H24 Cl2 N2 O Pd | |
| Formula weight | 437.67 | |
| Temperature | 130 K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| space group | P 1 21 1 | |
| Unit cell dimensions | a = 12.1876(18) Å | α = 90° |
| | b = 9.8204(14) Å | β = 96.796(3) ° |
| | c =15.854(2) Å | γ = 90° |
| Volume | 1884.2(5) $Å^3$ | |
| Z | 4 | |
| Calculated density | 11.543 $Mg/m^3$ | |
| Absorption coefficient | 1.271 $mm^{-1}$ | |
| F(000) | 888 | |

| | |
|---|---|
| Crystal size | 0.18 x 0.03 x 0.02 $mm^3$ |
| Theta range for data collection | 1.293 to 30.495 ° |
| Limiting indices | $-14 \leq h \leq 17$, $-13 \leq k \leq 13$, $-22 \leq 22$ |
| Reflections collected | 18529 |
| unique | 10955 [R(int) = 0.0640] |
| Completeness to theta = 26.00 ° | 99.9 % |
| Absorption correction | Empirical |
| Max. and min. transmission | 1.00000 and 0.46327 |
| Refinement method | Semi-empirical from equivalents |
| Data / restraints / parameters | 10955 / 40 / 407 |
| Goodness-of-fit on $F^2$ | 1.023 |
| Final R indices [$I>2\sigma(I)$] | $R_1 = 0.0638$, $wR_2 = 0.1180$ |
| R indices (all data) | $R_1 = 0.0974$, $wR_2 = 0.1327$ |
| Absolute structure parameter | -0.03(4) |
| Extinction coefficient | n/a |
| Largest diff. peak and hole | 1.263 and -1.046 $e.Å^{-3}$ |

## 4. Deuterium labeled experiments




The deuterium labeled styrene *E*-**1h**-*d* was synthesized and subjected to the standard condition, and the hydroamination product **2h**-*d* was obtained as a mixture of isomers with the deuterium incorporation on both $\alpha$ and β carbon (eq 1). The $^{1}$H NMR spectrum of **2h**-*d* was shown in Figure S1.

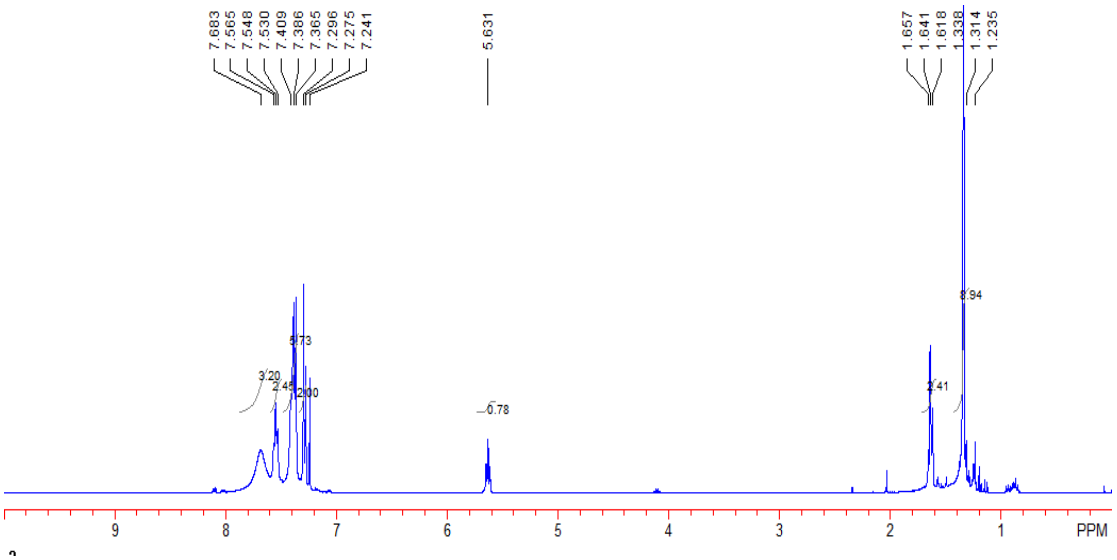
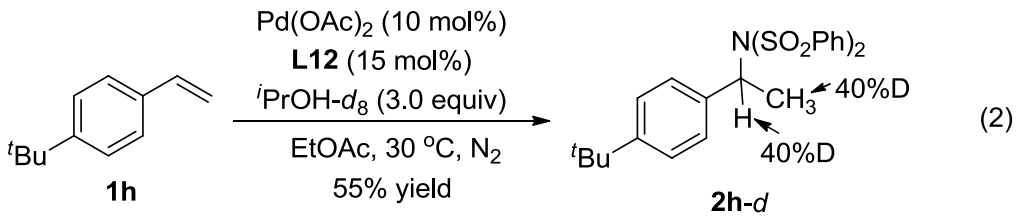



**Figure S1**. $^{1}$H NMR spectrum of **2h**-*d* obtained in eq 1




When styrene *E*-**1h**-*d* was subjected to the standard condition with iPrOH-$d_8$, the same product **2h**-*d* was given (eq 2), however with 40% deuterium incorporation on both $\alpha$ and β carbon (eq 2). The $^1$H NMR spectrum of **2h**-*d* was shown in Figure S2.

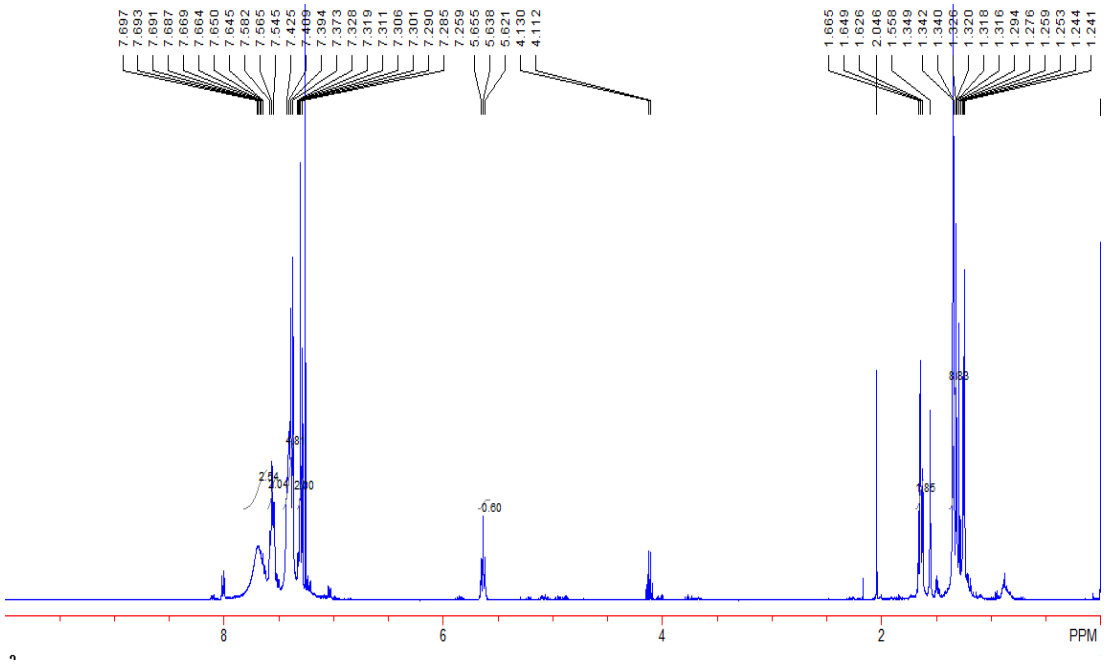



**Figure S2**. $^1$H NMR spectrum of **2h**-*d* obtained in eq 2

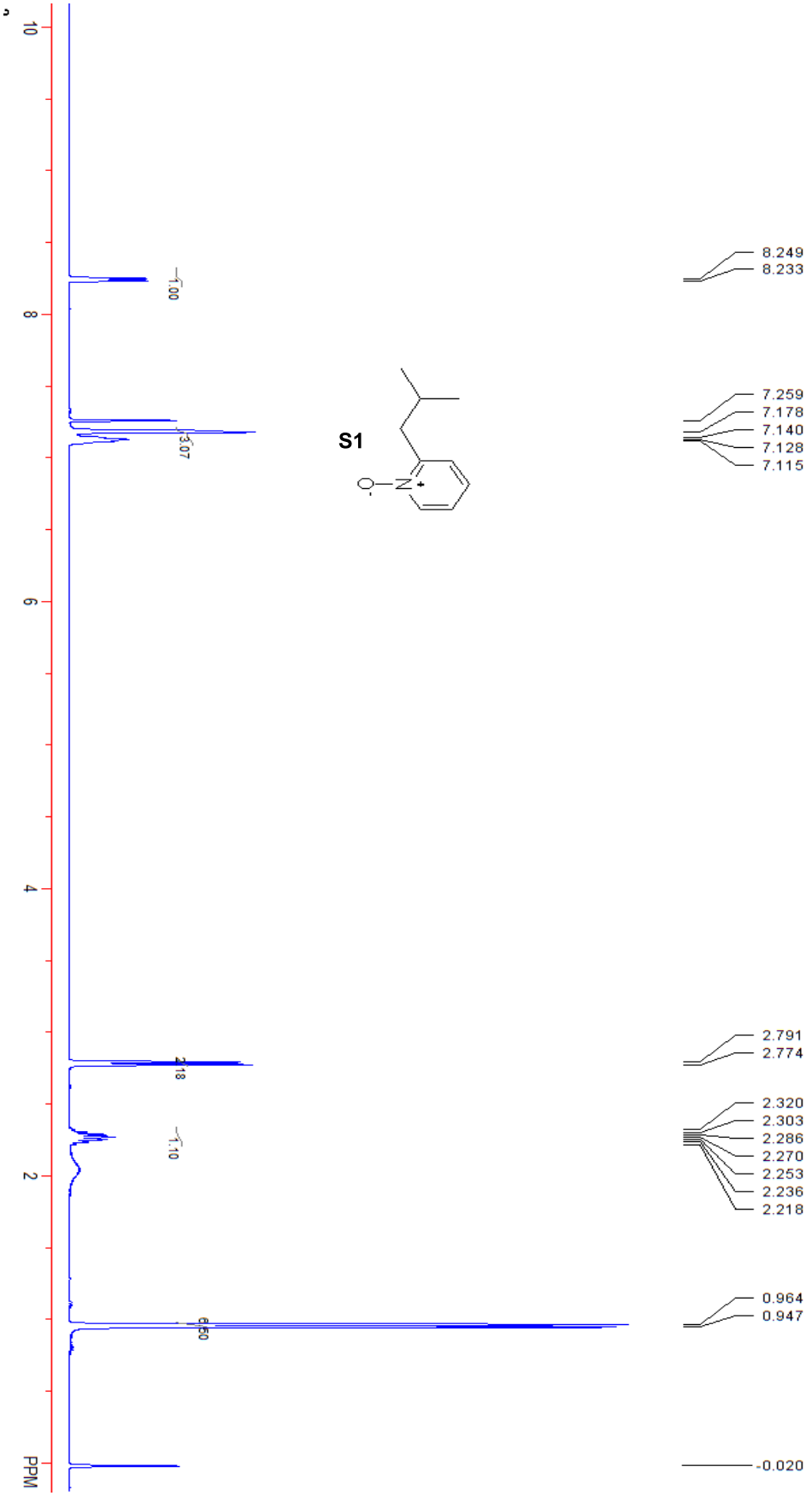
## Reference

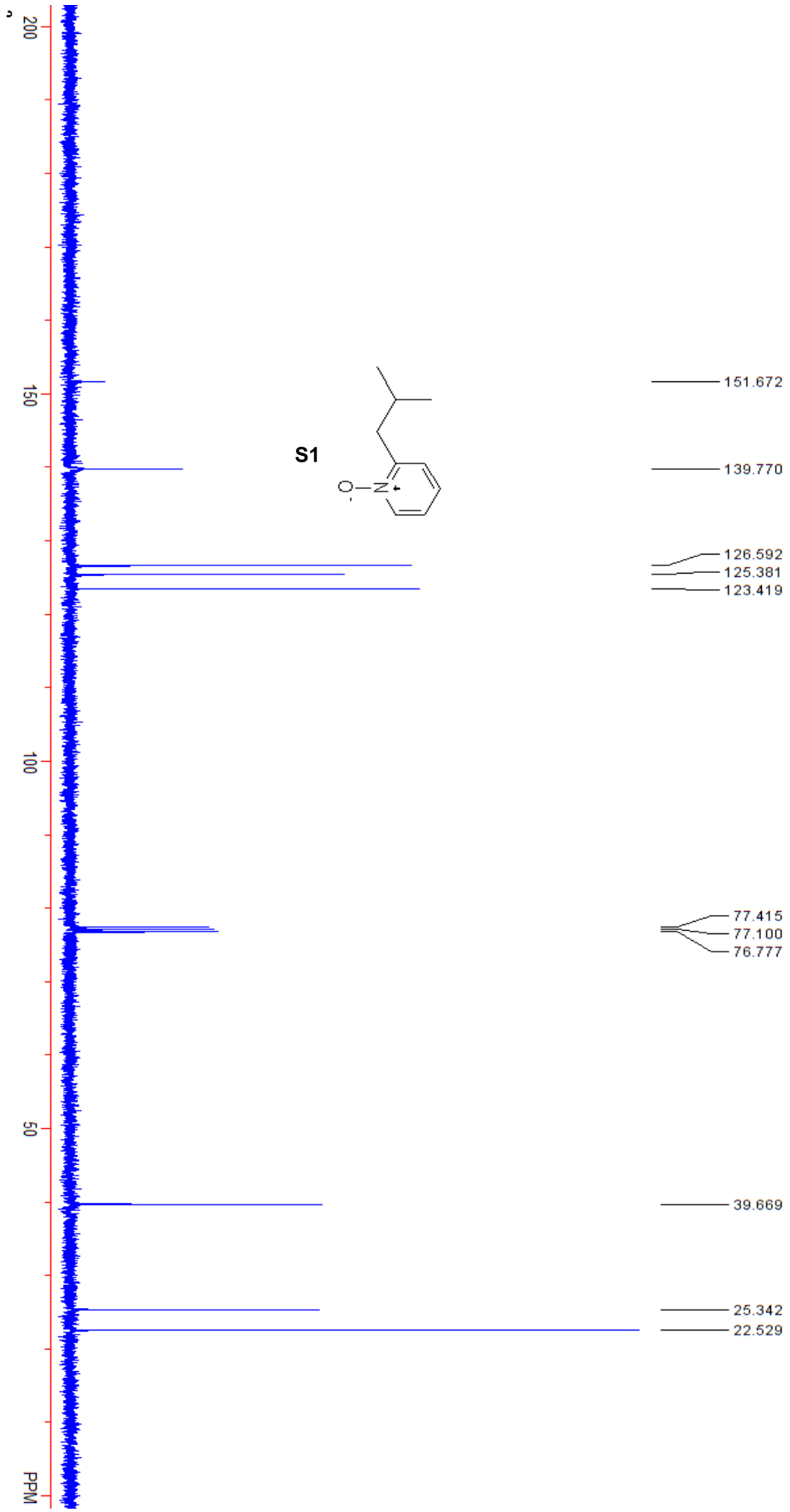
[S1] Shuman, R.T.; Ornstein, P. L.; Paschal, J. W.; Gesellchen, P. D. An improved synthesis of homoproline and derivatives. *J. Org. Chem*. **1990**, *55*, 738-741.

[S2] Kikushima, K.; Holder, J. C.; Gatti, M.; Stoltz, B. M. Palladium-catalyzed asymmetric conjugate addition of arylboronic acids to five-, six-, and seven-membered β-substituted cyclic enones: Enantioselective construction of all-carbon quaternary stereocenters. *J. Am. Chem. Soc*. **2011**, *133*, 6902–6905.

[S3] Werner, E. W.; Mei, T.-S.; Burckle, A. J.; Sigman, M. S. Enantioselective heck arylations of acyclic alkenyl alcohols using a redox-relay strategy. *Science*, **2012**, *338*, 1455-1458.

# 7. NMR spectra of ligands and products

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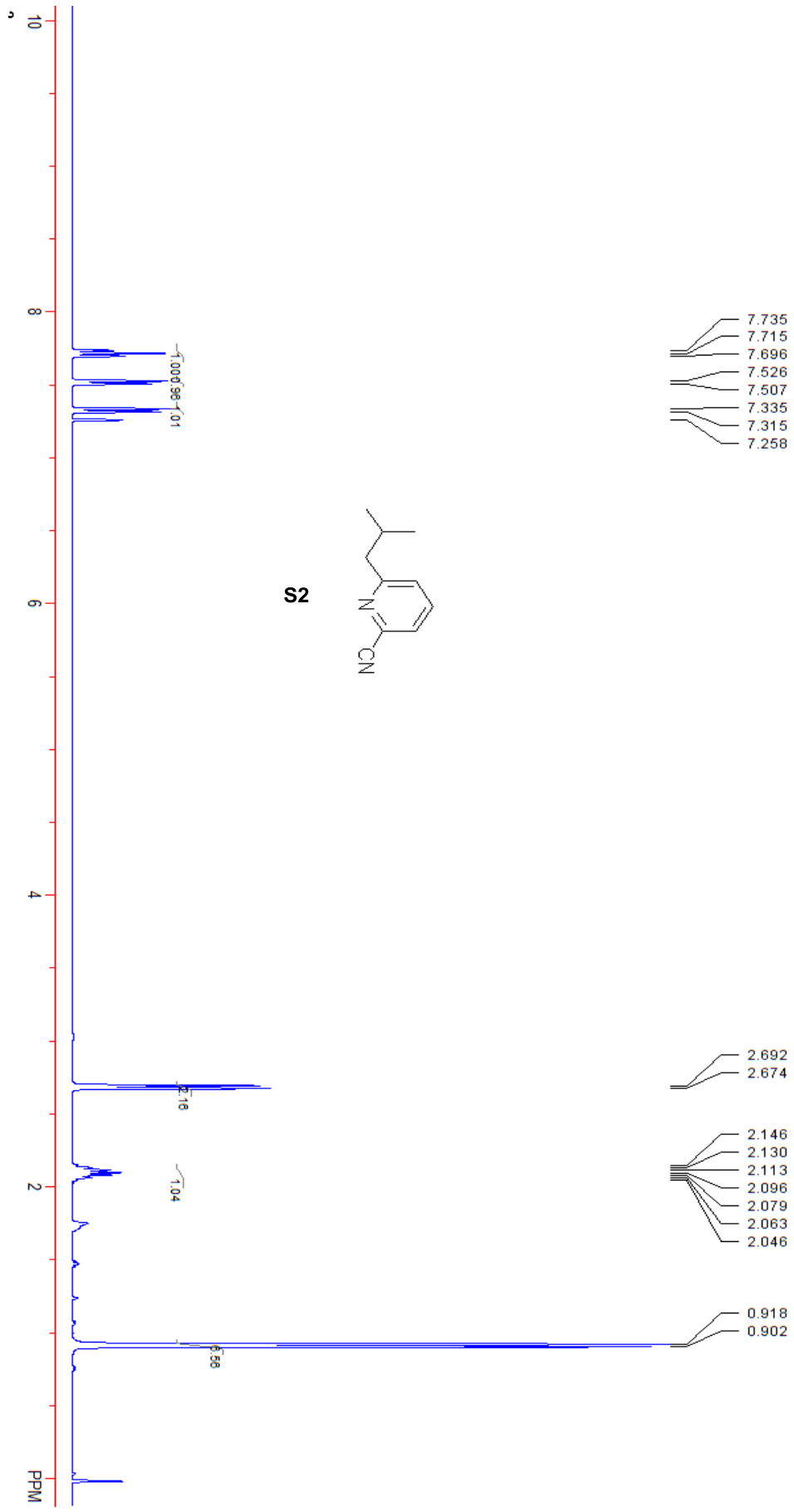
77.100
76.777
39.669
25.342
22.529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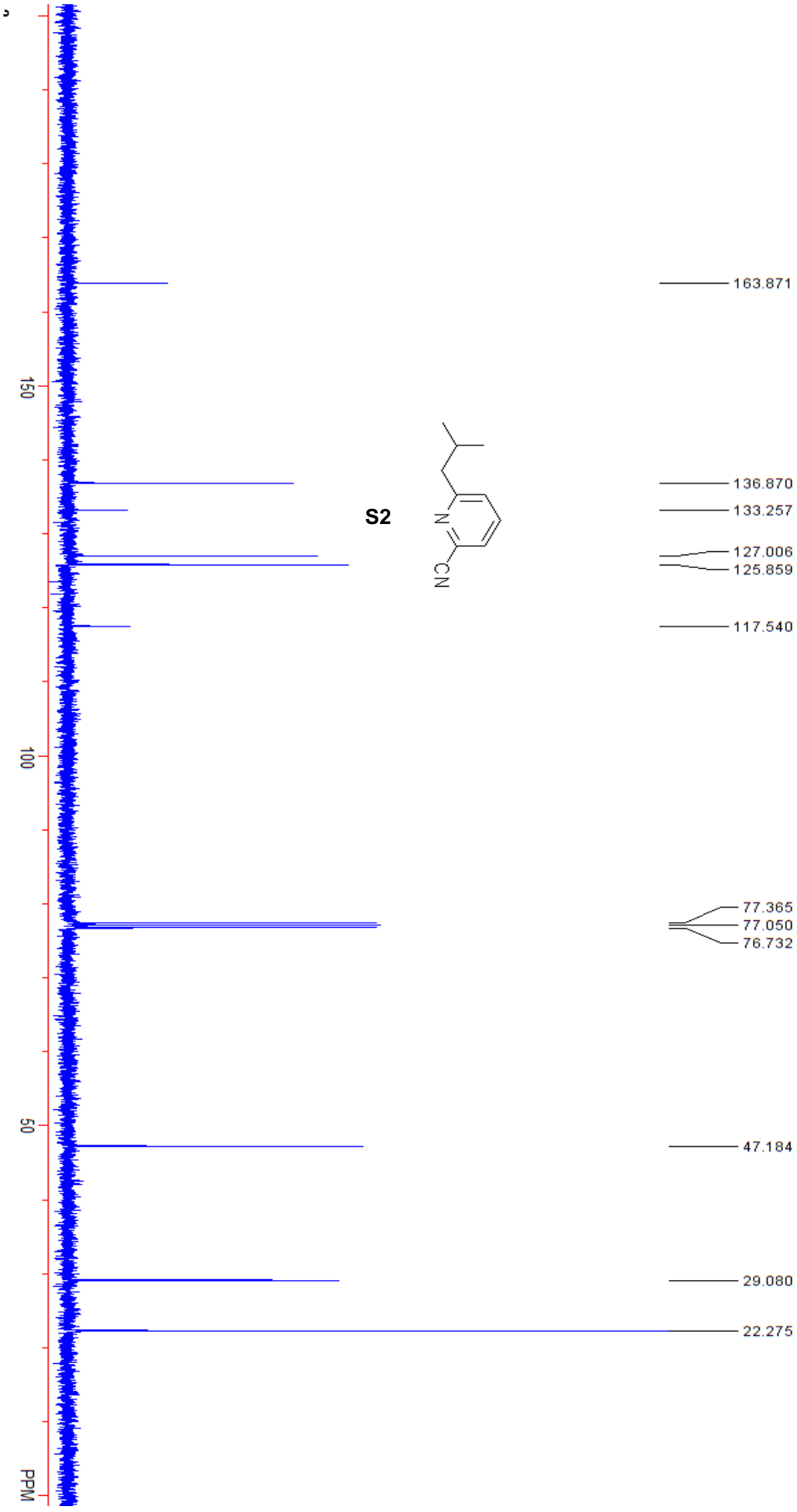
7.715
7.696
7.526
7.507
7.335
7.315
7.258
1.00
0.98
1.01
2.16
1.04
6.56
2.692
2.674
2.146
2.130
2.113
2.096
2.079
2.063
2.046
0.918
0.902
10
8
6
4
2
PPM

