Supplementary Information

NMR and LC-HR-MS Data of Protected Intermediates:

NMR Data of 2,2,2-Trichloroethyl-DOM-3-Sulfate:

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.57 (dq, *J* = 5.8, 1.6 Hz, 1H), 5.31 (s, 1H), 5.18 (s, 1H), 5.07 (dt, *J* = 11.9, 4.6 Hz, 1H), 4.82 (d, *J* = 10.9 Hz, 1H), 4.79 (d, *J* = 10.9 Hz, 1H), 4.68 (d, *J* = 5.9 Hz, 1H), 4.66 (s, 1H), 4.57 (d, *J* = 4.3 Hz, 1H), 3.80 (s, *2*H), 3.75 (s, 1H), 2.75 (dd, *J* = 15.6, 4.3 Hz, 1H), 2.09 (dd, *J* = 15.6, 11.6 Hz, 1H), 1.96 (br, 1H) 1.88 (s, 3H), 1.46 (s, 3H);
^{13}C-NMR (100 MHz, $CDCl_3$) δ 200.3 (s, 1C), 149.7 (s, 1C), 138.2 (d, 1C), 136.1 (s, 1C), 110 (t, 1C), 92.8 (s, 1C), 81.0 (d, 1C), 79.9 (t, 1C), 79.1 (d, 1C), 74.4 (d, 1C), 70.1 (d, 1C), 62.0 (t, 1C), 52.3 (s, 1C), 48.6 (s, 1C), 40.8 (t, 1C), 19.6 (q, 1C), 15.5 (q, 1C); LC-HR-MS *m/z* calcd for $C_{17}H_{20}O_8SCl_3^-$ $[M - H^+]^-$, 488.9950, found 488.9948.

NMR data of 2,2,2-Trichloroethyl-DOM-15-Sulfate:

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.67 (dq, *J* = 5.9, 1.6 Hz, 1H), 5.26 (s, 1H), 5.10 (s, 1H), 4.94 (d, *J* = 5.9 Hz, 1H), 4.72 (s, 1H), 4.62 (d, *J* = 10.1 Hz, 1H), 4.61 (d *J* = 10.9 Hz, 1H), 4.56 (d, *J* = 10.9 Hz; 1H), 4.45 (d, *J* = 10.5 Hz, 1H), 4.30 (dt, *J* = 10.7, 4.2 Hz, 1H), 4.26 (d, *J* = 4.3 Hz, 1H), 3.78 (s, 1H), 2.20 (dd, *J* = 15.0, 4.1 Hz, 1H), 1.94–2.06 (m, 2H), 1.89 (s, 3H), 1.37 (s, 3H);
^{13}C-NMR (100 MHz, $CDCl_3$) δ 199.9 (s, 1C), 151.1 (s, 1C), 139.1 (d, 1C), 136.0 (s, 1C), 109.8 (t, 1C), 92.6 (s, 1C), 81.2 (d, 1C), 79.8 (t, 1C), 73.1 (d, 1C), 72.2 (t, 1C), 69.5 (d, 1C), 69.3 (d, 1C), 51.8 (s, 1C), 49.0 (s, 1C), 44.3 (t, 1C), 19.4 (q, 1C), 15.5 (q, 1C); LC-HR-MS *m/z* calcd for $C_{17}H_{20}O_8SCl_3^-$ $[M - H^+]^-$, 488.9950, found 488.9942.

NMR Data of Bis (2,2,2-Trichloroethyl) DOM-3,15-Disulfate:

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.65 (dq, *J* = 5.6, 1.6 Hz, 1H), 5.39 (s, 1H), 5.24 (s, 1H), 5.09 (dt, *J* = 11.5, 4.4 Hz, 1H), 4.81 (s, 2H), 4.77 (d, *J* = 5.9 Hz, 1H), 4.70 (s, 1H), 4.61–4.66 (m, *2*H), 4.59 (d, *J* = 10.5 Hz, 1H), 4.54 (d, *J* = 10.5 Hz, 1H), 4.47 (d, *J* = 10.5 Hz, 1H), 3.76 (br, 1H), 2.58 (dd, *J* = 16.0, 4.3 Hz, 1H), 2.18 (dd, *J* = 15.6, 11.3 Hz, 1H), 1.91 (s, 3H), 1.43 (s, 3H);
^{13}C-NMR (100 MHz, $CDCl_3$) δ 199.3 (s, 1C), 148.3 (s, 1C), 138.3 (d, 1C), 136.4 (s, 1C), 112.0 (t, 1C), 92.7 (s, 1C), 92.5 (s, 1C), 80.2 (t, 1C), 80.0 (t, 1C), 79.8 (d, 1C), 78.9 (d, 1C), 73.0 (d, 1C), 71.8 (t, 1C), 69.3 (d, 1C), 51.6 (s, 1C), 48.4 (s, 1C), 40.9 (t, 1C), 19.1 (q, 1C), 15.4 (q, 1C); LC-HR-MS *m/z* calcd for $C_{19}H_{21}O_{11}S_2Cl_6^-$ $[M - H^+]^-$, 698.8662, found 698.8681.

NMR and LC-HR-MS Data of DOM-Sulfates:

NMR Data of DOM-3-Sulfate, Ammonium Salt:

^{1}H NMR (400 MHz, methanol-d_4) δ 6.59 (dq, *J* = 5.9, 1.6 Hz, 1H), 5.21 (s, 1H), 5.04 (s, 1H), 4.94–4.80 (m, 7H), 4.68 (dt, *J* = 11.3, 4.5 Hz, 1H), 4.60 (s, 1H), 4.41 (d, *J* = 4.3 Hz, 1H), 3.76 (d, *J* = 12.1 Hz, 1H), 3.72 (d, *J* = 12.1 Hz, 1H), 2.73 (dd, *J* = 15.0, 4.5 Hz, 1H), 1.93 (dd, *J* = 15.0, 11.5 Hz, 1H), 1.81 (q, *J* = 0.8 Hz, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, methanol-d_4) δ 202.3 (s, 1C), 153.9 (s, 1C), 139.6 (d, 1C), 136.9 (s, 1C), 108.3 (t, 1C), 81.5 (d, 1C), 75.7 (d, 1C), 75.6 (d, 1C), 71.7

(d, 1C), 61.7 (t, 1C), 54.1 (s, 1C), 49.5 (s, 1C), 43.3 (t, 1C), 20.1 (q, 1C), 15.4 (q, 1C); LC-HR-MS: *m/z* calculated for $C_{15}H_{19}O_8S^-$ $[M - H^+]^-$: 359.0806, *m/z* measured: 359.0807.

