Electronic Supplementary Material (ESI) for Organic and Biomolecular Chemistry

Electronic Supplementary Information for: Cyclodextrin-alloxazin conjugates for organocatalytic enantioselective sulfoxidations

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Contents

1 Characterizations of synthesized compounds

1.1 NMR spectra of compounds 4 and 5a

The NMR spectra of **4** were measured on Bruker AVANCE-600 instrument (^{1}H at 600.13 MHz and ^{13}C at 150.9 MHz) in DMSO. The NMR spectra of **5a** were measured on Bruker AVANCE-600 instrument (^{1}H at 600.13 MHz and ^{13}C at 150.9 MHz) with a cryo-probe in D_2O at 37 °C. Spectra are referenced to dioxane (added as internal standard) using δ_H(dioxane) = 3.75 ppm and δ_C(dioxane) = 69.3 ppm.

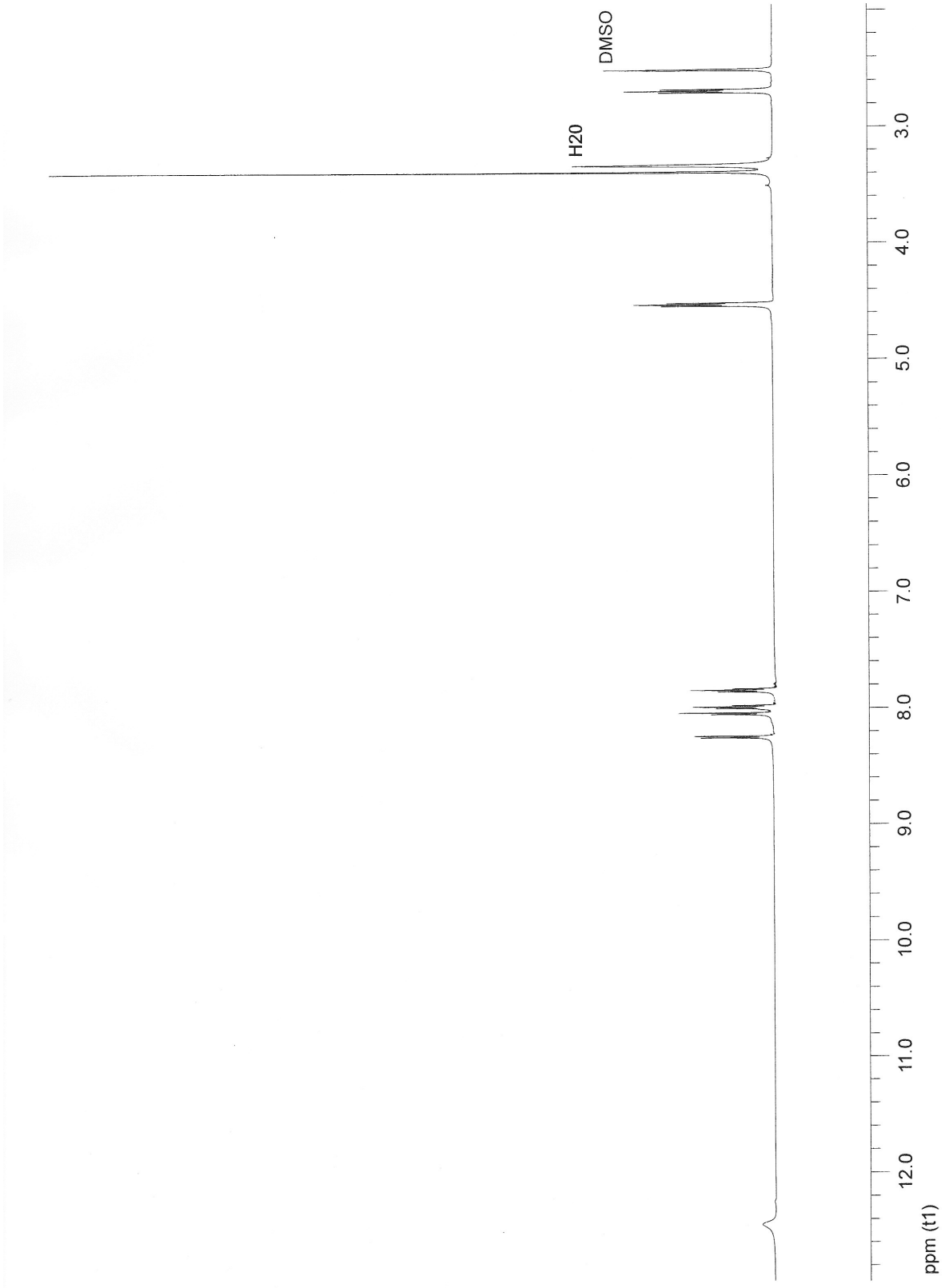


Figure S1: ^{1}H-NMR spectrum of compound **4**

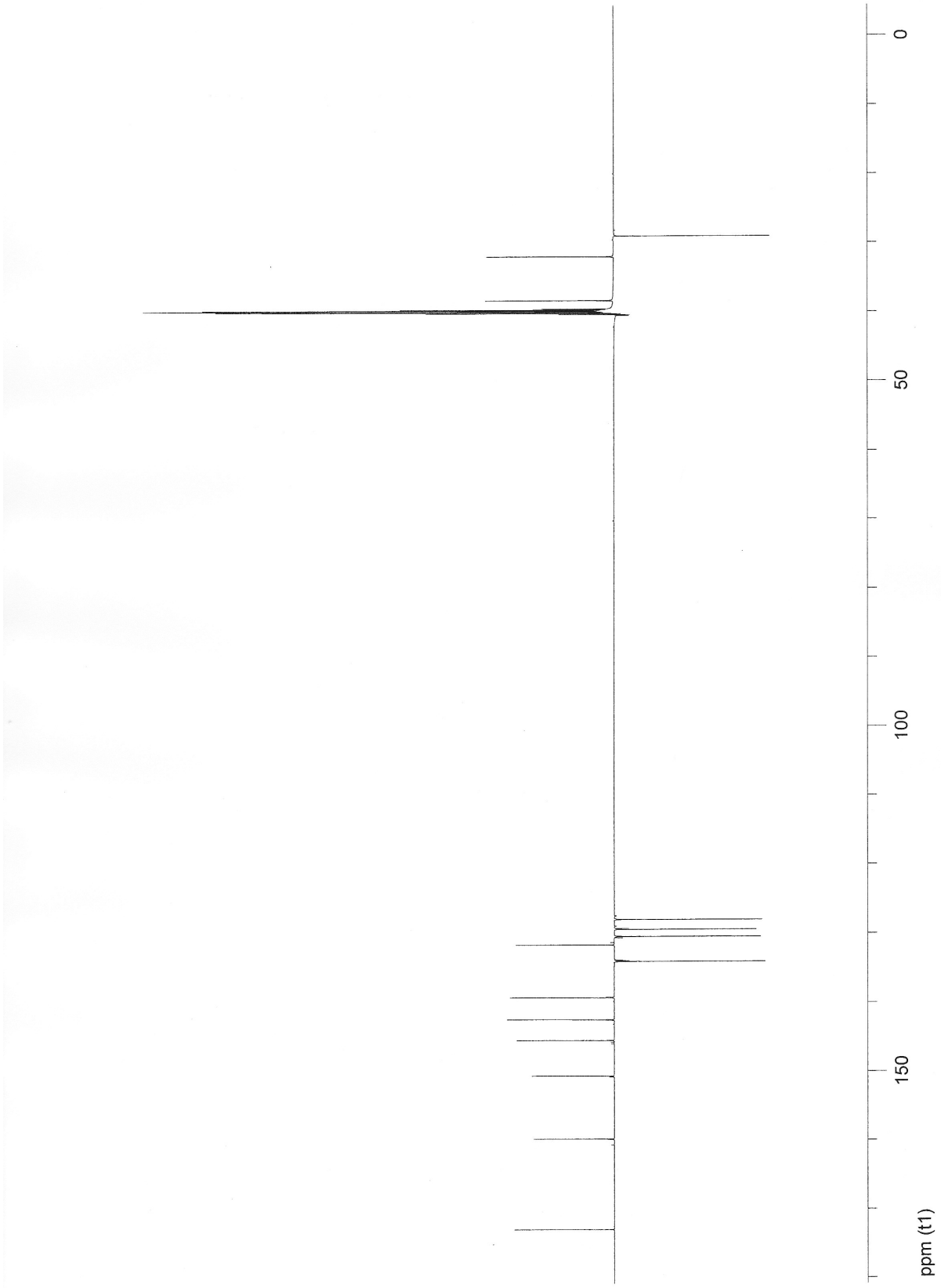


Figure S2: ^{13}C-NMR spectrum of compound **4**

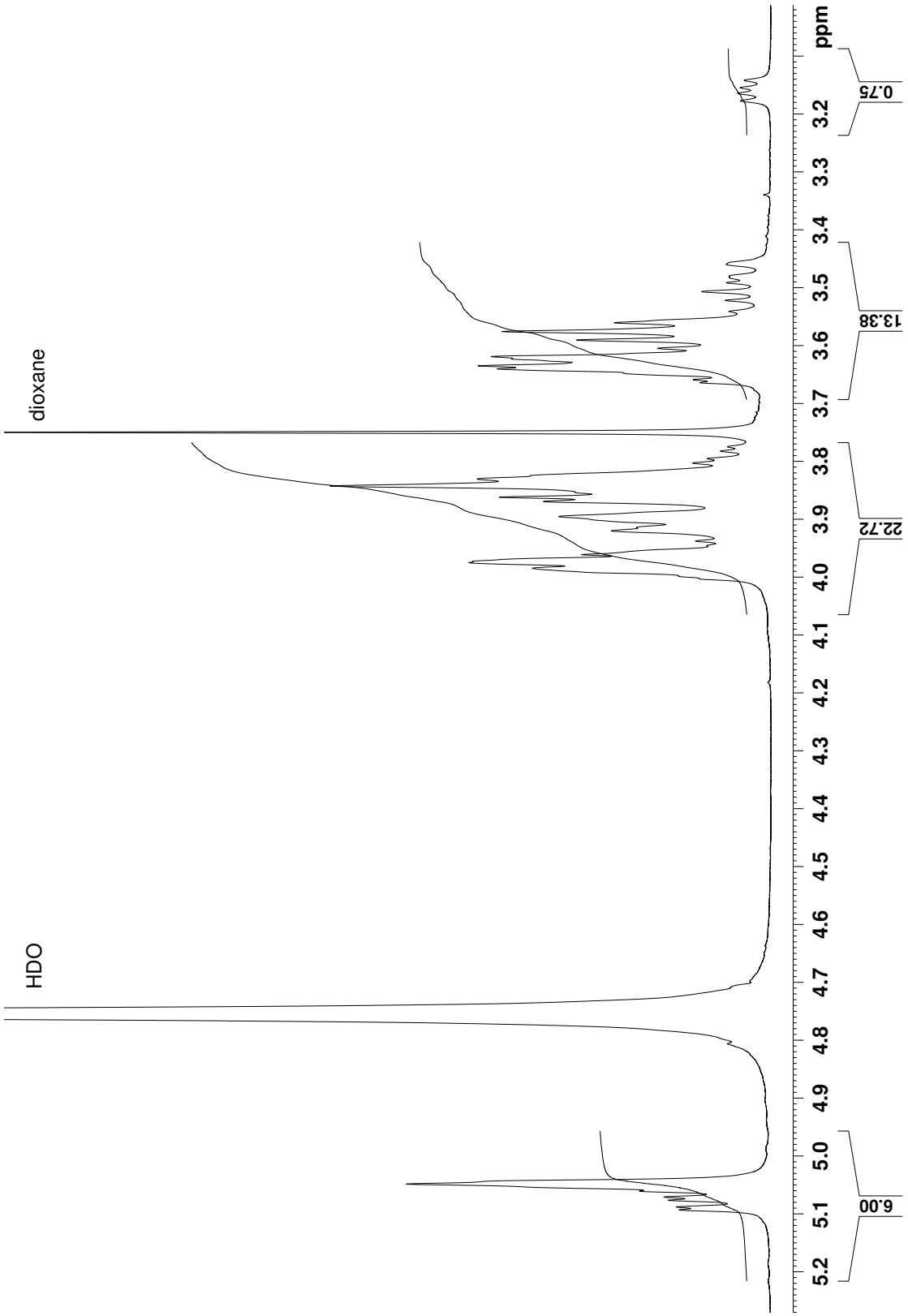


Figure S3: ^{1}H-NMR spectrum of compound **5a**

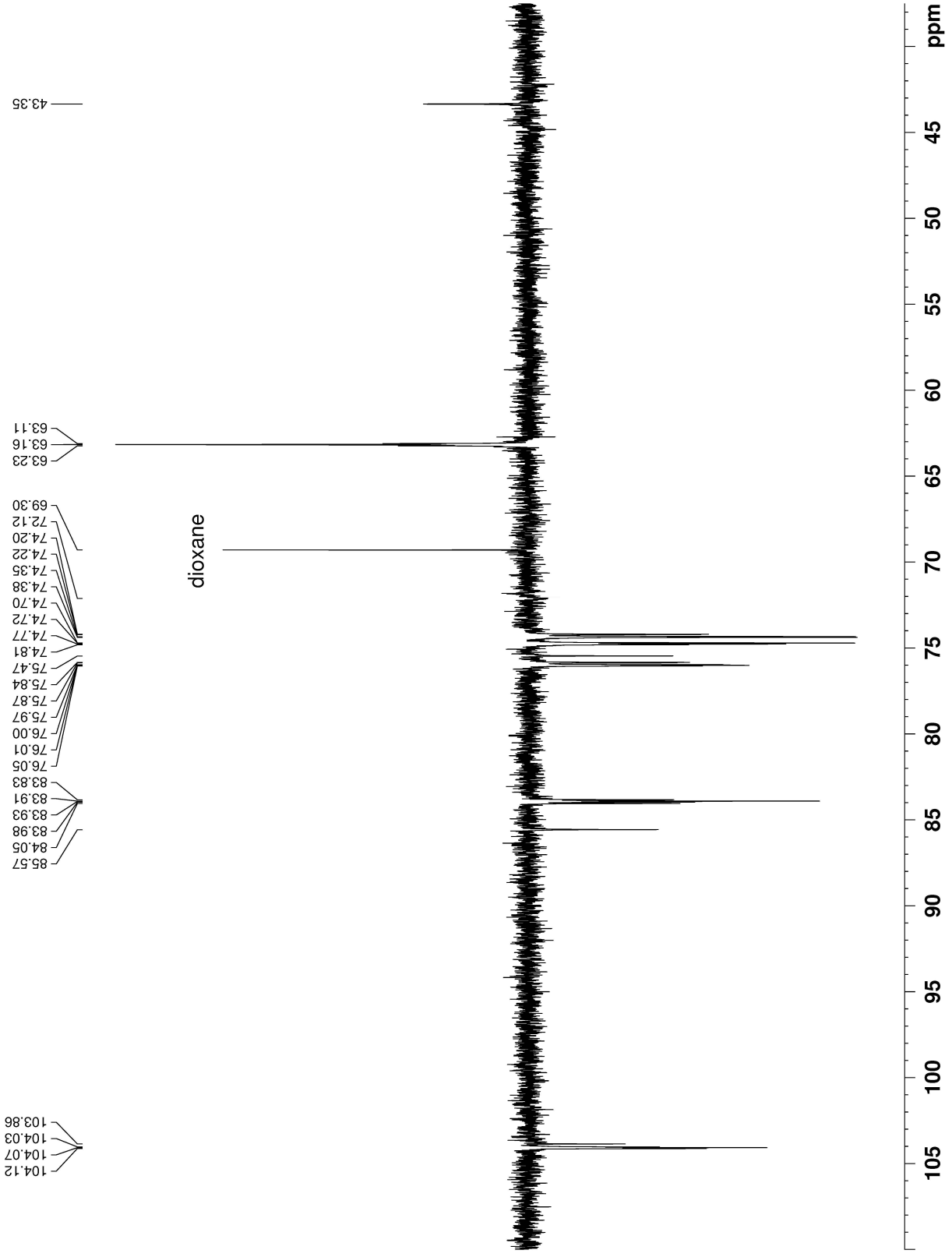


Figure S4: ^{13}C-NMR spectrum of compound **5a**

1.2 NMR data of conjugates 6a, 7a and 7b

The NMR spectra were measured on Bruker AVANCE-600 instrument (^{1}H at 600.13 MHz and ^{13}C at 150.9 MHz) with a cryo-probe in D_2O at 37 °C. Spectra are referenced to dioxane (added as internal standard) using δ_H(dioxane) = 3.75 ppm and δ_C(dioxane) = 69.3 ppm. For structural assignment of proton and carbon signals combination of 2D-H,H-COSY, 2D-H,H-TOCSY, 2D-H,C-HSQC and 2D-H,C-HMBC spectra was used. The complete intraresidue and sequential assignment of cyclodextrin proton and carbon signals was achieved only for compound **7b**.