**S2**


N
CN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133.257
127.006
125.859
117.540
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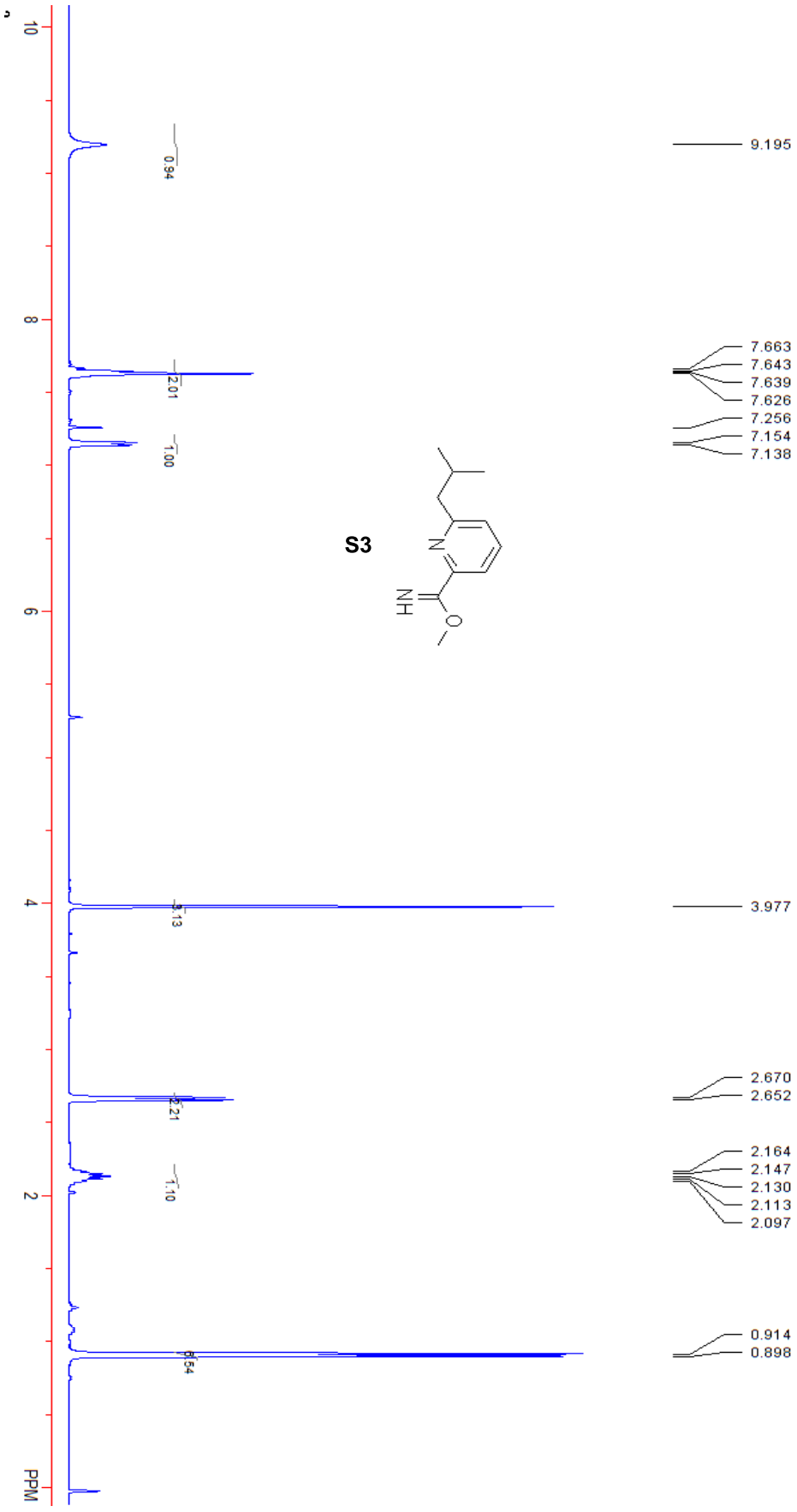
77.050
76.732
47.184
29.080
22.275
150
100
50
PPM
S2
N
CN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7.256
7.154
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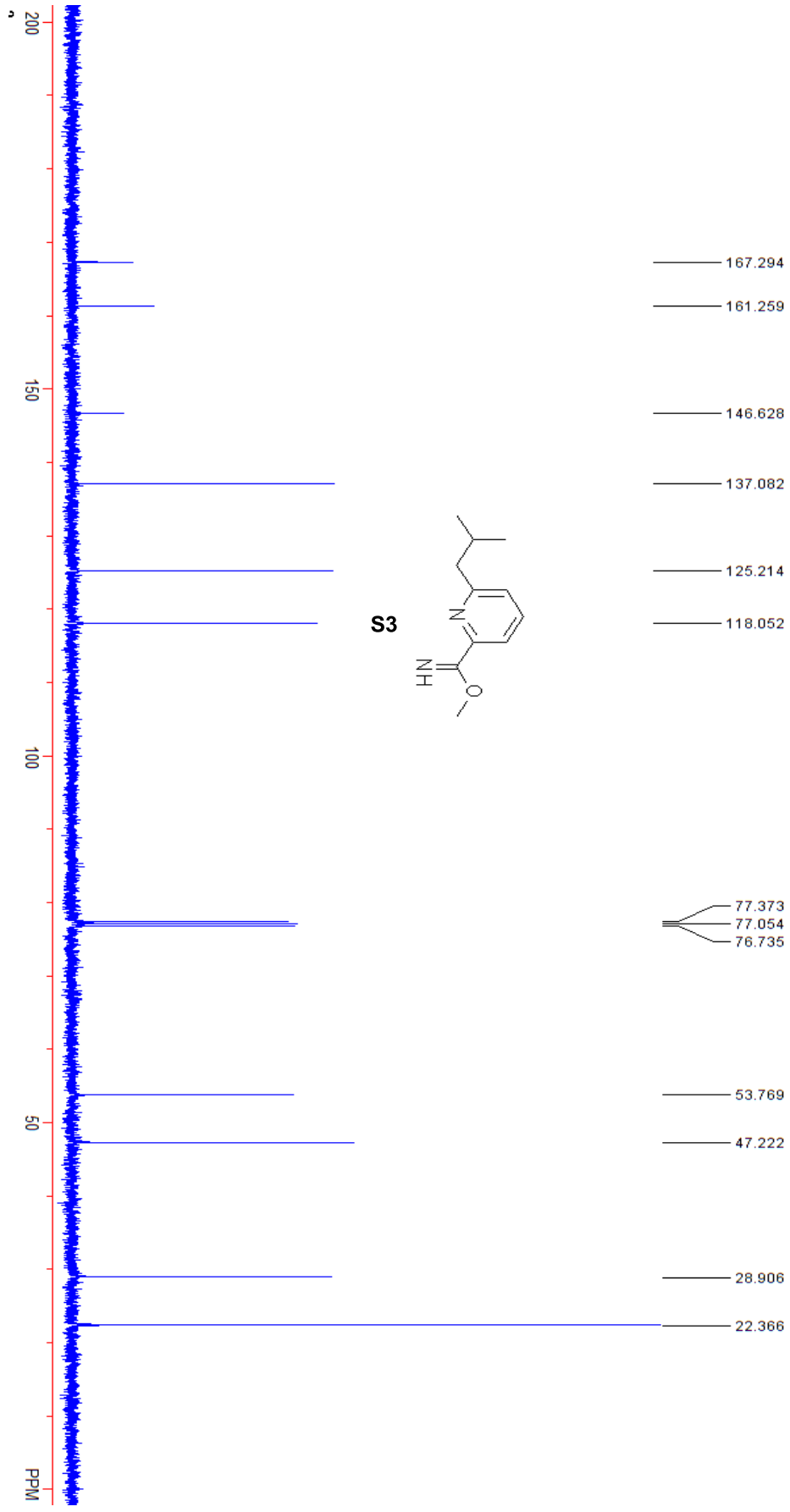
2.147
2.130
2.113
2.097
0.914
0.898
0.94
2.01
1.00
3.13
2.21
1.10
9.54
S3
N
NH
O
10
8
6
4
2
PPM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PPM
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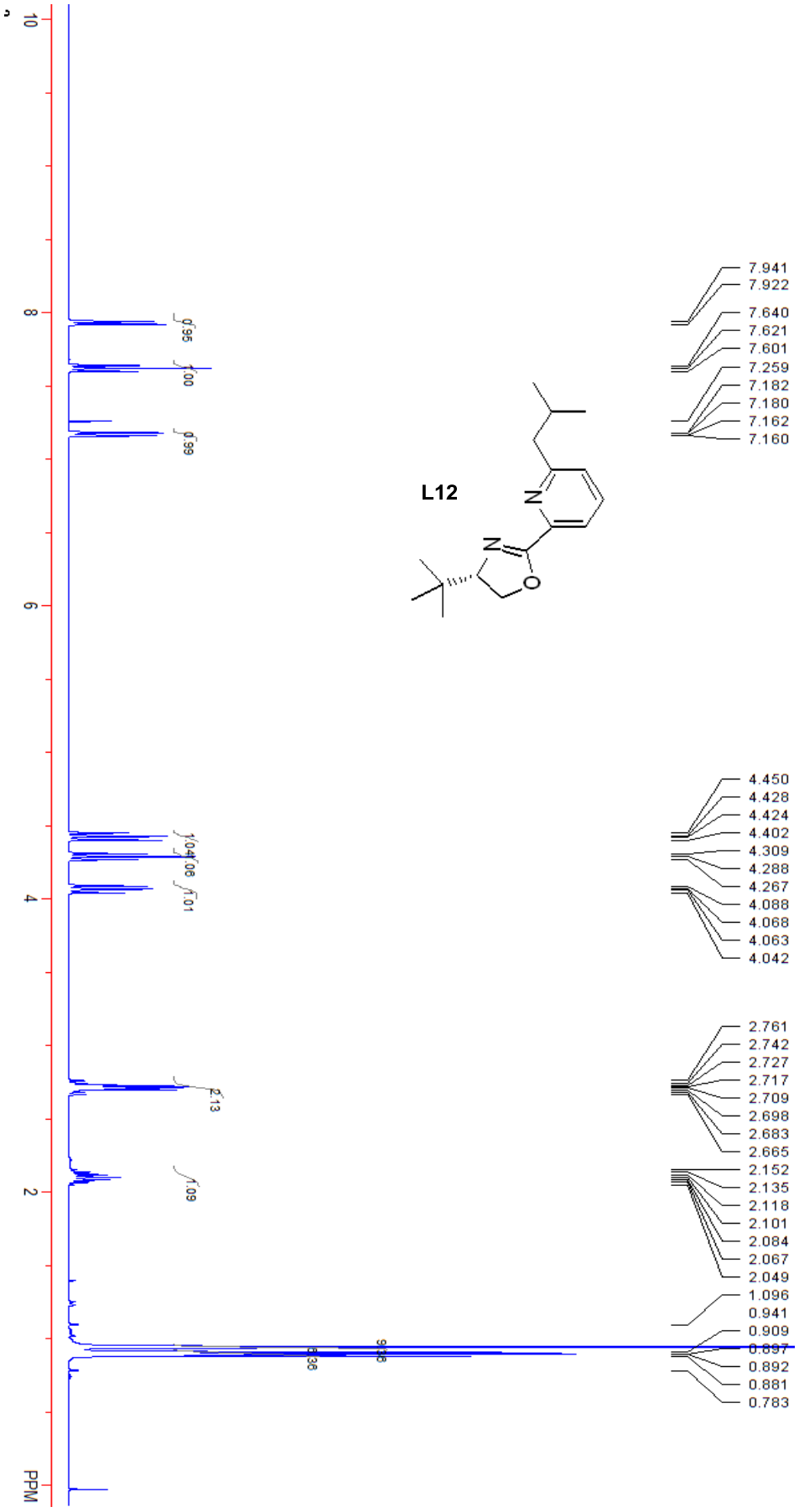
28.906
22.366
S3
N
HN
O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7.621
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7.162
7.160
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4.428
4.424
4.402
4.309
4.288
4.267
4.088
4.068
4.063
4.042
2.761
2.742
2.727
2.717
2.709
2.698
2.683
2.665
2.152
2.135
2.118
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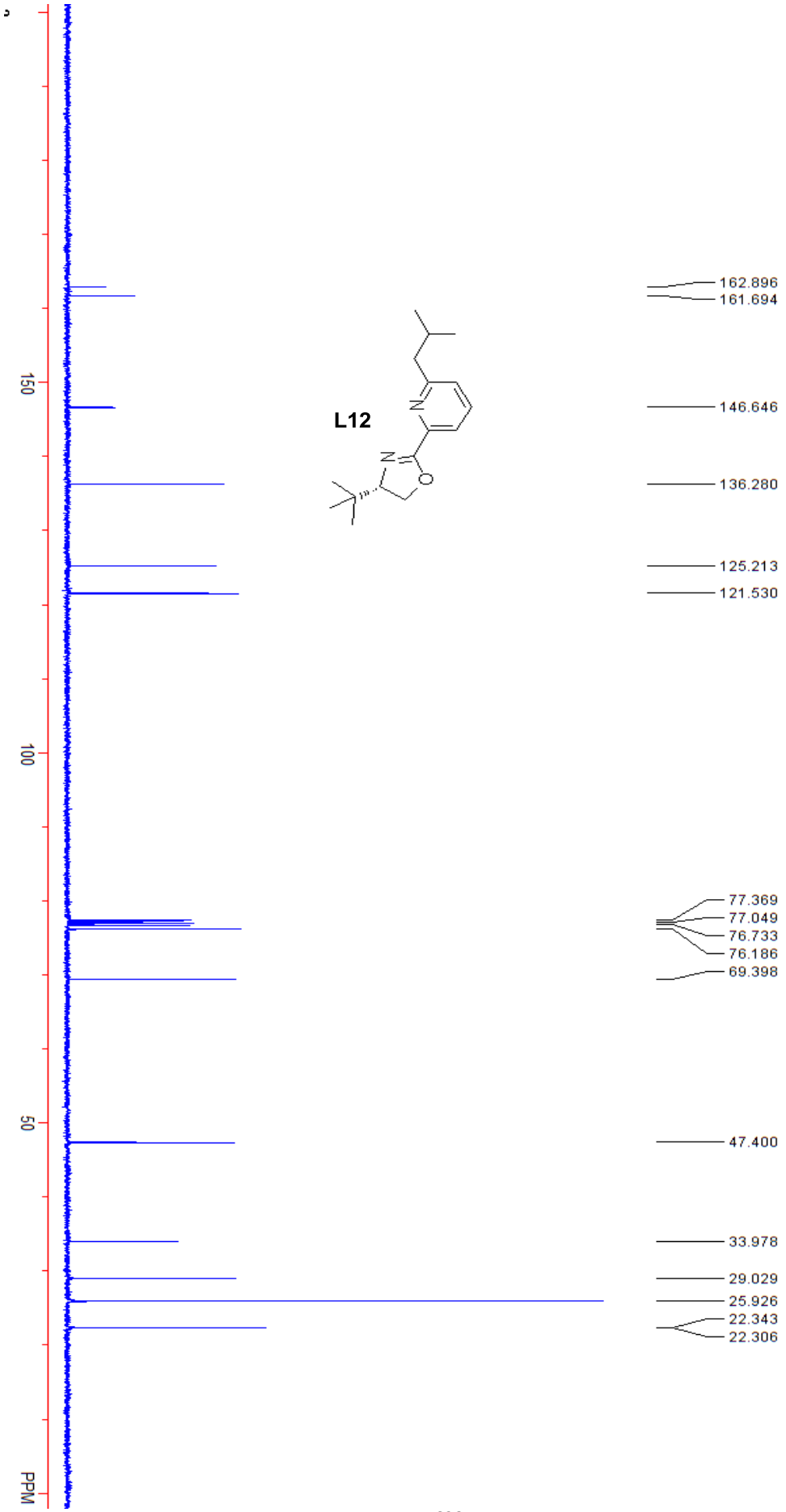
PPM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150
100
50
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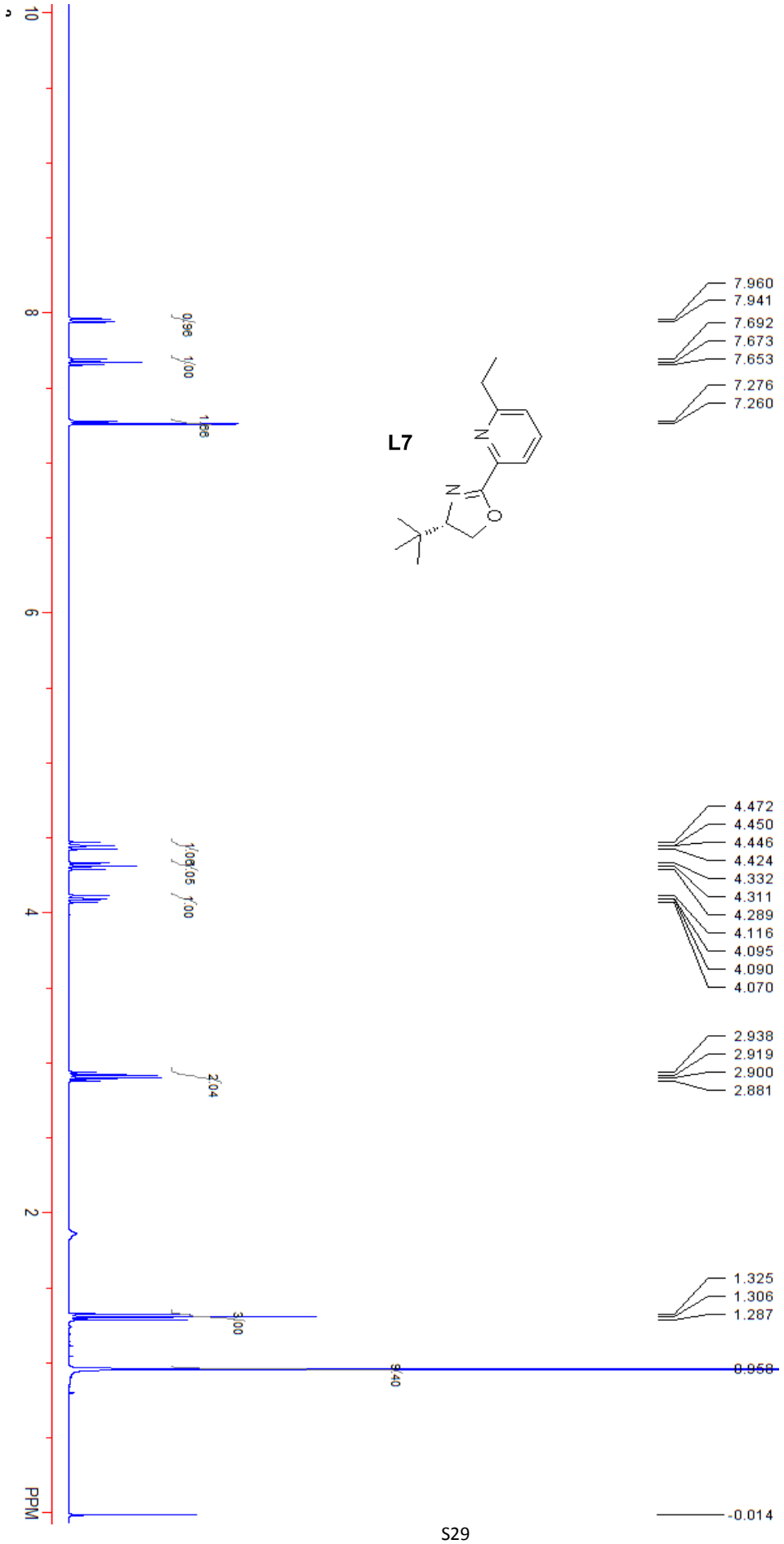
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4.446
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4.311
4.289
4.116
4.095
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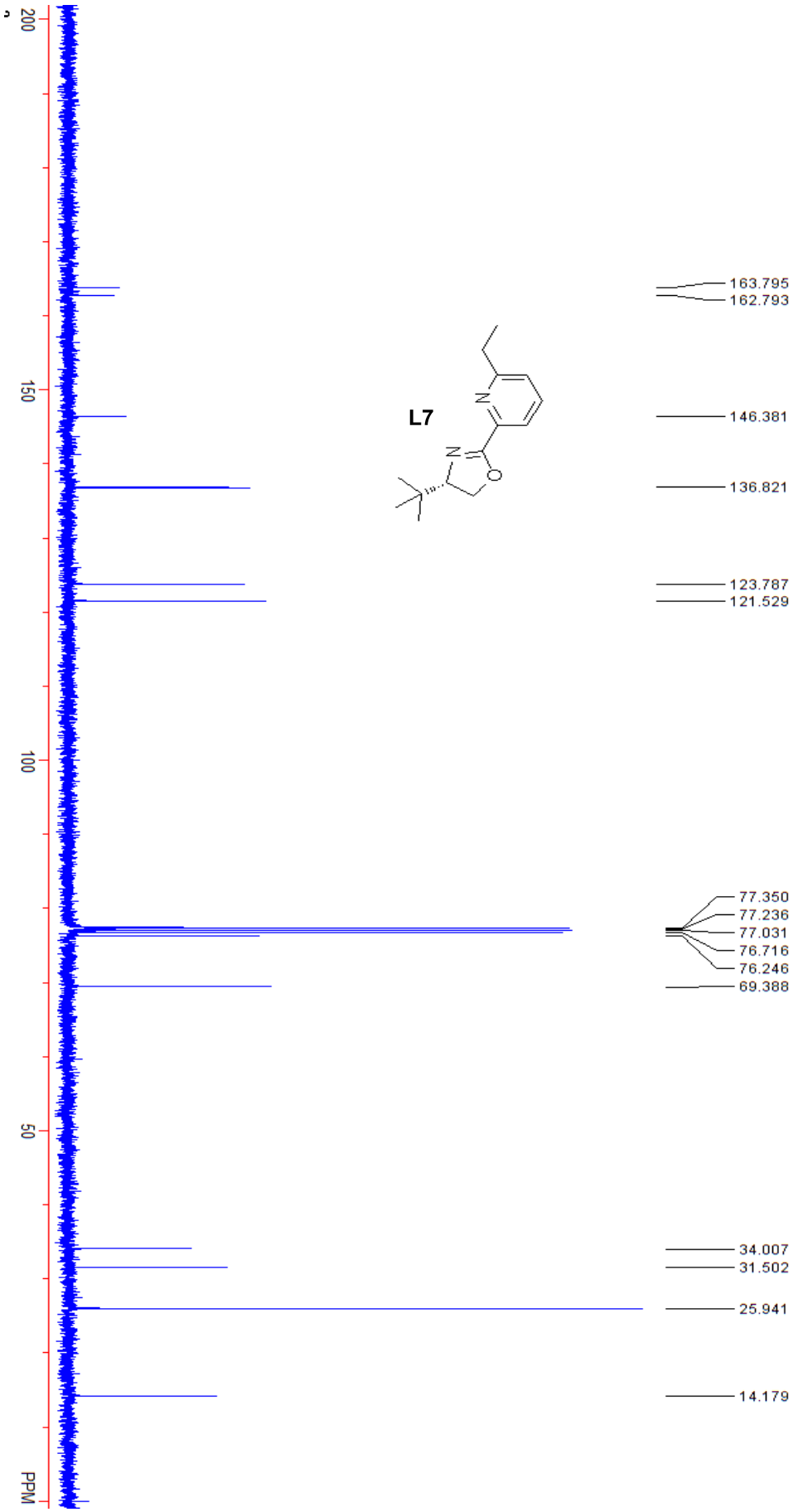
1.306
1.287
0.958
-0.014
PPM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150
100
50
PPM
L7
N
N
O
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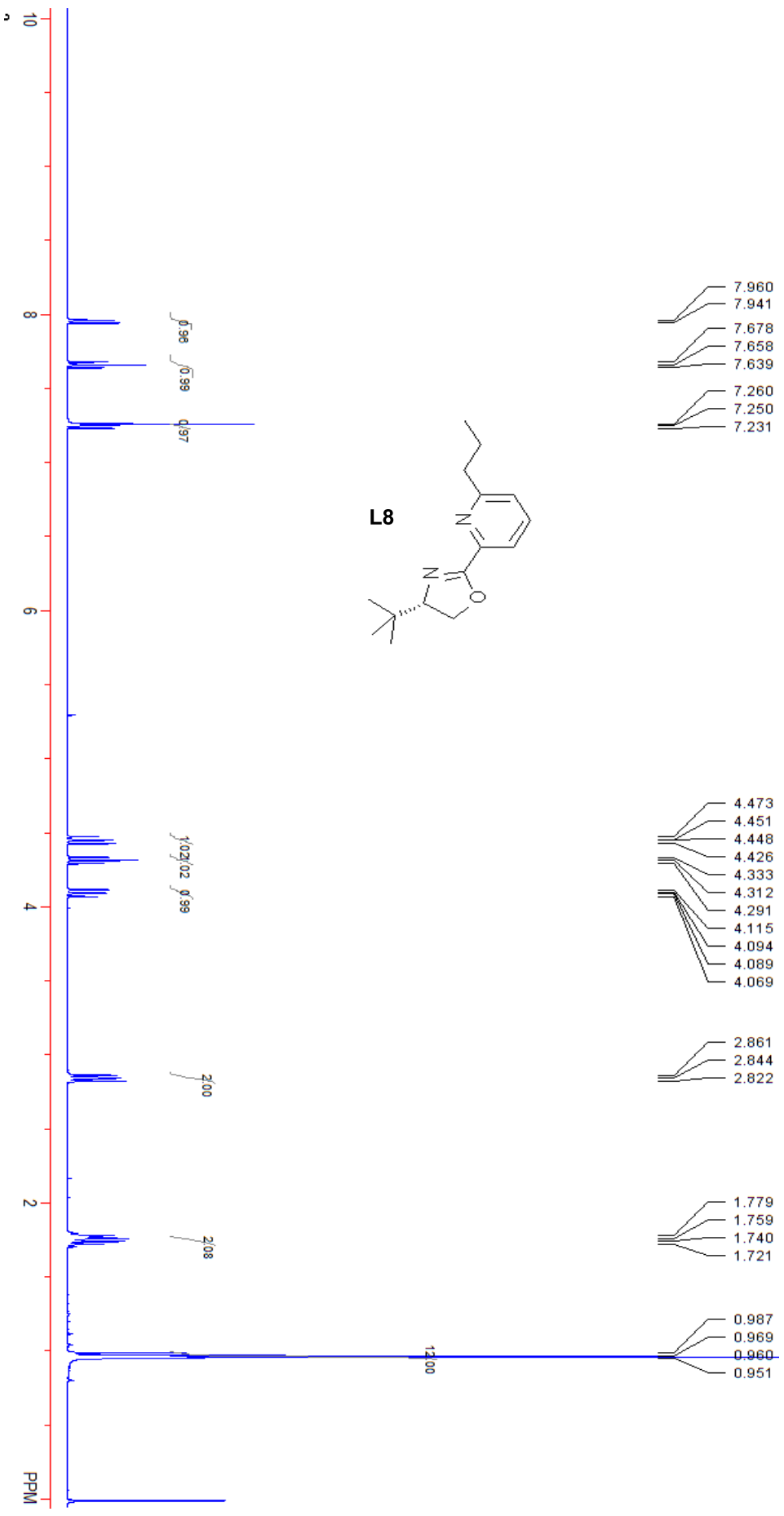
25.941
14.179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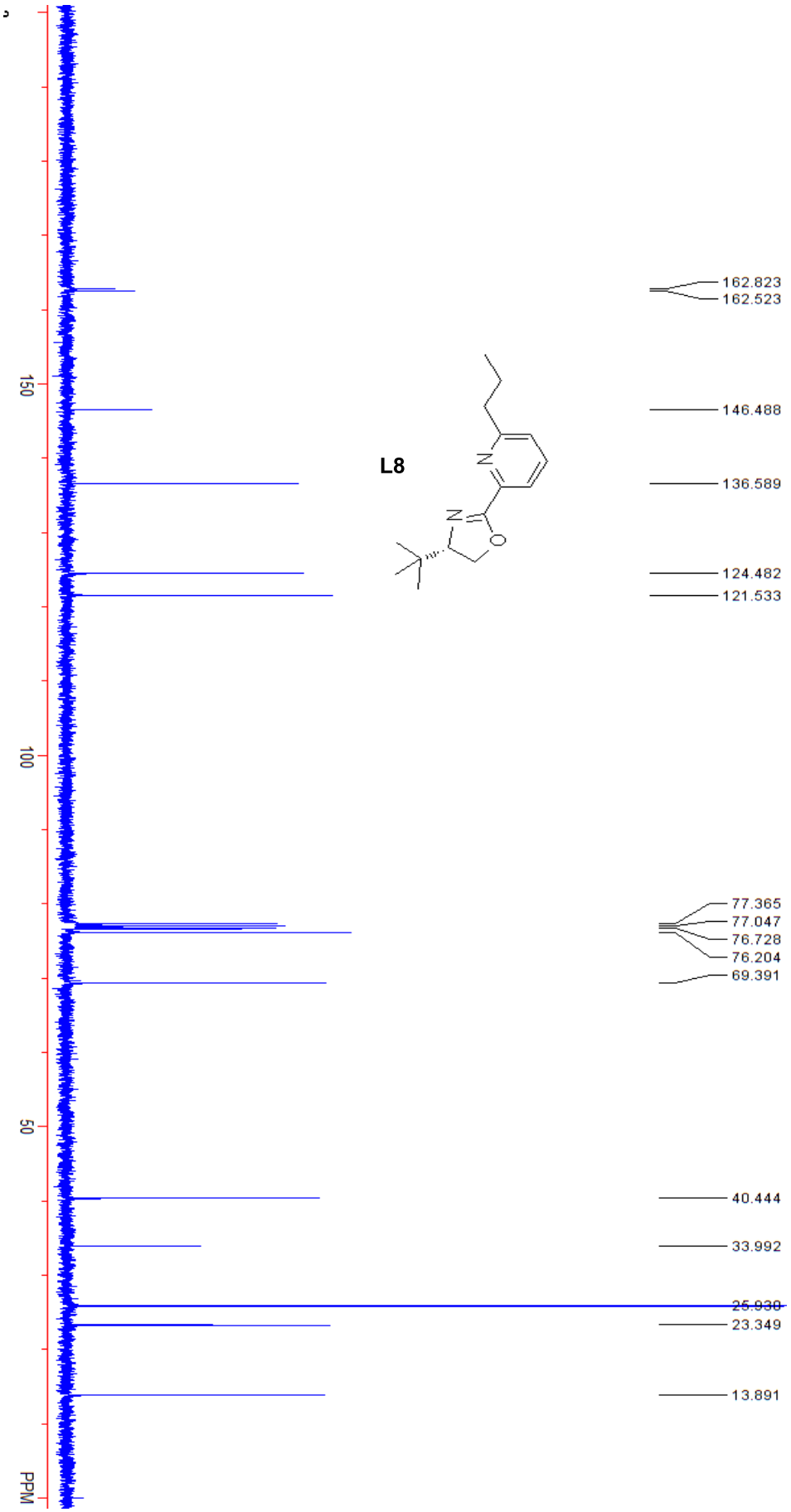
6
4
2
PPM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50
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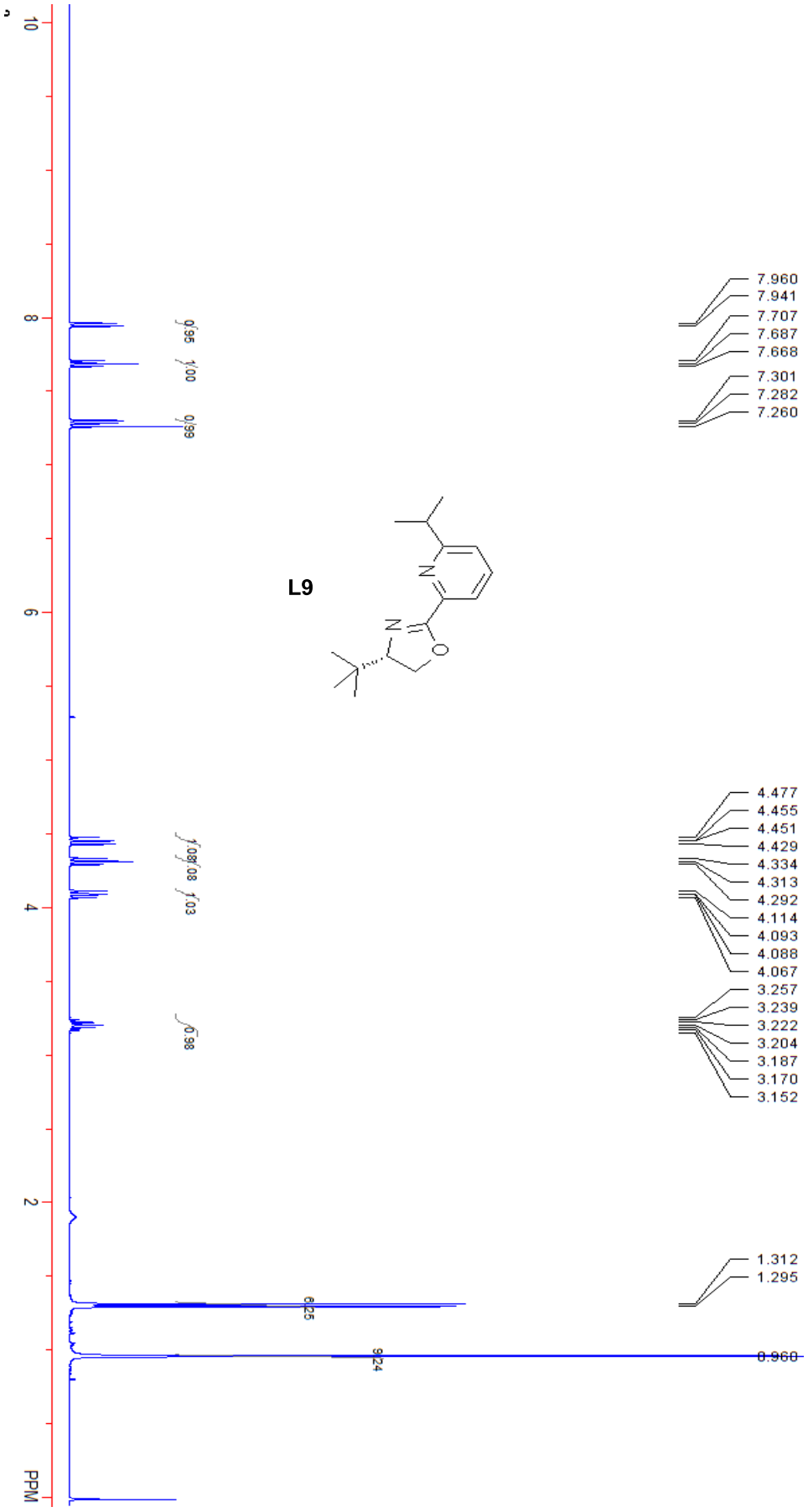
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4.429
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4.313
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4.114
4.093
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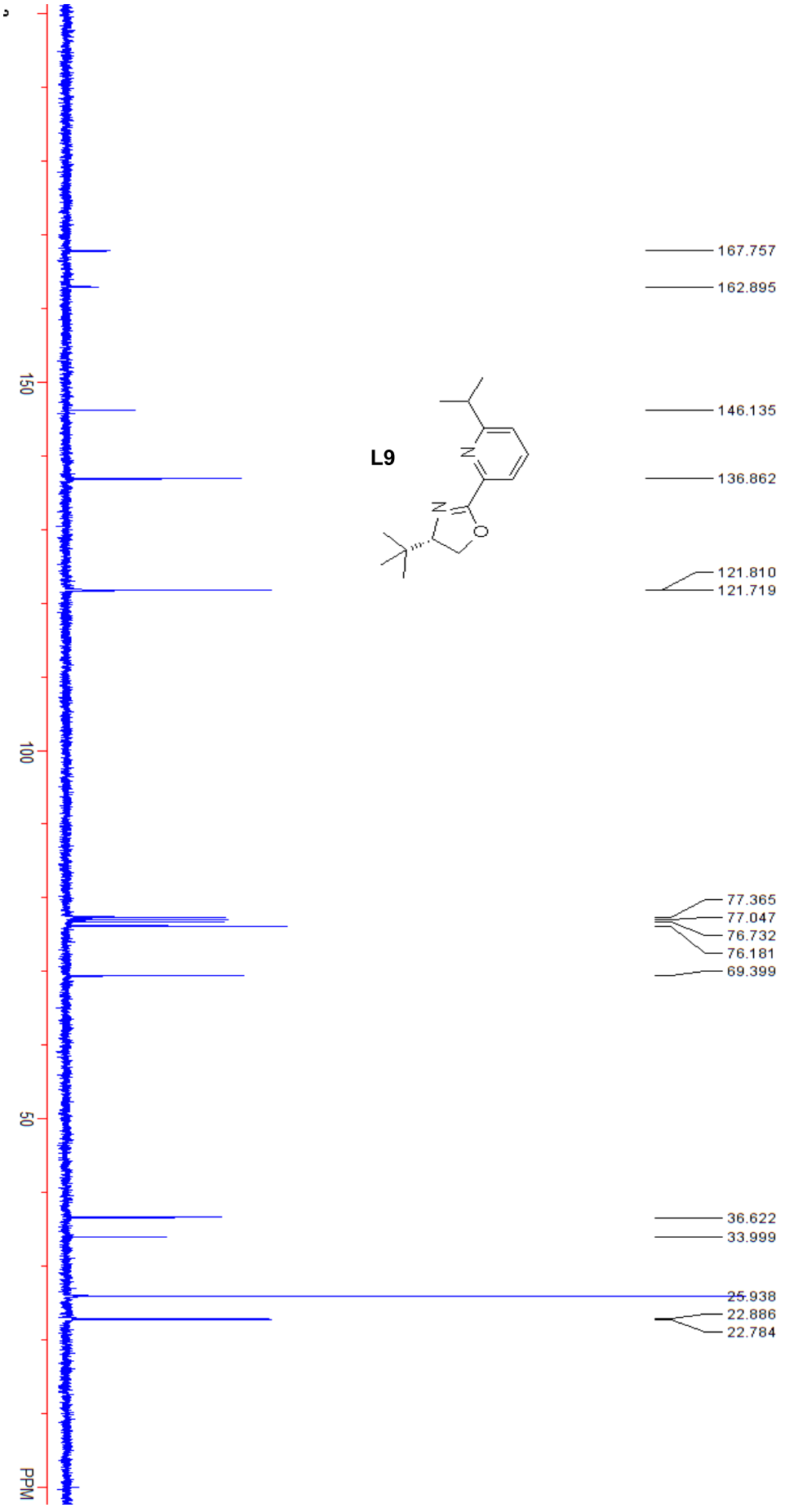
PPM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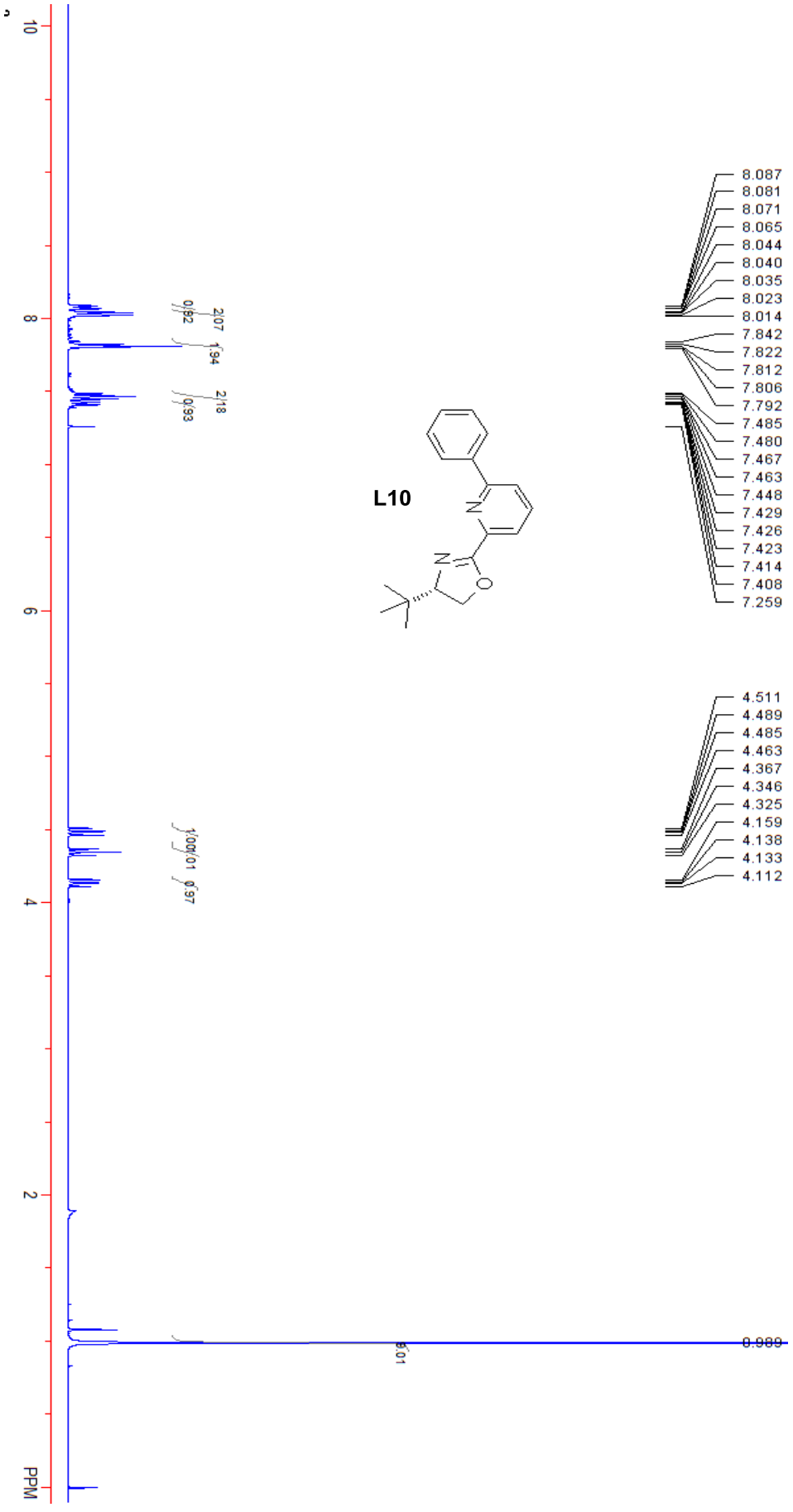
150
100
50
PPM
L9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7.806
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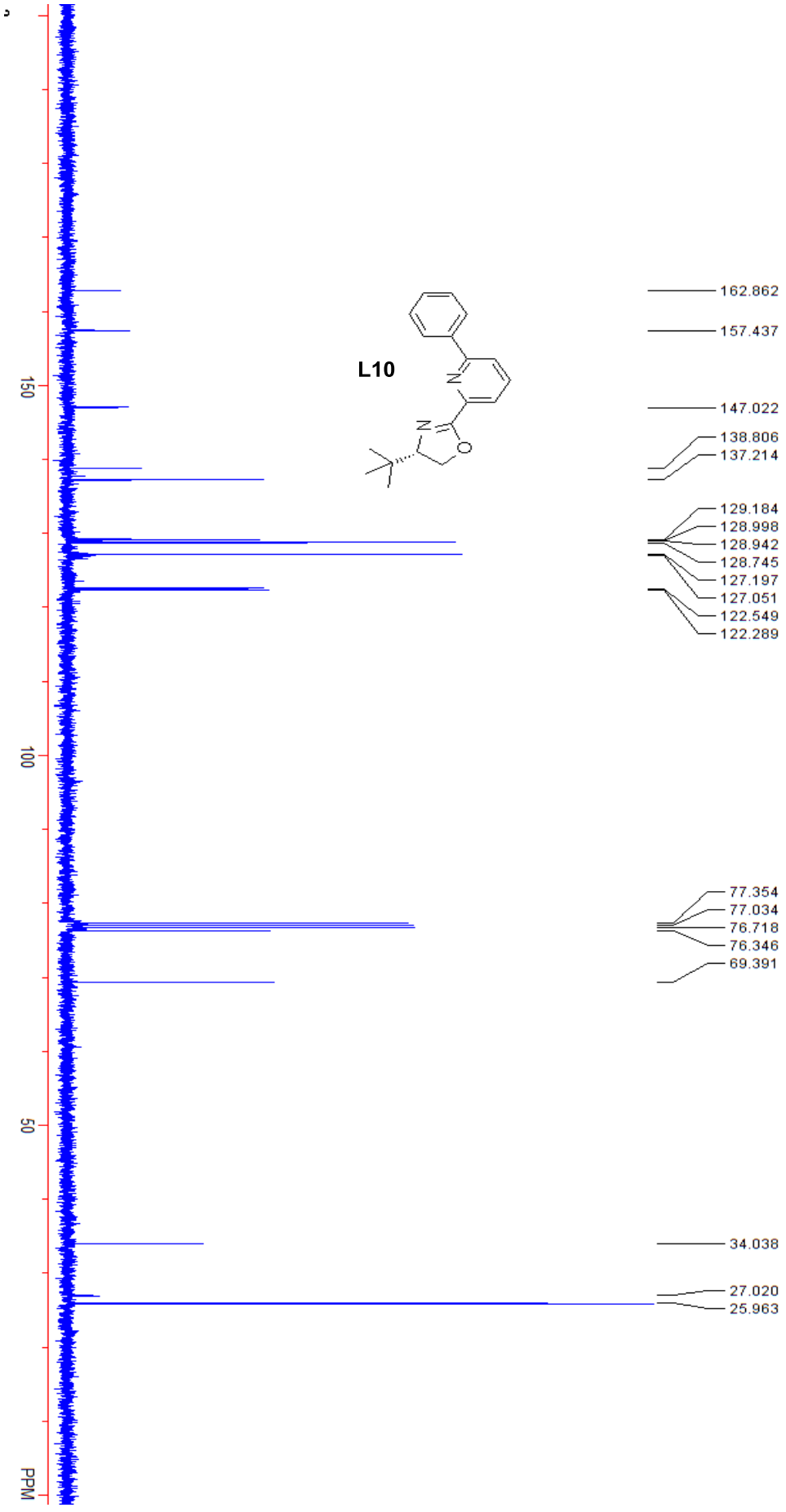
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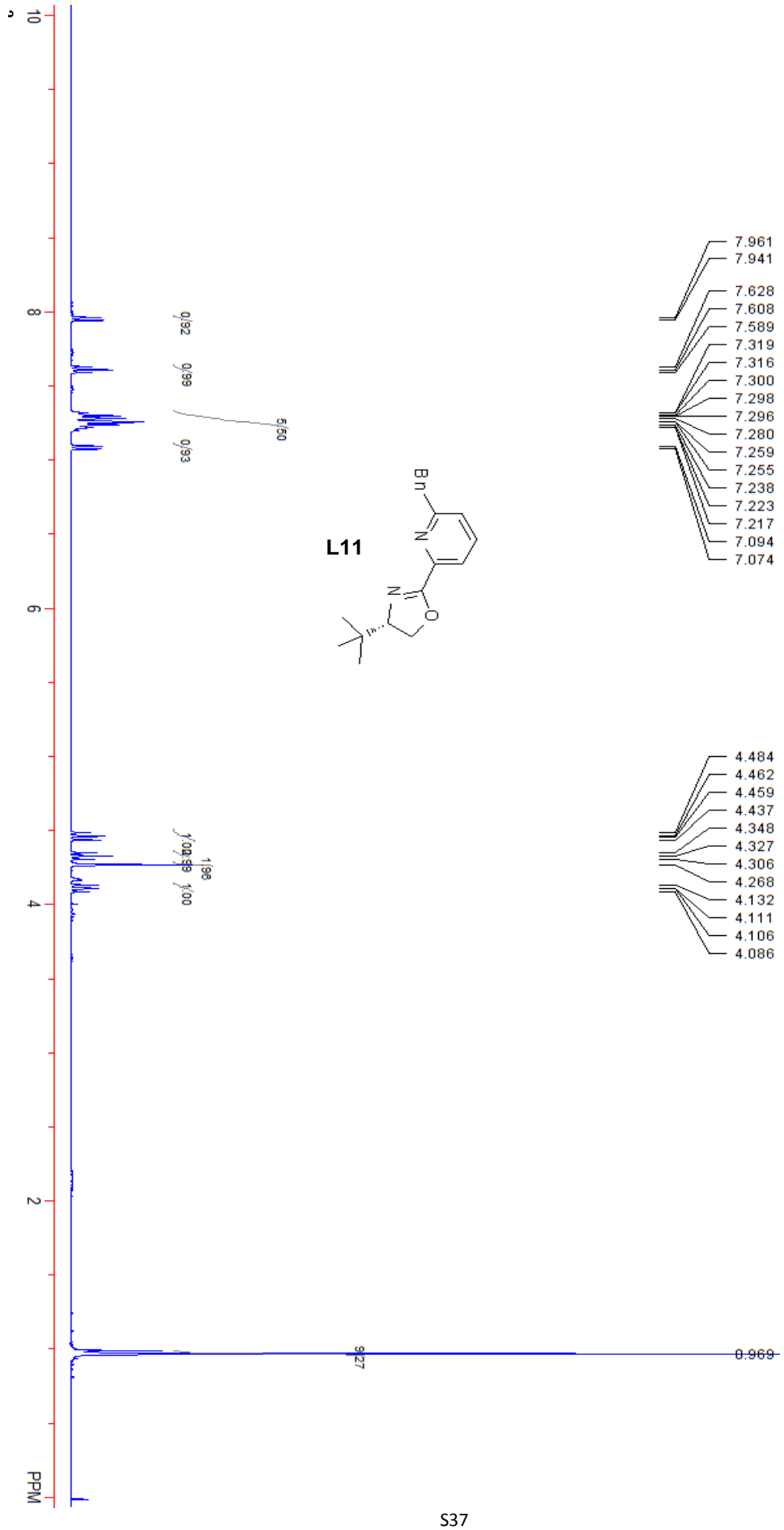
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7.074
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4.306
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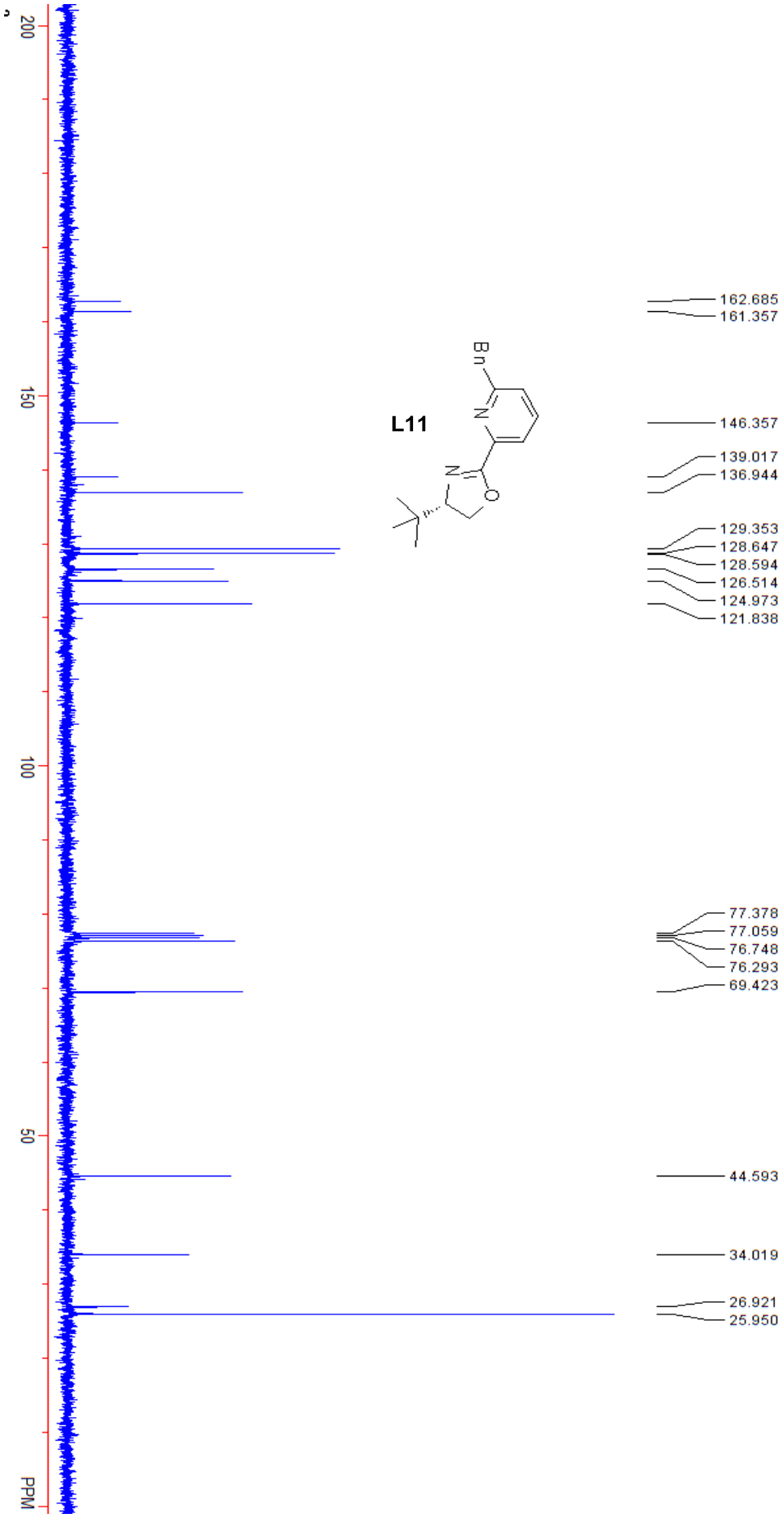
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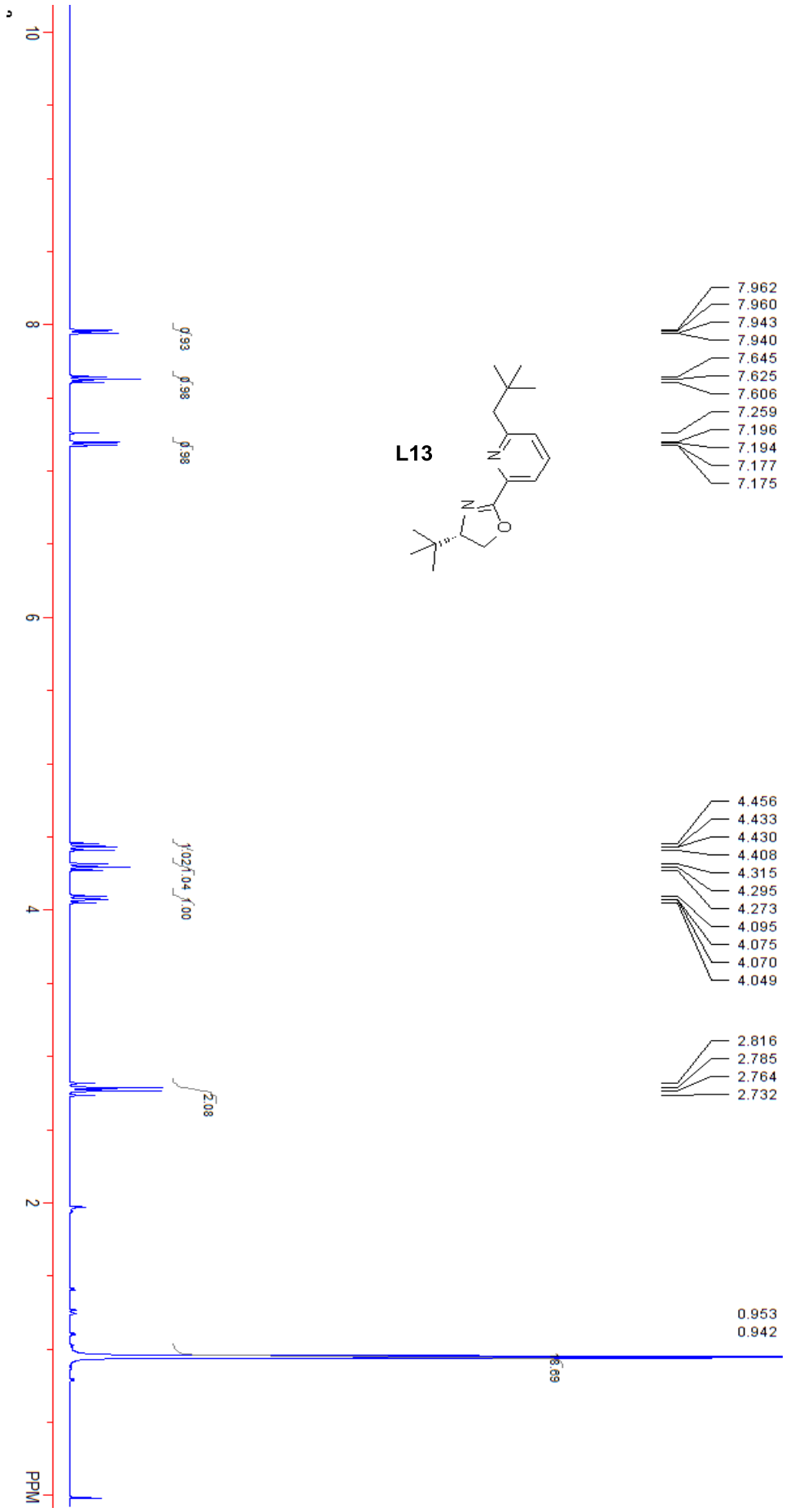
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4.315
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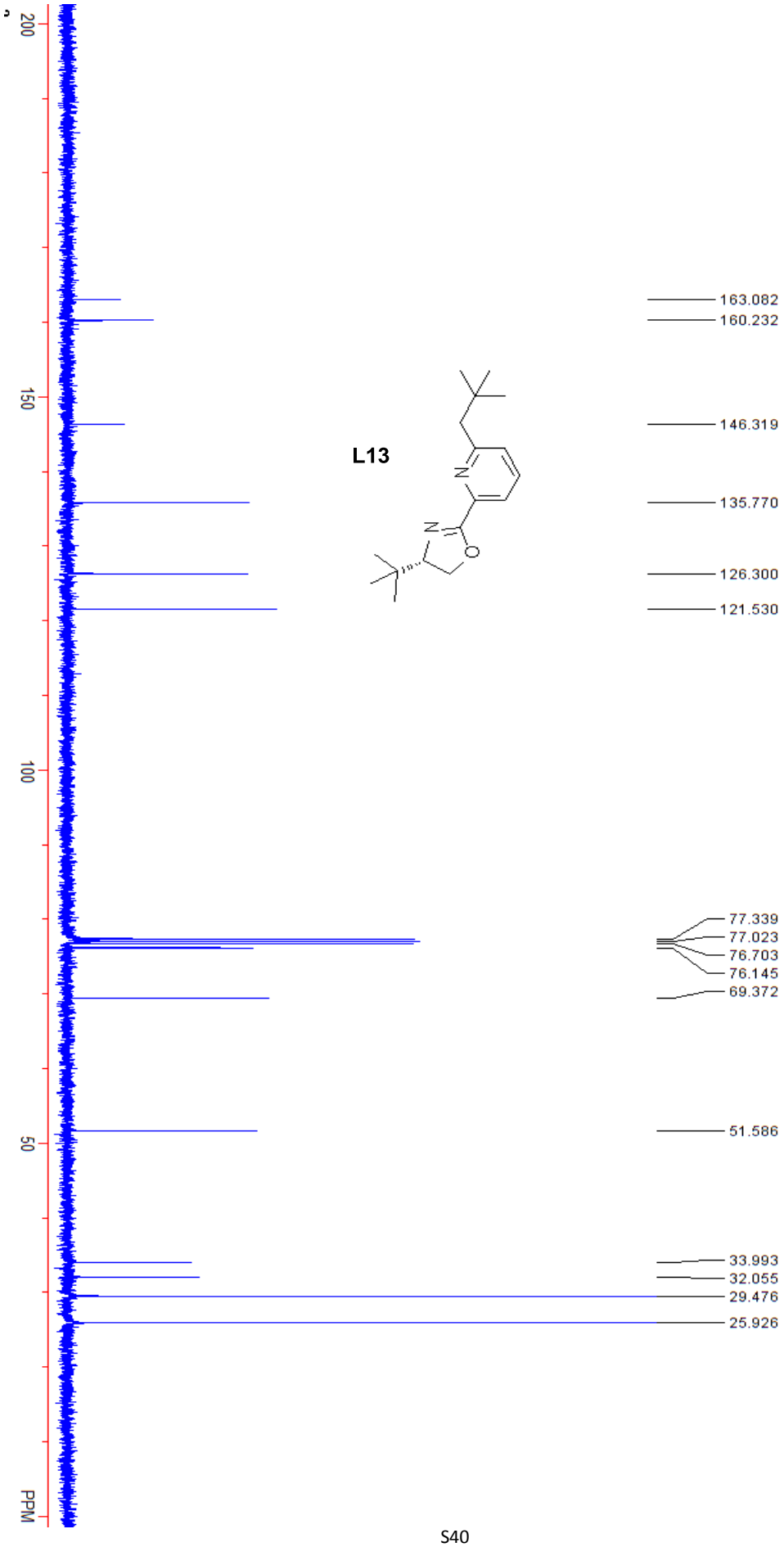
PPM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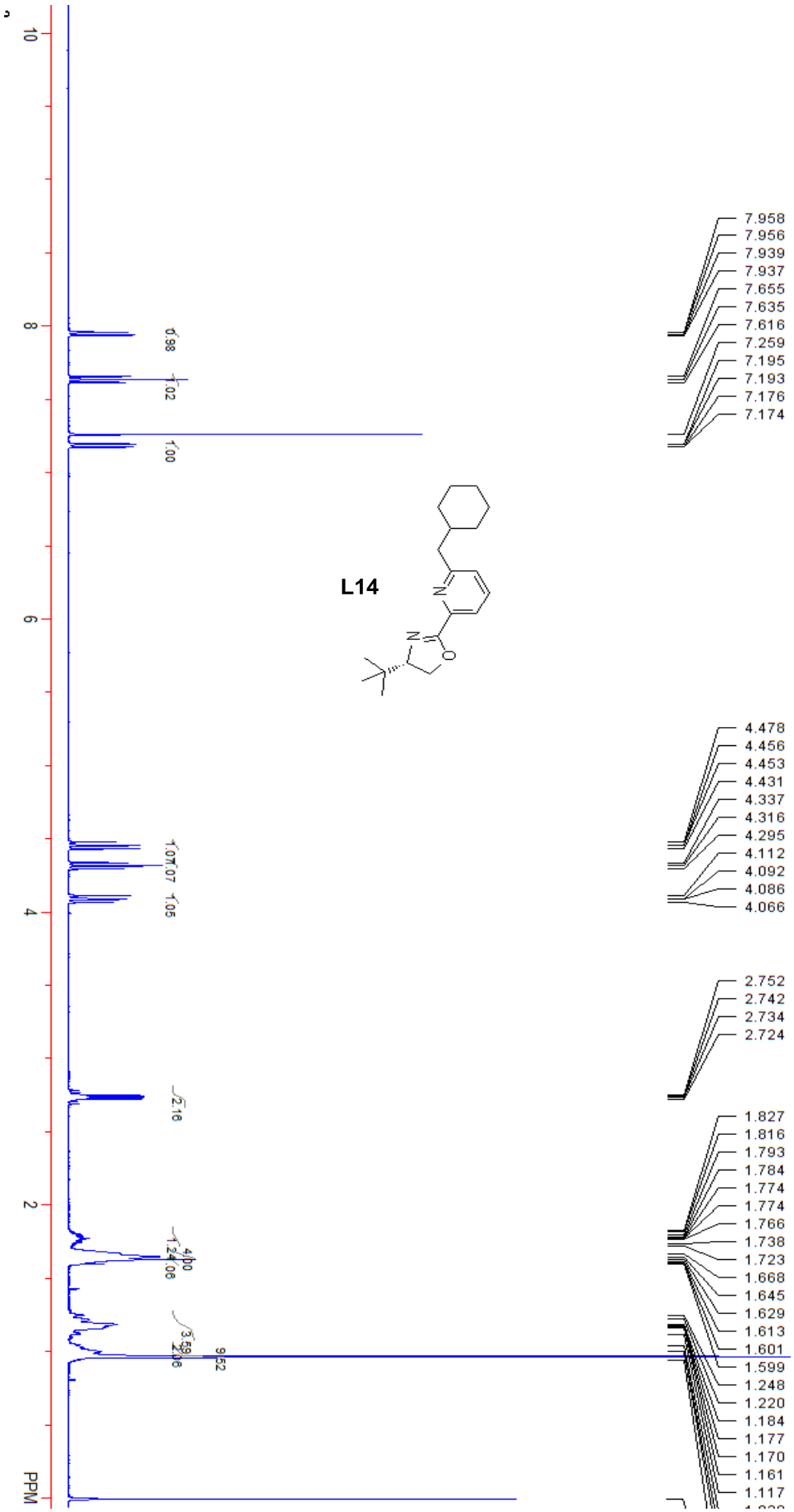
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1.07
1.07
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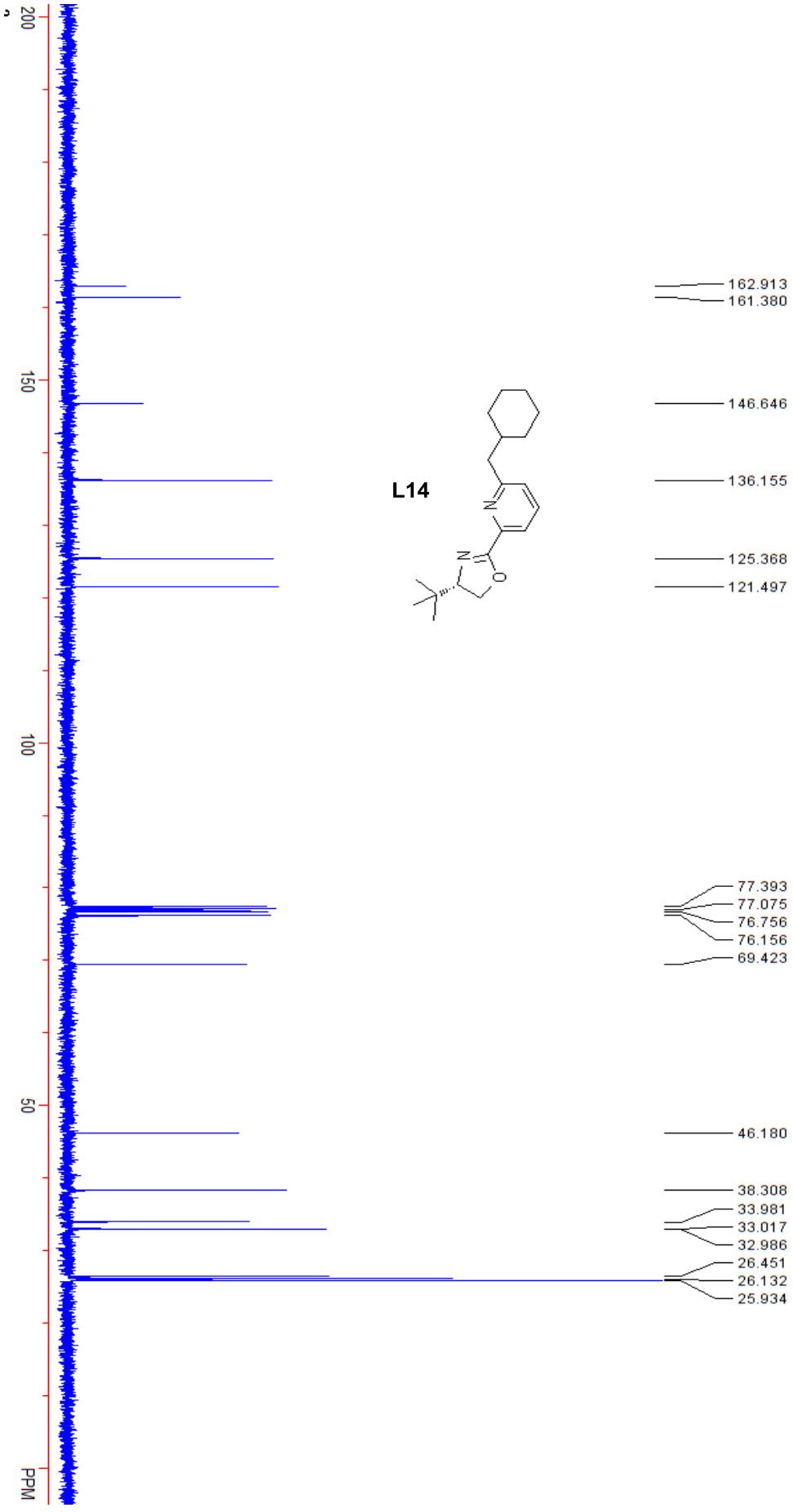
PPM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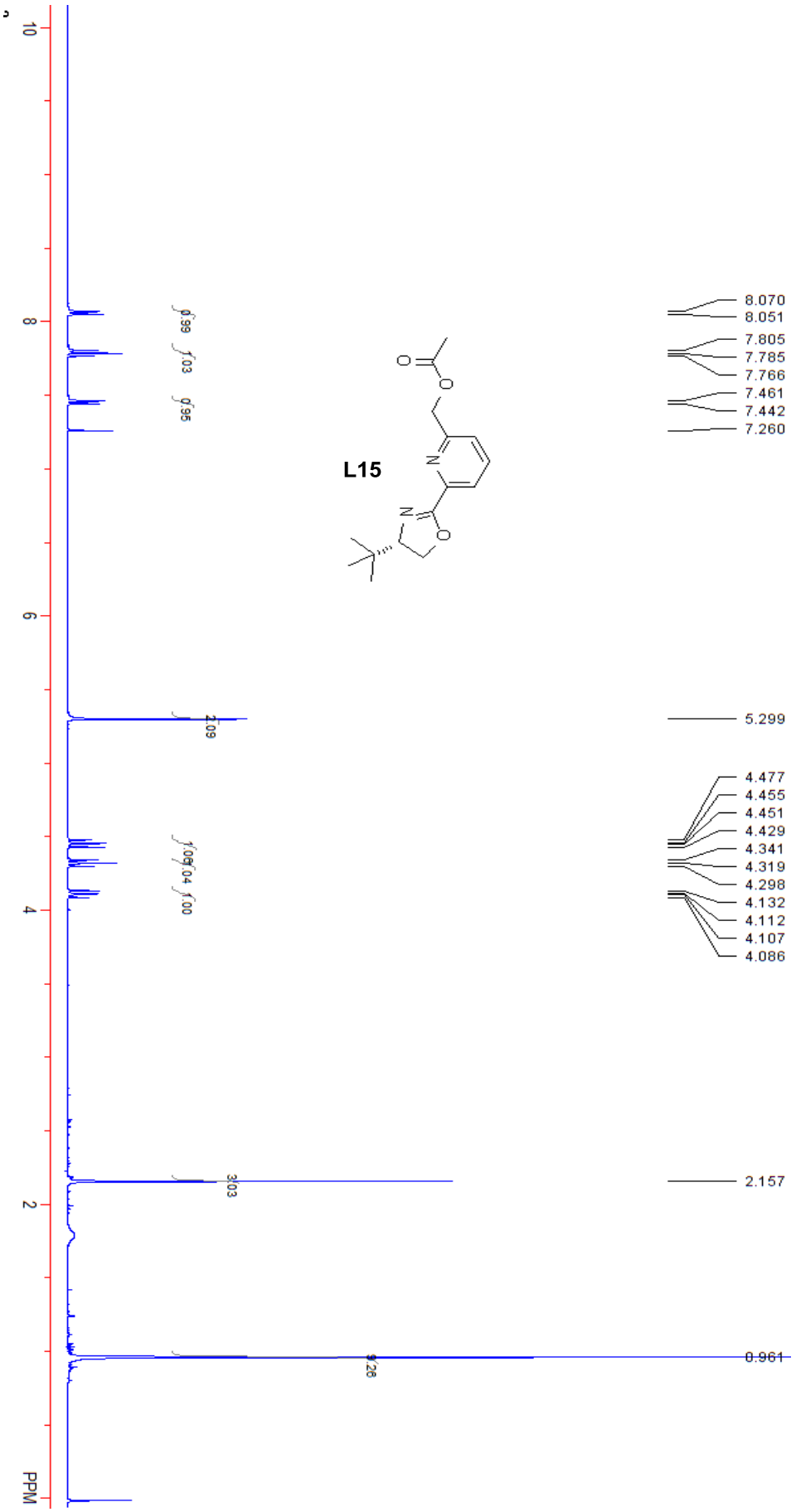
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4.086
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0.99
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9.26
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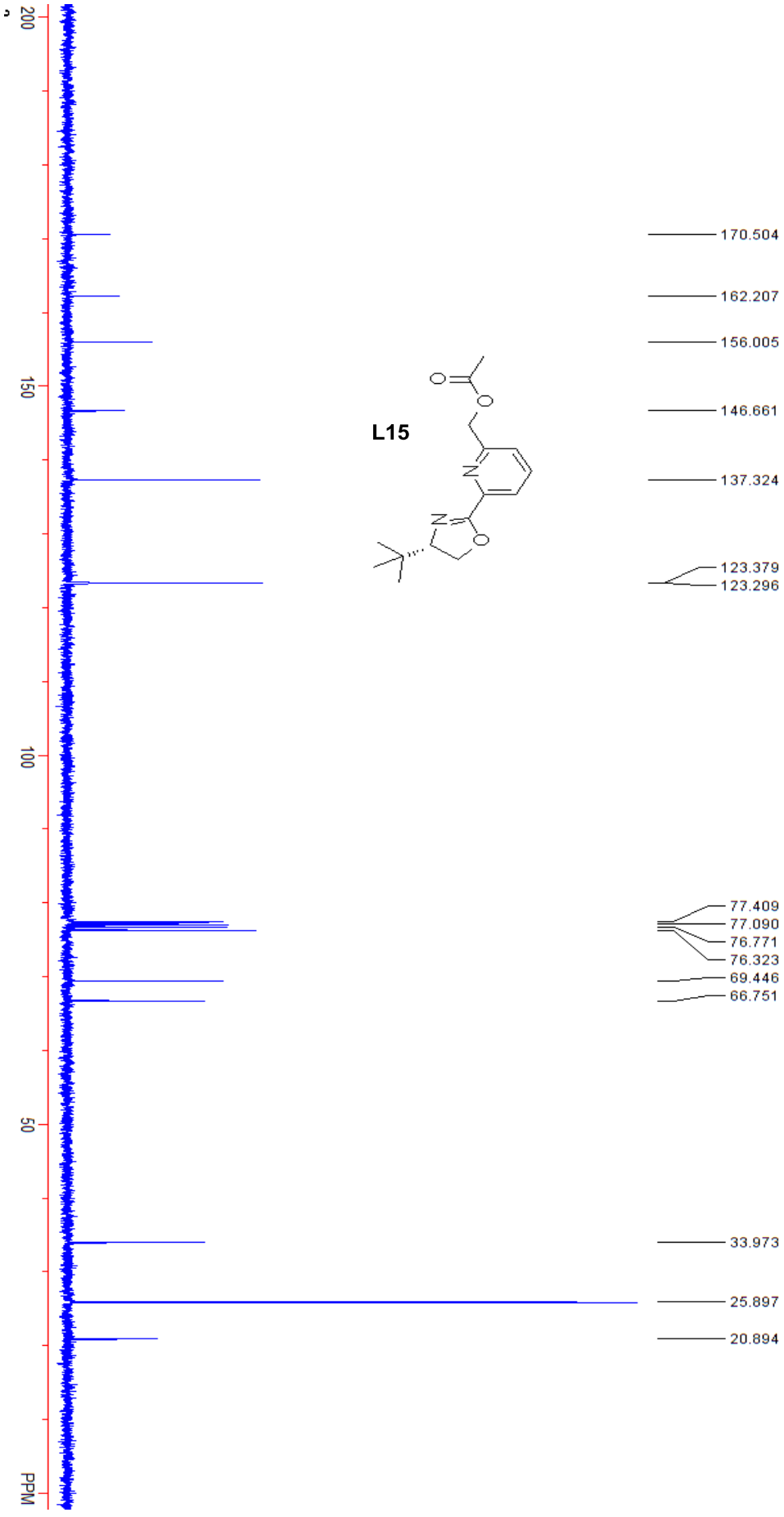
6
4
2
PPM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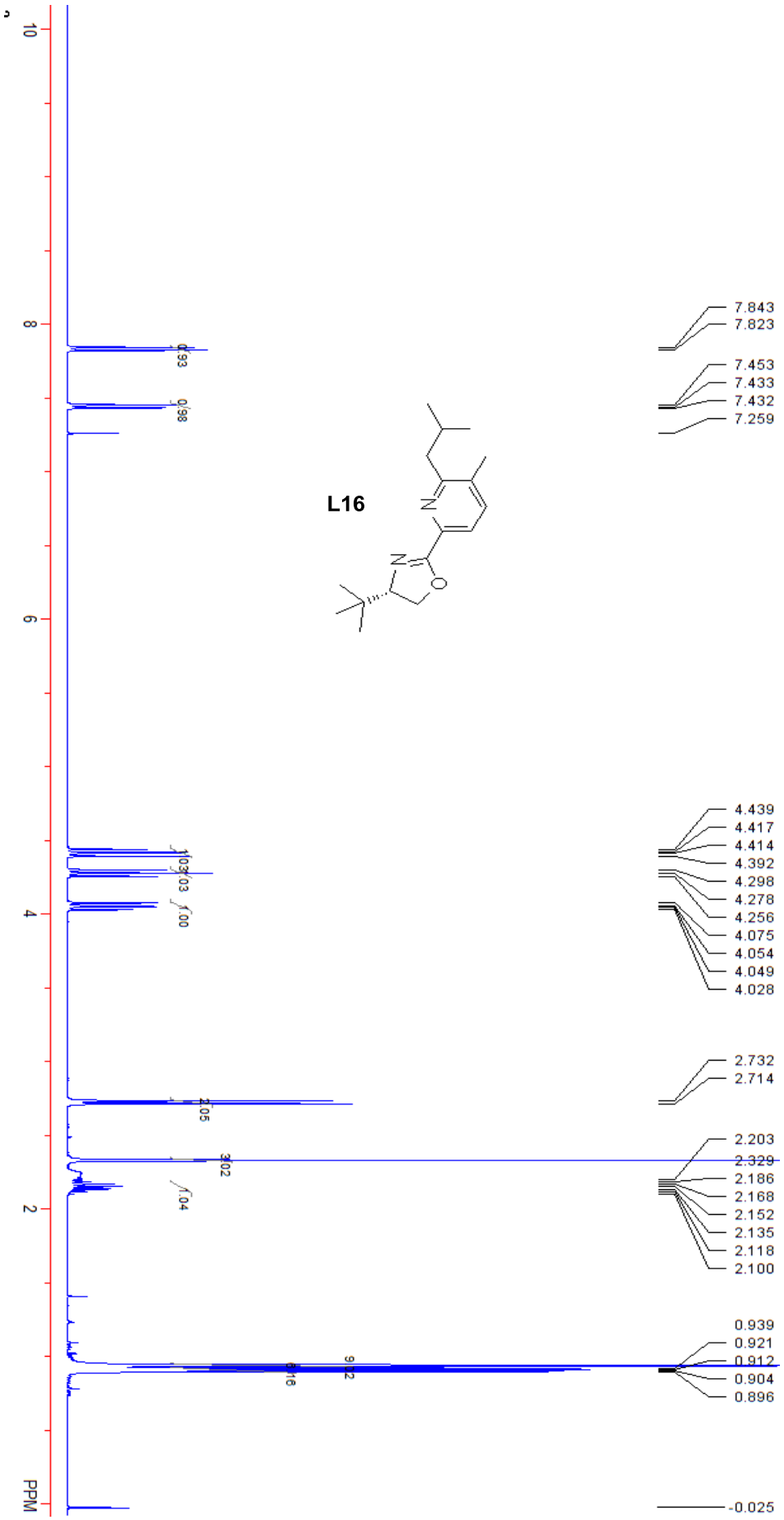
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4.278
4.256
4.075
4.054
4.049
4.028
2.732
2.714
2.203
2.329
2.186
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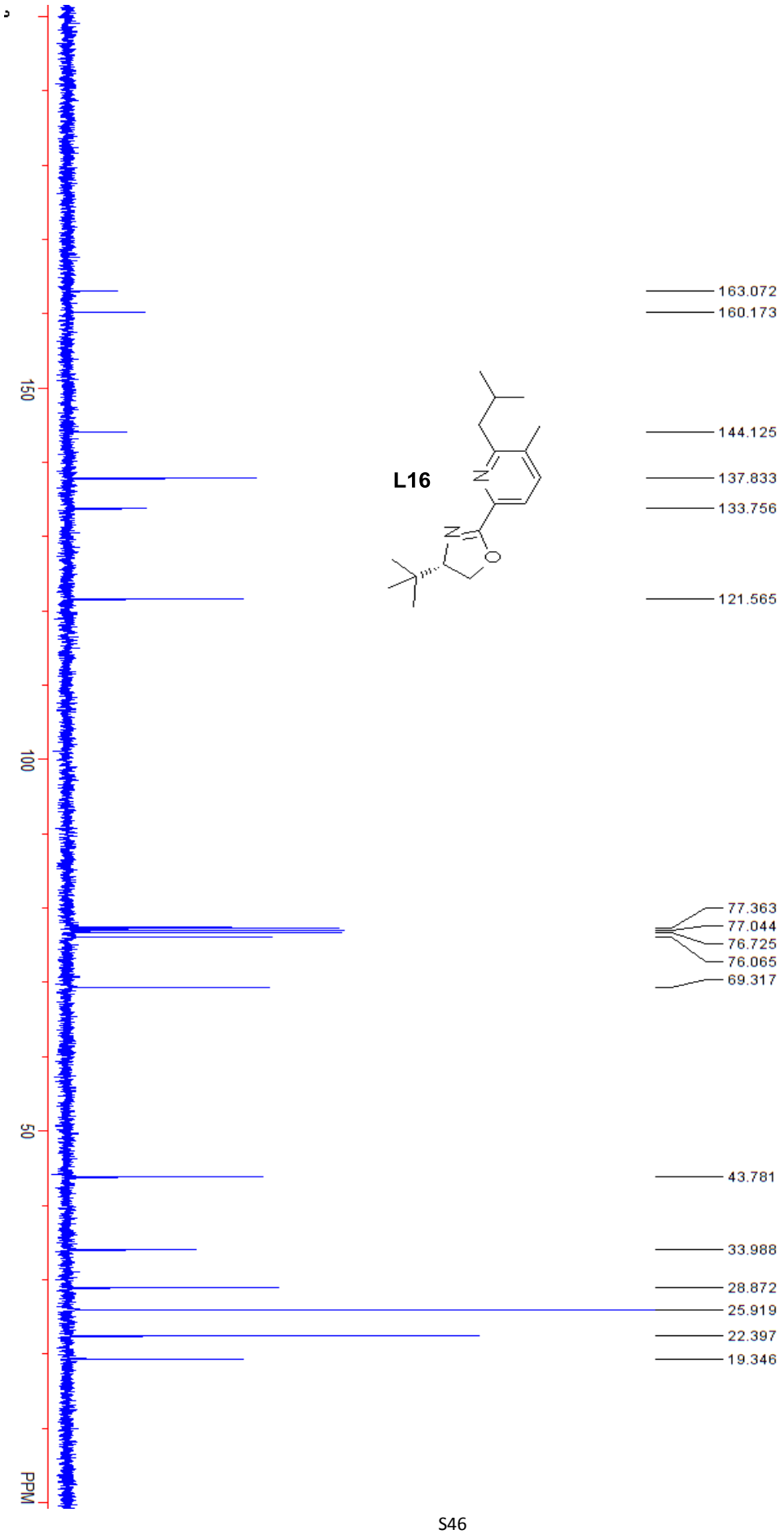
PPM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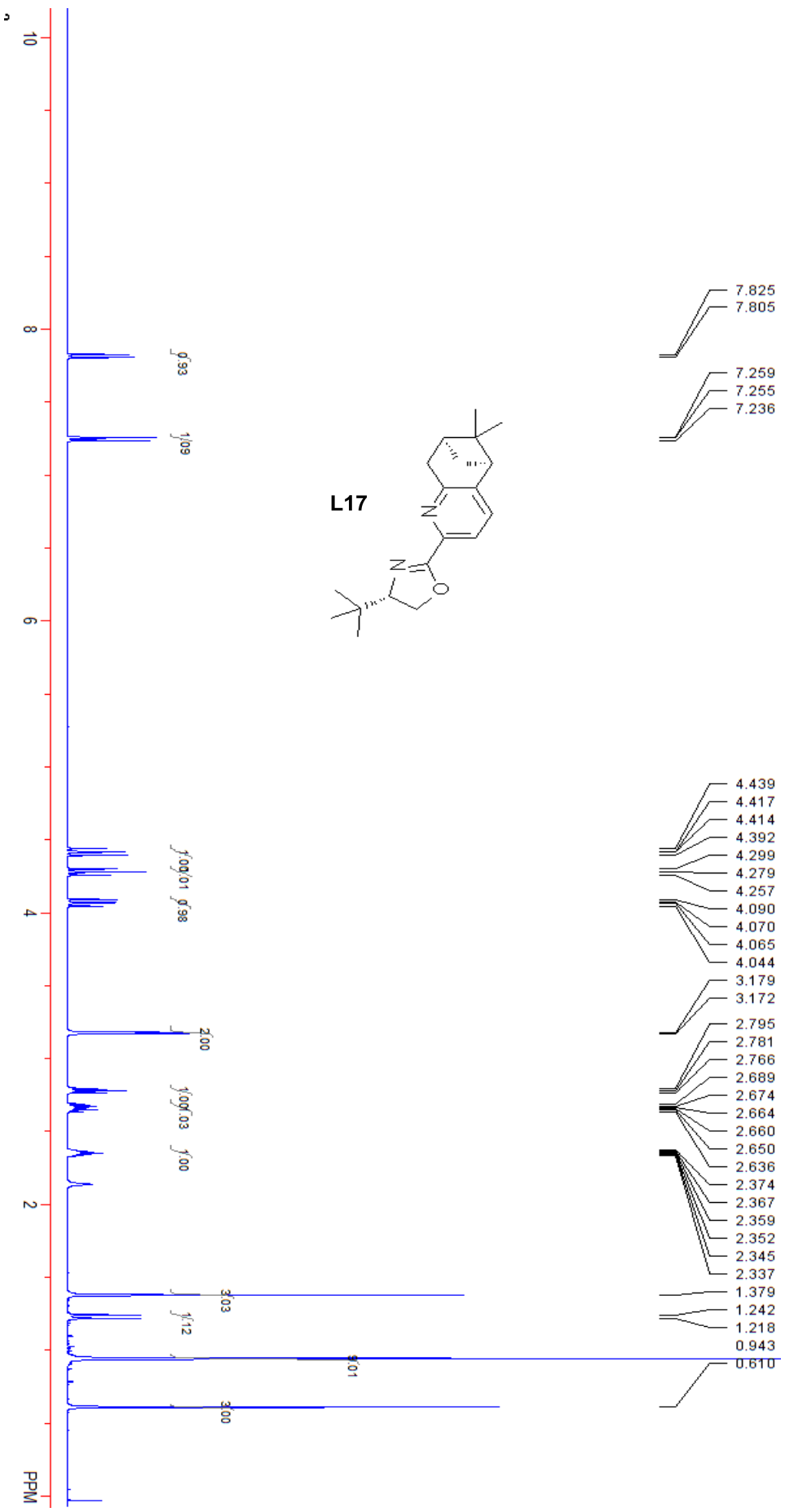
150
100
50
PPM
L16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7.259
7.255
7.236
4.439
4.417
4.414
4.392
4.299
4.279
4.257
4.090
4.070
4.065
4.044
3.179
3.172
2.795
2.781
2.766
2.689
2.674
2.664
2.660
2.650
2.636
2.374
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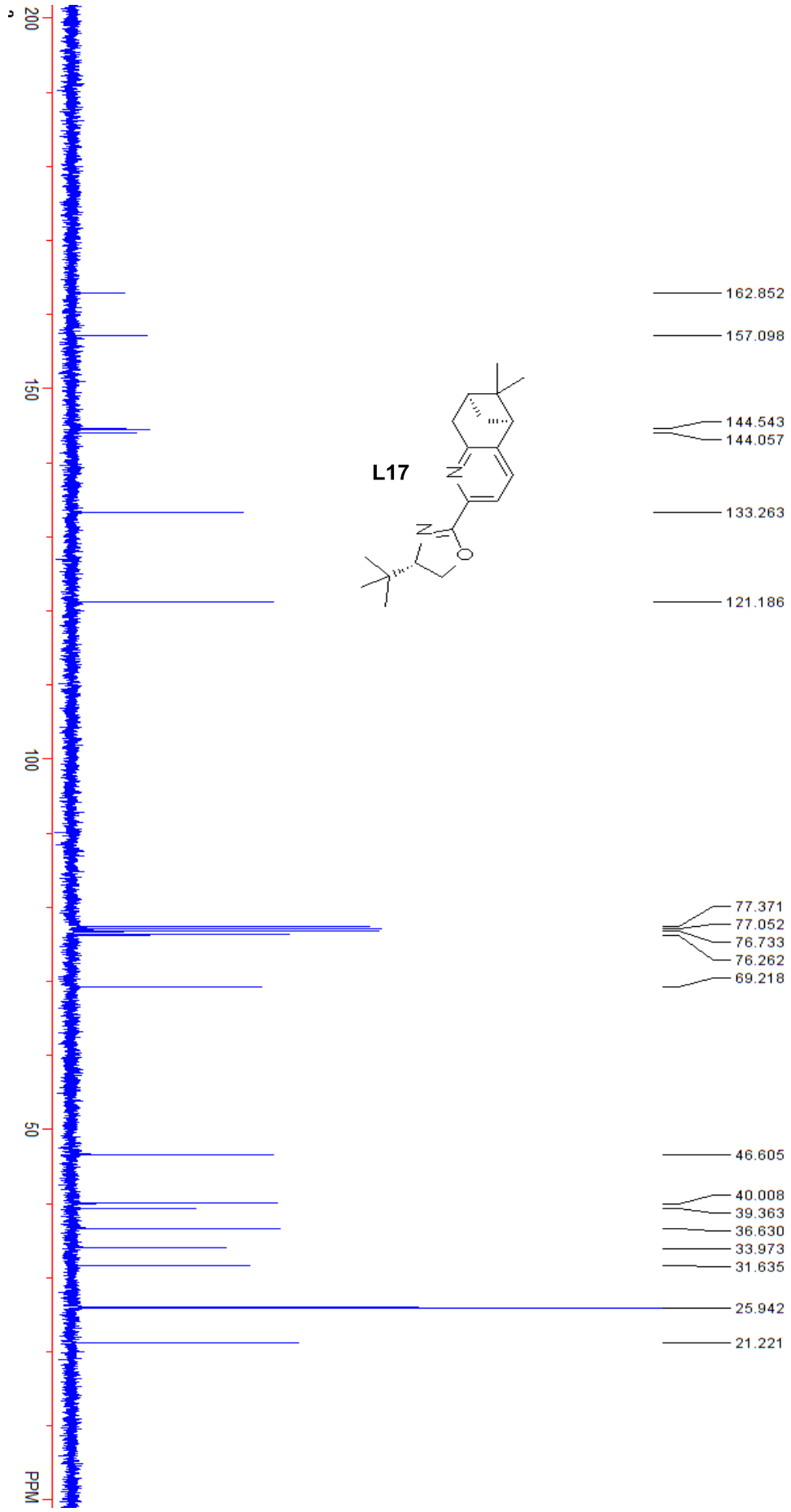
2.359
2.352
2.345
2.337
1.379
1.242
1.218
0.943
0.610
PPM


