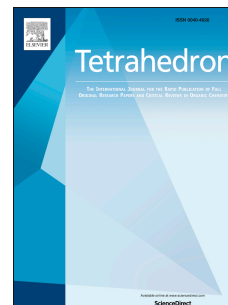


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Graphical Abstract

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Sulfation of deoxynivalenol, its acetylated derivatives and T2-toxin

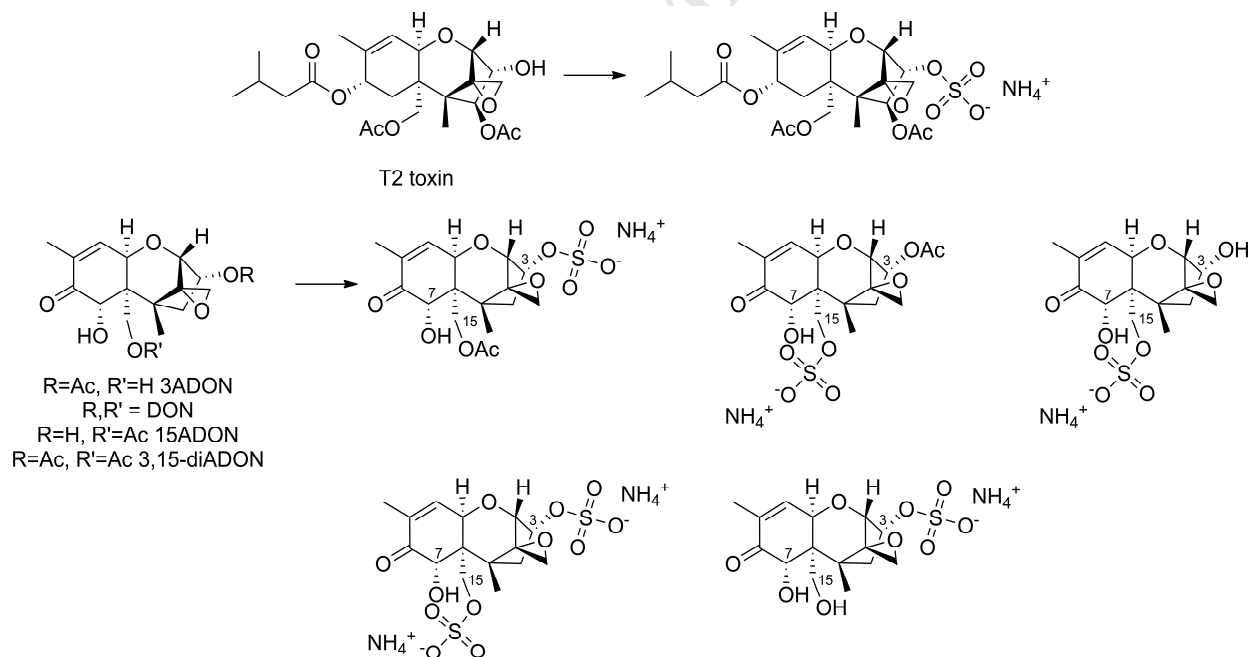
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ABSTRACT

The synthesis of several sulfates of trichothecene mycotoxins is presented. Deoxynivalenol (DON) and its acetylated derivatives were synthesized from 3-acetyldeoxynivalenol (3ADON) and used as substrate for sulfation in order to reach a series of five different DON-based sulfates as well as T2-toxin-3-sulfate. These substances are suspected to be formed during phase-II metabolism in plants and humans. The sulfation was performed using a sulfonyl imidazolium salt which was synthesized prior to use. All protected intermediates and final products were characterized via NMR and will serve as reference materials for further investigations in the fields of toxicology and bioanalytics of mycotoxins.

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1. Introduction

Deoxynivalenol (DON), its acetylated derivatives which occur as 3-acetyldeoxynivalenol (3ADON), 15-acetyldeoxynivalenol (15ADON) and 3,15-diacetyldeoxynivalenol (3,15-diADON) as well as T2-toxin (Fig. 1) are very common and widespread trichothecene mycotoxins. They are predominantly produced by different *Fusarium* species and can contaminate food and feed. They are known to act as a protein biosynthesis inhibitor^{1,2}, neurotoxin, immunosuppressive or nephrotoxin³ and can cause acute and chronic symptoms⁴ after uptake. Based on this knowledge, regulatory limits regarding the toxin concentration for food and feed were established to minimize the daily uptake.

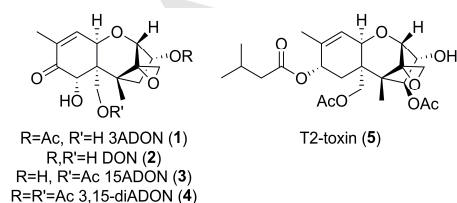


Fig. 1. Structures of DON, its derivatives and T2-toxin.

Although these limits cover the toxins themselves, there is still a lack of information regarding the occurrence and toxicity of their conjugated forms. DON, for example, can undergo glycosylation during late phase-II metabolism in the plant to end up as DON-3- β -D-O-glucoside, which is classified as masked mycotoxin⁵. A similar conjugation leads to the corresponding T2-toxin glucoside which was recently discovered⁶. These masking mechanisms can also lead to di- and triglycosides, which are difficult to investigate due to a lack of authentic reference standards. The main concern regarding these masked forms is the fact that the occurring conjugates can be cleaved in the stomach after uptake, whereby releasing the parent toxin. In addition to the stomach, this cleavage could also occur within the process of malting⁷, leading to an increase of free DON. Besides the formation of glycosides, other masking processes like sulfation or thia-michael addition can take place. Zearalenone is another prominent mycotoxin produced by *Fusarium* species and its metabolite zearalenone-14-sulfate⁸ was previously synthesized⁹ in order to serve as reference material for contamination and toxicity studies. Additionally, the synthesis of different *Alternaria* toxin sulfates¹⁰ was recently published showing the emerging research interest within the field of mycotoxin sulfur conjugates. Besides the occurrence of different masked mycotoxins in plants, many parent toxins also undergo phase-II

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metabolism in living organisms, leading mainly to their corresponding glucuronides¹¹ but also to their sulfates^{12,13}. Besides the occurring glucuronides, which are accessible¹⁴ and were already used for the successful development of a biomarker method¹⁵⁻¹⁷ for human deoxynivalenol exposure estimation, little is known about possible occurring trichothecene sulfates. Considering that sulfate conjugates are described for different substance classes like steroids¹⁸, pollutants¹⁹ and drugs^{20,21}, we assume that there might be even more trichothecene-derived sulfates occurring during metabolism.

The objective of this work was the development of a reliable synthetic strategy for the sulfation of trichothecenes to access all possible DON-sulfates incorporating their acetylated derivatives and T2-toxin-3-sulfate. Since the reactivity of the three hydroxyl groups in deoxynivalenol is somewhat different in the order $15 > 3 >> 7$ (Fig. 2), we expected all kinds of 3- and 15-sulfates to occur. However, we were interested in evaluating the possibility to obtain also some C7-sulfates.

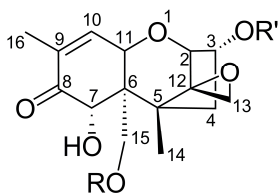
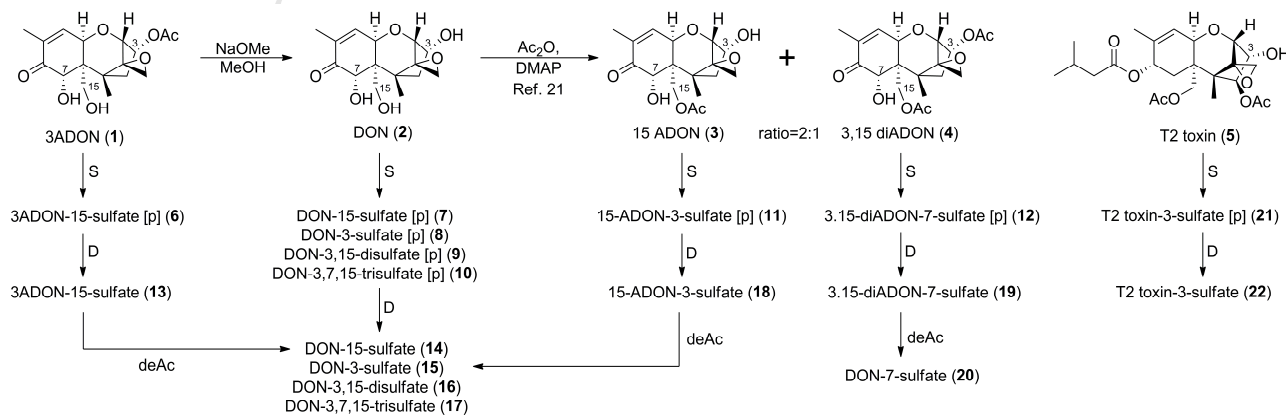


Fig. 2. Structural numbering of trichothecenes.

3ADON was isolated and crystallized²² from an extract of *F. graminearum* and was directly used after comparison with a 3ADON standard. Deprotection and the following acetylation of DON²³ led us to the four desired structures which we wanted to utilize for sulfation (Scheme 1) to access a series of different sulfates.

Regarding sulfation itself, numerous synthetic methods²⁴ can be found within the literature mainly based on the application of commercially available sulfur trioxide complexes. Since these methods are limited concerning possible chemical modifications after installation of the sulfate group as well as yield, reproducibility and regioselectivity, several newer protective groups for the sulfation of organic molecules are described in literature. Besides the fact that we already made good experiences in our group regarding the separation of intermediates and yields by the use of 2,2,2-trichloroethyl (TCE) protective groups²⁵ within chemical sulfation and glycosylation reactions, the use of this protective group also offers the possibility of mild deprotection conditions. The cleavage could therefore be done via catalytic transfer hydrogenation (Pd/C) or via different mild reductive methods including ammonium formate or Zn/ammonium formate.

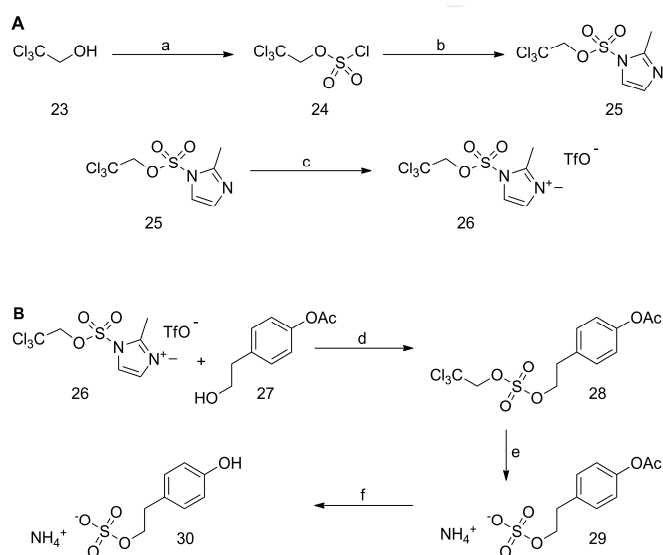


Scheme 1. Synthetic strategies toward all possible sulfate conjugates. S=sulfation, D=deprotection, deAc=deacetylation.

In addition, a very mild protocol using sulfonyl imidazolium salts was recently employed in the synthesis of trichloroethyl-protected sulfates on carbohydrates and offered a good possibility of the sulfation of the fragile trichothecene scaffold.

2. Results and discussion

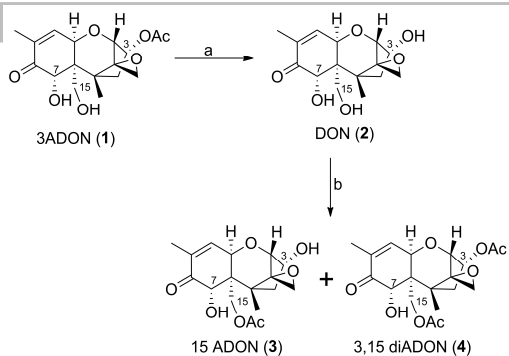
After synthesis of the proper sulfonyl imidazolium salt²⁶ (Scheme 2a, **26**), the method evaluation was done by the use of a simple mimic²⁷ (Scheme 2b, **27**), which was selected because of an incorporated primary alcohol group, a labile acetic ester group (like T2-toxin) and its UV activity.



Scheme 2. Synthesis of the protected sulfate donor (A, **26**) and method evaluation with a simple mimic (B, **27**). Conditions: (a) Pyridine, SO_2Cl_2 , -80°C to RT, 24 h, 97%, (b) 2-methyl imidazole, 2 h, 99%, (c) Et_2O , 0°C , MeOTf, 6 h, 94%, (d), 1,2-dimethylimidazole, 0°C , DCM, 24 h, 82%, (e) MeOH, HCOONH_4 , Zn, 20 min, 81%, (f) MeOH, NaOMe, 1 h, 42%.

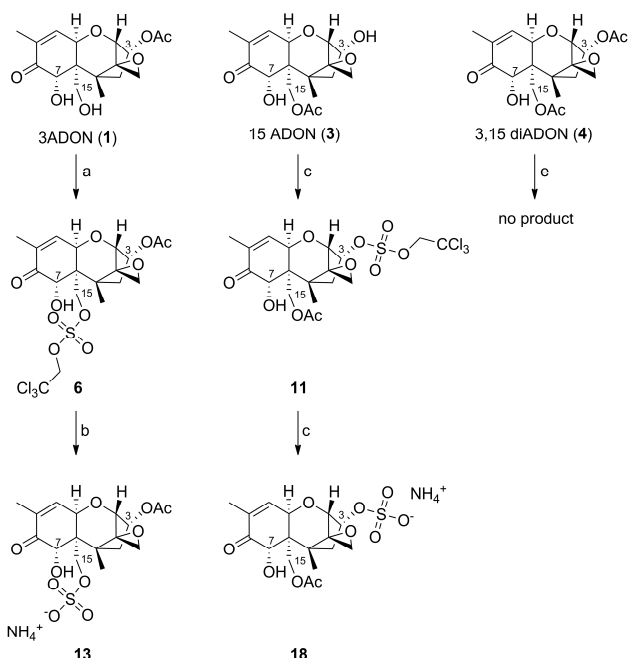
The reaction of **26** and **27** to **28** proceeds smoothly if freshly sublimed dimethylimidazole is used. The deprotection toward **29** was done in an ultrasonic bath at RT, which shortened the reaction time to 20 minutes. This is a crucial achievement since trichothecenes often own labile ester groups which are rapidly cleaved in different solvents. The deprotection toward **30** was done via NaOMe and was carried out as a proof of concept regarding the deprotection of acetylated sulfates.

The reactions toward the different acetylated DON derivatives (Scheme 3) were carried out as described in literature²³ and led to all desired products.



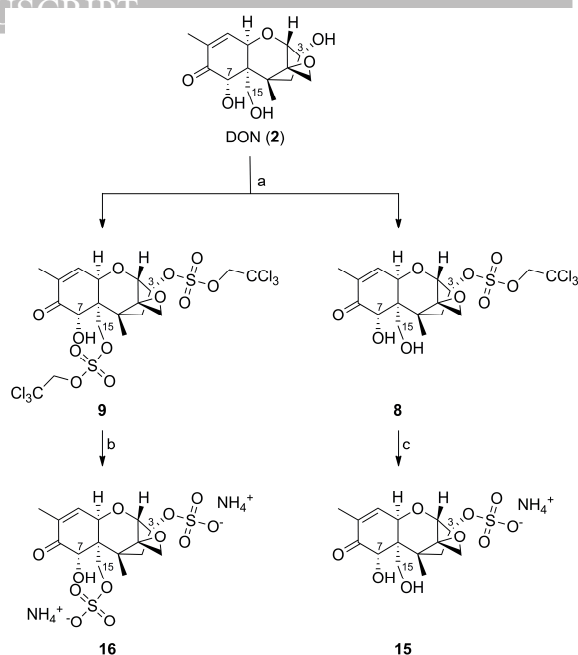
Scheme 3. Synthesis of different deoxynivalenol derivatives. Conditions: (a) NaOMe, MeOH, 2 h, 94%, (b) Ac₂O, DMAP, 18 h, 47% for **3**, 23% for **4**, sum=70%.

The sulfation of **1** yielded (as expected) only one product, which was identified as the protected 3ADON-15-sulfate, and sulfation of **3** yielded only the protected 15ADON-3-sulfate. Both substances were successfully deprotected to the free sulfates as ammonium salt. In case of **4** no reaction was observed at all, even with four equivalents of **26** and prolonged reaction time (Scheme 4).



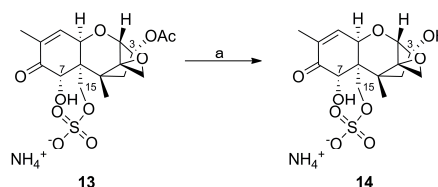
Scheme 4. Synthesis of different acetyldeoxynivalenol-derived sulfates. Conditions: (a) DCM, 2.5 eq. 1,2-dimethylimidazole, 1.25+0.75 eq. **26**, 0°C, 18 h, 52%, (b) MeOH, 9 eq. HCOONH₄, 3 eq. Zn, 20 min, 72%, (c) DCM, 2.7 eq. 1,2-dimethylimidazole, 1.35 eq. **26**, 0°C, 18 h, 34%, (d) MeOH, 9 eq. HCOONH₄, 3 eq. Zn, 20 min, 89%.

In case of **2** we isolated protected DON-3,15-disulfate surprisingly together with DON-3-sulfate instead of the expected 15-sulfate (Scheme 5). Additionally, these two products were the only ones which were isolated. In general, we were expecting position 7 to be somewhat unreactive due to steric hindrance and poor nucleophilicity. Nevertheless, since acetylation resulted in a 2:1 mixture of the 15- to the 3-product, we also expected a similar regioselectivity and ratio of the sulfation reaction. The reaction itself has not undergone any decomposition (observed via TLC) during workup, so we could also exclude the theory of degradation from the disulfate species toward the monosulfate. Deprotection of both substances yielded DON-3-sulfate as ammonium salt and DON-3,15-disulfate as the corresponding diammonium salt.



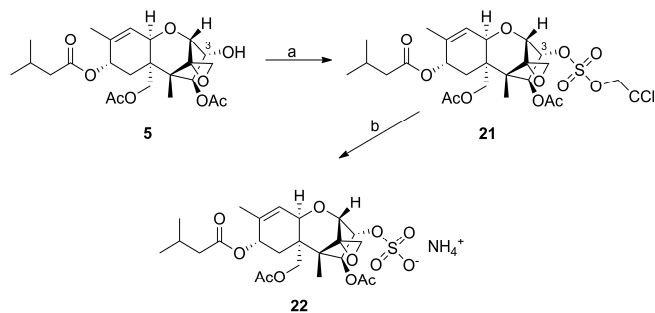
Scheme 5. Synthesis of DON-3-sulfate and DON-3,15-disulfate as their ammonium salts. Conditions: (a) DCM, 4 eq. 1,2-dimethylimidazole, 2 eq. **26**, 0°C, 18 h, 24% for **9**, 30% for **8**, (b) MeOH, 18 eq. HCOONH₄, 6 eq. Zn, 2 h, 54%, (c) MeOH, 9 eq. HCOONH₄, 3 eq. Zn, 30 min, 98%.

