One Pot Iridium-Catalyzed Asymmetrical Double Allylations of Sodium Sulfide: a Fast and Economic Way to Construct Chiral C_2-Symmetric Bis(1-Substituted-Allyl)Sulfane

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Supporting Information

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General: All manipulations were carried out under an argon atmosphere using standard Schlenk techniques. All glassware was oven or flame dried immediately prior to use. All solvents were purified and dried according to standard methods prior to use, unless stated otherwise.

All reagents were obtained from commercial sources and used without further purification. ^{1}H NMR spectra were obtained at 300 MHz or 400 MHz and recorded relative to tetramethylsilane signal (0 ppm) or residual protio-solvent. ^{13}C NMR spectra were obtained at 75 MHz or 100 MHz and chemical shifts were recorded relative to the solvent resonance ($CDCl_3$, 77.0 ppm). ^{19}F NMR spectra were obtained at 282 MHz, and CF_3CO_2H was used as internal standard. Data for ^{1}H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant(s) in Hz, integration). Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm).

The phosphoramidite ligands[1], substituted allylic carbonates[2] were prepared according to known procedures.

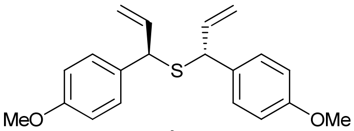
Reference:

1. a) A. Alexakis, S. Rosset, J. Allamand, S. March, F. Guillen, C. Benhaim, *Synlett* **2001**, *9*, 1375; b) R. Naasz, L. A. Arnold, A. J. Minnaard, B. L. Feringa, *Angew. Chem. Int. Ed.* **2001**, *40*, 927; c) K. Tissot-Croset, D. Polet, A. Alexakis, *Synthesis* **2004**, *15*, 2586.

2. P. G. M. Wuts, S. W. Ashford, A. M. Anderson,; J. R. Atkins, *Org. Lett.* **2003**, *5*, 1483.
3. **6f** was prepared according to the literature: Q. Yao, *Org. Lett.* **2002**, *4*, 427.

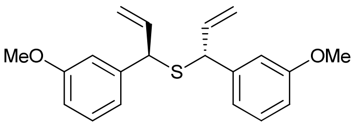
General procedure for the iridium-catalyzed regio-, diastereo-, and enantioselective allylic alkylation of Na_2S • $9H_2O$:

$[Ir(COD)Cl]_2$ (0.002 mmol, 1 mol%), phosphoramidite ligand **1a** [*O*,*O*'-(*S*)-(1,1'-dinaphthyl-2,2'-diyl)-*N*,*N*'-di-(*S,S*)-[phenylethylphosphoramidite] (0.004 mmol, 2 mol%) were dissolved in THF (0.5 mL) and propylamine (0.2 mL) in a dry Schlenk tube filled with argon. The reaction mixture was heated at 50°C for 30 min and then the volatile solvents were removed under vacuum to give a yellow solid. After that, allylic carbonate **3** (0.20 mmol, 100 mol%), Sodium sulfide hydrate **2c** (0.60 mmol, 300 mol %), cesium fluoride (0.60 mmol, 300 mol%), and DCM (2.0 mL) were added. The reaction was stirring at room temperature until **3** was completely consumed. Then the crude reaction mixture was filtrated with celite and the solvent was removed under reduced pressure. The crude residue was purified by flash column chromatography (petroleum ether/dichloromethane) to give the desired products **4**.



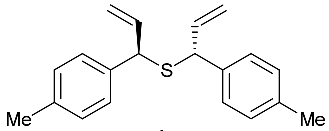
4a

bis((*R*)-1-(4-methoxyphenyl)allyl)sulfane (4a): colorless oil, 99% yield, b/l = >99/1 DL/Meso = 98/2, >99% *ee*. The *ee* of the product was determined by HPLC. [Diacel CHIRALPAK AD-H (0.46 cm x 25 cm); hexane/2-propanol = 98/2; flow rate = 0.7 mL/min; detection wavelength = 214 nm; t_R = 11.544 (meso), 14.450 (major) min]. $[\alpha]_D^{20}$ = -142.0° (c 0.5, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ = 7.24 (d, *J* = 8.4 Hz, 4H), 6.84 (d, *J* = 8.8 Hz, 4H), 6.04 (ddd, *J* = 16.8, 10.2, 8.7 Hz, 2H), 5.16(ddd, *J* = 10.2, J^8 = 1.5, J^8 = 0.6 Hz, 2H), 5.13(ddd, *J* = 16.8, J^8 = 1.2, J^8 = 0.9 Hz, 2H) 4.27 (d, *J* = 8.7 Hz, 1H), 3.77 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 158.7, 138.2, 132.0, 128.9, 115.5, 113.9, 55.2, 51.7. HRMS (EI) calcd for $C_{20}H_{22}O_2S$ (M^+): 326.1341, Found: 326.1345. IR(KBr): ν_{max} (cm^{-1}) = 3902, 3852, 3751, 3648, 3629, 2834, 1866, 1772, 1538, 1504, 1456, 1247, 1174, 1032, 830, 419.



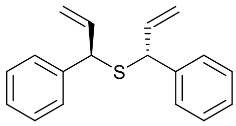
4b

bis((*R*)-1-(3-methoxyphenyl)allyl)sulfane (4b): colorless oil, 99% yield, b/l = >99/1, DL/Meso = 97/3, 97% *ee*. The *ee* of the product was determined by HPLC. [Diacel CHIRALCEL OJ-H (0.46 cm x 25 cm); hexane/2-propanol = 80/20; flow rate = 0.7 mL/min; detection wavelength = 214 nm; t_R = 31.400 (major), 34.869 (meso), 56.381 (minor) min]. $[\alpha]_D^{20}$ = -110.0° (c 0.7, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.23 (t, *J* = 7.8 Hz, 2H), 6.91 (d, *J* = 7.8 Hz, 2H), 6.87 (s, 2H), 6.79 (d, *J* = 8.1 Hz, 2H), 6.05 (ddd, *J* = 17.4, 9.6, 9.0 Hz, 2H), 5.18 (d, *J* = 9.3 Hz, 2H), 5.16 (d, *J* = 17.4 Hz, 2H), 4.29 (d, *J* = 8.7 Hz, 2H), 3.79 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 159.7, 141.5, 137.9, 129.5, 120.2, 115.9, 113.3, 112.9, 55.1, 52.5. HRMS (EI) calcd for $C_{20}H_{22}O_2S$ (M^+): 326.1341, Found: 326.1344. IR(KBr): ν_{max} (cm^{-1}) = 3851, 3749, 3645, 3000, 2967, 2833, 1595, 1485, 1264, 1149, 1047, 919, 764, 693, 455.



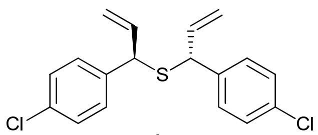
4c

bis((*R*)-1-*p*-tolylallyl)sulfane (4c): colorless oil, 99% yield, b/l = >99/1, DL/Meso = 96/4, >99% *ee*. The *ee* of the product was determined by HPLC. [Diacel CHIRALCEL OJ-H (0.46 cm x 15 cm); hexane/2-propanol = 97/3; flow rate = 1.0 mL/min; detection wavelength = 214 nm; t_R = 8.245 (major), 12.279 (minor), 14.984 (meso) min]. $[\alpha]_D^{20}$ = -97.8° (c 0.3, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.21 (d, *J* = 7.8 Hz, 4H), 7.12 (d, *J* = 7.8 Hz, 4H), 6.05 (ddd, *J* = 16.8, 9.9, 8.7 Hz, 2H), 5.15 (d, *J* = 9.3 Hz, 2H), 5.13 (d, *J* = 17.4 Hz, 2H), 4.28 (d, *J* = 8.7 Hz, 2H), 2.32 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 138.2, 137.0, 136.9, 129.3, 127.8, 115.7, 52.2, 20.9. HRMS (EI) calcd for $C_{20}H_{22}S$ (M^+): 294.1442, Found: 294.1444. IR(KBr): ν_{max} (cm^{-1}) = 2913, 2847, 1681, 1509, 1010, 798, 506, 470.



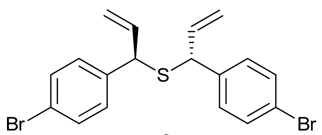
4d

bis((*R*)-1-phenylallyl)sulfane (4d): colorless oil, 99% yield, b/l = 96/4, DL/Meso = 95/5, 96% *ee*. The *ee* of the product was determined by HPLC. [Diacel CHIRALCEL OJ-H (0.46 cm x 25 cm); hexane/2-propanol = 90/10; flow rate = 0.7 mL/min; detection wavelength = 214 nm; t_R = 28.335 (meso), 29.566 (minor), 30.970 (major) min]. $[\alpha]_D^{20}$ = -130.4° (c 0.5, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.32-7.23 (m, 10H), 6.07 (ddd, *J* = 17.1, 9.6 Hz, 8.7, 2H), 5.18 (d, *J* = 9.6, 2H), 5.15 (d, *J* = 17.1, 2H), 4.32 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 140.1, 138.1, 128.6, 127.9, 127.3, 115.9, 52.4. HRMS (EI) calcd for $C_{18}H_{18}S$ (M^+): 226.1129, Found: 226.1128. IR(KBr): ν_{max} (cm^{-1}) = 3903, 3853, 3750, 3735, 3675, 3648, 3628, 3566, 3028, 1868, 1770, 1730, 1679, 1557, 1505, 1455, 985, 914, 696, 423.



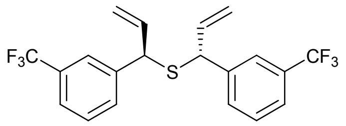
4e

bis((*R*)-1-(4-chlorophenyl)allyl)sulfane (4e): colorless oil. 80% yield, b/l = >99/1, DL/Meso = 96/4, >99% ee. The ee of the product was determined by HPLC. [Diacel CHIRALPAK AD-H (0.46 cm x 25 cm); hexane/2-propanol = 98/2; flow rate = 0.7 mL/min; detection wavelength = 214 nm; t_R = 5.382 (meso), 5.662 (major) min]. $[\alpha]_D^{20}$ = -166.8° (c 0.5, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.29 (d, *J* = 8.4 Hz, 4H), 7.23 (d, *J* = 8.4 Hz, 4H), 6.00 (ddd, *J* = 17.1, 9.9, 8.4 Hz, 2H), 5.20 (d, *J* = 10.5 Hz, 2H), 5.14 (d, *J* = 17.1 Hz, 2H), 4.26 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 138.4, 137.3, 133.2, 129.3, 128.8, 116.6, 51.7. HRMS (EI) calcd for $C_{18}H_{16}SCl_2$ (M^+): 334.0350, Found: 334.0349. IR(KBr): ν_{max} (cm^{-1}) = 3903, 3869, 3852, 3837, 3750, 3735, 3710, 3689, 3676, 3648, 3627, 3565, 3077, 1718, 1651, 1557, 1539, 1505, 1487, 1455, 1091, 1013, 920, 820.



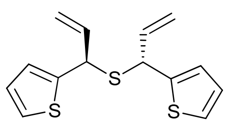
4f

bis((*R*)-1-(4-bromophenyl)allyl)sulfane (4f): colorless oil, 72% yield, b/l = 98/2, DL/Meso = 97/3, >99% ee. The ee of the product was determined by HPLC. [Diacel CHIRALCEL AY-H (0.46 cm x 25 cm); hexane/2-propanol = 99.5/0.5; flow rate = 0.8 mL/min; detection wavelength = 214 nm; t_R = 6.285 (meso), 7.103 (major) min]. $[\alpha]_D^{20}$ = -117.6° (c 0.5, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.44 (d, *J* = 8.4 Hz, 4H), 7.17 (d, *J* = 8.4 Hz, 4H), 5.99 (ddd, *J* = 16.8, 9.9, 8.7 Hz, 2H), 5.20 (d, *J* = 9.9 Hz, 2H), 5.13 (d, *J* = 16.8 Hz, 2H), 4.24 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 138.8, 137.3, 131.7, 129.7, 121.2, 116.6, 51.7. HRMS (EI) calcd for $C_{18}H_{16}S\ ^{79}Br_2$ (M^+): 421.9339, Found: 421.9348. IR(KBr): ν_{max} (cm^{-1}) = 3903, 3852, 3802, 3751, 3734, 3676, 3649, 3628, 3567, 2925, 2363, 1870, 1732, 1717.89, 1653, 1560, 1486, 1455, 1286, 1071, 1009, 919, 815, 750, 517, 418.



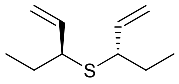
4g

bis((*R*)-1-(3-(trifluoromethyl)phenyl)allyl)sulfane (4g): colorless oil, 67% yield, b/l = 97/3, DL/Meso = 96/4, >99% ee. The ee of the product was determined by HPLC. [Diacel CHIRALCEL OJ-H (0.46 cm x 15 cm); hexane/2-propanol = 99.8/0.2; flow rate = 1.0 mL/min; detection wavelength = 214 nm; t_R = 3.162 (meso), 3.631 (major) min]. $[\alpha]_D^{20}$ = -113.8° (c 0.7, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.54-7.51 (m, 6H), 7.49-7.39 (m, 2H), 6.05 (ddd, *J* = 17.1, 9.9, 8.7 Hz, 2H), 5.26 (d, *J* = 9.9 Hz, 2H), 5.19 (d, *J* = 17.1 Hz, 2H), 4.37 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 140.8, 136.9, 131.4, 131.1 (q, *J* = 30.0 Hz), 129.2, 124.8 (q, *J* = 3.8 Hz), 124.4 (q, *J* = 3.8 Hz), 124.0 (q, *J* = 270.7 Hz), 117.2, 52.1. ^{19}F NMR (282 MHz, $CDCl_3$) δ = -62.60(s). HRMS (EI) calcd for $C_{20}H_{16}F_6S$ (M^+): 402.0877, Found: 402.0878. IR(KBr): ν_{max} (cm^{-1}) = 3851, 3733, 3646, 1714, 1633, 1555, 1447, 1332, 1165, 1125, 1073, 924, 770, 702, 418.



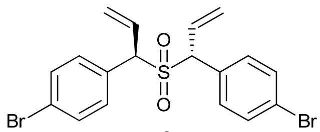
4h

bis((*R*)-1-(thiophen-2-yl)allyl)sulfane (4h): colorless oil. 99% yield, b/l = >99/1, DL/Meso = >99/1, >99% ee. The ee of the product was determined by HPLC. [Diacel CHIRALCEL OJ-H (0.46 cm x 15 cm); Hexane/ n-propanol (0.1%DEA) =99/1 (v/v); flow rate = 1.0 mL/min; detection wavelength = 214 nm; t_R = 13.363 (meso), 14.090 (major), 19.642 (minor) min]. $[\alpha]_D^{20}$ = -63.6° (c 0.5, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.23 (d, *J* = 5.1 Hz, 2H), 7.08-6.89 (m, 4H), 6.06 (ddd, *J* = 16.8, 9.9, 8.7 Hz, 2H), 5.24 (d, *J* = 9.0 Hz, 2H), 5.19 (d, *J* = 15.9 Hz, 2H), 4.65 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 143.3, 137.6, 126.7, 125.2, 124.9, 116.6, 47.7. HRMS (EI) calcd for $C_{14}H_{14}S\ _3$(M^+): 278.0258, Found: 278.0260. IR(KBr): ν_{max} (cm^{-1}) = 3903, 3852, 3819, 3749, 3735, 3675, 3648 3628, 3566, 3080, 1869, 1716, 1653, 1635, 1558, 1541, 1506, 1232, 986, 920, 853, 696.



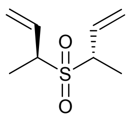
4i

di(*S*)-pent-1-en-3-ylsulfane 4i: colorless oil, 84% yield, b/l = 81/19, DL/Meso =85/15, 98% ee. Determination of the ee of **4i** was performed by GC on a Rtx-13100 (30m x 0.25mm x 0.25um) column, He-flow 1.0 ml/min., split ratio: 100:1. oven temp. 150 °C, Hold 10 min, inlet temp: 250 °C., t_R =3.226 (major), 3.415 (minor), 4.103 (meso) min. $[\alpha]_D^{20}$ = -85.0° (c 0.1, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.57 (dt, *J* = 16.8, 10.0 Hz, 2H), 5.05 (dd, *J* = 10.0, J^5 = 1.6 Hz, 2H), 4.94 (ddd, *J* = 16.8, J^5 = 1.6 Hz, J^5 = 0.4 Hz, 2H), 3.08-3.00 (m, 2H), 1.70-1.48 (m, 4H), 0.95 (t, *J* = 7.6 Hz, 6H).



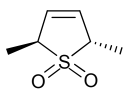
6f

4,4'-(1*R*,1'*R*)-1,1'-sulfonylbis(prop-2-ene-1,1-diyl)bis(bromobenzene) (6f) [3]: white powder. mp: 147.6-149.5, 73% yield, 99% ee. The ee of the product was determined by [Diacel CHIRALPAK AD (0.46 cm x 25 cm); Hexane/ *i*-propanol=90/10 (v/v); flow rate = 1.0 mL/min; detection wavelength = 214 nm; t_R = 21.719 (major)]. $[\alpha]_D^{20}$ = -69.0° (c 0.6, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.52 (d, *J* = 8.4 Hz, 4H), 7.24 (d, *J* = 8.0 Hz, 4H), 6.28 (ddd, *J* = 16.8, 10.0, 8.8 Hz, 2H), 5.56 (d, *J* = 10.4 Hz, 2H), 5.40 (d, *J* = 17.2 Hz, 1H), 4.76 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 132.1, 131.4, 130.2, 129.8, 123.8, 123.6, 69.0. HRMS (ESI-) calcd for $C_{18}H_{15}O_2SBr_2(M^-)$: 452.9159, Found: 452.9165. IR(KBr): ν_{max} (cm^{-1}) = 3850, 3647, 1482, 1402, 1314, 1275, 1257, 1128, 1071, 1005, 939, 833, 762, 749, 713, 630, 510.



6j

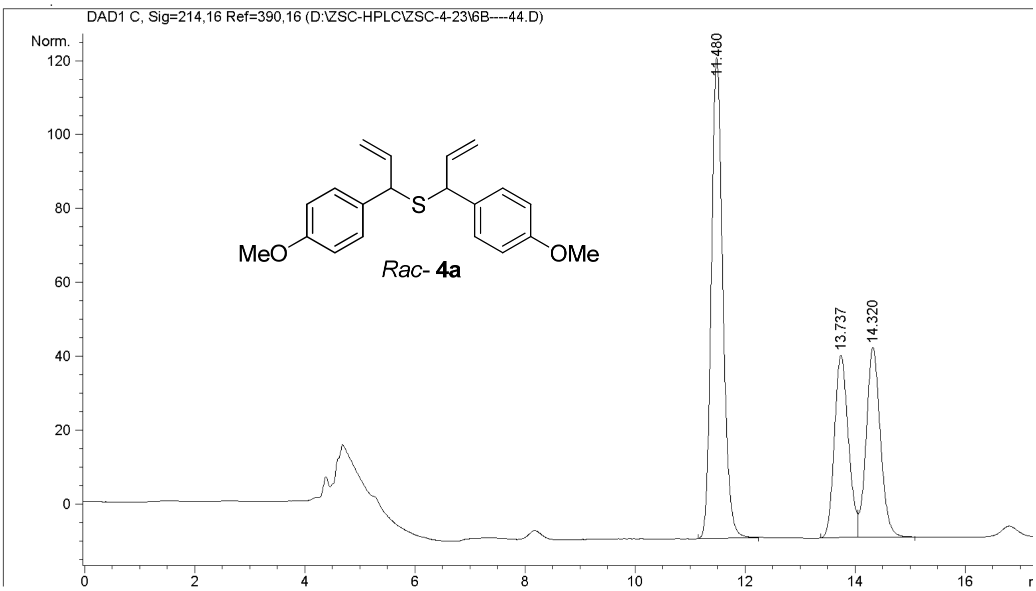
(*S*)-3-((*S*)-but-3-en-2-ylsulfonyl)but-1-ene (6j): (51% one pot yield from **3j**, DL/Meso =89/11,b/l =87/13, 99% ee. The ee of the product was determined by [Diacel CHIRALCEL IC (0.46 cm x 15 cm); Hexane/ n-propanol=90/10 (v/v); flow rate = 1.0 mL/min; detection wavelength = 214 nm; t_R = 29.080 (major), 34.088 (minor) min]. $[\alpha]_D^{20}$ = -118.3° (c 0.1, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.86 (ddd, *J* = 17.2, 10.4, 8.8 Hz, 2H), 5.41 (d, *J* = 10.0Hz, 2H), 5.34 (d, *J* = 17.2 Hz, 2H), 3.88 (dq, *J* = 8.8, 7.2 Hz, 1H).



7j

(2*S*,5*S*)-2,5-dimethyl-2,5-dihydrothiophene 1,l-dioxide (7j): To a solution of **6j** (17.0 mg, 0.1 mmol) in 4 mL CH_2Cl_2 was added Grubbs catalyst 1st (3.3 mg, 0.004 mmol) and the reaction mixture was heated to reflux under Ar over night and could be monitored by TLC. The reaction was then cooled to rt and concentrated to dryness under vacuum after **6j** was completely consumed. Flash chromatography on silica gel (petroleum ether/ethyl acetate = 5:1) gave **7j** (12.3 mg, 86% yield, 94% ee) as a colorless thick oil. The ee of the product was determined by [Diacel CHIRALPAK AD-H (0.46 cm x 25 cm); Hexane/ *i*-propanol=90/10 (v/v); flow rate = 1.0 mL/min; detection wavelength = 214 nm; t_R = 15.424 (minor), 17.364 (major) min] to be 94%. $[\alpha]_D^{20}$ = -128.2° (c 0.2, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ =5.95 (s, 1H), 3.73 (q, *J* = 6.8 Hz, 1H), 1.44 (d, *J* = 7.2 Hz, 3H).