200
150
100
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PPM
L17
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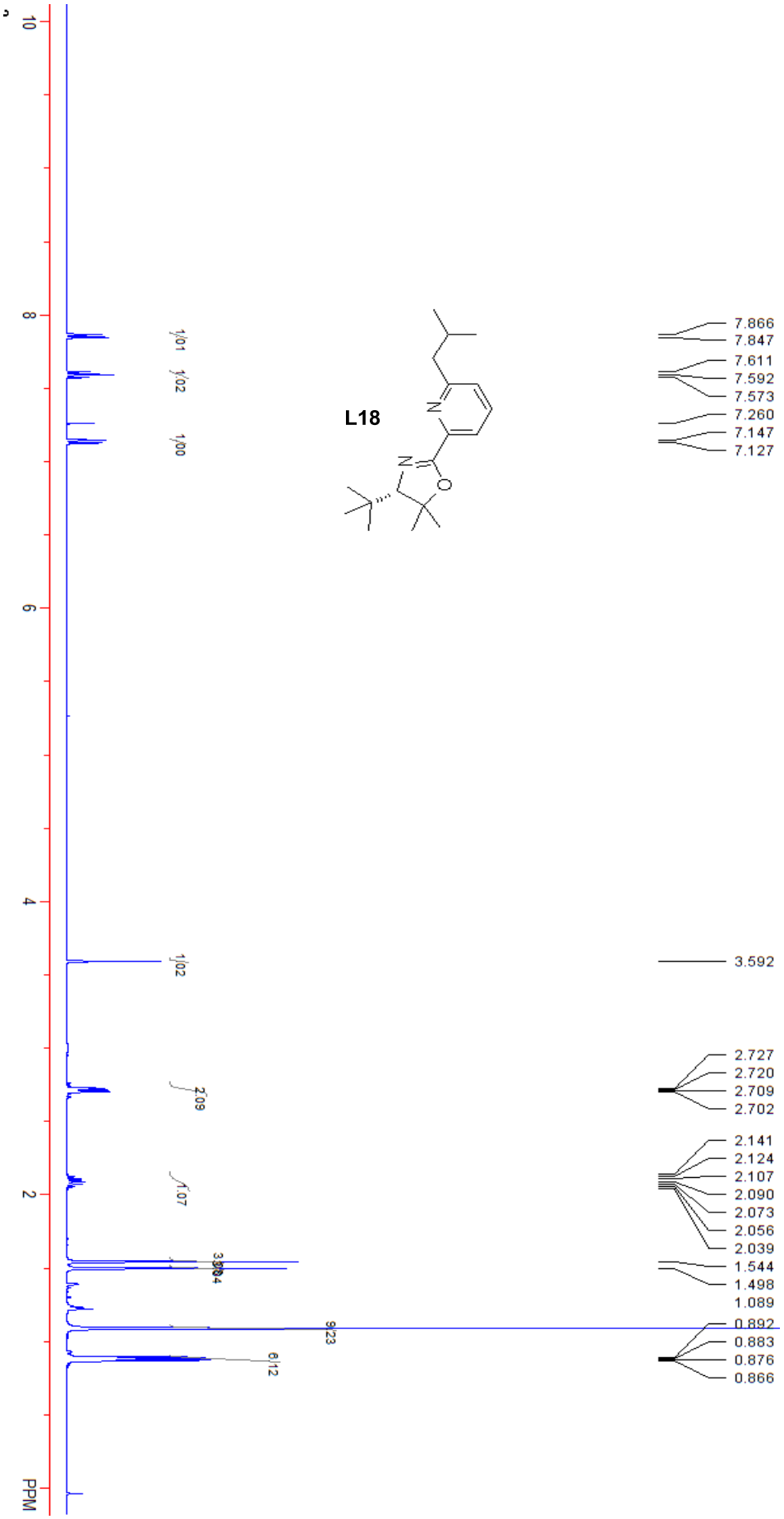
157.098
144.543
144.057
133.263
121.186
77.371
77.052
76.733
76.262
69.218
46.605
40.008
39.363
36.630
33.973
31.635
25.942
21.221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7.847
7.611
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7.573
7.260
7.147
7.127
3.592
2.727
2.720
2.709
2.702
2.141
2.124
2.107
2.090
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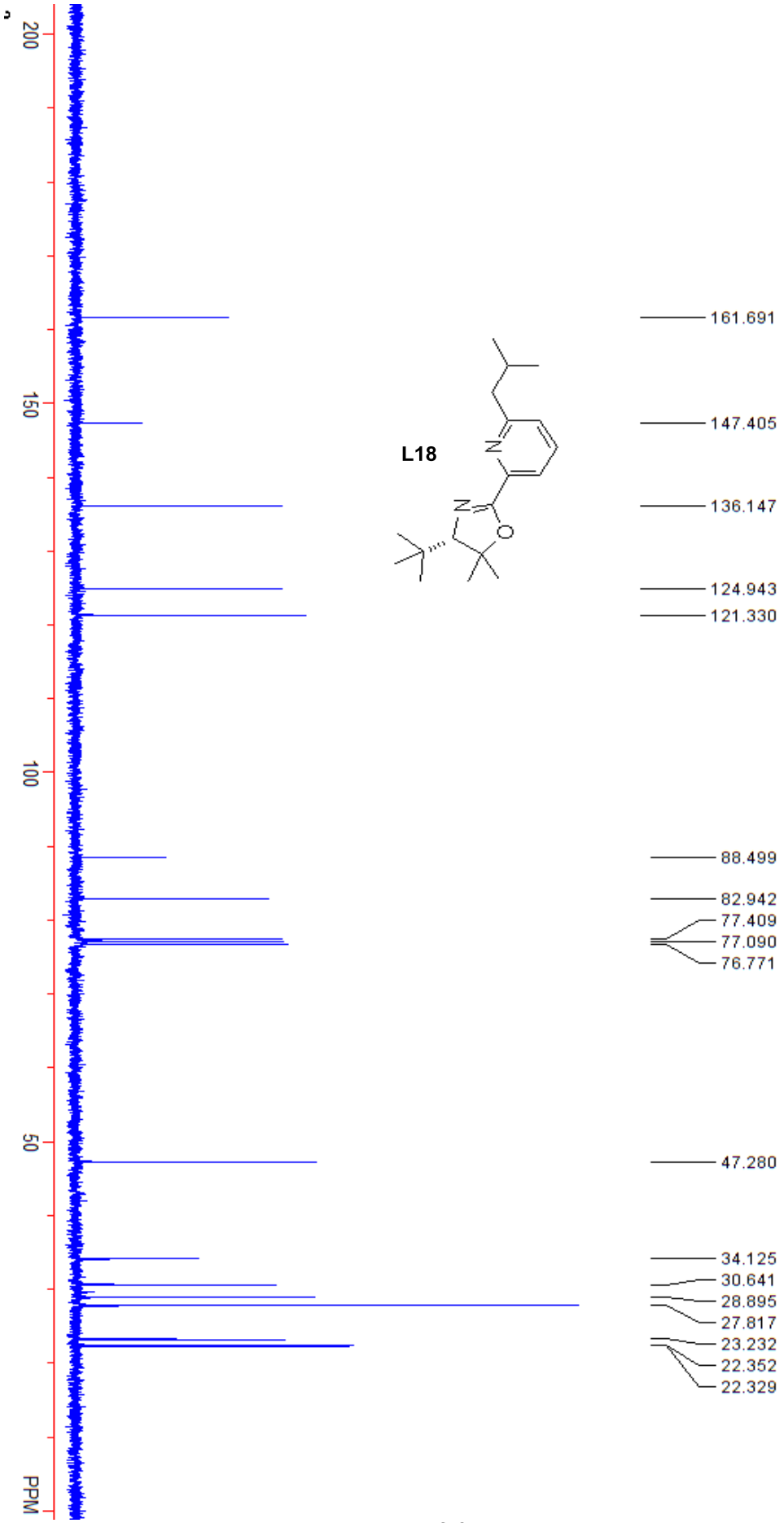
2.056
2.039
1.544
1.498
1.089
0.892
0.883
0.876
0.866
PPM