Position 7 was not sulfated during any reaction. Of the remaining five theoretically possible sulfates, only DON-15-sulfate was not isolated after direct sulfation (Scheme 1). For this reason we used deacetylation of 3ADON-15-sulfate (Scheme 6) to complete the set of sulfates.



Scheme 6. Synthesis of DON-15-sulfate as ammonium salt. Conditions: (a) MeOH, NaOMe, 2 h, 69%.

Having proven that the method is well working for trichothecenes, we aimed also for the sulfation of T2-toxin, which was carried out in a similar way, whereby leading to the corresponding 3-sulfate as ammonium salt (Scheme 7).



Scheme 7. Synthesis of T2-toxin-3-sulfate as ammonium salt. Conditions: (a) DCM, 4 eq. 1,2-dimethylimidazole, 2 eq. **26**, 0°C, 18 h, 49%, (b) MeOH, 9 eq. HCOONH₄, 3 eq. Zn, 1 h, 29%.

Finally, all isolated protected intermediates as well as all ammonium sulfates were characterized via NMR, and ¹H shifts were assigned using several NMR references^{28,29} for DON and its

derivatives. For the assignment of the ^1H shifts of T2-toxin-3-sulfate, COSY and HMBC spectra were recorded. We tried to purify all intermediates very quickly over short columns (10–15 g silica), since we have encountered deacetylation of all types of trichothecenes during column chromatography (silica as well as RP). Therefore, we ended up with traces of 1,2-dimethylimidazole within the protected intermediates. After the deprotection step, column chromatography was done with very polar solvents (DCM:MeOH:NH₄OH=10:4:1 or 10:2.5:0.5), which led to HCOONH₄ impurities in our products. Because of this we usually made a second column chromatography, followed by lyophilization, and continued drying for several days to remove remaining HCOONH₄. All NMR spectra and ^1H chemical shift assignments can be found in the supporting information.

3. Conclusion

Considering the proven unreactivity of the C7 position to chemical sulfation, we have synthesized, isolated and characterized all possible DON-sulfates including its acetylated derivatives. In case of T2-toxin we were also able to synthesize the desired T2-toxin-3-sulfate. Therefore, we have proven that the utilized method is well working for trichothecenes and provides a good way to access the class of trichothecene sulfates via sulfation of the parent toxins including their acetylated derivatives. Separation of the protected intermediates was done using column chromatography, followed by fast deprotection by the use of Zn/HCOONH₄ within an ultrasonic bath. All products and intermediates were characterized by ^1H and ^{13}C NMR, and all ^1H chemical shifts were assigned to the substances. The gathered ^1H NMR information will serve as a valuable reference for naturally isolated material. Finally, all standards will be used for identification and quantification of their occurrence and formation within human and plant metabolism.

4. Experimental section

4.1. General remarks

CAUTION: All used toxins are strong protein biosynthesis inhibitors and can cause a series of acute and chronic symptoms. Therefore, we strongly recommend considering their toxicity within all reactions!

All reactions were carried out under an argon atmosphere and the progress of all reactions was monitored using thin-layer chromatography (TLC) over silica gel 60 F₂₅₄ (Merck, Germany). All chromatograms were visualized by heat staining using ceric ammonium molybdate/Hanessian's stain³⁰ in ethanol/sulfuric acid. Chromatographic separation was done on silica gel 60 (40–63 μm) using a Sepacore™ Flash System (Büchi, Switzerland) or glass columns. All samples were measured via LC-ESI-MS/MS and LC-APCI-MS/MS and in a negative ionization mode. These measurements were performed on an HCT ion trap mass spectrometer (Bruker, Germany). A TLC-MS interface (Camag, Germany) was used for ESI-MS analysis after TLC. ^1H and ^{13}C spectra were recorded upon using a Bruker DPX-200 spectrometer as well as an Avance DRX-400 MHz spectrometer (both Bruker, Germany). Data were recorded and evaluated using TOPSPIN 1.3 (Bruker Biospin). All chemical shifts are given in ppm relative to tetramethylsilane. The calibration was done using residual solvent signals³¹. Multiplicities are abbreviated as s (singlet), d (doublet), t (triplet), q (quartet) and b (broad signal). 3-ADON was obtained from BOKU, Dept. for Agrarbiotechnology (IFA-Tulln) as crude fermentation extract, purified via column chromatography, and used after the ^1H NMR

purity check. All other chemicals were purchased from ABCR (Germany) and Sigma Aldrich (Austria/Germany).

4.2. 2,2,2-Trichloroethyl 2-(4-acetoxyphenyl)ethylsulfate (28)

27 (121.8 mg, 0.68 mmol, 1.0 eq.) was dissolved in 3 mL DCM and a solution of 1,2-dimethylimidazole (194.9 mg, 2.03 mmol, 3.0 eq.) in 1 mL DCM was added at 0°C. Then, **26** (464.0 mg, 1.01 mmol, 1.5 eq.) was added in one portion and the reaction was allowed to warm to room temperature over night. After TLC indicated full conversion the reaction was directly purified via column chromatography (hexane:EtOAc = 1:1) to yield **28** (215.8 mg, 82%) as white solid. ^1H NMR (200 MHz, CDCl₃) δ 7.28 (s, 4H), 4.82 (s, 2H), 4.27 (t, *J*=6.8 Hz, 2H), 2.95 (t, *J*=6.8 Hz, 2H), 2.02 (s, 3H); ^{13}C NMR (50 MHz, CDCl₃) δ 171.0 (s, 1C), 148.9 (s, 1C), 138.1 (s, 1C), 130.6 (d, 2C), 121.2 (d, 2C), 92.5 (s, 1C), 80.5 (t, 1C), 64.5 (t, 1C), 34.5 (t, 1C), 21.0 (q, 1C). HRMS *m/z* calcd for C₁₂H₁₄O₆SCl₃⁺ [M+H]⁺, 390.9571, found 390.9578.

4.3. 2-(4-Acetoxyphenyl)ethylsulfate, ammonium salt (29)

28 (190.0 mg, 0.49 mmol, 1.0 eq.) was dissolved in 5 mL MeOH. Ammonium formate (276.5 mg, 4.39 mmol, 9.0 eq.) and Zn dust (95.6 mg, 1.46 mmol, 3.0 eq.) were added and the reaction was placed in an ultrasonic bath. After 20 minutes, TLC revealed complete conversion of the starting material and the reaction mixture was filtered through celite and concentrated to 1 mL. Direct purification of this solution using column chromatography (DCM:MeOH:NH₄OH = 10:2.5:0.5) yielded **29** (109.5 mg, 81%) as white solid. ^1H NMR (200 MHz, methanol-*d*₄) δ = 7.22 (b, 4H), 4.93 (b, NH₄⁺, H₂O), 4.23 (t, *J*=6.9, 2H), 2.91 (t, *J*=6.9 Hz, 2H), 2.00 (s, 3H); ^{13}C NMR (50 MHz, methanol-*d*₄) δ 172.9 (s, 1C), 152.5 (s, 1C), 136.0 (s, 1C), 130.6 (d, 2C), 122.5 (d, 2C), 66.2 (t, 1C), 35.3 (t, 1C), 20.8 (q, 1C). HRMS *m/z* calcd for C₁₀H₁₁O₆S⁻ [M-NH₄]⁻, 259.0282, found 259.0278.

4.4. 2-(4-Hydroxyphenyl)ethylsulfate, ammonium salt (30)

29 (69.0 mg, 0.25 mmol, 1.00 eq.) was dissolved in 2 mL dry MeOH and NaOMe (14.1 mg, 0.26 mmol, 1.05 eq.) was added. Since no reaction occurred after 30 minutes we added the same amount of NaOMe again. After stirring for 30 minutes, TLC indicated full conversion and the reaction was directly purified via column chromatography (DCM:MeOH:NH₄OH = 10:2.5:0.5) to yield **9** (28.7 mg, 42%) as white solid. ^1H NMR (200 MHz, methanol-*d*₄) δ = 7.21 (s, 4H), 4.90 (b, NH₄⁺, H₂O), 3.73 (t, *J*=6.9, 2H), 2.80 (t, *J*=6.9 Hz, 2H); ^{13}C NMR (50 MHz, methanol-*d*₄) δ 152.3 (s, 1C), 137.0 (s, 1C), 130.6 (d, 2C), 122.5 (d, 2C), 64.2 (t, 1C), 39.5 (t, 1C). HRMS *m/z* calcd for C₈H₉O₅S⁻ [M-NH₄]⁻, 217.0176, found 217.0174.

4.5. 15-ADON (3) and 3,15-diADON (4)

3-ADON (**1**) (95.6 mg, 0.28 mmol, 1.0 eq.) was dissolved in 5 mL methanol, followed by the addition of NaOMe (13.7 mg, 0.25 mmol, 0.9 eq.). After 2 h, TLC showed full conversion of the **1**. The reaction was concentrated to 1 mL and directly purified by the use of column chromatography (CHCl₃:MeOH = 9:1), which yielded deoxynivalenol (**2**, 79.0 mg, 94%) as white solid. The reaction product was proven to be identical to an authentic sample by TLC and, thus, was directly used for acetylation. For this purpose, **2** (79.0 mg, 0.27 mmol) was dissolved in 50 mL dry dichloromethane. Pyridine (1 mL) and 4-DMAP (app. 10 mg) were added, followed by the dropwise addition of acetic anhydride (27.2 mg, 0.27 mmol). The reaction was stirred over night, treated with 20 mL 2N HCl, and extracted with dichloromethane. After drying with Na₂SO₄, filtration and

evaporation of the solvent, the remaining residue was subjected to column chromatography (CHCl₃:MeOH = 95:5) to yield **3** (42.0 mg, 47%) and **4** (23.5 mg, 23%) as white solid. Total yield = 70%, 93% conversion. 15-ADON (**3**): ¹H NMR (200 MHz, CDCl₃) δ 6.61 (dq, *J*=5.7, 1.6 Hz, 1H), 4.89 (d, *J*=5.7 Hz, 1H), 4.83 (d, *J*=1.6 Hz, 1H), 4.52 (dt, *J*=10.2, 4.7 Hz, 1H), 4.24 (s, 2H), 3.78 (d, *J*=1.8 Hz, 1H), 3.63 (d, *J*=4.5 Hz, 1H), 3.13 (d, *J*=4.3 Hz, 1H), 3.08 (d, *J*=4.3 Hz, 1H), 2.22 (dd, *J*=14.8, 4.7 Hz, 1H), 2.08 (dd, *J*=14.7, 10.4 Hz, 1H), 1.88 (s, 3 H), 1.87 (s, 3H), 1.07 (s, 3H); ¹³C NMR (50 MHz, CDCl₃) δ = 199.6 (s), 170.3 (s), 138.8 (d), 135.6 (s), 80.7 (d), 73.5 (d), 70.1 (d), 68.9 (s), 65.5 (s), 62.2 (t), 51.4 (s), 47.4 (t), 46.3 (s), 43.3 (t), 20.7 (q), 15.4 (q), 13.8 (q). 3,15-diADON (**4**): ¹H NMR (200 MHz, CDCl₃) δ 6.56 (dq, *J*=5.8, 1.4 Hz, 1H), 5.20 (dt, *J*=10.9, 4.6 Hz, 1H), 4.80 (d, *J*=2.0 Hz, 1H), 4.69 (d, *J*=5.8 Hz, 1H), 4.27 (d, *J*=12.1 Hz, 1H), 4.20 (d, *J*=12.1 Hz, 1H), 3.89 (d, *J*=4.3 Hz, 1H), 3.80 (d, *J*=2.0 Hz, 1H), 3.14 (d, *J*=4.3 Hz, 1H), 3.09 (d, *J*=4.3 Hz, 1H), 2.31 (dd, *J*=15.2, 4.8 Hz, 1H), 2.15 (dd, *J*=15.2, 10.9 Hz, 1H), 2.12 (s, 3H), 1.88 (s, 3 H), 1.87 (s, 3H), 1.08 (s, 3H); ¹³C NMR (50 MHz, CDCl₃) δ 199.3 (s), 170.3 (s), 170.2 (s), 138.4 (d), 135.6 (s), 78.9 (d), 73.4 (d), 71.1 (d), 70.1 (d), 64.9 (s), 62.1 (t), 51.5 (s), 47.4 (t), 45.8 (s), 40.3 (t), 21.0 (q), 20.6 (q), 15.3 (q), 13.6 (q).

4.6. 2,2,2-Trichloroethyl 3-acetyl-DON-15-sulfate (**6**)

1 (45.1 mg, 133 μmol, 1.0 eq.) was dissolved in 2.5 mL of DCM and cooled to 0°C and 1,2-dimethylimidazole (32.0 mg, 333 μmol, 2.5 eq.) in 1 mL DCM was added to the reaction. Then, **26** (76.2 mg, 167 μmol, 1.25 eq.) was added and the reaction was allowed to reach room temperature over night. Since TLC showed remaining starting material after 18 h, the reaction was cooled again to 0°C and another 0.75 eq. of **26** (45.7 mg, 100 μmol) were added. After another 48 hours, TLC showed still starting material, but also the formation of substantial amounts of product. The reaction was directly used for column chromatography (CHCl₃:MeOH = 95:5), yielding **6** (37.8 mg, 52%) as white solid. ¹H NMR (200 MHz, CDCl₃) δ 6.66 (dq, *J*=5.9, 1.4 Hz, 1H), 5.24 (ddd, *J*=9.5, 6.0, 4.5, 1H), 4.89 (d, *J*=1.4 Hz, 1H), 4.80 (d, *J*=5.9 Hz, 1H), 4.64 (d, *J*=10.8 Hz, 1H), 4.57 (d, *J*=10.8 Hz, 1H), 4.56 (d, *J*=10.6 Hz, 1H), 4.43 (d, *J*=10.6 Hz, 1H), 3.95 (d, *J*=4.5 Hz, 1H), 3.84 (d, *J*=1.4 Hz, 1H), 3.16 (d, *J*=4.1 Hz, 1H), 3.13 (d, *J*=4.1 Hz, 1H), 2.10–2.36 (m, 2H), 2.16 (s, 3H), 1.91 (b, 3H), 1.11 (s, 3H); ¹³C NMR (50 MHz, CDCl₃) δ 198.8 (s, 1C), 170.2 (s, 1C), 138.7 (d, 1C), 136.1 (s, 1C), 92.4 (s, 1C), 79.7 (t, 1C), 78.8 (d, 1C), 73.0 (d, 1C), 71.6 (t, 1C), 70.7 (d, 1C), 69.1 (d, 1C), 64.6 (s, 1C), 51.3 (s, 1C), 47.4 (t, 1C), 45.8 (s, 1C), 40.4 (t, 1C), 20.9 (q, 1C), 15.2 (q, 1C), 13.5 (q, 1C); HRMS *m/z* calcd for C₁₉H₂₄O₁₀SCl₃⁺ [M+H]⁺, 549.0150, found 549.0160.

4.7. 2,2,2-Trichloroethyl 15-acetyl-DON-3-sulfate (**11**)

3 (76.5 mg, 226 μmol, 1.0 eq.) was dissolved in 3 mL of DCM and cooled to 0°C and 1,2-dimethylimidazole (58.7 mg, 610 μmol, 2.7 eq.) in 1 mL DCM was added to reaction. Then **26** (139.7 mg, 305 μmol, 1.35 eq.) was added and the reaction was allowed to reach room temperature over night. TLC showed substantial amounts of product and the reaction was directly used for column chromatography (DCM:MeOH = 95:5) yielding **11** (42.6 mg, 34%) as white solid. ¹H NMR (200 MHz, CDCl₃) δ 6.60 (dq, *J*=5.8, 1.5 Hz, 1H), 5.31 (dt, *J*=11.1, 4.3, 1H), 4.81 (s, 1H), 4.79 (s, 2H), 4.70 (d, *J*=5.8 Hz, 1H), 4.27 (d, *J*=12.1 Hz, 1H), 4.18 (d, *J*=12.1 Hz, 1H), 4.00 (d, *J*=4.3 Hz, 1H), 3.71 (s, 1H), 3.21 (d, *J*=4.1 Hz, 1H), 3.15 (d, *J*=4.1 Hz, 1H), 2.64 (dd, *J*=15.7, 4.3 Hz, 1H), 2.29 (dd, *J*=15.7, 11.1 Hz, 1H), 1.93 (s, 3H), 1.91 (b, 3H), 1.12 (s, 3H); ¹³C NMR (50 MHz, CDCl₃) δ 198.9 (s, 1C), 170.2 (s, 1C), 137.9 (d, 1C), 136.1 (s, 1C), 92.7 (s, 1C), 80.4

(d, 1C), 80.0 (t, 1C), 78.9 (d, 1C), 73.5 (d, 1C), 70.1 (d, 1C), 64.5 (s, 1C), 62.0 (t, 1C), 51.2 (s, 1C), 47.4 (t, 1C), 46.1 (s, 1C), 40.2 (t, 1C), 20.8 (q, 1C), 15.4 (q, 1C), 13.7 (q, 1C); HRMS *m/z* calcd for C₁₉H₂₄O₁₀SCl₃⁺ [M+H]⁺, 549.0150, found 549.0146.