NMR Data of DOM-15-Sulfate, Ammonium Salt:

^{1}H NMR (400 MHz, methanol-*d*$_4$) δ 6.62 (dq, *J* = 6.0, 1.5 Hz, 1H), 5.16 (s, 1H), 5.02 (d, *J* = 6.2 Hz, 1H), 5.01 (s, 1H), 4.95–4.80 (s, NH_4^+, 2 x CH, H_2O), 4.70 (s, 1H), 4.20 (d, *J* = 10.5 Hz, 1H), 4.16 (s, 1H), 4.14 (dt, *J* = 15.2, 4.5 Hz, 1H), 4.01 (d, 10.9 Hz, 1H), 2.56 (dd, *J* = 14.6, 4.1 Hz, 1H), 1.88 (dd, *J* = 15.0, 10.7 Hz, 1H), 1.83 (s, 3H), 1.43 (s, 3H); ^{13}C NMR (100 MHz, methanol-*d*$_4$) δ 201.7 (s, 1C), 154.7 (s, 1C), 140.1 (d, 1C), 136.9 (s, 1C), 107.8 (t, 1C), 82.8 (d, 1C), 76.0 (d, 1C), 70.9 (d, 1C), 70.1 (d, 1C), 67.1 (t, 1C), 52.8 (s, 1C), 50.2 (s, 1C), 45.7 (t, 1C), 20.0 (q, 1C), 15.4 (q, 1C); LC-HR-MS: *m/z* calculated for $C_{15}H_{19}O_8S^-$ $[M - H^+]^-$: 359.0806, *m/z* measured: 359.0806.

NMR Data of DOM-3,15-Disulfate, Diammonium Salt:

^{1}H NMR (600 MHz, methanol-*d*$_4$) δ 6.64 (dq, *J* = 5.7, 1.5 Hz, 1H), 5.23 (s, 1H), 5.07 (s, 1H), 5.05 – 4.80 (m, 2 x NH_4^+ + 1H + H_2O), 4.72–4.60 (m, 3H), 4.45 (d, *J* = 4.4 Hz, 1H), 4.23 (d, *J* = 10.9 Hz, 1H), 4.03 (d, *J* = 10.9 Hz, 1H), 2.81 (dd, *J* = 15.3, 4.4 Hz, 1H), 1.99 (dd, *J* = 15.3, 11.4 Hz, 1H), 1.83 (s, 3H), 1.46 (s, 3H); ^{13}C NMR (150 MHz, methanol-*d*$_4$) δ 201.6 (s, 1C), 153.6 (s, 1C), 139.9 (d, 1C), 137.1 (s, 1C), 108.6 (t, 1C), 81.5 (d, 1C), 75.7 (d, 1C), 75.5 (d, 1C), 71.0 (d, 1C), 67.1 (t, 1C), 52.9 (s, 1C), 49.1 (s, 1C), 43.4 (t, 1C), 19.9 (q, 1C), 15.5 (q, 1C); LC-HR-MS: *m/z* calculated for $C_{15}H_{19}O_{11}S_2^-$ $[M - H^+]^-$: 439.0374, *m/z* measured: 439.0375.

Table S1. Matrix effects of the long gradient method. Dilution of extracts: 1 + 2, v + v.

	Average ± standard deviation (*n* = 3)									
	DON-3-sulfate	**DON-15-sulfate**	**DOM-3-sulfate**	**DOM-15-Sulfate**	**DON**	**DOM**	**DONS 1**	**DONS 2**	**DONS 3**	**DOMS 2**
Excreta of turkey	87 ± 3	95 ± 1	89 ± 5	103 ± 2	89 ± 1	90 ± 1	89 ± 4	105 ± 5	101 ± 5	108 ± 0
Excreta of broiler (1)	105 ± 2	135 ± 0	104 ± 1	114 ± 2	107 ± 2	107 ± 2	108 ± 1	119 ± 4	188 ± 4	117 ± 1

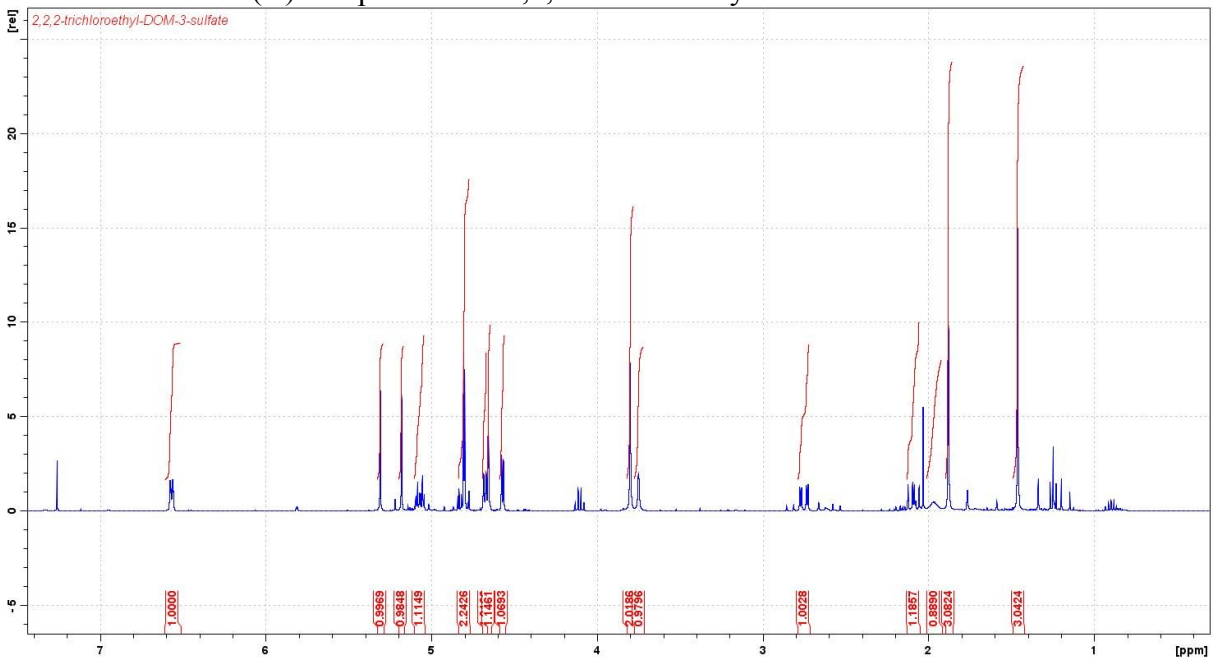
Table S2. Calculation of biological recoveries in the pullet experiment. -: no *A. galli* infection, +: *A.galli* infection.