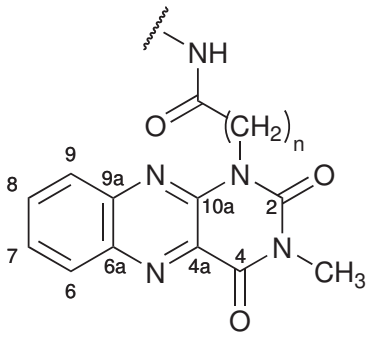


Figure S5: Numbering of alloxazine skeleton used in Table S1

Table S1: Carbon and proton NMR data of compounds **6a**, **7a** and **7b** (aglycone part) in D_2O at 37 °C.

Compound	C-2	C-4	C-4a	C-6a	C-6	C-7	C-8	C-9	C-9a	C-10a	N-CH_3	N-CH_2	CH_2-CO	C=O
6a	153.95	163.71	131.62	141.96	132.13	133.08	137.61	130.28	145.59	146.99	31.71	47.21	–	172.42
7a	153.77	163.81	131.64	141.72	132.15	132.90	137.45	130.39	145.84	147.19	31.60	42.19	36.68	176.27
7b	153.82	163.42	132.64	142.17	132.39	132.72	137.25	129.78	145.00	148.18	31.55	42.39	36.44	175.84

Compound	H-6	H-7	H-8	H-9	N-CH_3	N-CH_2		CH_2-CO
6a	8.16 m	7.88 m	8.01 m	8.01 m	3.51 s	5.30 d (16.5)	5.00 d (16.5)	–
7a	8.19 bdd (8.5; 1.4)	7.89 ddd (8.5; 7.0; 1.4)	8.03 ddd (8.5; 7.0; 1.4)	8.11 bdd (8.5; 1.4)	3.49 s	4.67 m (2H)		2.75 m (2H)
7b	8.36 m	8.07 m	8.18 m	8.20 m	3.55 s	5.15 ddd (14; 10; 4)	4.44 dt (14; 4; 4)	2.68 m (2H)

Table S2: Carbon and proton NMR data of compounds **6a**, **7a** and **7b** (cyclodextrin part) in D_2O at 37 °C.

Compound	Glucose residue	C-1'	C-2'	C-3'	C-4'	C-5'	C-6'
6a		104.18	74.38	76.06	86.02	74.64	63.05
		104.13	74.35 (3)	75.99	83.83	74.61	62.96 (2)
		104.10 (2)	74.33 (2)	75.97	83.80	74.56	62.83
		104.00 (2)		75.95	83.78	74.63	62.68
				75.92	83.70	74.49	43.50
				75.69	83.60	72.68	
7a		104.24	74.40	76.09	85.98	74.65	63.07
		104.21 (3)	74.38 (2)	76.06	83.96	74.61	63.02
		104.10	74.34 (2)	76.01	83.91	74.58 (2)	62.89
		103.94	74.31	75.98	83.80	74.55	62.77
				75.95	83.76	73.07	62.66
				75.84	83.66		43.01
7b	**I**	104.27	74.93	75.96	85.91	75.68	42.00
	II	104.92	74.60	75.78	83.44	75.10	62.93
	III	104.42	74.32	74.32	83.64	73.16	62.74
	IV	103.74	74.96	75.77	83.42	74.24	62.38
	V	105.23	74.87	76.05	82.83	74.85	63.14
	VI	104.55	74.51	76.29	84.24	75.63	63.00
	VII	104.47	74.73	75.89	82.71	73.98	61.75
		H-1'	**H-2'**	**H-3'**	**H-4'**	**H-5'**	**H-6'a+H-6'b**
6a		5.050	3.609	3.88 – 3.95	3.51 – 3.62	3.809	3.625 – 3.73 (4)
		5.032	3.619			3.70–3.75 (4)	3.99 + 3.868
		5.002	3.589 (2)			3.961	3.860 + 3.482
		4.996	3.518				
		4.965	3.565				
		4.954					
7a		5.026	3.543	3.84 – 3.94	3.51 – 3.60	3.63	3.51 + 3.63
		5.013	3.553			3.67	3.72 – 3.80 (3)
		4.999	3.576			3.74	3.81
		4.987	3.598 (2)			3.78	3.658 + 3.192
		4.943	3.603			3.80 (2)	
		4.897					
7b	**I**	5.165	3.692	3.922	3.298	3.717	4.07 + 2.70
	II	5.07	3.704	4.128	3.828	4.175	4.231 + 4.093
	III	4.77	~3.44	3.659	3.374	2.607	3.643 + 3.373
	IV	4.965	3.481	3.482	3.256	2.92	3.249 + 3.187
	V	5.19	3.744	4.152	3.711	4.508	4.168 + 3.995
	VI	4.91	3.625	3.995	3.659	~3.715	4.063 + 3.818
	VII	4.845	3.464	3.368	3.313	2.42	3.272 + 3.135