4.8. 2,2,2-Trichloroethyl DON-3-sulfate (**8**) and bis(2,2,2-trichloroethyl) DON-3,15-disulfate (**9**)

2 (40.6 mg, 137 μmol, 1.0 eq.) was dissolved in 5 mL of DCM and cooled to 0°C and 1,2-dimethylimidazole (52.7 mg, 548 μmol, 4.0 eq.) in 1 mL DCM was added to the reaction. Then, **26** (125.4 mg, 274 μmol, 2.0 eq.) was added and the reaction was allowed to reach room temperature over night. TLC after 24 h showed nearly full conversion of the starting material and the reaction was directly used for column chromatography (DCM:MeOH gradient from 100:0→95:5), yielding **8** (22.4 mg, 30%) and **9** (23.9 mg, 24%) as white solid. ¹H NMR of **8** (200 MHz, CDCl₃) δ 6.62 (dq, *J*=5.9, 1.4 Hz, 1H), 5.31 (dt, *J*=11.2, 4.4, 1H), 4.81 (s, 1H), 4.78 (s, 2H), 4.74 (d, *J*=5.9 Hz, 1H), 3.99 (d, *J*=4.5 Hz, 1H), 3.86 (d, *J*=11.5 Hz, 1H), 3.77 (b, 1H), 3.76 (d, *J*=11.5 Hz, 1H), 3.22 (d, *J*=4.1 Hz, 1H), 3.14 (d, *J*=4.1 Hz, 1H), 2.80 (dd, *J*=15.7, 4.3 Hz, 1H), 2.25 (dd, *J*=15.7, 11.2 Hz, 1H), 1.91 (b, 3H), 1.71 (b, 1H), 1.17 (s, 3H); ¹³C NMR of **8** (50 MHz, CDCl₃) δ 199.6 (s, 1C), 138.0 (d, 1C), 136.3 (s, 1C), 92.7 (s, 1C, -CCl₃ tiny signal!), 80.8 (d, 1C), 79.9 (t, 1C), 79.1 (d, 1C), 74.5 (d, 1C), 70.1 (d, 1C), 64.8 (s, 1C), 62.0 (t, 1C), 51.8 (s, 1C), 47.7 (t, 1C), 46.1 (s, 1C), 40.3 (t, 1C), 15.4 (q, 1C), 14.1 (q, 1C); HRMS *m/z* calcd for C₁₇H₂₀O₉SCl₃⁻ [M-H]⁻, 504.9899, found 504.9878. ¹H NMR of **9** (200 MHz, CDCl₃) δ 6.69 (dq, *J*=5.9, 1.5 Hz, 1H), 5.32 (dt, *J*=11.1, 4.4, 1H), 4.88 (s, 1H), 4.81 (dt, *J*=5.7, 1.5 Hz, 1H), 4.79 (s, 2H), 4.66 (d, *J*=11.0 Hz, 1H), 4.60 (d, *J*=11.0 Hz, 1H), 4.48 (s, 2H), 4.04 (d, *J*=4.4 Hz, 1H), 3.83 (b, 1H), 3.21 (d, *J*=4.1 Hz, 1H), 3.17 (d, *J*=4.1 Hz, 1H), 2.57 (dd, *J*=15.7, 4.2 Hz, 1H), 2.33 (dd, *J*=15.7, 11.1 Hz, 1H), 1.93 (b, 3H), 1.15 (s, 3H); ¹³C NMR of **9** (50 MHz, CDCl₃) δ 198.5 (s, 1C), 138.2 (d, 1C), 136.6 (s, 1C), 2 x 80.0 (d, 1C/t, 1C), 79.9 (t, 1C), 78.9 (d, 1C), 73.3 (d, 1C), 71.5 (t, 1C), 69.2 (d, 1C), 64.3 (s, 1C), 51.3 (s, 1C), 47.7 (t, 1C), 46.2 (s, 1C), 40.3 (t, 1C), 15.3 (q, 1C), 13.6 (q, 1C), 2 x CCl₃ between 92 and 93 ppm are missing, but the corresponding CH₂ groups are located at 80.0 and 79.9; HRMS *m/z* calcd for C₁₇H₂₀O₁₂S₂Cl₃⁻ [M-TCE]⁻, 584.9467, found 584.9505.

4.9. General deprotection procedure

The protected intermediate was dissolved in MeOH (1 mL/10 μmol starting material). HCOONH₄ (3 eq.) as well as Zn dust (9 eq.) were added and the reaction was placed in an ultrasonic bath at room temperature. The reaction was followed via TLC until substantial amounts of products were formed (20 to 90 min). After filtration through celite the remaining residue was subjected to column chromatography to end up with the corresponding sulfates as ammonium salts. For all acetylated DON derivatives and T2-toxin, DCM:MeOH:NH₄OH=10:2.5:0.5 was used for purification. In case of DON-3- and 15-sulfate and 3,15-disulfate, a mixture of DCM:MeOH:NH₄OH = 10:4:1 was used. Since all products contained accompanying HCOONH₄, we tried to purify some products via a second and third column chromatography as well as via lyophilization. Nevertheless, we obtained all desired products as a white misty veil.

4.10. 3-Acetyl-DON-15-sulfate, ammonium salt (**13**)

Following the general deprotection procedure, **6** (34.0 mg, 62 μmol) was converted into **13** (19.3 mg, 72%). ¹H NMR (200 MHz, methanol-*d*₄) δ 6.63 (dq, *J*=6.1, 1.4 Hz, 1H), 5.11 (dt, *J*=11.3, 4.4 Hz, 1H), 4.92 (d, *J*=6.1 Hz, 1H), 4.89 (s, 1H), 4.87 (b, NH₄⁺, H₂O), 4.27 (d, *J*=11.0 Hz, 1H), 3.94 (d, *J*=11.1 Hz,

1H), 3.85 (d, $J=4.5$ Hz, 1H), 3.16 (d, $J=4.3$ Hz, 1H), 3.12 (d, $J=4.3$ Hz, 1H), 2.79 (dd, $J=15.3, 4.3$ Hz, 1H), 2.08 (dd, $J=15.3, 11.3$ Hz, 1H), 2.13 (s, 3H), 1.85 (b, 3H), 1.18 (s, 3H); ^{13}C NMR (50 MHz, methanol- d_4) δ 201.0 (s, 1C), 172.5 (s, 1C), 139.4 (d, 1C), 137.1 (s, 1C), 80.5 (d, 1C), 75.8 (d, 1C), 72.7 (d, 1C), 70.7 (d, 1C), 67.1 (t, 1C), 66.3 (s, 1C), 52.3 (s, 1C), 48.4 (t, 1C), 46.8 (s, 1C), 41.7 (t, 1C), 20.8 (q, 1C), 15.3 (q, 1C), 14.4 (q, 1C); HRMS m/z calcd for $\text{C}_{17}\text{H}_{21}\text{O}_{10}\text{S}^- [\text{M}-\text{NH}_4^+]$, 417.0861, found 417.0834.

4.11. 15-Acetyl-DON-3-sulfate, ammonium salt (18)

Following the general deprotection procedure, **11** (13.1 mg, 24 μmol) was converted into **18** (9.2 mg, 89%). ^1H NMR (400 MHz, methanol- d_4) δ 6.65 (dq, $J=5.9, 1.5$ Hz, 1H), 4.70–5.10 (m, NH_4^+ , C3-H, C7-H, C11-H, H_2O), 4.30 (d, $J=12.1$ Hz, 1H), 4.23 (d, $J=12.1$ Hz, 1H), 3.82 (d, $J=4.5$ Hz, 1H), 3.12 (s, 2H), 2.63 (dd, $J=15.3, 4.3$ Hz, 1H), 2.11 (dd, $J=15.3, 11.2$ Hz, 1H), 1.90 (s, 3H), 1.85 (b, 3H), 1.11 (s, 3H); ^{13}C NMR (100 MHz, methanol- d_4) δ 201.1 (s, 1C), 171.9 (s, 1C), 139.7 (d, 1C), 137.0 (s, 1C), 81.2 (d, 1C), 75.2 (d, 1C), 75.0 (d, 1C), 71.3 (d, 1C), 65.9 (s, 1C), 63.3 (t, 1C), 52.6 (s, 1C), 48.2 (t, 1C), 47.0 (s, 1C), 42.5 (t, 1C), 20.6 (q, 1C), 15.4 (q, 1C), 14.4 (q, 1C); HRMS m/z calcd for $\text{C}_{17}\text{H}_{21}\text{O}_{10}\text{S}^- [\text{M}-\text{NH}_4^+]$, 417.0861, found 417.0824.

4.12. DON-3-sulfate, ammonium salt (15)

Following the general deprotection procedure, **8** (18.8 mg, 37 μmol) was converted into **15** (14.3 mg, 98%). ^1H NMR (400 MHz, methanol- d_4) δ 6.61 (dq, $J=6.1, 1.4$ Hz, 1H), 4.75–4.95 (m, NH_4^+ , C3-H, C11-H, H_2O), 4.79 (s, 1H), 3.80 (d, $J=4.4$ Hz, 1H), 3.78 (d, $J=12.3$ Hz, 1H), 3.68 (d, $J=12.3$ Hz, 1H), 3.12 (d, $J=4.5$ Hz, 1H), 3.09 (d, $J=4.5$ Hz, 1H), 2.75 (dd, $J=15.2, 4.4$ Hz, 1H), 2.06 (dd, $J=15.2, 11.4$ Hz, 1H), 1.83 (b, 3H), 1.12 (s, 3H); ^{13}C NMR (100 MHz, methanol- d_4) δ 201.7 (s, 1C), 139.4 (d, 1C), 137.0 (s, 1C), 81.2 (d, 1C), 75.8 (d, 1C), 75.4 (d, 1C), 71.6 (d, 1C), 66.3 (s, 1C), 61.8 (t, 1C), 53.6 (s, 1C), 48.2 (t, 1C), 46.7 (s, 1C), 42.5 (t, 1C), 15.4 (q, 1C), 14.6 (q, 1C); HRMS m/z calcd for $\text{C}_{15}\text{H}_{19}\text{O}_9\text{S}^- [\text{M}-\text{NH}_4^+]$, 375.0755, found 375.0741.

4.13. DON-15-sulfate, ammonium salt (14)

13 (12.0 mg, 28 μmol , 1.0 eq.) was dissolved in 5 mL MeOH and NaOMe (3.0 mg, 55 mmol, 2.0 eq.) was added. After stirring for 2 hours, TLC indicated full conversion of the starting material and the reaction was subjected to column chromatography (DCM:MeOH: $\text{NH}_4\text{OH} = 10:4:1$), yielding **14** (7.5 mg, 69%) as white solid. ^1H NMR (200 MHz, methanol- d_4) δ 6.65 (dq, $J=6.1, 1.4$ Hz, 1H), 5.04 (d, $J=6.1$ Hz, 1H), 4.85–4.95 (m, NH_4^+ , C7-H, H_2O), 4.76 (dt, $J=11.1, 4.5$, 1H), 4.25 (d, $J=11.0$ Hz, 1H), 3.96 (d, $J=11.0$ Hz, 1H), 3.55 (d, $J=4.5$ Hz, 1H), 3.12 (d, $J=4.5$ Hz, 1H), 3.06 (d, $J=4.5$ Hz, 1H), 2.57 (dd, $J=14.8, 4.4$ Hz, 1H), 1.99 (dd, $J=14.8, 11.1$ Hz, 1H), 1.84 (b, 3H), 1.14 (s, 3H); ^{13}C NMR (50 MHz, methanol- d_4) δ 201.1 (s, 1C), 139.9 (d, 1C), 136.9 (s, 1C), 82.3 (d, 1C), 75.9 (d, 1C), 70.7 (d, 1C), 69.6 (d, 1C), 67.2 (s, 1C), 66.7 (t, 1C), 52.5 (s, 1C), 48.2 (t, 1C), 47.5 (s, 1C), 45.0 (t, 1C), 15.3 (q, 1C), 14.4 (q, 1C); HRMS m/z calcd for $\text{C}_{15}\text{H}_{19}\text{O}_9\text{S}^- [\text{M}-\text{NH}_4^+]$, 375.0755, found 375.0746.

4.14. DON-3,15-disulfate, diammonium salt (16)

Following the general deprotection procedure, twice the amount of HCOONH_4 (6 eq.) and Zn dust (18 eq.) were used to convert **9** (20.0 mg, 28 μmol) into **16** (7.4 mg, 54%). ^1H NMR (400 MHz, methanol- d_4) δ 6.67 (dq, $J=6.0, 1.5$ Hz, 1H), 4.80–5.00 (m, NH_4^+ , C3-H, C7-H, C11-H, H_2O), 4.24 (d, $J=10.9$ Hz, 1H), 3.98 (d, $J=10.9$ Hz, 1H), 3.84 (d, $J=4.5$ Hz, 1H), 3.15 (d, $J=4.3$ Hz, 1H), 3.10 (d, $J=4.3$ Hz, 1H), 2.97 (dd, $J=15.3, 4.5$ Hz, 1H), 2.10 (dd, $J=15.3, 11.4$ Hz, 1H), 1.85 (b, 3H), 1.16 (s, 3H);

^{13}C NMR (100 MHz, methanol- d_4) δ 201.3 (s, 1C), 139.6 (d, 1C), 137.2 (s, 1C), 81.3 (d, 1C), 75.8 (d, 1C), 75.7 (d, 1C), 70.5 (d, 1C), 67.4 (s, 1C), 66.1 (t, 1C), 52.4 (s, 1C), 48.4 (t, 1C), 47.1 (s, 1C), 42.4 (t, 1C), 15.3 (q, 1C), 14.5 (q, 1C); HRMS m/z calcd for $\text{C}_{15}\text{H}_{20}\text{O}_{12}\text{S}_2^- [\text{M}-2\text{xNH}_4^+ + \text{H}^+]$, 455.0323, found 455.0300.

4.15. 2,2,2-Trichloroethyl T2-toxin-3-sulfate (21)

5 (37.4 mg, 80 μmol , 1.0 eq.) was dissolved in 3 mL of DCM, cooled to 0°C , and 1,2-dimethylimidazole (30.8 mg, 321 mmol, 4.0 eq.) in 1 mL DCM was added to the reaction. Then, **26** (73.4 mg, 160 mmol, 2.00 eq.) was added and the reaction was allowed to reach room temperature over night. After 18 hours, TLC showed substantial amounts of product and the reaction was directly used for column chromatography (DCM:MeOH = 95:5), yielding **21** (26.4 mg, 49%) as white solid. ^1H NMR (200 MHz, CDCl_3) δ 6.16 (d, $J=3.1$ Hz, 1H), 5.77 (dt, $J=5.7, 1.4$ Hz, 1H), 5.28 (d, $J=5.5$ Hz, 1H), 5.10 (dd, $J=4.9, 3.1$ Hz, 1H), 4.80 (s, 2H), 4.31 (d, $J=12.7$ Hz, 1H), 4.25 (d, $J=5.7$ Hz, 1H), 4.10 (d, $J=12.7$ Hz, 1H), 3.96 (d, $J=4.9$ Hz, 1H), 3.09 (d, $J=3.9$ Hz, 1H), 2.85 (d, $J=3.9$ Hz, 1H), 2.35 (dd, $J=15.2, 6.0$ Hz, 1H), 2.11 (s, 3H), 2.09 (s, 3H), 2.00–2.25 (m, 3H), 1.80 (d, $J=16.6$ Hz, 1H), 1.76 (s, 3H), 0.96 (d, $J=6.3$ Hz, 3H), 0.95 (d, $J=6.3$ Hz, 3H), 0.74 (s, 3H); ^{13}C NMR (50 MHz, CDCl_3) δ 172.8 (s, 1C), 170.4 (s, 1C), 170.2 (s, 1C), 137.1 (s, 1C), 123.0 (d, 1C), 92.6 (s, 1C), 87.1 (d, 1C), 80.1 (t, 1C), 78.4 (d, 1C), 77.4 (d, 1C), 67.7 (d, 1C), 67.5 (d, 1C), 64.6 (t, 1C), 63.8 (s, 1C), 48.7 & 47.5 (1t, 1s, 2x1C), 43.7 (t, 1C), 43.1 (s, 1C), 28.4 (t, 1C), 25.9 (d, 1C), 22.6 (q, 1C), 22.5 (q, 1C), 21.2 (q, 1C), 20.8 (q, 1C), 20.4 (q, 1C), 6.5 (q, 1C); HRMS m/z calcd for $\text{C}_{24}\text{H}_{33}\text{O}_{12}\text{S}^- [\text{M}-\text{TCE-group}]$, 545.1698, found 545.1679.

4.16. T2-toxin-3-sulfate, ammonium salt (22)

Following the general deprotection procedure, **21** (20.8 mg, 31 μmol) was converted into **22** (5.0 mg, 29%). ^1H NMR (400 MHz, methanol- d_4) δ 6.03 (d, $J=3.1$ Hz, 1H), 5.78 (dt, $J=5.5$ Hz, 1H), 5.33 (d, $J=5.5$ Hz, 1H), 4.80–4.94 (m, NH_4^+ , C3-H, H_2O), 4.33 (d, $J=12.1$, 1H), 4.32 (d, $J=5.5$ Hz, 1H), 4.16 (d, $J=12.1$ Hz, 1H), 3.79 (d, $J=5.1$ Hz, 1H), 3.04 (d, $J=3.9$ Hz, 1H), 2.87 (d, $J=3.9$ Hz, 1H), 2.38 (dd, $J=15.3, 5.5$ Hz, 1H), 2.13–2.18 (m, 2H), 2.07 (s, 3H), 2.06 (s, 3H), 2.00–2.12 (m, 1H), 1.92 (d, $J=15.3$ Hz, 1H), 1.74 (s, 3H), 0.97 (d, $J=6.7$ Hz, 3H), 0.96 (d, $J=6.7$ Hz, 3H), 0.72 (s, 3H); ^{13}C NMR (100 MHz, methanol- d_4) δ 174.0 (s, 1C), 172.3 (s, 1C), 172.2 (s, 1C), 137.2 (s, 1C), 125.1 (d, 1C), 82.3 (d, 1C), 81.5 (d, 1C), 79.7 (d, 1C), 69.4 (d, 1C), 68.5 (d, 1C), 65.8 (t, 1C), 65.0 (s, 1C), 50.1 & 47.8 (1t, 1s, 2x1C), 44.5 (t, 1C), 44.3 (s, 1C), 28.8 (t, 1C), 26.9 (d, 1C), 22.8 (q, 1C), 22.7 (q, 1C), 21.3 (q, 1C), 20.7 (q, 1C), 20.4 (q, 1C), 6.9 (q, 1C); HRMS m/z calcd for $\text{C}_{24}\text{H}_{33}\text{O}_{12}\text{S}^- [\text{M}-\text{NH}_4^+]$, 545.1698, found 545.1682.

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Supplementary Material

NMR spectra of all protected and isolated sulfates as well as tables for the ^1H chemical shifts of all substances can be found in the supporting information.