	Amount of Lyophilized Excreta (g)	**µg Excreted Over 7 Days (in DON Equivalents)**						
		DON-3-sulfate	**DOM-3-sulfate**	**DON**	**DONS 2**	**SUM**	**µg DON Ingested**	**Biological Recovery (%)**
DON -	109 ± 3	2496 ± 104	379 ± 20	84 ± 3	16 ± 1	2975 ± 121	1973 ± 23	151 ± 5
DON +	110 ± 9	2227 ± 182	272 ± 113	85 ± 7	14 ± 5	2598 ± 214	1981 ± 0	131 ± 11

Table S3. Optimized SRM parameters.

Analyte	Retention Time (min)		Precursor Ion	Ion Spezies	DP	Product Ions	CE	Relative Intensity
	Short Method	Long Method	(m/z)		(V)	(quant/qual) (m/z)	(eV)	(qual/quant)
DONS 1	2.60	2.76	377.1	$[M-H]^-$	−130	80.0/331.0	−98/−52	0.25
DONS 2	3.40	3.81	377.1	$[M-H]^-$	−105	81.0/347.0	−68/−36	0.21
DONS 3	3.92	4.66	377.1	$[M-H]^-$	−125	347.0/80.0	−36/−98	0.29
DOMS 2	4.19	5.10	361.1	$[M-H]^-$	−25	81.0/249.0	−65/−30	0.51
DON-3-sulfate	4.43	5.58	375.1	$[M-H]^-$	−125	344.9/247.0	−36/−38	0.58
DON-15-sulfate	4.38	5.46	375.1	$[M-H]^-$	−110	97.0/229.0	−38/−42	0.20
DOM-3-sulfate	4.74	6.18	359.1	$[M-H]^-$	−125	97.0/329.0	−38/−34	0.65
DOM-15-sulfate	4.80	6.28	359.1	$[M-H]^-$	−125	97.0/80.0	−28/−118	0.26
DON	4.95	6.43	341.1	$[M+HCO_2]^-$	−25	265.1/45.0	−14/−38	1.01
DOM	5.44	7.50	325.1	$[M+HCO_2]^-$	−25	45.0/249.1	−40/−17	0.32

(**A**) ^{1}H spectrum of 2,2,2-trichloroethyl-DOM-3-sulfate



(**B**) ^{13}C spectrum of 2,2,2-trichloroethyl-DOM-3-sulfate

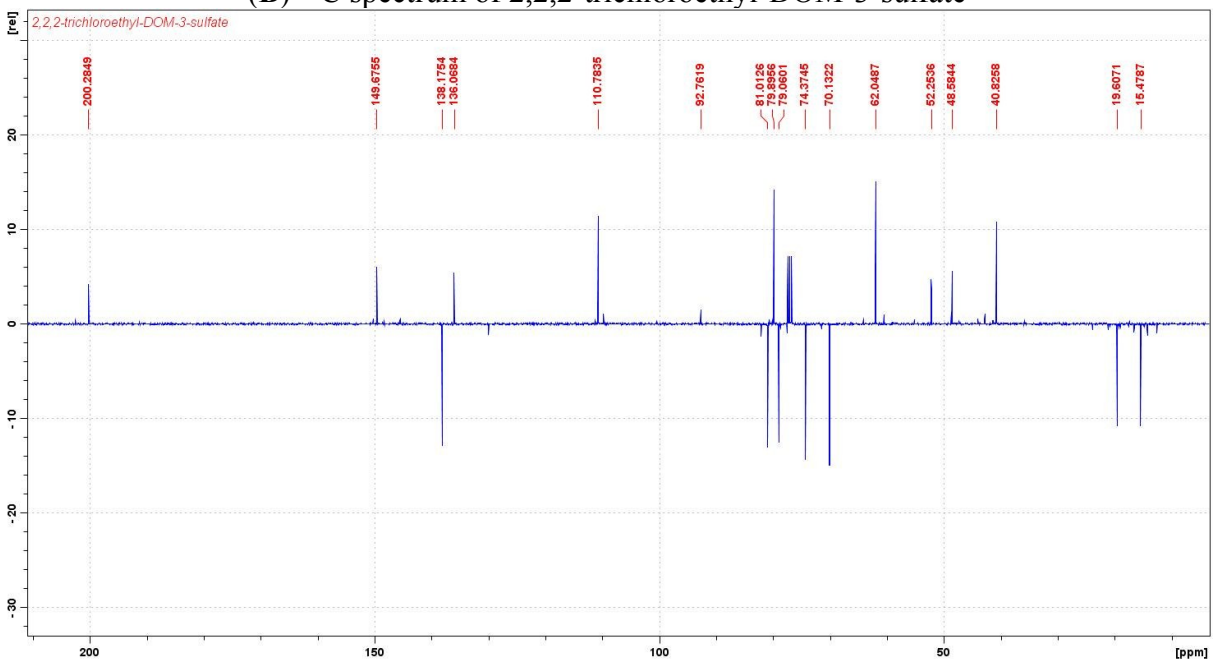
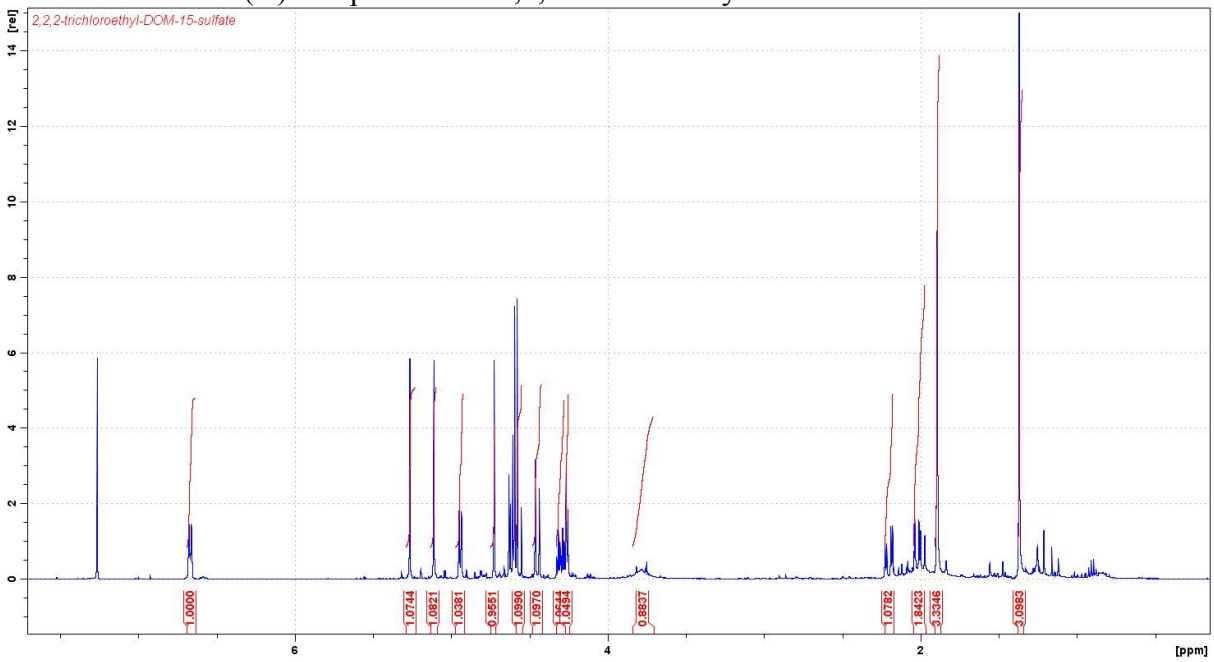