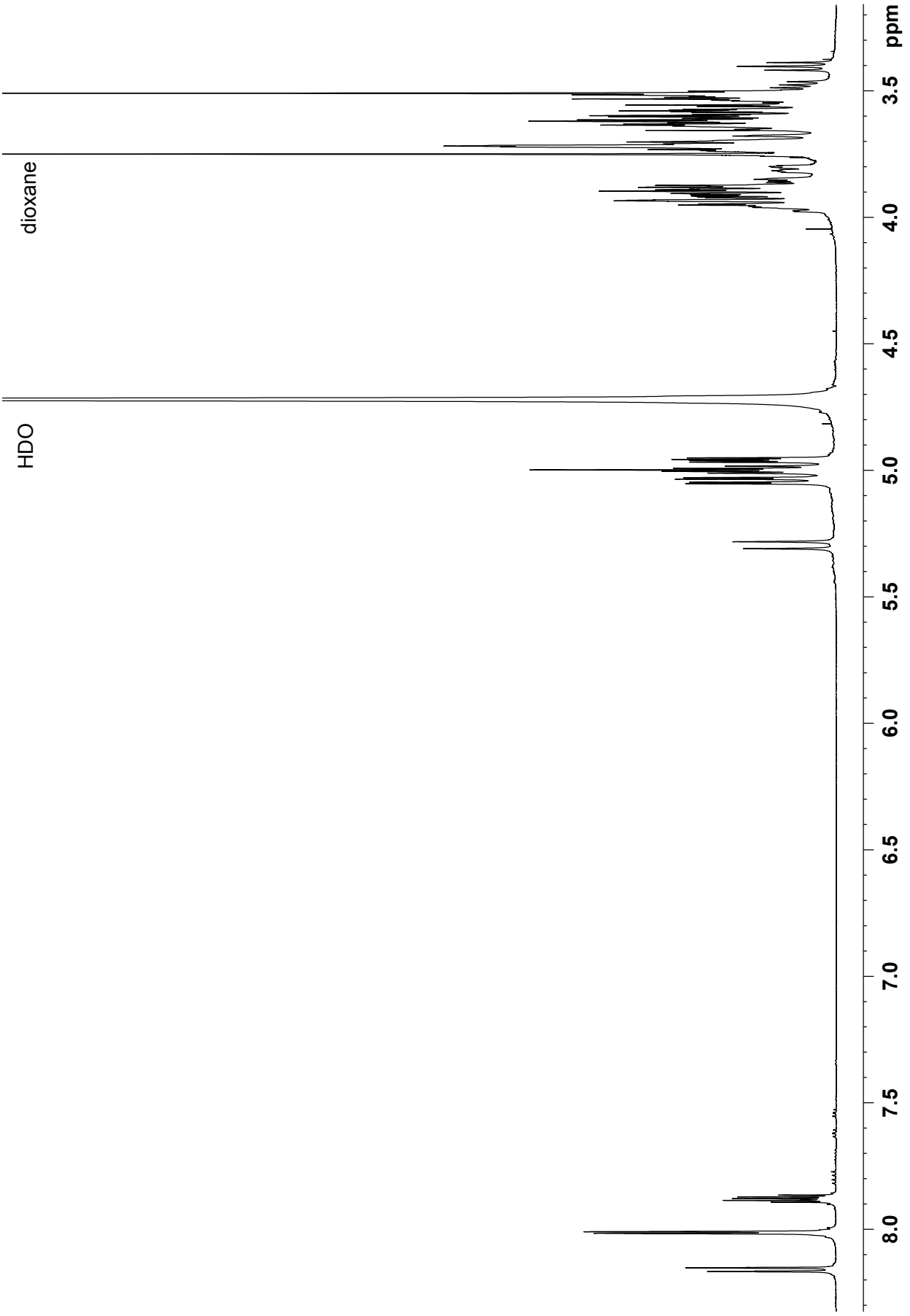


Figure S6: ^{1}H-NMR spectrum of conjugate **6a**

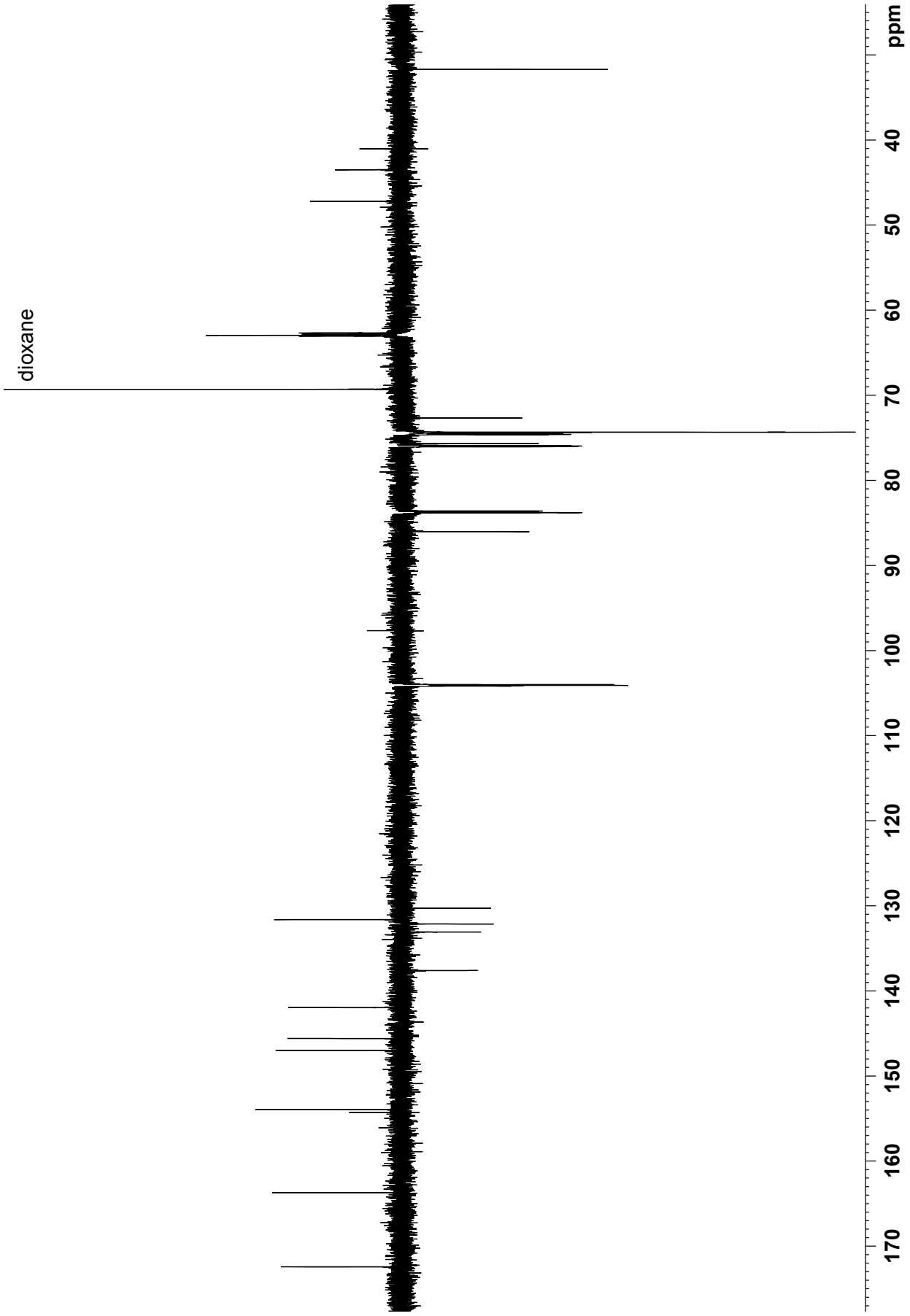


Figure S7: ^{13}C-NMRspectrum of conjugate **6a**

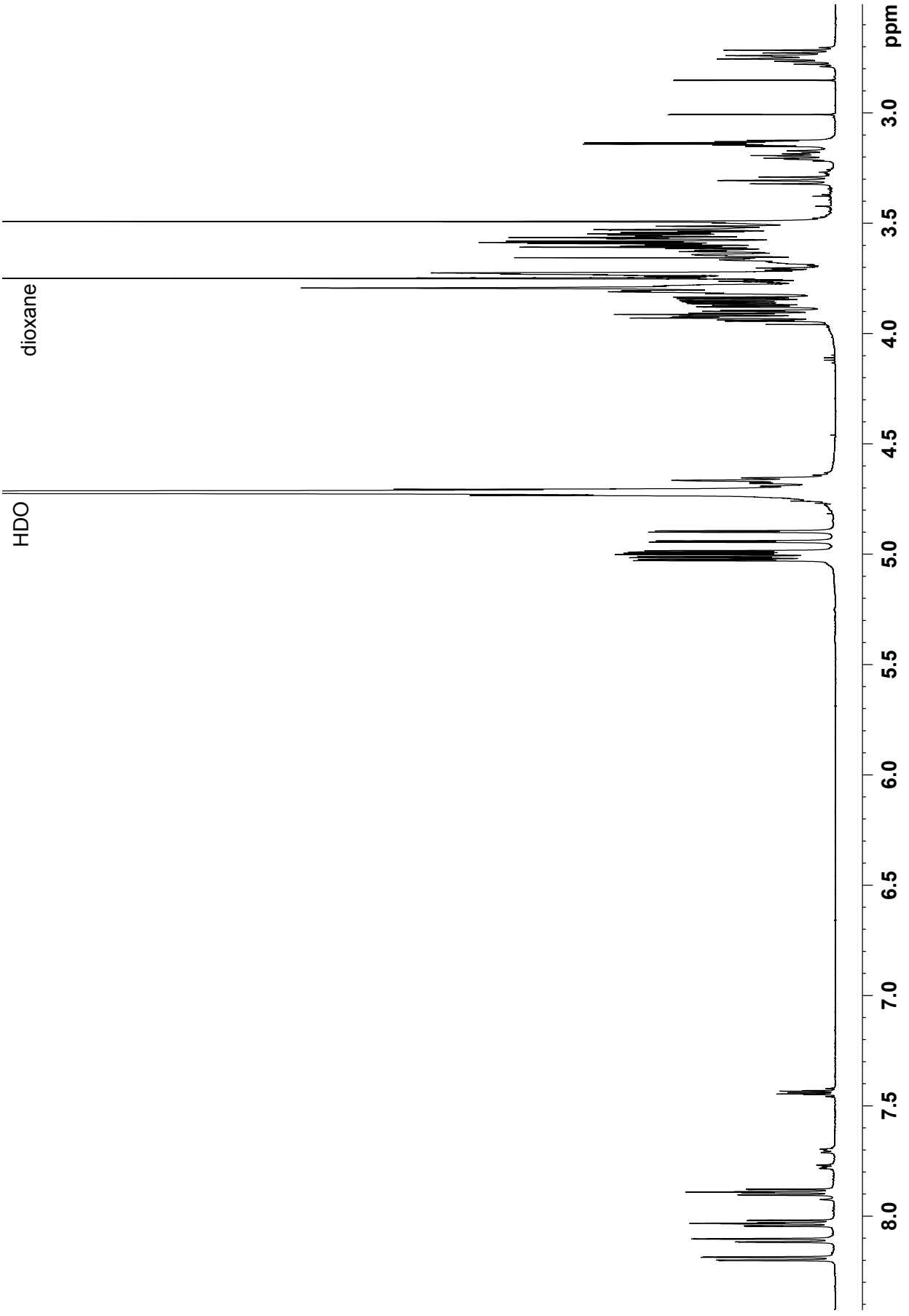


Figure S8: ^{1}H-NMR spectrum of conjugate **7a**

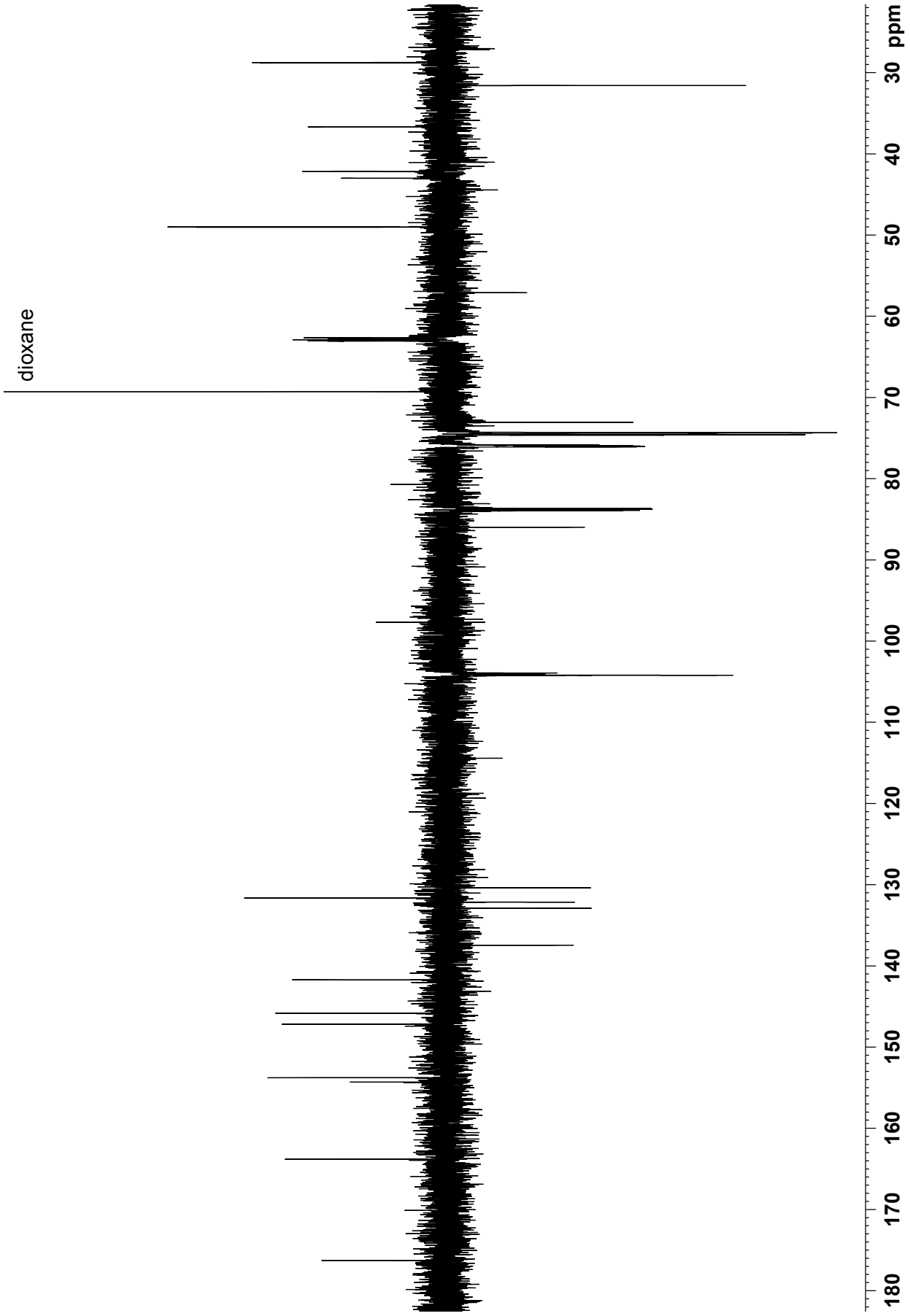


Figure S9: ^{13}C-NMRspectrum of conjugate **7a**

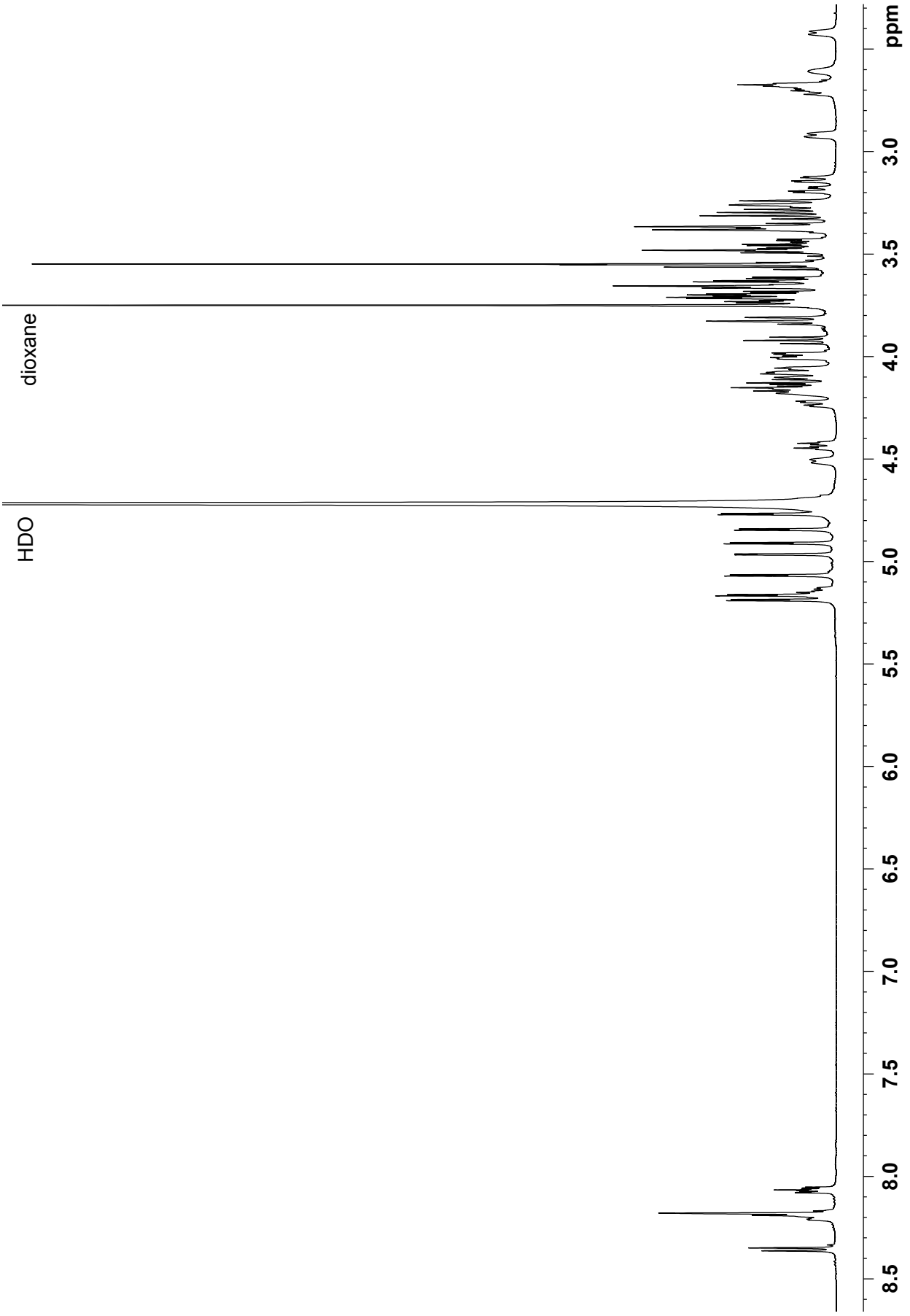


Figure S10: ^{1}H-NMR spectrum of conjugate **7b**

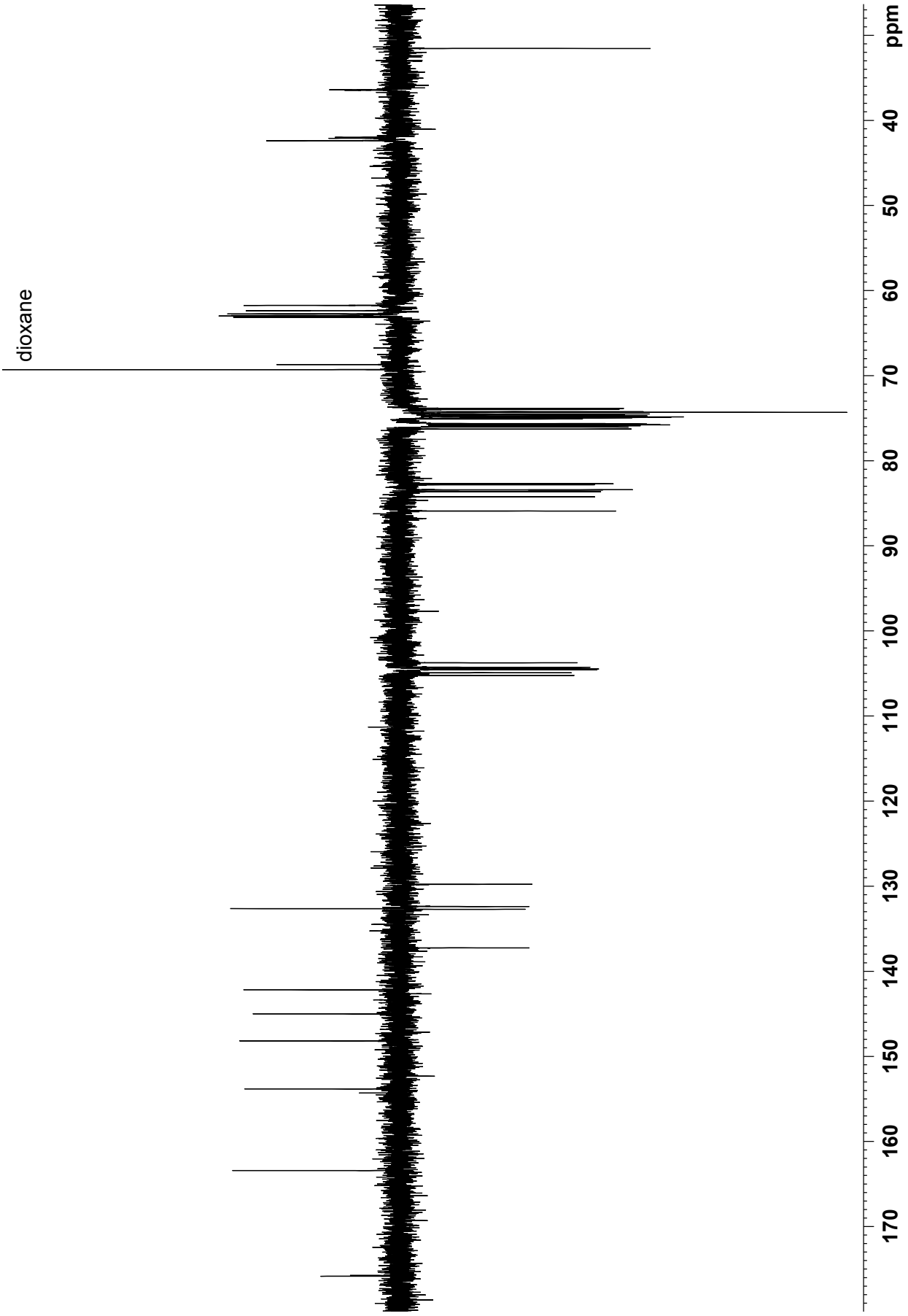


Figure S11: ^{13}C-NMR spectrum of conjugate **7b**

1.3 HR-MS spectra of flavinium salts 1a, 2a and 2b

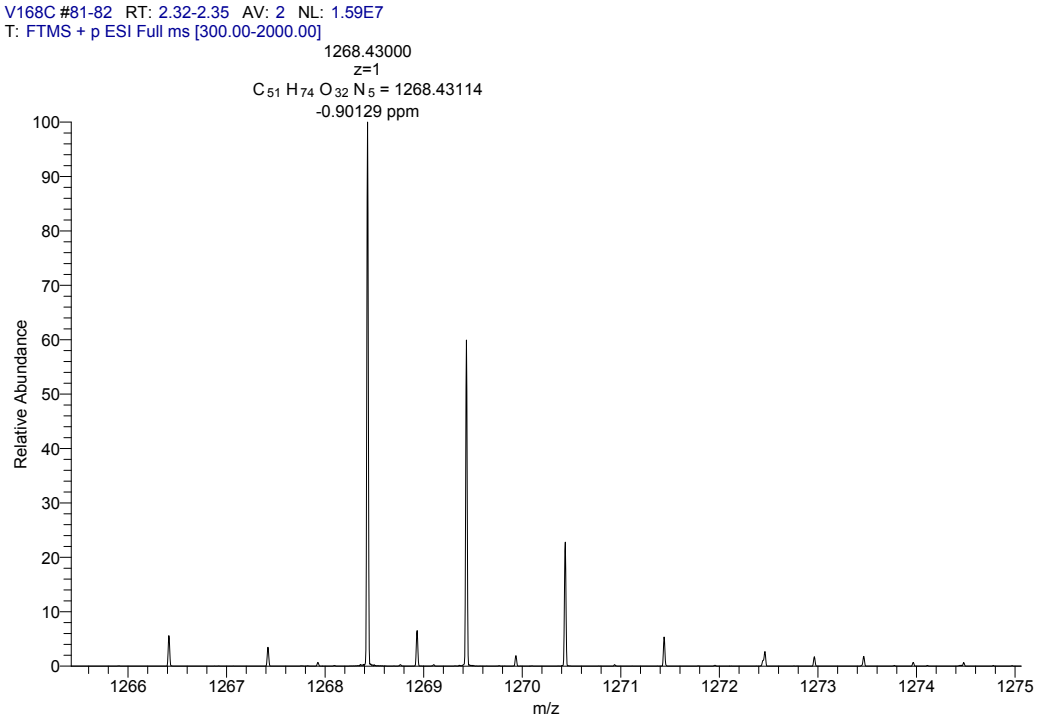


Figure S12: High resolution mass spectrum of compound **1a**

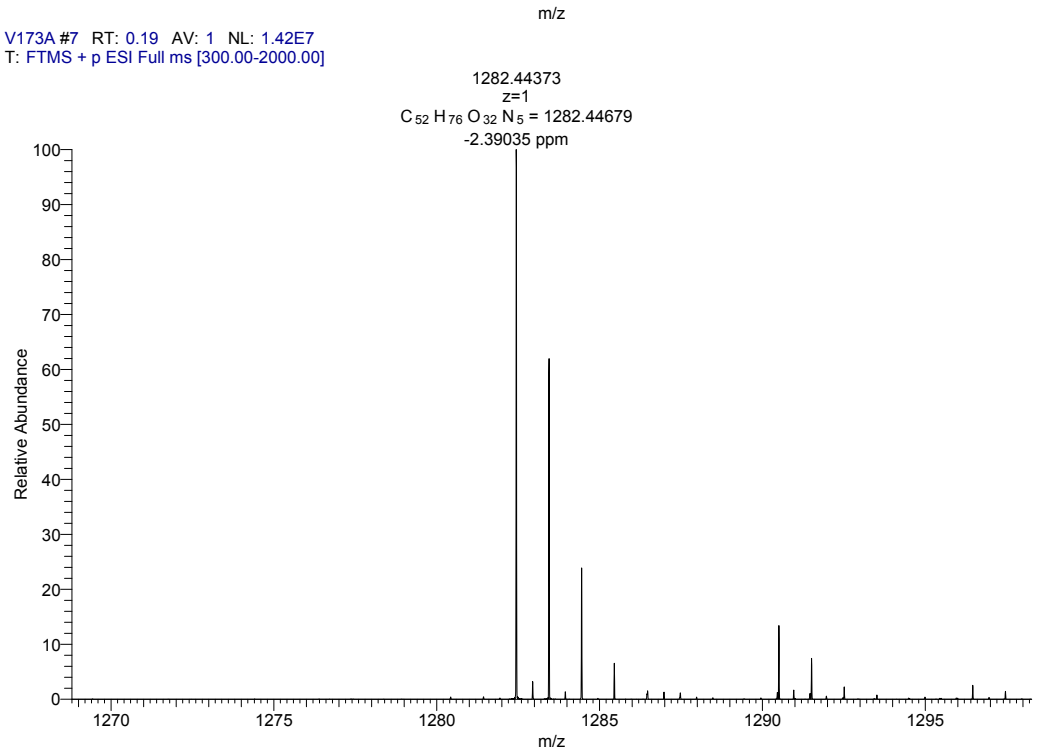


Figure S13: High resolution mass spectrum of compound **2a**

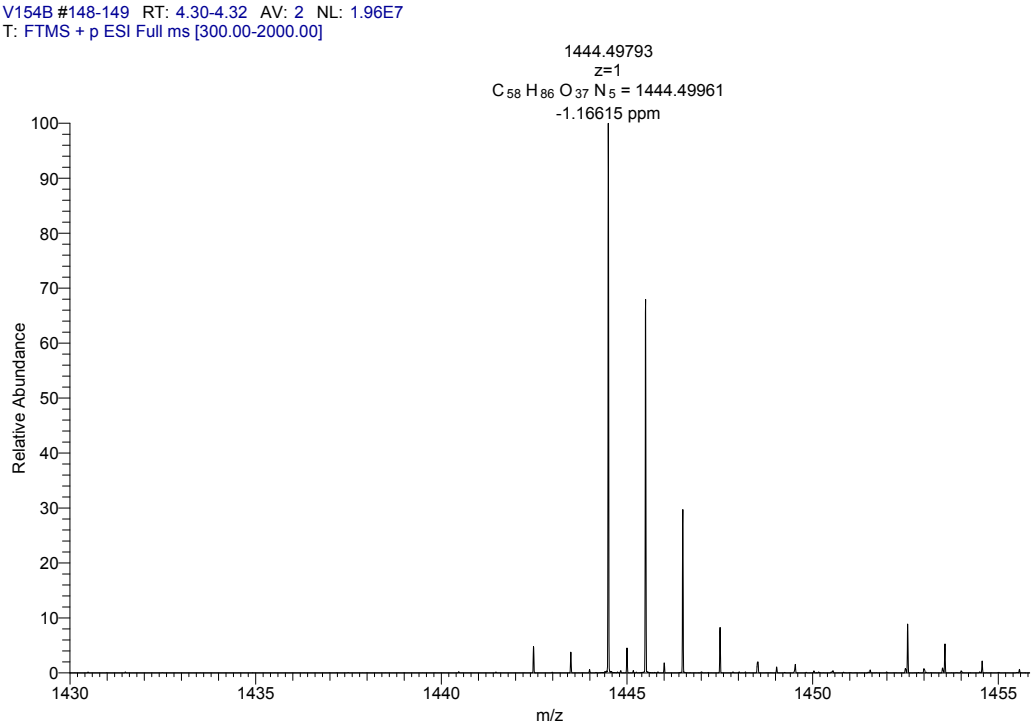