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Supplementary Information

Sulfation of deoxynivalenol, its acetylated derivatives and T2-toxin

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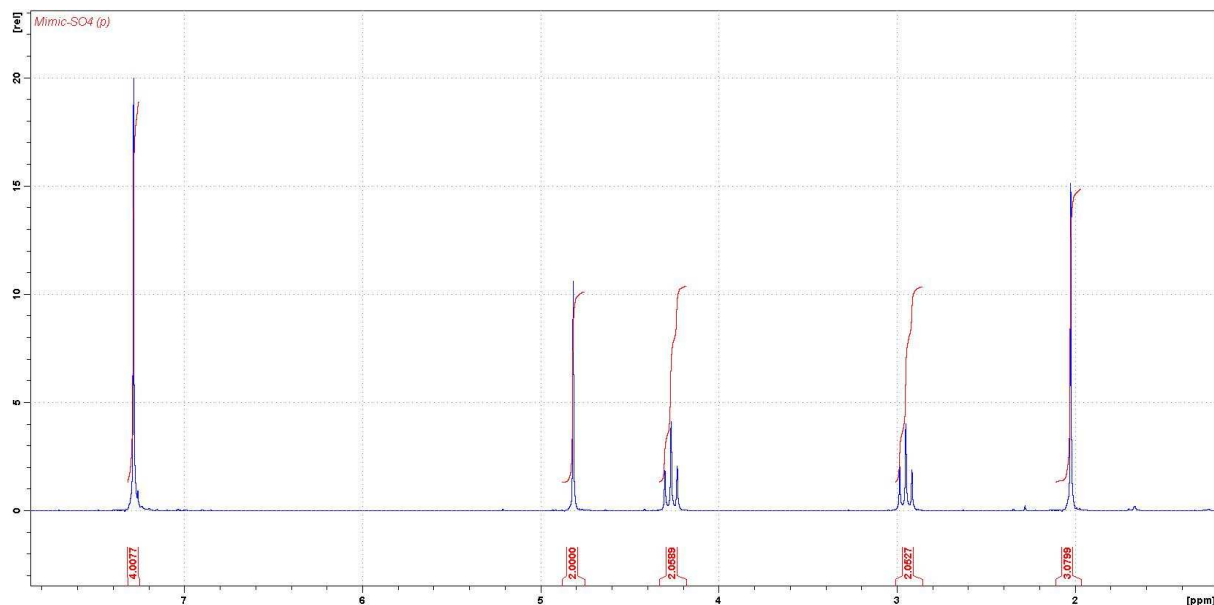
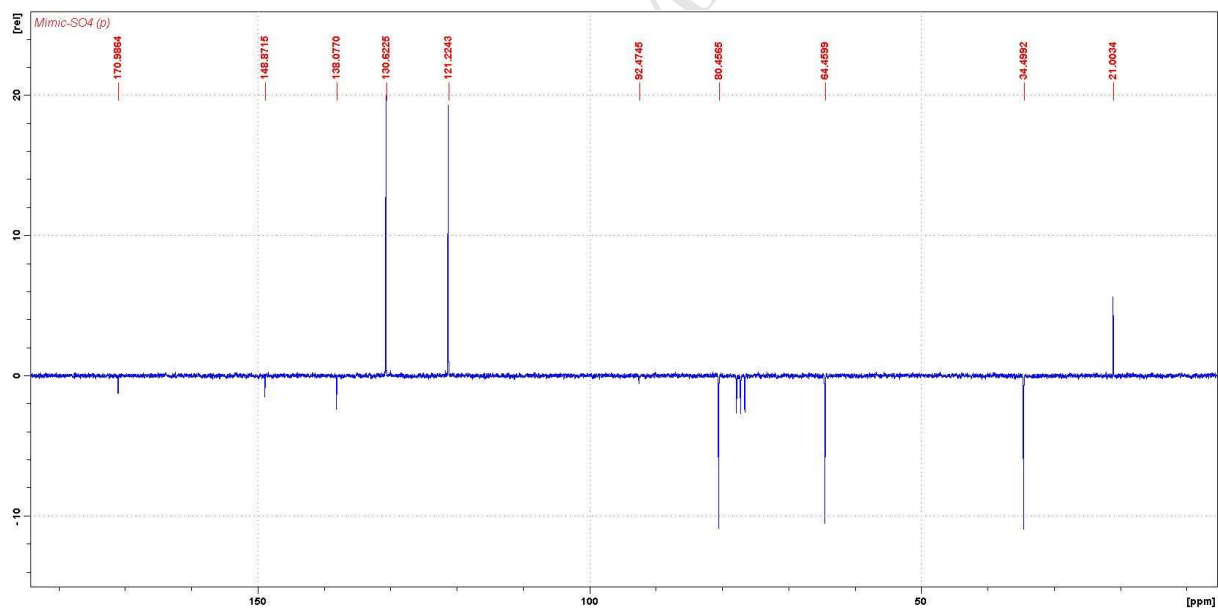
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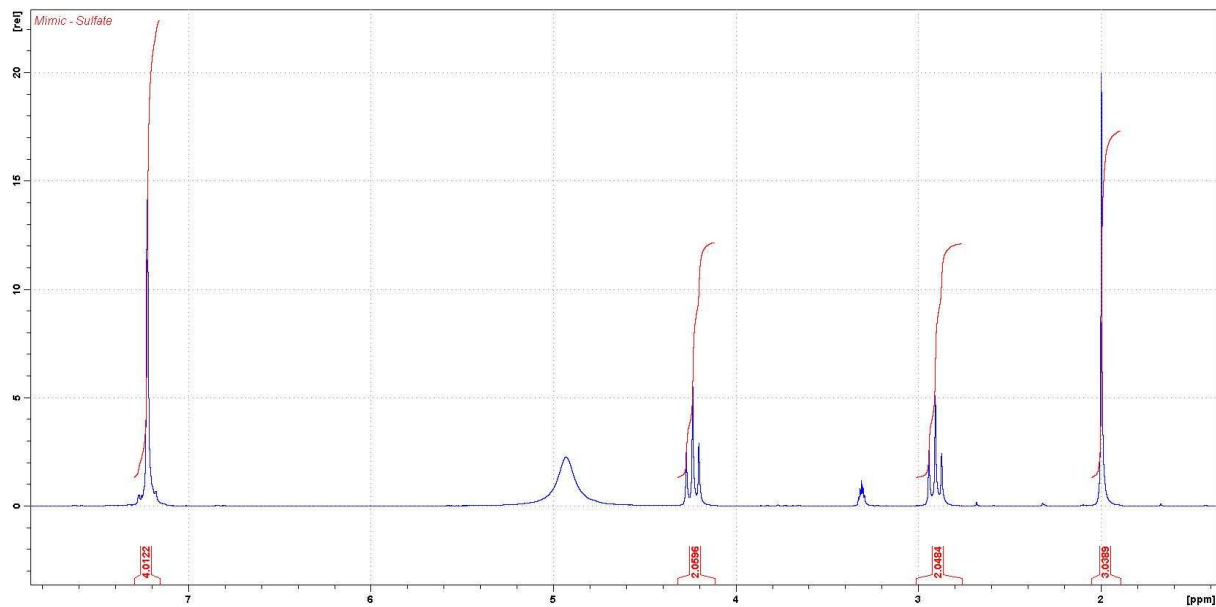
Name / Position	2	3	4 α	4 β	7 α	7 β	8 β	10	11	13 a	13 b	14	15 a	15 b	16	3 Ac/OH	7 Ac/OH	15 Ac/OH	3 TCE	15 TCE
DON (2) in CDCl ₃	3.63 d [4.5]	4.53 bt	2.23 dd [14.8; 4.6]	2.08 dd [14.9; 10.6]	x	4.84 bs	x	6.62 dq [5.9; 1.4]	4.81 d [5.7]	3.08 d [4.3]	3.17 d [4.3]	1.1 s	3.74 d [11.5]	3.89 d [11.5]	1.9 bs	2.37 d [2.4]	3.86 bs, 1H	1.75 bs, 1H	x	x
DON (2) in MeOD	3.53 d [4.5]	4.37 dt [11.0; 4.5]	2.39 dd [14.6; 4.5]	1.95 dd [14.6; 11.0]	x	4.72 s	x	6.50 dq [5.9; 1.5]	4.93 d [5.7]	3.05 d [4.3]	3.10 d [4.3]	1.1 s	3.73 s, 2H	3.73	2 bs	x	x	x	x	x
3 ADON (1) in CDCl ₃	3.91 d [4.7]	5.23 dt [11.0; 4.5]	2.38 dd [15.1; 4.5]	2.17 dd [15.1; 11.2]	x	4.84 d [1.8]	x	6.61 dq [5.8; 1.5]	4.70 d [5.9]	3.12 d [4.3]	3.18 d [4.3]	1.2 s	3.72 - 3.88 m, 3H	1.90 bs	2.14 s, 3H	3.72 - 3.88 m, 3H	1.67 bt, 1H	x	x	x
15 ADON (3) in CDCl ₃	3.60 d [4.5]	4.49 dt [9.8; 4.9]	2.20 dd [14.8; 4.8]	2.06 dd [14.8; 10.5]	x	4.81 bs	x	6.59 dq [5.9; 1.4]	4.88 d [5.7]	3.06 d [4.3]	3.11 d [4.3]	1.1 s	4.22 bs	4.22 bs	1.9 bs	2.92 bs, 1H	3.8 bs, 1H	1.86 bs	x	x
3,15 diADON (4) in CDCl ₃	3.89 d [4.5]	5.19 dt [10.9; 4.6]	2.30 dd [15.2; 4.8]	2.14 dd [15.2; 10.9]	x	4.79 d [2.0]	x	6.55 dq [5.7; 1.4]	4.68 d [5.7]	3.08 d [4.3]	3.13 d [4.3]	1.1 s	4.19 d [12.1]	4.27 d [12.1]	1.9 bs	2.11 s, 3H	3.79 d [2.0], 1H	1.86 bs	x	x
T2 toxin (5) in CDCl ₃	3.64 d [4.9]	4.18 dd [4.9; 2.9]	5.34 d [2.9]	x	1.9-2.2 m	2.38 dd [15.1; 5.4]	5.3 d [5.5]	5.78 dt [5.9; 1.2]	4.35 d [6.3]	2.79 d [3.9]	3.05 d [3.9]	0.8 s	4.06 d [12.7]	4.30 d [12.7]	1.7 bs	2.00 - 2.20 2xs, 2x3H, 6H	x	2.00 - 2.20 2xs, 2x3H, 6H	x	x
3 ADON-15-sulfate [p] (6) in CDCl ₃	3.95 d [4.5]	5.24 ddd [9.5; 6.0; 4.5]	2.10 - 2.36 m, 2 H	2.10 - 2.36 m, 2 H	x	4.89 d [1.4]	x	6.66 dq [5.9; 1.4]	4.8 d [5.9]	3.13 d [4.1]	3.16 d [4.1]	1.1 s	4.43 d [10.6]	4.56 d [10.6]	1.9 bs	2.16 s, 3H	3.84 d [1.4], 1H	x	x	4.64; 4.57 2 x d [10.8]
15 ADON-3-sulfate [p] (11) in CDCl ₃	4.00 d [4.3]	5.31 dt [11.1; 4.3]	2.64 dd [15.7; 4.3]	2.29 dd [15.7; 11.1]	x	4.81 s	x	6.60 dq [5.8; 1.5]	4.70 d [5.8]	3.15 d [4.1]	3.21 d [4.1]	1.1 s	4.18 d [12.1]	4.27 d [12.1]	1.9 bs	x	3.71 s, 1H	1.93 s	4.79 s, 2H	x
DON-3,15-disulfate [pp] (9) in CDCl ₃	4.04 d [4.4]	5.32 dt [11.1; 4.4]	2.57 dd [15.7; 4.2]	2.33 dd [15.7; 11.1]	x	4.88 bs	x	6.69 dq [5.9; 1.5]	4.81 dt [5.7; 1.5]	3.17 d [4.1]	3.21 d [4.1]	1.2 s	4.60 d [11.0]	4.66 d [11.0]	1.9 bs	x	3.83 bs, 1H	x	4.79 s, 2H	4.48 s, 2H
DON-3-sulfate [p] (15) in CDCl ₃	3.99 [4.5]	5.31 dt [11.2; 4.4]	2.80 dd [15.7; 4.3]	2.25 dd [15.7; 11.2]	x	4.81 s	x	6.62 dq [5.9; 1.4]	4.74 d [5.9]	3.14 d [4.1]	3.22 d [4.1]	1.2 s	3.76 d [11.5]	3.86 d [11.5]	1.9 bs	x	3.77 bs, 1H	1.71 bs, 1H	x	4.78 s, 2H
T2 toxin-3-sulfate [p] (21) in CDCl ₃	3.96 d [4.9]	5.10 dd [4.9; 3.1]	6.16 d [3.1]	x	1.80 d [16.6]	2.35 dd [15.2; 6.0]	5.3 d [5.5]	5.77 dt [5.7; 1.4]	4.25 d [5.7]	2.85 d [3.9]	3.09 d [3.9]	0.7 s	4.1 d [12.7]	4.31 d [12.7]	1.8 bs	2.00 - 2.20 2xs, 2x3H, 6H	x	2.00 - 2.20 2xs, 2x3H, 6H	4.80 s, 2H	x
3 ADON-15-sulfate (13) in MeOD	3.85 d [4.5]	5.11 dt [11.3; 4.4]	2.79 dd [15.3; 4.3]	2.08 dd [15.3; 11.3]	x	4.89 s	x	6.63 dq [6.1; 1.4]	4.92 d [6.1]	3.12 d [4.3]	3.16 d [4.3]	1.2 s	3.94 d [11.0]	4.27 d [11.0]	1.9 bs	2.13 s, 3H	x	x	x	x
15 ADON-3-sulfate (18) in MeOD	3.82 d [4.5]	4.70-5.10 m [+NH ₄ , H ₂ O]	2.63 dd [15.3; 4.3]	2.11 dd [15.3; 11.2]	x	4.70-5.10 m [+NH ₄ , H ₂ O]	x	6.65 dq [5.9; 1.5]	4.70-5.10 m [+NH ₄ , H ₂ O]	3.12 s	3.12 s	1.1 s	4.23 d [12.1]	4.30 d [12.1]	1.9 bs	x	x	1.90 s, 3H	x	x
DON-3-sulfate (15) in MeOD	3.80 d [4.4]	4.90 dt [11.2; 4.5]	2.75 dd [15.2; 4.4]	2.06 dd [15.2; 11.4]	x	4.79 s	x	6.61 dq [6.1; 1.4]	4.86 d [5.8]	3.09 d [4.5]	3.12 d [4.5]	1.1 s	3.68 d [12.3]	3.78 d [12.3]	1.8 bs	x	x	x	x	x
DON-15-sulfate (14) in MeOD	3.55 d [4.5]	4.76 dt [11.1; 4.45]	2.57 dd [14.8; 4.4]	1.99 dd [14.8; 11.1]	x	4.85-4.95 m [+NH ₄ , H ₂ O]	x	6.65 dq [6.1; 1.4]	5.04 d [6.1]	3.06 d [4.5]	3.12 d [4.5]	1.1 s	3.96 d [11.0]	4.25 d [11.0]	1.8 bs	x	x	x	x	x
DON-3,15-disulfate (16) in MeOD	3.84 d [4.5]	4.80 - 5.00 m [+NH ₄ , H ₂ O]	2.97 dd [15.3; 4.5]	2.10 dd [15.3; 11.4]	x	4.80-5.00 m [+NH ₄ , H ₂ O]	x	6.67 dq [6.0; 1.5]	4.80-5.00 m [+NH ₄ , H ₂ O]	3.10 d [4.3]	3.15 d [4.3]	1.2 s	4.24 d [10.9]	3.98 d [10.9]	1.9 bs	x	x	x	x	x
T2 toxin-3-sulfate (22) in MeOD	3.79 d [5.1]	5.10 dd [4.9; 3.1]	6.03 d [3.1]	x	1.92 d [16.6]	2.38 dd [15.3; 5.9]	5.3 d [5.5]	5.78 d [5.5]	4.32 d [5.5]	2.87 d [3.9]	3.04 d [3.9]	0.7 s	4.16 d [12.1]	4.33 d [12.1]	1.7 bs	2.00 - 2.20 2xs, 2x3H, 6H	x	2.00 - 2.20 2xs, 2x3H, 6H	x	x

28.) 2,2,2-Trichloroethyl 2-(4-acetoxyphenyl)ethylsulfate

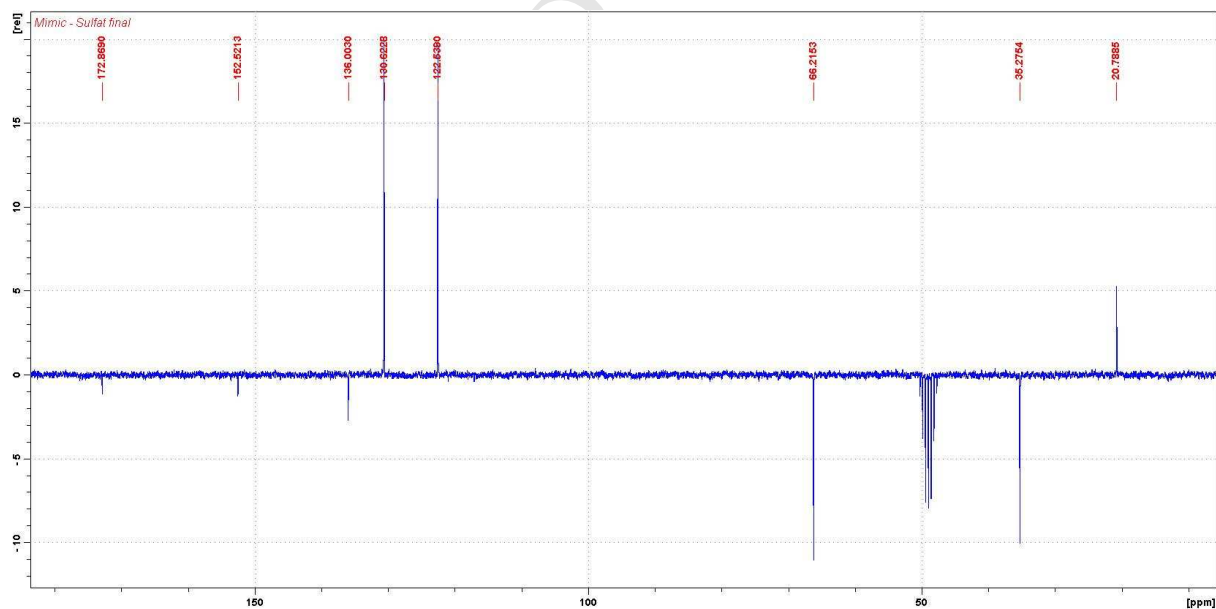
 ^1H NMR (200 MHz, CDCl_3) δ 7.28 (s, 4H), 4.82 (s, 2H), 4.27 (t, $J=6.8$ Hz, 2H), 2.95 (t, $J=6.8$ Hz, 2H), 2.02 (s, 3H) ^{13}C NMR (50 MHz, CDCl_3) δ 171.0 (s, 1C), 148.9 (s, 1C), 138.1 (s, 1C), 130.6 (d, 2C), 121.2 (d, 2C), 92.5 (s, 1C), 80.5 (t, 1C), 64.5 (t, 1C), 34.5 (t, 1C), 21.0 (q, 1C)

29.) 2-(4-Acetoxyphenyl)ethylsulfate, ammonium salt

^1H NMR (200 MHz, methanol- d_4) δ = 7.22 (b, 4H), 4.93 (b, NH_4^+ , H_2O), 4.23 (t, $J=6.9$, 2H), 2.91 (t, $J=6.9$ Hz, 2H), 2.00 (s, 3H);