Figure S1. *Cont.*

(**C**) ^{1}H spectrum of 2,2,2-trichloroethyl-DOM-15-sulfate



(**D**) ^{13}C spectrum of 2,2,2-trichloroethyl-DOM-15-sulfate

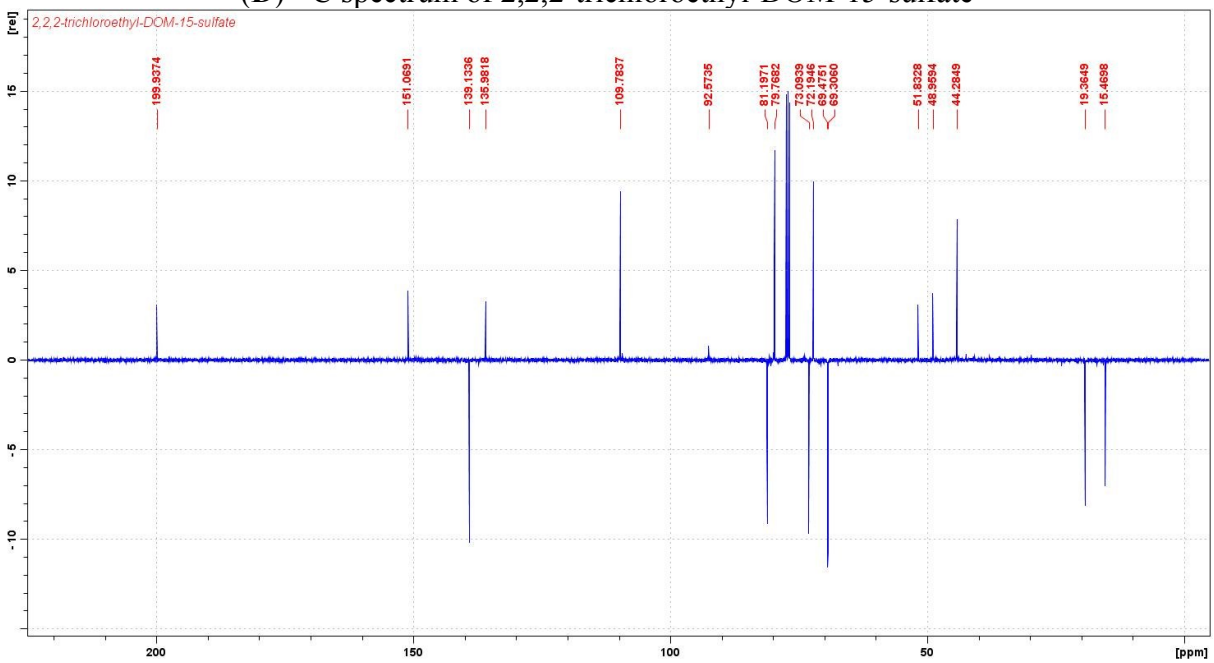
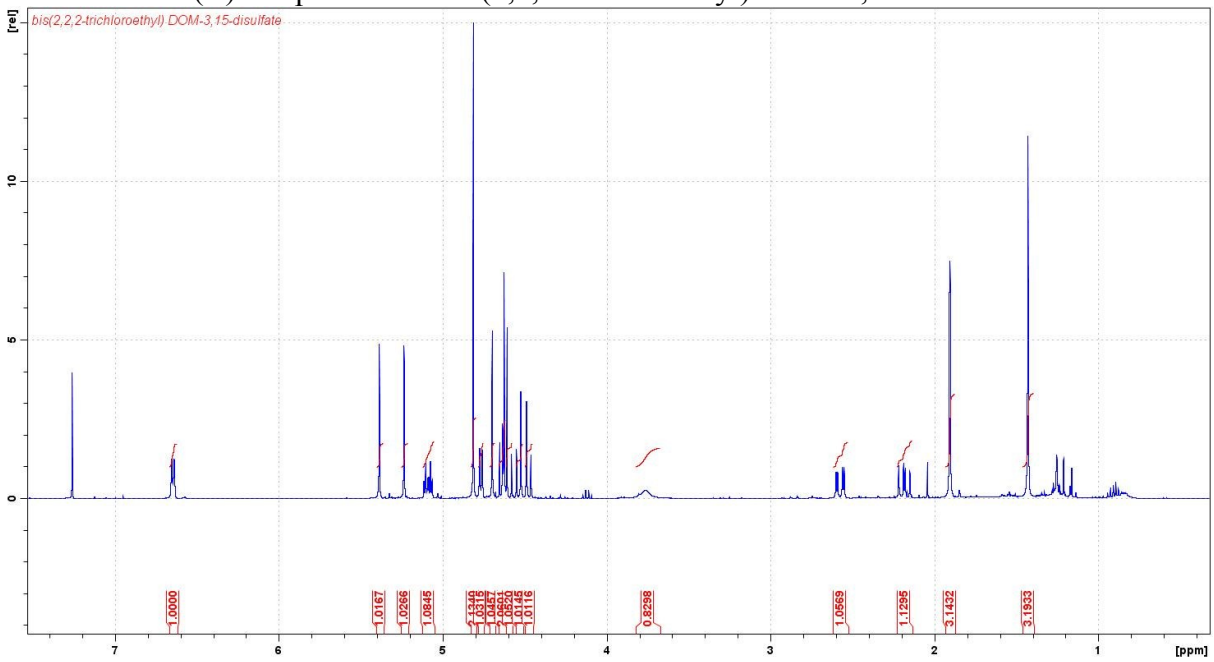