200
150
100
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PPM
L18
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N
O
161.691
147.405
136.147
124.943
121.330
88.499
82.942
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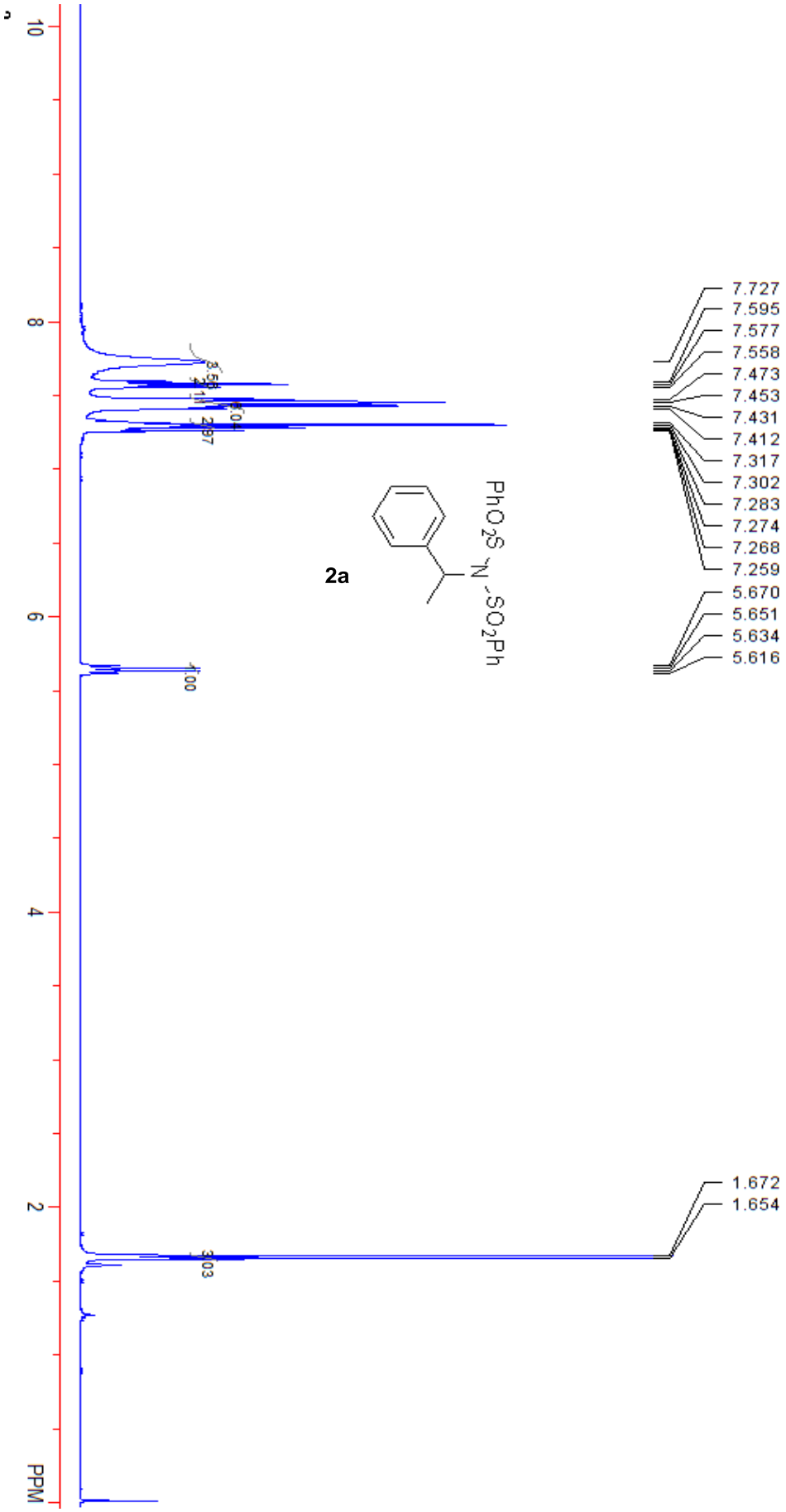
76.771
47.280
34.125
30.641
28.895
27.817
23.232
22.352
22.329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7.577
7.558
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7.453
7.431
7.412
7.317
7.302
7.283
7.274
7.268
7.259
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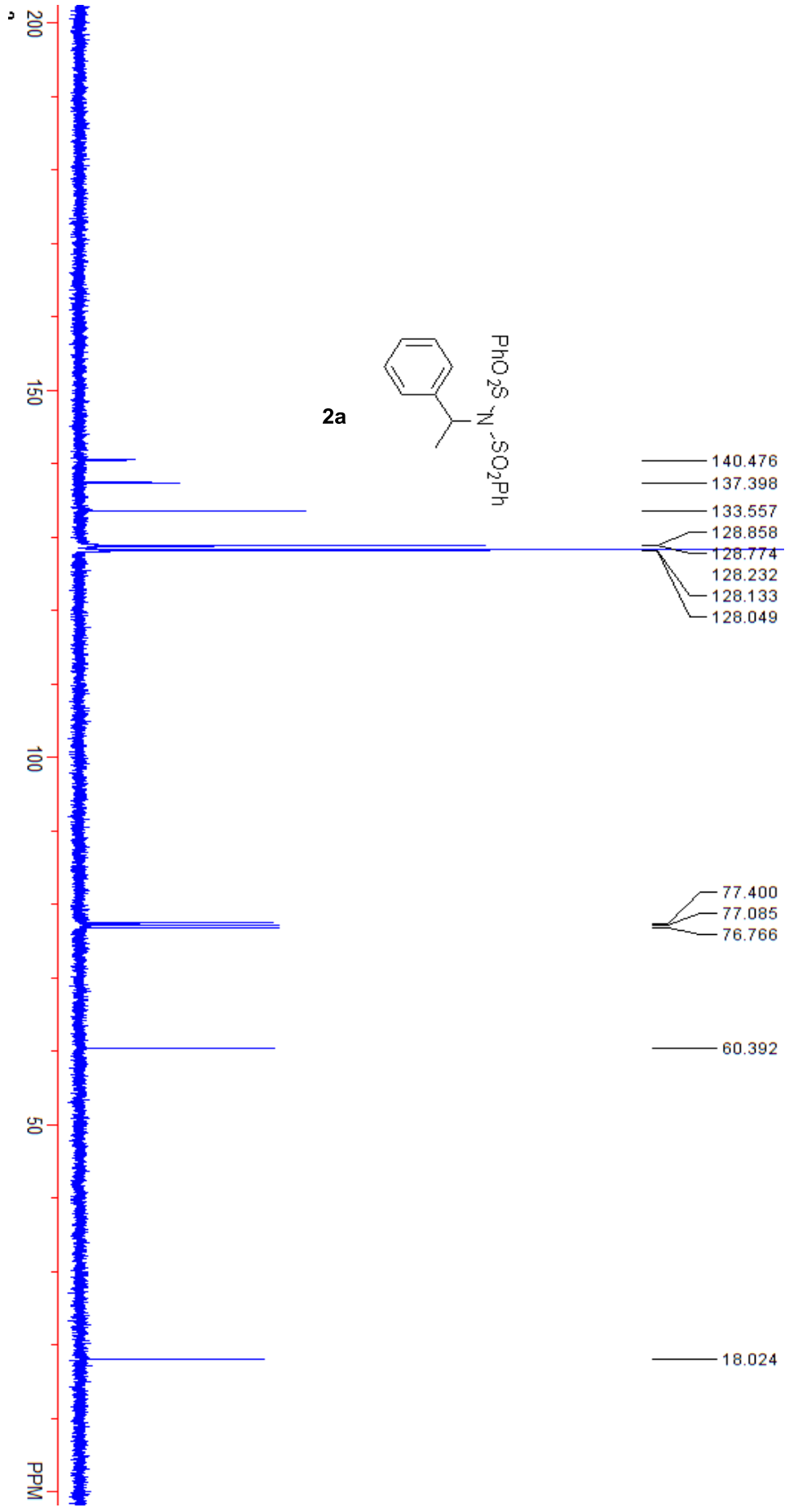
5.651
5.634
5.616
1.672
1.654
2a
PhO2S
N
SO2Ph
PPM


2a
PhO2S
N
SO2Ph
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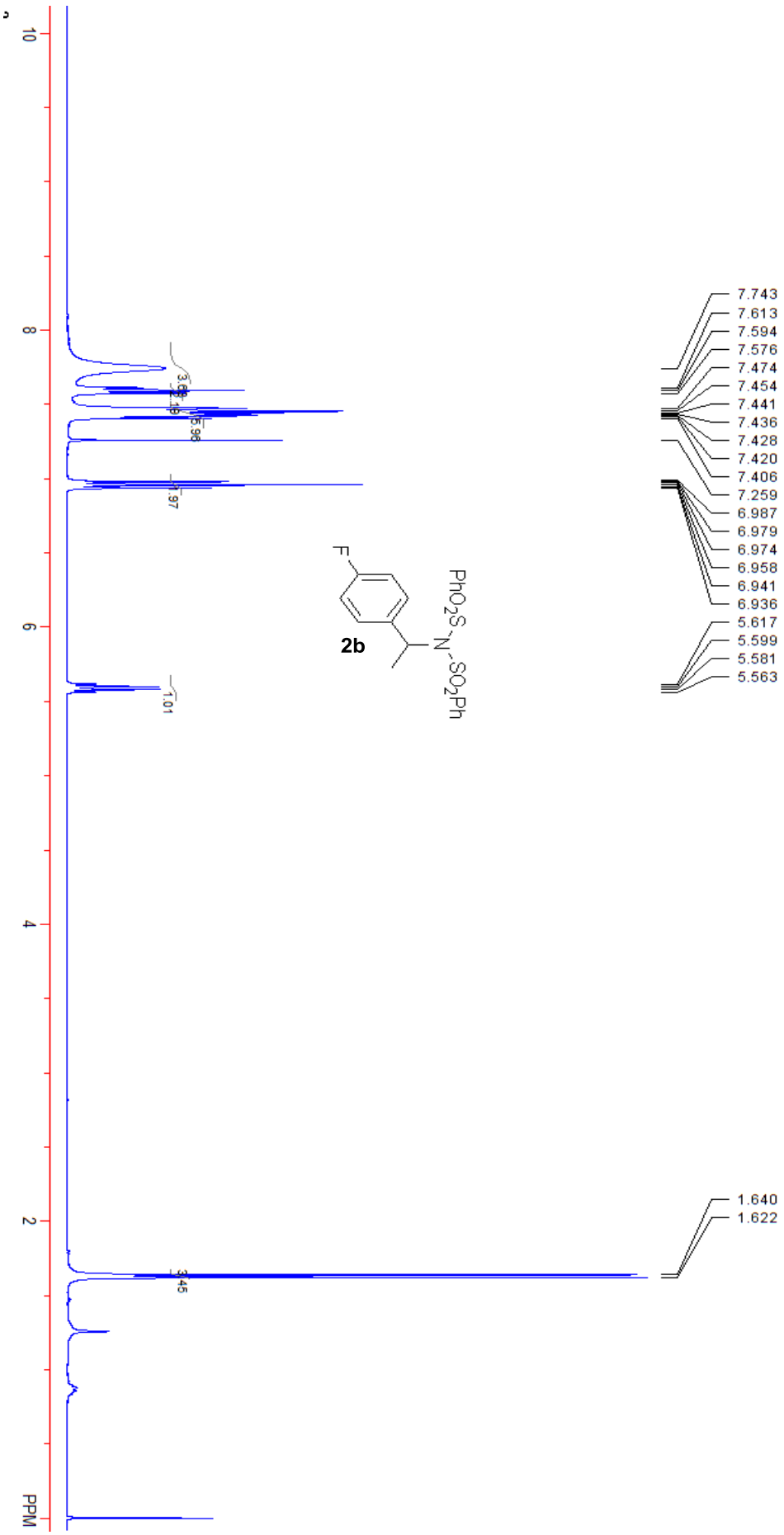
137.398
133.557
128.858
128.774
128.232
128.133
128.049
77.400
77.085
76.766
60.392
18.024
200
150
100
50
PPM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7.594
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7.454
7.441
7.436
7.428
7.420
7.406
7.259
6.987
6.979
6.974
6.958
6.941
6.936
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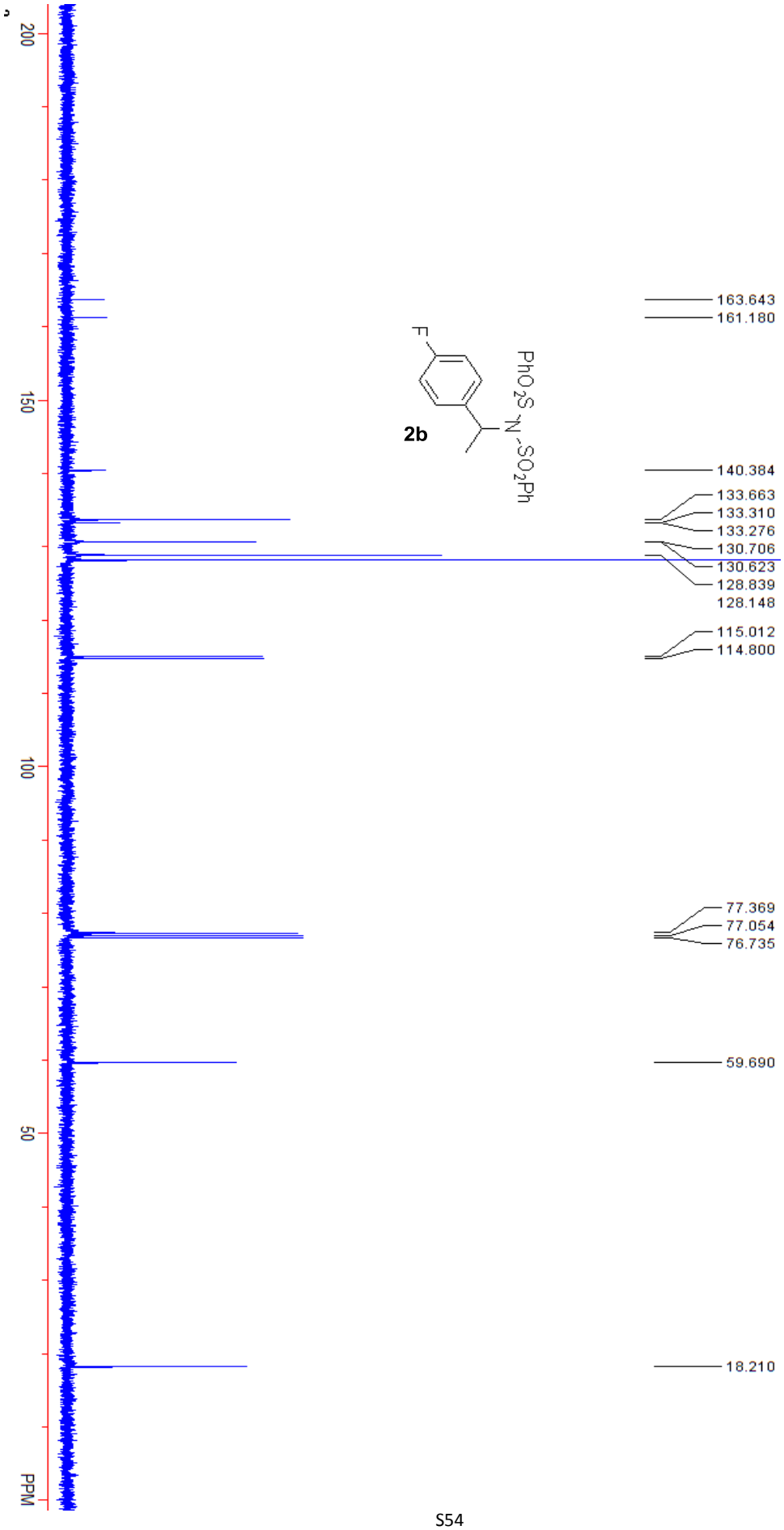
5.599
5.581
5.563
1.640
1.622
2b
F
PhO2S
N
SO2Ph
PPM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150
100
50
PPM
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161.180
140.384
133.663
133.310
133.276
130.706
130.623
128.839
128.148
115.012
114.800
77.369
77.054
76.735
59.690
18.210
F
PhO2S
N
SO2Ph
2b


7.748
7.621
7.603
7.584
7.479
7.459
7.440
7.371
7.349
7.260
7.238
7.217
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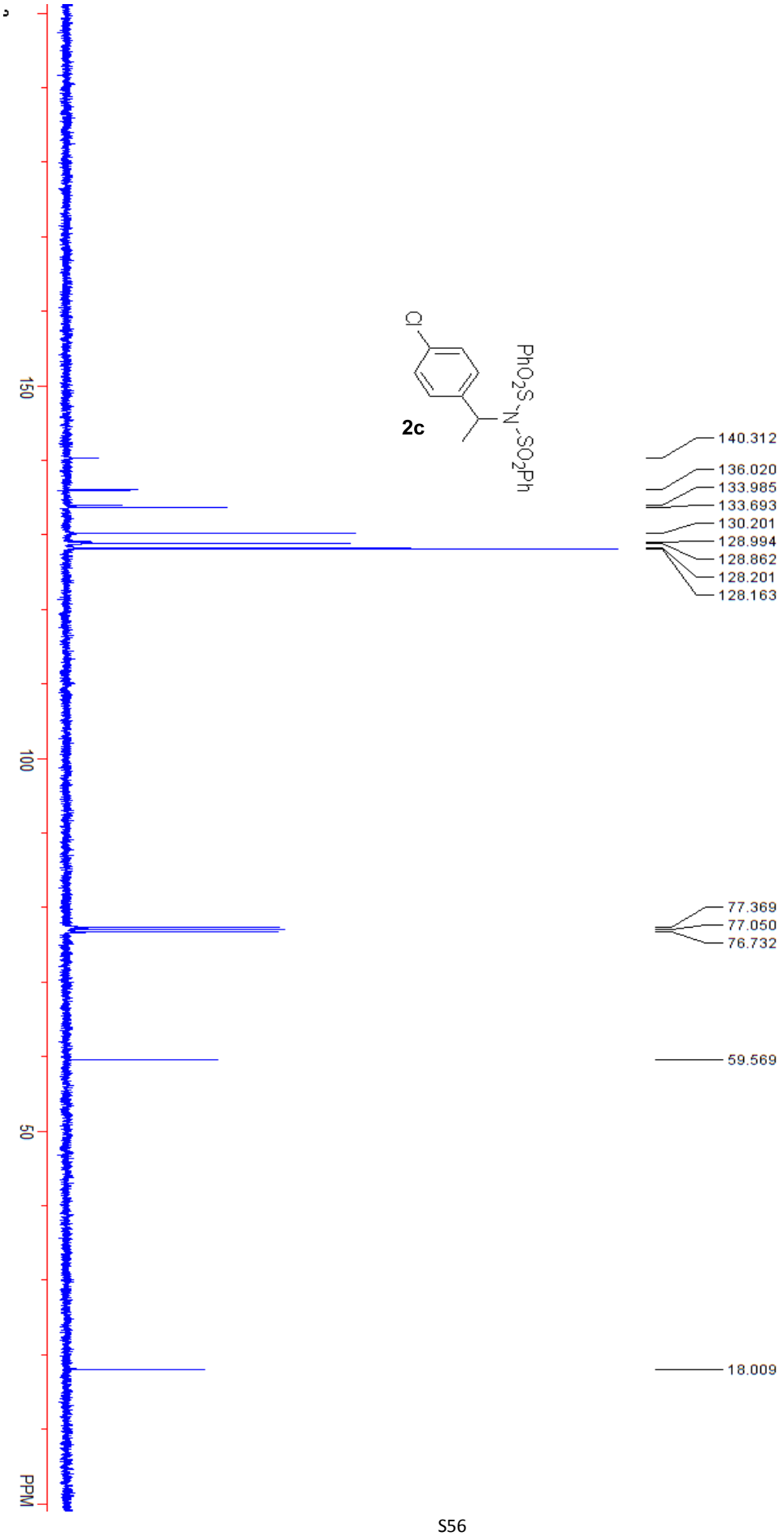
5.586
5.568
5.551
1.637
1.619
Cl
PhO2S
N
SO2Ph
2c
PPM


Cl
PhO2S
N
SO2Ph
2c
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136.020
133.985
133.693
130.201
128.994
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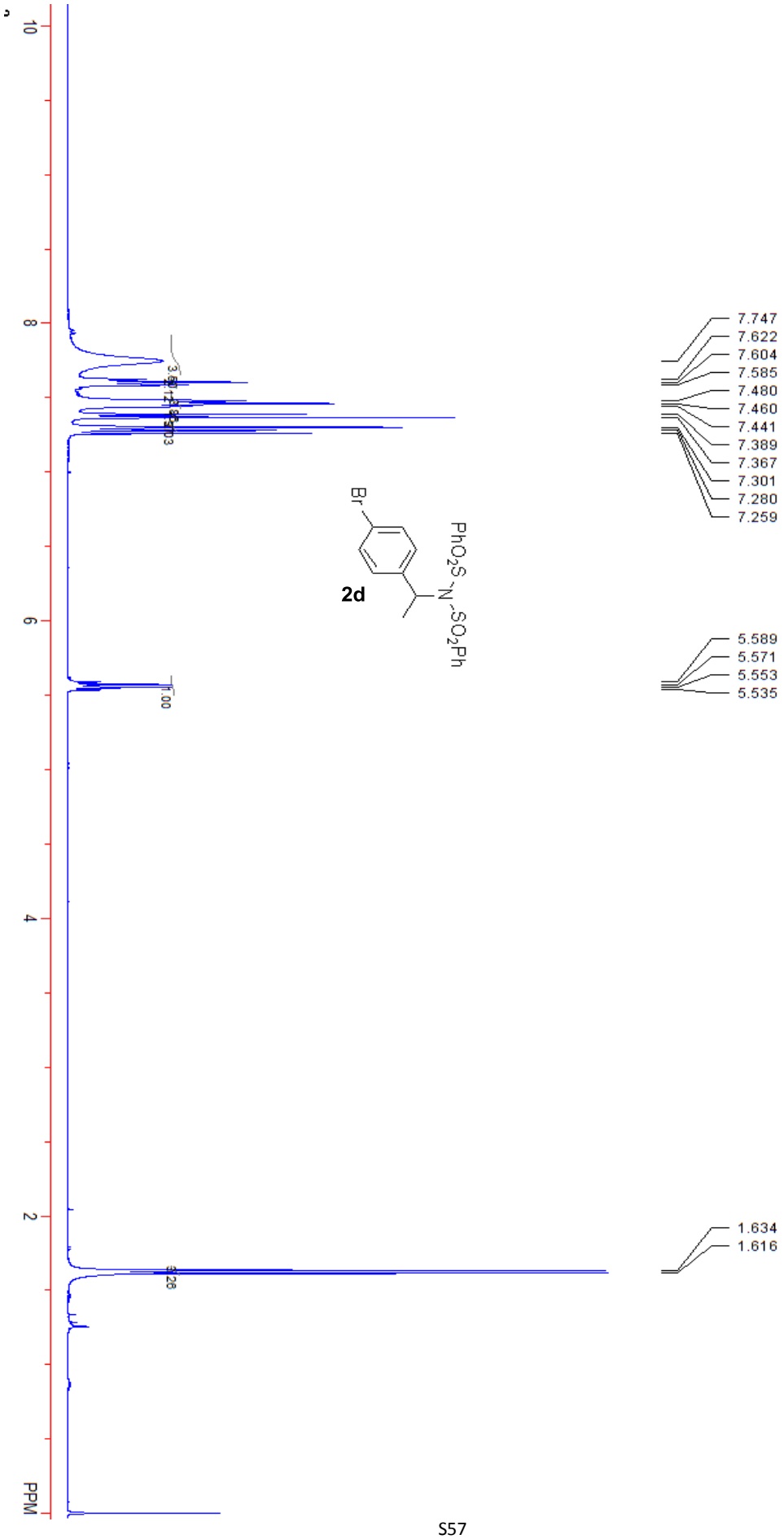
128.201
128.163
77.369
77.050
76.732
59.569
18.009
150
100
50
PPM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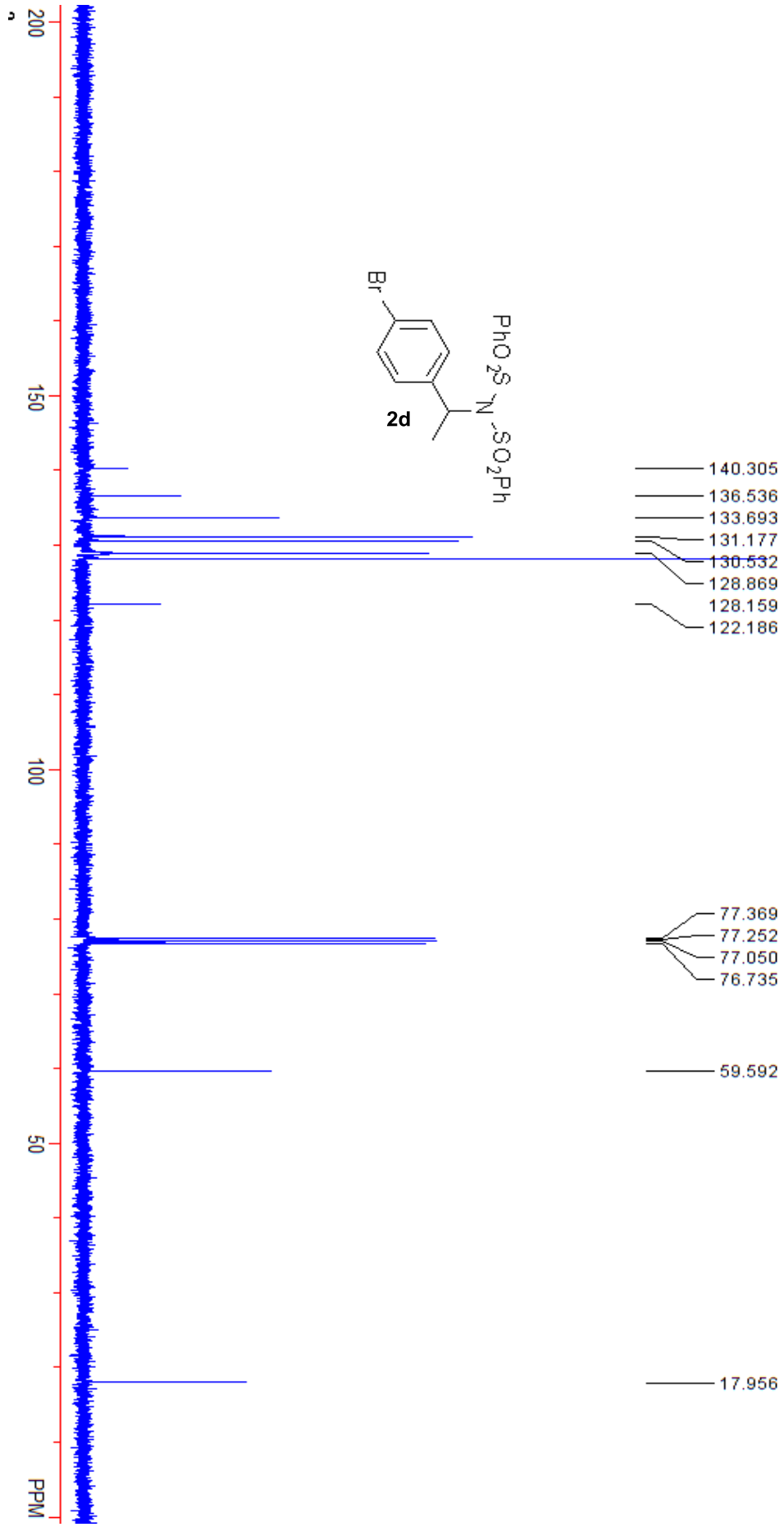
7.585
7.480
7.460
7.441
7.389
7.367
7.301
7.280
7.259
5.589
5.571
5.553
5.535
1.634
1.616
Br
PhO2S
N
SO2Ph
2d
PPM


200
150
100
50
PPM
Br
PhO2S
N
SO2Ph
2d
140.305
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128.869
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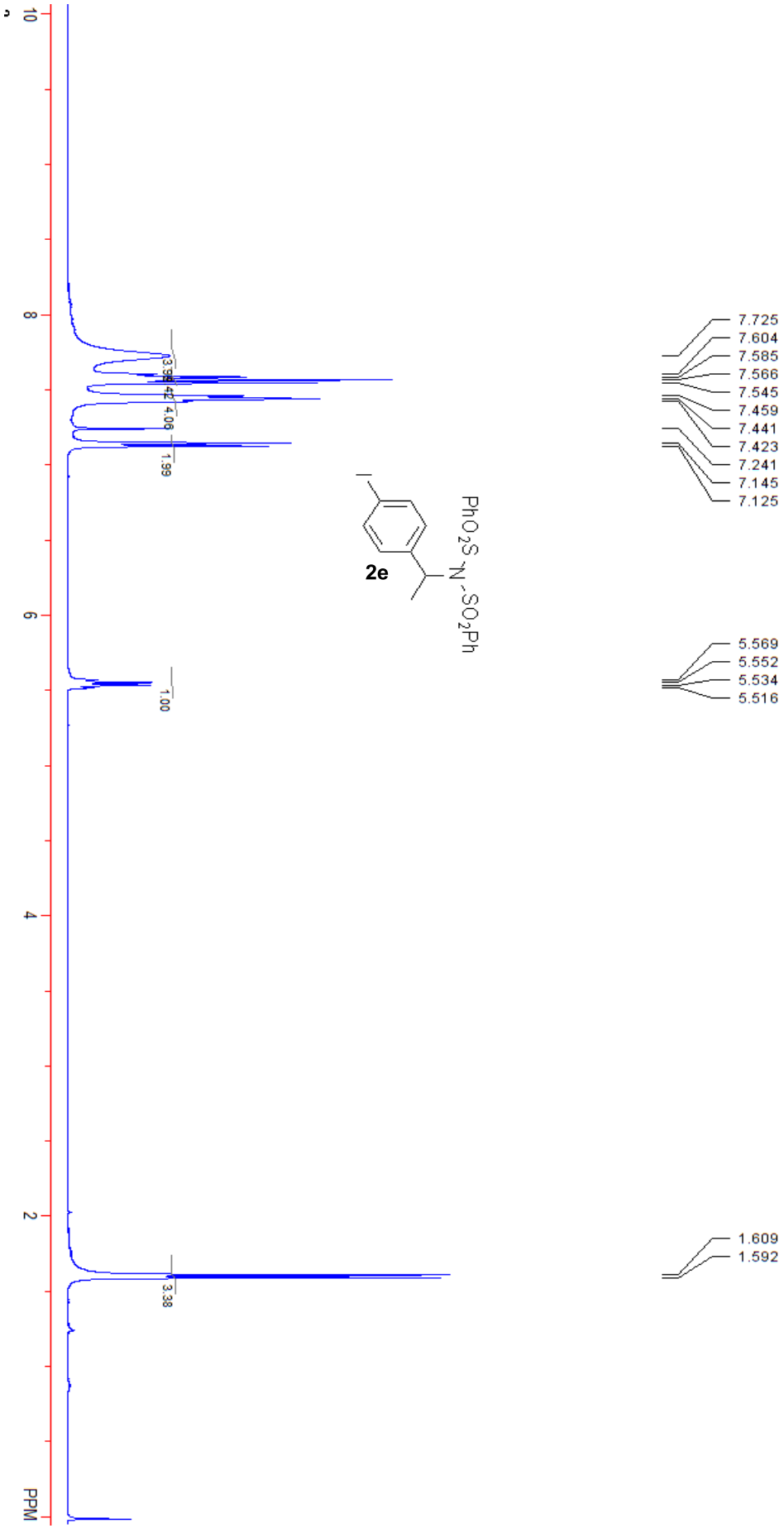
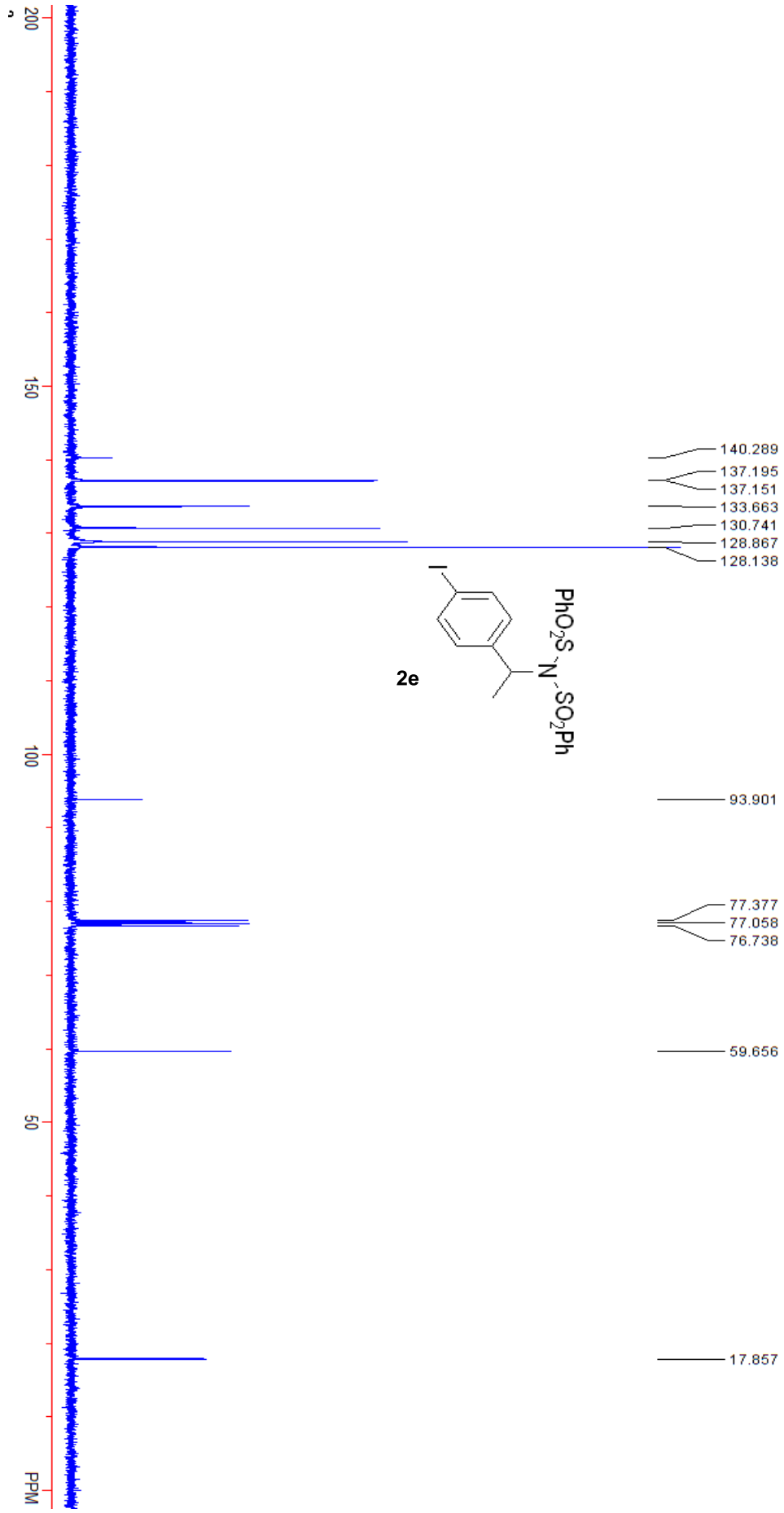
122.186
77.369
77.252
77.050
76.735
59.592
17.956