Figure S14: High resolution mass spectrum of compound **2b**

1.4 HR-MS spectra of conjugates 6a, 7a and 7b

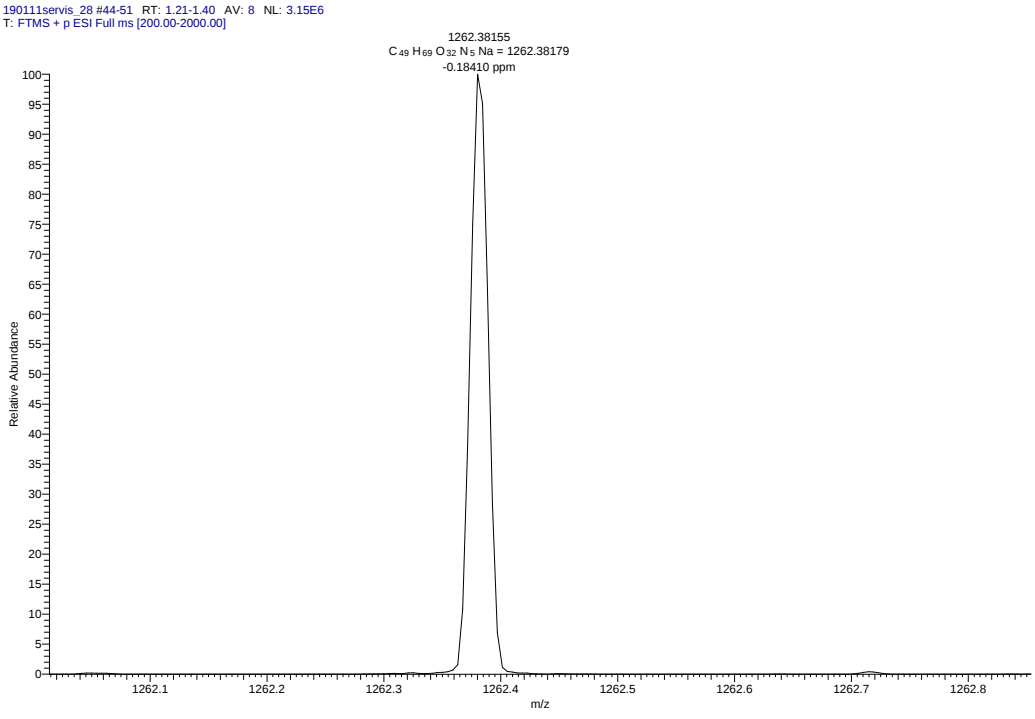


Figure S15: High resolution mass spectrum of compound **6a**

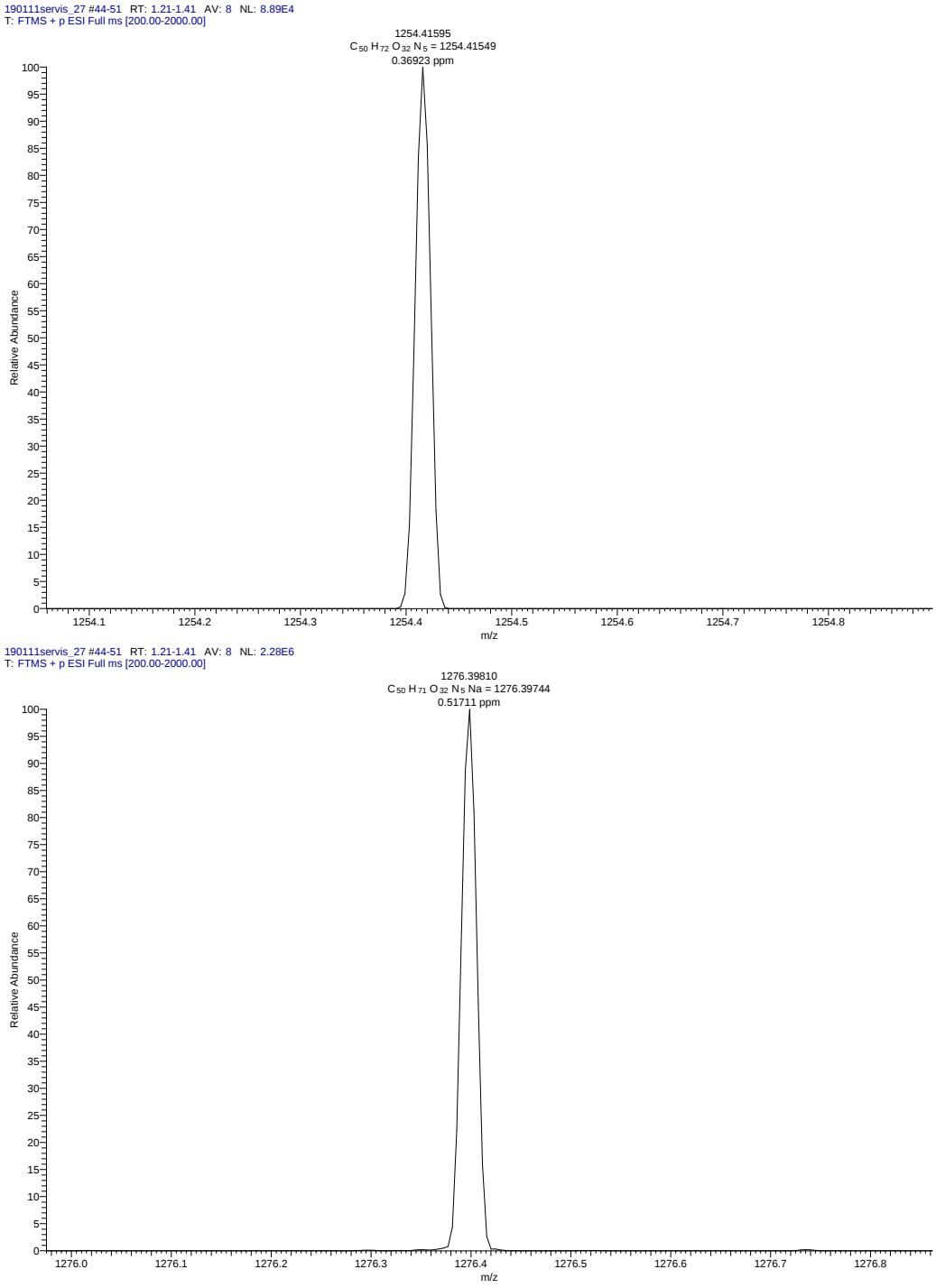


Figure S16: High resolution mass spectra of compound **7a**

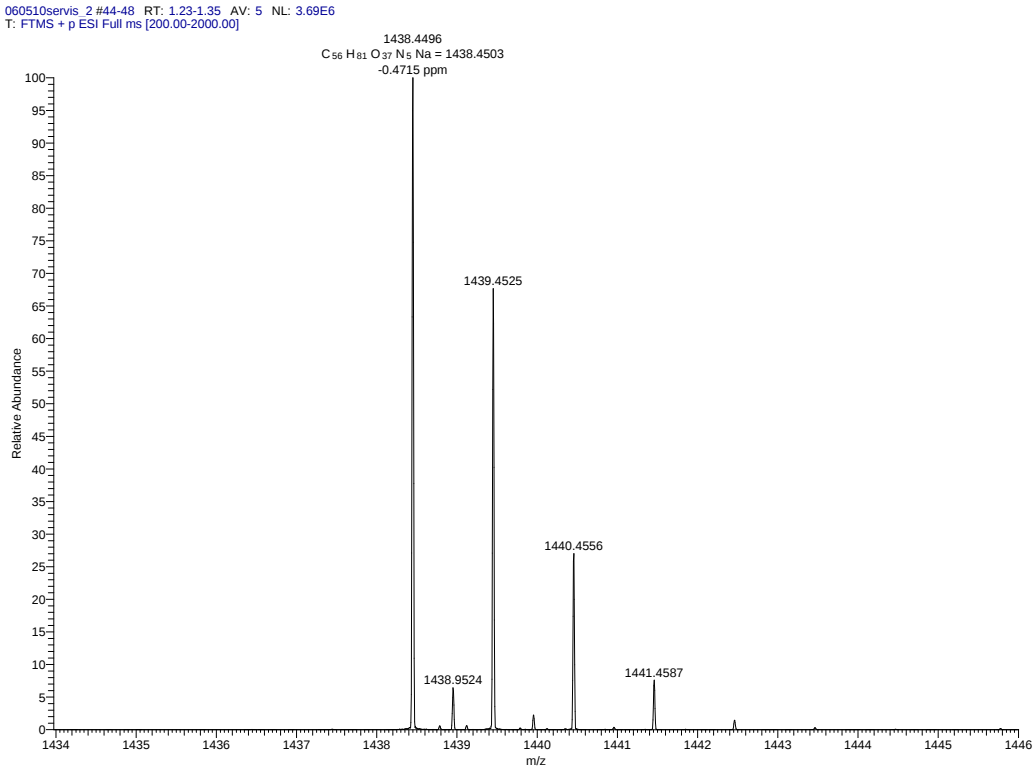


Figure S17: High resolution mass spectrum of compound **7b**

1.5 UV-VIS spectra of conjugates 1a, 2a and 2b

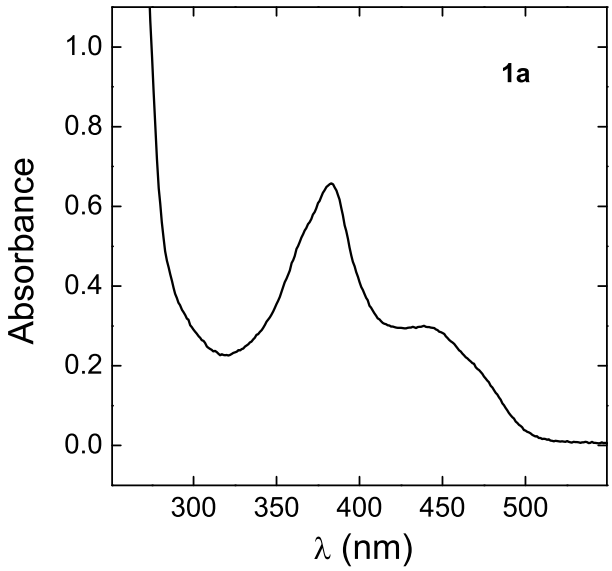


Figure S18: UV-VIS spectrum of compound **1a**. Phosphate buffer (pH 2), c(**1a**) = 5 x 10^{-5}M

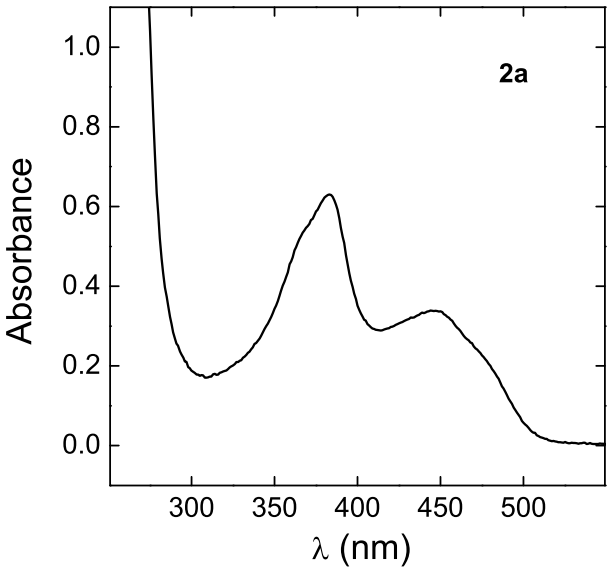


Figure S19: UV-VIS spectrum of compound **2a**. Phosphate buffer (pH 2), c(**2a**) = 5 x 10^{-5}M

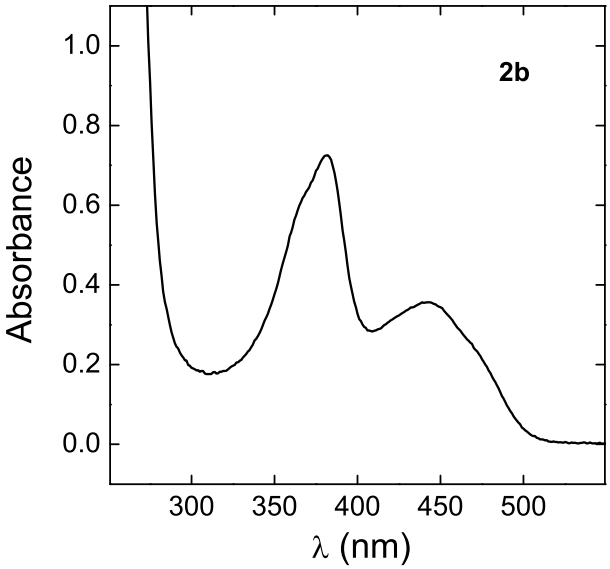


Figure S20: UV-VIS spectrum of compound **2b**. Phosphate buffer (pH 2), c(**2b**) = 5 x 10^{-5}M