Figure S1. *Cont.*

(**E**) ^{1}H spectrum of bis(2,2,2-trichloroethyl) DOM-3,15-disulfate



(**F**) ^{13}C spectrum of bis(2,2,2-trichloroethyl) DOM-3,15-disulfate

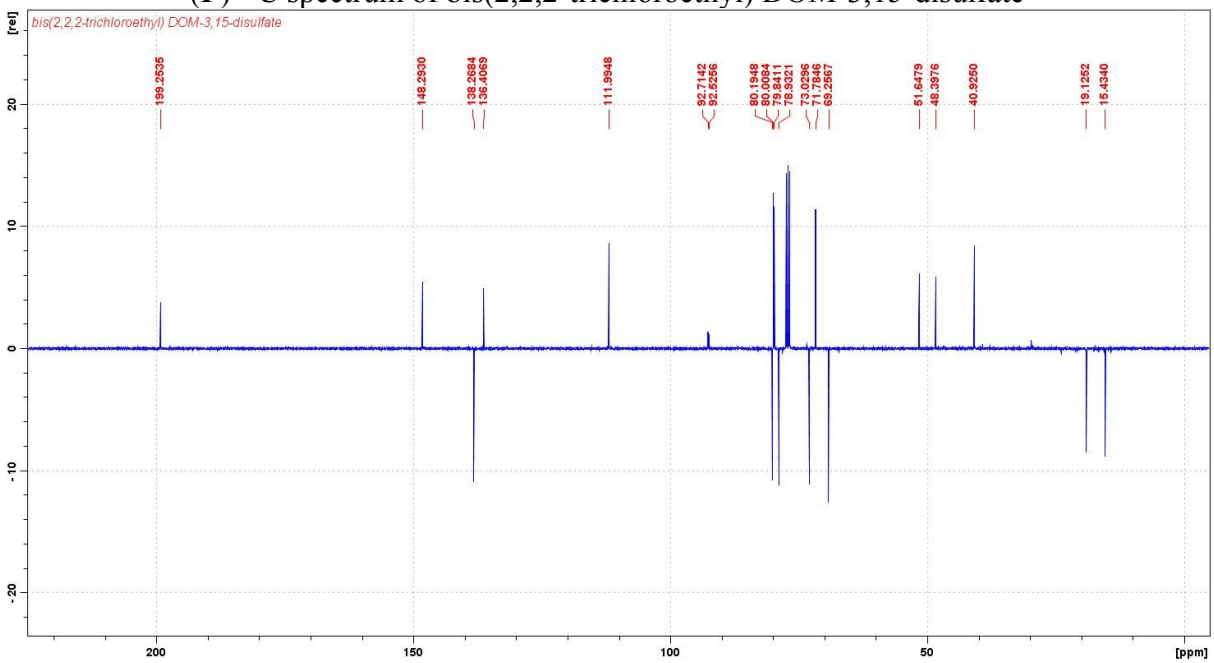
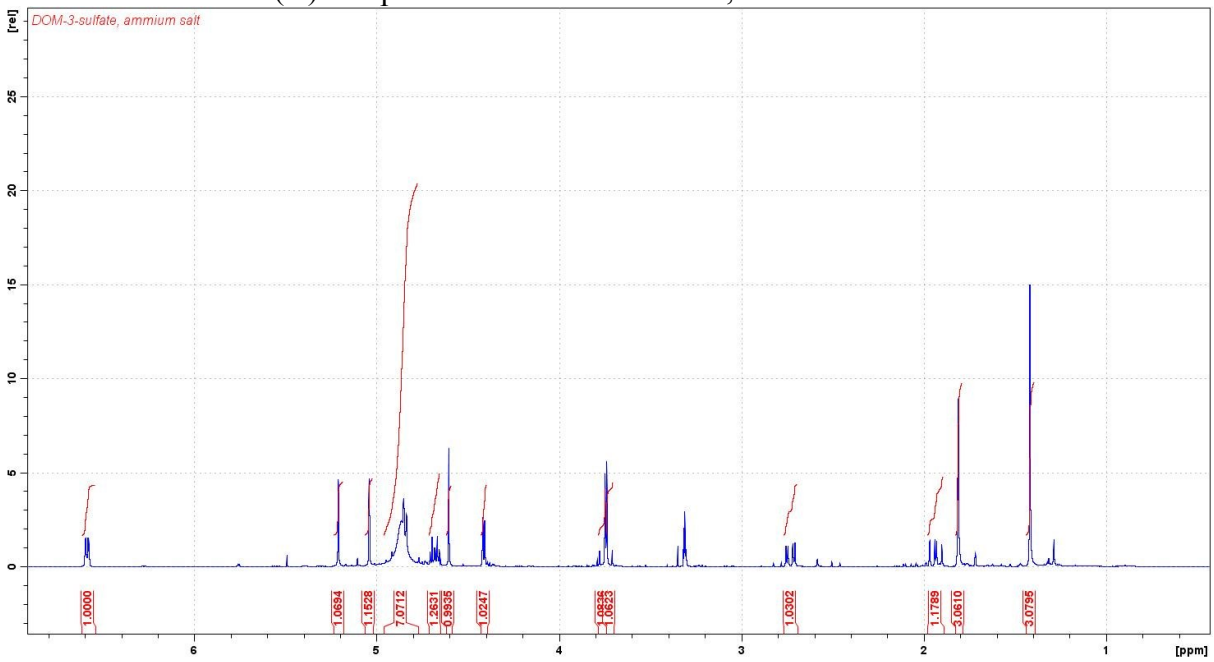


Figure S1. NMR spectra of protected intermediates.