HPLC Chromatograms of the Chiral Compounds



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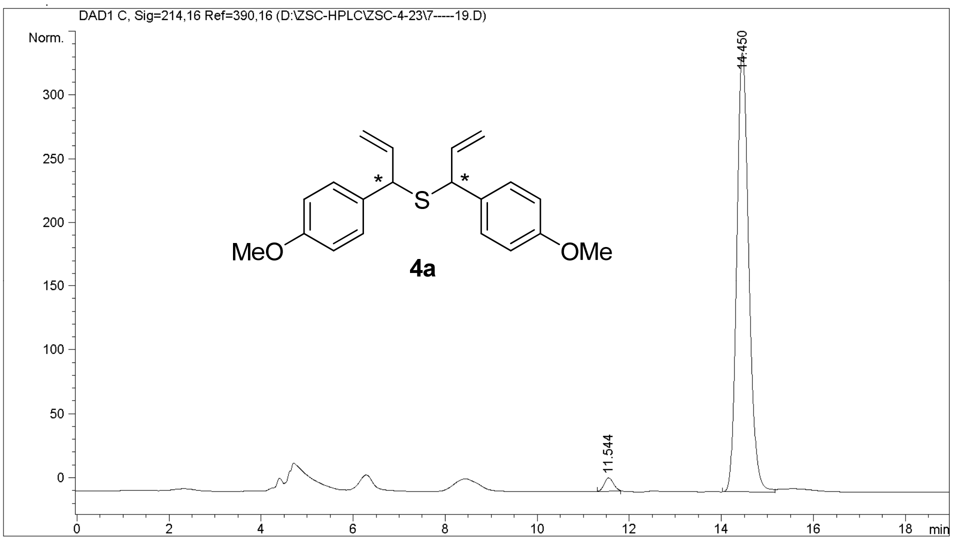
2	13.737	BV	0.2736	870.00647	23.5615
3	14.320	VB	0.2785	926.35590	25.0875

Totals : 3692.49493



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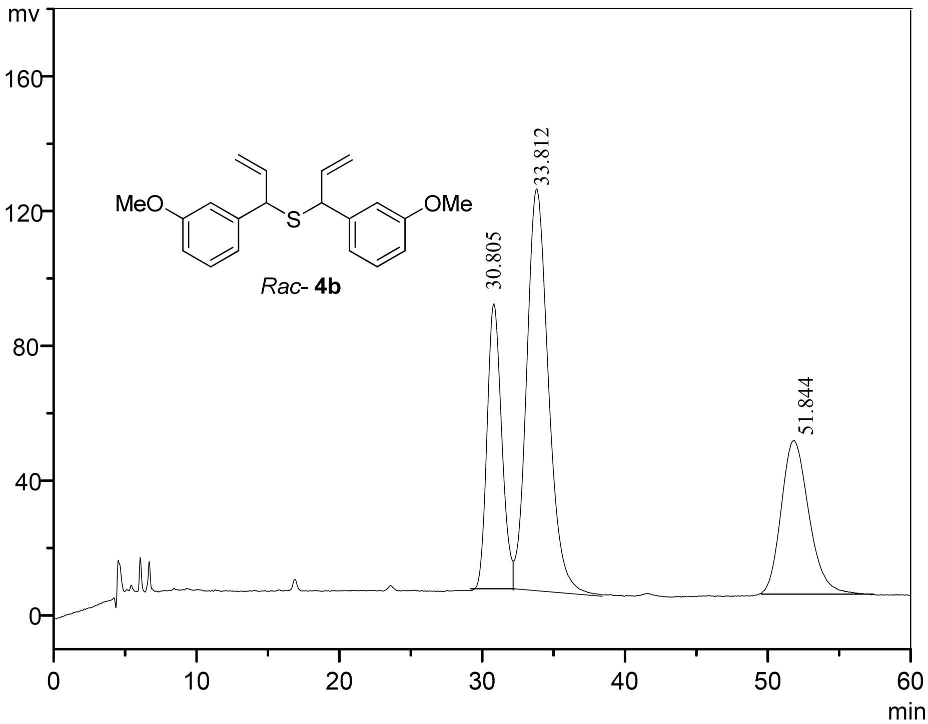
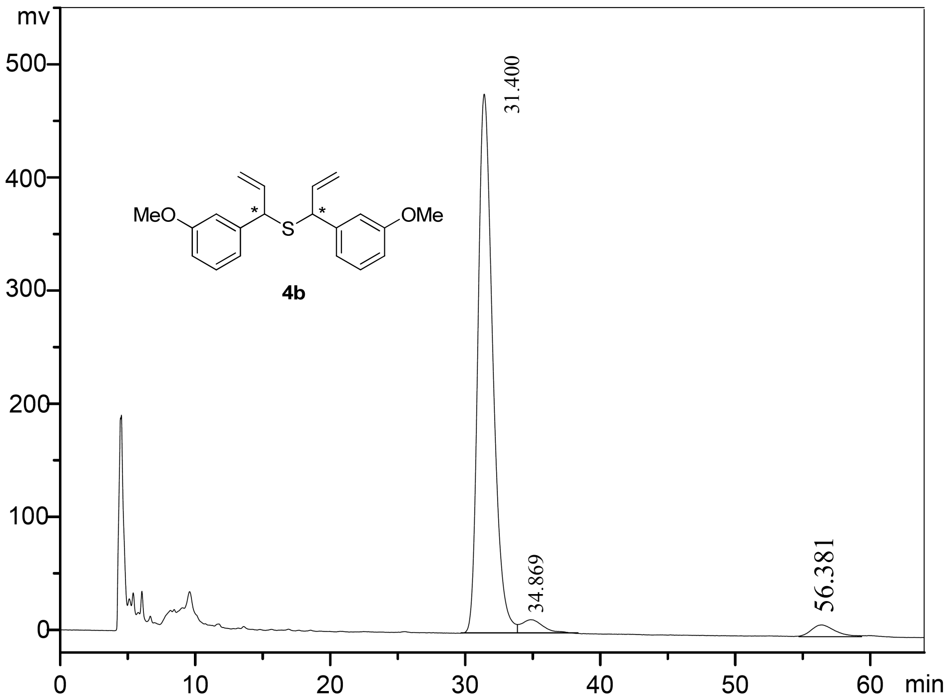
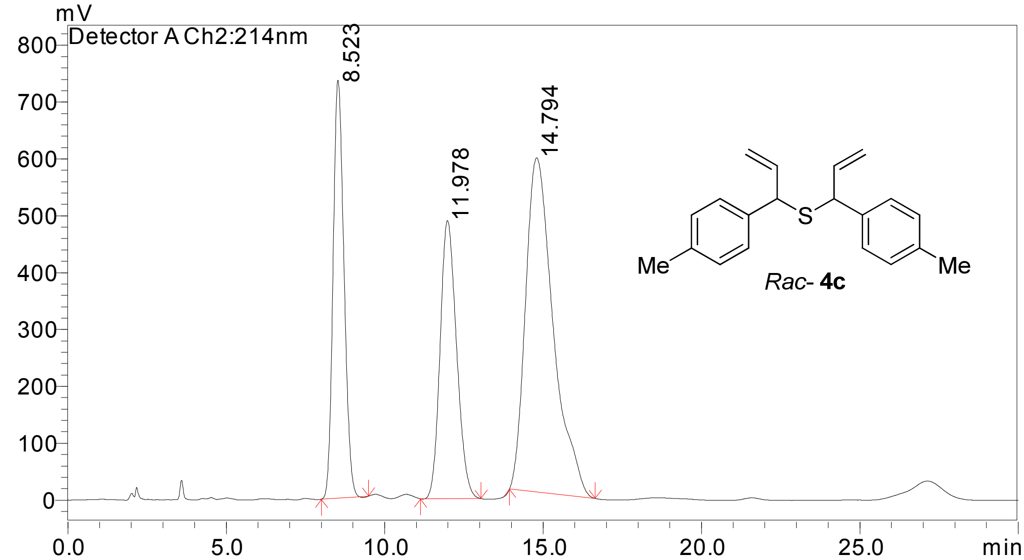


Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Area(%)
1	30.805	29.250	32.200	6155106	85245	72.2050	24.8525
2	33.812	32.200	37.408	12625318	19595	105.5680	50.9773
3	51.844	49.208	55.117	5986117	45389	131.8840	24.1702



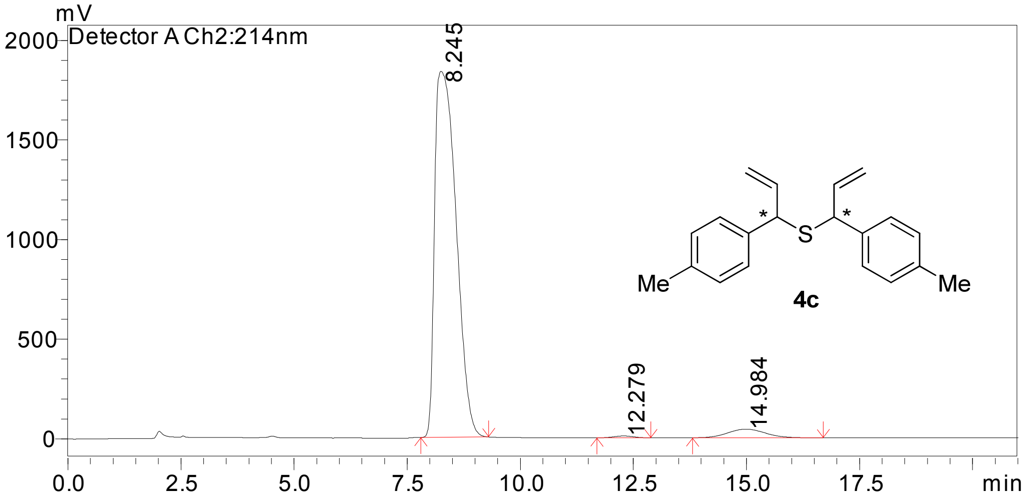
Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Area(%)
1	31.400	29.975	33.992	36501911	476972	76.5280	94.1760
2	34.869	33.992	37.208	1154867	11566	99.8500	2.9796
3	56.381	54.542	59.208	1102470	9828	112.1790	2.8444

p-Me



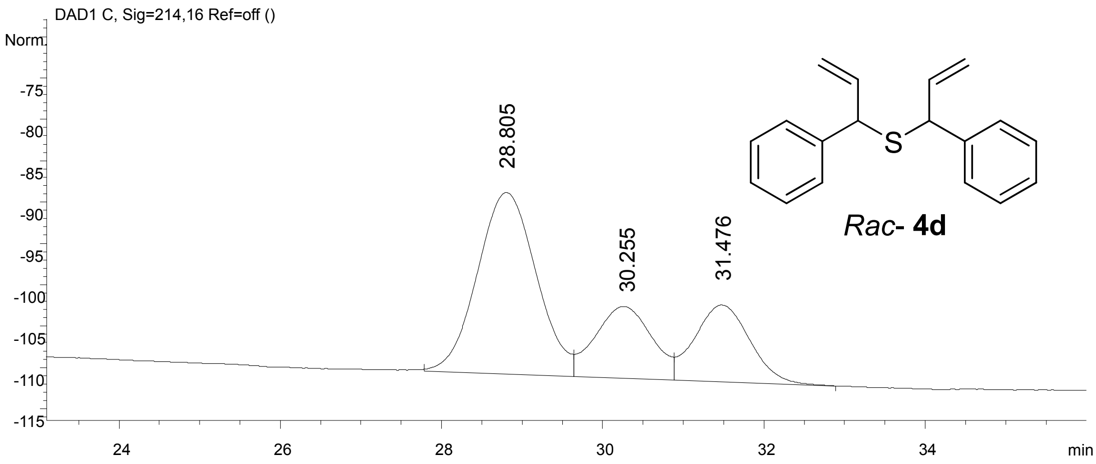
<Column Performance Report>

Peak No.	Time	Area	Area %	Plate number	Tailing	Resolution
1	8.523	17601834	24.7754	2826.184	1.180	--
2	11.978	17682081	24.8883	2477.761	1.189	4.309
3	14.794	35761802	50.3363	1463.590	1.482	2.244



<Column Performance Report>

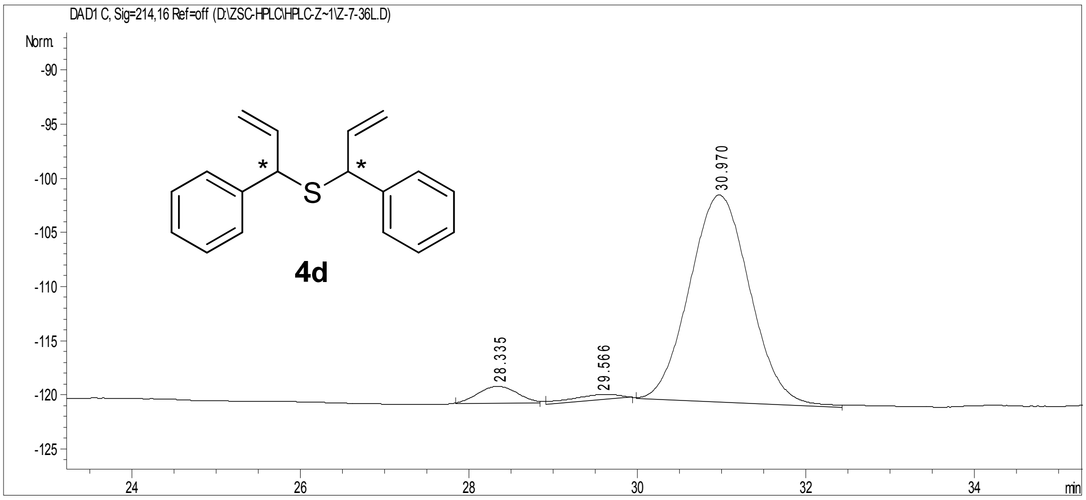
Peak No.	Time	Area	Area %	Plate number	Tailing	Resolution
1	8.245	60716124	95.5165	1630.170	1.834	--
2	12.279	293811	0.4622	3637.236	1.013	4.947
3	14.984	2556169	4.0213	1528.926	1.091	2.305



Signal 2: DAD1 C, Sig=214,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	28.805	BV	0.7885	1114.59607	56.3202
2	30.255	VV	0.7390	422.14209	21.3307
3	31.476	VB	0.7354	442.29727	22.3491

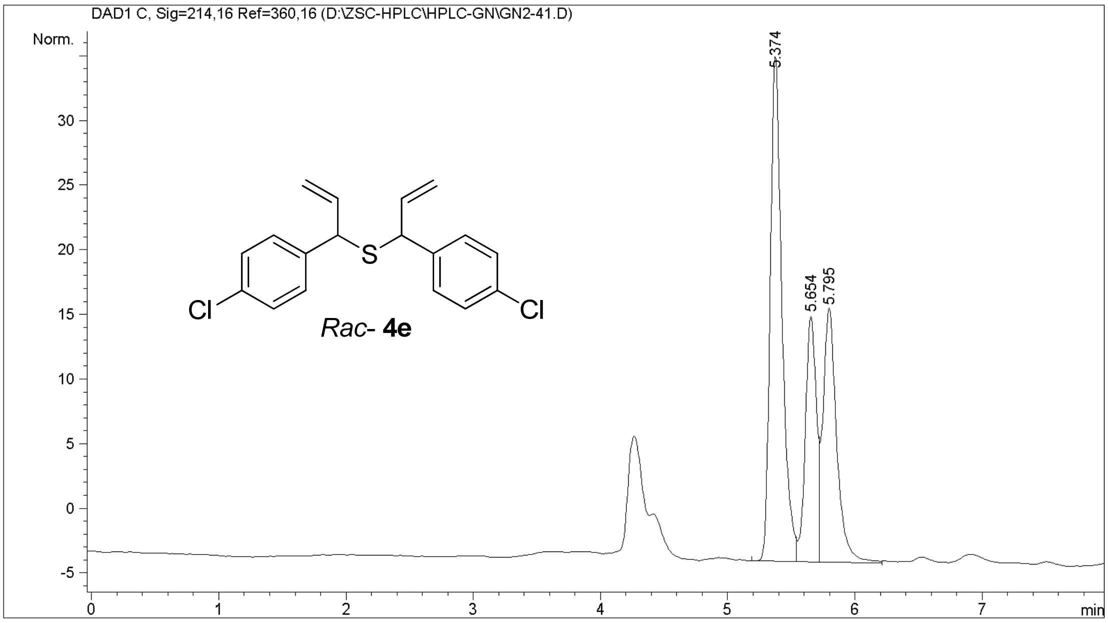
Totals : 1979.03543



Signal 2: DAD1 C, Sig=214,16 Ref=off

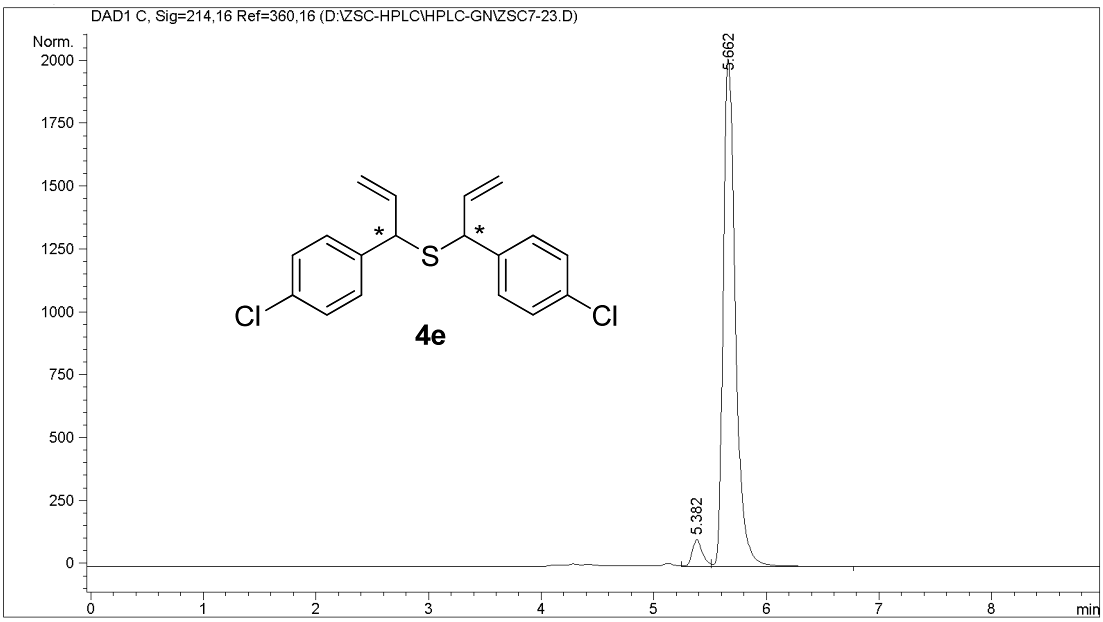
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	28.335	MM	0.5436	50.97386	4.9860
2	29.566	MM	0.6712	19.84482	1.9411
3	30.970	MM	0.8289	951.51422	93.0728

Totals : 1022.33290



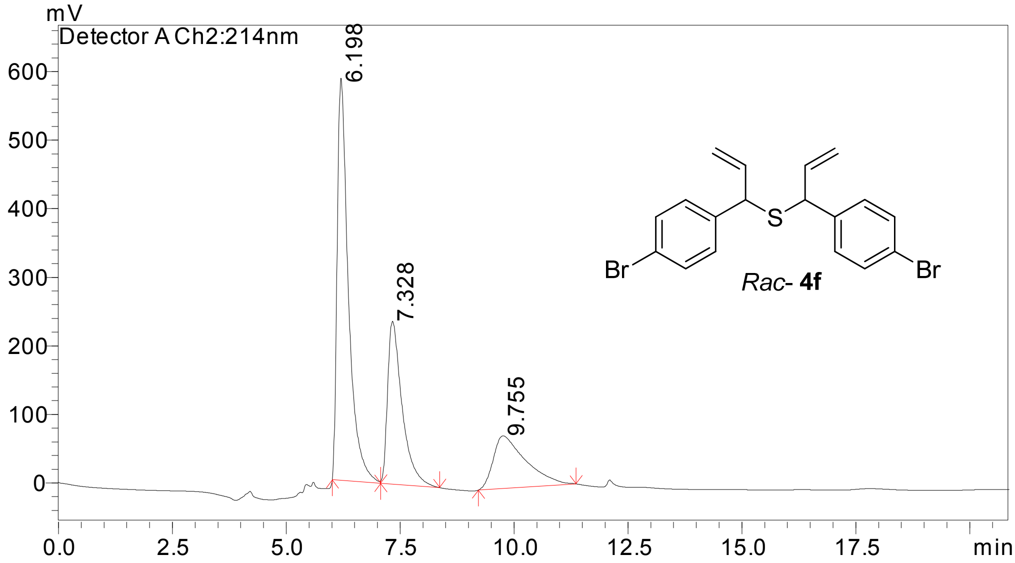
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	5.374	VV	0.1034	252.31023	48.8393
2	5.654	VV	0.0955	116.52006	22.5546
3	5.795	VB	0.1120	147.78236	28.6060