1.6 UV-VIS spectra of conjugates 6a, 7a and 7b

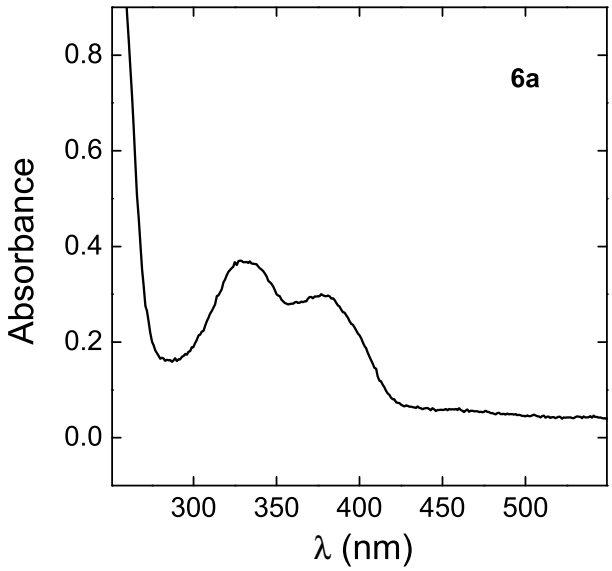


Figure S21: UV-VIS spectrum of compound **6a**. Phosphate buffer (pH 2), c(**6a**) = 5 x 10^{-5} M

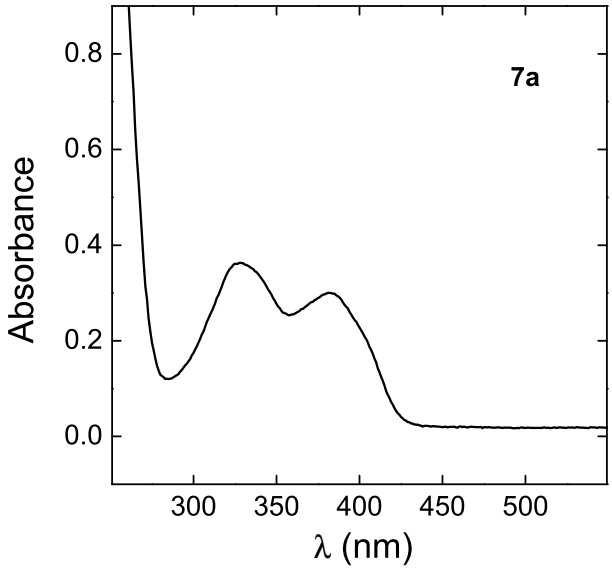


Figure S22: UV-VIS spectrum of compound **2a**. Phosphate buffer (pH 2), c(**7a**) = 5 x 10^{-5} M

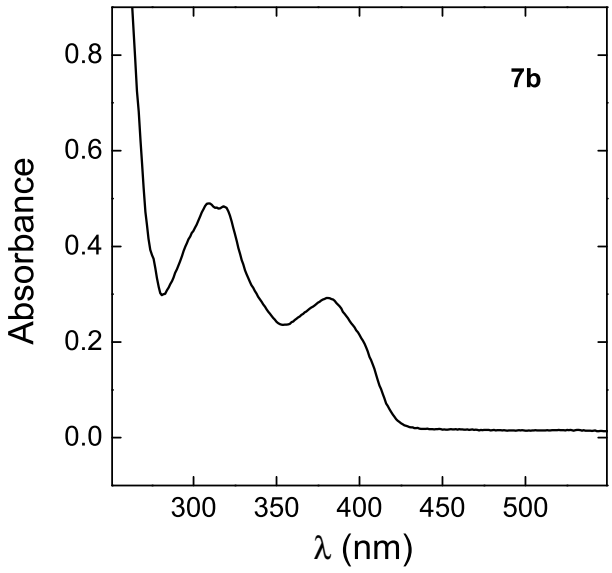


Figure S23: UV-VIS spectrum of compound **2b**. Phosphate buffer (pH 2), c(**7b**) = 5 x 10^{-5} M

2 Determination of ee by ^{1}H-NMR or chiral HPLC

All catalytic oxidations of sulfides were performed in the same manner as discribed by us.[1] Product of catalytic reaction was isolated by extraction with CCl_4 ($2\times500\,\mu L$) and then with $CDCl_3$ ($500\,\mu L$). The conversion of catalytic oxidations was determined as the ratio of peaks of the respective *S*-methyl groups in ^{1}H-NMR spectra. The enantiomeric excesses of *n*-alkyl methyl sulfoxides were determined by ^{1}H-NMR using (*R*)-(–)-3,5-dinitro-*N*-(1-phenylethyl)-benzamide as a shift reagent.[2] ^{1}H-NMR shifts may slightly vary according to the composition of the $CDCl_3$–CCl_4 mixture. In cases of aryl-, benzyl-, *tert*-butyl- and cyclohexyl- methyl sulfoxides, enantiomeric excess was determined by HPLC equipped with a chiral column. In addition, a set of samples representing all alkyl methyl sulfoxide structures was also analyzed by HPLC method using both UV and optical rotation (Chiralyser).

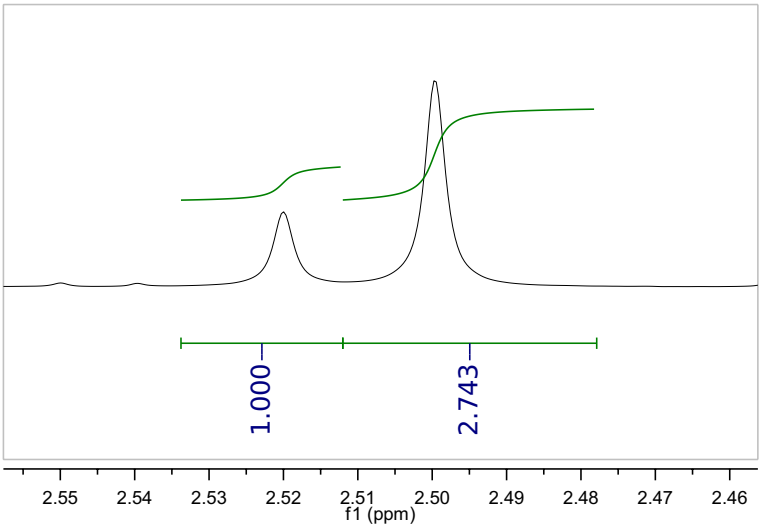


Figure S24: Part of the ^{1}H-NMR spectrum of product of hexyl(methyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 1, Table 1, main text).

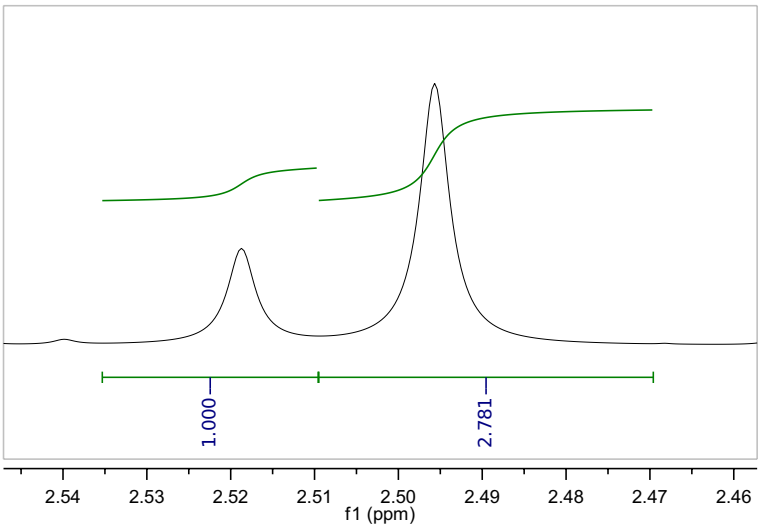


Figure S25: Part of the ^{1}H-NMR spectrum of product of hexyl(methyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 2, Table 1, main text).

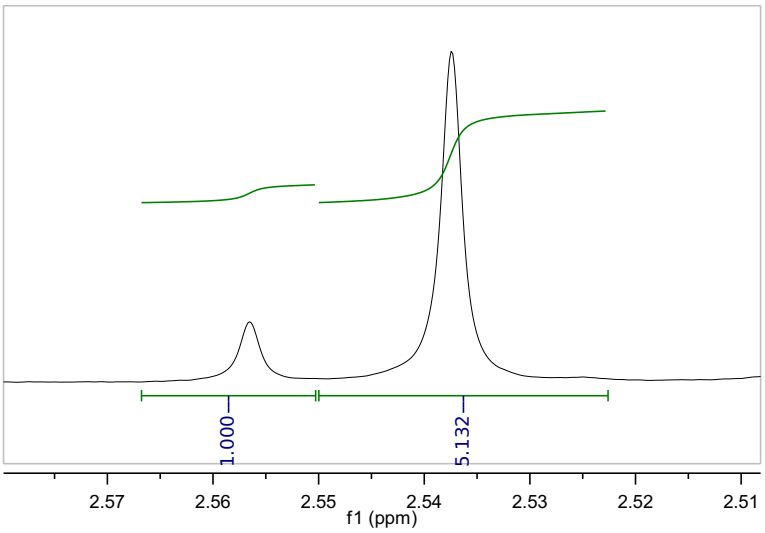
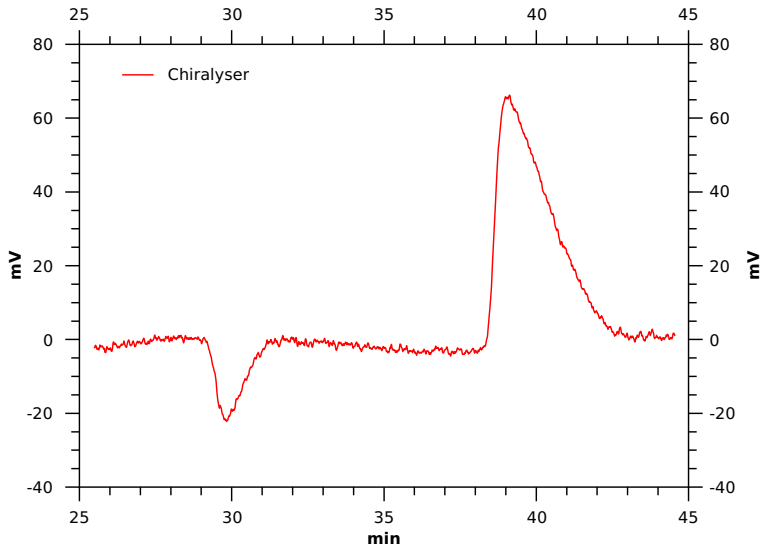


Figure S26: Part of the ^{1}H-NMR spectrum of product of methyl(octyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 3, Table 1, main text).



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	29.833	28.893	31.673	14.9	1.07
2	39.113	37.593	43.24	85.1	1.95

Figure S27: Part of the HPLC chromatogram of product of methyl(octyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 3, Table 1, main text). Conditions: **Daicel® Chiralpak AS-H** column, heptane/i-PrOH (17:3), 1 mL/min.

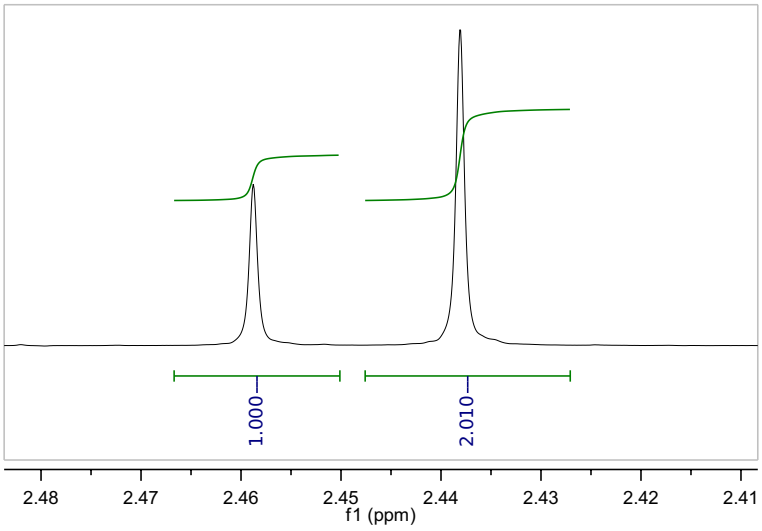
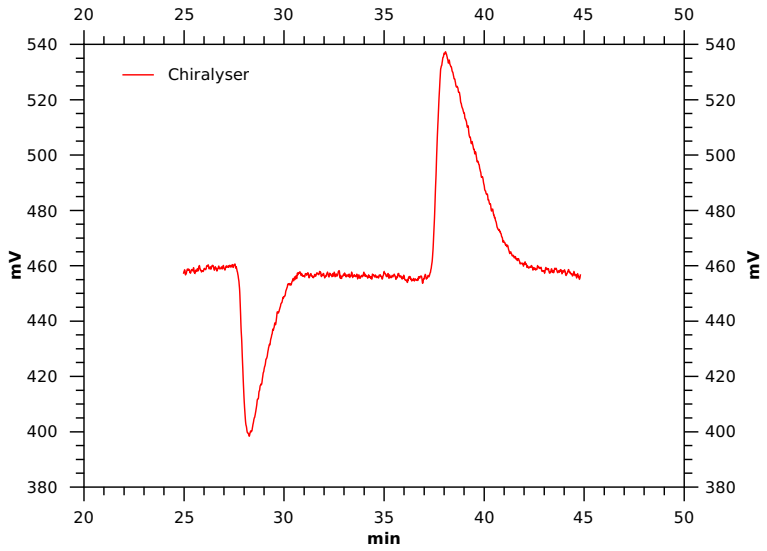


Figure S28: Part of the ^{1}H-NMR spectrum of product of methyl(octyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 4, Table 1, main text).



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	28.260	27.540	31.187	31.4	1.30
2	38.067	36.920	43.020	68.6	2.16

Figure S29: Part of the HPLC chromatogram of product of methyl(octyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 4, Table 1, main text). Conditions: **Daicel® Chiralpak AS-H** column, heptane/i-PrOH (17:3), 1 mL/min.

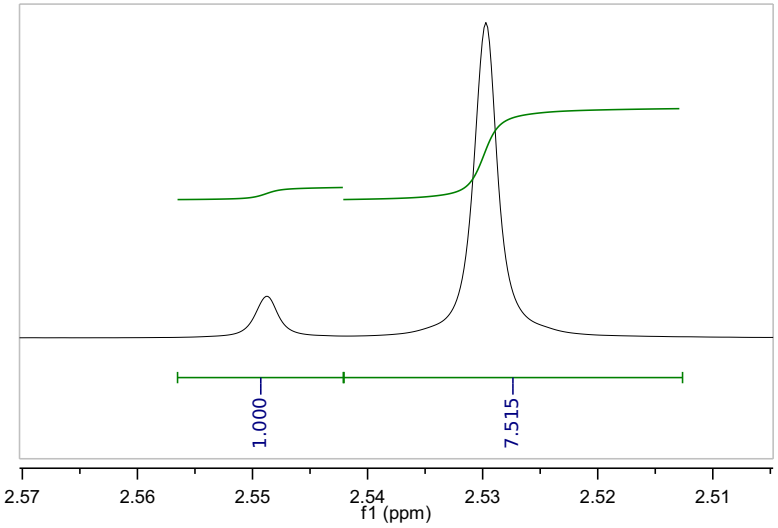
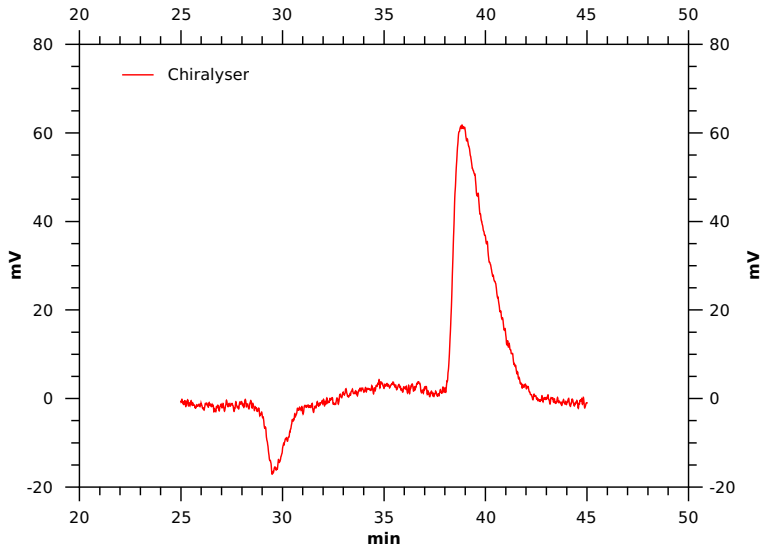


Figure S30: Part of the ^{1}H-NMR spectrum of product of methyl(octyl)sulfide oxidation catalyzed by **1a** (5 mol%), (Entry 5, Table 1, main text).