10
8
6
4
2
PPM
4.06
1.99
1.00
3.38
2e
PhO2S
N
SO2Ph
7.725
7.604
7.585
7.566
7.545
7.459
7.441
7.423
7.241
7.145
7.125
5.569
5.552
5.534
5.516
1.609
1.592


200
150
100
50
PPM
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137.195
137.151
133.663
130.741
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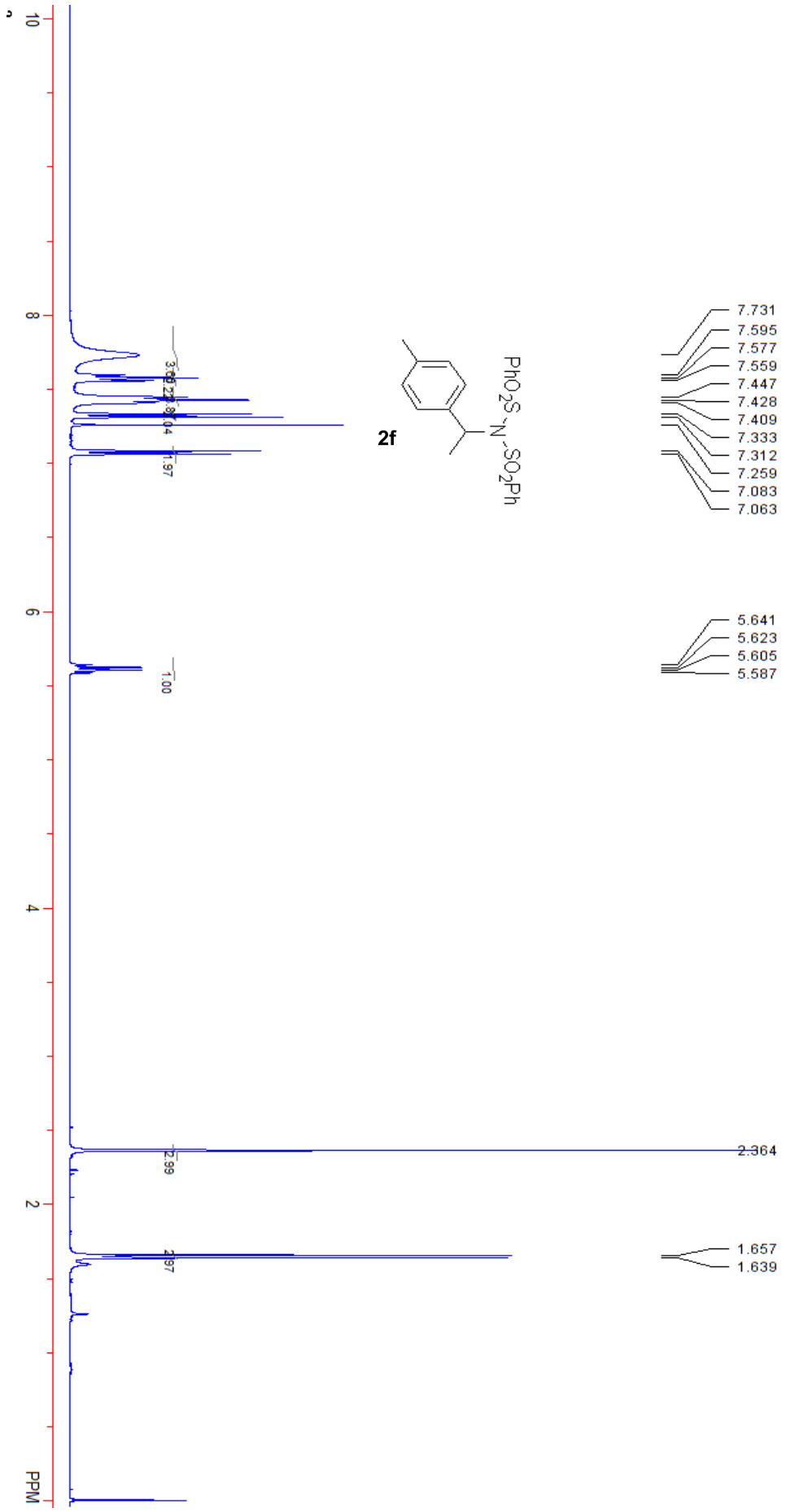
77.377
77.058
76.738
59.656
17.857
2e
I
PhO2S
N
SO2Ph


2f
PhO2S
N
SO2Ph
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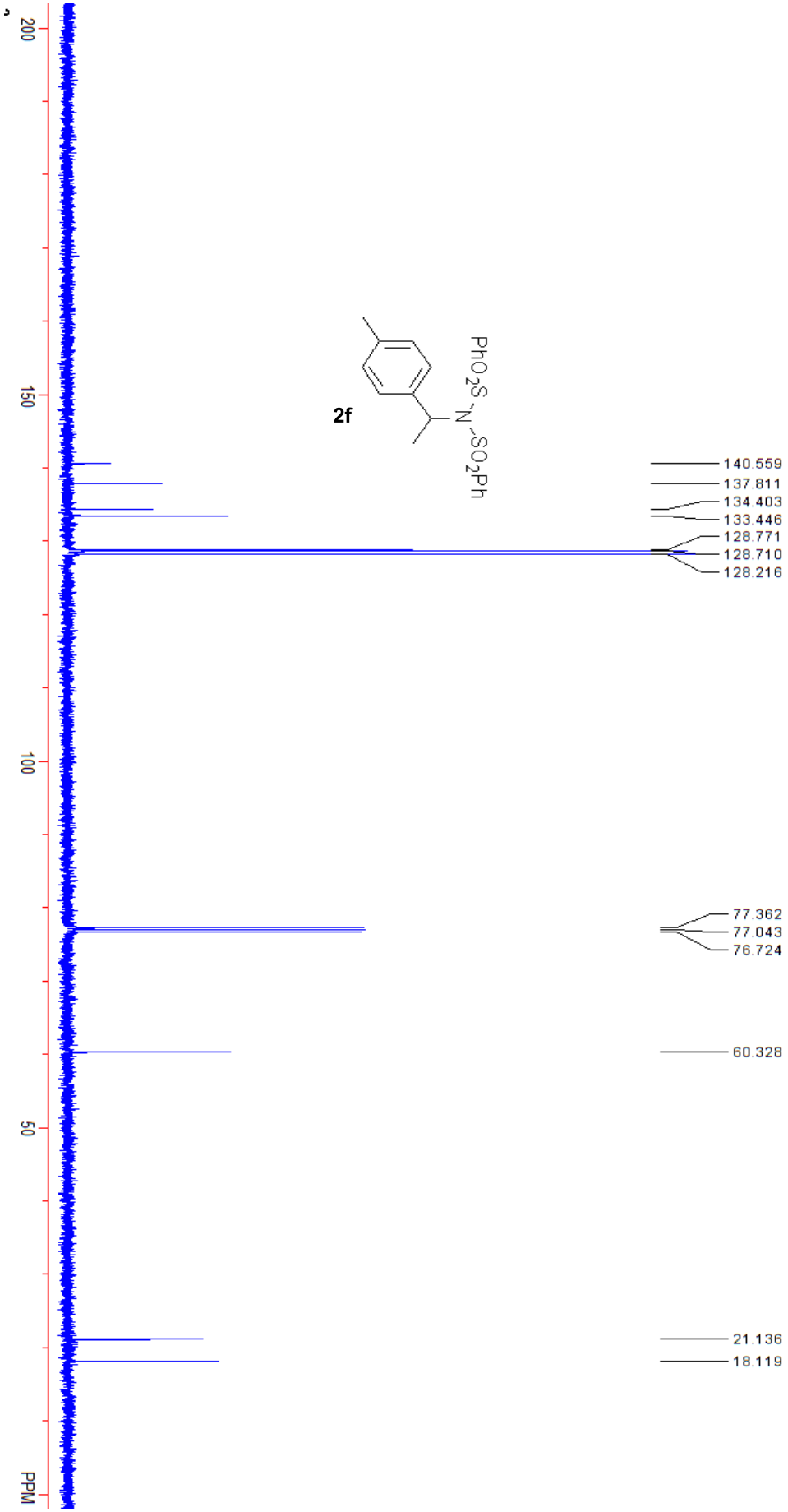
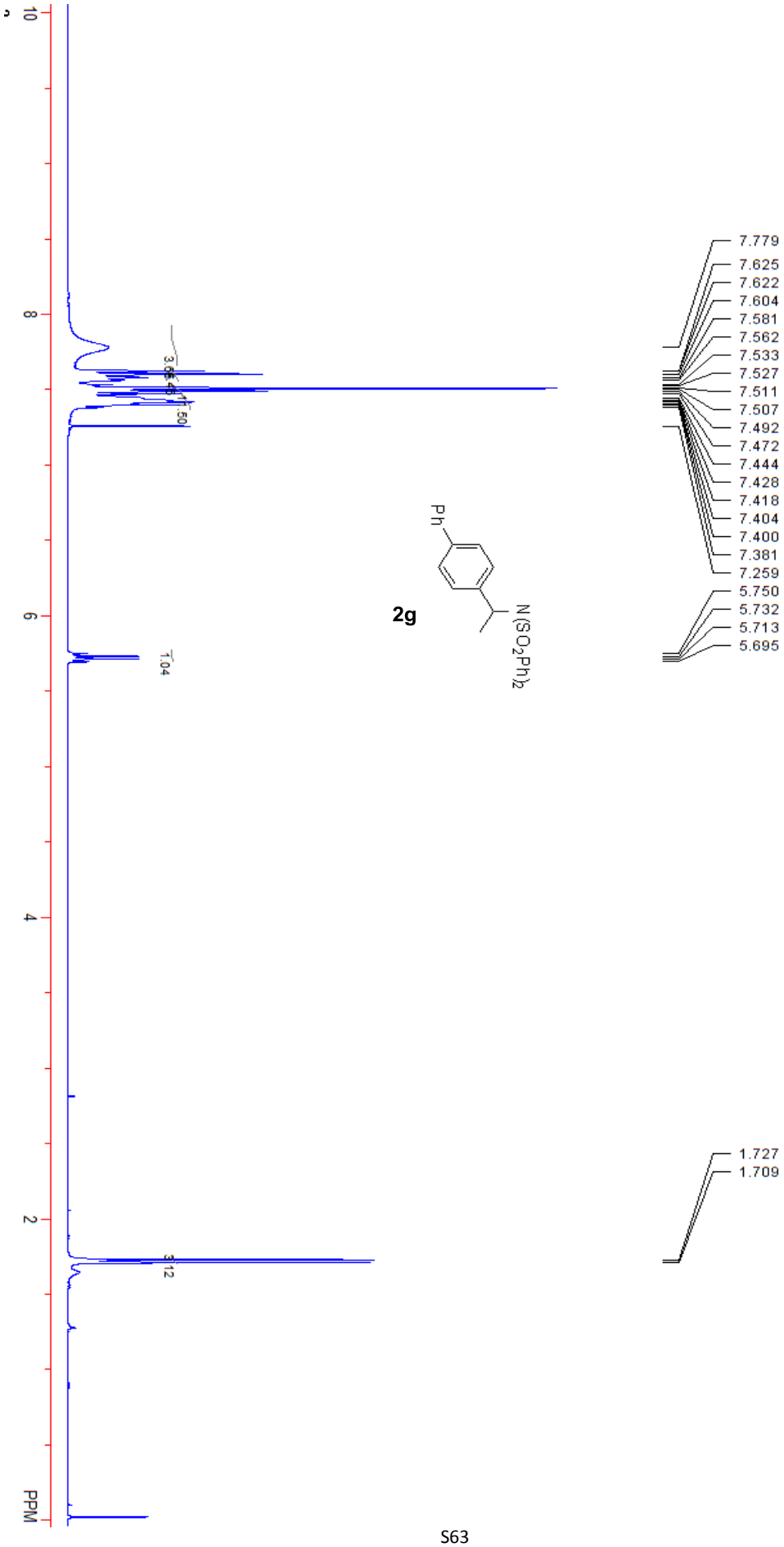
7.595
7.577
7.559
7.447
7.428
7.409
7.333
7.312
7.259
7.083
7.063
5.641
5.623
5.605
5.587
2.364
1.657
1.639
3.09
2.23
3.04
1.97
1.00
2.99
2.97
PPM


200
150
100
50
PPM
2f
PhO2S
N
SO2Ph
140.559
137.811
134.403
133.446
128.771
128.710
128.216
77.362
77.043
76.724
60.328
21.136
18.119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7.581
7.562
7.533
7.527
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7.507
7.492
7.472
7.444
7.428
7.418
7.404
7.400
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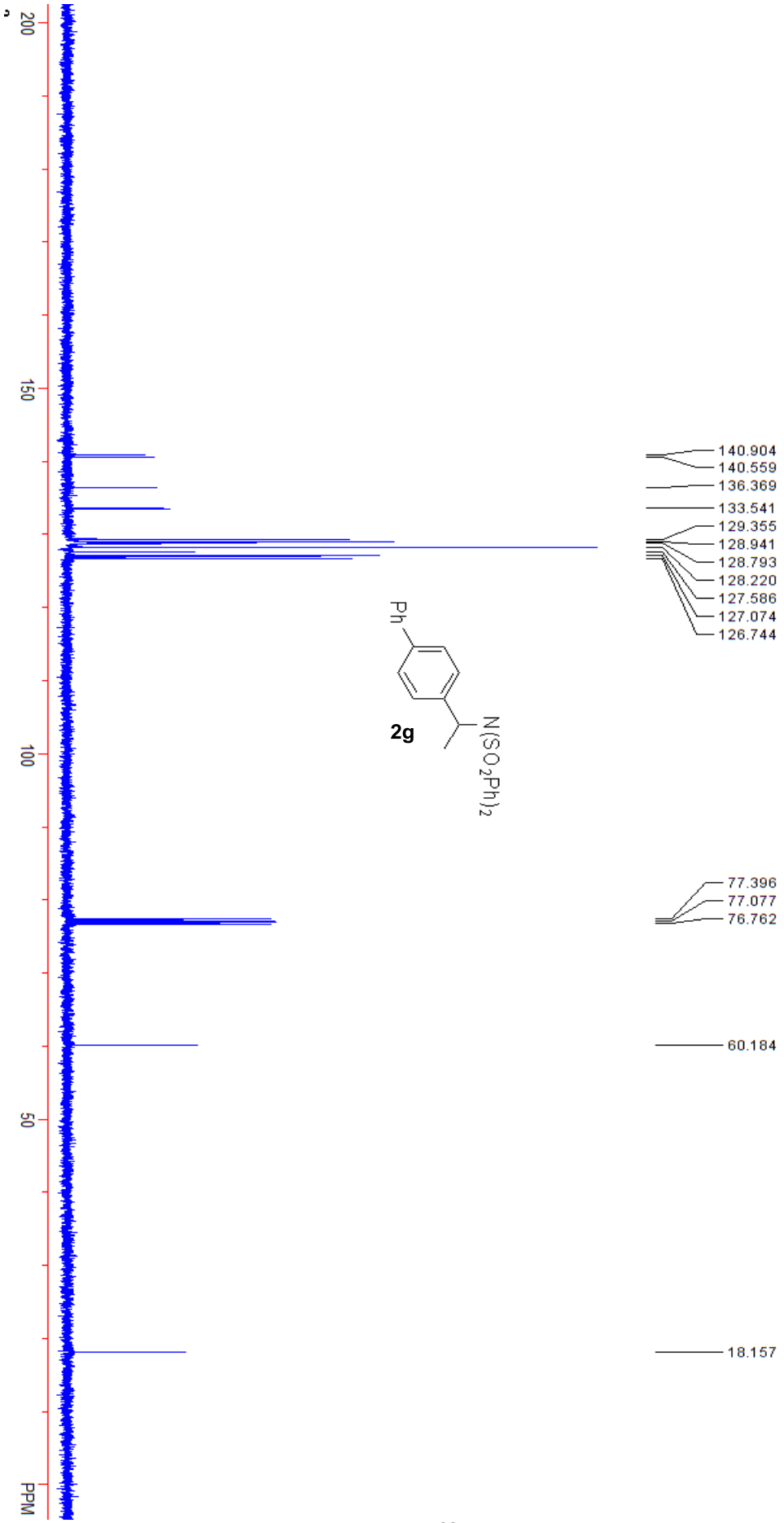
7.259
5.750
5.732
5.713
5.695
1.727
1.709
Ph
N(SO2Ph)2
2g
PPM


200
150
100
50
PPM
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140.559
136.369
133.541
129.355
128.941
128.793
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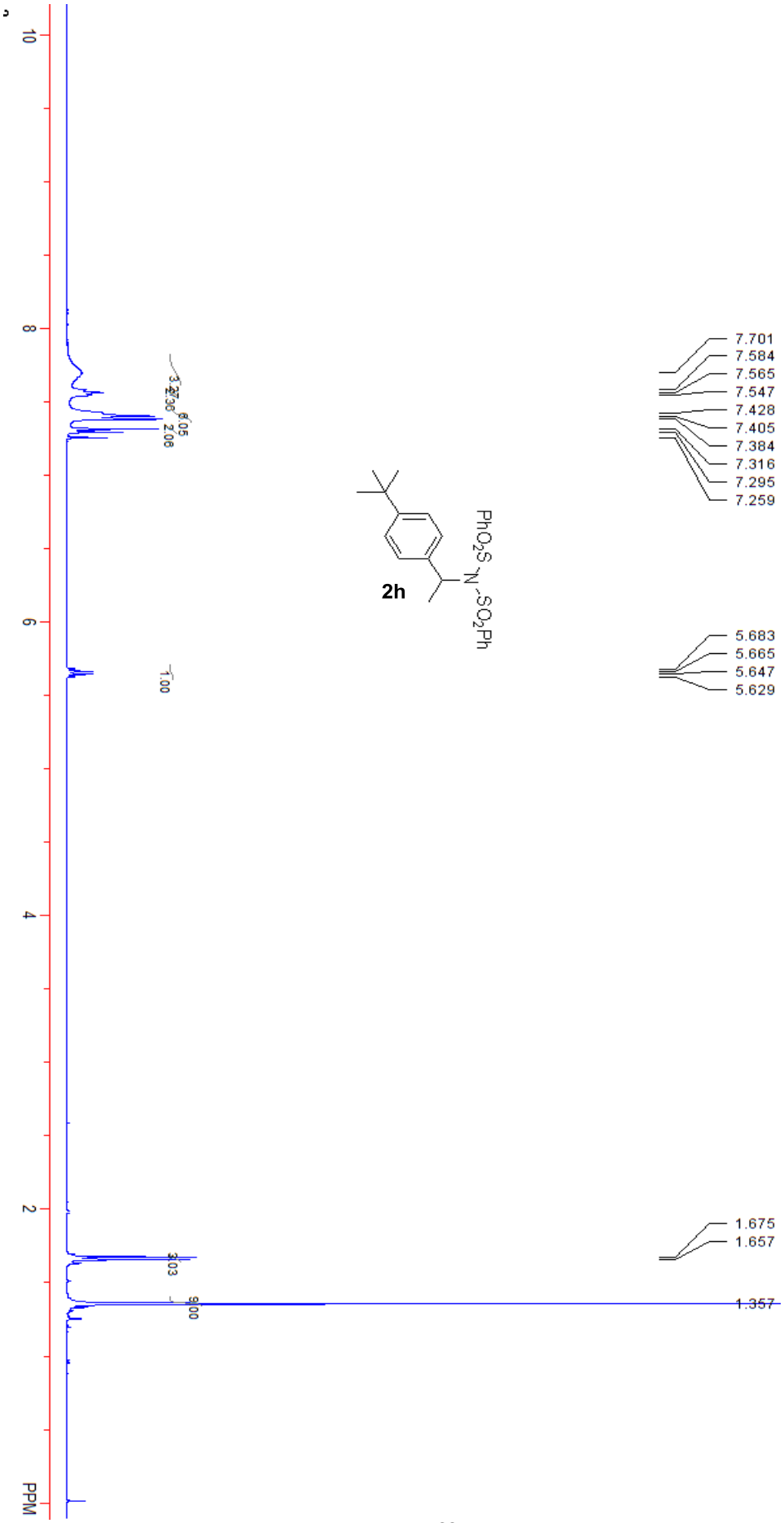
126.744
77.396
77.077
76.762
60.184
18.157
Ph
N(SO2Ph)2
2g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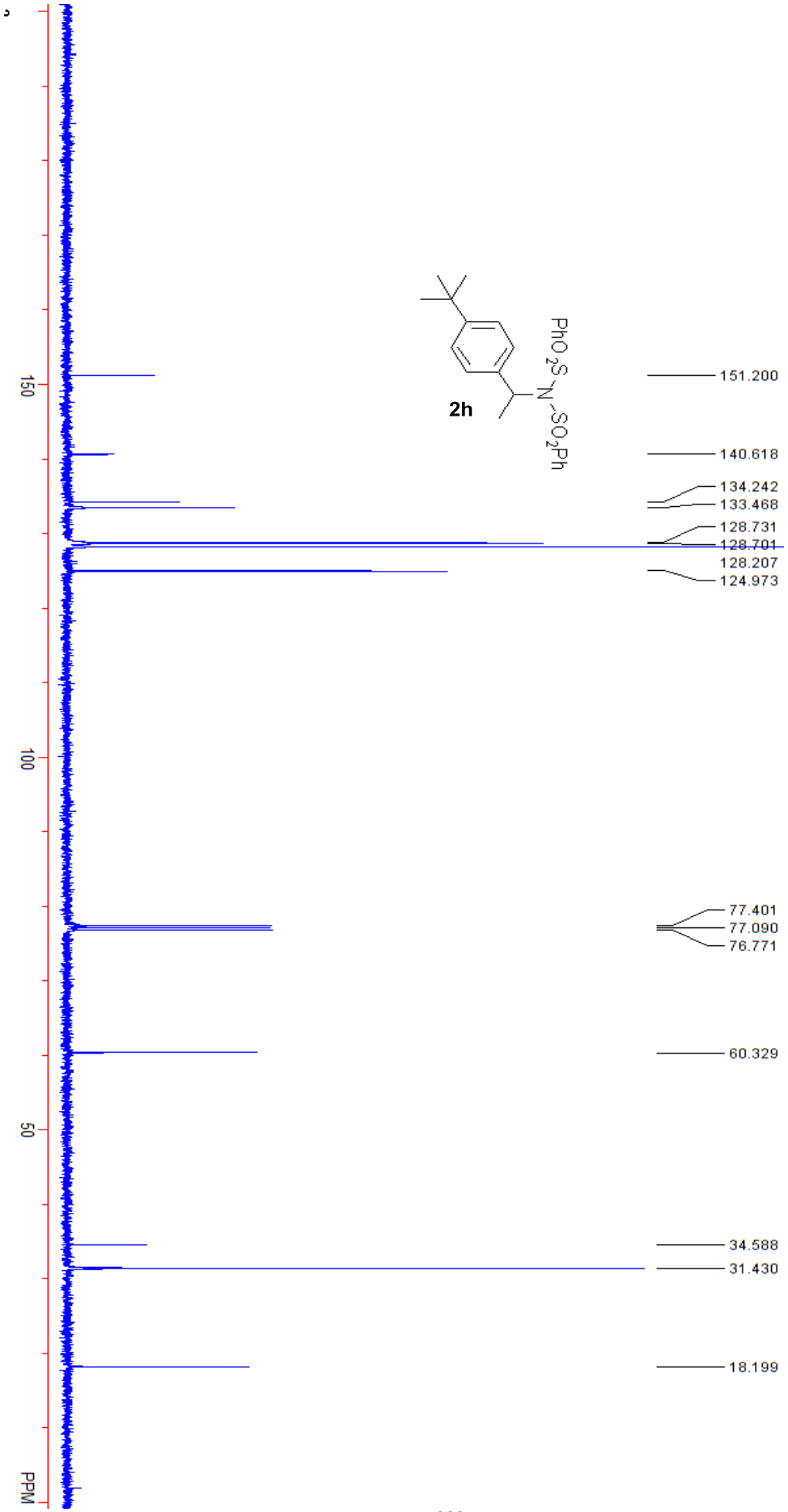
7.584
7.565
7.547
7.428
7.405
7.384
7.316
7.295
7.259
5.683
5.665
5.647
5.629
1.675
1.657
1.357
3.27
2.08
2.06
1.00
3.03
9.00
9.05
2h
PhO2S
N
SO2Ph
10
8
6
4
2
PPM


2h
PhO2S
N
SO2Ph
151.200
140.618
134.242
133.468
128.731
128.701
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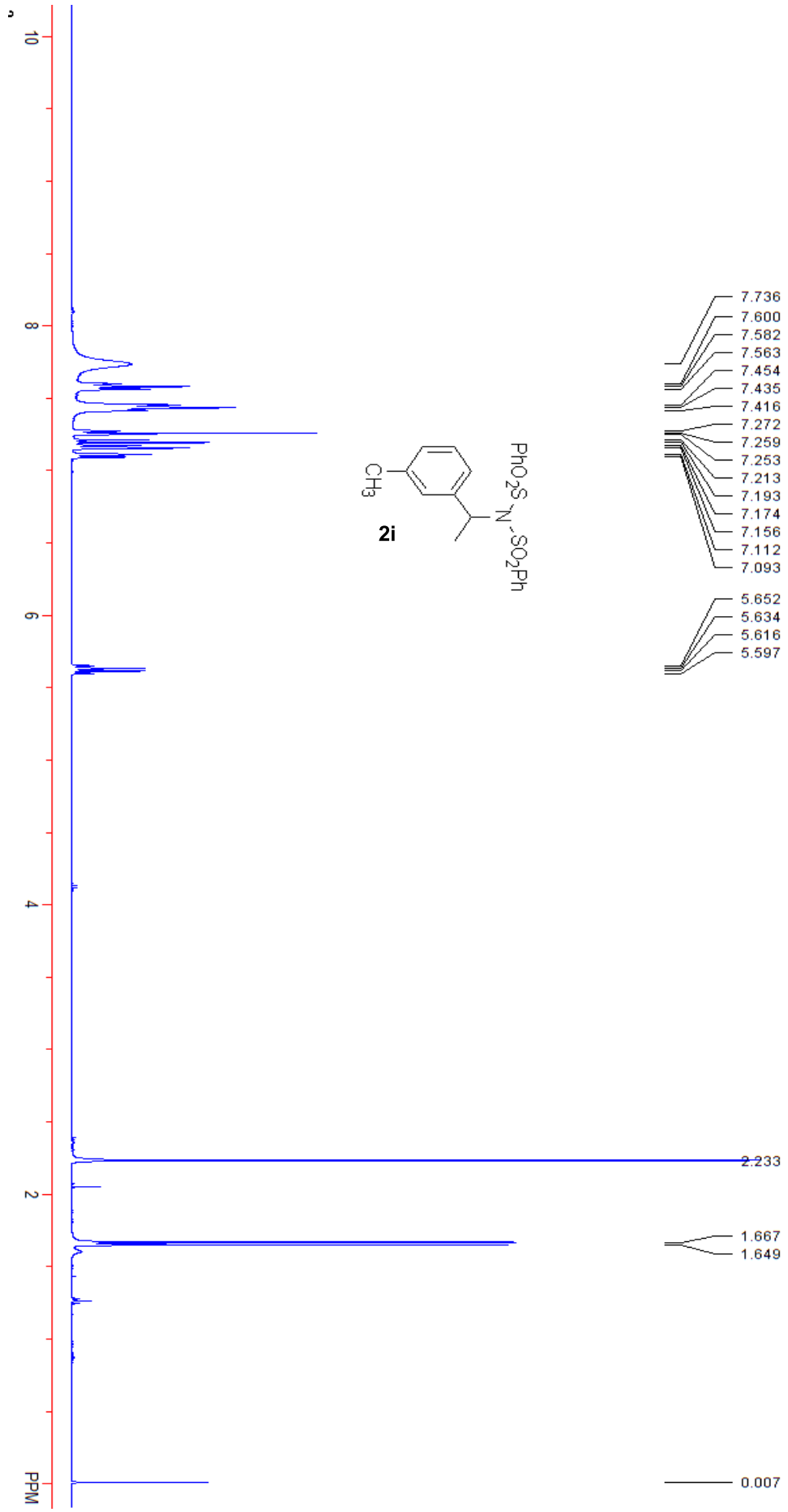
77.401
77.090
76.771
60.329
34.588
31.430
18.199
150
100
50
PPM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7.600
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7.563
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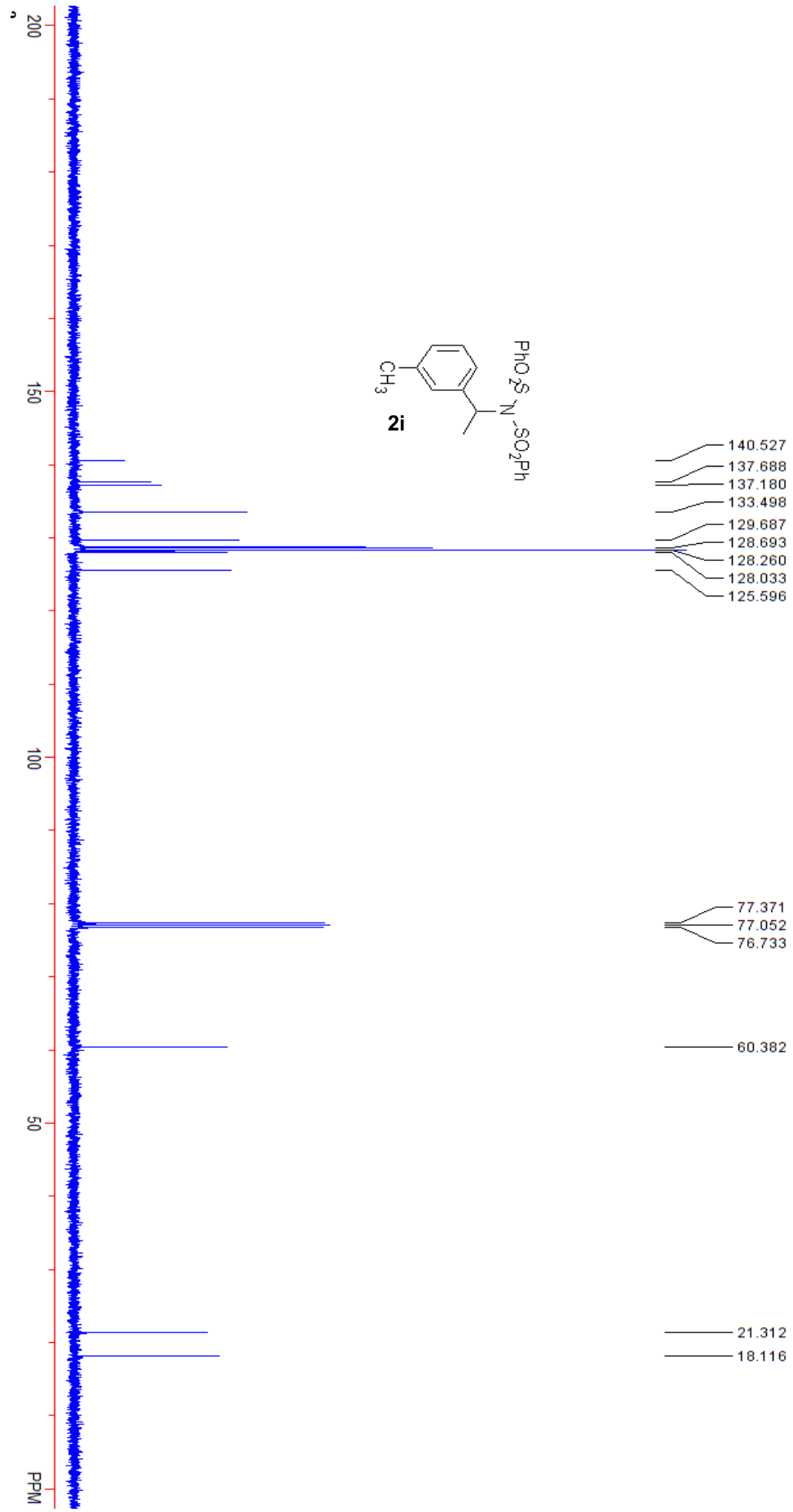
7.435
7.416
7.272
7.259
7.253
7.213
7.193
7.174
7.156
7.112
7.093
5.652
5.634
5.616
5.597
2.233
1.667
1.649
0.007
CH3
PhO2S
N
SO2Ph
2i
10
8
6
4
2
PPM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150
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50
PPM
CH3
PhO2S
N
SO2Ph
2i
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137.180
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128.260
128.033
125.596
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77.052
76.733
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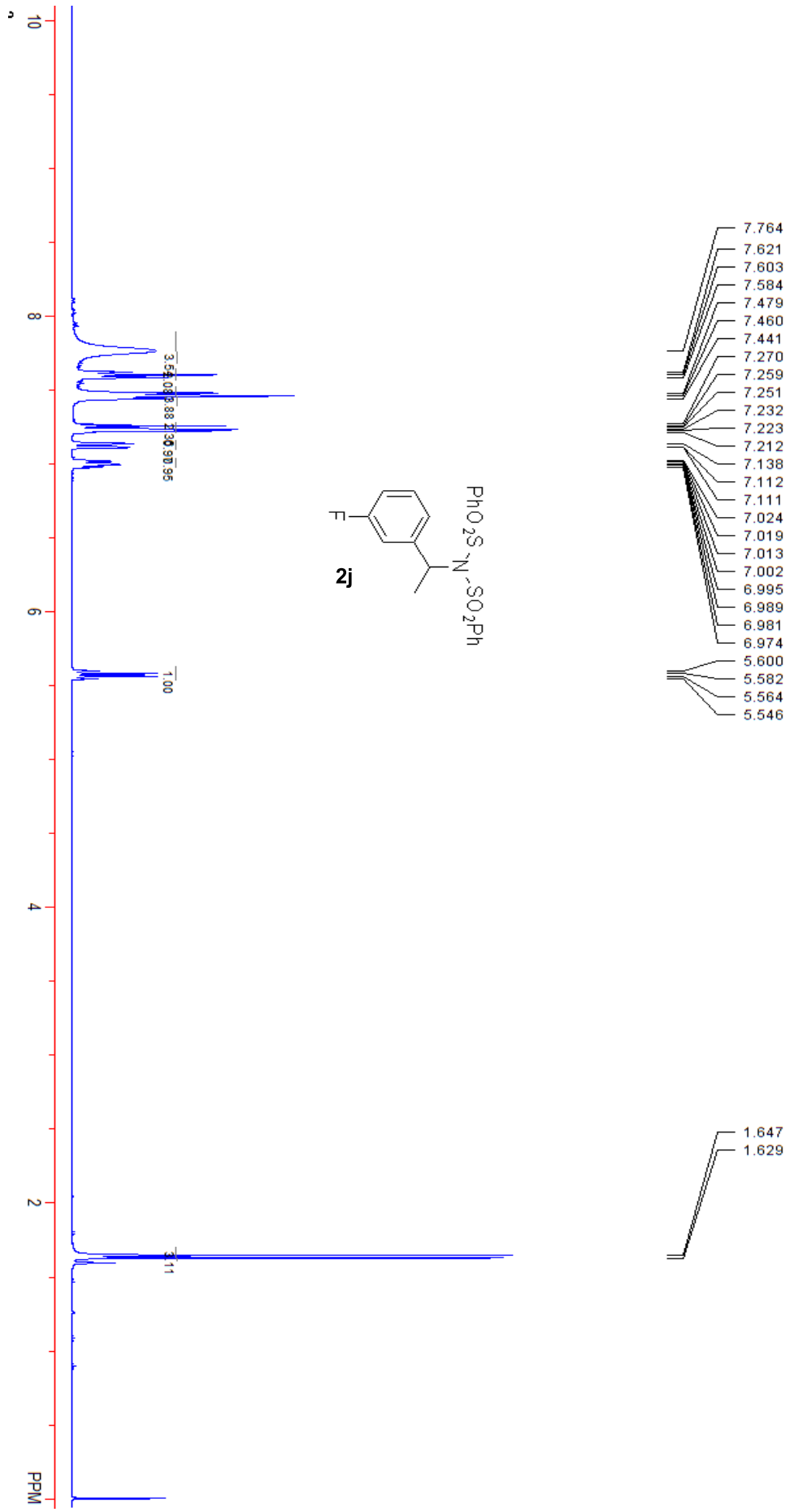
21.312
18.116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7.584
7.479
7.460
7.441
7.270
7.259
7.251
7.232
7.223
7.212
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7.112
7.111
7.024
7.019
7.013
7.002
6.995
6.989
6.981
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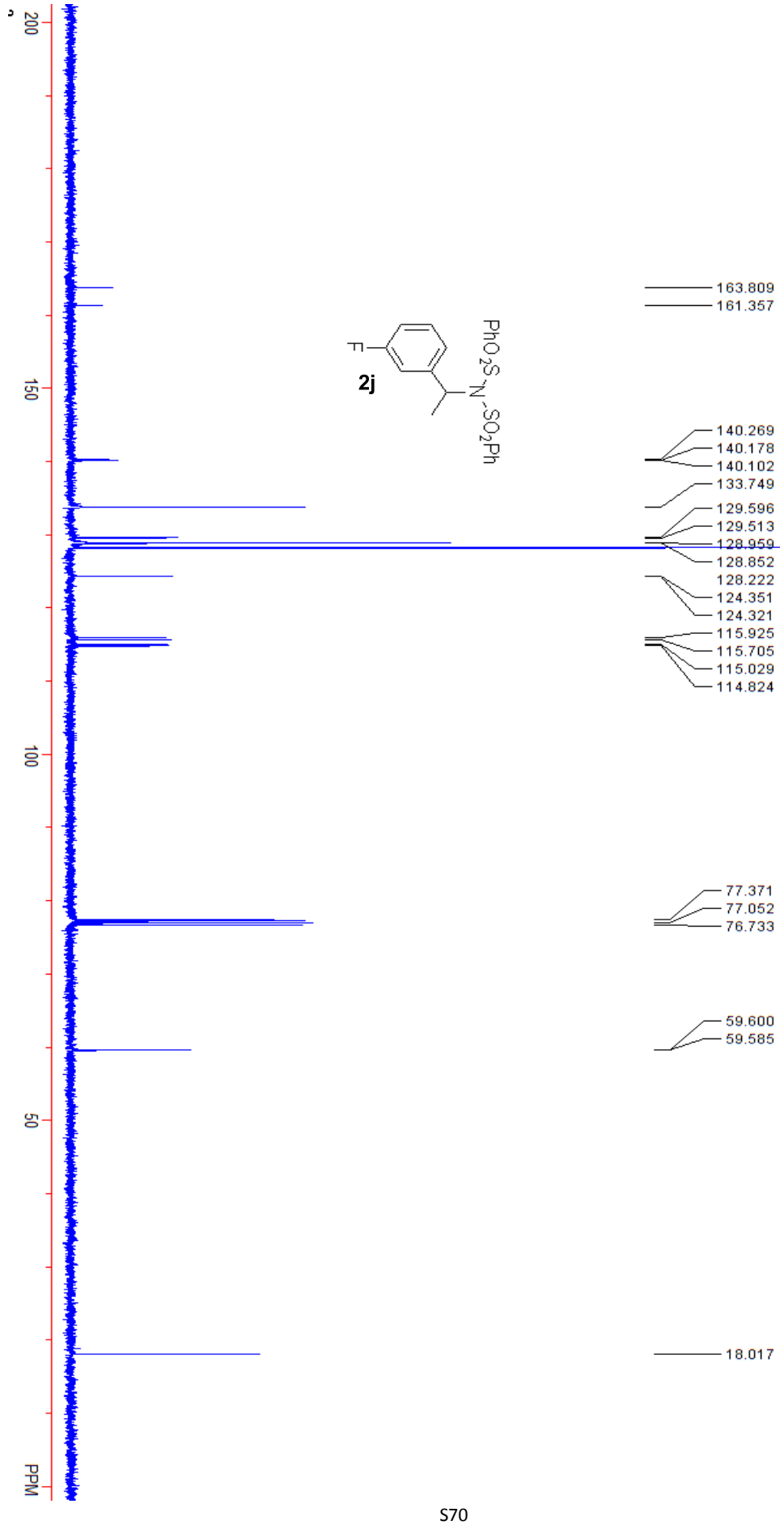
5.564
5.546
1.647
1.629
F
PhO2S
N
SO2Ph
2j
PPM