Totals : 516.61265



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	5.382	VV	0.0985	674.67163	4.2379
2	5.662	VV	0.1208	1.52454e4	95.7621

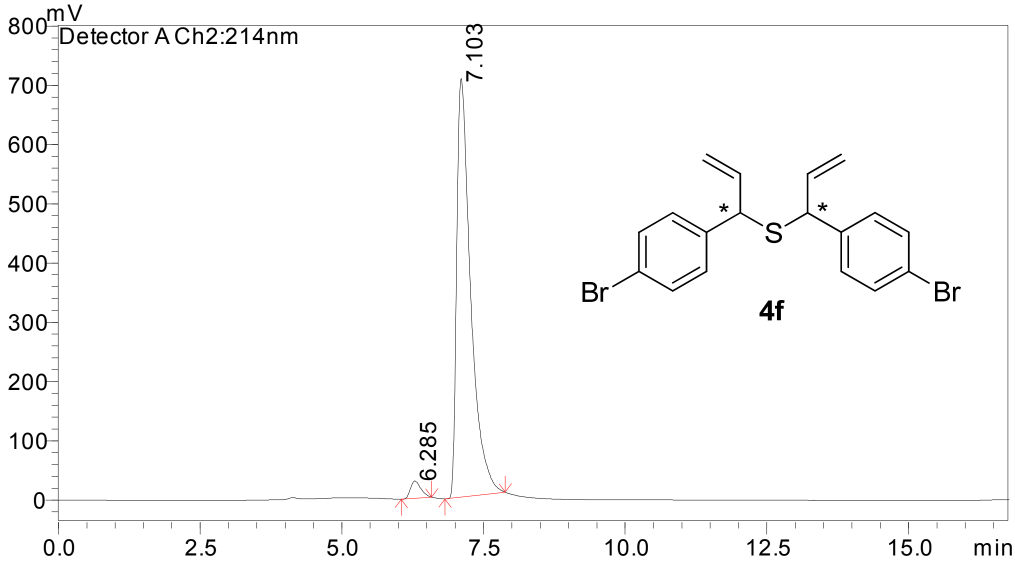
< Chromatogram >



<Column Performance Report>

Peak No.	Time	Area	Area %	Plate number	Tailing	Resolution
1	6.198	10084571	52.5781	3407.147	2.055	--
2	7.328	5288720	27.5739	2789.017	1.977	2.307
3	9.755	3806895	19.8481	913.973	1.938	2.631

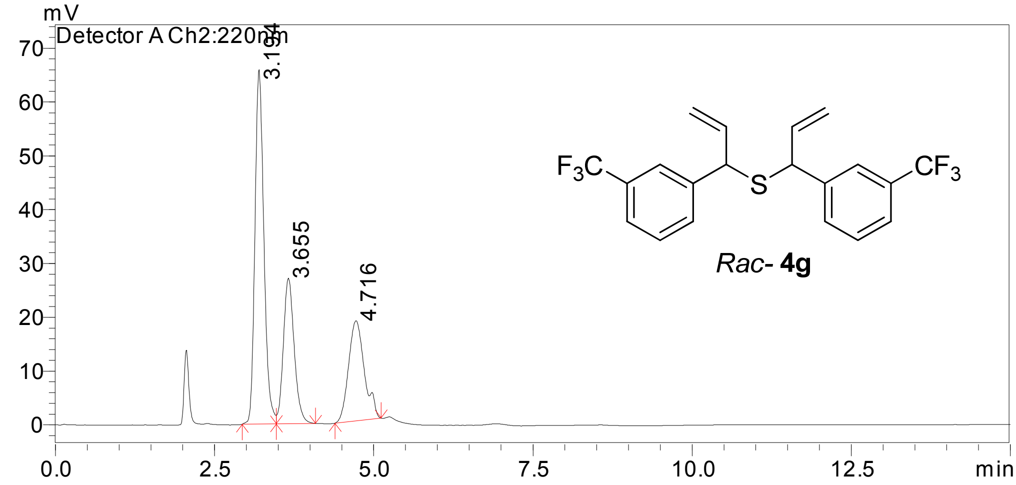
< Chromatogram >



<Column Performance Report>

Peak No.	Time	Area	Area %	Plate number	Tailing	Resolution
1	6.285	373963	2.9947	5111.440	1.354	--
2	7.103	12113495	97.0053	4338.391	2.158	2.089

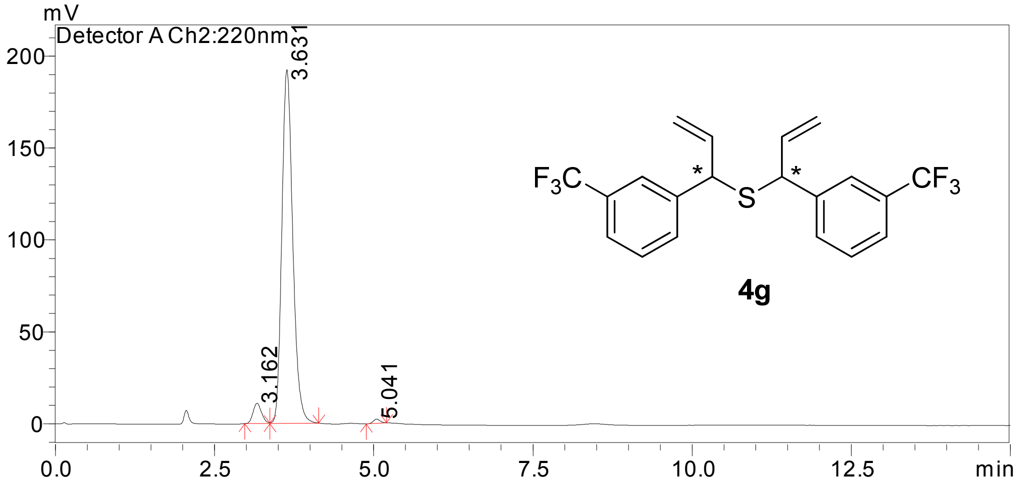
< Chromatogram >



<Column Performance Report>

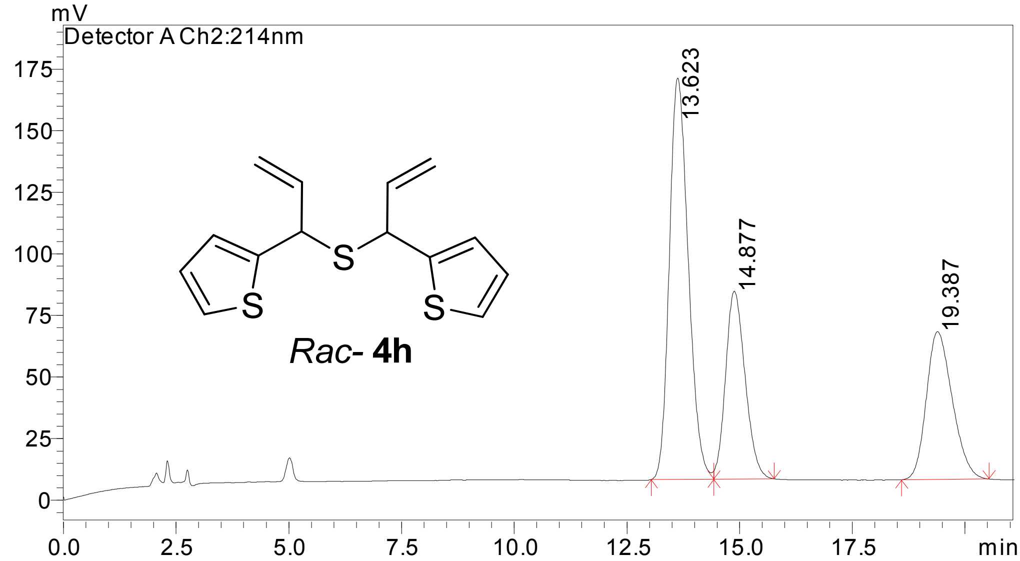
Peak No.	Time	Area	Area %	Plate number	Tailing	Resolution
1	3.194	640033	49.7984	2404.719	1.272	--
2	3.655	321517	25.0160	2128.590	--	1.597
3	4.716	323697	25.1856	1830.644	1.175	2.800

< Chromatogram >



<Column Performance Report>

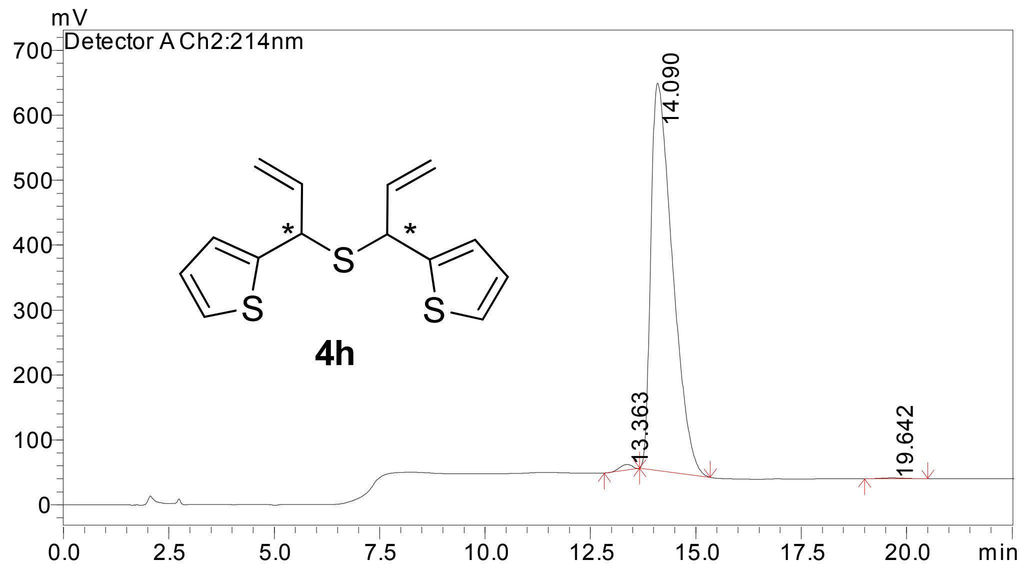
Peak No.	Time	Area	Area %	Plate number	Tailing	Resolution
1	3.162	101463	4.1895	2513.122	1.189	--
2	3.631	2303395	95.1091	2079.799	1.236	1.643
3	5.041	16988	0.7014	10159.847	1.153	5.439



<Column Performance Report>

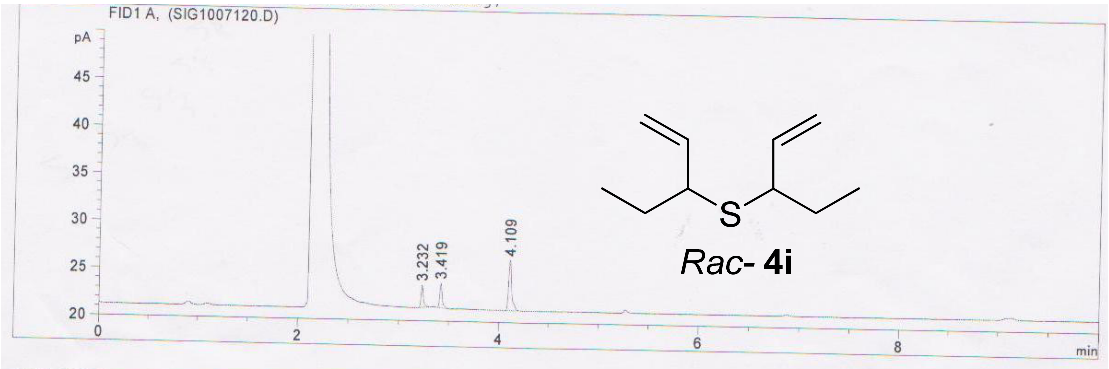
Peak No.	Time	Area	Area %	Plate number	Tailing	Resolution
1	13.623	4778519	50.3680	4909.432	1.227	--
2	14.877	2303029	24.2751	5510.176	1.182	1.589
3	19.387	2405669	25.3570	5288.922	1.245	4.829

< Chromatogram >



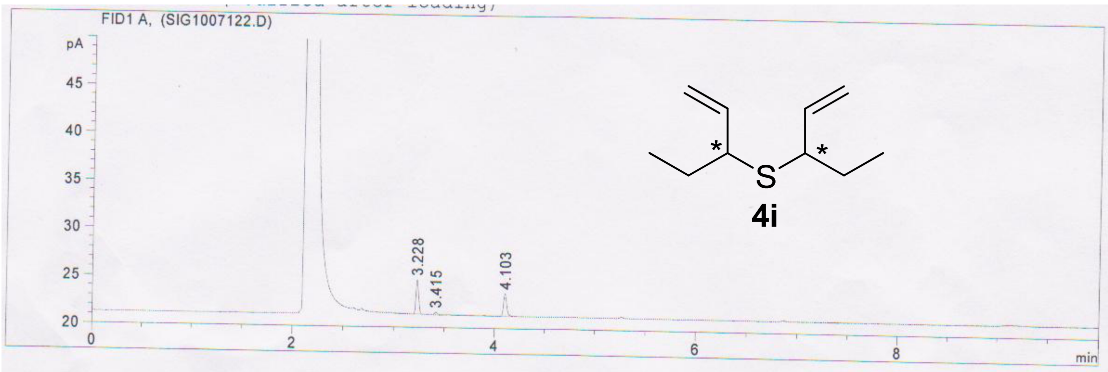
<Column Performance Report>

Peak No.	Time	Area	Area %	Plate number	Tailing	Resolution
1	13.363	164194	0.7609	10088.564	0.858	--
2	14.090	21362529	99.0000	3419.560	1.871	0.972
3	19.642	51585	0.2391	6865.840	1.075	5.808



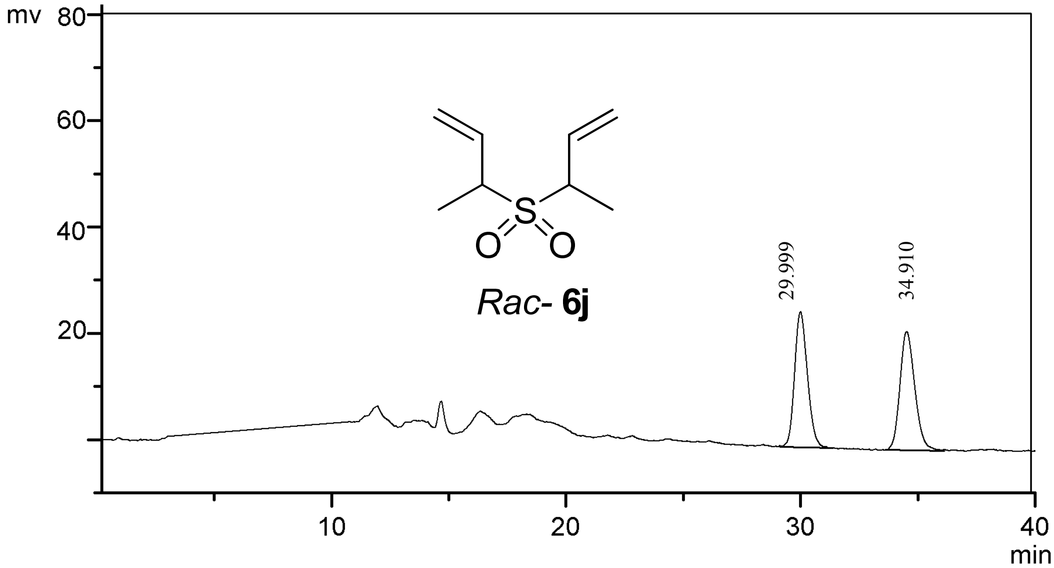
Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	3.232	BB	0.0258	3.97708	2.33805	19.66776
2	3.419	BB	0.0269	4.57960	2.55253	22.64735
3	4.109	BB	0.0328	11.66465	5.27358	57.68488

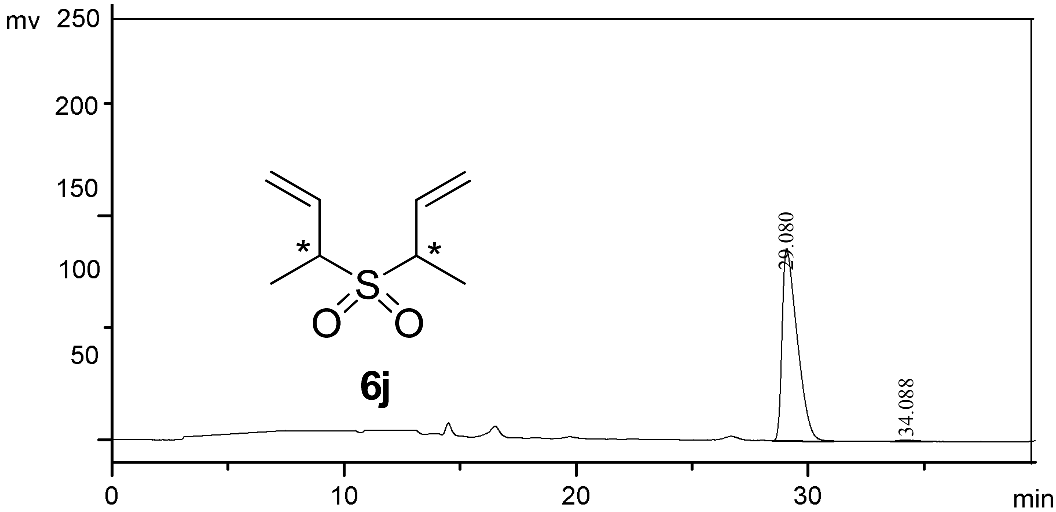


Signal 1: FID1 A,

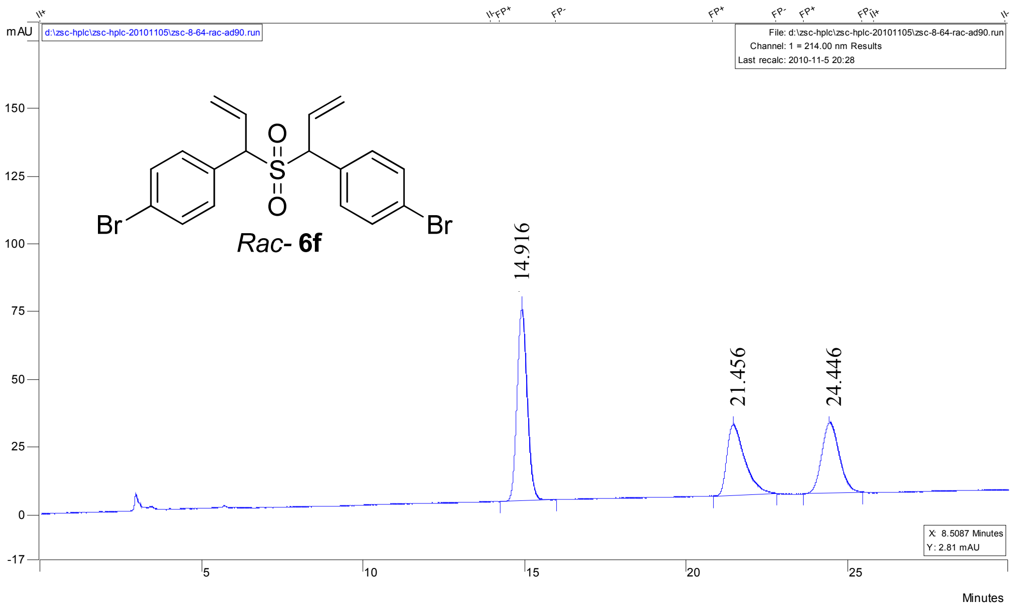
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	3.228	BB	0.0284	6.49595	3.53871	53.63368
2	3.415	BB	0.0308	5.16381e-1	2.53605e-1	4.26349
3	4.103	BB	0.0336	5.09937	2.32008	42.10283



Peak#	R.Time	I.Time	F.Time	Area	Height	Area (%)
1	29.999	29.150	31.317	982440	25591	49.8045
2	34.516	33.658	36.233	990152	22325	50.1955

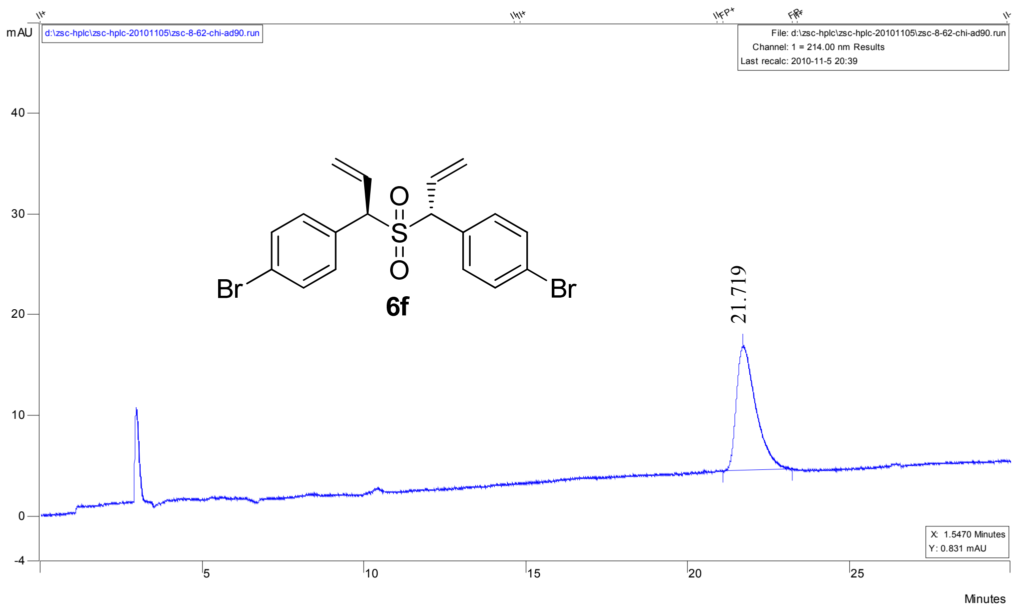


Peak#	R.Time	I.Time	F.Time	Area	Height	Area (%)
1	29.080	28.500	31.000	7754165	171530	99.2779
2	34.088	33.433	35.250	56403	1037	0.7221