(**A**) ^{1}H spectrum of DOM-3-sulfate, ammonium salt



(**B**) ^{13}C spectrum of DOM-3-sulfate, ammonium salt

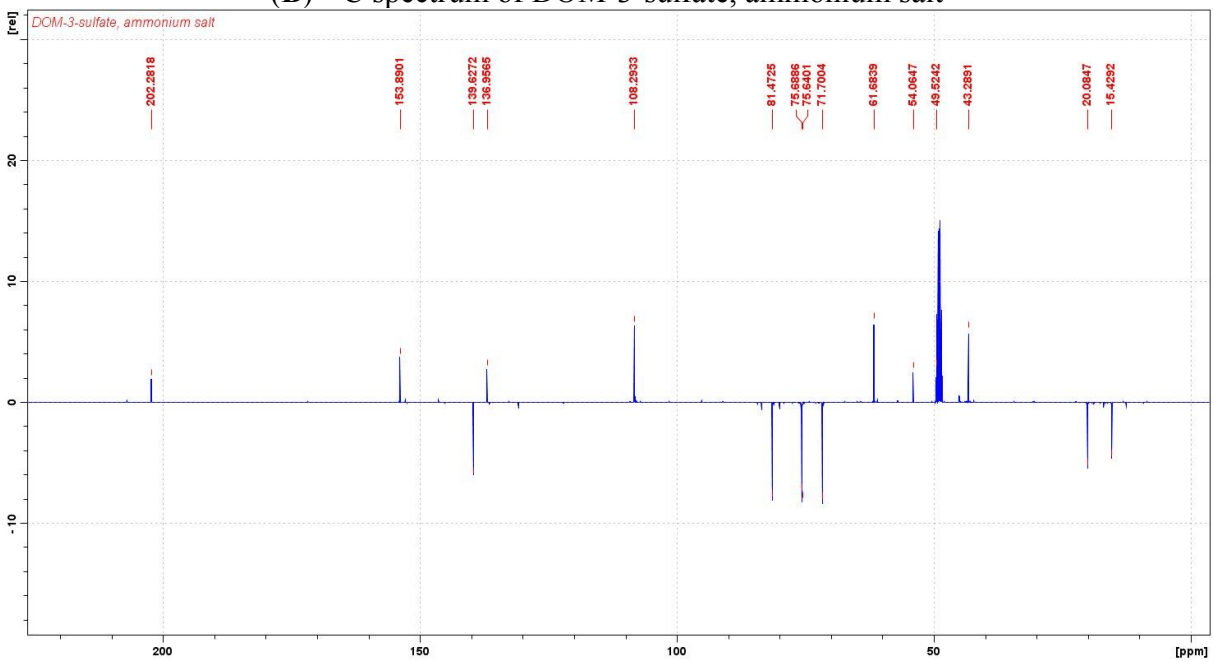
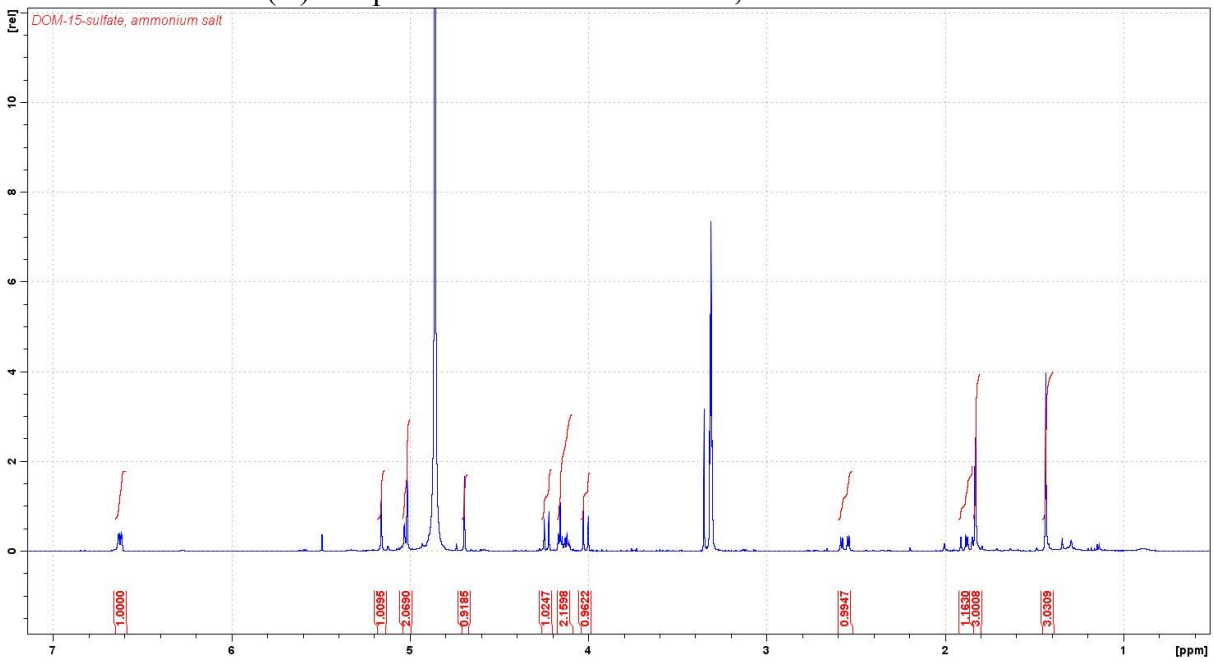


Figure S2. *Cont.*

(**C**) ^{1}H spectrum of DOM-15-sulfate, ammonium salt



(**D**) ^{13}C spectrum of DOM-15-sulfate, ammonium salt

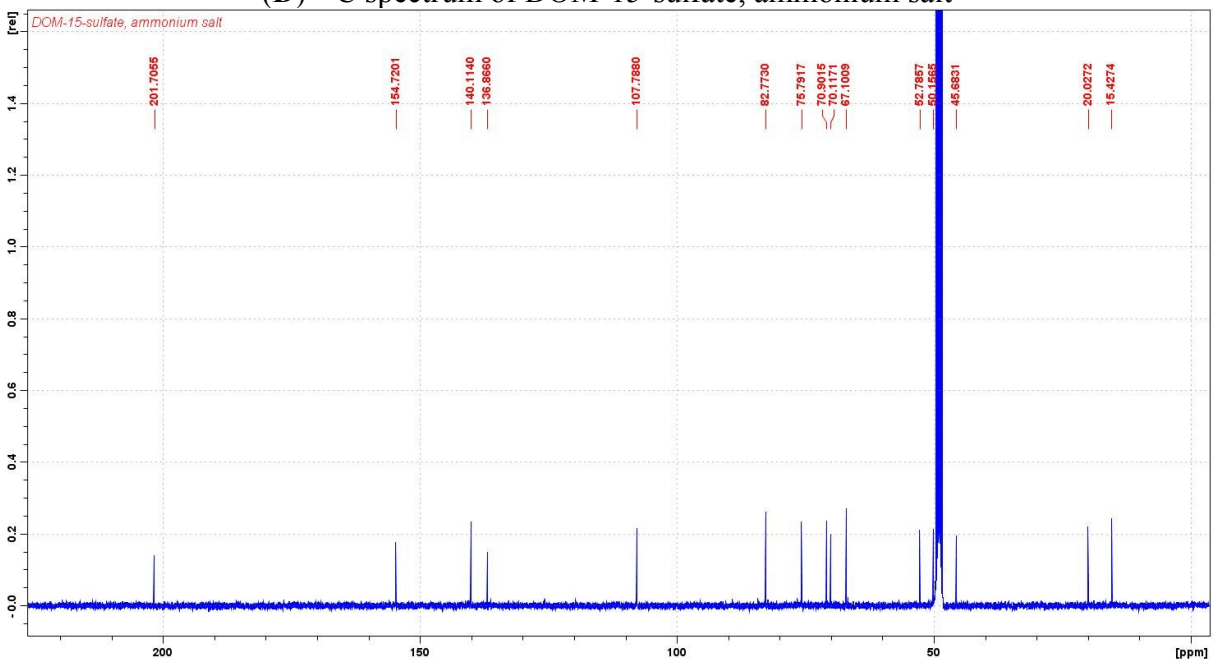
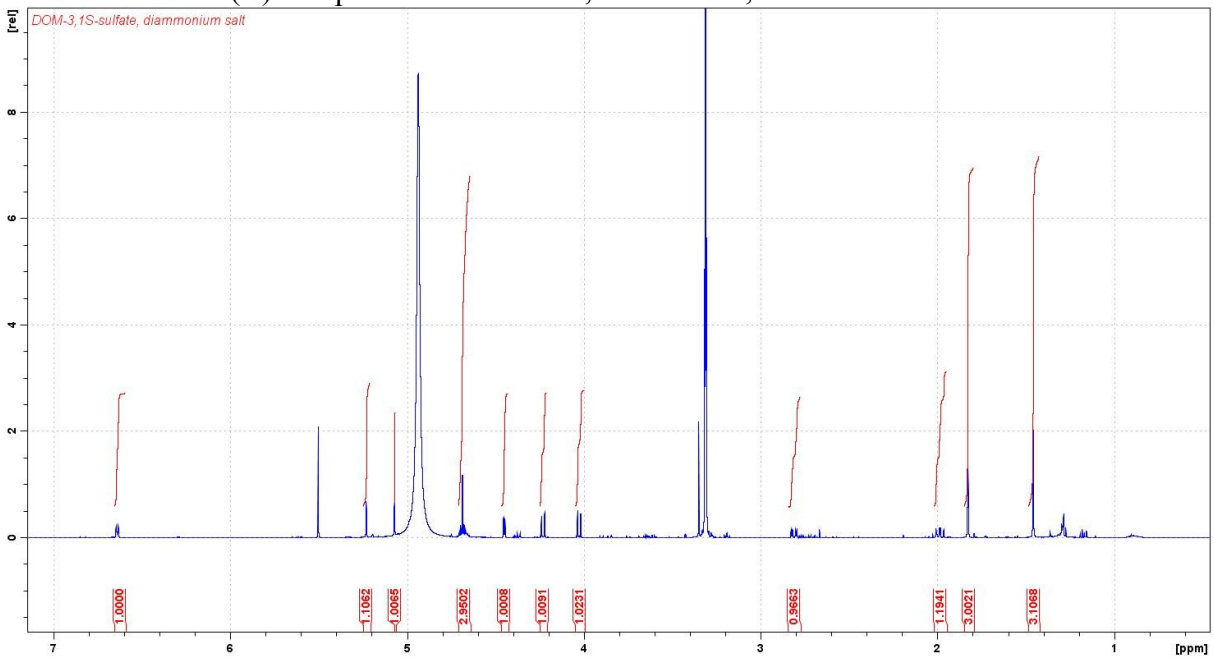