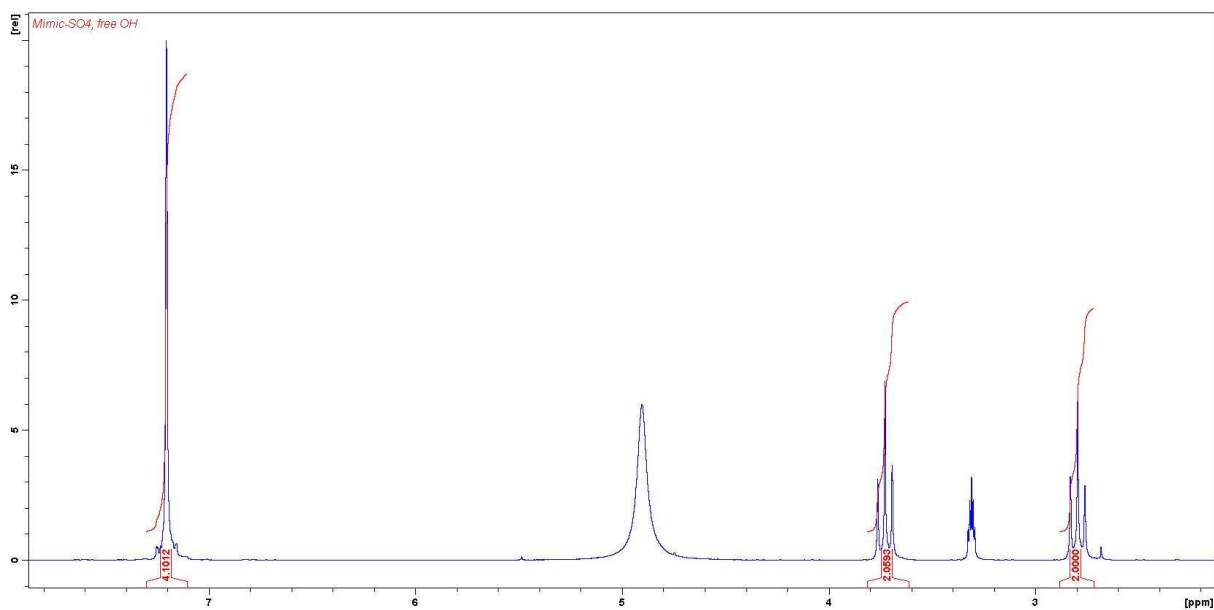


^{13}C NMR (50 MHz, methanol- d_4) δ 172.9 (s, 1C), 152.5 (s, 1C), 136.0 (s, 1C), 130.6 (d, 2C), 122.5 (d, 2C), 66.2 (t, 1C), 35.3 (t, 1C), 20.8 (q, 1C)

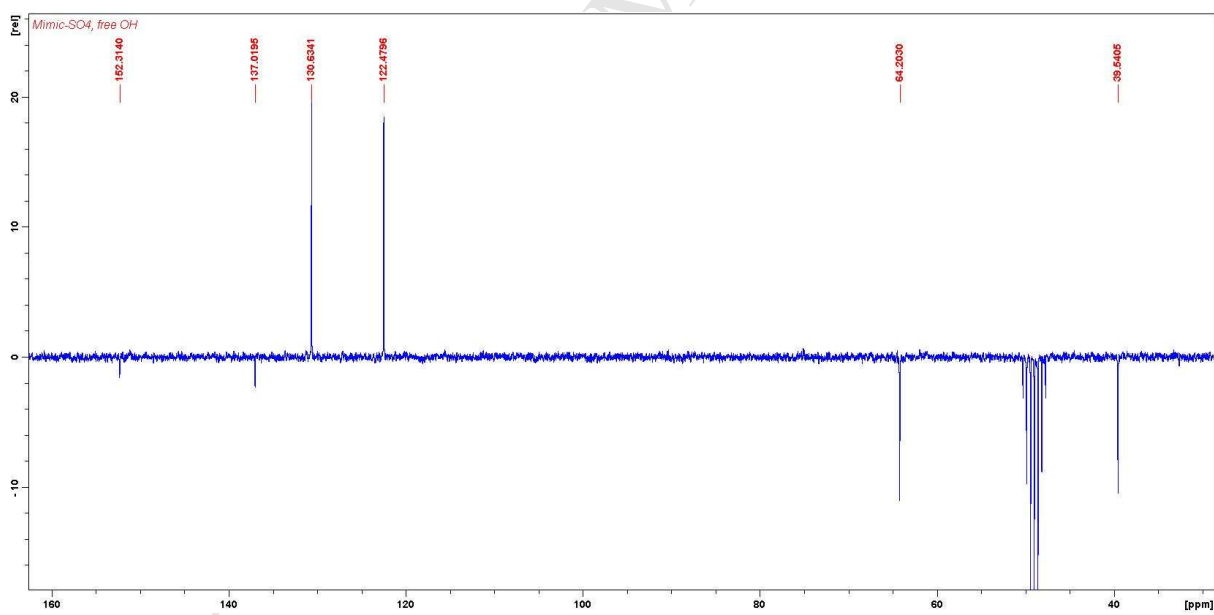


30.) 2-(4-Hydroxyphenyl)ethylsulfate, ammonium salt

^1H NMR (200 MHz, methanol- d_4) δ = 7.21 (s, 4H), 4.90 (b, NH_4^+ , H_2O), 3.73 (t, $J=6.9$, 2H), 2.80 (t, $J=6.9$ Hz, 2H)

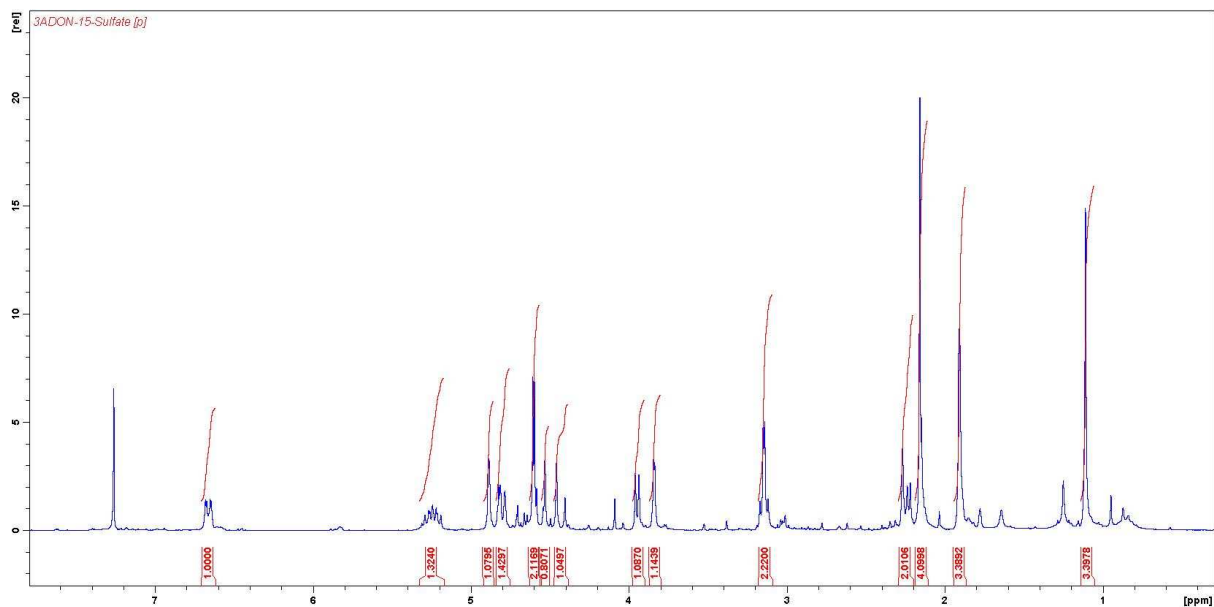


^{13}C NMR (50 MHz, methanol- d_4) δ 152.3 (s, 1C), 137.0 (s, 1C), 130.6 (d, 2C), 122.5 (d, 2C), 64.2 (t, 1C), 39.5 (t, 1C)

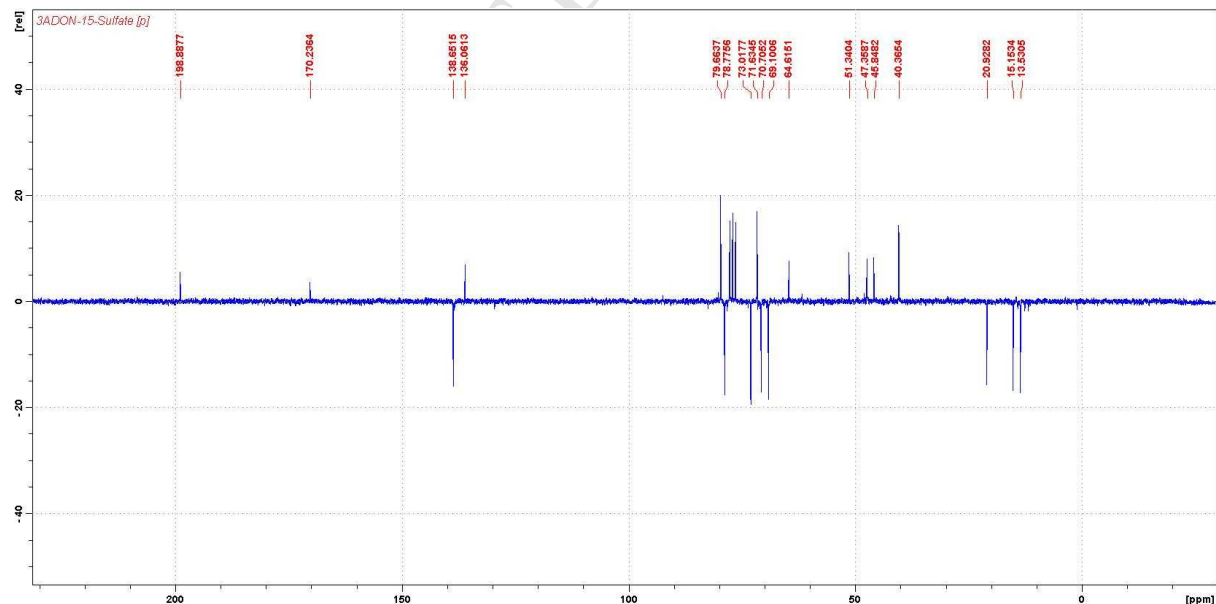


6.) 2,2,2-Trichloroethyl 3-acetyl-DON-15-sulfate

^1H NMR (200 MHz, CDCl_3) δ 6.66 (dq, $J=5.9, 1.4$ Hz, 1H), 5.24 (ddd, $J=9.5, 6.0, 4.5$ Hz, 1H), 4.89 (d, $J=1.4$ Hz, 1H), 4.80 (d, $J=5.9$ Hz, 1H), 4.64 (d, $J=10.8$ Hz, 1H), 4.57 (d, $J=10.8$ Hz, 1H), 4.56 (d, $J=10.6$ Hz, 1H), 4.43 (d, $J=10.6$ Hz, 1H), 3.95 (d, $J=4.5$ Hz, 1H), 3.84 (d, $J=1.4$ Hz, 1H), 3.16 (d, $J=4.1$ Hz, 1H), 3.13 (d, $J=4.1$ Hz, 1H), 2.10-2.36 (m, 2H), 2.16 (s, 3H), 1.91 (b, 3H), 1.11 (s, 3H)

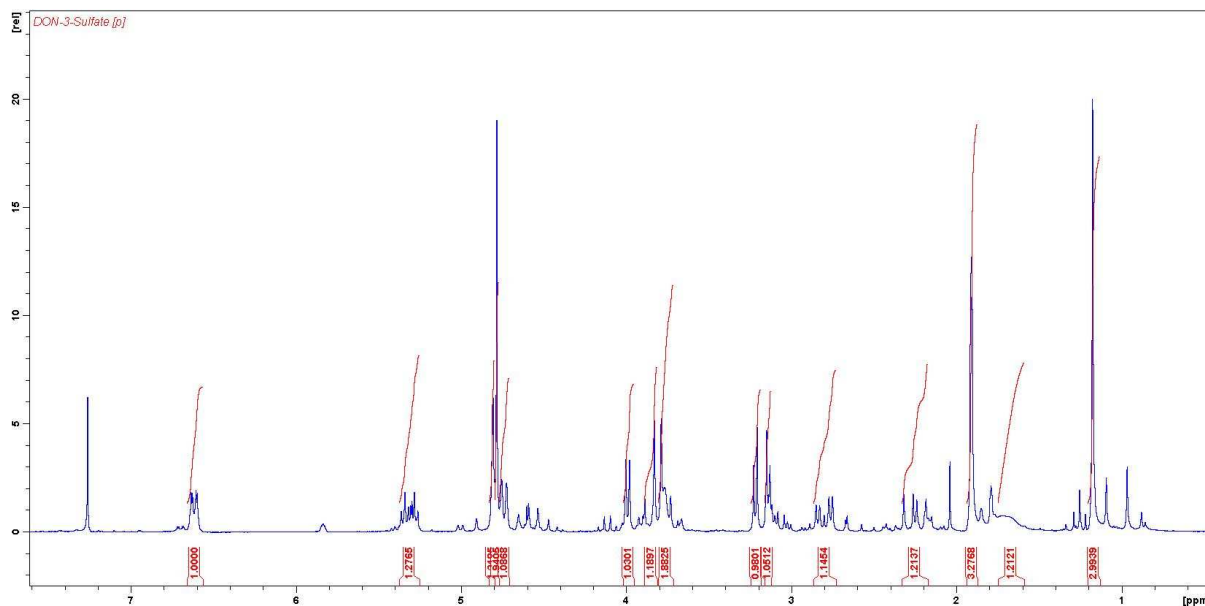


^{13}C -NMR (50 MHz, CDCl_3) δ 198.8 (s, 1C), 170.2 (s, 1C), 138.7 (d, 1C), 136.1 (s, 1C), 92.4 (s, 1C, $-\text{CCl}_3$ tiny signal!), 79.7 (t, 1C), 78.8 (d, 1C), 73.0 (d, 1C), 71.6 (t, 1C), 70.7 (d, 1C), 69.1 (d, 1C), 64.6 (s, 1C), 51.3 (s, 1C), 47.4 (t, 1C), 45.8 (s, 1C), 40.4 (t, 1C), 20.9 (q, 1C), 15.2 (q, 1C), 13.5 (q, 1C)

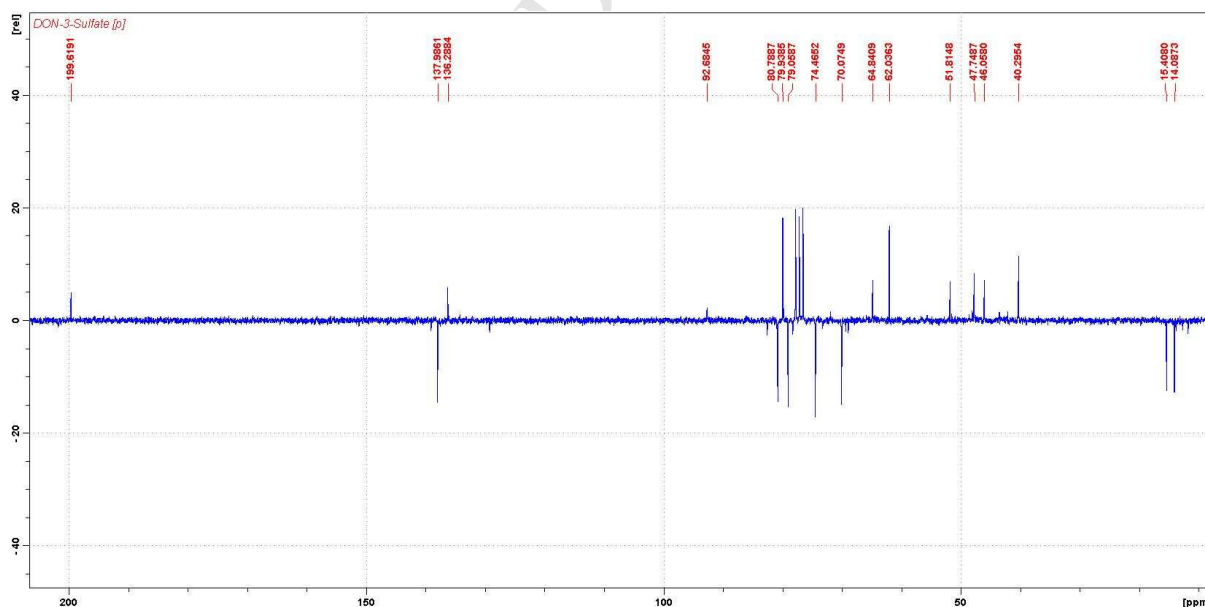


8.) 2,2,2-Trichloroethyl DON-3-sulfate

^1H NMR (200 MHz, CDCl_3) δ 6.62 (dq, $J=5.9, 1.4$ Hz, 1H), 5.31 (dt, $J=11.2, 4.4$, 1H), 4.81 (s, 1H), 4.78 (s, 2H), 4.74 (d, $J=5.9$ Hz, 1H), 3.99 (d, $J=4.5$ Hz, 1H), 3.86 (d, $J=11.5$ Hz, 1H), 3.77 (b, 1H), 3.76 (d, $J=11.5$ Hz, 1H), 3.22 (d, $J=4.1$ Hz, 1H), 3.14 (d, $J=4.1$ Hz, 1H), 2.80 (dd, $J=15.7, 4.3$ Hz, 1H), 2.25 (dd, $J=15.7, 11.2$ Hz, 1H), 1.91 (b, 3H), 1.71 (b, 1H), 1.17 (s, 3H)

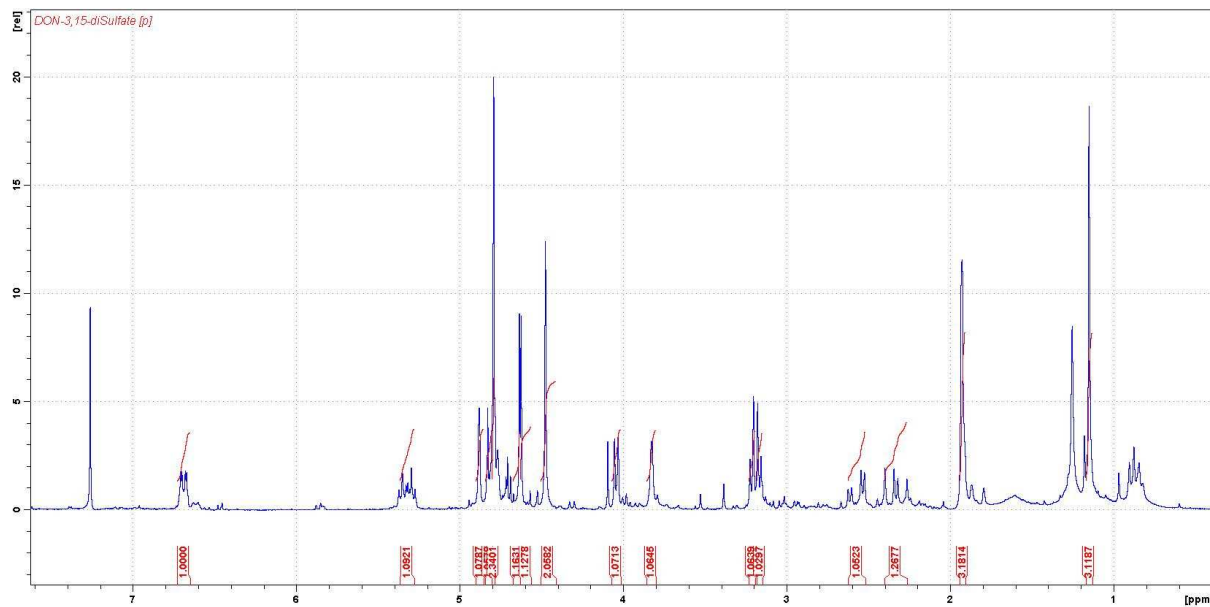


^{13}C -NMR (50 MHz, CDCl_3) δ 199.6 (s, 1C), 138.0 (d, 1C), 136.3 (s, 1C), 92.7 (s, 1C, $-\text{CCl}_3$ tiny signal!), 80.8 (d, 1C), 79.9 (t, 1C), 79.1 (d, 1C), 74.5 (d, 1C), 70.1 (d, 1C), 64.8 (s, 1C), 62.0 (t, 1C), 51.8 (s, 1C), 47.7 (t, 1C), 46.1 (s, 1C), 40.3 (t, 1C), 15.4 (q, 1C), 14.1 (q, 1C)