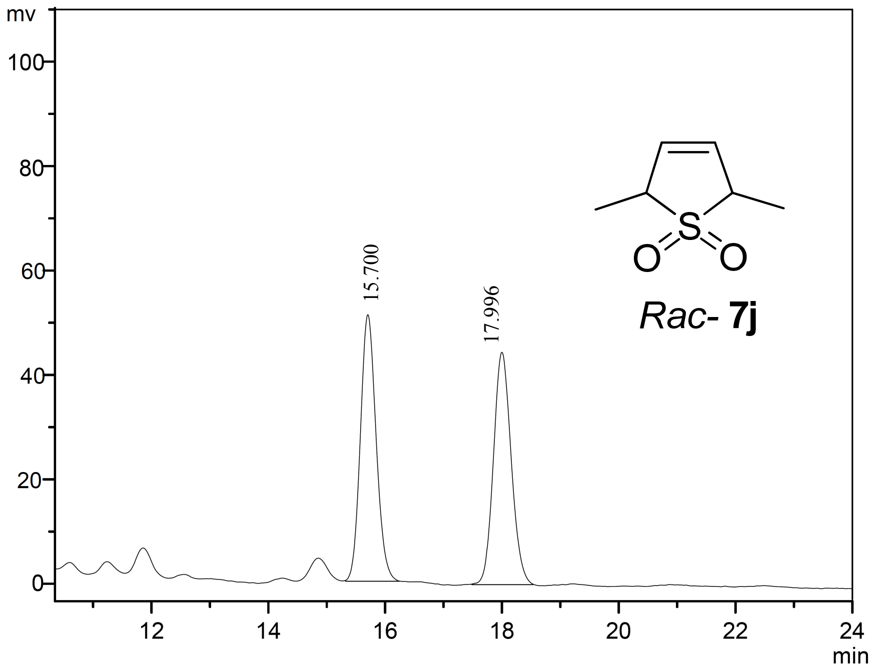
Run Mode: Analysis Peak Measurement: Area Calc. Type: % Normalize Results: No

Peak No.	Name	tR (min)	Result	Sep. Code	W 1/2 (sec)	Efficiency Plates	Plates/m	k'	R	Tailing (5.0%)
1		14.916	43.922	BB	20.35	10722	71481	13.92		1.10
2		21.456	28.242	BB	34.90	7545	50297	20.46	8.4	1.75
3		24.446	27.836	BB	35.40	9518	63456	23.45	3.0	1.14

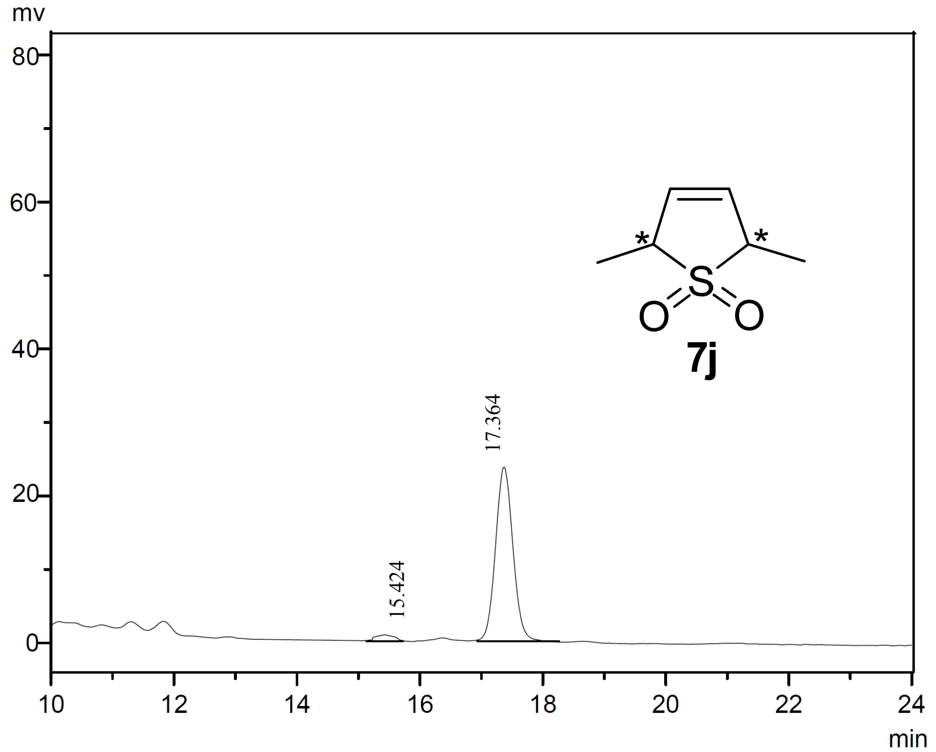


Run Mode: Analysis Peak Measurement: Area Calc. Type: % Normalize Results: No

Peak No.	Name	tR (min)	Result	Sep. Code	W 1/2 (sec)	Efficiency Plates	Plates/m	k'	R	Tailing (5.0%)
1		21.719	100.000	BB	37.60	6660	44398	20.72		1.67

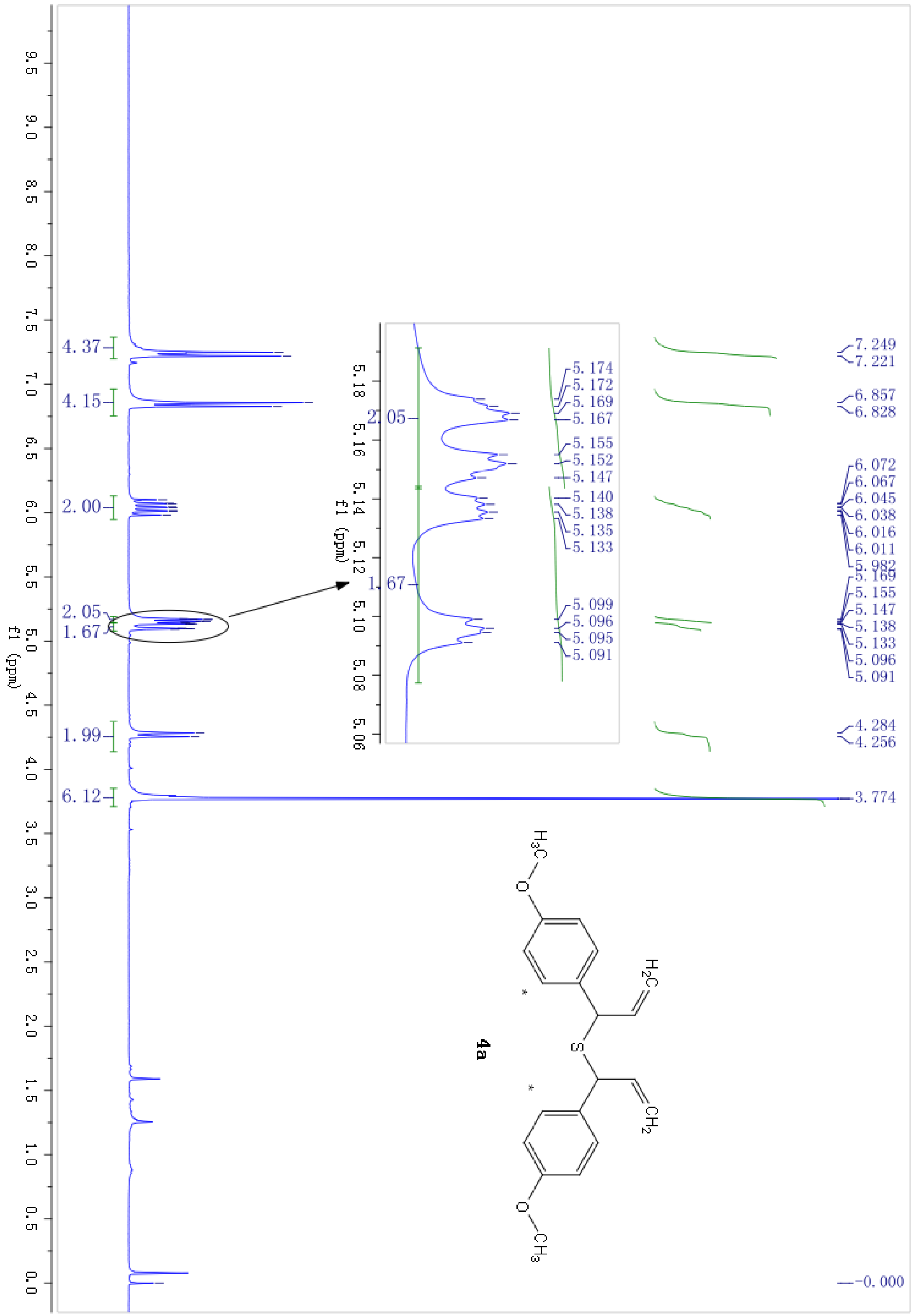


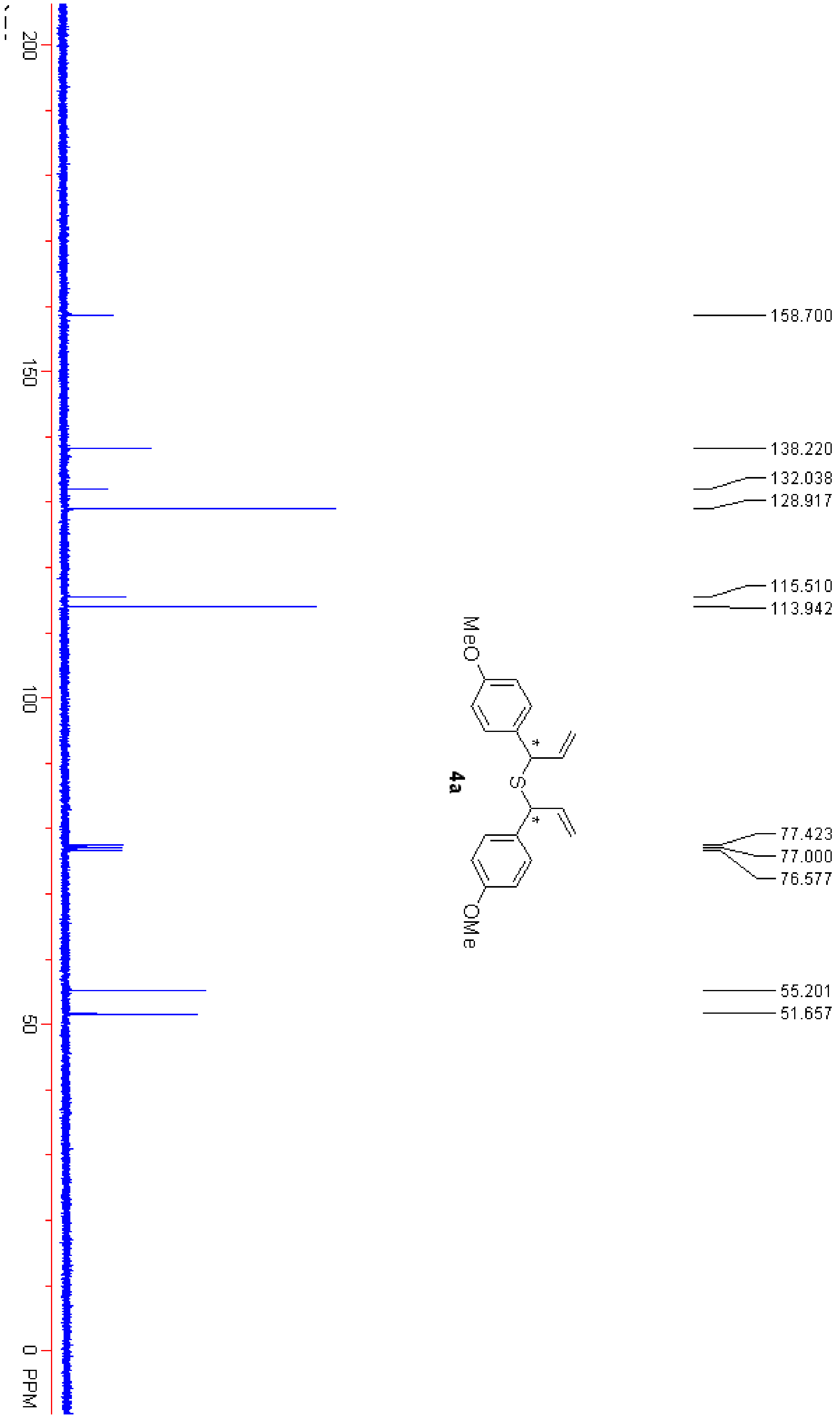
Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Area (%)
1	15.700	15.267	16.308	1437071	76599	18.7610	50.0857
2	17.996	17.258	18.650	1432152	67038	21.3630	49.9143



Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Area (%)
1	15.424	15.183	15.592	39404	2927	13.4610	3.2417
2	17.364	16.850	18.142	1176160	59300	19.8340	96.7583

NMR Spectra of the compounds 4, 5, 6 and 7.

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132.038
128.917
115.510
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77.423
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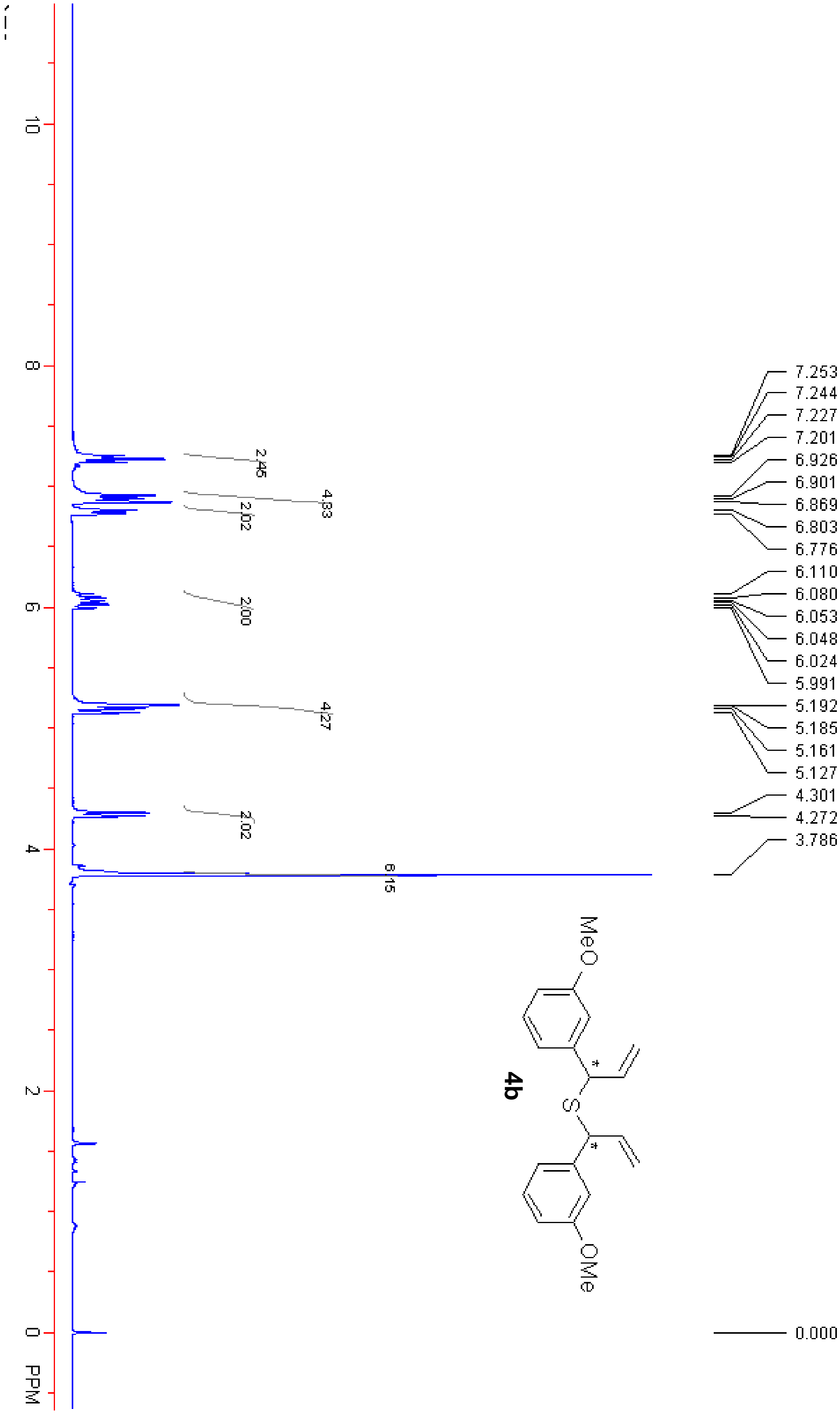
76.577
55.201
51.657
MeO
S
OMe
4a
200
150
100
50
0 PPM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7.201
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6.901
6.869
6.803
6.776
6.110
6.080
6.053
6.048
6.024
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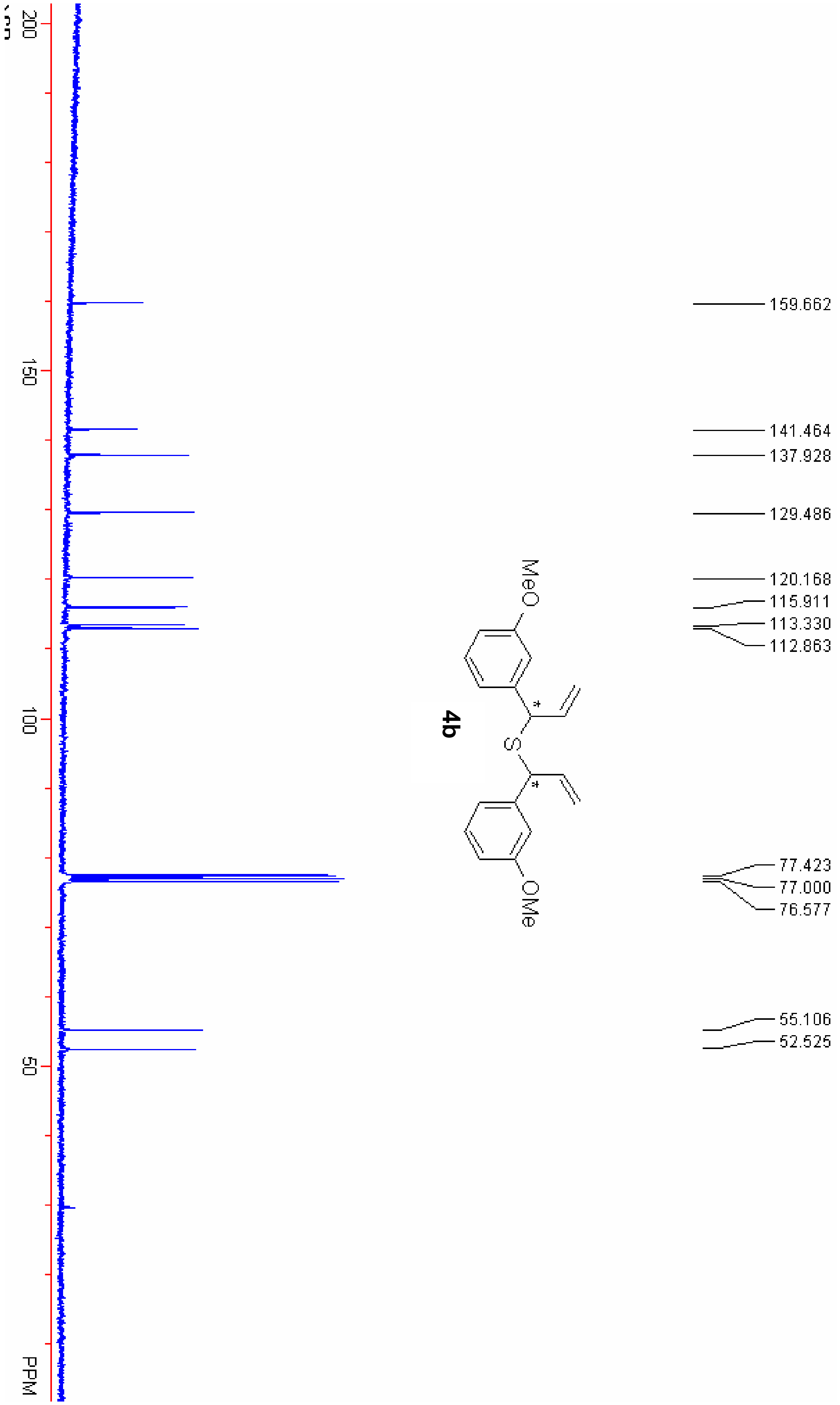
5.192
5.185
5.161
5.127
4.301
4.272
3.786
0.000
4b
PPM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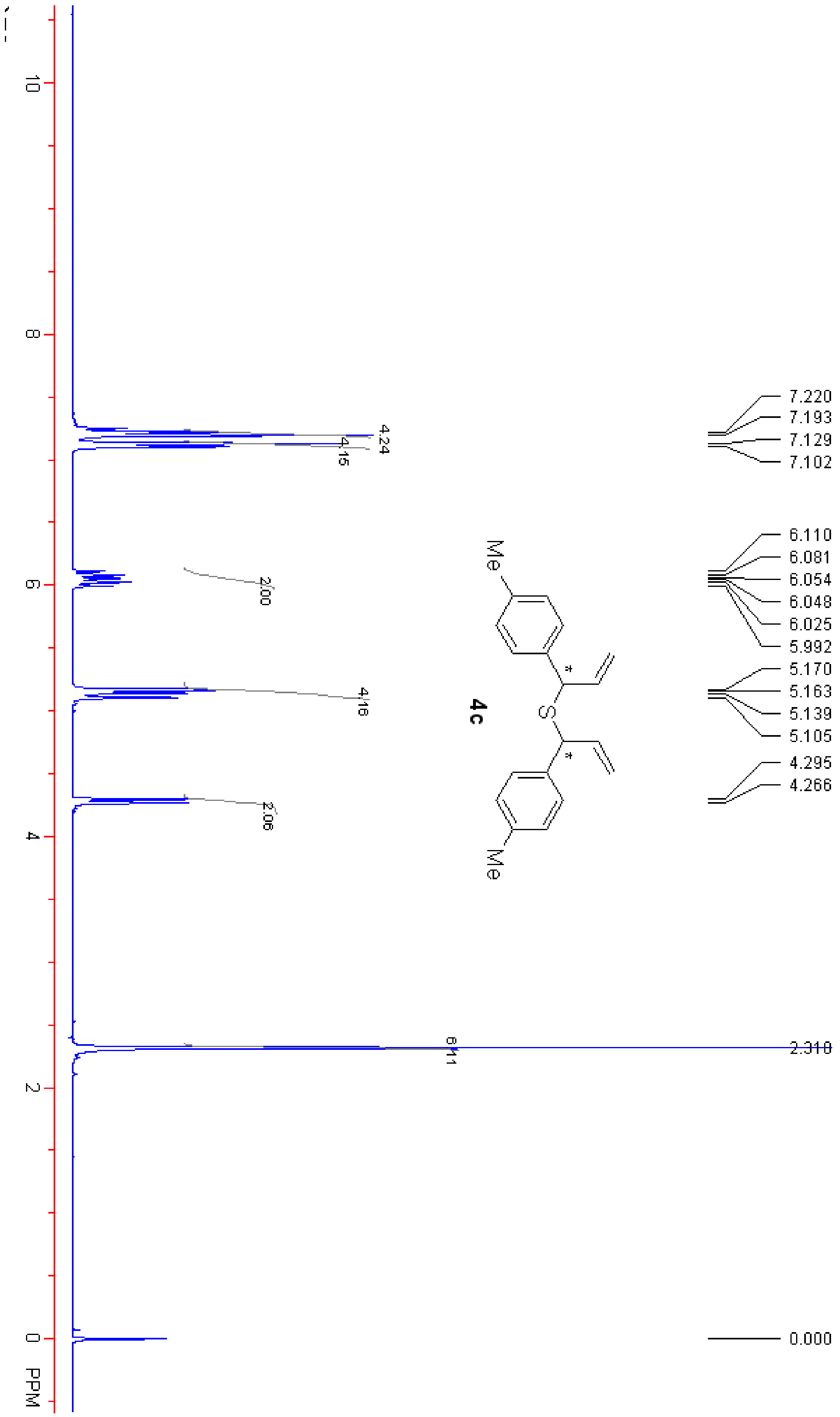
52.525
MeO
S
OMe
4b
200
150
100
50
PPM