Figure S2. *Cont.*

(**E**) ^{1}H spectrum of DOM-3,15-disulfate, diammonium salt



(**F**) ^{13}C spectrum of DOM-3,15-disulfate, diammonium salt

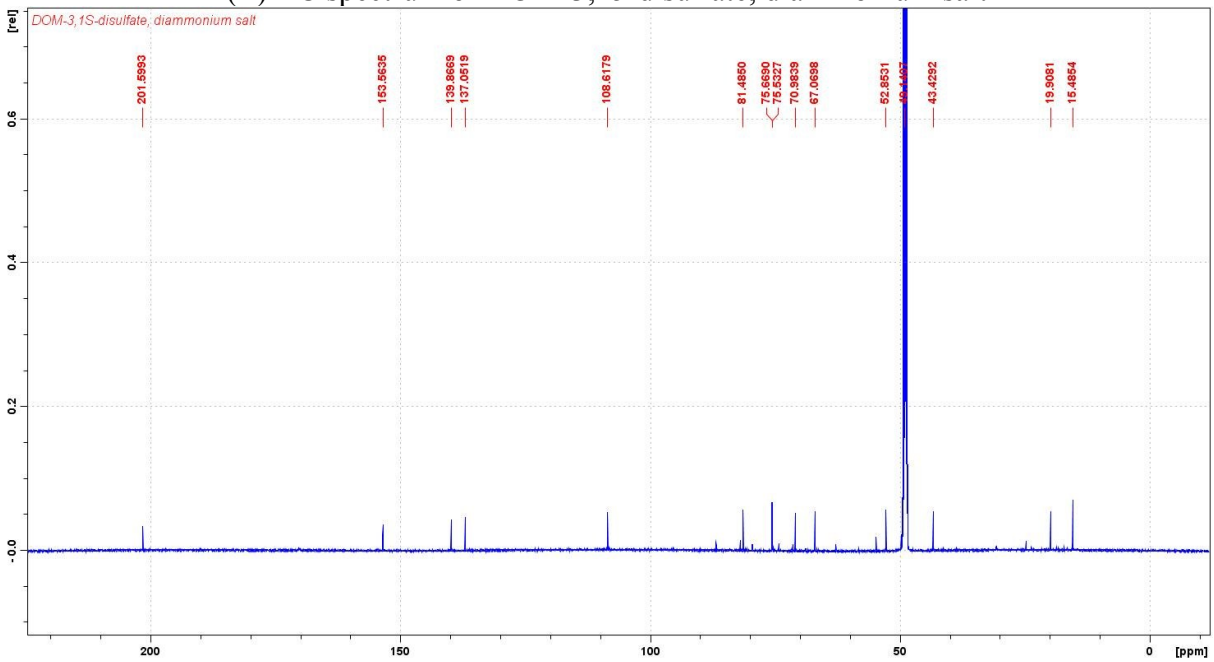


Figure S2. NMR Spectra of DOM-sulfates (ammonium salts).