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	29.487	28.440	31.100	12.2	1.12
2	38.847	37.620	44.180	87.8	1.77

Figure S31: Part of the HPLC chromatogram of product of methyl(octyl)sulfide oxidation catalyzed by **1a** (5 mol%), (Entry 5, Table 1, main text). Conditions: **Daicel® Chiralpak AS-H** column, heptane/i-PrOH (17:3), 1 mL/min.

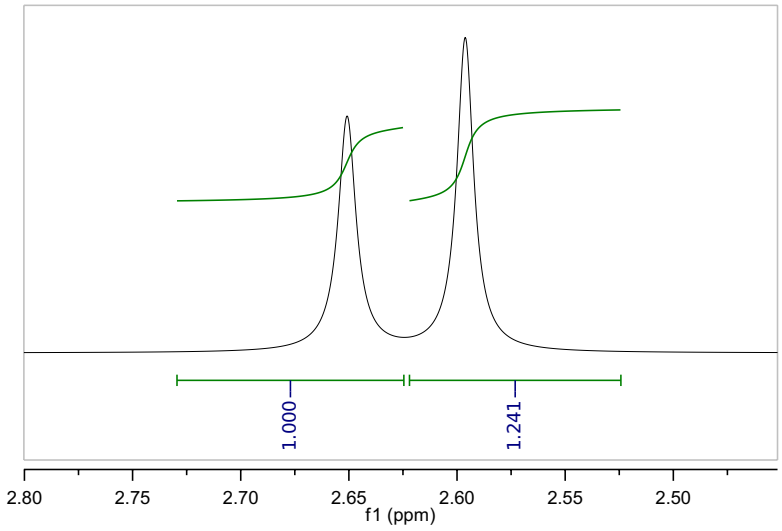


Figure S32: Part of the ^{1}H-NMR spectrum of product of decyl(methyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 6, Table 1, main text).

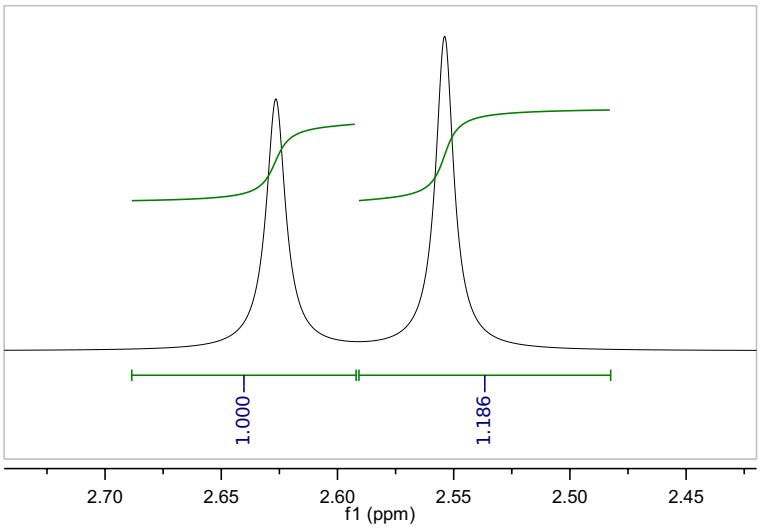


Figure S33: Part of the ^{1}H-NMR spectrum of product of decyl(methyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 7, Table 1, main text).

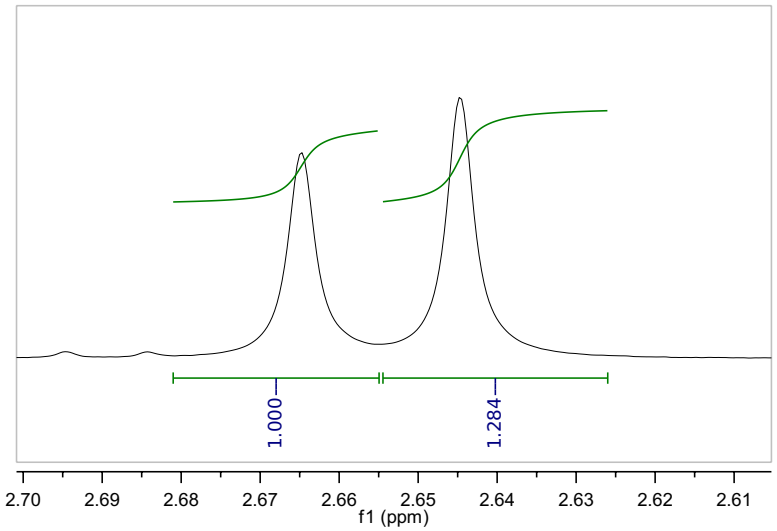


Figure S34: Part of the ^{1}H-NMR spectrum of product of dodecyl(methyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 8, Table 1, main text).

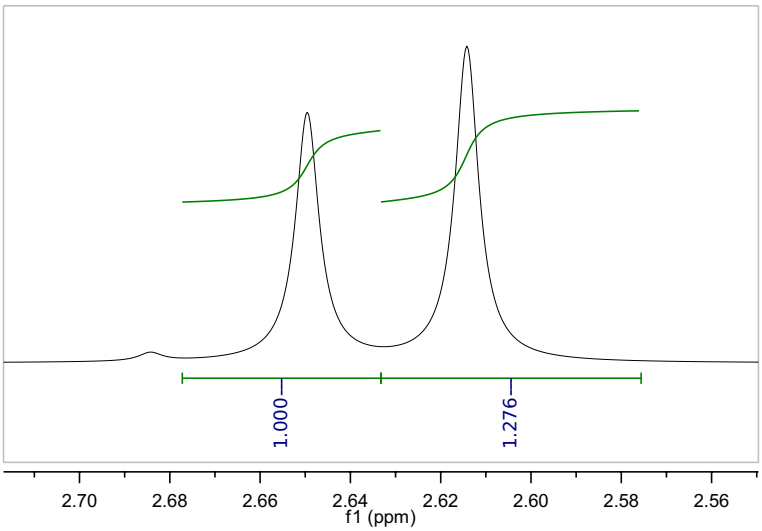


Figure S35: Part of the ^{1}H-NMR spectrum of product of dodecyl(methyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 9, Table 1, main text).

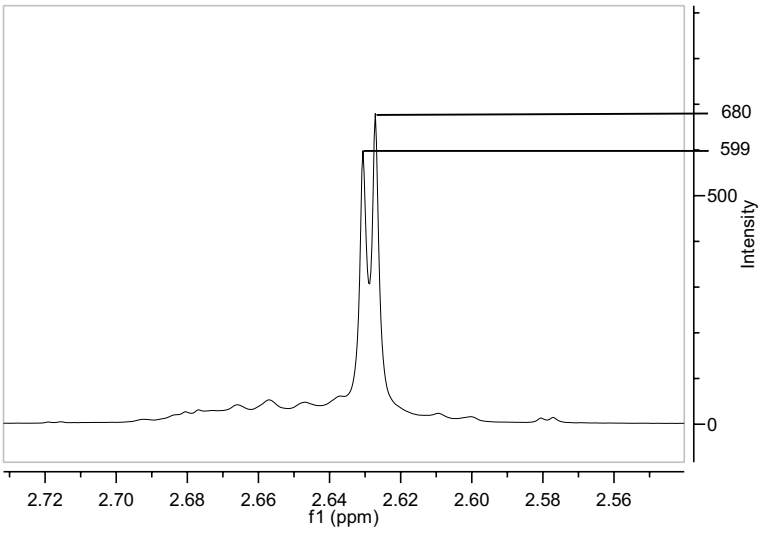
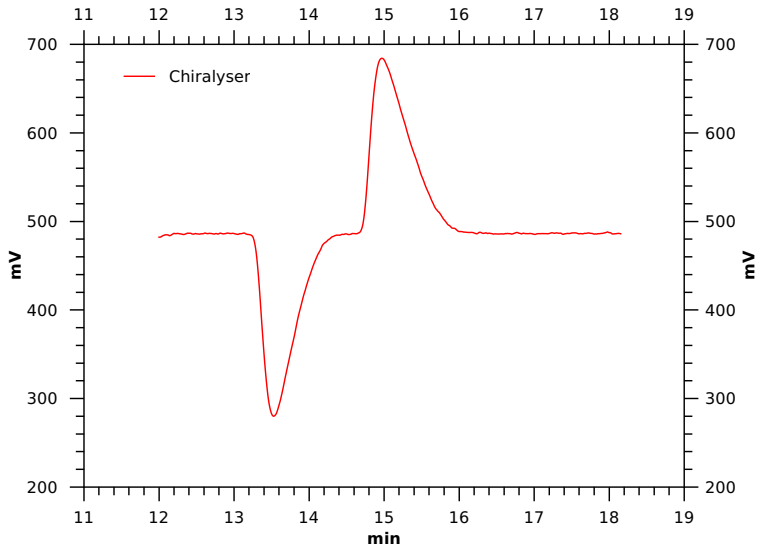


Figure S36: Part of the ^{1}H-NMR spectrum of product of cyclohexyl(methyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 11, Table 1, main text). Enantiomeric excess was estimated from the differences of peak's height.



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	13.527	13.133	14.440	45.9	0.47
2	14.967	14.533	16.453	54.1	0.57

Figure S37: Part of the HPLC chromatogram of product of cyclohexyl(methyl)sulfide oxidation catalyzed by **1a** (1 mol%), (Entry 12, Table 1, main text). Conditions: **Phenomenex® Lux 5u Amylose-2** column, heptane/i-PrOH (17:3), 1 mL/min.

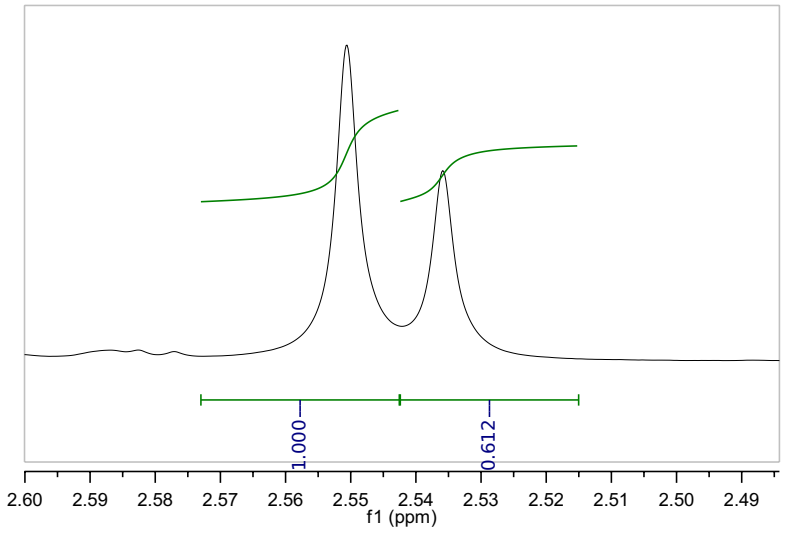


Figure S38: Part of the ^{1}H-NMR spectrum of product of hexyl(methyl)sulfide oxidation catalyzed by **2a** (1 mol%), (Entry 1, Table 2, main text).

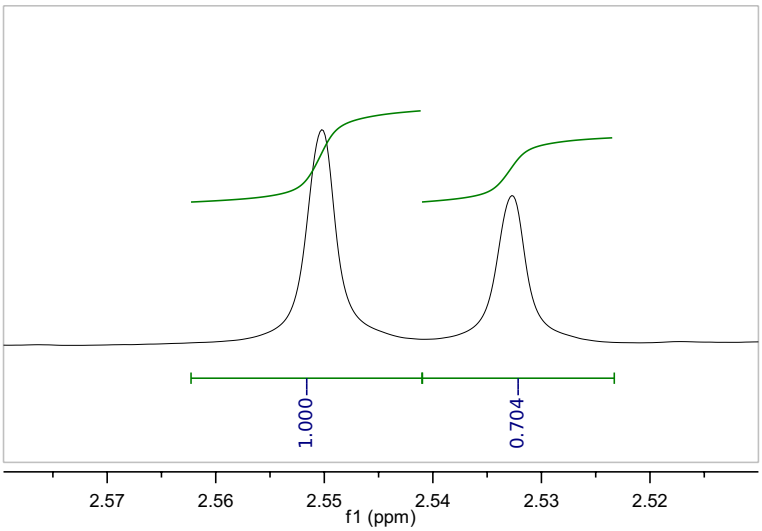


Figure S39: Part of the ^{1}H-NMR spectrum of product of methyl(octyl)sulfide oxidation catalyzed by **2a** (1 mol%), (Entry 2, Table 2, main text).

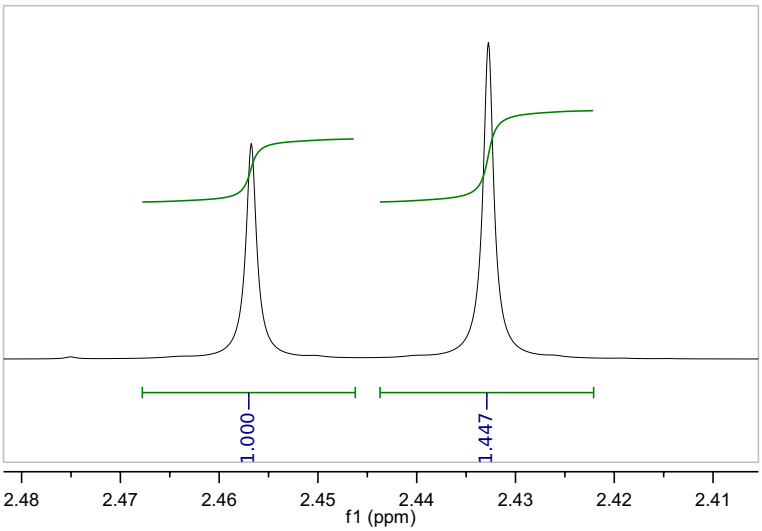
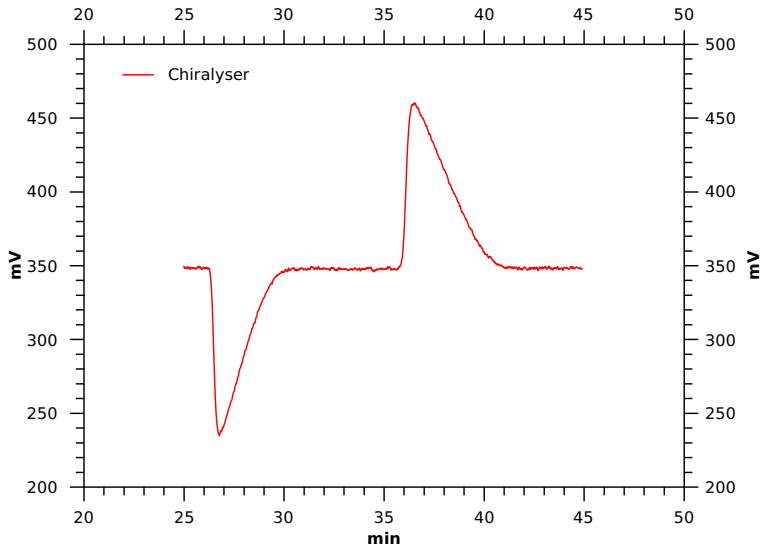


Figure S40: Part of the ^{1}H-NMR spectrum of product of methyl(octyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 2, Table 3, main text).



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	26.760	25.733	30.220	41.6	1.57
2	36.487	35.353	41.680	58.4	2.27

Figure S41: Part of the HPLC chromatogram of product of methyl(octyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 2, Table 3, main text). Conditions: **Daicel® Chiralpak AS-H** column, heptane/i-PrOH (17:3), 1 mL/min.