4c
Me
S
Me
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5.992
5.170
5.163
5.139
5.105
4.295
4.266
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10
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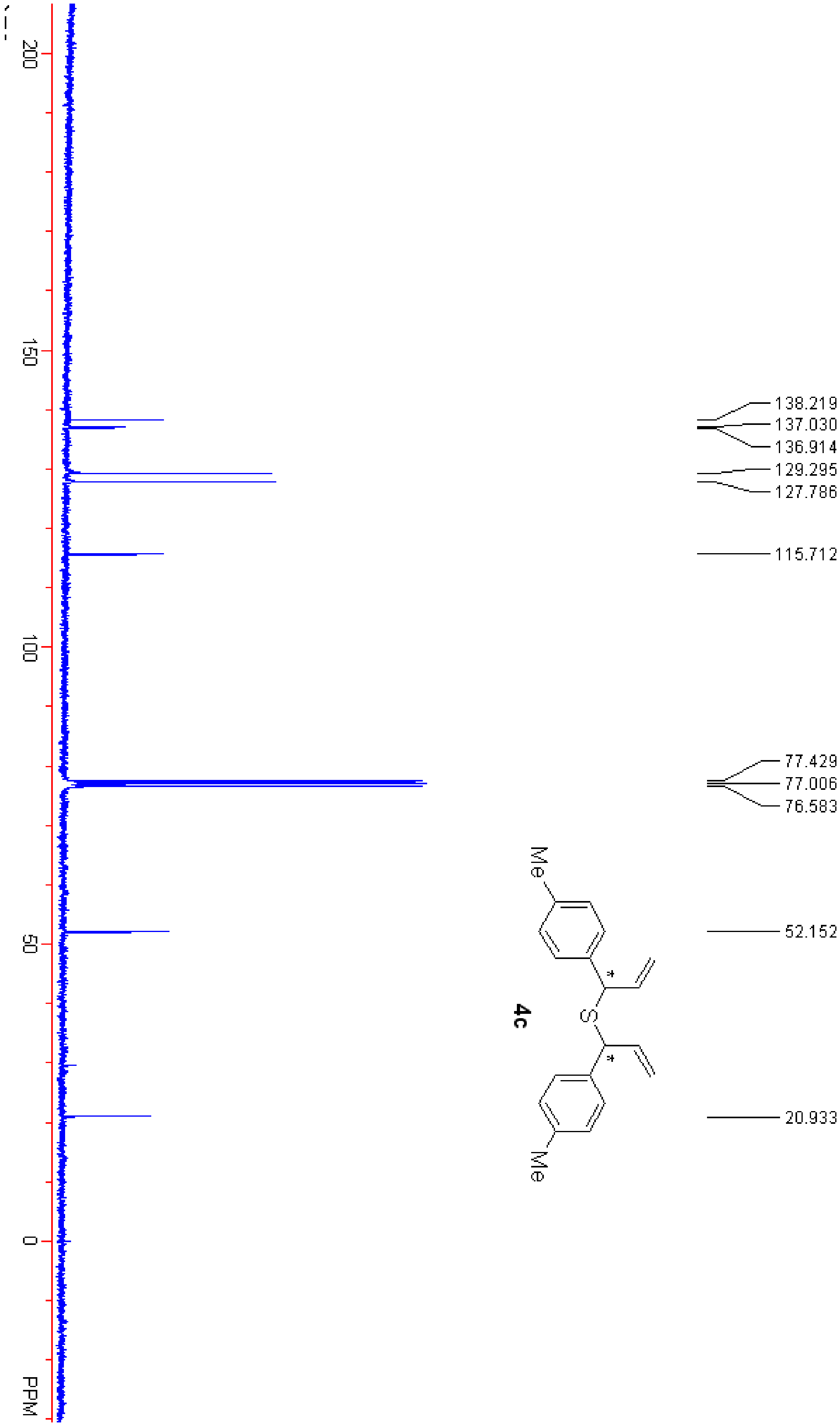
6
4
2
0
PPM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50
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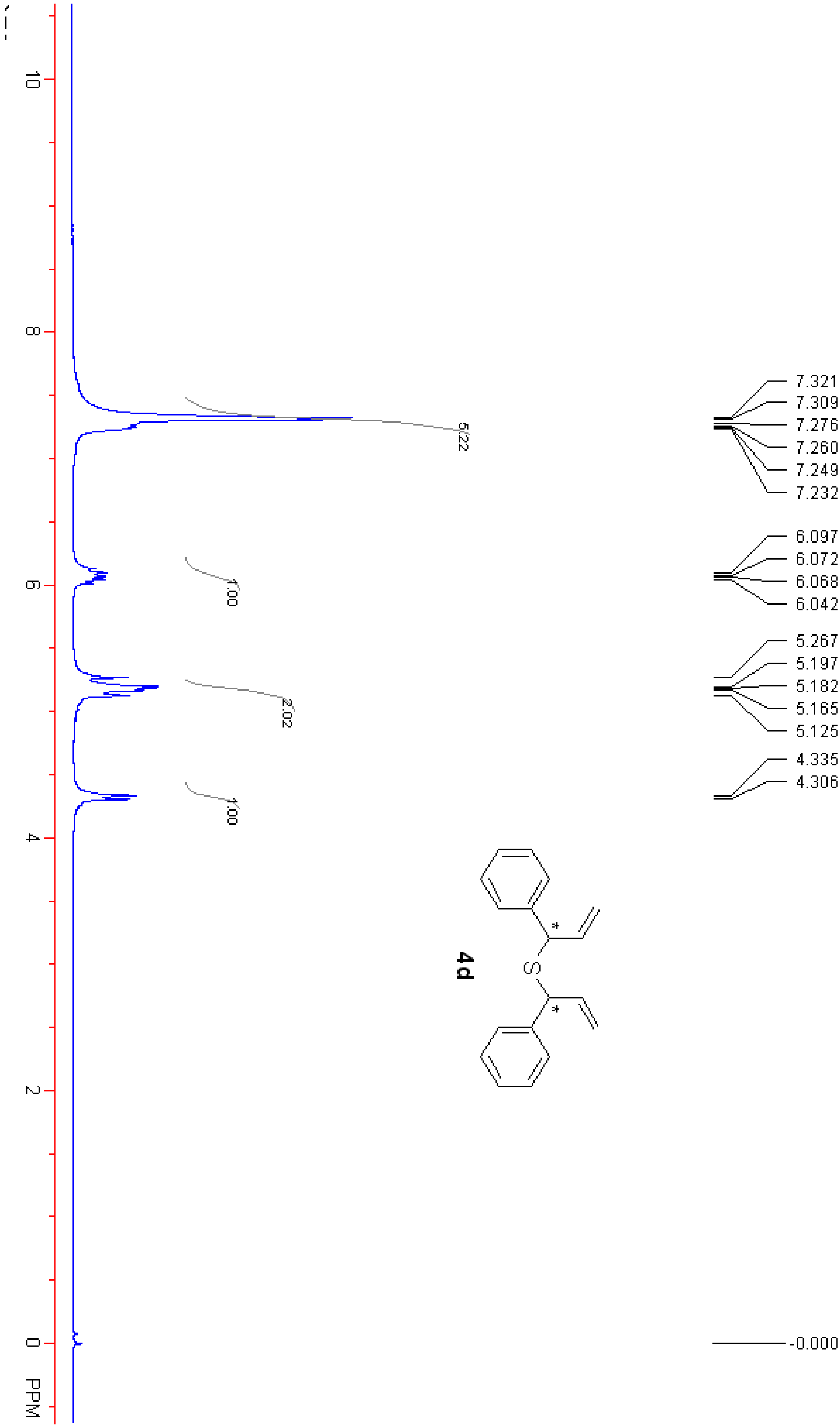
Me
S
Me