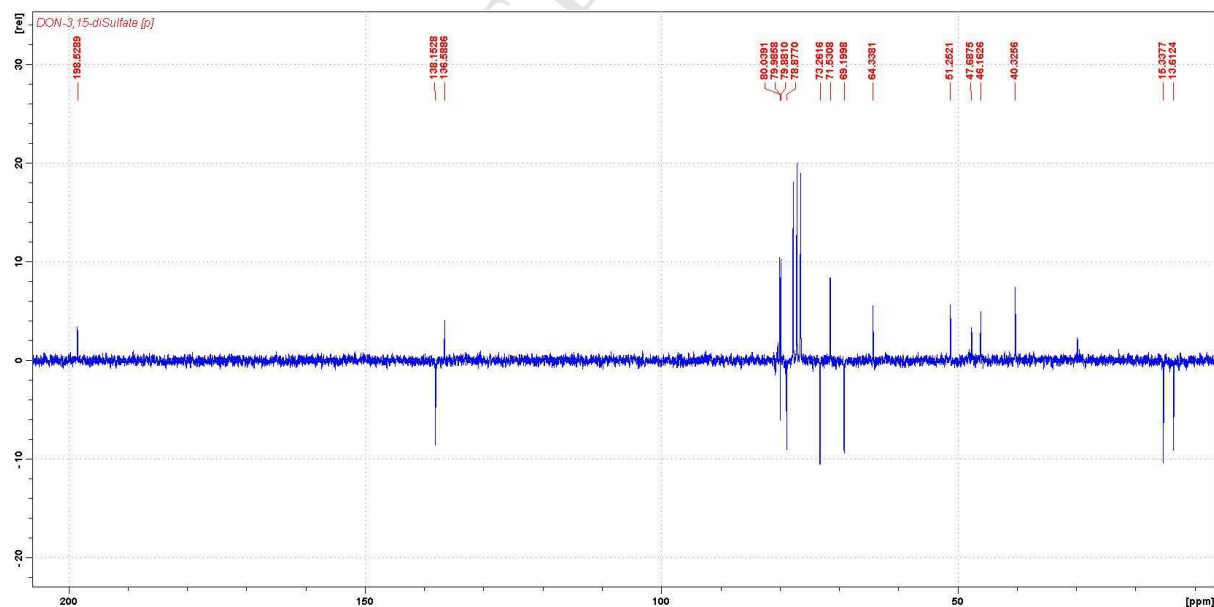


9.) bis(2,2,2-Trichloroethyl) DON-3,15-disulfate

^1H NMR (200 MHz, CDCl_3) δ 6.69 (dq, $J=5.9, 1.5$ Hz, 1H), 5.32 (dt, $J=11.1, 4.4$, 1H), 4.88 (s, 1H), 4.81 (dt, $J=5.7, 1.5$ Hz, 1H), 4.79 (s, 2H), 4.66 (d, $J=11.0$ Hz, 1H), 4.60 (d, $J=11.0$ Hz, 1H), 4.48 (s, 2H), 4.04 (d, $J=4.4$ Hz, 1H), 3.83 (b, 1H), 3.21 (d, $J=4.1$ Hz, 1H), 3.17 (d, $J=4.1$ Hz, 1H), 2.57 (dd, $J=15.7, 4.2$ Hz, 1H), 2.33 (dd, $J=15.7, 11.1$ Hz, 1H), 1.93 (b, 3H), 1.15 (s, 3H)

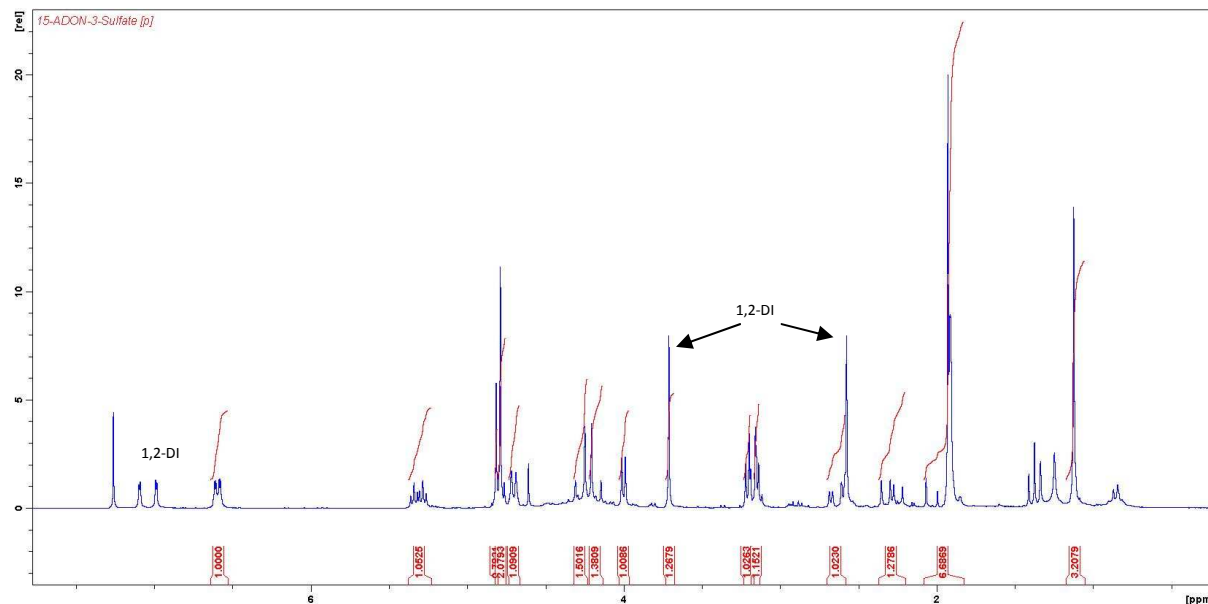


^{13}C -NMR (50 MHz, CDCl_3) δ 198.5 (s, 1C), 138.2 (d, 1C), 136.6 (s, 1C), 2 x 80.0 (d, 1C/t, 1C), 79.9 (t, 1C), 78.9 (d, 1C), 73.3 (d, 1C), 71.5 (t, 1C), 69.2 (d, 1C), 64.3 (s, 1C), 51.3 (s, 1C), 47.7 (t, 1C), 46.2 (s, 1C), 40.3 (t, 1C), 15.3 (q, 1C), 13.6 (q, 1C), 2 x CCl_3 between 92 and 93 ppm are missing, but the corresponding CH_2 groups are located at 80.0 and 79.9

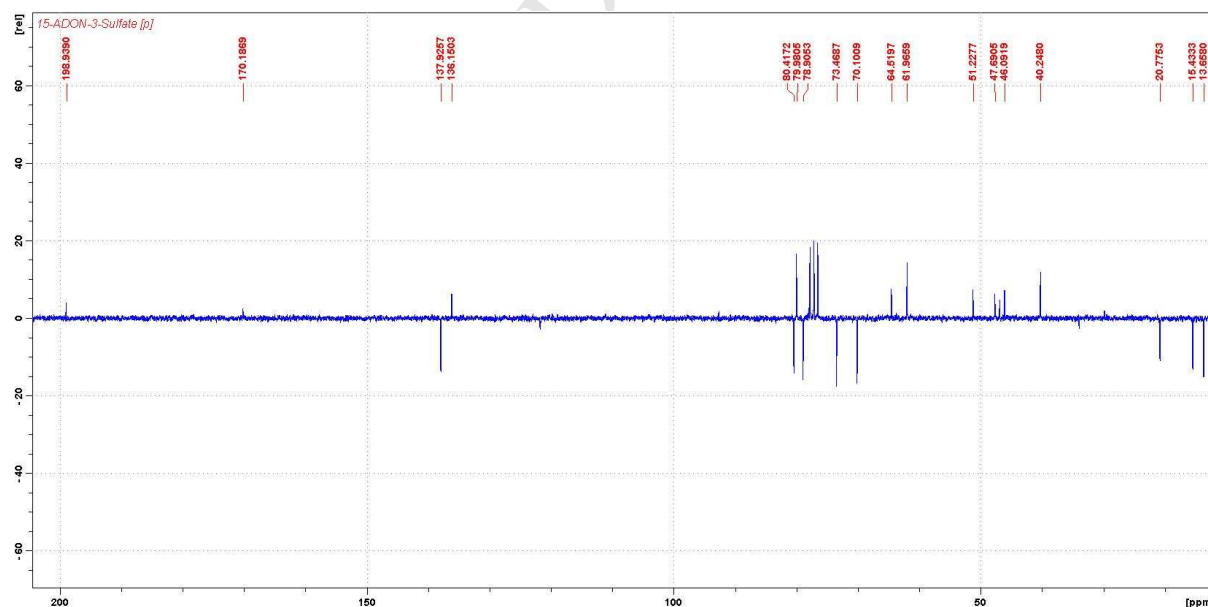


11.) 2,2,2-Trichloroethyl 15-acetyl-DON-3-sulfate

^1H NMR (200 MHz, CDCl_3) δ 6.60 (dq, $J=5.8, 1.5$ Hz, 1H), 5.31 (dt, $J=11.1, 4.3$, 1H), 4.81 (s, 1H), 4.79 (s, 2H), 4.70 (d, $J=5.8$ Hz, 1H), 4.27 (d, $J=12.1$ Hz, 1H), 4.18 (d, $J=12.1$ Hz, 1H), 4.00 (d, $J=4.3$ Hz, 1H), 3.71 (s, 1H), 3.21 (d, $J=4.1$ Hz, 1H), 3.15 (d, $J=4.1$ Hz, 1H), 2.64 (dd, $J=15.7, 4.3$ Hz, 1H), 2.29 (dd, $J=15.7, 11.1$ Hz, 1H), 1.93 (s, 3H), 1.91 (b, 3H), 1.12 (s, 3H); 1,2-DI = traces of 1,2-dimethylimidazole

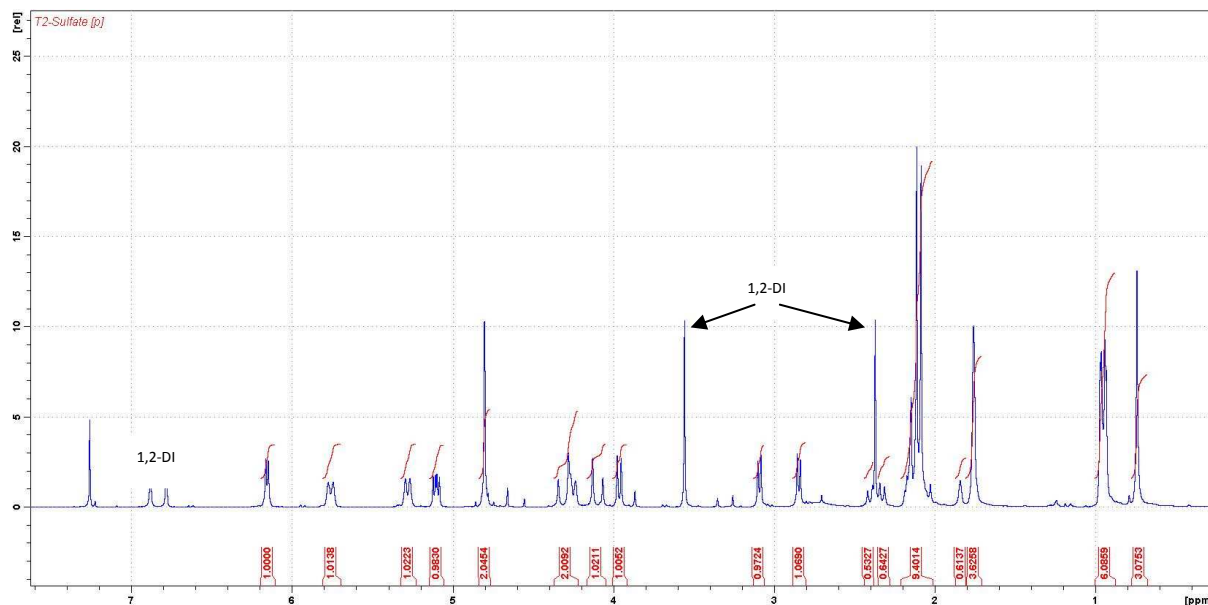


^{13}C -NMR (50 MHz, CDCl_3) δ 198.9 (s, 1C), 170.2 (s, 1C), 137.9 (d, 1C), 136.1 (s, 1C), 92.7 (s, 1C, $-\text{CCl}_3$ tiny signal!), 80.4 (d, 1C), 80.0 (t, 1C), 78.9 (d, 1C), 73.5 (d, 1C), 70.1 (d, 1C), 64.5 (s, 1C), 62.0 (t, 1C), 51.2 (s, 1C), 47.4 (t, 1C), 46.1 (s, 1C), 40.2 (t, 1C), 20.8 (q, 1C), 15.4 (q, 1C), 13.7 (q, 1C)

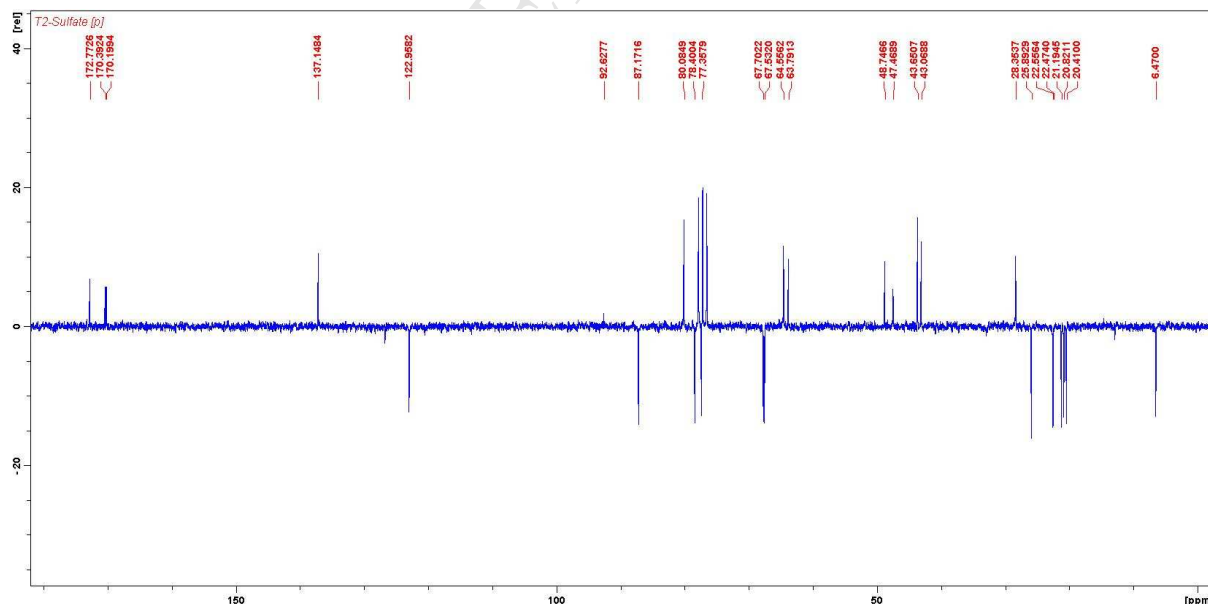


21.) 2,2,2-Trichloroethyl T2-toxin-3-sulfate

^1H NMR (200 MHz, CDCl_3) δ 6.16 (d, $J=3.1$ Hz, 1H), 5.77 (dt, $J=5.7, 1.4$ Hz, 1H), 5.28 (d, $J=5.5$ Hz, 1H), 5.10 (dd, $J=4.9, 3.1$ Hz, 1H), 4.80 (s, 2H), 4.31 (d, $J=12.7$ Hz, 1H), 4.25 (d, $J=5.7$ Hz, 1H), 4.10 (d, $J=12.7$ Hz, 1H), 3.96 (d, $J=4.9$ Hz, 1H), 3.09 (d, $J=3.9$ Hz, 1H), 2.85 (d, $J=3.9$ Hz, 1H), 2.35 (dd, $J=15.2, 6.0$ Hz, 1H), 2.11 (s, 3H), 2.09 (s, 3H), 2.00-2.25 (m, 3H), 1.80 (d, $J=16.6$ Hz, 1H), 1.76 (s, 3H), 0.96 (d, $J=6.3$ Hz, 3H), 0.95 (d, $J=6.3$ Hz, 3H), 0.74 (s, 3H); 1,2-DI = traces of 1,2-dimethylimidazole

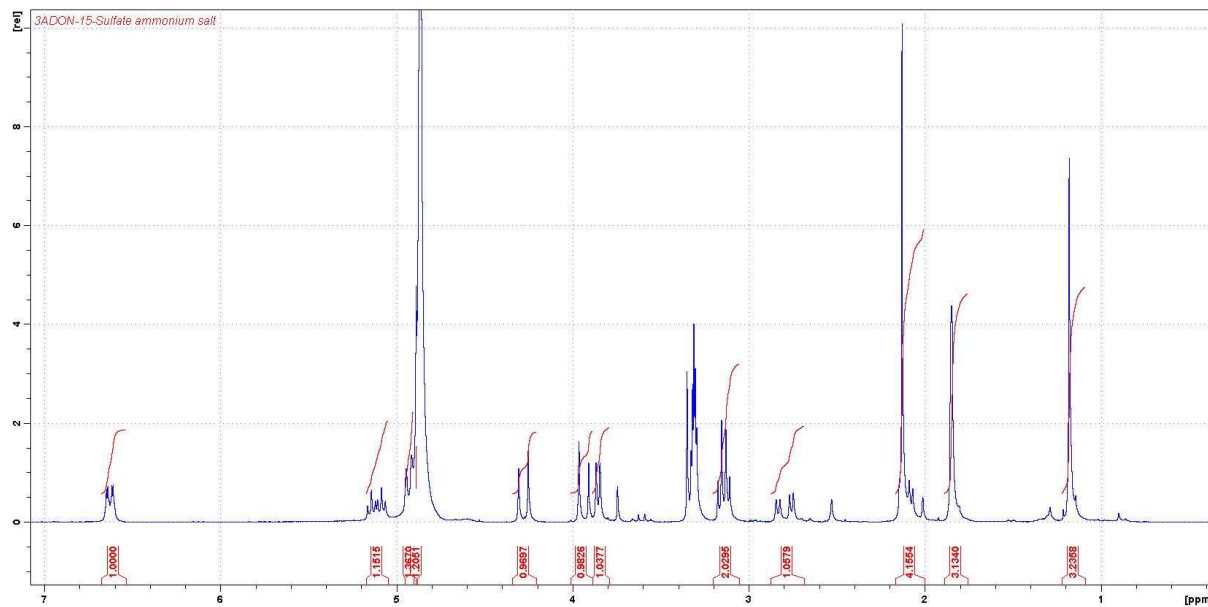


^{13}C NMR (50 MHz, CDCl_3) δ 172.8 (s, 1C), 170.4 (s, 1C), 170.2 (s, 1C), 137.1 (s, 1C), 123.0 (d, 1C), 92.6 (s, 1C), 87.1 (d, 1C), 80.1 (t, 1C), 78.4 (d, 1C), 77.4 (d, 1C), 67.7 (d, 1C), 67.5 (d, 1C), 64.6 (t, 1C), 63.8 (s, 1C), 48.7 & 47.5 (1t, 1s, 2x1C), 43.7 (t, 1C), 43.1 (s, 1C), 28.4 (t, 1C), 25.9 (d, 1C), 22.6 (q, 1C), 22.5 (q, 1C), 21.2 (q, 1C), 20.8 (q, 1C), 20.4 (q, 1C), 6.5 (q, 1C)