200
150
100
50
PPM
F
2j
PhO2S
N
SO2Ph
163.809
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140.269
140.178
140.102
133.749
129.596
129.513
128.959
128.852
128.222
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77.052
76.733
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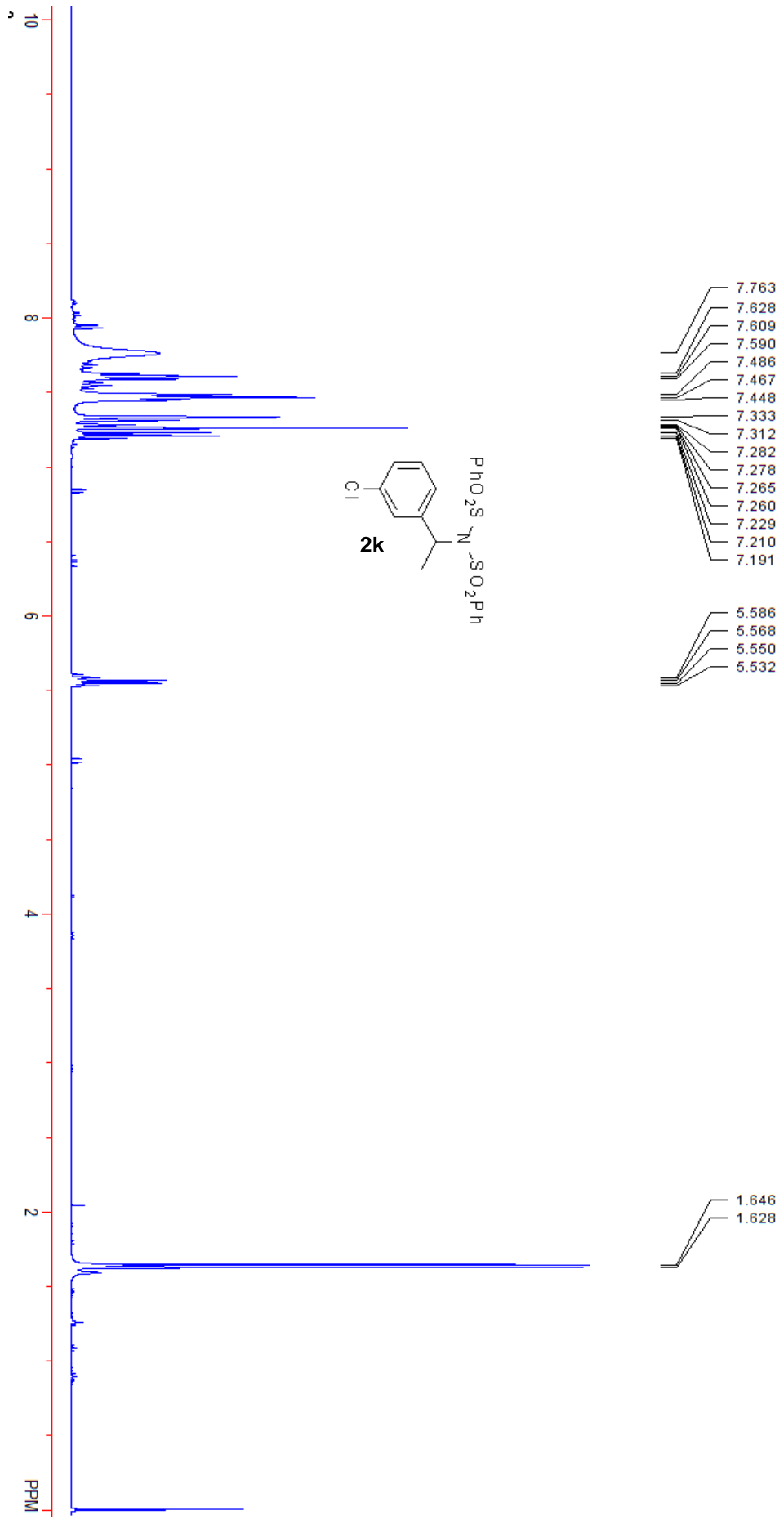
59.585
18.017


10
8
6
4
2
PPM
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7.628
7.609
7.590
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7.467
7.448
7.333
7.312
7.282
7.278
7.265
7.260
7.229
7.210
7.191
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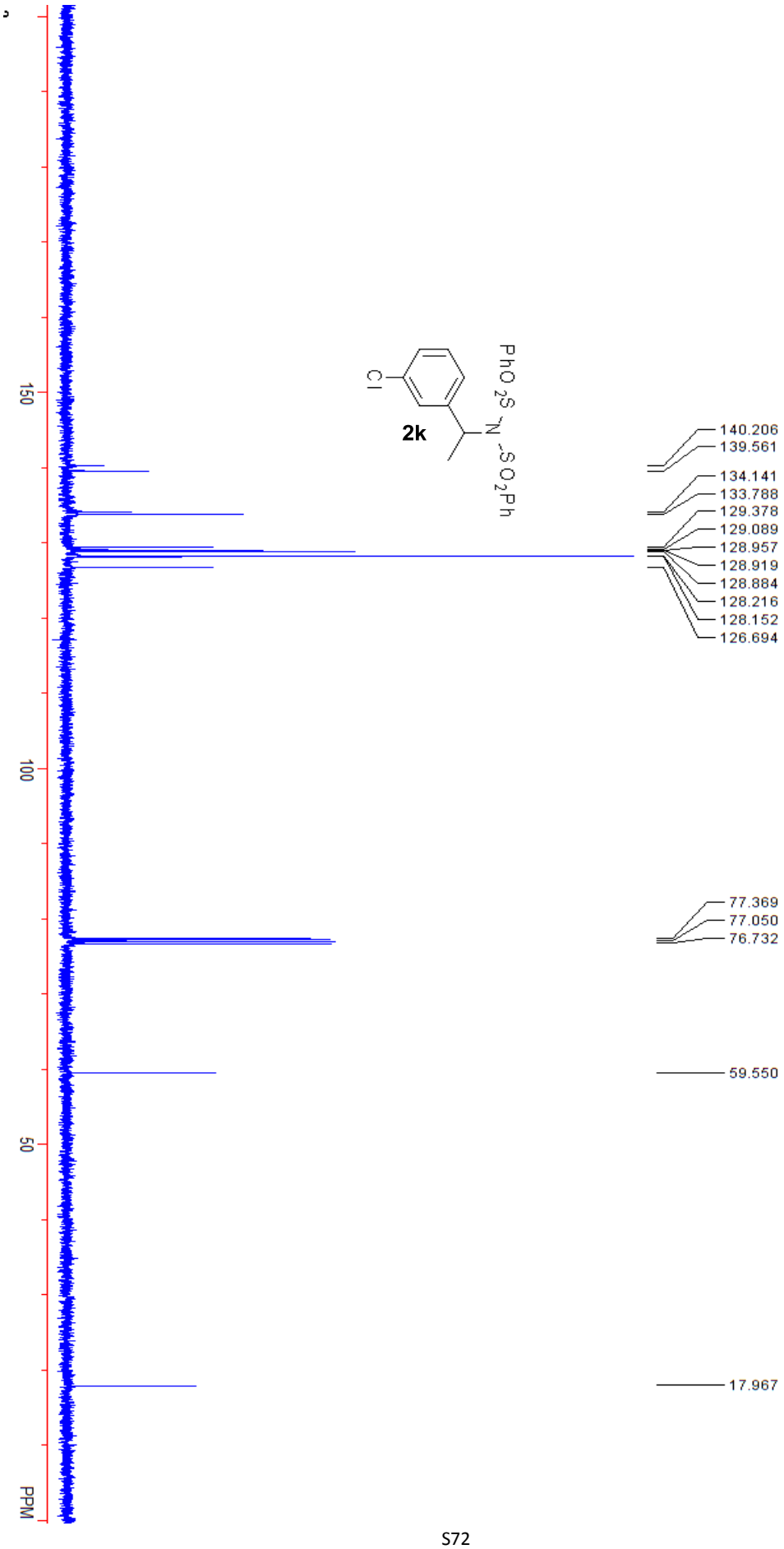
5.568
5.550
5.532
1.646
1.628
Cl
PhO2S
N
SO2Ph
2k


2k
Cl
PhO2S
N
SO2Ph
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129.089
128.957
128.919
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128.152
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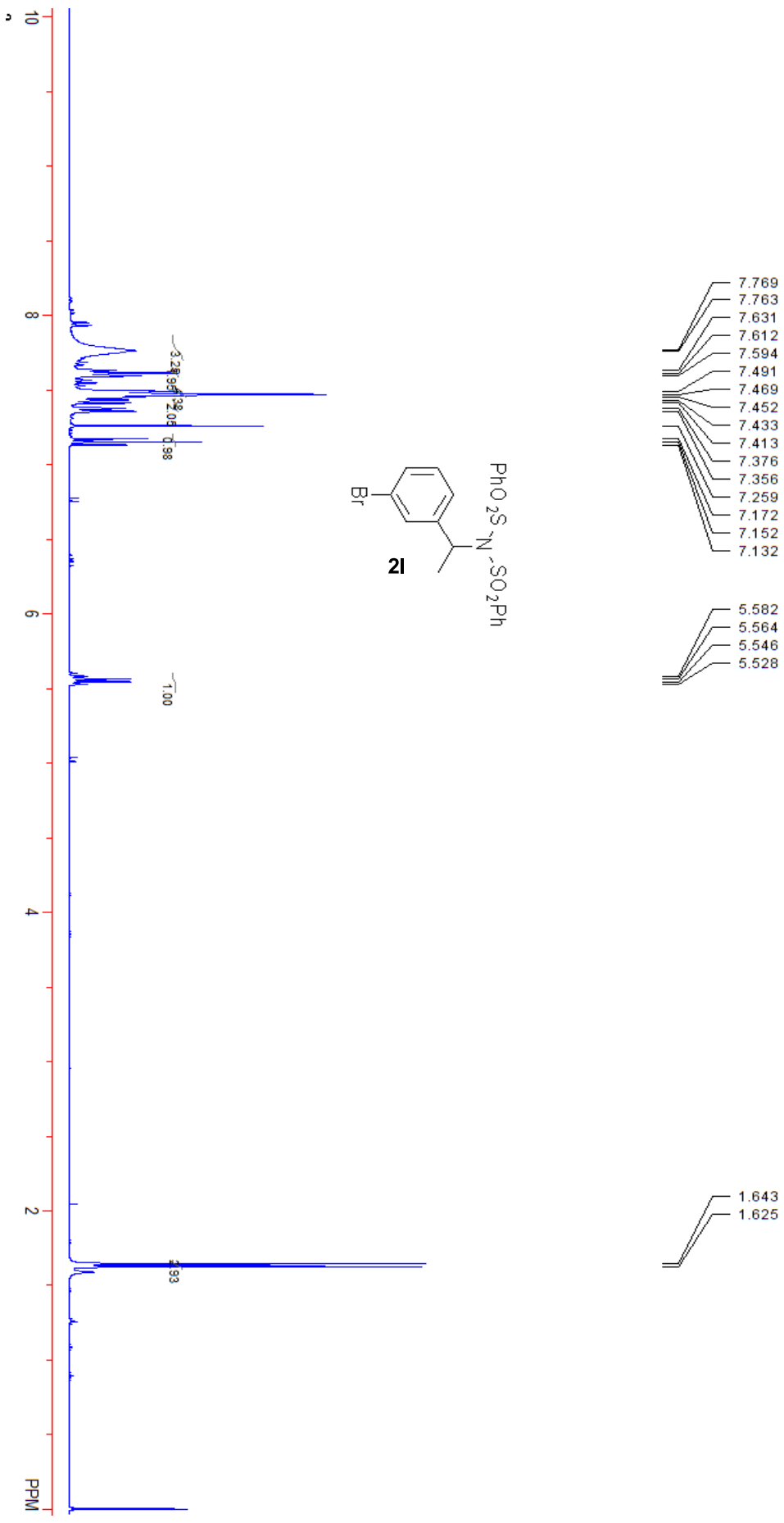
77.369
77.050
76.732
59.550
17.967
150
100
50
PPM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7.612
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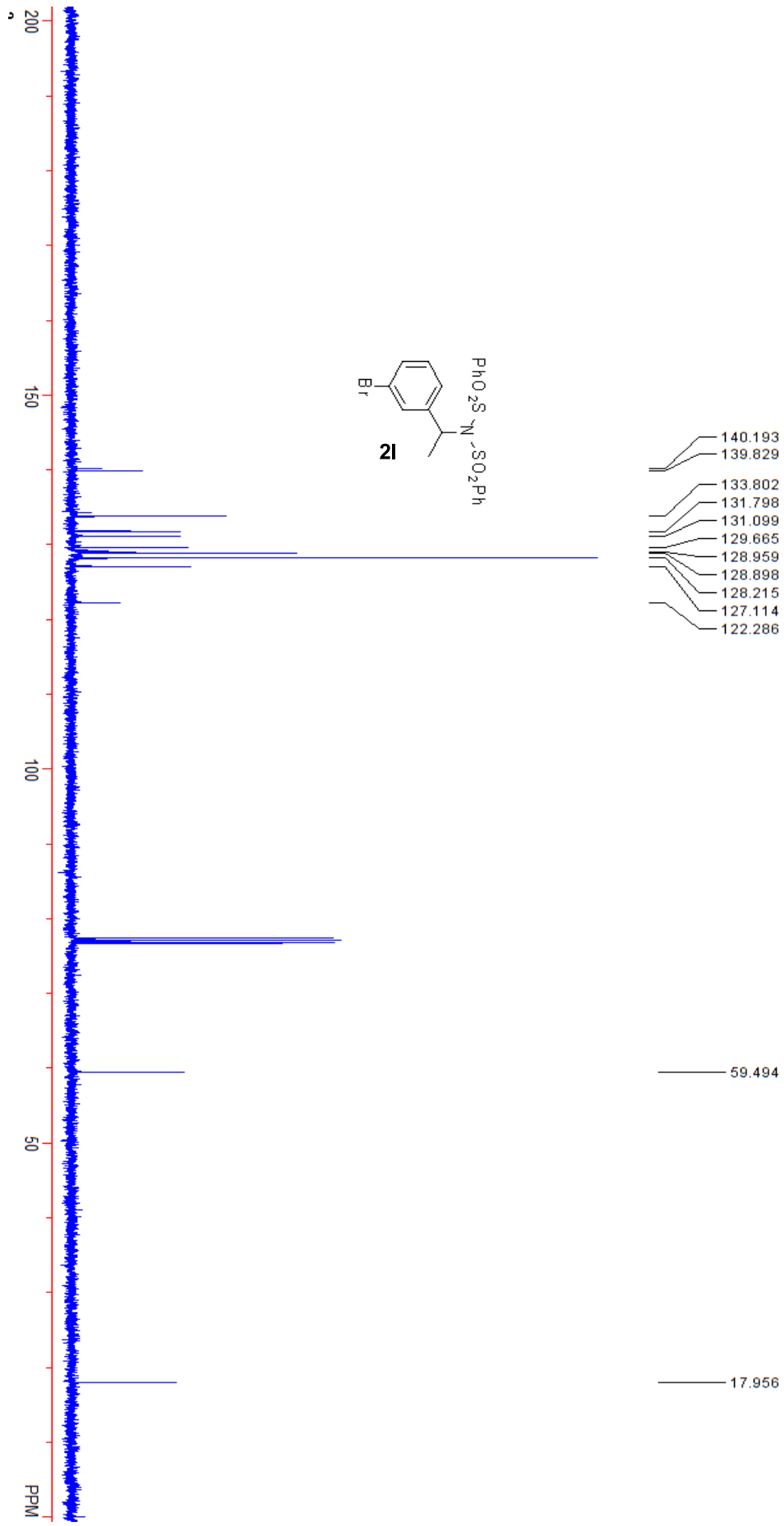
7.491
7.469
7.452
7.433
7.413
7.376
7.356
7.259
7.172
7.152
7.132
5.582
5.564
5.546
5.528
1.643
1.625
Br
PhO2S
N
SO2Ph
2l
PPM


200
150
100
50
PPM
Br
PhO2S
N
SO2Ph
2l
140.193
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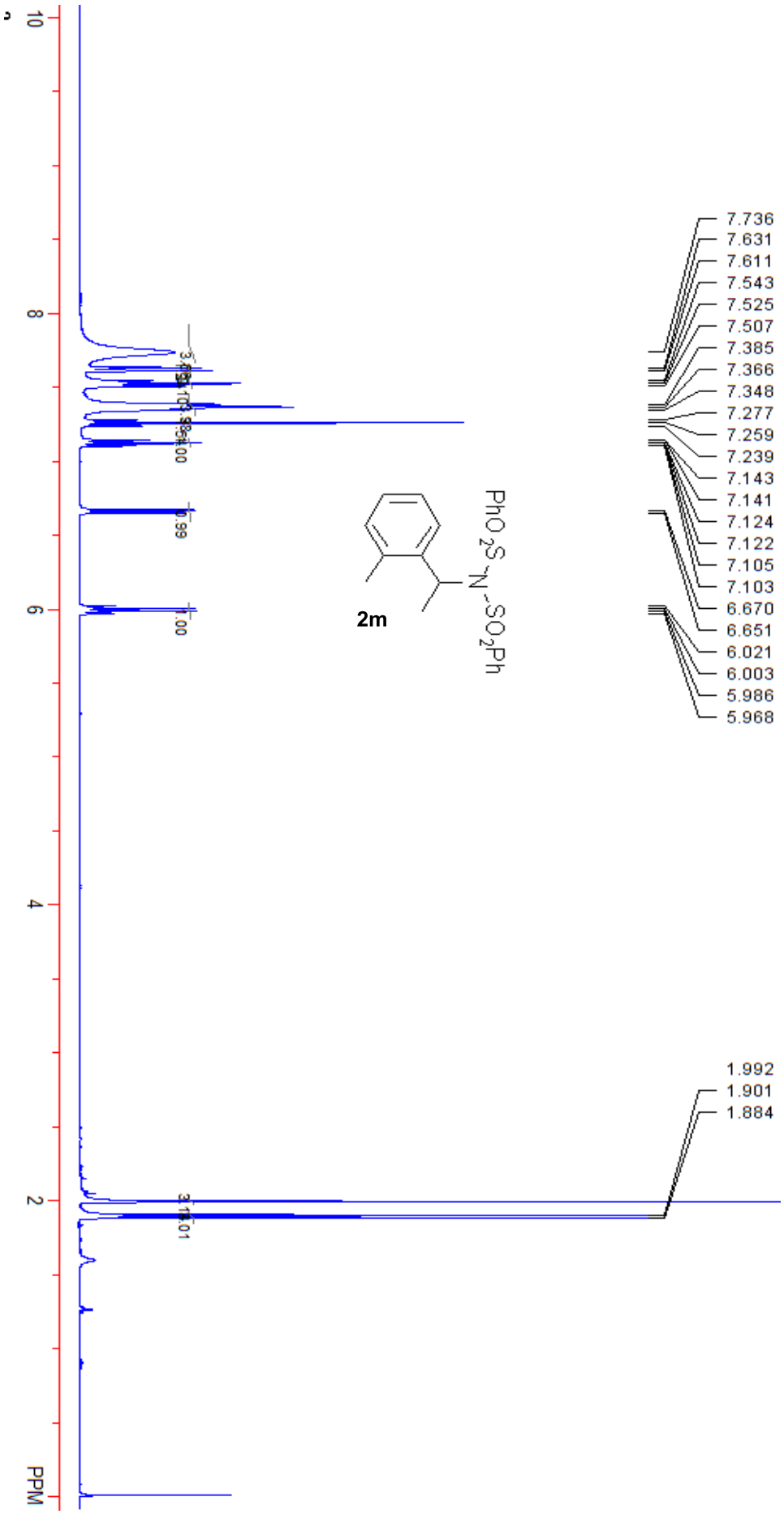
131.099
129.665
128.959
128.898
128.215
127.114
122.286
59.494
17.956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7.277
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7.143
7.141
7.124
7.122
7.105
7.103
6.670
6.651
6.021
6.003
5.986
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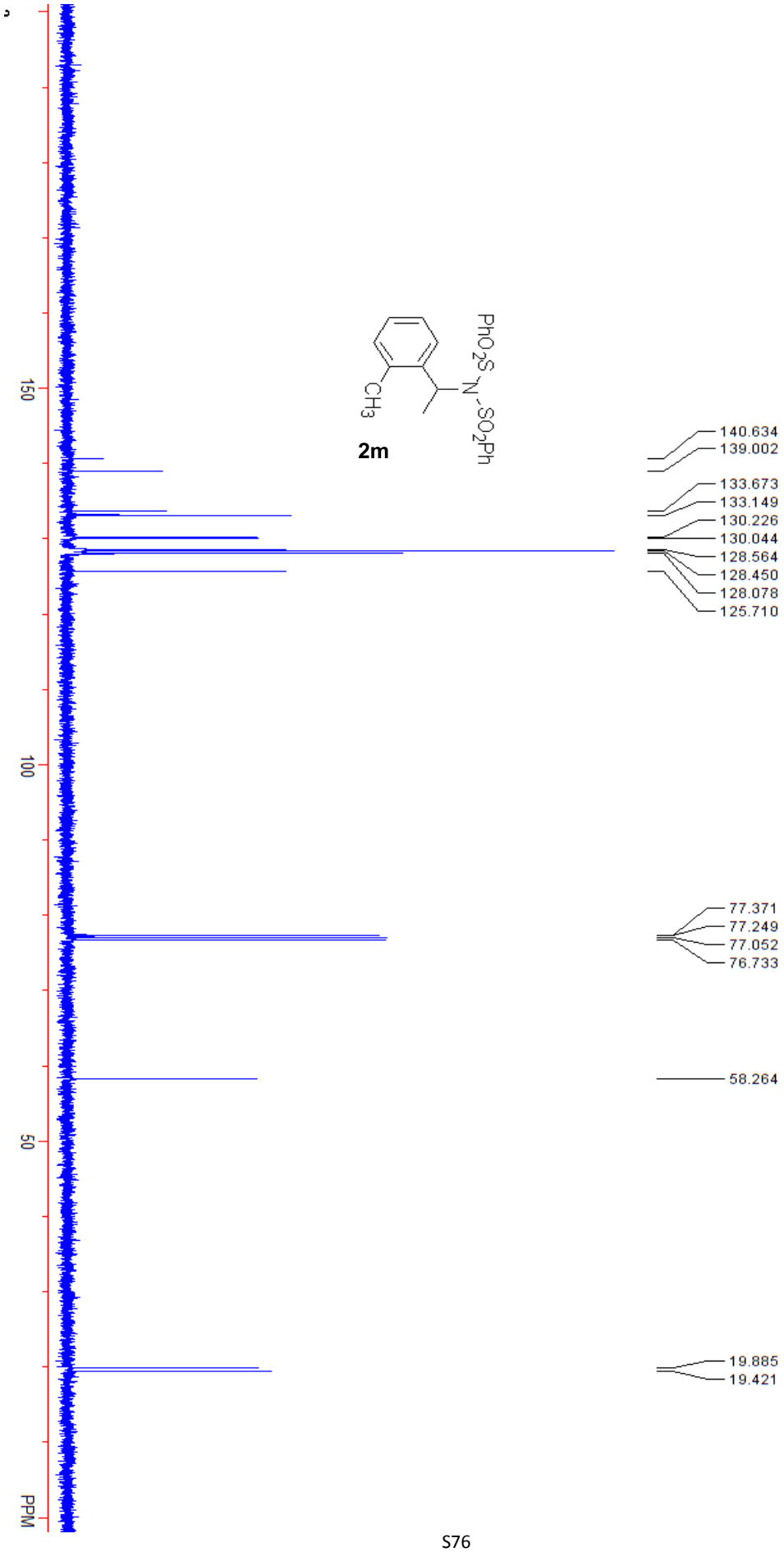
1.901
1.884
PhO2S
N
SO2Ph
2m
10
8
6
4
2
PPM


PhO2S
N
SO2Ph
CH3
2m
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133.149
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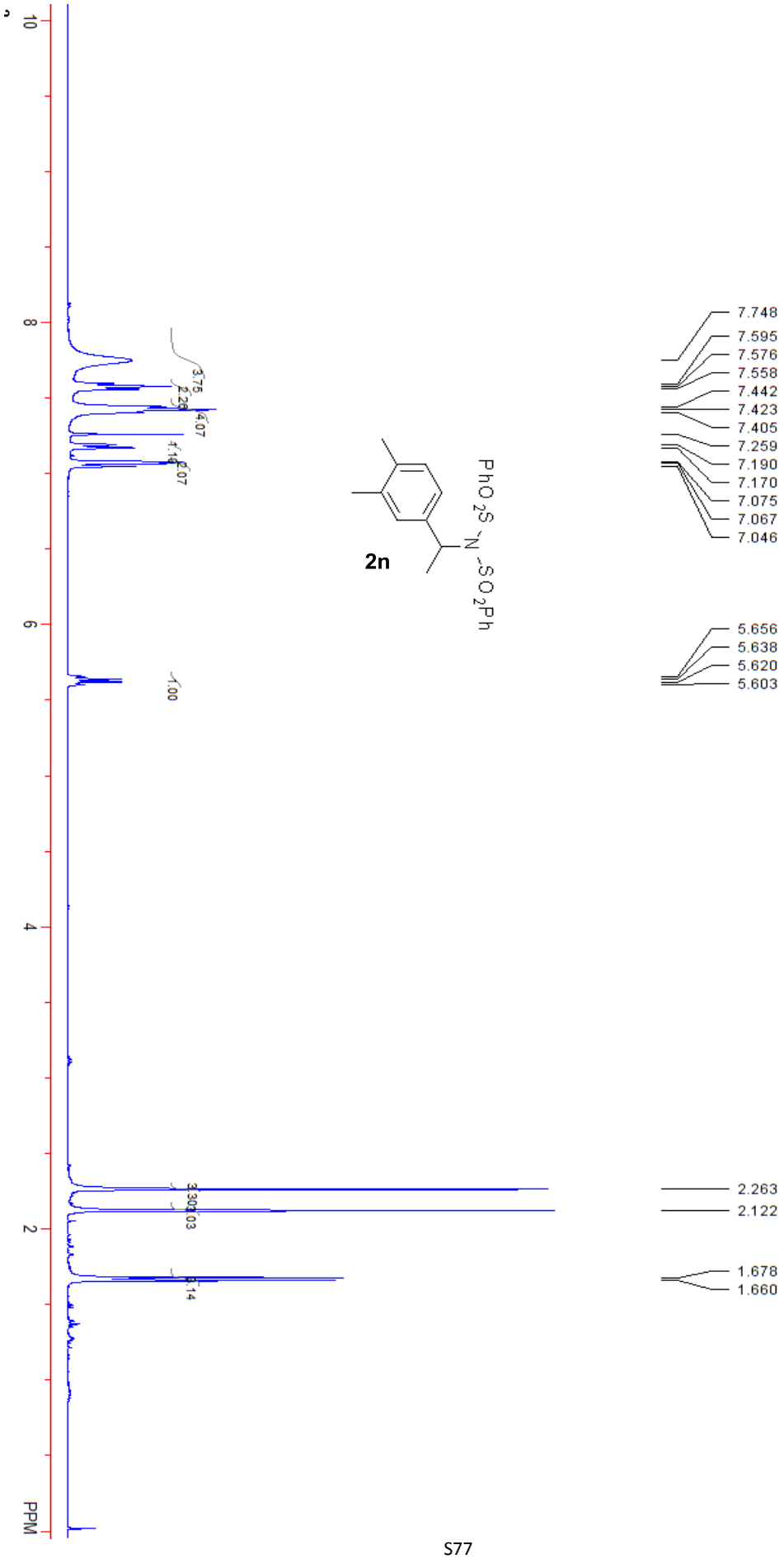
130.044
128.564
128.450
128.078
125.710
77.371
77.249
77.052
76.733
58.264
19.885
19.421
150
100
50
PPM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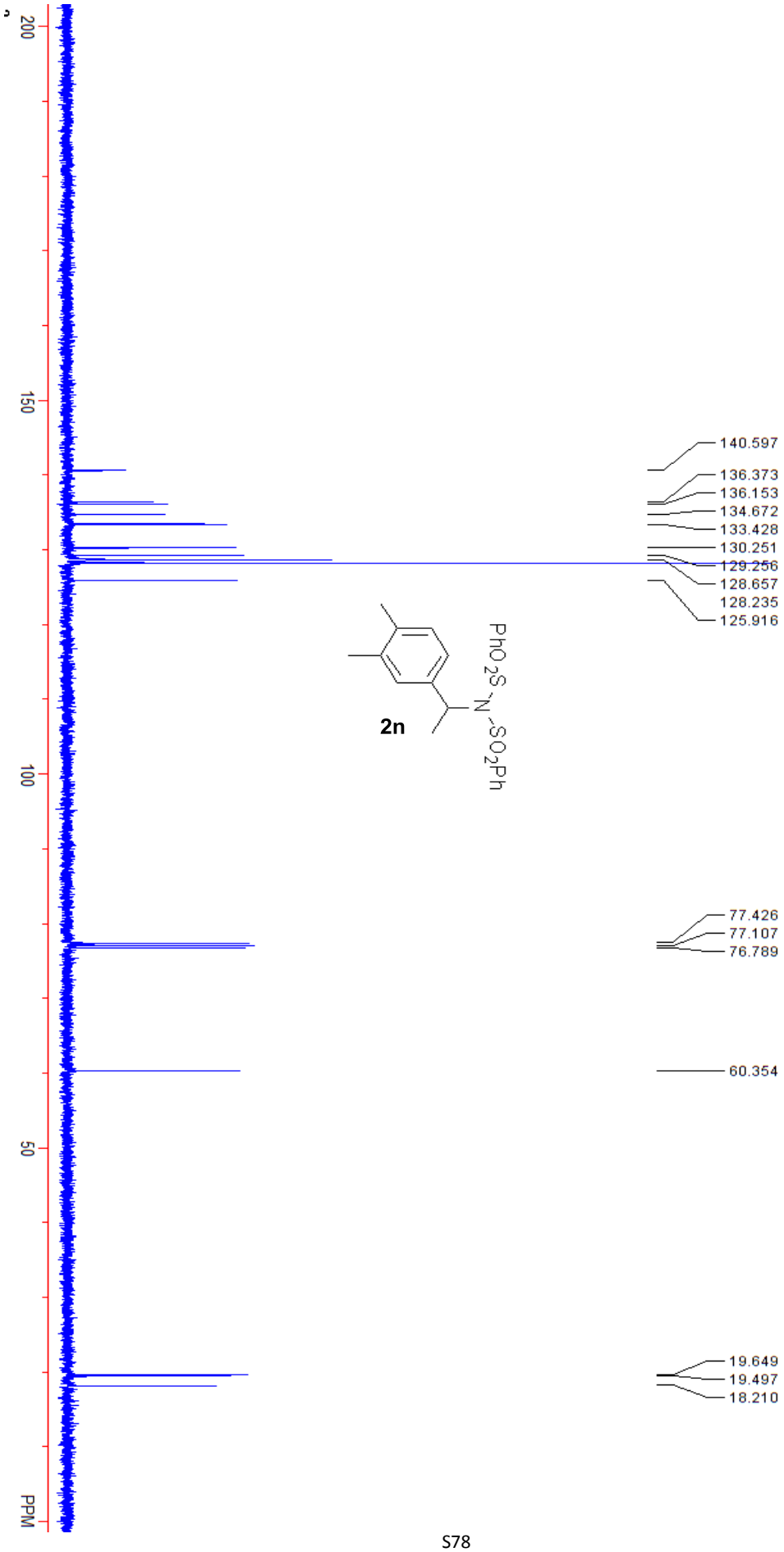
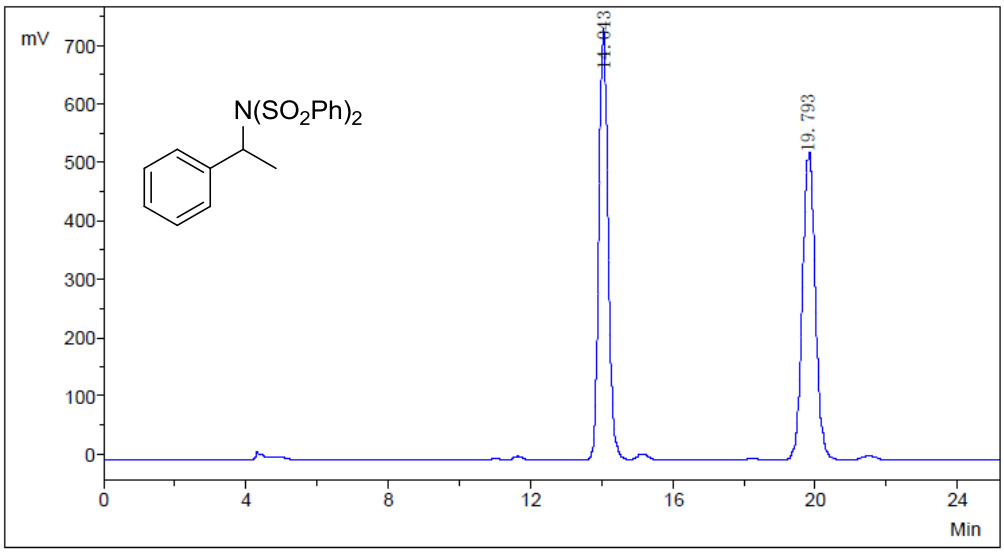
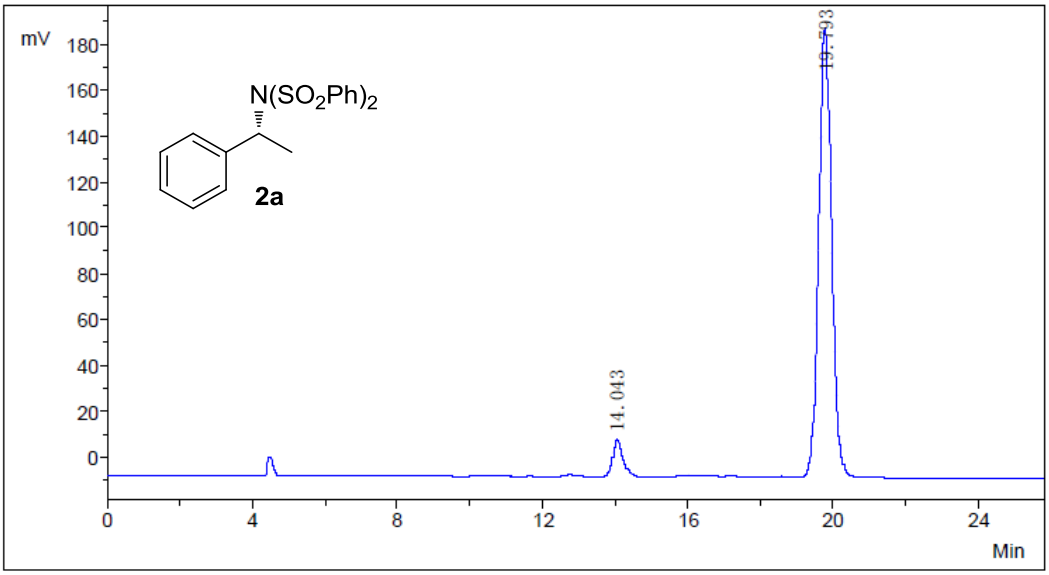
7.259
7.190
7.170
7.075
7.067
7.046
5.656
5.638
5.620
5.603
2.263
2.122
1.678
1.660
2n
PhO2S
N
SO2Ph
PPM