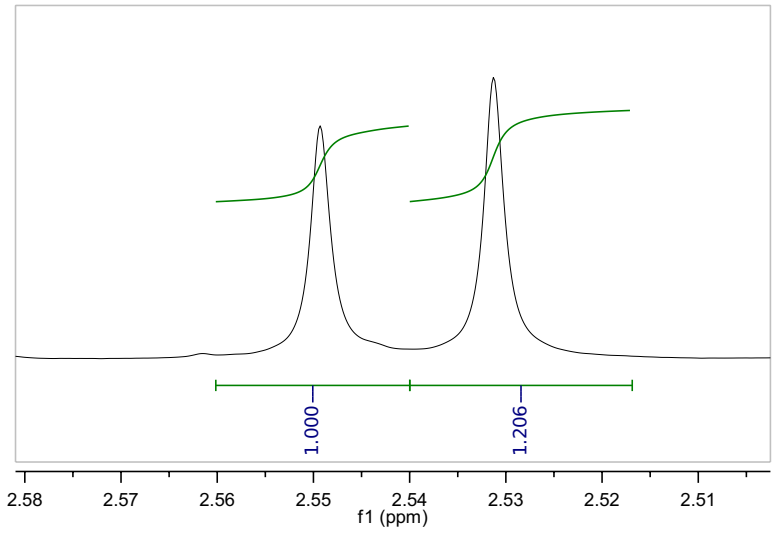
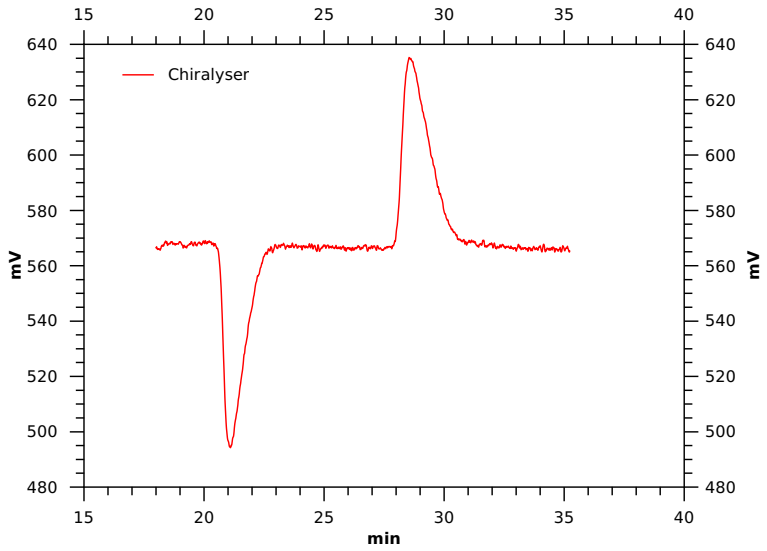


Figure S42: Part of the ^{1}H-NMR spectrum of product of decyl(methyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 3, Table 3, main text).



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	21.100	20.213	22.960	44.9	0.94
2	28.540	27.427	31.433	55.1	1.23

Figure S43: Part of the HPLC chromatogram of product of decyl(methyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 3, Table 3, main text). Conditions: **Daicel® Chiralpak AS-H** column, heptane/i-PrOH (17:3), 1 mL/min.

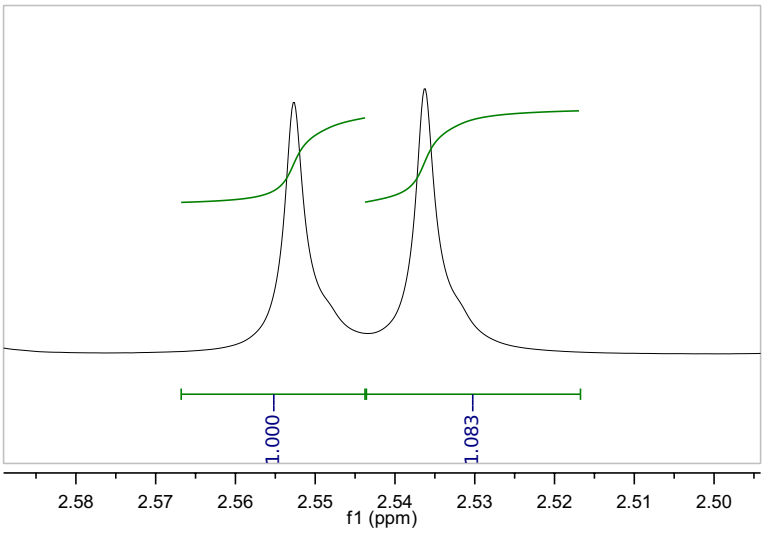
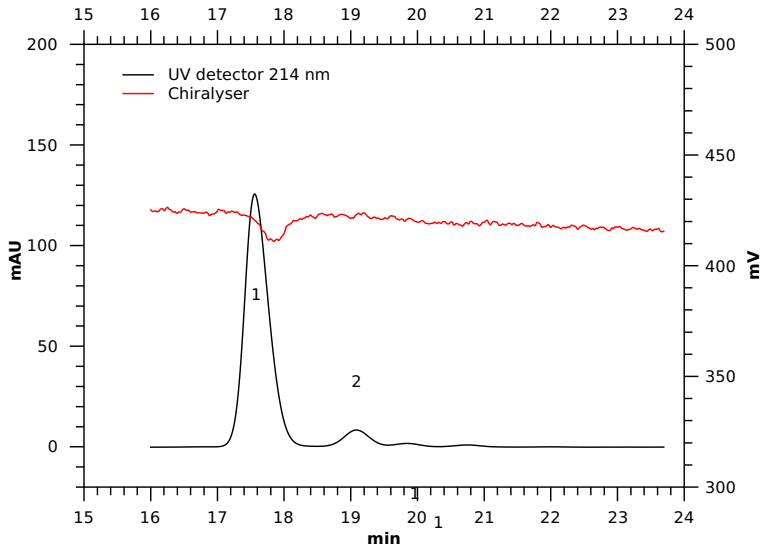
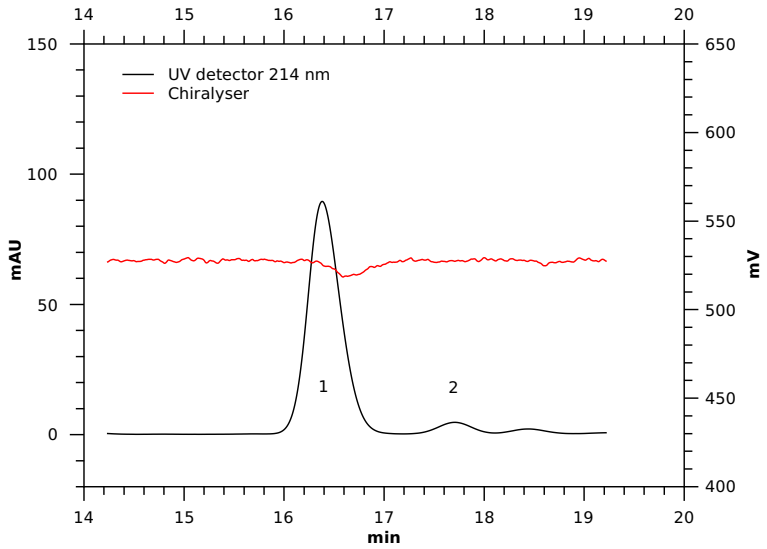


Figure S44: Part of the ^{1}H-NMR spectrum of product of dodecyl(methyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 4, Table 3, main text).



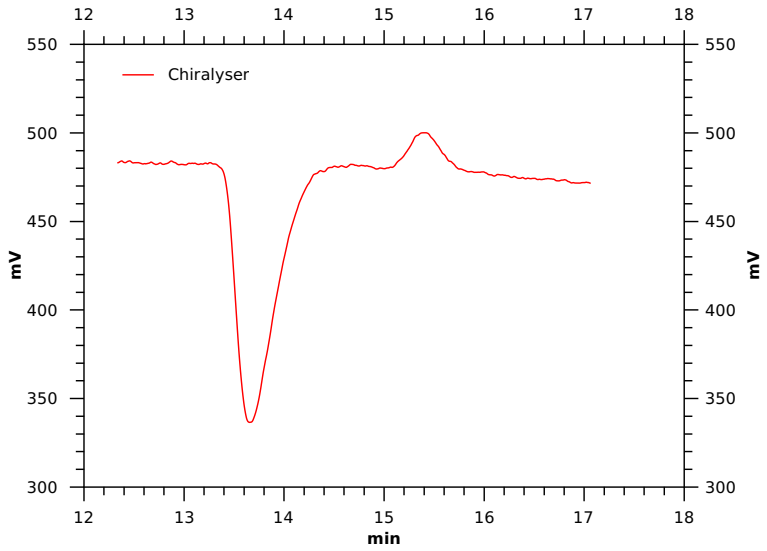
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	17.560	17.000	18.487	94.2	0.40
2	19.080	18.527	19.567	5.8	0.41

Figure S45: Part of the HPLC chromatogram of product of *tert*-butyl(methyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 5, Table 3, main text). Conditions: **Phenomenex® Lux 5u Cellulose-4** column, heptane/i-PrOH (9:1), 1 mL/min.



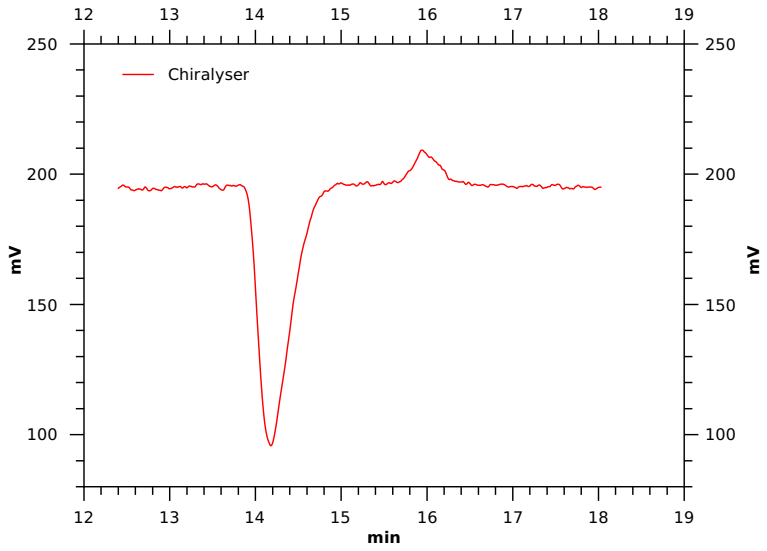
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	16.380	15.487	17.193	95.6	0.37
2	17.707	17.207	18.113	4.4	0.37

Figure S46: Part of the HPLC chromatogram of product of *tert*-butyl(methyl)sulfide oxidation catalyzed by **1b** (5 mol%), (Entry 6, Table 3, main text). Conditions: **Phenomenex® Lux 5u Cellulose-4** column, heptane/i-PrOH (9:1), 1 mL/min.



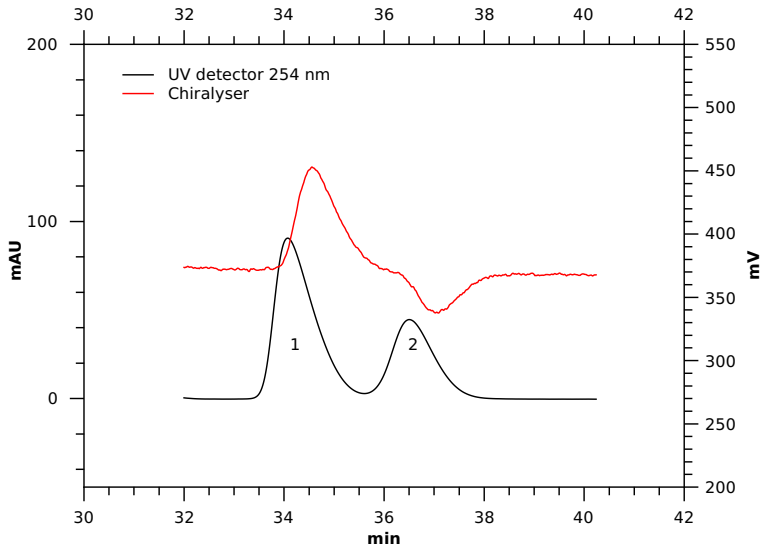
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	13.653	13.260	14.573	89.2	0.43
2	15.407	15.000	15.900	10.8	0.35

Figure S47: Part of the HPLC chromatogram of product of cyclohexyl(methyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 7, Table 3, main text). Conditions: **Phenomenex® Lux 5u Amylose-2** column, heptane/i-PrOH (17:3), 1 mL/min.



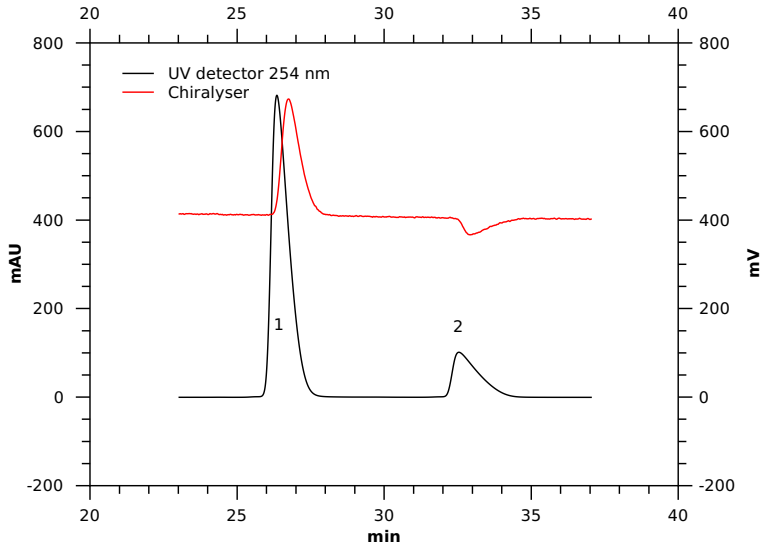
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	14.180	13.820	14.993	89.8	0.41
2	15.940	15.520	16.520	10.2	0.34

Figure S48: Part of the HPLC chromatogram of product of cyclohexyl(methyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 8, Table 3, main text). Conditions: **Phenomenex® Lux 5u Amylose-2** column, heptane/i-PrOH (17:3), 1 mL/min.



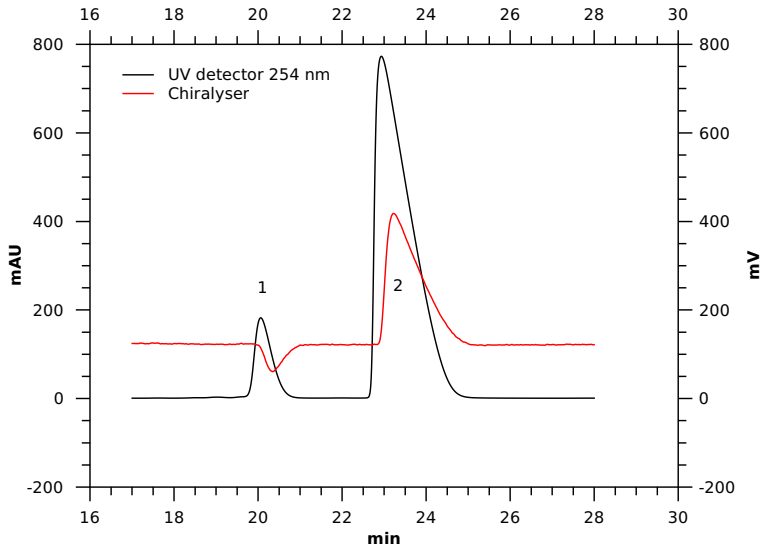
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	34.073	33.307	35.607	65.2	0.87
2	36.500	35.607	38.660	34.8	0.93

Figure S49: Part of the HPLC chromatogram of product of benzyl(methyl)sulfide oxidation catalyzed by **1b** (1 mol%), (Entry 9, Table 3, main text). Conditions: **Knauer® Eurocel 01 5μm** column, heptane/i-PrOH (17:3), 0.5 mL/min.