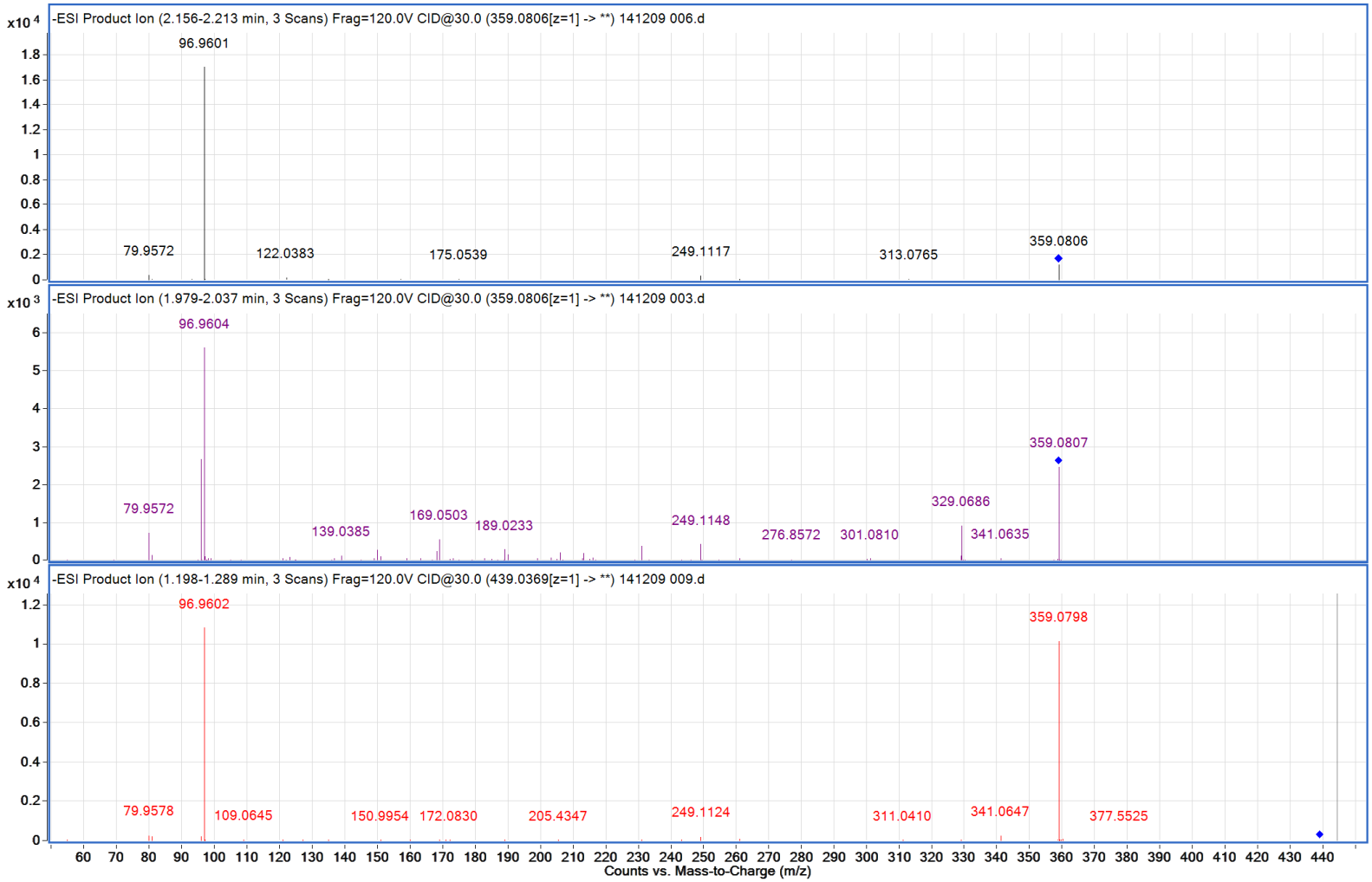


Figure S3. HR-MS/MS spectra of DOM-3-sulfate, DOM-15-sulfate and DOM-3,15-disulfate (CE 30 eV).

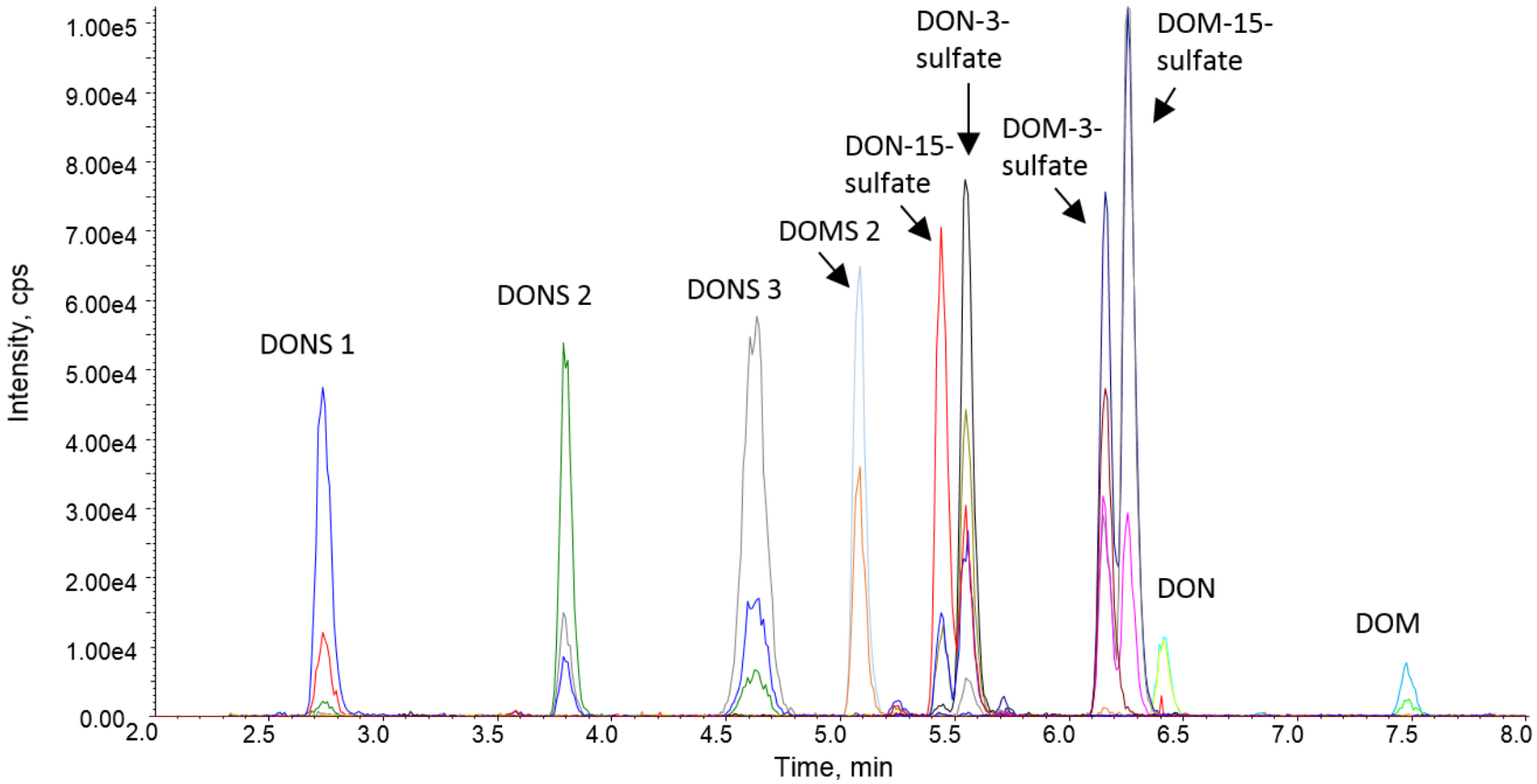


Figure S4. LC-MS/MS chromatogram of a standard solution containing 30 ng/mL of all analytes (long gradient method).