200
150
100
50
PPM
140.597
136.373
136.153
134.672
133.428
130.251
129.256
128.657
128.235
125.916
77.426
77.107
76.789
60.354
19.649
19.497
18.210
PhO2S
N
SO2Ph
2n

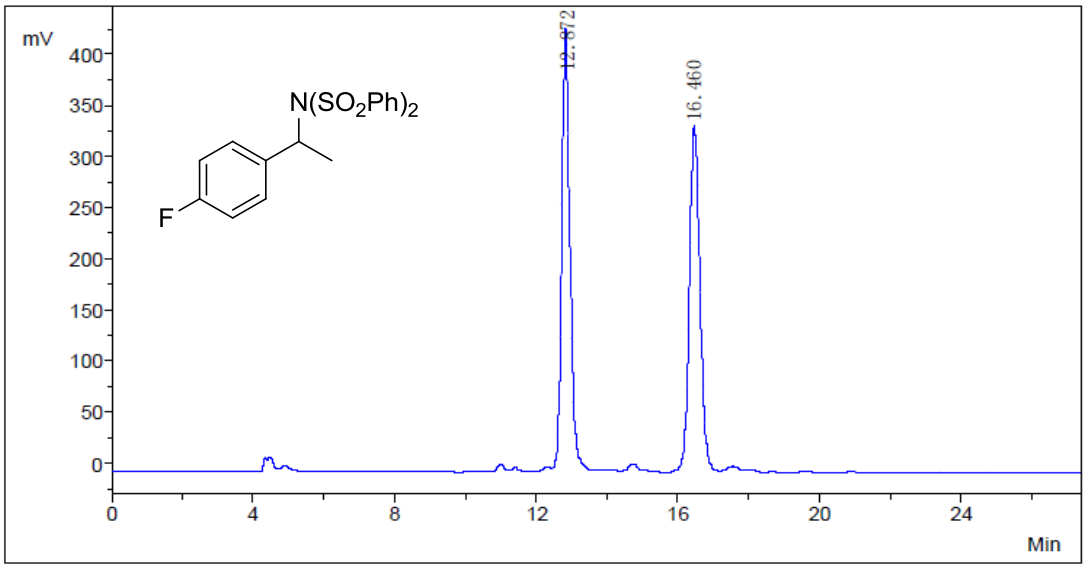
# 8. HPLC of products



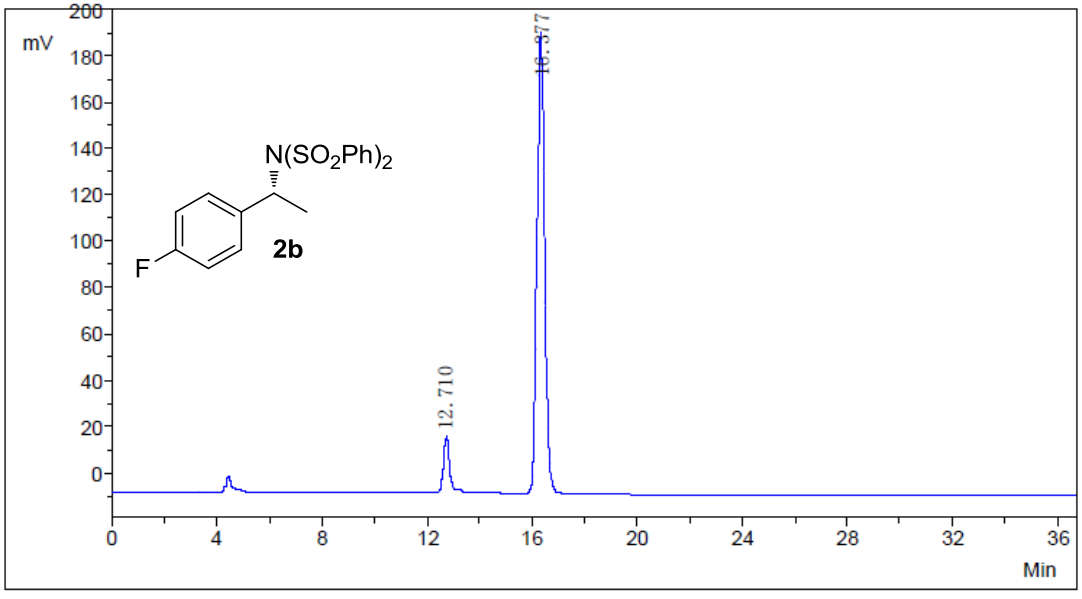

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 14.043 | 736454.7 | 13128351.8 | 50.1081 |
| 2 | 2 | | 19.793 | 511380.3 | 13071682.9 | 49.8919 |
| 合计: | | | | 1247835.0 | 26200034.8 | 100.0000 |



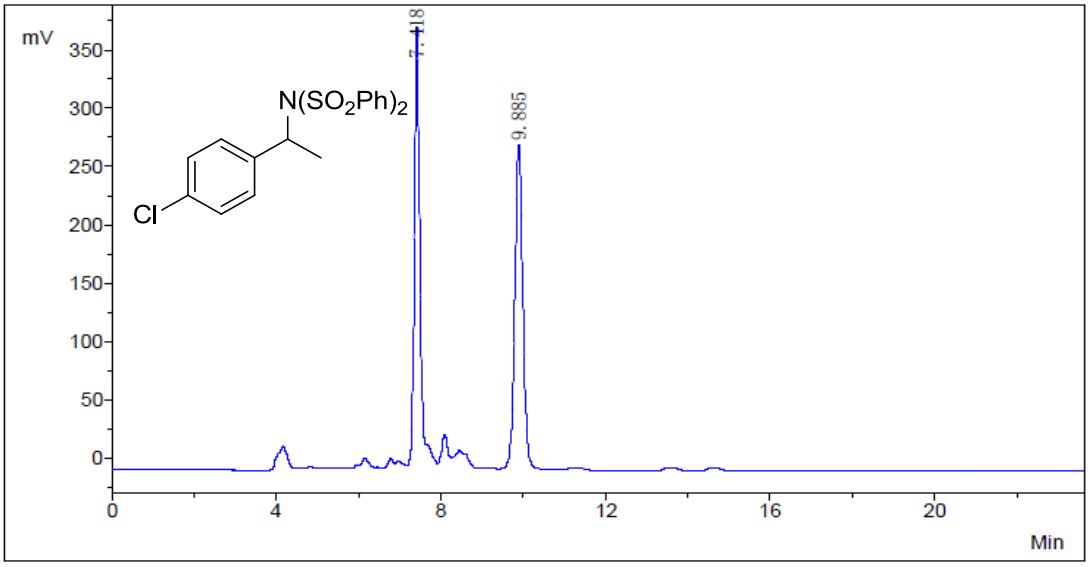

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 14.043 | 15976.1 | 318451.2 | 6.1314 |
| 2 | 2 | | 19.793 | 195677.6 | 4875286.5 | 93.8686 |
| 合计: | | | | 211653.7 | 5193737.7 | 100.0000 |



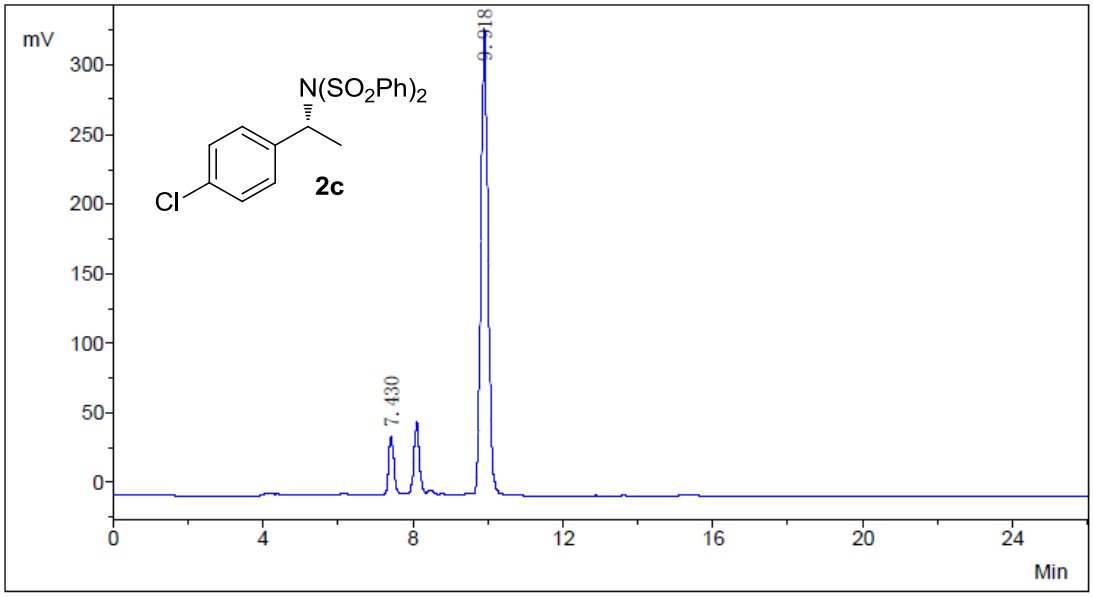

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 12.872 | 401725.2 | 6989038.7 | 50.0161 |
| 2 | 2 | | 16.460 | 334584.0 | 6984544.4 | 49.9839 |
| 合计: | | | | 736309.2 | 13973583.1 | 100.0000 |



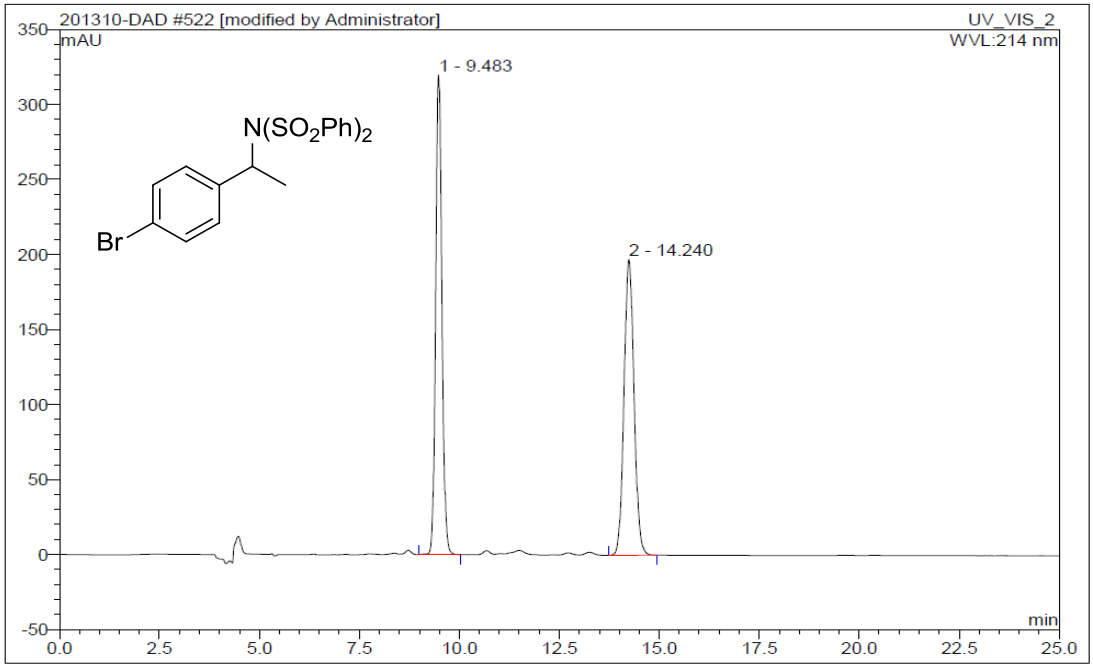

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 12.710 | 23547.8 | 407834.4 | 9.2311 |
| 2 | 2 | | 16.377 | 189036.3 | 4010217.3 | 90.7689 |
| 合计: | | | | 212584.1 | 4418051.8 | 100.0000 |



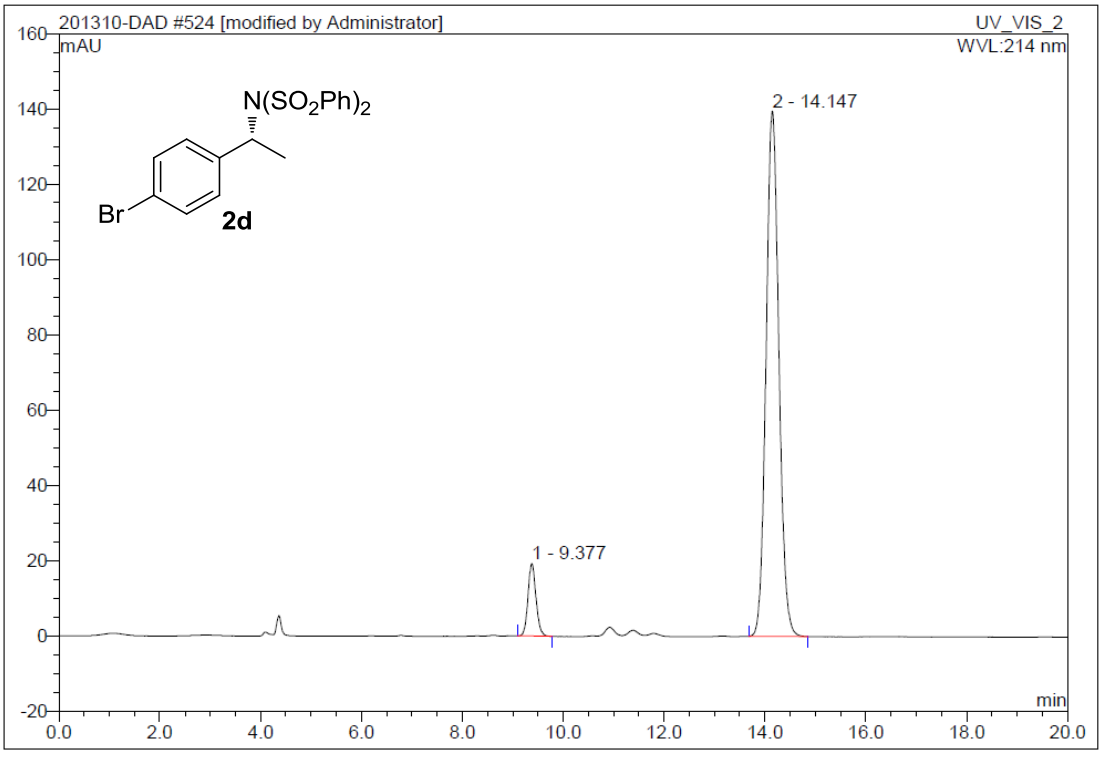

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 7.418 | 374387.1 | 3624424.5 | 49.2498 |
| 2 | 2 | | 9.885 | 274034.4 | 3734842.6 | 50.7502 |
| 合计: | | | | 648421.5 | 7359267.1 | 100.0000 |



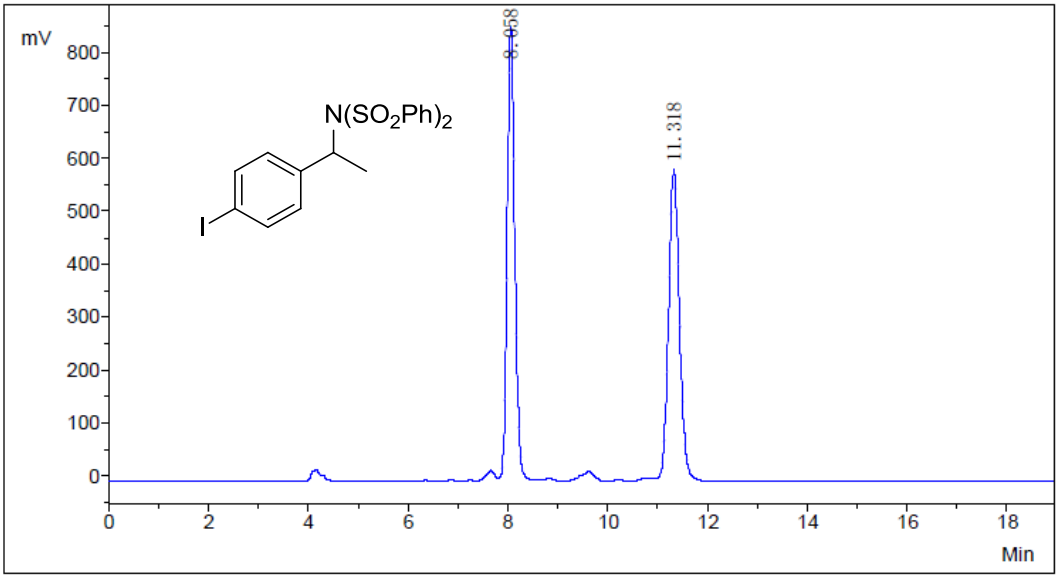

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 7.430 | 41128.6 | 375897.6 | 8.0045 |
| 2 | 2 | | 9.918 | 334839.8 | 4320206.2 | 91.9955 |
| 合计: | | | | 375968.3 | 4696103.8 | 100.0000 |



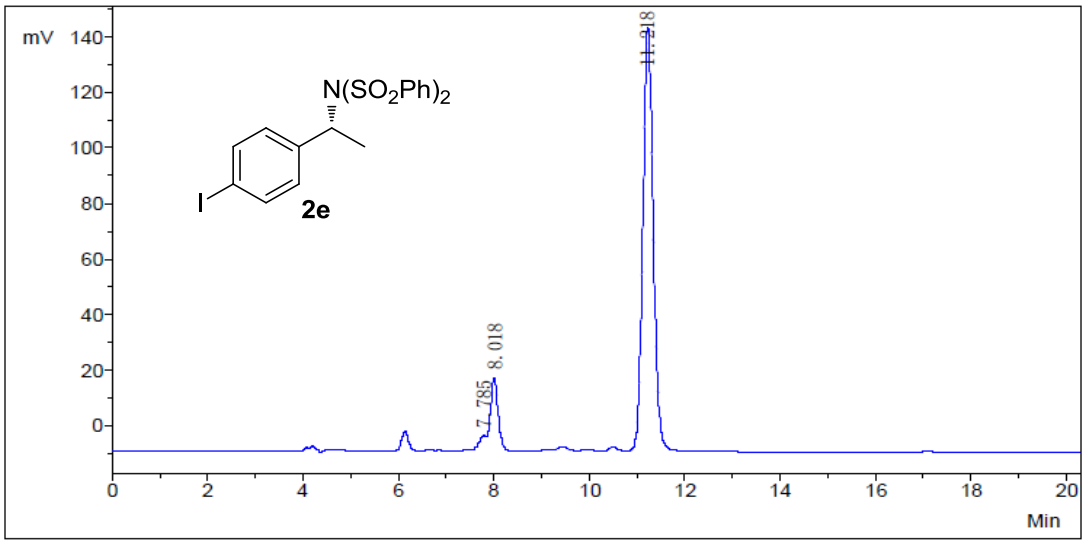

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 9.48 | n.a. | 319.714 | 57.933 | 50.25 | n.a. | BMB |
| 2 | 14.24 | n.a. | 197.126 | 57.355 | 49.75 | n.a. | BMB |
| Total: | | | 516.840 | 115.288 | 100.00 | 0.000 | |



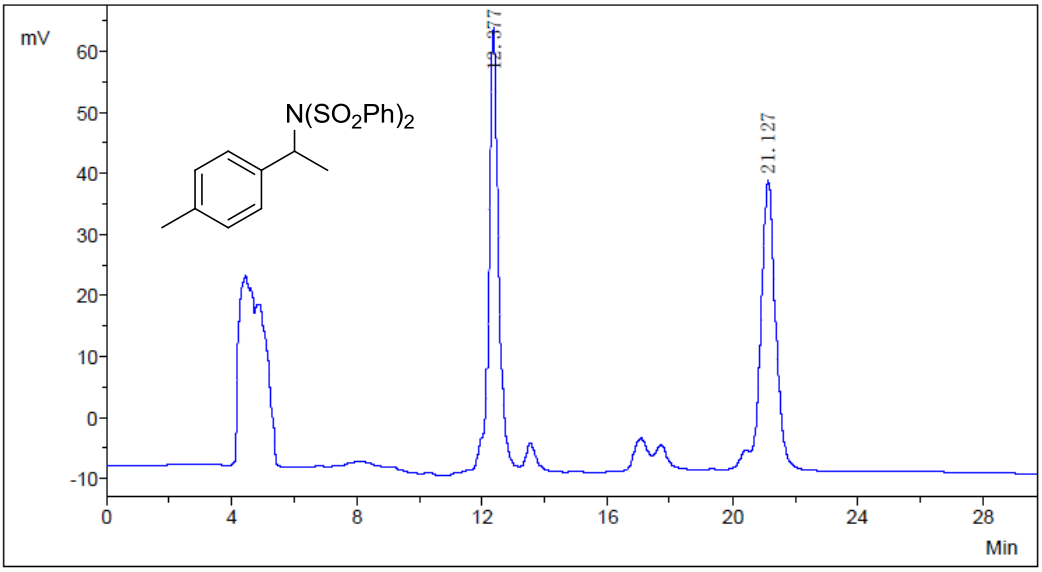

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 9.38 | n.a. | 19.333 | 3.551 | 7.96 | n.a. | BMB |
| 2 | 14.15 | n.a. | 139.599 | 41.047 | 92.04 | n.a. | BMB |
| Total: | | | 158.931 | 44.599 | 100.00 | 0.000 | |



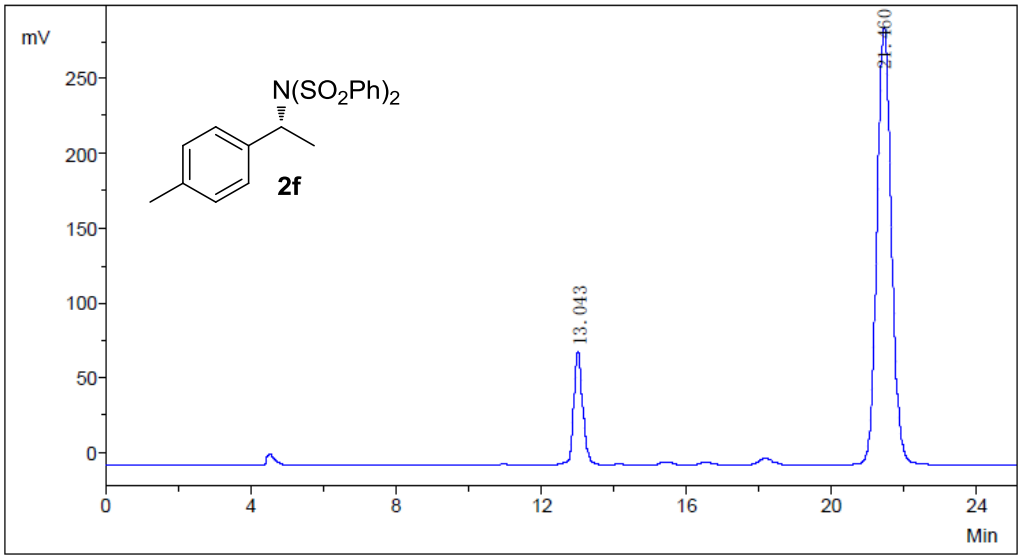

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 8.058 | 849964.0 | 8882609.6 | 50.1567 |
| 2 | 2 | | 11.318 | 582939.3 | 8827121.5 | 49.8433 |
| 合计: | | | | 1432903.3 | 17709731.1 | 100.0000 |



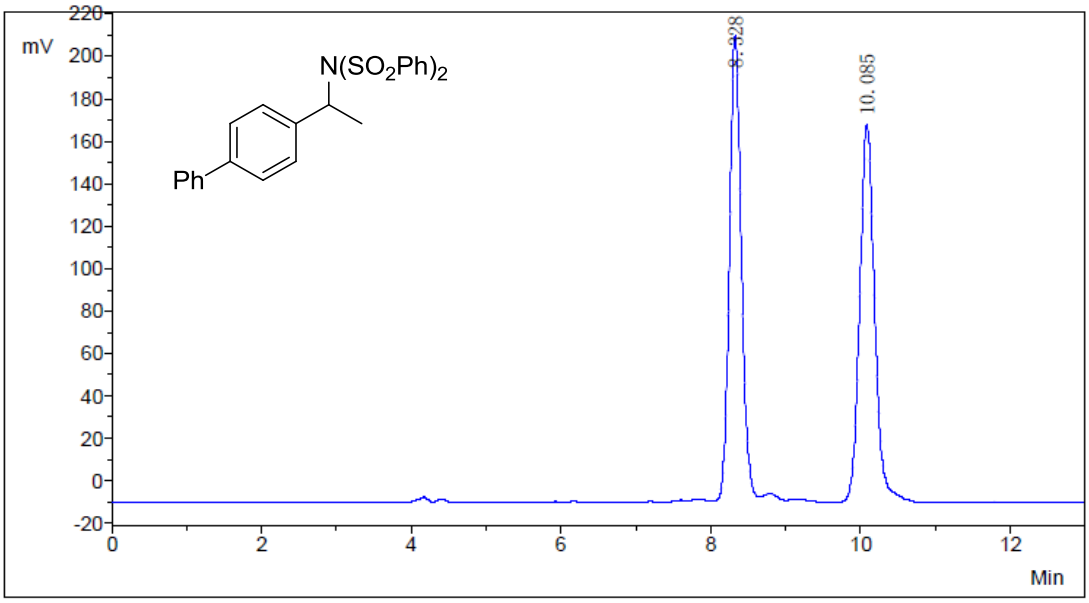

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 7.785 | 5282.2 | 52273.4 | 1.9877 |
| 2 | 2 | | 8.018 | 26149.4 | 288979.6 | 10.9887 |
| 3 | 3 | | 11.218 | 150506.3 | 2288542.7 | 87.0236 |
| 合计: | | | | 181937.8 | 2629795.6 | 100.0000 |



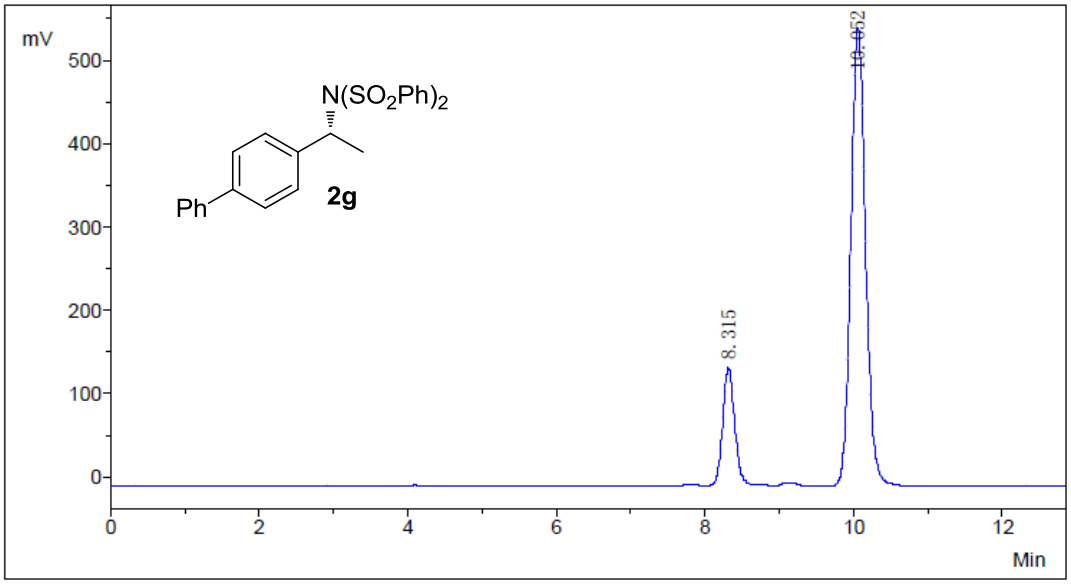

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 12.377 | 72522.3 | 1564777.0 | 49.6294 |
| 2 | 2 | | 21.127 | 47358.5 | 1588146.0 | 50.3706 |
| 合计: | | | | 119880.8 | 3152923.0 | 100.0000 |



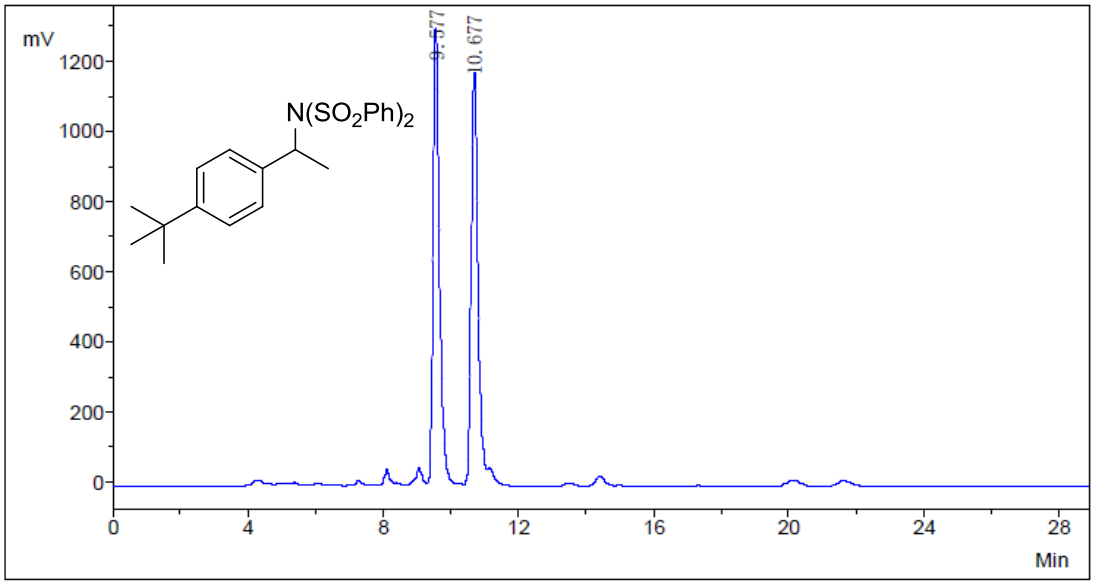

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 13.043 | 73784.6 | 1295520.7 | 13.8367 |
| 2 | 2 | | 21.460 | 292124.6 | 8067422.1 | 86.1633 |
| 合计: | | | | 365909.2 | 9362942.9 | 100.0000 |



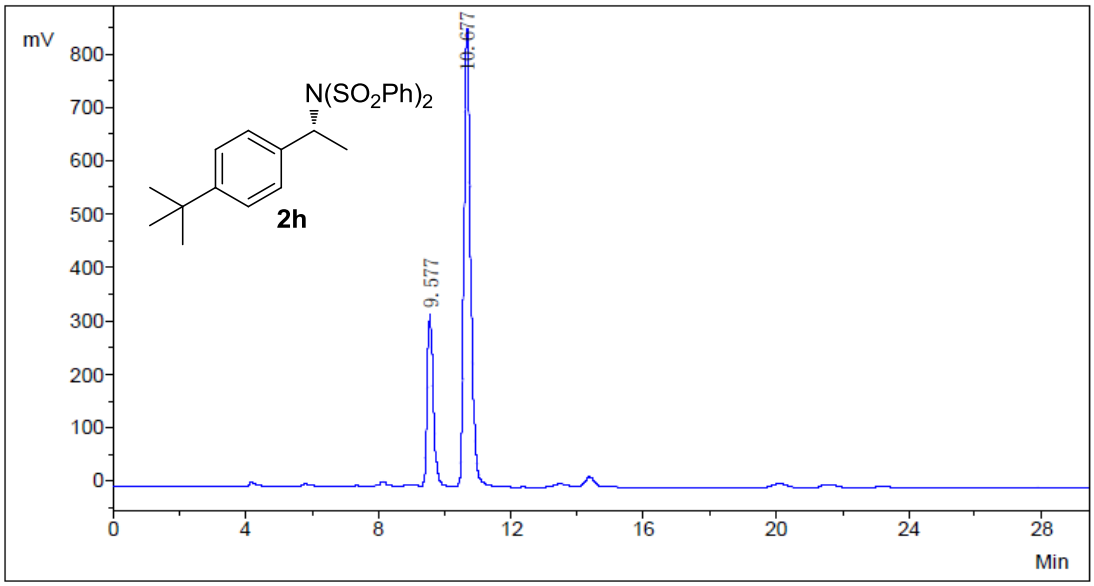

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 8.328 | 218484.8 | 2369452.3 | 49.2637 |
| 2 | 2 | | 10.085 | 178040.7 | 2440279.1 | 50.7363 |
| 合计: | | | | 396525.5 | 4809731.4 | 100.0000 |



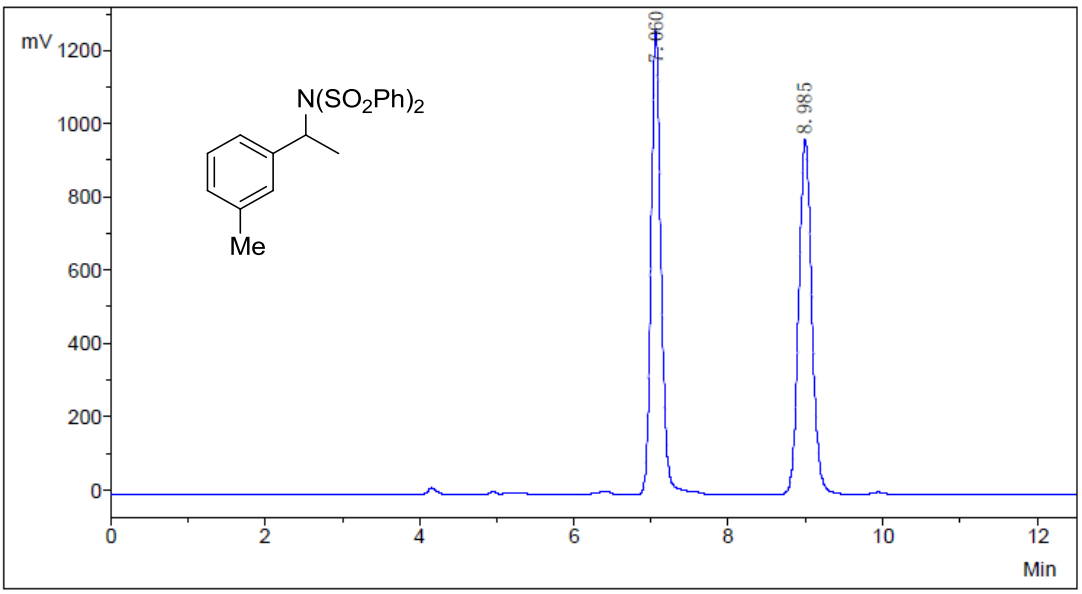

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 8.315 | 136477.8 | 1520759.7 | 16.7276 |
| 2 | 2 | | 10.052 | 545972.4 | 7570554.4 | 83.2724 |
| 合计: | | | | 682450.2 | 9091314.0 | 100.0000 |



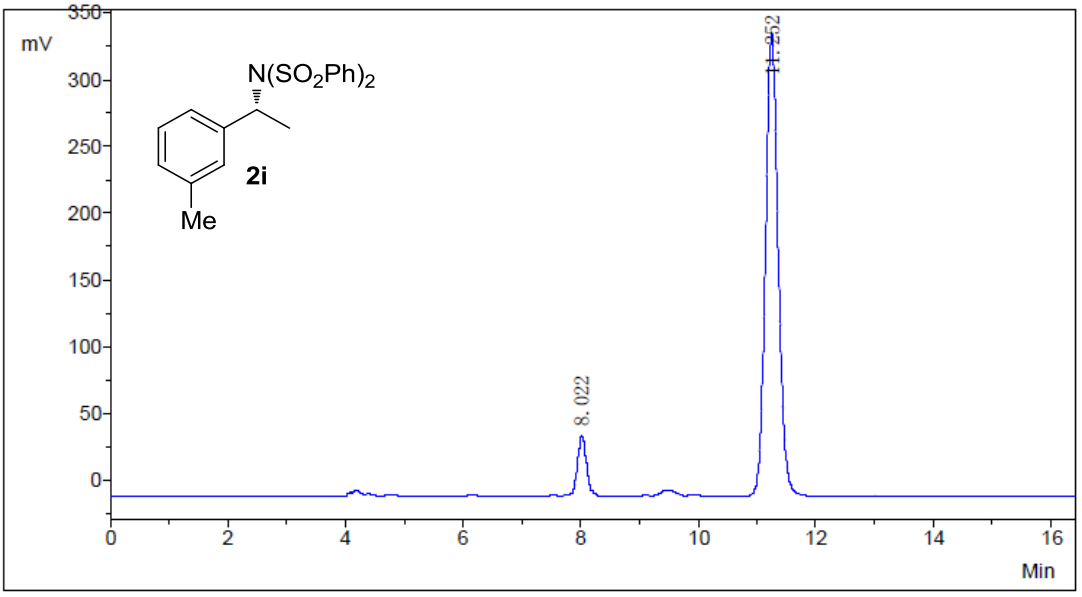

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 9.577 | 1249213.6 | 17374564.7 | 49.2154 |
| 2 | 2 | | 10.677 | 1143939.2 | 17928515.7 | 50.7846 |
| 合计: | | | | 2393152.8 | 35303080.4 | 100.0000 |