4c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6.068
6.042
5.267
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5.182
5.165
5.125
4.335
4.306
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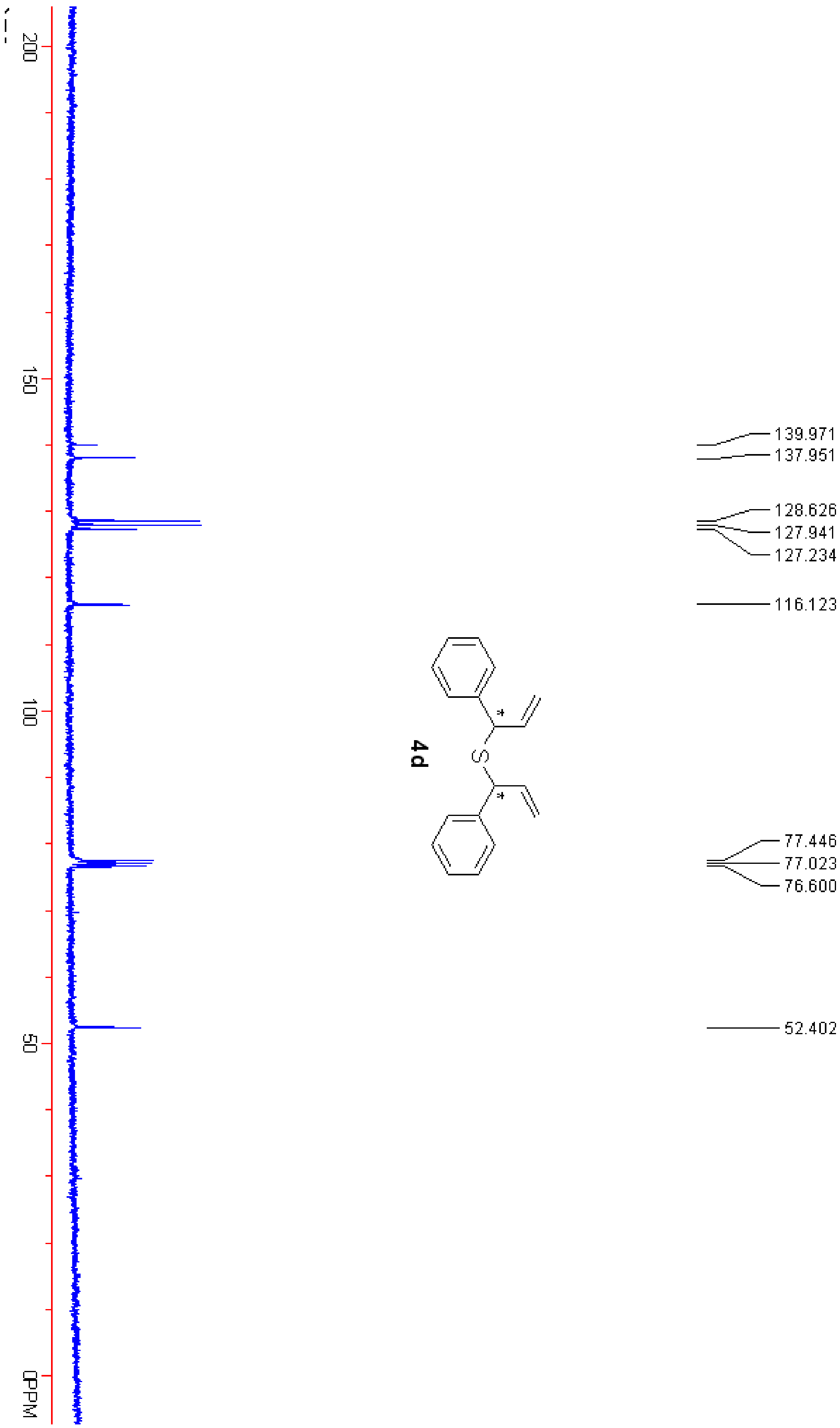
5.22
1.00
2.02
1.00
10
8
6
4
2
0
PPM
S
4d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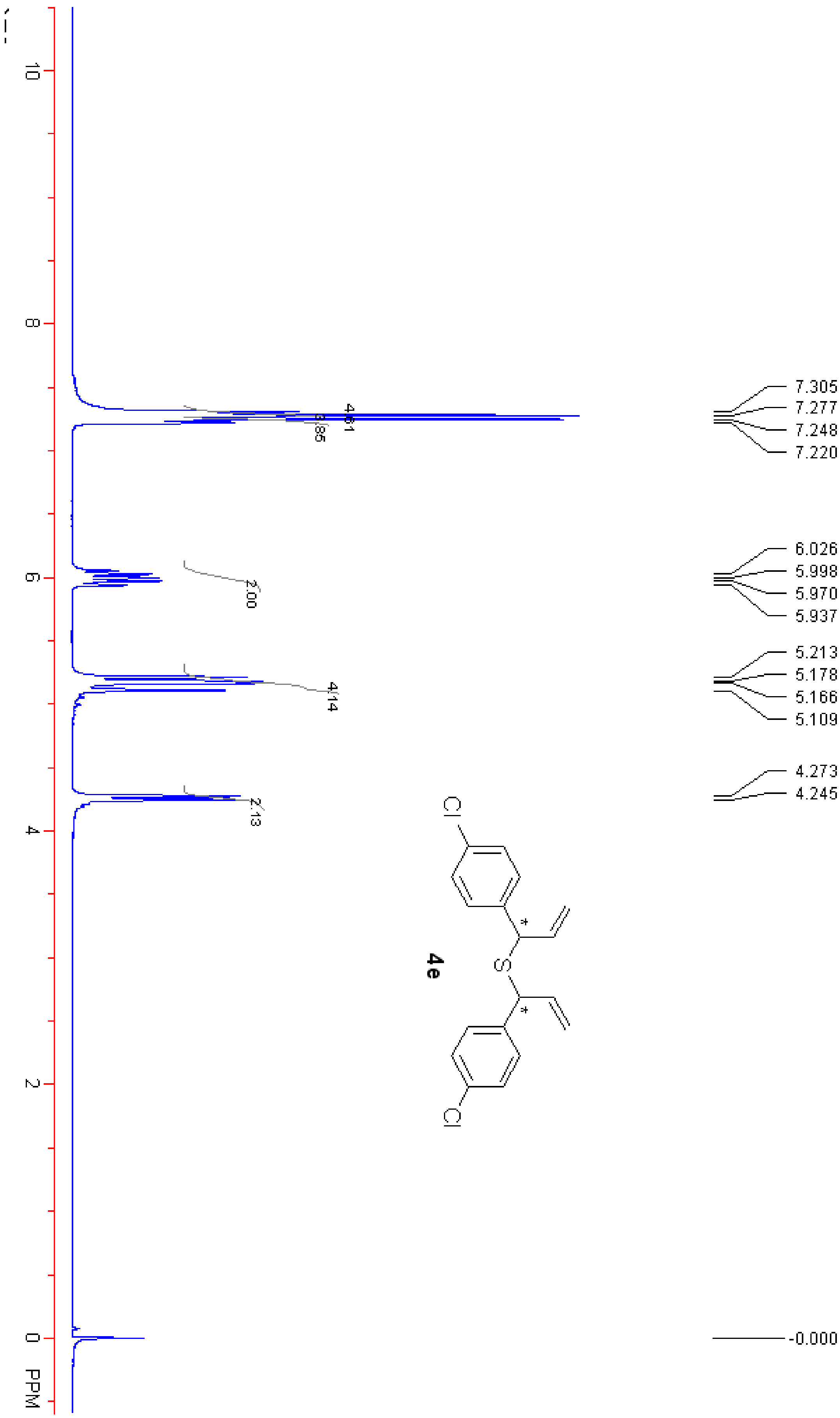
4d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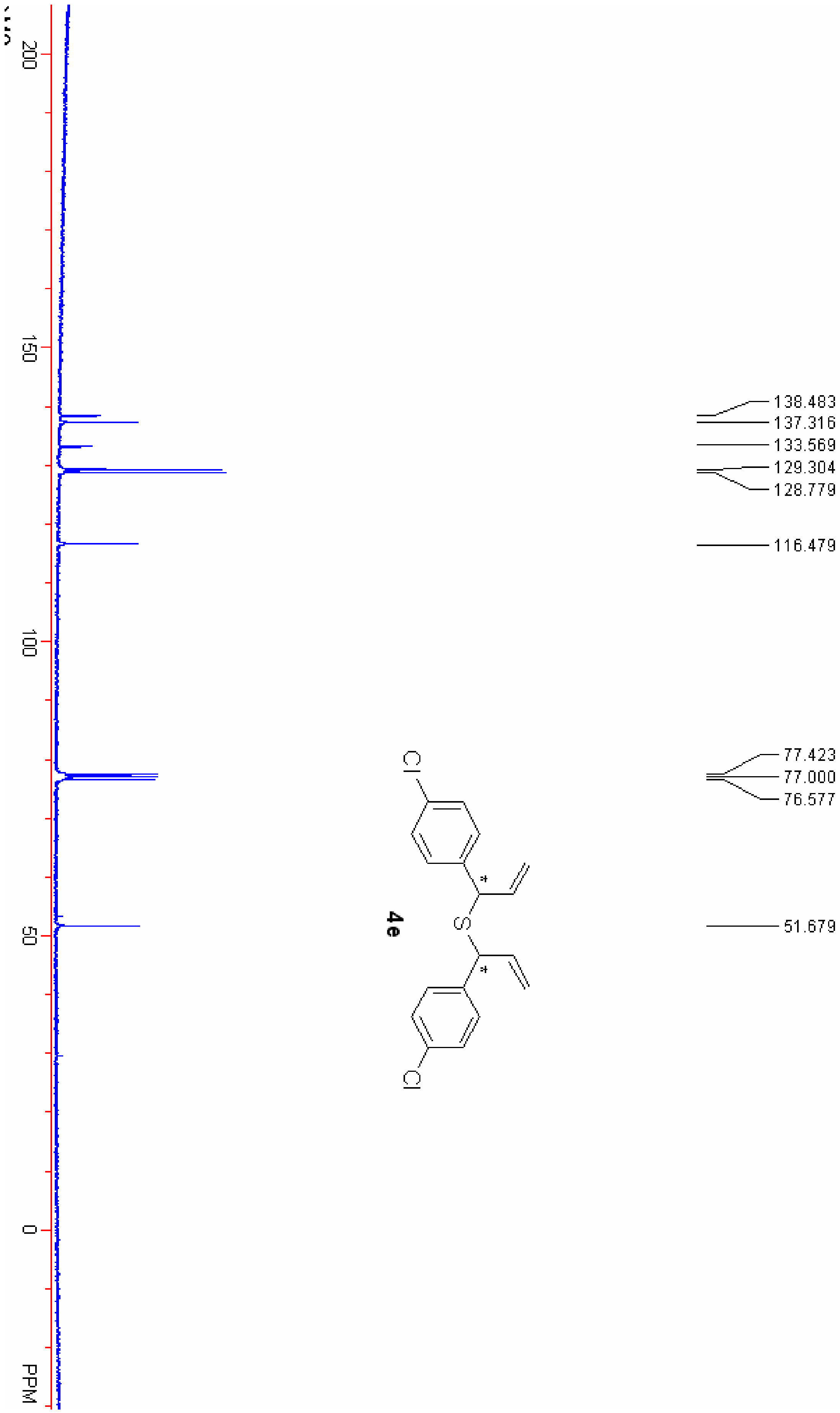
5.178
5.166
5.109
4.273
4.245
-0.000
4.61
3.85
2.00
4.14
2.13
10
8
6
4
2
0
PPM
Cl
S
Cl
4e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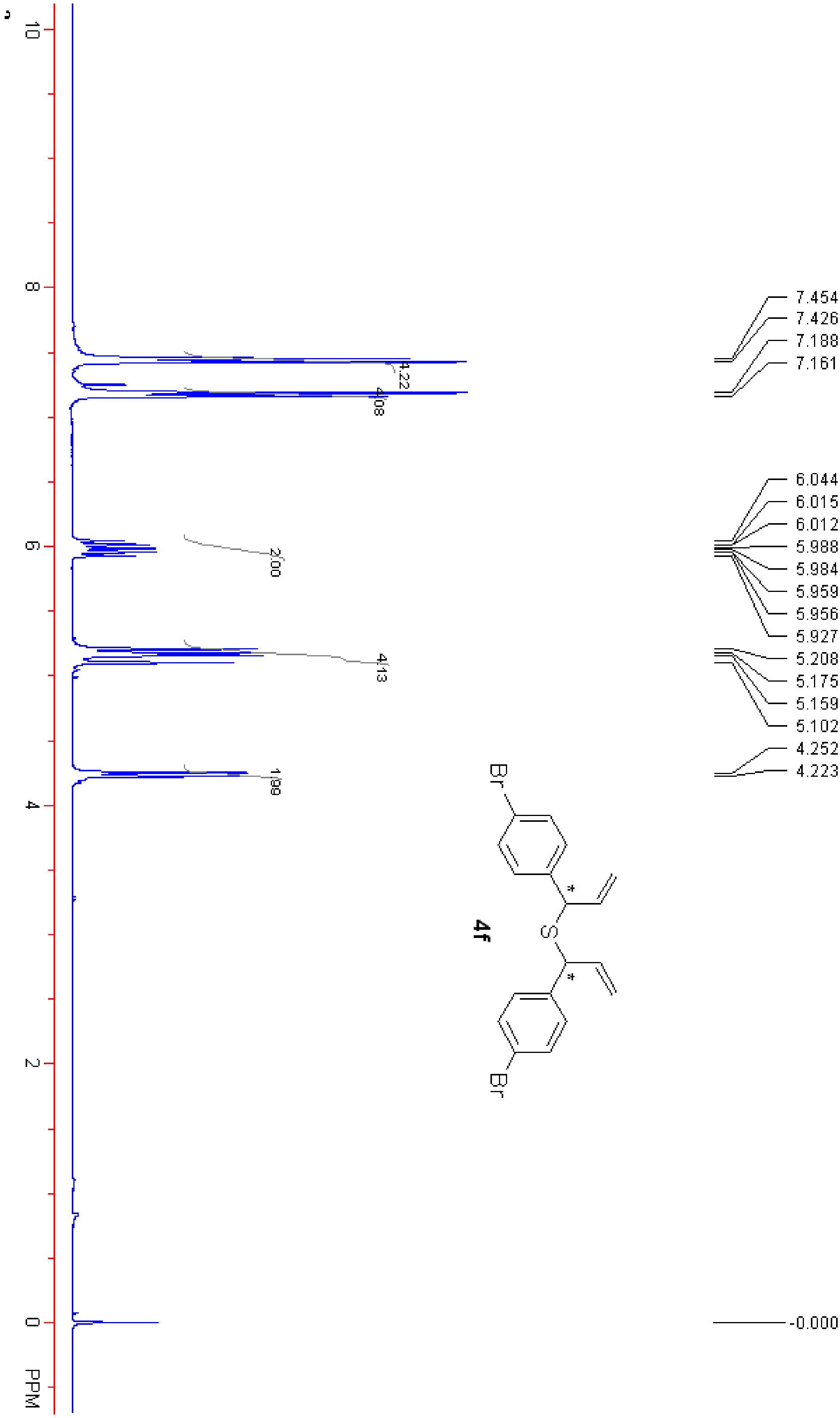
51.679
4e
PPM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5.959
5.956
5.927
5.208
5.175
5.159
5.102
4.252
4.223
-0.000
4.22
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4.13
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10
8
6
4
2
0
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Br
S
Br
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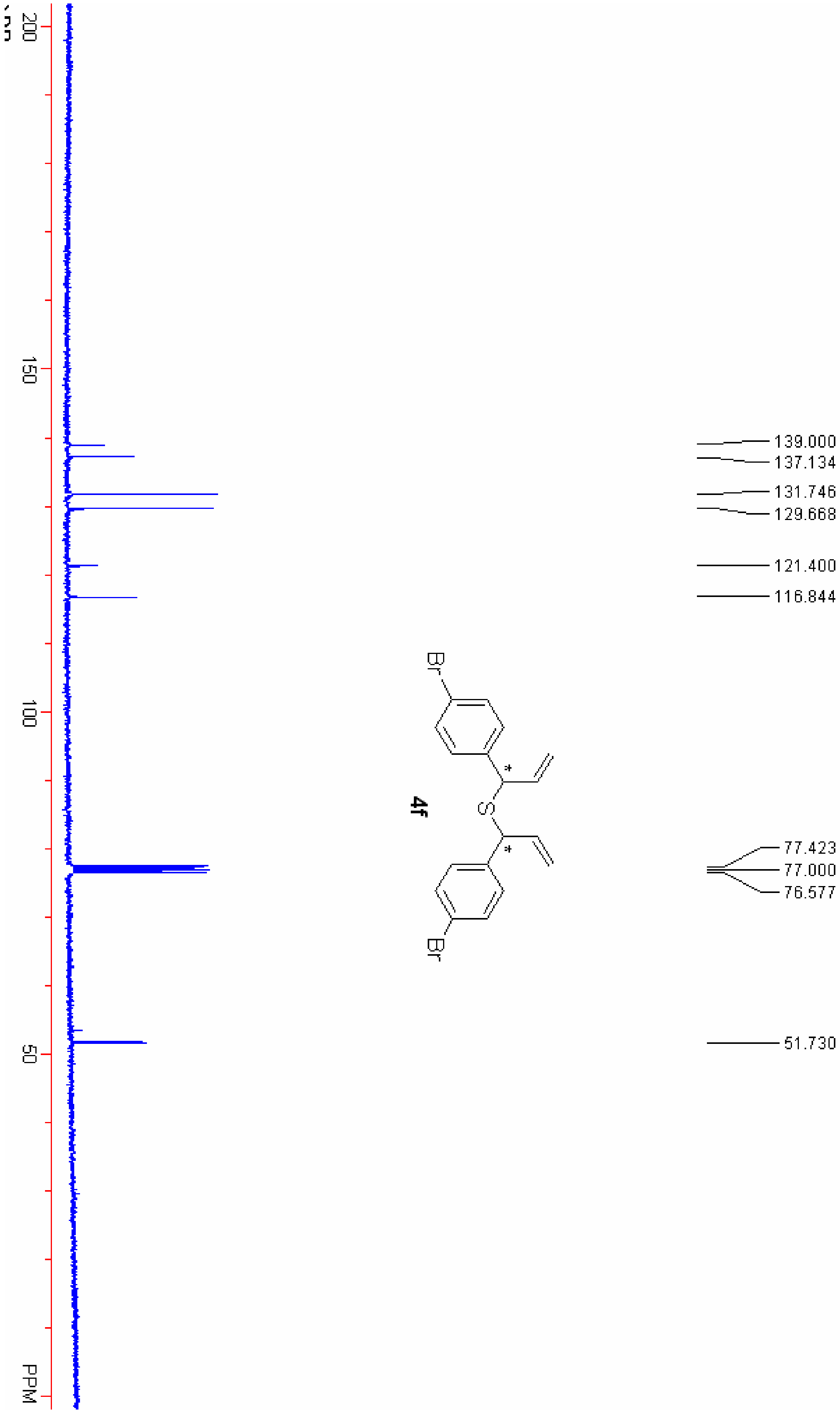
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100
50
PPM
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S
Br
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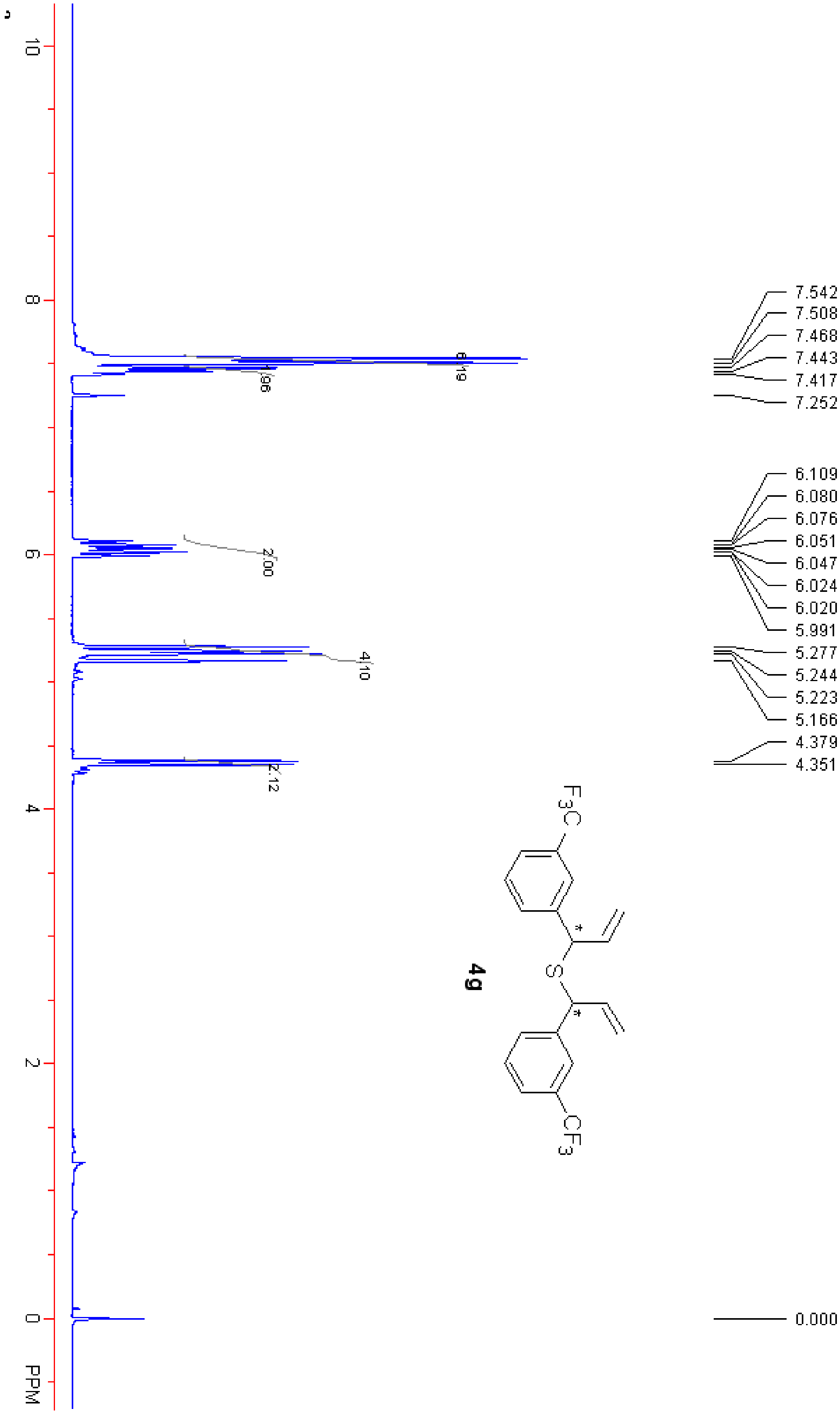
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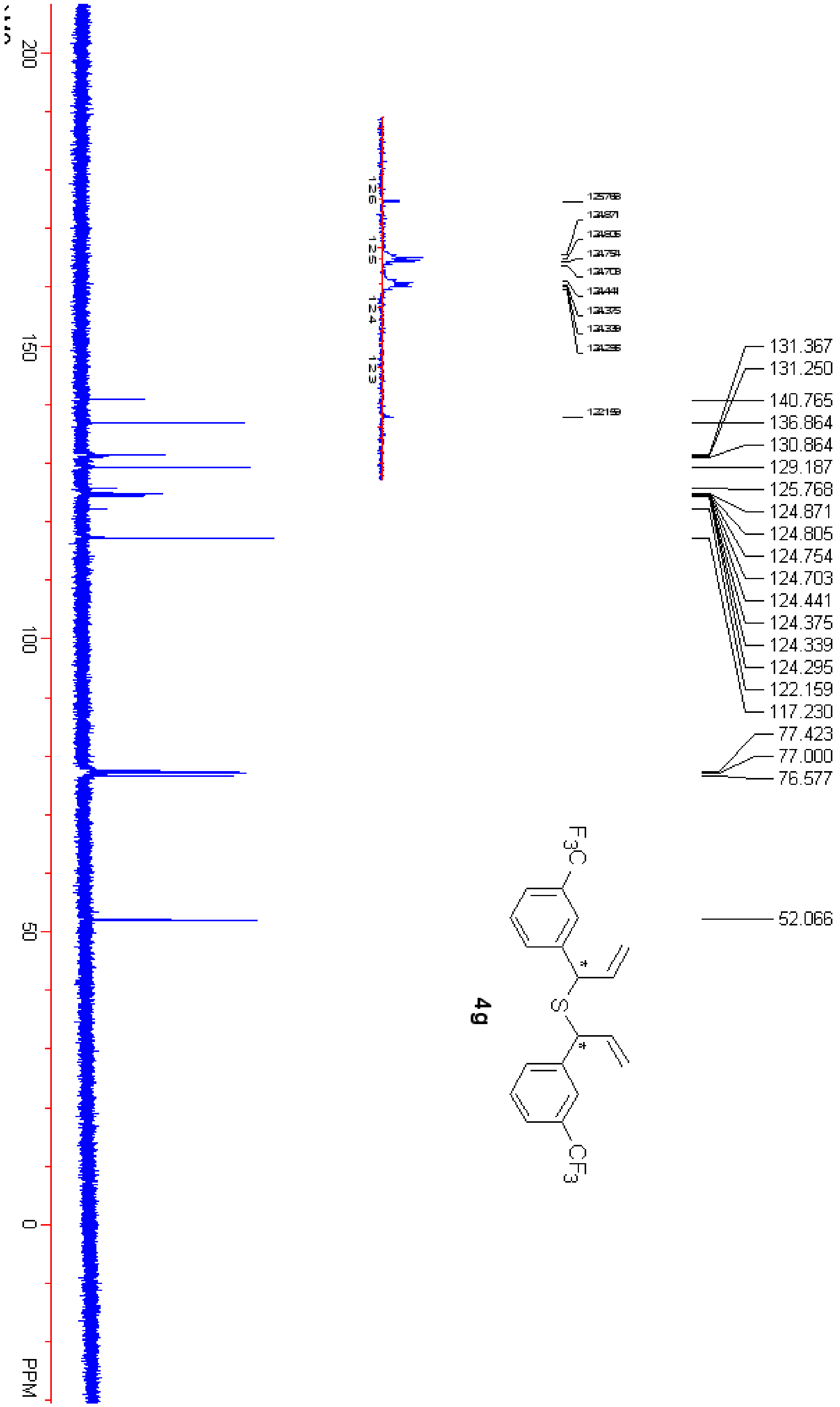
PPM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124.375
124.339
124.295
122.159
117.230
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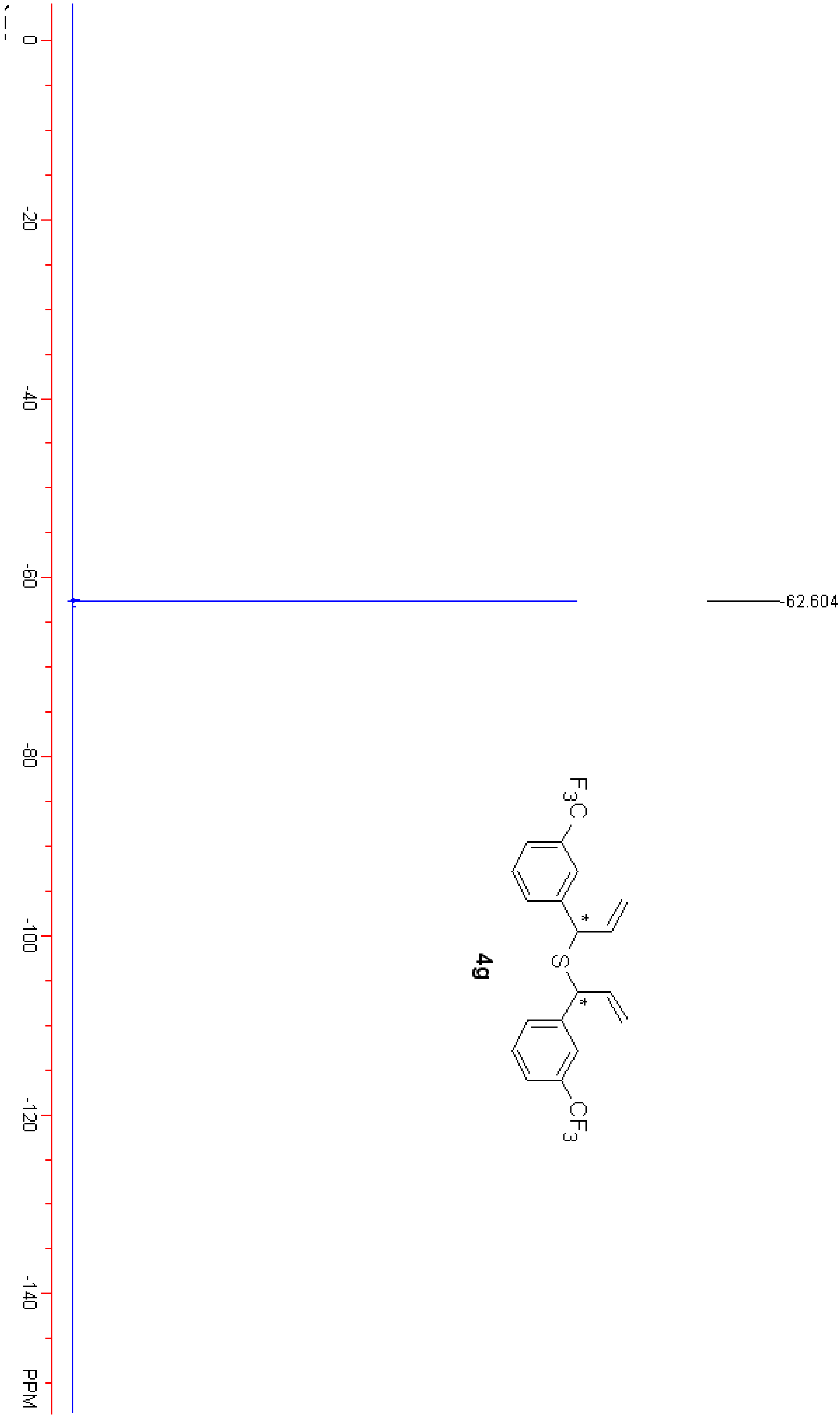
77.000
76.577
52.066
200
150
100
50
0
PPM
F3C
S
CF3