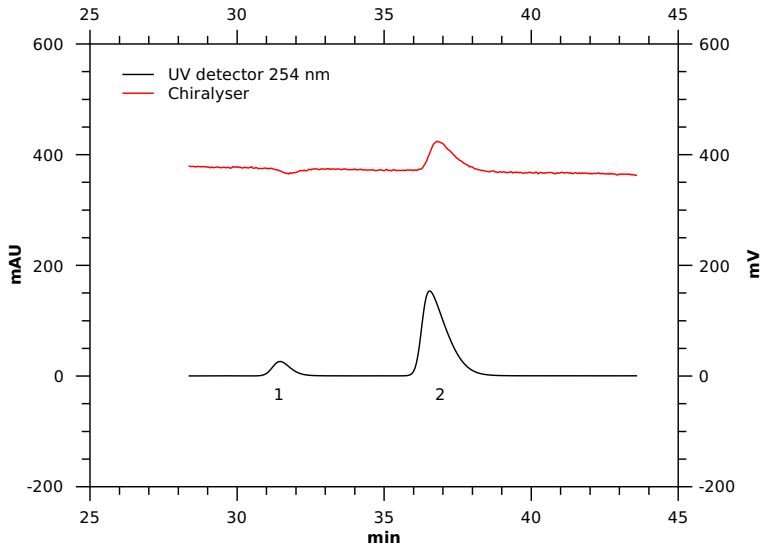
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	26.353	25.720	28.160	82.1	0.65
2	32.540	31.953	34.787	17.9	0.96

Figure S50: Part of the HPLC chromatogram of product of thioanisole oxidation catalyzed by **1b** (1 mol%), (Entry 11, Table 3, main text). Conditions: **Knauer® Eurocel 01 5μm** column, heptane/i-PrOH (9:1), 0.65 mL/min.



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	20.060	19.347	21.213	10.3	0.45
2	22.933	22.433	25.860	89.7	0.93

Figure S51: Part of the HPLC chromatogram of product of 4-methylthioanisole oxidation catalyzed by **1b** (1 mol%), (Entry 13, Table 3, main text). Conditions: **Phenomenex® Lux 5u Amylose-2** column, heptane/i-PrOH (17:3), 1 mL/min.



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	31.460	30.713	33.273	11.1	0.69
2	36.547	35.613	39.453	88.9	0.92

Figure S52: Part of the HPLC chromatogram of product of 4-methylthioanisole oxidation catalyzed by **1b** (0.3 mol%), (Entry 14, Table 3, main text). Conditions: **Phenomenex® Lux 5u Amylose-2** column, heptane/i-PrOH (9:1), 1 mL/min.

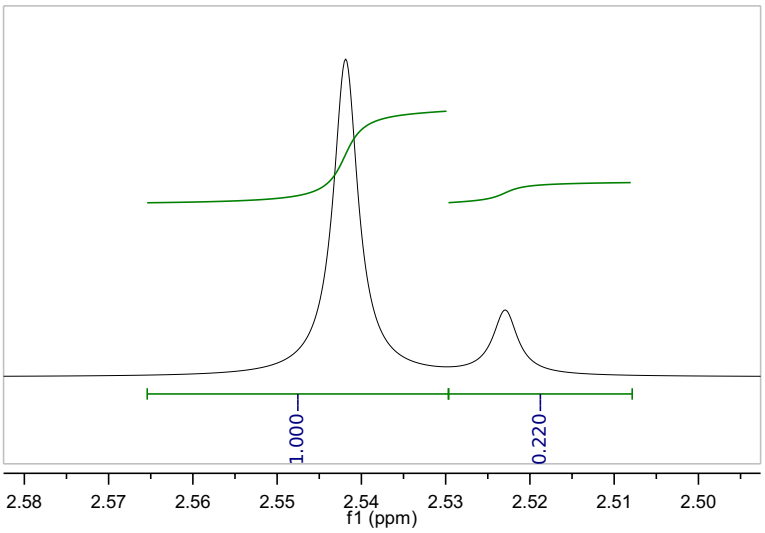
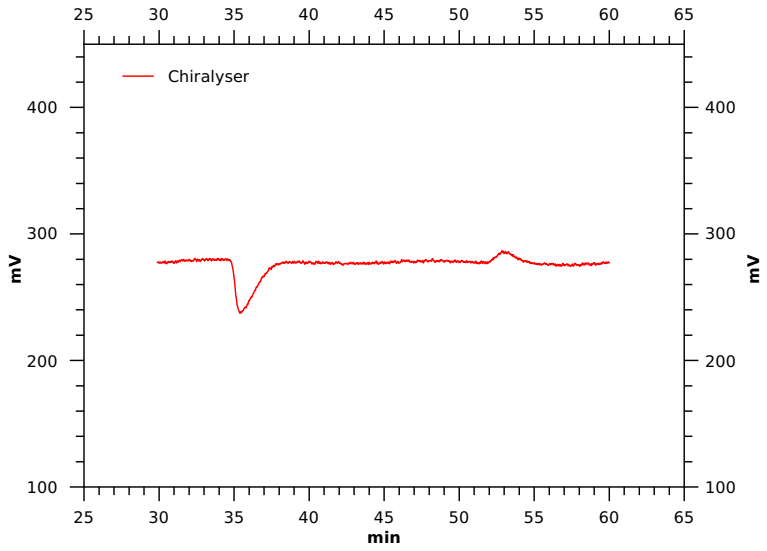
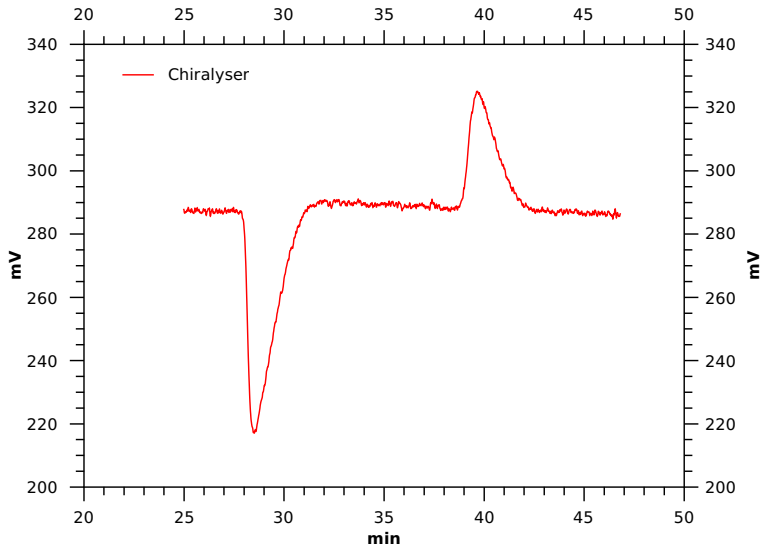


Figure S53: Part of the ^{1}H-NMR spectrum of product of hexyl(octyl)sulfide oxidation catalyzed by **2b** (1 mol%), (Entry 1, Table 4, main text).



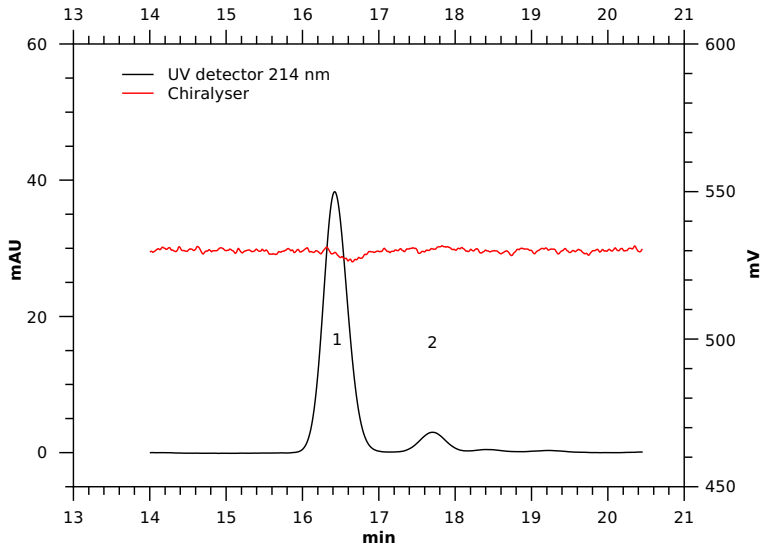
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	35.380	34.440	38.487	80.4	1.51
2	52.853	51.847	55.467	19.6	1.31

Figure S54: Part of the HPLC chromatogram of product of hexyl(octyl)sulfide oxidation catalyzed by **2b** (1 mol%), (Entry 1, Table 4, main text). Conditions: **Daicel® Chiralpak AS-H** column, heptane/i-PrOH (17:3), 1 mL/min.



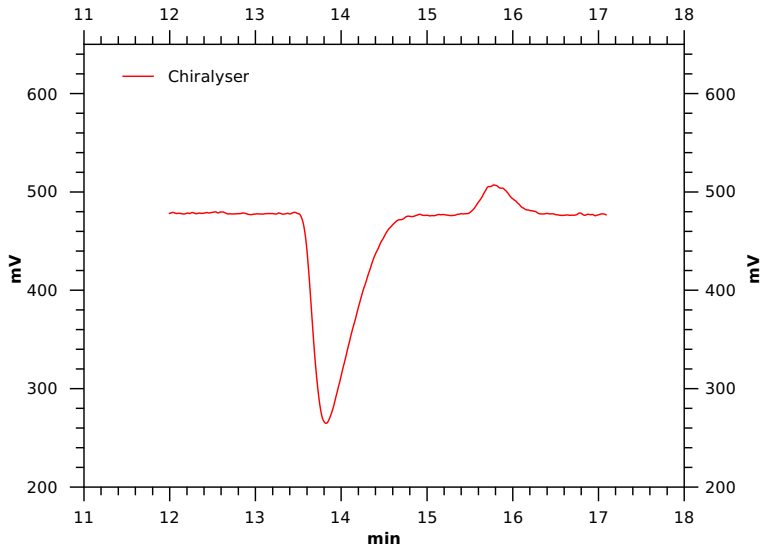
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	28.513	27.467	32.033	64.3	1.45
2	39.653	38.307	42.553	35.7	1.52

Figure S55: Part of the HPLC chromatogram of product of methyl(octyl)sulfide oxidation catalyzed by **2b** (1 mol%), (Entry 2, Table 4, main text). Conditions: **Daicel® Chiralpak AS-H** column, heptane/i-PrOH (17:3), 1 mL/min.



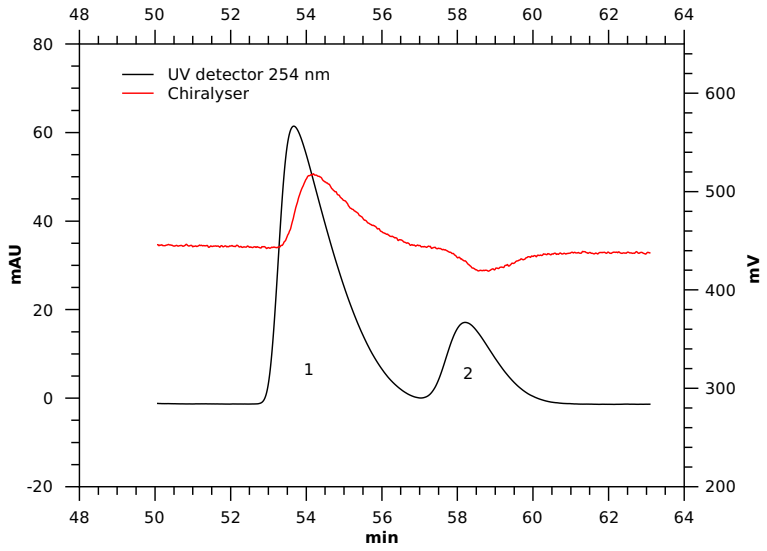
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	16.420	15.913	17.127	93	0.37
2	17.700	17.193	18.160	7	0.38

Figure S56: Part of the HPLC chromatogram of product of *tert*-butyl(methyl)sulfide oxidation catalyzed by **2b** (1 mol%), (Entry 5, Table 4, main text). Conditions: **Phenomenex® Lux 5u Cellulose-4** column, heptane/i-PrOH (9:1), 1 mL/min.



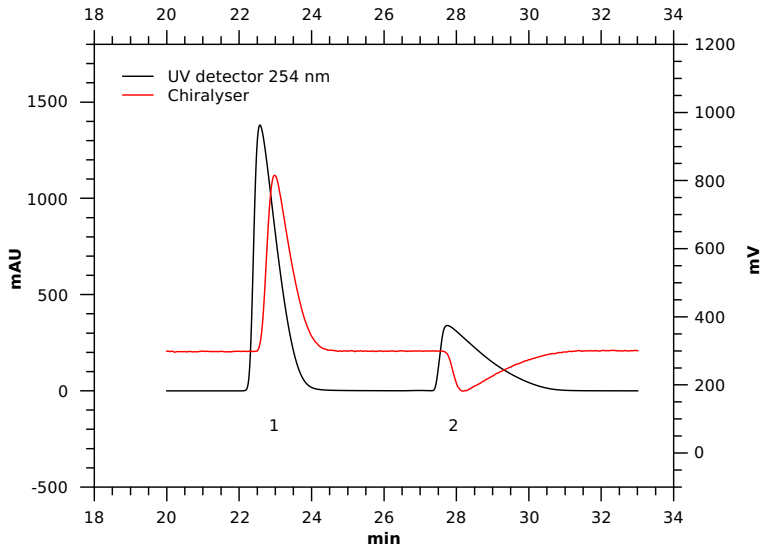
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	13.820	13.453	15.140	89.8	0.51
2	15.780	15.373	16.567	10.2	0.40

Figure S57: Part of the HPLC chromatogram of product of cyclohexyl(methyl)sulfide oxidation catalyzed by **2b** (1 mol%), (Entry 6, Table 4, main text). Conditions: **Phenomenex® Lux 5u Amylose-2** column, heptane/i-PrOH (17:3), 1 mL/min.



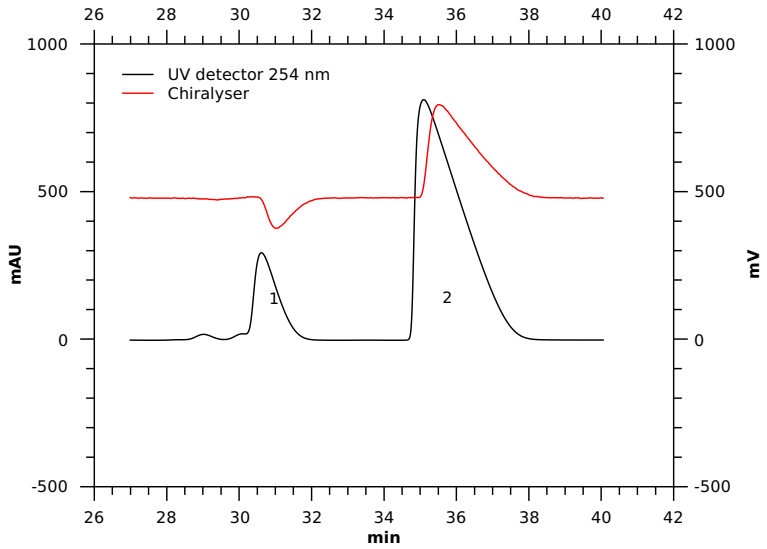
	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	53.667	52.767	57.033	79.1	1.57
2	58.200	57.033	60.993	20.9	1.43

Figure S58: Part of the HPLC chromatogram of product of benzyl(methyl)sulfide oxidation catalyzed by **2b** (1 mol%), (Entry 7, Table 4, main text). Conditions: **Knauer® Eurocel 01 5μm** column, heptane/i-PrOH (9:1), 0.65 mL/min.



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	22.573	22.033	24.800	67.8	0.70
2	27.747	27.300	31.373	32.2	1.32

Figure S59: Part of the HPLC chromatogram of product of thioanisole oxidation catalyzed by **2b** (1 mol%), (Entry 8, Table 4, main text). Conditions: **Knauer® Eurocel 01 5μm** column, heptane/i-PrOH (9:1), 0.65 mL/min.



	Reten. Time [min]	Start Time [min]	End Time [min]	Area [%]	W05 [min]
1	30.613	30.167	32.040	15.5	0.71
2	35.093	34.473	39.087	84.5	1.44

Figure S60: Part of the HPLC chromatogram of product of 4-methylthioanisole oxidation catalyzed by **2b** (1 mol%), (Entry 9, Table 4, main text). Conditions: **Phenomenex® Lux 5u Amylose-2** column, heptane/i-PrOH (9:1), 0.65 mL/min.

References

[1] V. Mojr, V. Herzig, M. Buděšínský, R. Cibulka and T. Kraus, *Chem. Commun.*, 2010, **46**, 7599–7601.

[2] M. Deshmukh, E. Duñach, S. Juge and H. B. Kagan, *Tetrahedron Letters*, 1984, **25**, 3467–3470.