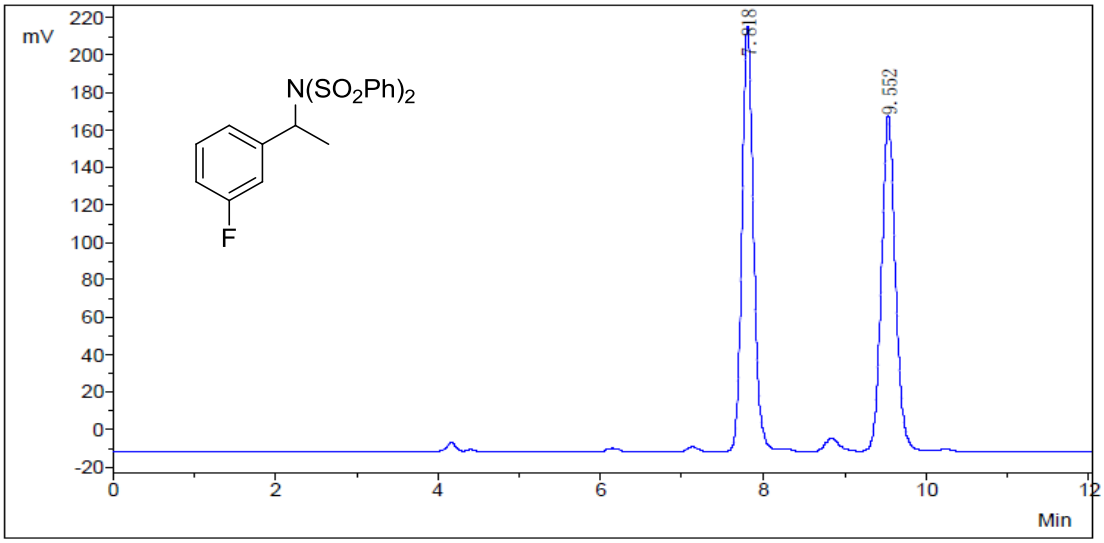

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 9.577 | 316911.5 | 3998401.1 | 24.3810 |
| 2 | 2 | | 10.677 | 853237.6 | 12401285.0 | 75.6190 |
| 合计: | | | | 1170149.1 | 16399686.2 | 100.0000 |



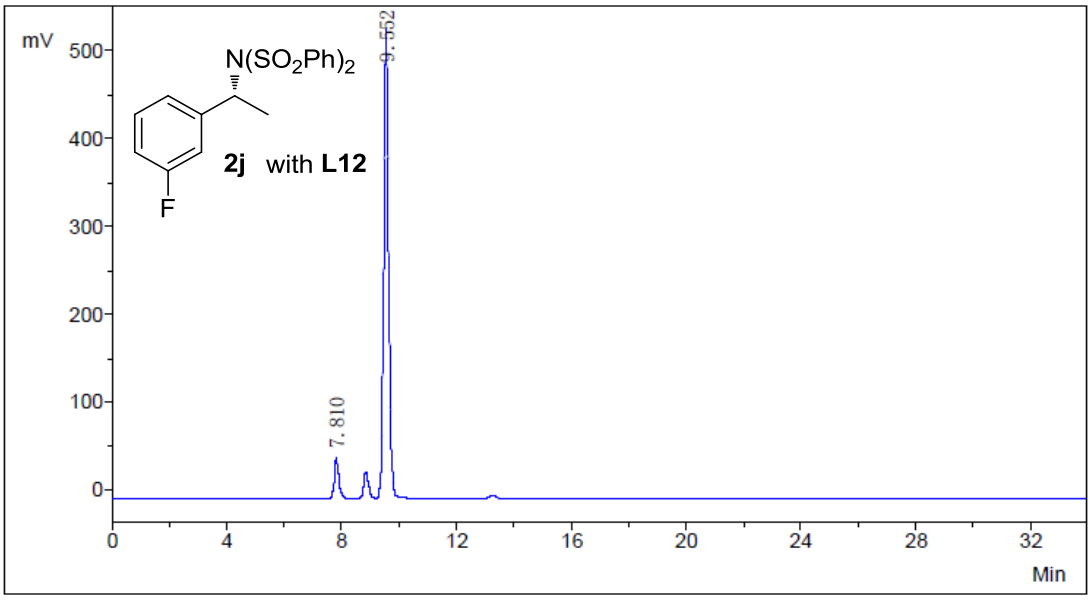

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 7.060 | 1260558.1 | 11413083.5 | 50.0947 |
| 2 | 2 | | 8.985 | 958044.7 | 11369949.0 | 49.9053 |
| 合计: | | | | 2218602.8 | 22783032.5 | 100.0000 |



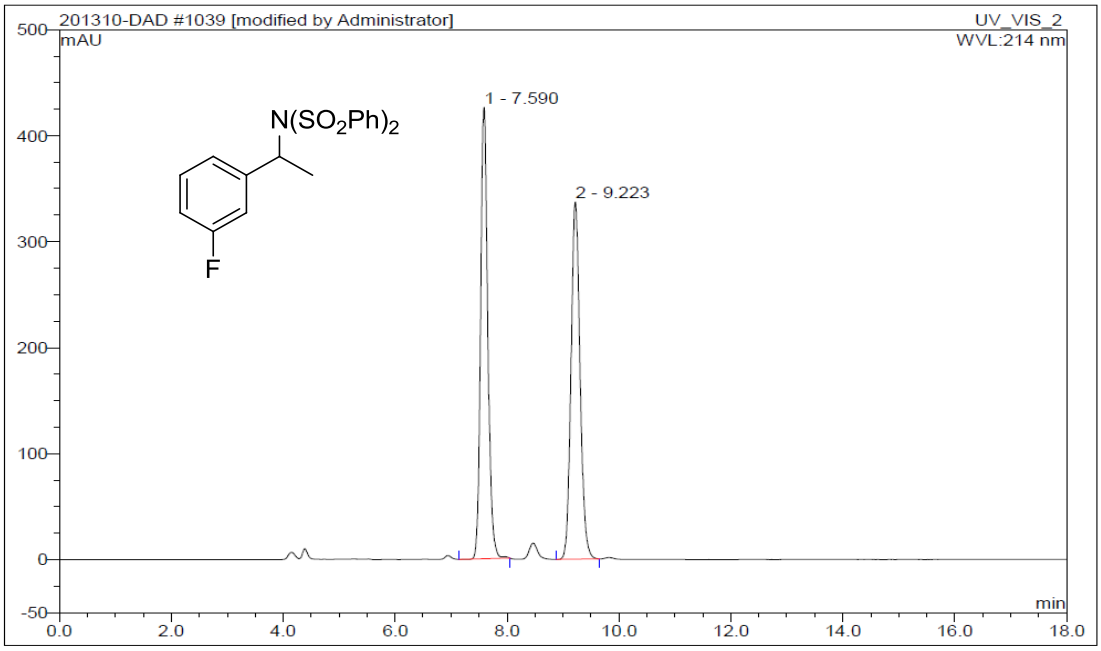

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 8.022 | 44943.8 | 455048.0 | 7.9783 |
| 2 | 2 | | 11.252 | 346228.2 | 5248538.5 | 92.0217 |
| 合计: | | | | 391172.0 | 5703586.5 | 100.0000 |



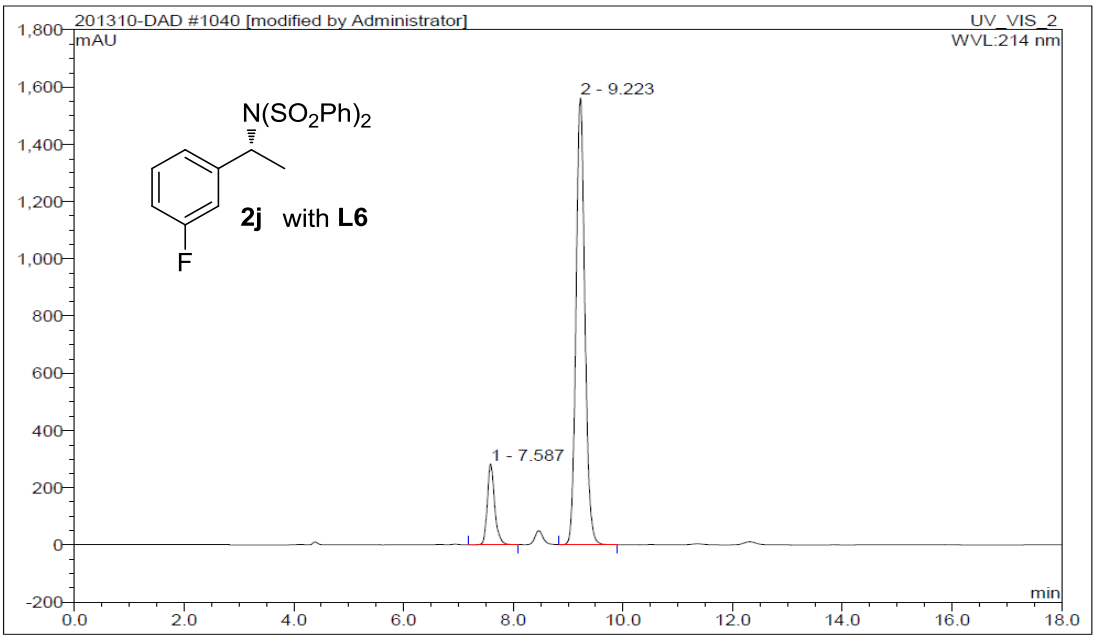

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 7.818 | 221728.6 | 2223199.3 | 50.7337 |
| 2 | 2 | | 9.552 | 175184.6 | 2158894.1 | 49.2663 |
| 合计: | | | | 396913.2 | 4382093.3 | 100.0000 |



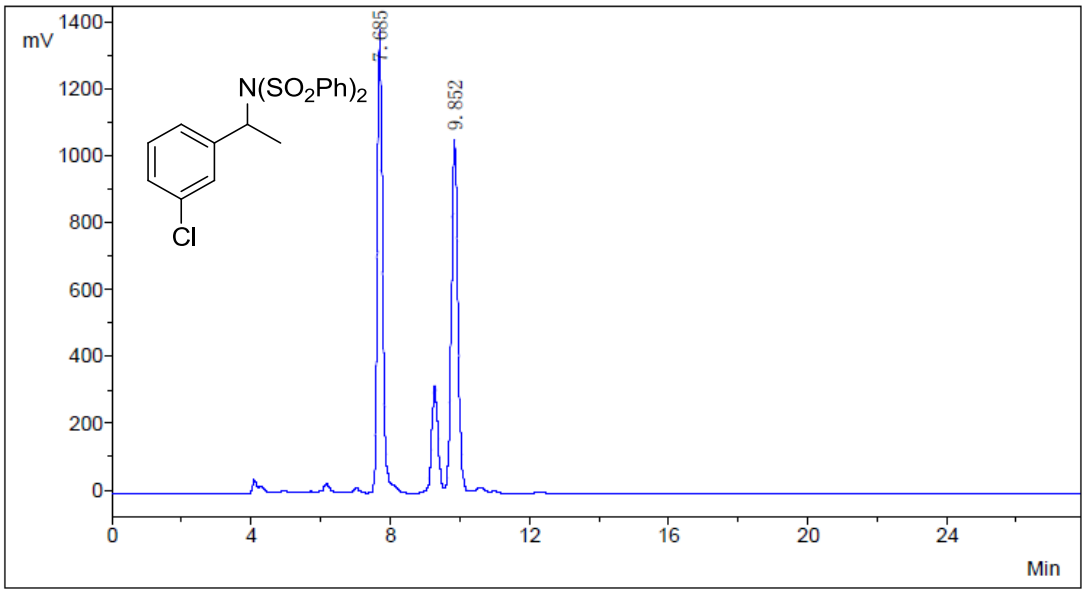

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 7.810 | 46473.5 | 501725.3 | 7.0742 |
| 2 | 2 | | 9.552 | 533082.0 | 6590562.7 | 92.9258 |
| 合计: | | | | 579555.5 | 7092287.9 | 100.0000 |



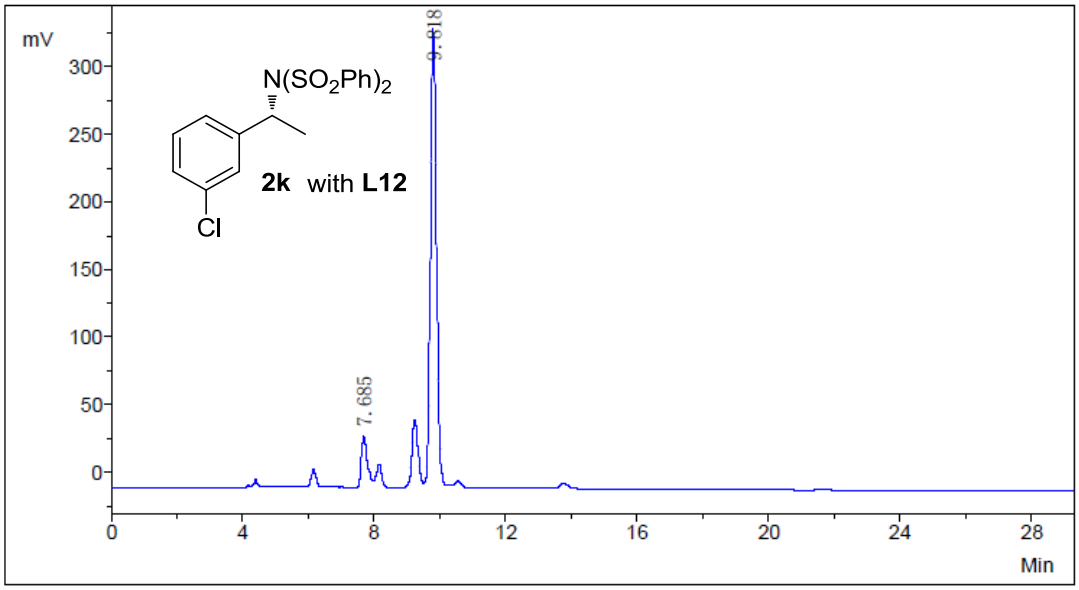

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 7.59 | n.a. | 425.948 | 63.452 | 50.19 | n.a. | BMB* |
| 2 | 9.22 | n.a. | 336.927 | 62.976 | 49.81 | n.a. | BMB* |
| **Total:** | | | 762.875 | 126.428 | 100.00 | 0.000 | |



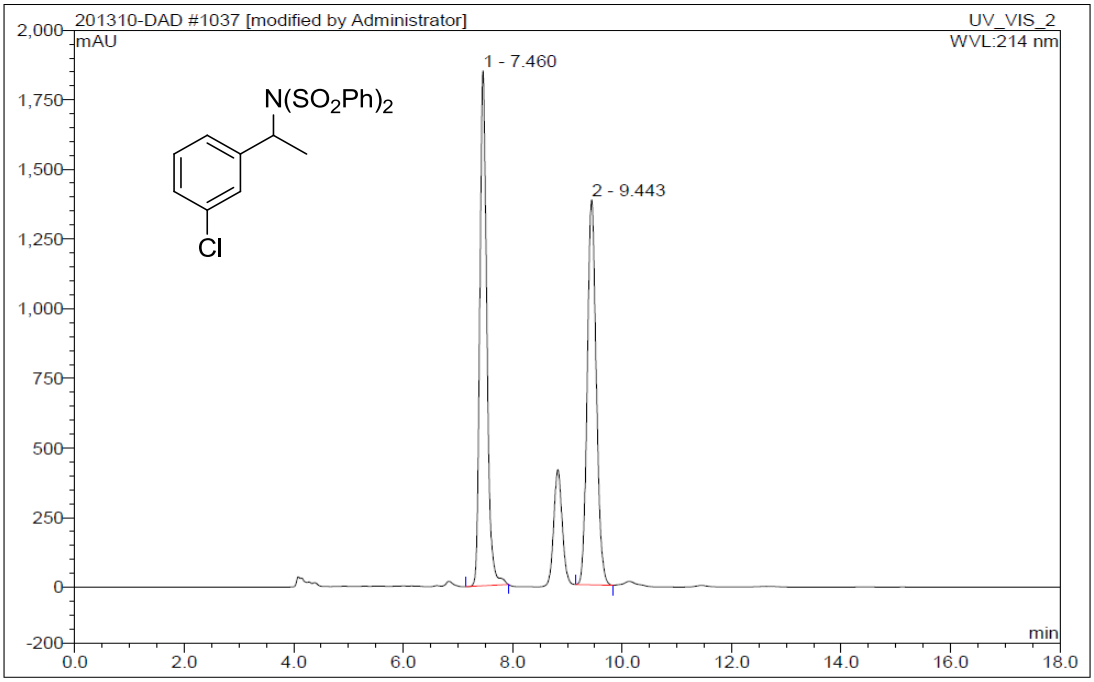

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 7.59 | n.a. | 282.216 | 43.802 | 13.02 | n.a. | BMB* |
| 2 | 9.22 | n.a. | 1561.318 | 292.532 | 86.98 | n.a. | BMB* |
| **Total:** | | | 1843.534 | 336.334 | 100.00 | 0.000 | |



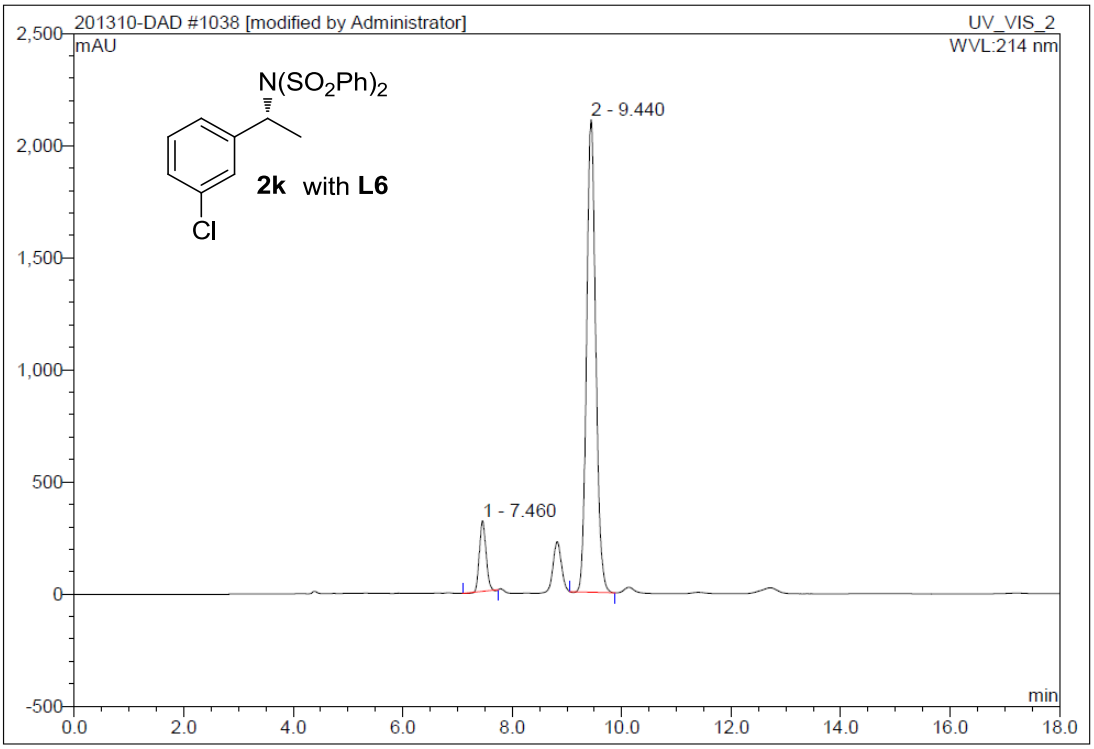

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 7.685 | 1347372.0 | 14490683.6 | 51.6646 |
| 2 | 2 | | 9.852 | 1048232.1 | 13556941.1 | 48.3354 |
| 合计: | | | | 2395604.1 | 28047624.7 | 100.0000 |



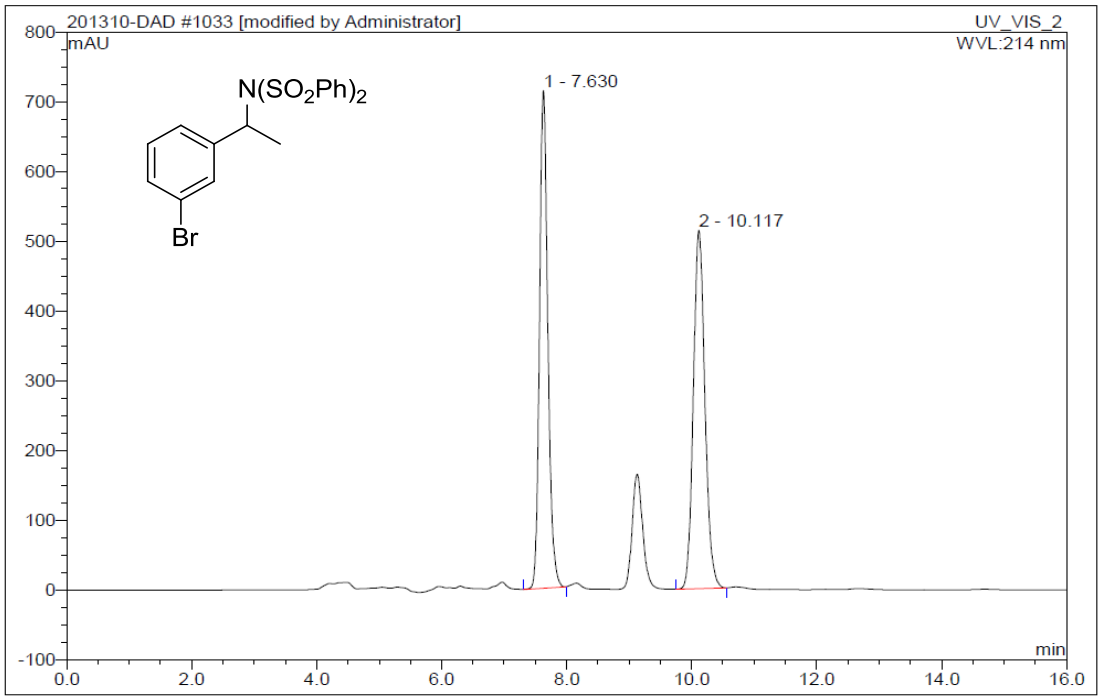

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 7.685 | 36263.9 | 400079.7 | 8.6279 |
| 2 | 2 | | 9.818 | 332571.9 | 4236940.8 | 91.3721 |
| 合计: | | | | 368835.7 | 4637020.5 | 100.0000 |



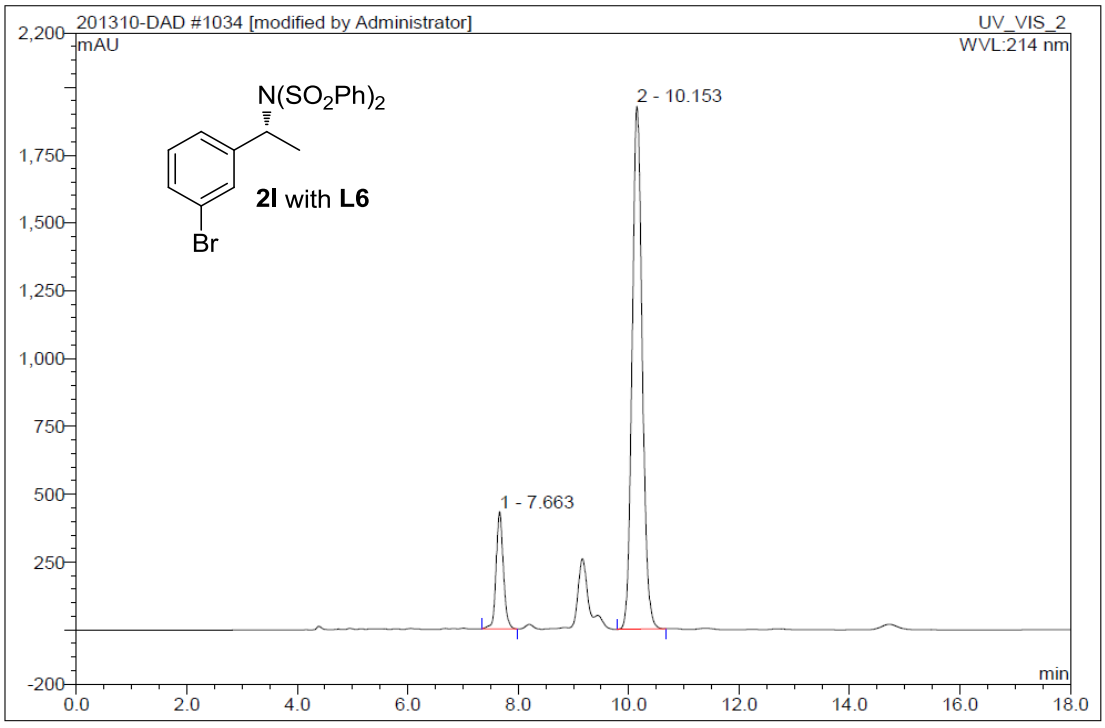

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 7.46 | n.a. | 1850.254 | 274.974 | 50.82 | n.a. | BMB* |
| 2 | 9.44 | n.a. | 1382.561 | 266.113 | 49.18 | n.a. | BMB* |
| **Total:** | | | 3232.815 | 541.087 | 100.00 | 0.000 | |



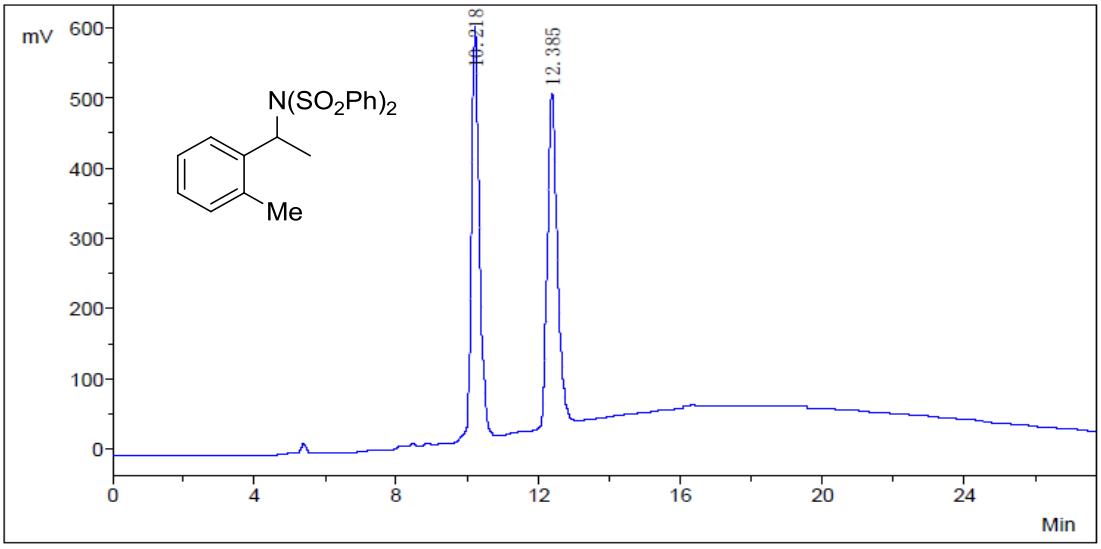

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 7.46 | n.a. | 315.979 | 43.543 | 9.63 | n.a. | BMB* |
| 2 | 9.44 | n.a. | 2107.685 | 408.589 | 90.37 | n.a. | BMB* |
| **Total:** | | | 2423.665 | 452.132 | 100.00 | 0.000 | |



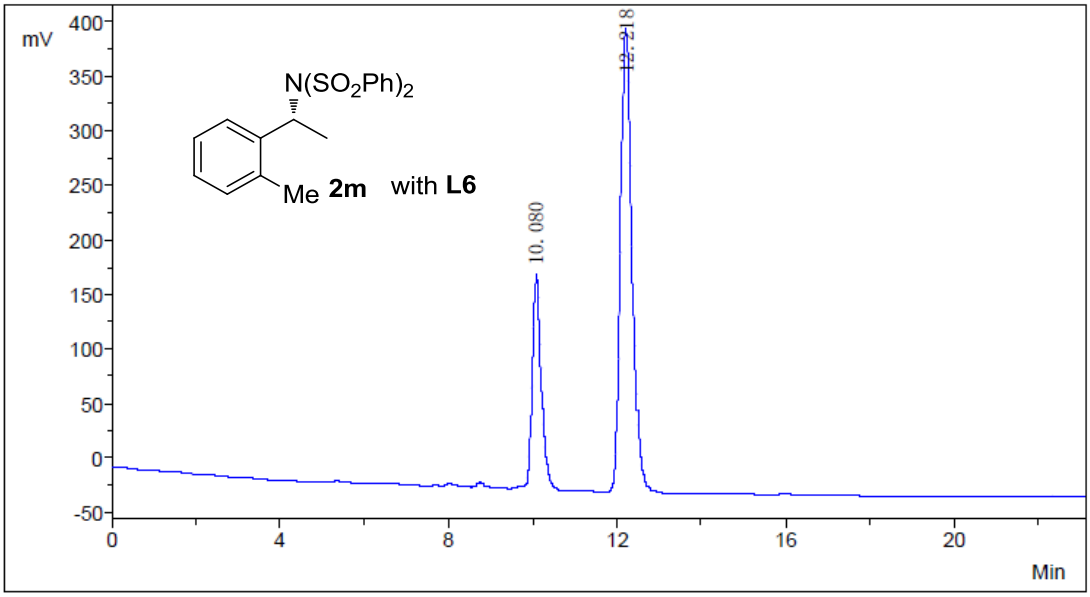

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 7.63 | n.a. | 713.767 | 110.417 | 50.02 | n.a. | BMB* |
| 2 | 10.12 | n.a. | 513.879 | 110.325 | 49.98 | n.a. | BMB* |
| **Total:** | | | 1227.646 | 220.743 | 100.00 | 0.000 | |



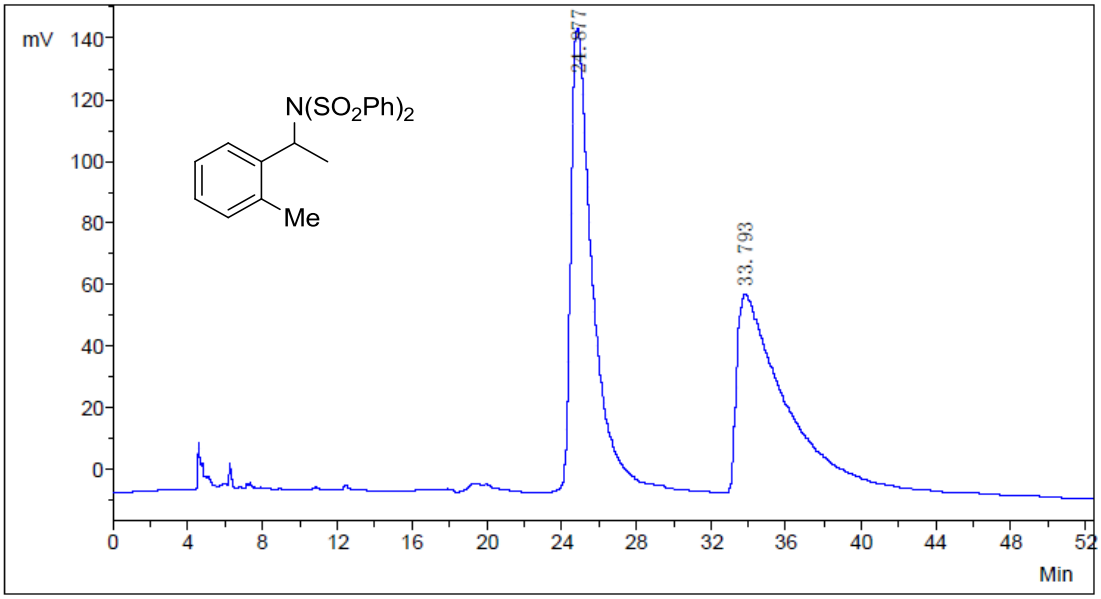

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 7.66 | n.a. | 432.373 | 65.879 | 13.88 | n.a. | BMB* |
| 2 | 10.15 | n.a. | 1927.830 | 408.918 | 86.12 | n.a. | BMB* |
| **Total:** | | | 2360.203 | 474.797 | 100.00 | 0.000 | |



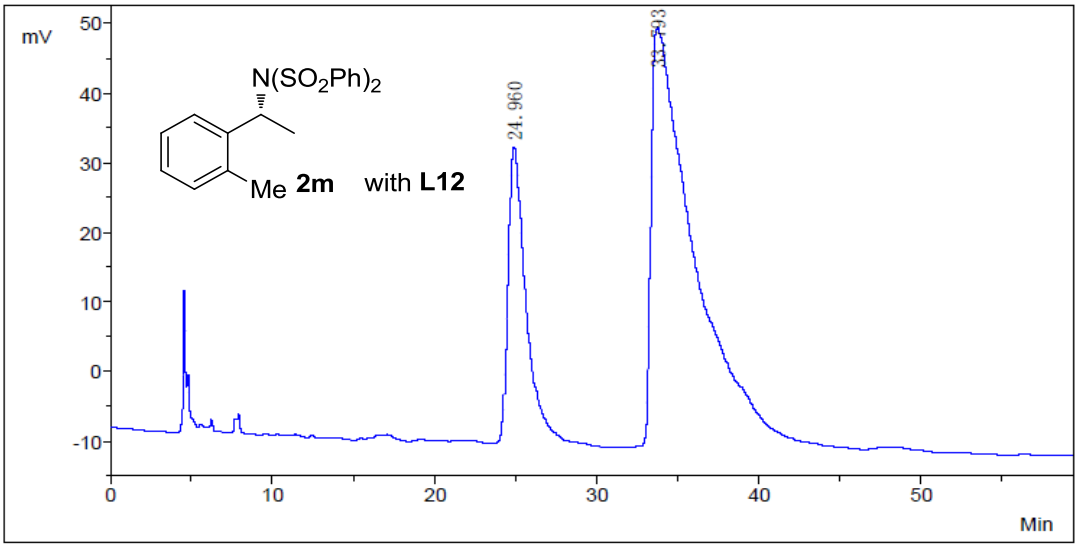

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 10.218 | 586610.1 | 9216559.7 | 49.9724 |
| 2 | 2 | | 12.385 | 472255.2 | 9226731.3 | 50.0276 |
| 合计: | | | | 1058865.3 | 18443291.0 | 100.0000 |



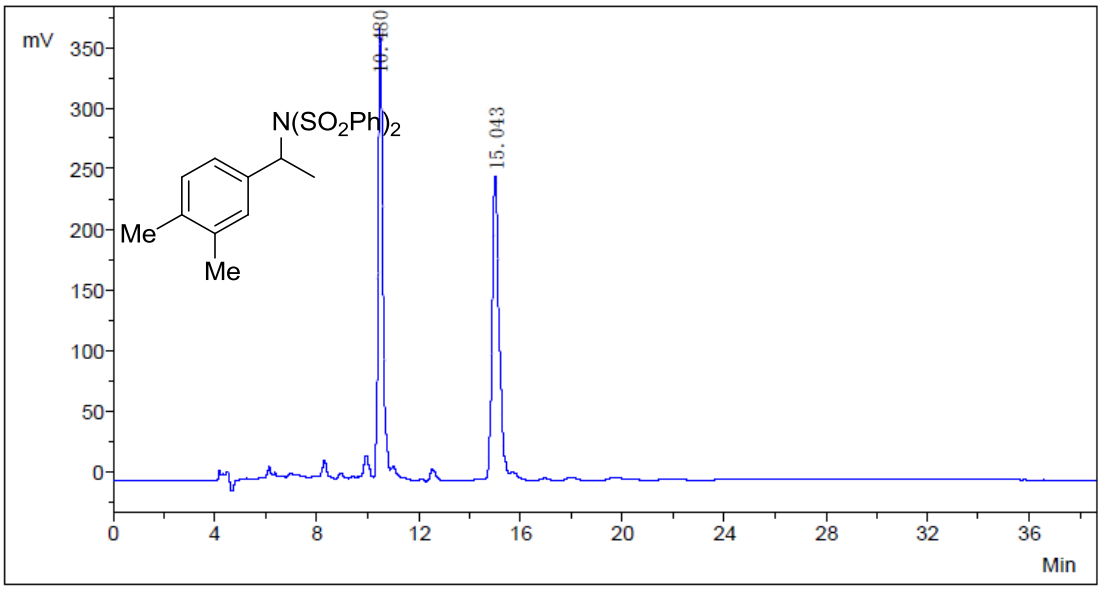

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 10.080 | 197004.6 | 2996825.3 | 26.8654 |
| 2 | 2 | | 12.218 | 423991.2 | 8158134.3 | 73.1346 |
| 合计: | | | | 620995.8 | 11154959.6 | 100.0000 |



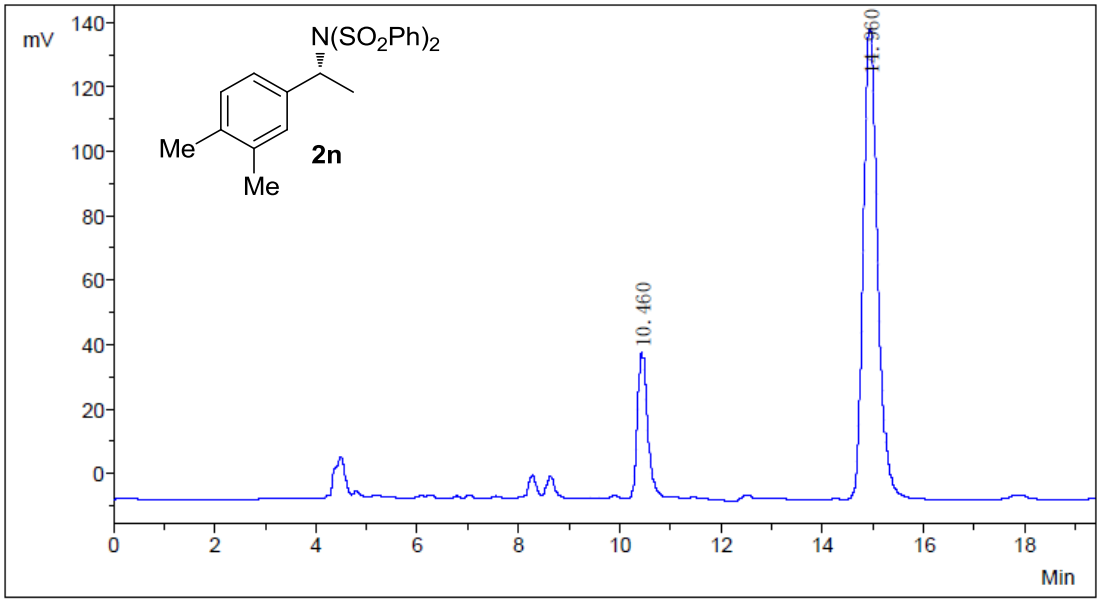

| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 24.877 | 150376.1 | 12326445.8 | 50.0069 |
| 2 | 2 | | 33.793 | 64291.8 | 12323042.2 | 49.9931 |
| 合计: | | | | 214668.0 | 24649488.0 | 100.0000 |




| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 24.960 | 42637.1 | 3383178.6 | 22.4199 |
| 2 | 2 | | 33.793 | 60596.5 | 11706889.4 | 77.5801 |
| 合计: | | | | 103233.6 | 15090068.1 | 100.0000 |




| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 10.480 | 354210.7 | 4445085.6 | 48.0697 |
| 2 | 2 | | 15.043 | 248163.4 | 4802084.6 | 51.9303 |
| 合计: | | | | 602374.1 | 9247170.2 | 100.0000 |




| 序号 | 峰号 | 组份名 | 保留时间 | 峰高 | 峰面积 | 面积百分比(%) |
|---|---|---|---|---|---|---|
| 1 | 1 | | 10.460 | 44688.2 | 683248.7 | 19.1841 |
| 2 | 2 | | 14.960 | 144825.3 | 2878281.4 | 80.8159 |
| 合计: | | | | 189513.5 | 3561530.1 | 100.0000 |