4g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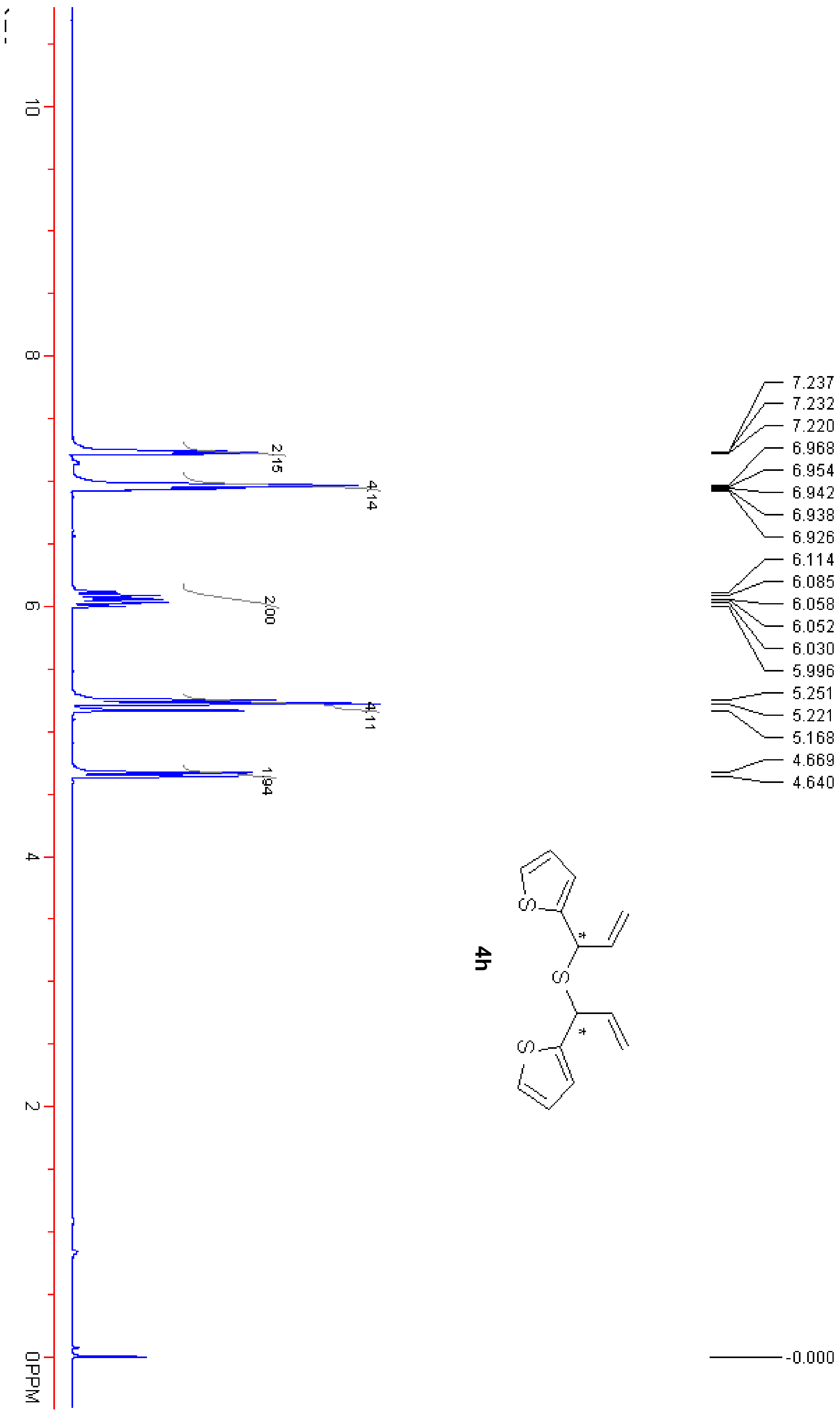
PPM
F3C
S
CF3
4g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6.926
6.114
6.085
6.058
6.052
6.030
5.996
5.251
5.221
5.168
4.669
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10
8
6
4
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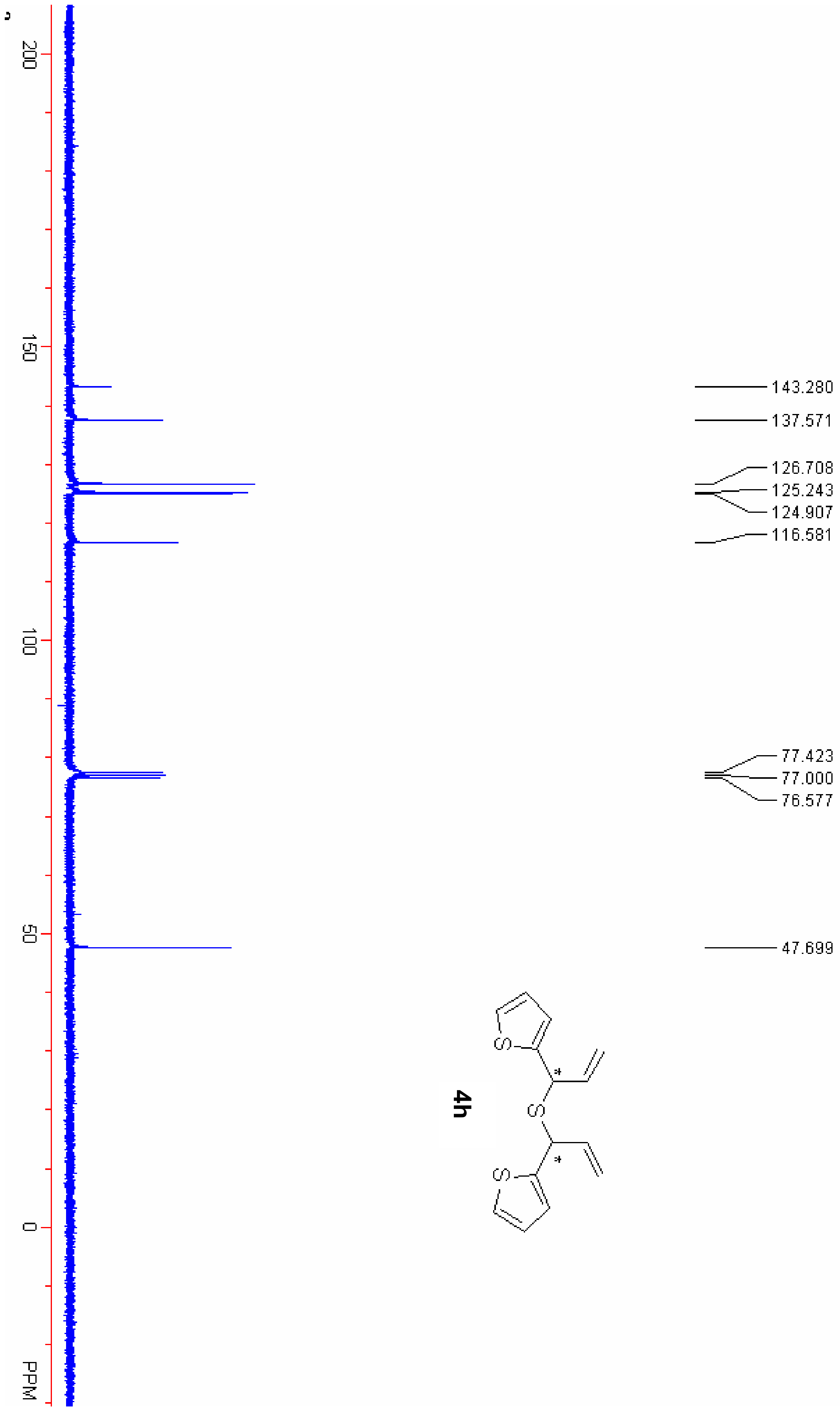
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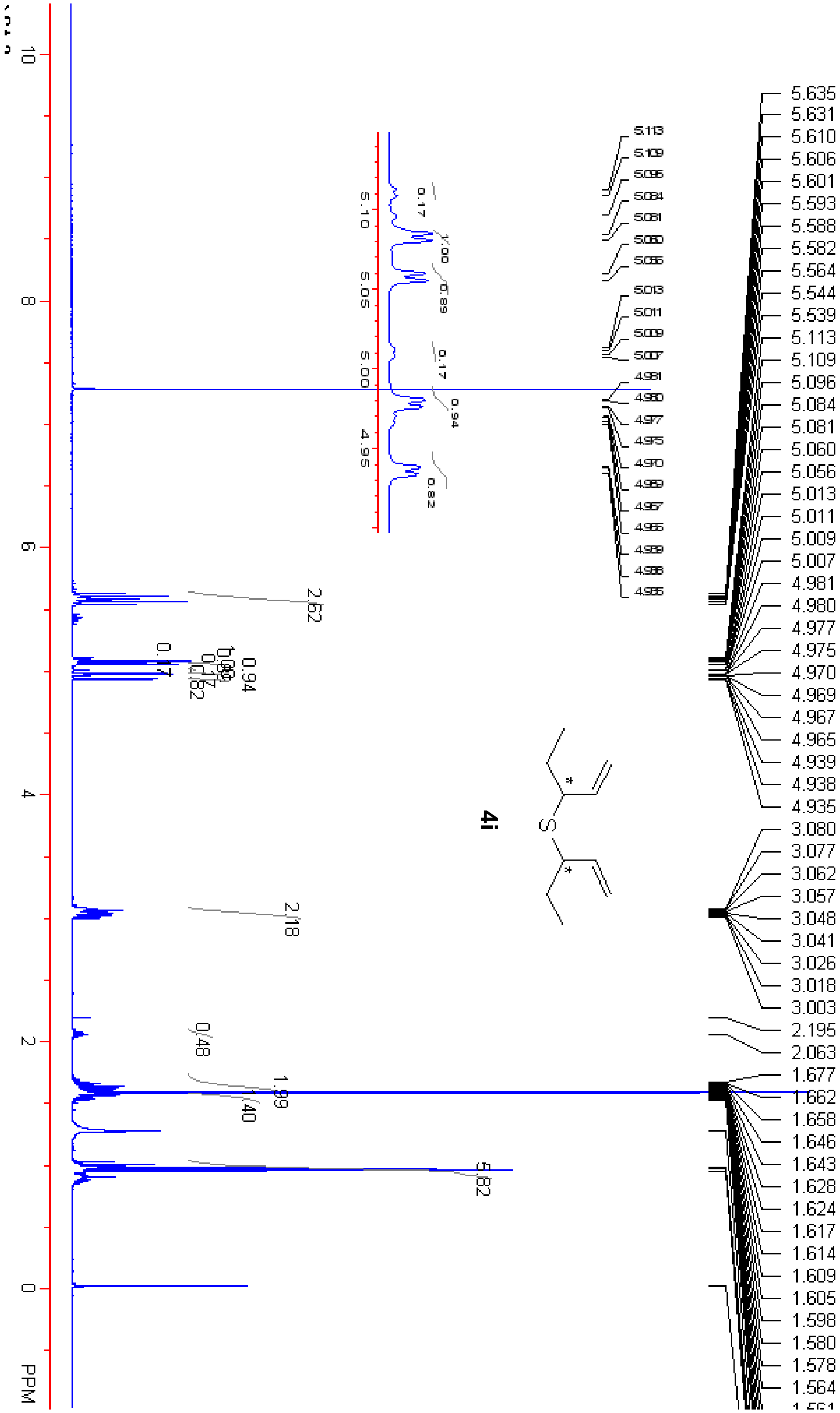
47.699
200
150
100
50
0
PPM
4h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4.95
0.17
1.00
0.89
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0.94
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5.631
5.610
5.606
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5.588
5.582
5.564
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4.965
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3.018
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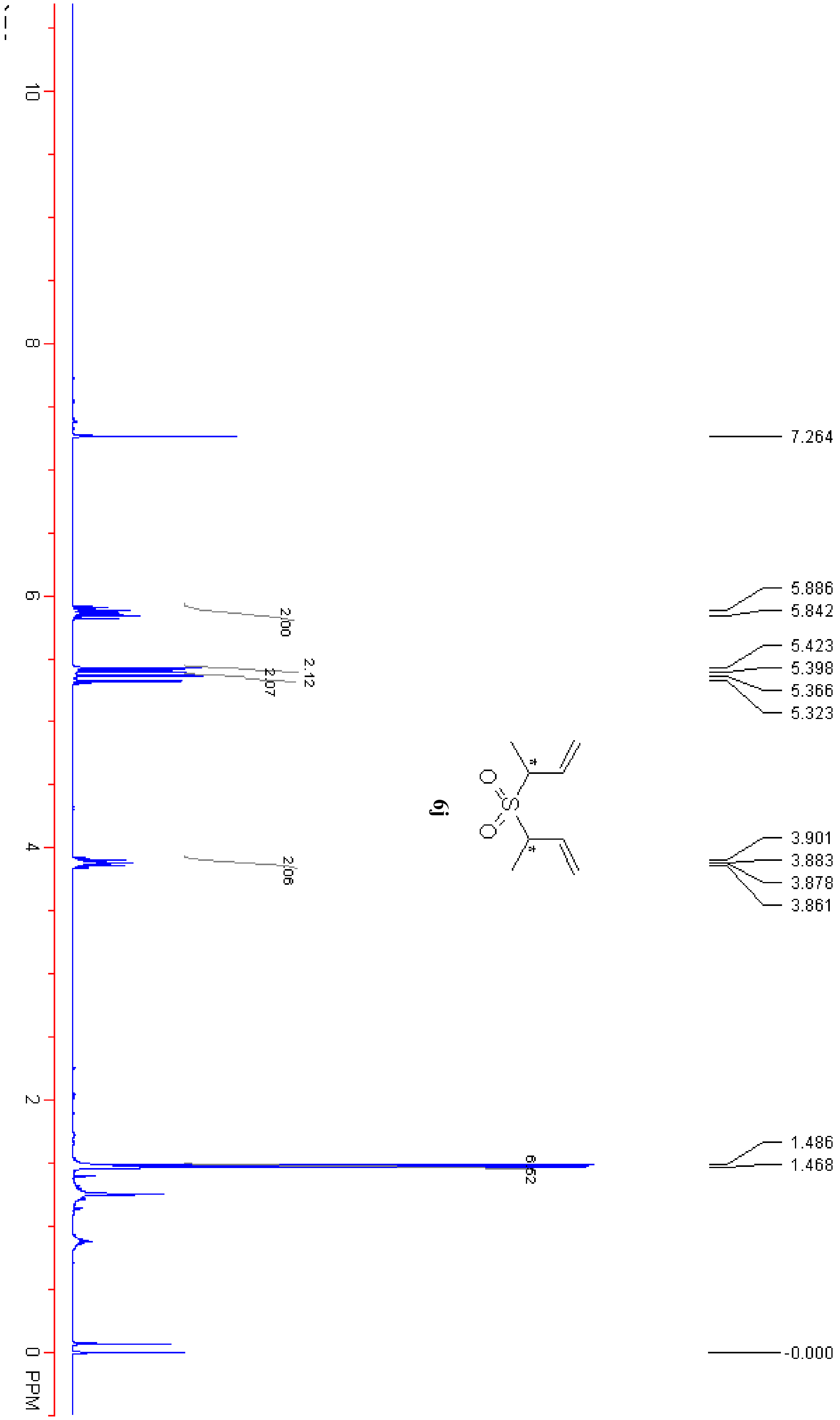
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2.12
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6j
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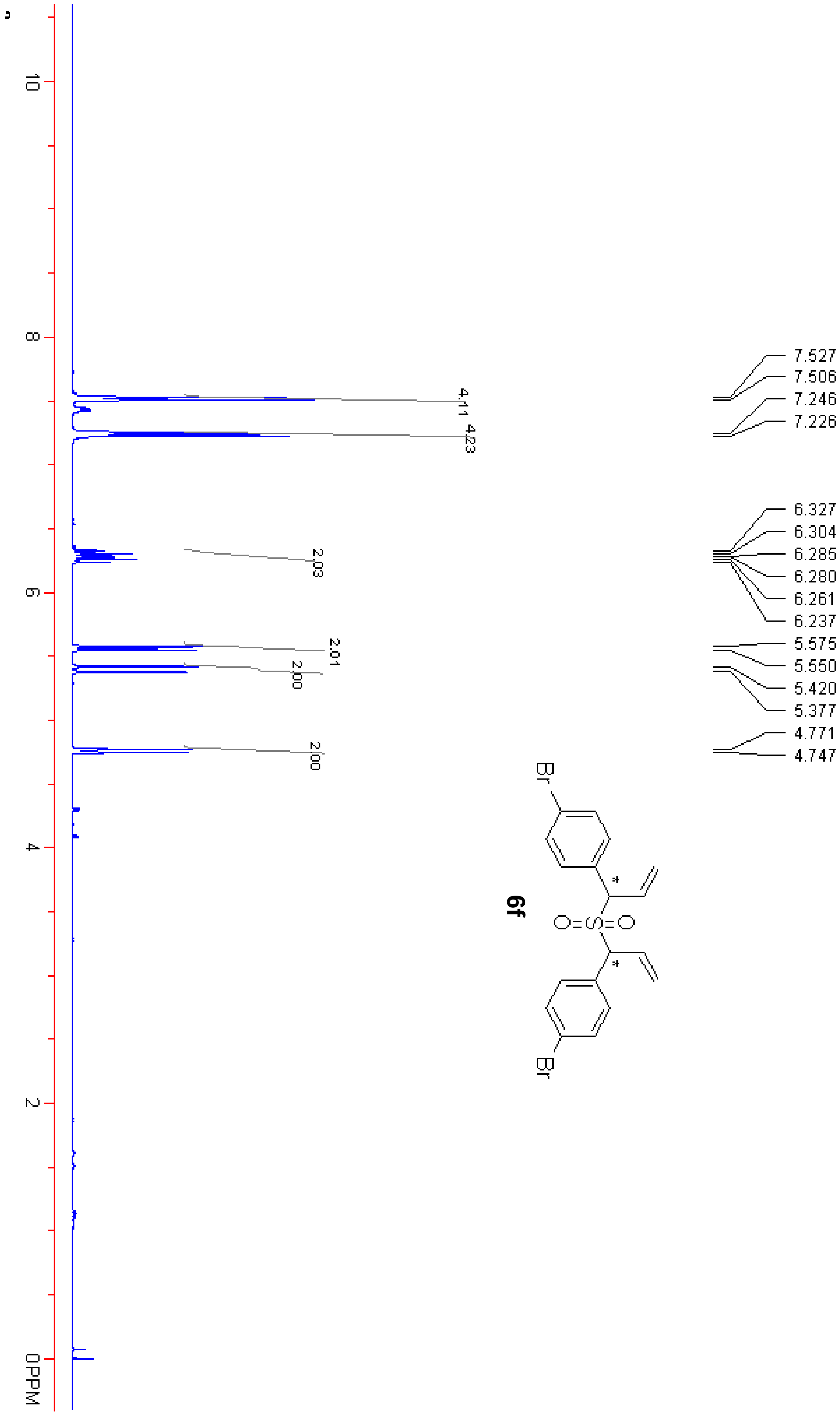
0 PPM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5.420
5.377
4.771
4.747
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6
4
2
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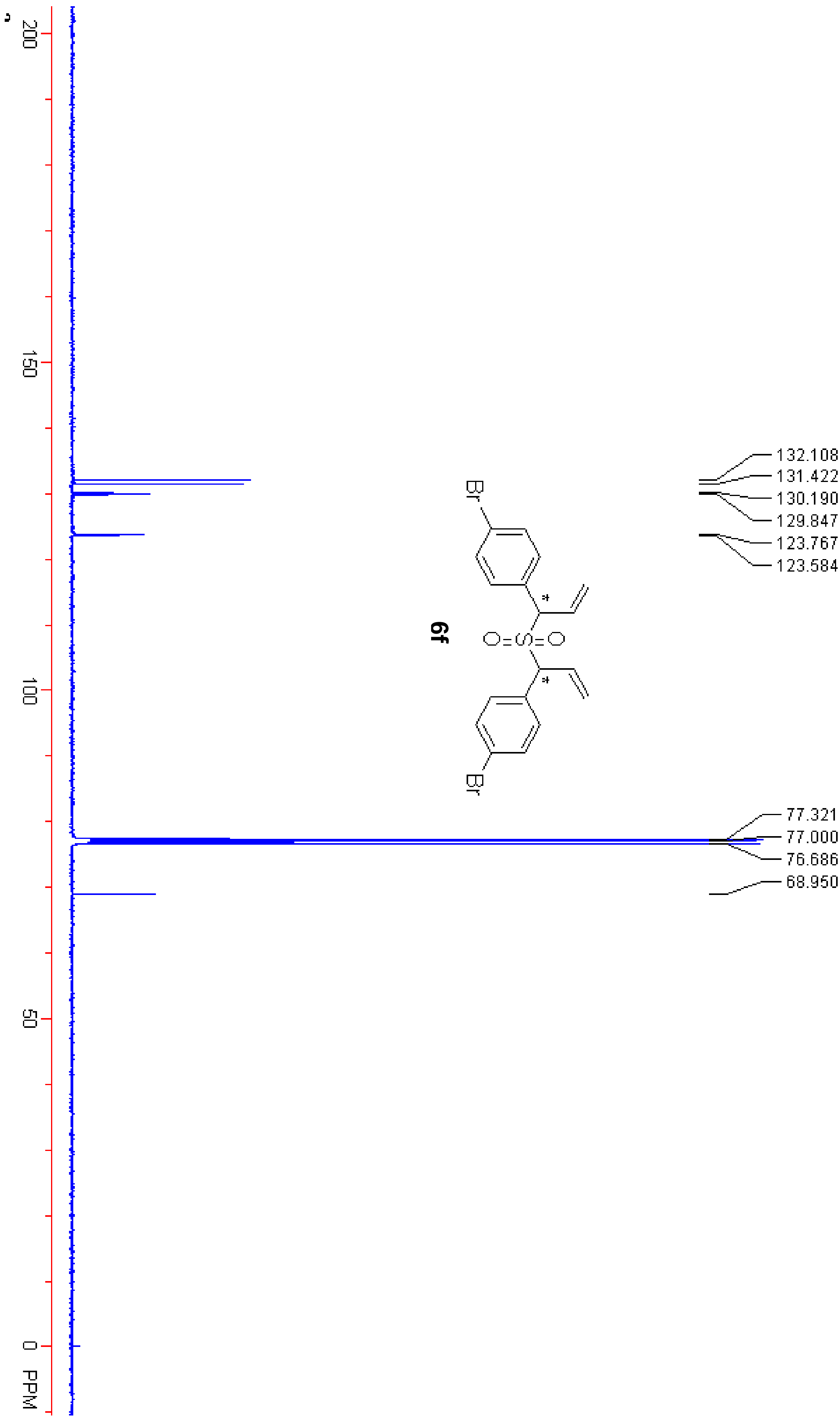
Br
Br
6f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Br
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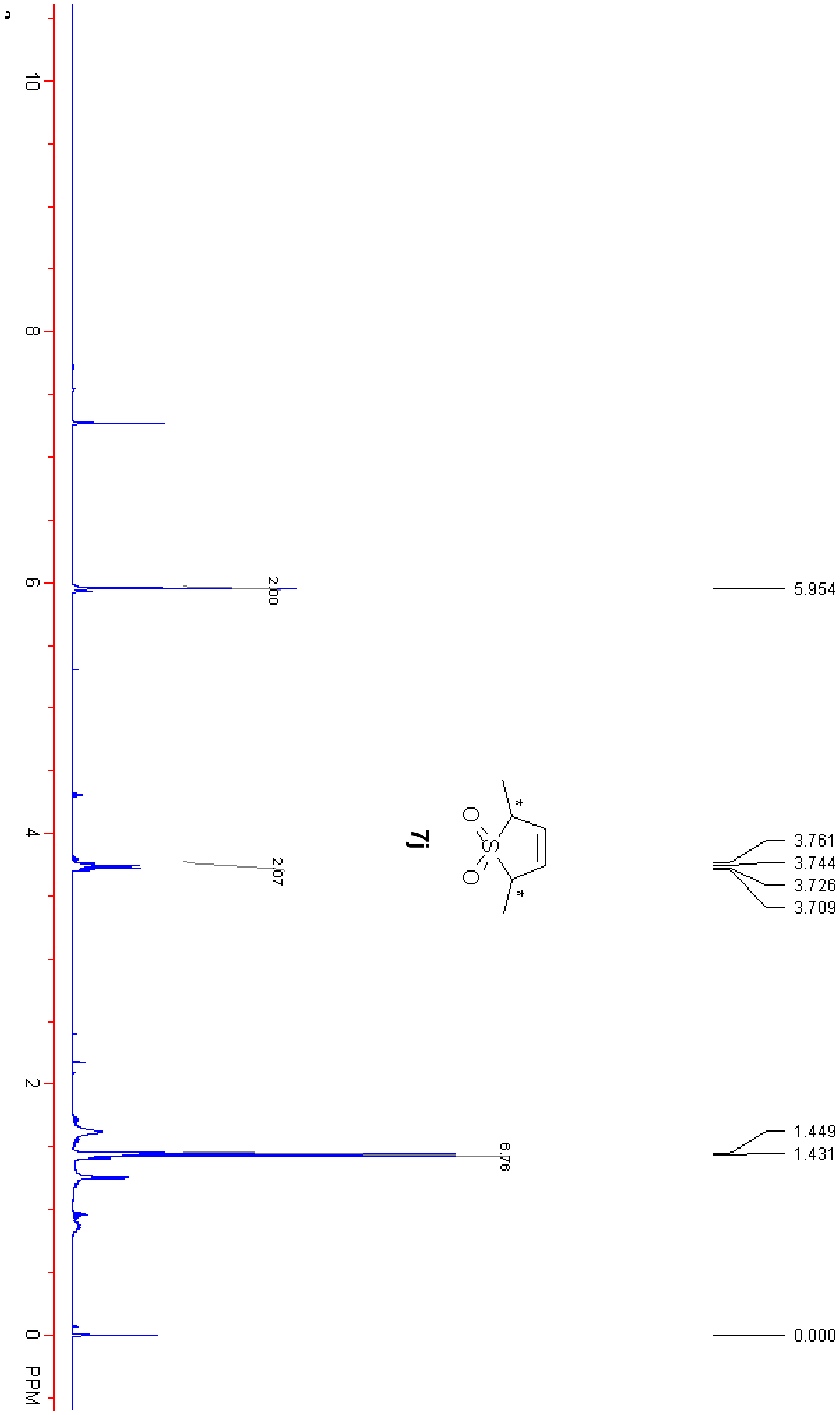
150
100
50
0
PPM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10
8
6
4
2
0
PPM