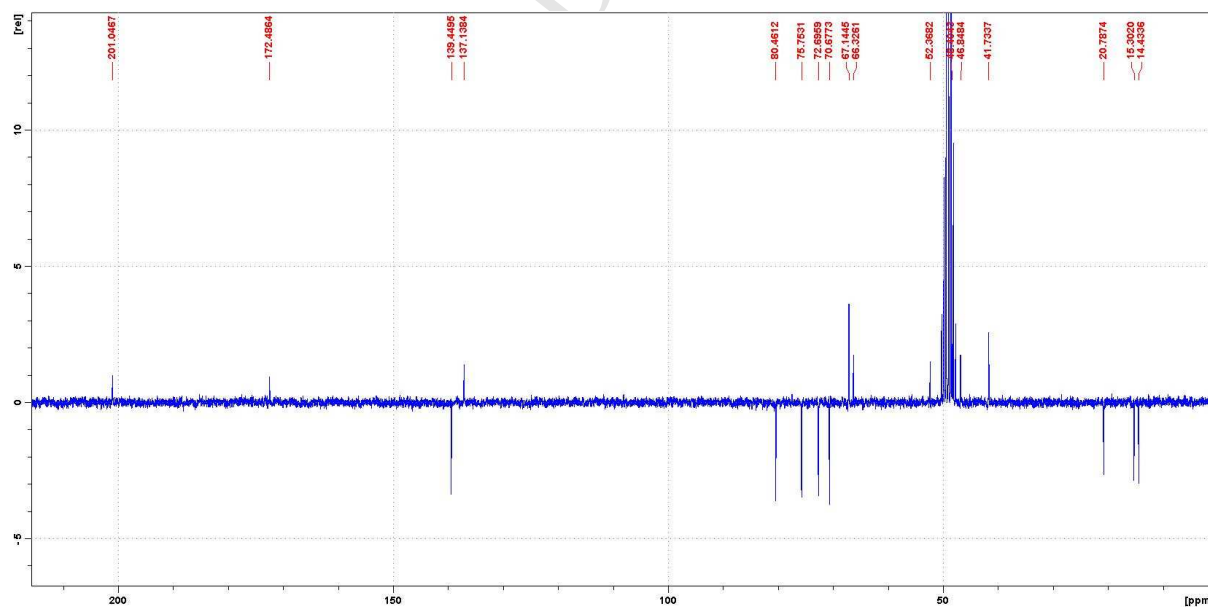


13.) 3-Acetyl-DON-15-sulfate, ammonium salt

^1H NMR (200 MHz, methanol- d_4) δ 6.63 (dq, $J=6.1, 1.4$ Hz, 1H), 5.11 (dt, $J=11.3, 4.4$ Hz, 1H), 4.92 (d, $J=6.1$ Hz, 1H), 4.89 (s, 1H), 4.87 (b, NH_4^+ , H_2O) 4.27 (d, $J=11.0$ Hz, 1H), 3.94 (d, $J=11.1$ Hz, 1H), 3.85 (d, $J=4.5$ Hz, 1H), 3.16 (d, $J=4.3$ Hz, 1H), 3.12 (d, $J=4.3$ Hz, 1H), 2.79 (dd, $J=15.3, 4.3$ Hz, 1H), 2.08 (dd, $J=15.3, 11.3$ Hz, 1H), 2.13 (s, 3H), 1.85 (b, 3H), 1.18 (s, 3H)

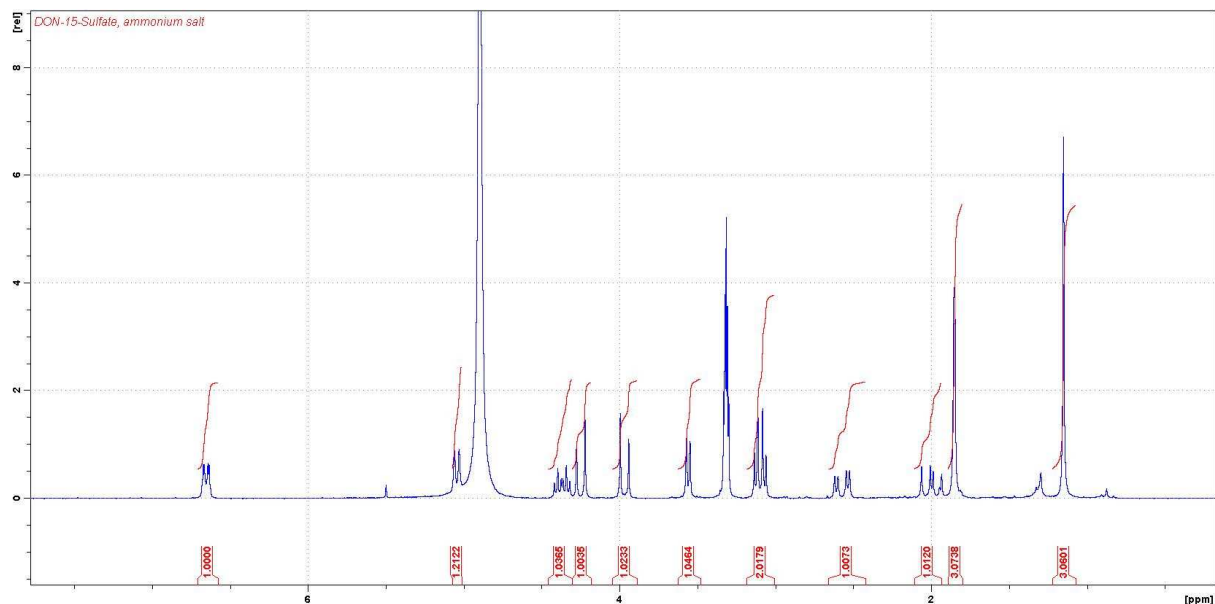


^{13}C NMR (50 MHz, methanol- d_4) δ 201.0 (s, 1C), 172.5 (s, 1C), 139.4 (d, 1C), 137.1 (s, 1C), 80.5 (d, 1C), 75.8 (d, 1C), 72.7 (d, 1C), 70.7 (d, 1C), 67.1 (t, 1C), 66.3 (s, 1C), 52.3 (s, 1C), 48.4 (t, 1C), 46.8 (s, 1C), 41.7 (t, 1C), 20.8 (q, 1C), 15.3 (q, 1C), 14.4 (q, 1C)

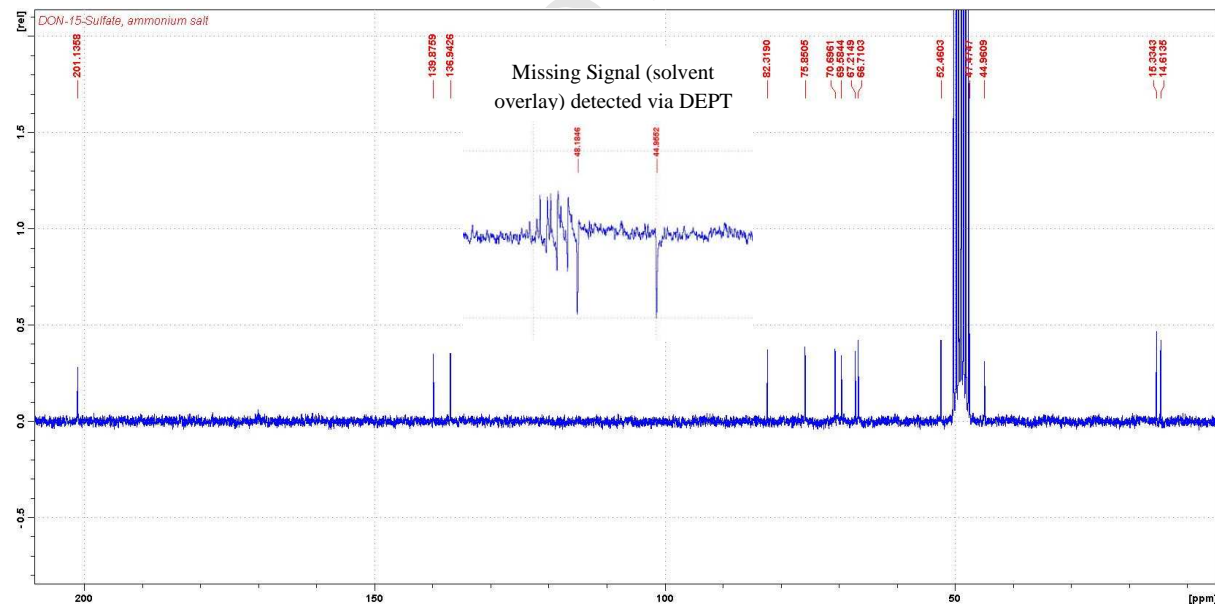


14.) DON-15-sulfate, ammonium salt

^1H NMR (200 MHz, methanol- d_4) δ 6.65 (dq, $J=6.1$, 1.4 Hz, 1H), 5.04 (d, $J=6.1$ Hz, 1H), 4.85-4.95 (m, NH_4^+ , C7-H, H_2O), 4.76 (dt, $J=11.1$, 4.5, 1H), 4.25 (d, $J=11.0$ Hz, 1H), 3.96 (d, $J=11.0$ Hz, 1H), 3.55 (d, $J=4.5$ Hz, 1H), 3.12 (d, $J=4.5$ Hz, 1H), 3.06 (d, $J=4.5$ Hz, 1H), 2.57 (dd, $J=14.8$, 4.4 Hz, 1H), 1.99 (dd, $J=14.8$, 11.1 Hz, 1H), 1.84 (b, 3H), 1.14 (s, 3H)

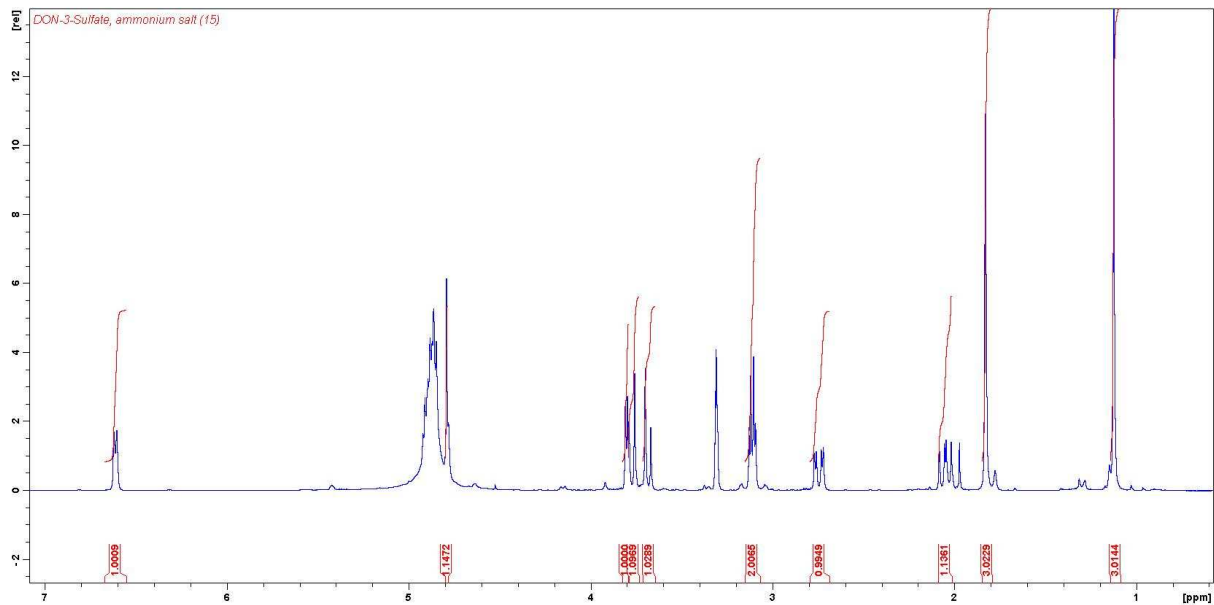


^{13}C NMR (50 MHz, methanol- d_4) δ 201.1 (s, 1C), 139.9 (d, 1C), 136.9 (s, 1C), 82.3 (d, 1C), 75.9 (d, 1C), 70.7 (d, 1C), 69.6 (d, 1C), 67.2 (s, 1C), 66.7 (t, 1C), 52.5 (s, 1C), 48.2 (t, 1C), 47.5 (s, 1C), 45.0 (t, 1C), 15.3 (q, 1C), 14.4 (q, 1C)

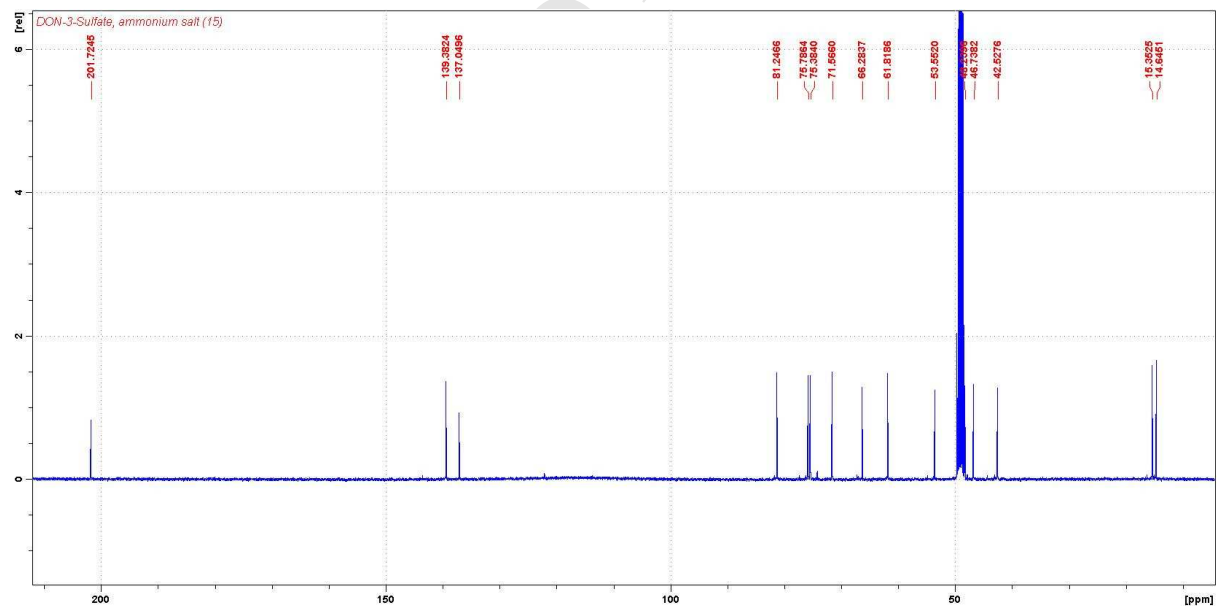


15.) DON-3-sulfate, ammonium salt

^1H NMR (400 MHz, methanol- d_4) δ 6.61 (dq, $J=6.1, 1.4$ Hz, 1H), 4.75-4.95 (m, NH_4^+ , C3-H, C11-H, H_2O), 4.79 (s, 1H), 3.80 (d, $J=4.4$ Hz, 1H), 3.78 (d, $J=12.3$ Hz, 1H), 3.68 (d, $J=12.3$ Hz, 1H), 3.12 (d, $J=4.5$ Hz, 1H), 3.09 (d, $J=4.5$ Hz, 1H), 2.75 (dd, $J=15.2, 4.4$ Hz, 1H), 2.06 (dd, $J=15.2, 11.4$ Hz, 1H), 1.83 (b, 3H), 1.12 (s, 3H)

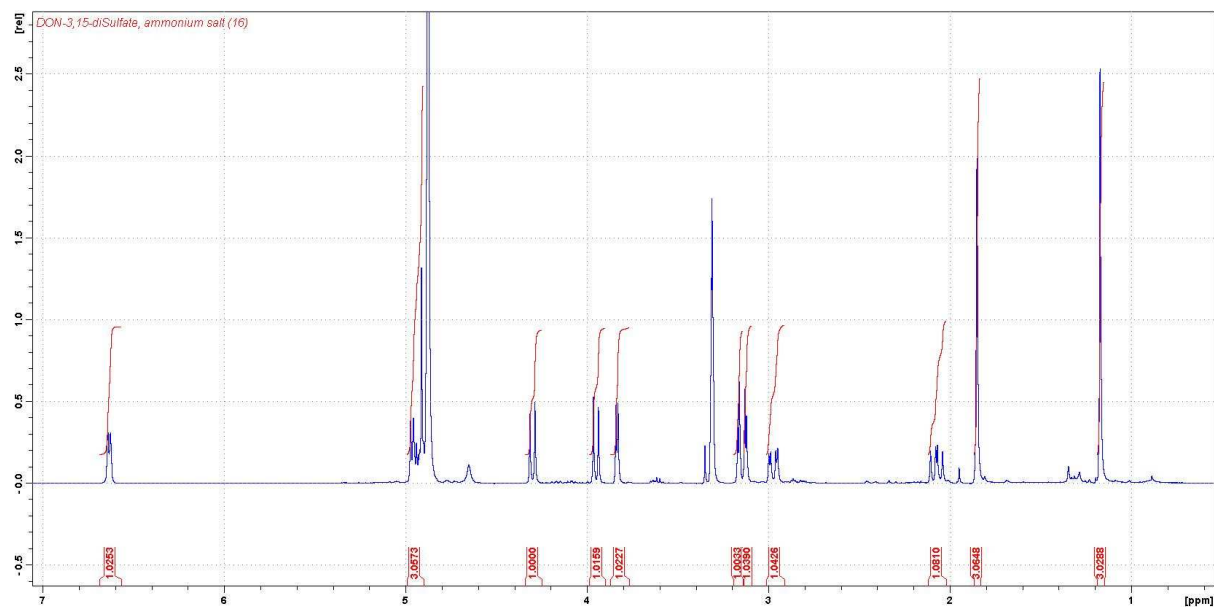


^{13}C NMR (100 MHz, methanol- d_4) δ 201.7 (s, 1C), 139.4 (d, 1C), 137.0 (s, 1C), 81.2 (d, 1C), 75.8 (d, 1C), 75.4 (d, 1C), 71.6 (d, 1C), 66.3 (s, 1C), 61.8 (t, 1C), 53.6 (s, 1C), 48.2 (t, 1C), 46.7 (s, 1C), 42.5 (t, 1C), 15.4 (q, 1C), 14.6 (q, 1C)