X-ray Crystallography of (*R*,*R*)-6f

Single Crystal X-Ray Analysis. A representative crystal was surveyed on a Bruker APEX diffractometer. All crystallographic calculations were facilitated by the SHELXL-97 system.

_computing_data_collection	'Bruker SMART'
_computing_cell_refinement	'Bruker SMART'
_computing_data_reduction	'Bruker SAINT'
_computing_structure_solution	'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement	'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics	XP
_computing_publication_material	XCIF

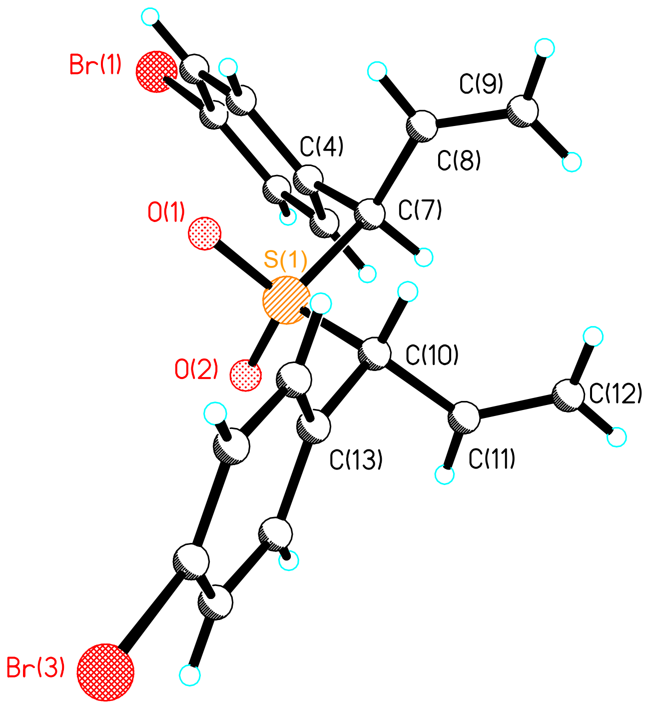

Table 1. Crystal data and structure refinement for xl.

Identification code	xl
Empirical formula	C18 H16 Br2 O2 S
Formula weight	456.19
Temperature	293(2) K

Wavelength	0.71073 A
Crystal system, space group	Triclinic, P1
Unit cell dimensions	a = 6.941(6) A alpha = 90.096(10) deg. b = 7.130(6) A beta = 90.099(12) deg. c = 18.450(15) A gamma = 90.035(12) deg.
Volume	913.0(13) A^3
Z, Calculated density	2, 1.659 Mg/m^3
Absorption coefficient	4.560 mm^-1
F(000)	452
Crystal size	0.25 x 0.08 x 0.04 mm
Theta range for data collection	2.21 to 27.53 deg.
Limiting indices	-9<=h<=7, -9<=k<=8, -23<=l<=22
Reflections collected / unique	4462 / 4462 [R(int) = 0.0000]
Completeness to theta = 27.53	90.3 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4462 / 11 / 367
Goodness-of-fit on F^2	1.060
Final R indices [I>2sigma(I)]	R1 = 0.0775, wR2 = 0.2218
R indices (all data)	R1 = 0.0942, wR2 = 0.2412
Absolute structure parameter	0.49(3)
Largest diff. peak and hole	0.800 and -1.459 e.A^-3

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

	x	y	z	U(eq)
Br(1)	6980(2)	21779(2)	2571(1)	72(1)
Br(2)	1982(2)	23244(2)	-9538(1)	72(1)
Br(3)	6627(2)	23245(2)	-2432(1)	72(1)
Br(4)	11627(2)	21776(2)	-4539(1)	72(1)
O(1)	5830(30)	24920(18)	-6238(7)	114(7)
O(2)	2570(30)	24921(19)	-5773(7)	107(6)
O(3)	7590(30)	20055(19)	-1216(7)	118(7)
O(4)	10910(30)	20069(17)	-775(7)	102(5)
S(1)	4260(9)	25942(5)	-5996(2)	72(1)
S(2)	9239(9)	19022(6)	-997(2)	74(1)
C(1)	5981(14)	24439(12)	-3309(4)	53(3)
C(2)	4265(12)	25414(14)	-3402(4)	54(3)
C(3)	3907(11)	26355(14)	-4048(4)	61(4)
C(4)	5265(13)	26320(13)	-4601(4)	49(3)
C(5)	6981(12)	25344(14)	-4508(4)	61(4)
C(6)	7339(12)	24404(13)	-3862(5)	65(4)
C(7)	4911(18)	27505(17)	-5270(6)	47(3)
C(8)	6620(20)	28684(17)	-5476(9)	63(4)
C(9)	6490(30)	30516(19)	-5468(10)	76(5)
C(10)	3558(19)	27496(16)	-6726(6)	48(3)
C(11)	1860(20)	28666(16)	-6494(8)	58(4)
C(12)	1960(30)	30531(18)	-6502(9)	73(5)
C(13)	3165(14)	26388(12)	-7390(4)	53(3)
C(14)	4543(11)	26393(12)	-7936(5)	57(3)
C(15)	4198(12)	25436(13)	-8580(4)	52(3)
C(16)	2475(14)	24474(12)	-8679(4)	53(3)
C(17)	1097(11)	24469(13)	-8133(5)	67(4)
C(18)	1442(12)	25426(13)	-7489(4)	58(4)
C(19)	11011(15)	20503(12)	-3691(4)	54(3)
C(20)	9288(13)	19543(14)	-3592(4)	63(4)
C(21)	8936(11)	18614(14)	-2944(5)	60(4)

C(22)	10307(14)	18645(14)	-2394(4)	52(3)
C(23)	12030(13)	19605(16)	-2492(5)	76(6)
C(24)	12383(12)	20534(15)	-3141(5)	67(4)
C(25)	9930(20)	17434(18)	-1713(7)	52(3)
C(26)	11630(20)	16260(20)	-1480(9)	80(6)
C(27)	11560(30)	14410(20)	-1457(10)	73(5)
C(28)	8530(20)	17469(16)	-272(7)	49(3)
C(29)	6840(20)	16230(20)	-475(9)	74(6)
C(30)	6890(30)	14370(20)	-479(10)	68(4)
C(31)	8149(14)	18592(12)	404(4)	51(3)
C(32)	6425(12)	19545(14)	508(4)	70(5)
C(33)	6088(11)	20486(14)	1156(5)	65(4)
C(34)	7475(14)	20474(13)	1700(4)	53(3)
C(35)	9199(12)	19520(14)	1596(4)	67(4)
C(36)	9536(11)	18580(13)	948(5)	60(4)

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

Br(1)-C(34)	1.888(7)
Br(2)-C(16)	1.842(6)
Br(3)-C(1)	1.884(6)
Br(4)-C(19)	1.861(6)
O(1)-S(1)	1.384(18)
O(2)-S(1)	1.443(16)
O(3)-S(2)	1.422(17)
O(4)-S(2)	1.439(17)
S(1)-C(7)	1.797(13)
S(1)-C(10)	1.812(11)
S(2)-C(25)	1.803(13)
S(2)-C(28)	1.807(12)
C(1)-C(2)	1.3900
C(1)-C(6)	1.3900
C(2)-C(3)	1.3900
C(3)-C(4)	1.3900
C(4)-C(5)	1.3900
C(4)-C(7)	1.518(13)
C(5)-C(6)	1.3900
C(7)-C(8)	1.501(9)
C(8)-C(9)	1.309(10)
C(10)-C(13)	1.482(14)
C(10)-C(11)	1.506(9)
C(11)-C(12)	1.331(9)
C(13)-C(14)	1.3900
C(13)-C(18)	1.3900
C(14)-C(15)	1.3900
C(15)-C(16)	1.3900
C(16)-C(17)	1.3900
C(17)-C(18)	1.3900
C(19)-C(20)	1.3900
C(19)-C(24)	1.3900
C(20)-C(21)	1.3900
C(21)-C(22)	1.3900
C(22)-C(23)	1.3900
C(22)-C(25)	1.548(13)
C(23)-C(24)	1.3900
C(25)-C(26)	1.512(10)
C(26)-C(27)	1.321(10)
C(28)-C(31)	1.505(14)

C(28)-C(29)	1.510(10)
C(29)-C(30)	1.327(9)
C(31)-C(32)	1.3900
C(31)-C(36)	1.3900
C(32)-C(33)	1.3900
C(33)-C(34)	1.3900
C(34)-C(35)	1.3900
C(35)-C(36)	1.3900
O(1)-S(1)-O(2)	117.9(10)
O(1)-S(1)-C(7)	111.7(9)
O(2)-S(1)-C(7)	107.7(7)
O(1)-S(1)-C(10)	107.1(7)
O(2)-S(1)-C(10)	107.6(8)
C(7)-S(1)-C(10)	104.0(6)
O(3)-S(2)-O(4)	117.5(10)
O(3)-S(2)-C(25)	109.4(8)
O(4)-S(2)-C(25)	108.5(9)
O(3)-S(2)-C(28)	107.9(9)
O(4)-S(2)-C(28)	109.2(7)
C(25)-S(2)-C(28)	103.4(6)
C(2)-C(1)-C(6)	120.0
C(2)-C(1)-Br(3)	122.4(5)
C(6)-C(1)-Br(3)	117.5(5)
C(1)-C(2)-C(3)	120.0
C(4)-C(3)-C(2)	120.0
C(3)-C(4)-C(5)	120.0
C(3)-C(4)-C(7)	118.6(7)
C(5)-C(4)-C(7)	121.2(7)
C(6)-C(5)-C(4)	120.0
C(5)-C(6)-C(1)	120.0
C(8)-C(7)-C(4)	113.0(11)
C(8)-C(7)-S(1)	110.8(9)
C(4)-C(7)-S(1)	107.5(8)
C(9)-C(8)-C(7)	120.3(14)
C(13)-C(10)-C(11)	112.7(11)
C(13)-C(10)-S(1)	109.8(7)
C(11)-C(10)-S(1)	109.8(9)
C(12)-C(11)-C(10)	120.6(13)
C(14)-C(13)-C(18)	120.0
C(14)-C(13)-C(10)	118.1(8)
C(18)-C(13)-C(10)	121.8(8)
C(15)-C(14)-C(13)	120.0
C(14)-C(15)-C(16)	120.0

C(15)-C(16)-C(17)	120.0
C(15)-C(16)-Br(2)	120.3(5)
C(17)-C(16)-Br(2)	119.6(5)
C(18)-C(17)-C(16)	120.0
C(17)-C(18)-C(13)	120.0
C(20)-C(19)-C(24)	120.0
C(20)-C(19)-Br(4)	123.3(5)
C(24)-C(19)-Br(4)	116.7(5)
C(19)-C(20)-C(21)	120.0
C(20)-C(21)-C(22)	120.0
C(23)-C(22)-C(21)	120.0
C(23)-C(22)-C(25)	121.9(8)
C(21)-C(22)-C(25)	117.9(8)
C(22)-C(23)-C(24)	120.0
C(23)-C(24)-C(19)	120.0
C(26)-C(25)-C(22)	114.1(11)
C(26)-C(25)-S(2)	110.4(10)
C(22)-C(25)-S(2)	106.8(8)
C(27)-C(26)-C(25)	122.4(14)
C(31)-C(28)-C(29)	112.2(11)
C(31)-C(28)-S(2)	109.7(7)
C(29)-C(28)-S(2)	112.8(10)
C(30)-C(29)-C(28)	124.2(14)
C(32)-C(31)-C(36)	120.0
C(32)-C(31)-C(28)	121.7(7)
C(36)-C(31)-C(28)	118.3(7)
C(31)-C(32)-C(33)	120.0
C(34)-C(33)-C(32)	120.0
C(35)-C(34)-C(33)	120.0
C(35)-C(34)-Br(1)	121.0(5)
C(33)-C(34)-Br(1)	119.0(5)
C(34)-C(35)-C(36)	120.0
C(35)-C(36)-C(31)	120.0

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for xl.
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
Br(1)	95(1)	81(1)	40(1)	-13(1)	20(1)	-5(1)
Br(2)	98(1)	81(1)	38(1)	-12(1)	-15(1)	9(1)
Br(3)	98(1)	79(1)	39(1)	15(1)	-15(1)	-5(1)
Br(4)	96(1)	79(1)	41(1)	14(1)	19(1)	6(1)
O(1)	240(20)	62(8)	44(7)	-17(6)	-26(9)	61(10)
O(2)	203(17)	76(9)	43(7)	18(6)	-12(8)	-69(10)
O(3)	240(20)	65(8)	47(7)	16(6)	30(10)	82(11)
O(4)	200(17)	58(7)	47(7)	-10(6)	12(8)	-48(8)
S(1)	145(4)	40(2)	31(2)	-3(2)	-9(2)	7(2)
S(2)	149(4)	43(2)	29(2)	-2(1)	16(2)	3(2)
C(1)	84(10)	43(7)	32(6)	-9(5)	0(6)	9(6)
C(2)	82(10)	45(8)	37(7)	14(6)	8(6)	6(6)
C(3)	66(9)	83(11)	35(7)	-14(7)	16(6)	5(7)
C(4)	65(8)	59(9)	22(6)	-3(5)	17(5)	10(6)
C(5)	83(10)	49(9)	52(9)	-4(7)	20(7)	19(7)
C(6)	96(12)	52(9)	46(8)	7(7)	5(8)	13(8)
C(7)	68(8)	48(7)	24(5)	-1(5)	13(5)	0(6)
C(8)	94(11)	48(10)	45(9)	13(7)	27(8)	12(7)
C(9)	87(12)	90(14)	50(10)	9(9)	3(8)	-21(9)
C(10)	84(9)	33(6)	27(6)	11(5)	4(6)	-1(6)
C(11)	87(11)	46(9)	41(8)	3(6)	8(7)	-3(7)
C(12)	92(12)	75(12)	53(10)	-23(8)	-2(8)	25(9)
C(13)	79(10)	42(8)	37(7)	6(6)	3(6)	-1(6)
C(14)	71(9)	57(9)	44(8)	3(7)	7(7)	-2(6)
C(15)	78(9)	52(8)	27(6)	1(5)	15(6)	1(6)
C(16)	84(10)	43(7)	31(6)	-2(5)	-5(6)	9(6)
C(17)	76(10)	69(10)	56(10)	-4(8)	8(8)	-14(8)
C(18)	76(10)	62(10)	35(7)	6(7)	10(6)	-8(7)
C(19)	97(11)	35(7)	29(6)	-6(5)	16(6)	3(6)
C(20)	105(12)	47(8)	35(7)	6(6)	1(7)	-14(7)
C(21)	63(9)	79(10)	38(7)	-15(7)	1(6)	-3(7)

C(22)	69(9)	60(9)	26(6)	0(6)	6(6)	-1(6)
C(23)	85(11)	102(14)	40(9)	21(9)	-24(8)	-25(10)
C(24)	80(11)	65(10)	55(9)	4(8)	18(8)	-14(8)
C(25)	86(10)	42(7)	28(6)	-1(5)	4(6)	1(6)
C(26)	86(12)	115(17)	38(9)	14(9)	-5(8)	-31(11)
C(27)	86(11)	62(11)	70(11)	10(9)	2(9)	25(8)
C(28)	90(10)	30(6)	27(6)	3(5)	-1(6)	9(6)
C(29)	73(11)	110(16)	39(8)	-23(9)	-7(7)	30(10)
C(30)	84(11)	56(10)	64(11)	8(8)	9(8)	-13(8)
C(31)	87(10)	34(7)	34(6)	11(5)	0(6)	8(6)
C(32)	110(13)	74(11)	26(7)	-10(7)	-2(7)	32(10)
C(33)	65(9)	75(10)	54(9)	4(8)	8(7)	11(7)
C(34)	72(9)	49(8)	37(7)	11(6)	22(6)	5(6)
C(35)	90(11)	75(11)	36(7)	-12(7)	-10(7)	6(8)
C(36)	74(9)	64(10)	41(8)	-5(7)	7(7)	8(7)

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

	x	y	z	U(eq)
H(2B)	3357	25438	-3032	65
H(3B)	2759	27008	-4110	73
H(5A)	7889	25321	-4878	74
H(6A)	8487	23751	-3800	78
H(7A)	3818	28339	-5175	56
H(8A)	7765	28110	-5609	75
H(9A)	5346	31093	-5335	91
H(9B)	7554	31240	-5596	91
H(10A)	4639	28341	-6826	58
H(11A)	734	28081	-6345	69
H(12A)	3087	31122	-6652	88
H(12B)	906	31241	-6358	88
H(14A)	5696	27037	-7870	69
H(15A)	5121	25440	-8945	63
H(17A)	-56	23826	-8199	80
H(18A)	519	25423	-7124	69
H(20A)	8371	19523	-3960	75
H(21A)	7783	17972	-2878	72
H(23A)	12948	19626	-2125	91
H(24A)	13536	21176	-3206	80
H(25A)	8841	16596	-1815	63
H(26A)	12767	16855	-1347	95
H(27A)	10432	13783	-1588	87
H(27B)	12633	13726	-1311	87
H(28A)	9622	16643	-171	59
H(29A)	5691	16815	-605	89
H(30A)	8021	13752	-352	82
H(30B)	5803	13692	-609	82
H(32A)	5496	19554	144	84
H(33A)	4934	21124	1225	77
H(35A)	10127	19512	1961	80
H(36A)	10690	17942	879	72