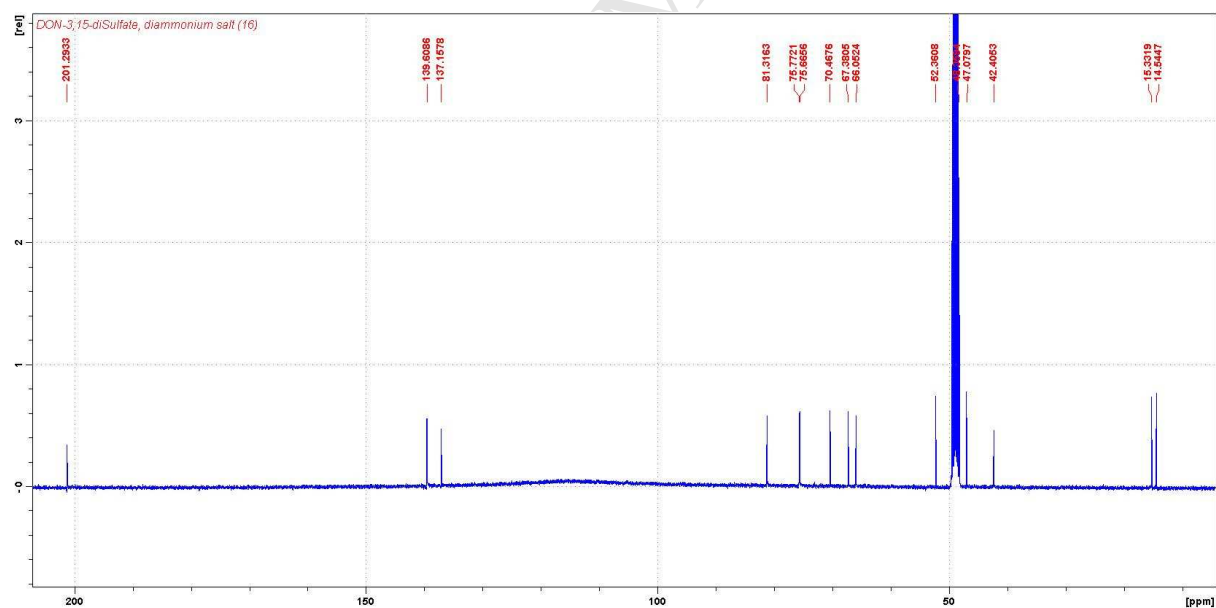


16.) DON-3,15-disulfate, diammonium salt

^1H NMR (400 MHz, methanol- d_4) δ 6.67 (dq, $J=6.0, 1.5$ Hz, 1H), 4.80-5.00 (m, NH_4^+ , C3-H, C7-H, C11-H, H_2O), 4.24 (d, $J=10.9$ Hz, 1H), 3.98 (d, $J=10.9$ Hz, 1H), 3.84 (d, $J=4.5$ Hz, 1H), 3.15 (d, $J=4.3$ Hz, 1H), 3.10 (d, $J=4.3$ Hz, 1H), 2.97 (dd, $J=15.3, 4.5$ Hz, 1H), 2.10 (dd, $J=15.3, 11.4$ Hz, 1H), 1.85 (b, 3H), 1.16 (s, 3H)

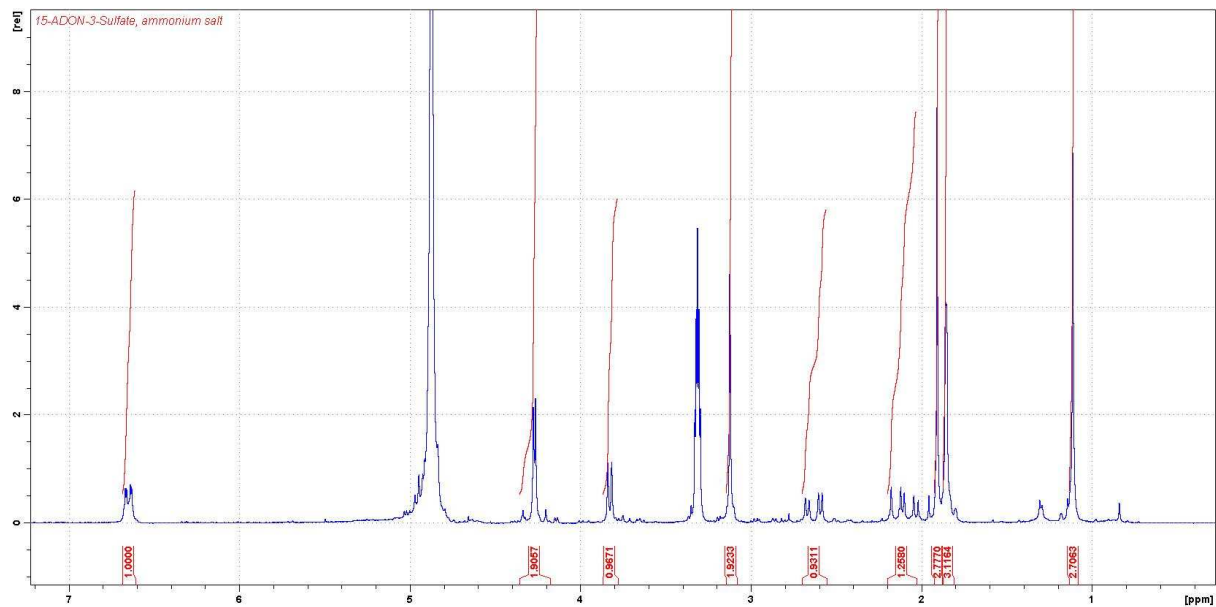


^{13}C NMR (100 MHz, methanol- d_4) δ 201.3 (s, 1C), 139.6 (d, 1C), 137.2 (s, 1C), 81.3 (d, 1C), 75.8 (d, 1C), 75.7 (d, 1C), 70.5 (d, 1C), 67.4 (s, 1C), 66.1 (t, 1C), 52.4 (s, 1C), 48.4 (t, 1C), 47.1 (s, 1C), 42.4 (t, 1C), 15.3 (q, 1C), 14.5 (q, 1C)

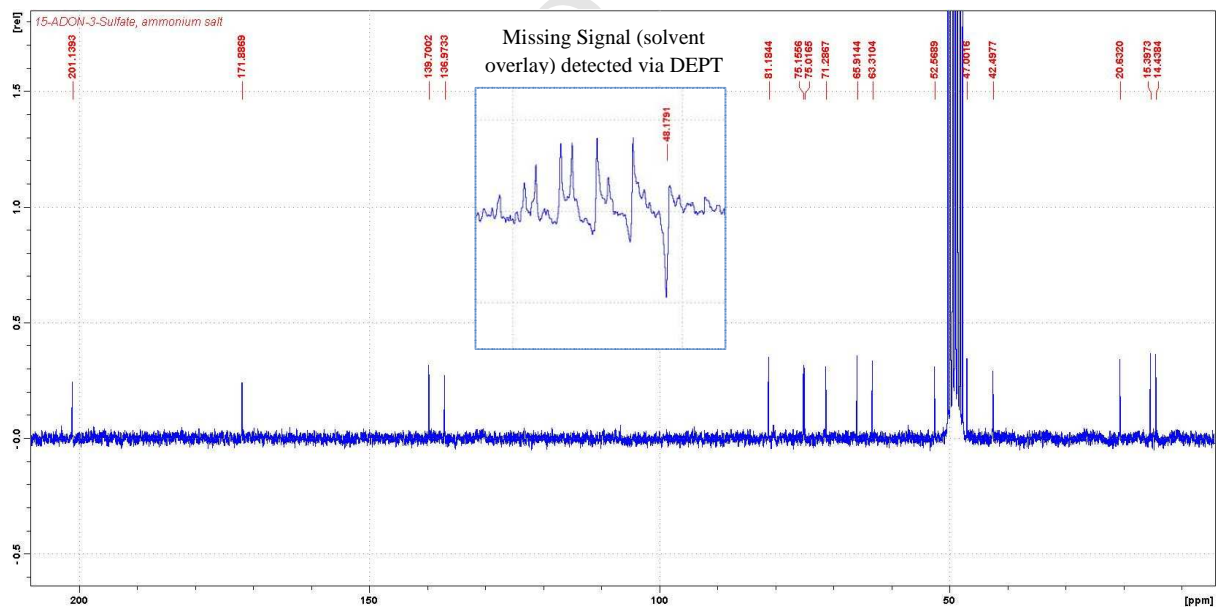


18.) 15-Acetyl-DON-3-sulfate, ammonium salt

^1H NMR (400 MHz, methanol- d_4) δ 6.65 (dq, $J=5.9, 1.5$ Hz, 1H), 4.70-5.10 (m, NH_4^+ , C3-H, C7-H, C11-H, H_2O), 4.30 (d, $J=12.1$ Hz, 1H), 4.23 (d, $J=12.1$ Hz, 1H), 3.82 (d, $J=4.5$ Hz, 1H), 3.12 (s, 2H), 2.63 (dd, $J=15.3, 4.3$ Hz, 1H), 2.11 (dd, $J=15.3, 11.2$ Hz, 1H), 1.90 (s, 3H), 1.85 (b, 3H), 1.11 (s, 3H)

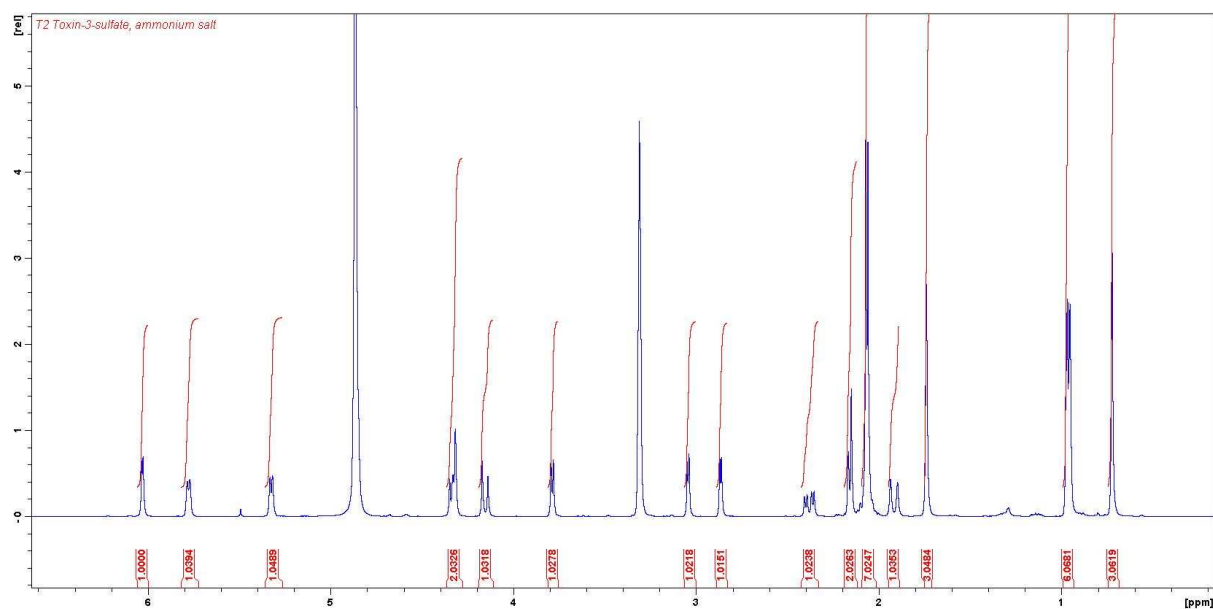


^{13}C -NMR (100 MHz, methanol- d_4) δ 201.1 (s, 1C), 171.9 (s, 1C), 139.7 (d, 1C), 137.0 (s, 1C), 81.2 (d, 1C), 75.2 (d, 1C), 75.0 (d, 1C), 71.3 (d, 1C), 65.9 (s, 1C), 63.3 (t, 1C), 52.6 (s, 1C), 48.2 (t, 1C), 47.0 (s, 1C), 42.5 (t, 1C), 20.6 (q, 1C), 15.4 (q, 1C), 14.4 (q, 1C)

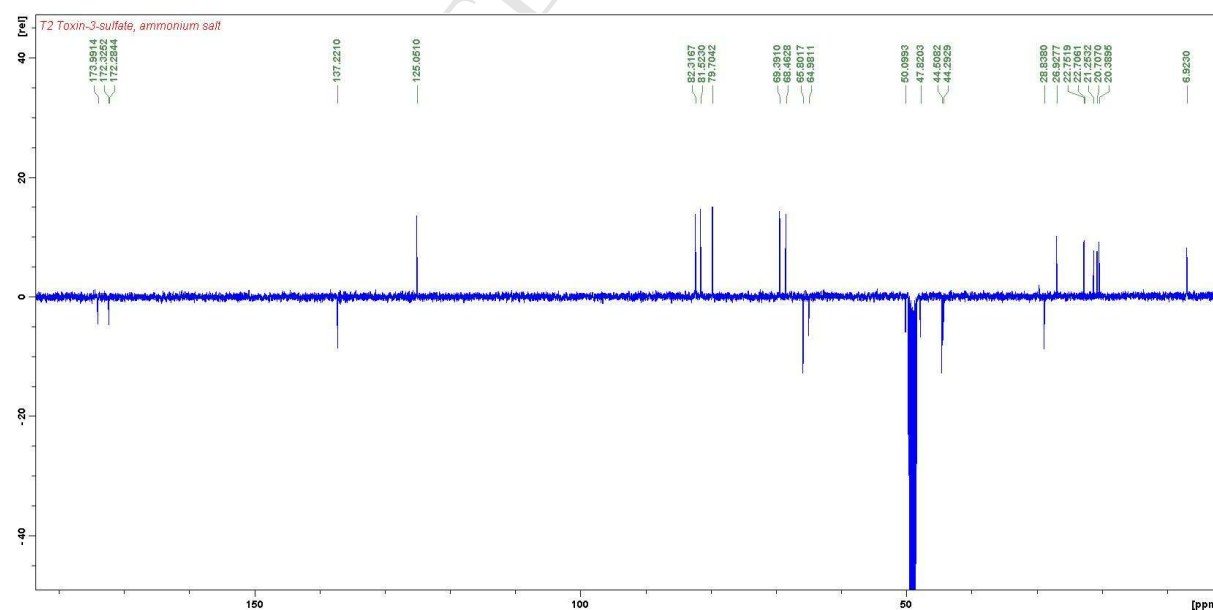


22.) T2-toxin-3-sulfate, ammonium salt

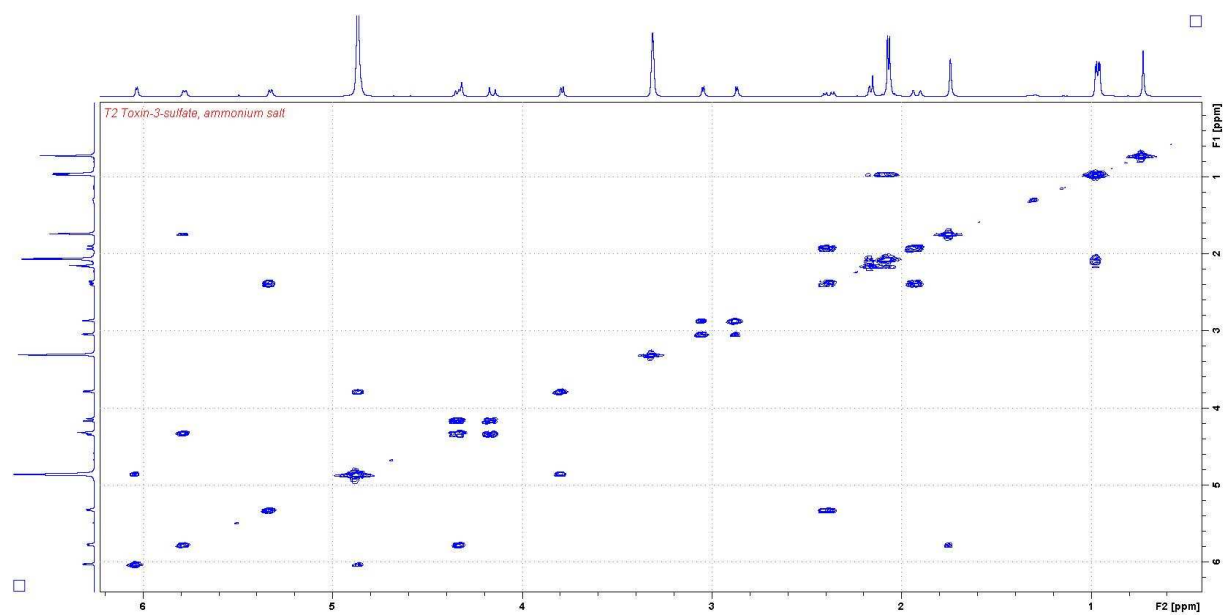
^1H NMR (400 MHz, methanol- d_4) δ 6.03 (d, $J=3.1$ Hz, 1H), 5.78 (dt, $J=5.5$ Hz, 1H), 5.33 (d, $J=5.5$ Hz, 1H), 4.80-4.94 (m, NH_4^+ , C3-H, H_2O), 4.33 (d, $J=12.1$ Hz, 1H), 4.32 (d, $J=5.5$ Hz, 1H), 4.16 (d, $J=12.1$ Hz, 1H), 3.79 (d, $J=5.1$ Hz, 1H), 3.04 (d, $J=3.9$ Hz, 1H), 2.87 (d, $J=3.9$ Hz, 1H), 2.38 (dd, $J=15.3, 5.5$ Hz, 1H), 2.13-2.18 (m, 2H), 2.07 (s, 3H), 2.06 (s, 3H), 2.00-2.12 (m, 1H), 1.92 (d, $J=15.3$ Hz, 1H), 1.74 (s, 3H), 0.97 (d, $J=6.7$ Hz, 3H), 0.96 (d, $J=6.7$ Hz, 3H), 0.72 (s, 3H)



^{13}C NMR (100 MHz, methanol- d_4) δ 174.0 (s, 1C), 172.3 (s, 1C), 172.2 (s, 1C), 137.2 (s, 1C), 125.1 (d, 1C), 82.3 (d, 1C), 81.5 (d, 1C), 79.7 (d, 1C), 69.4 (d, 1C), 68.5 (d, 1C), 65.8 (t, 1C), 65.0 (s, 1C), 50.1 & 47.8 (1t, 1s, 2x1C), 44.5 (t, 1C), 44.3 (s, 1C), 28.8 (t, 1C), 26.9 (d, 1C), 22.8 (q, 1C), 22.7 (q, 1C), 21.3 (q, 1C), 20.7 (q, 1C), 20.4 (q, 1C), 6.9 (q, 1C)



22.) T2-toxin-3-sulfate, ammonium salt - COSY



22.) T2-toxin-3-sulfate, ammonium salt - HMBC

