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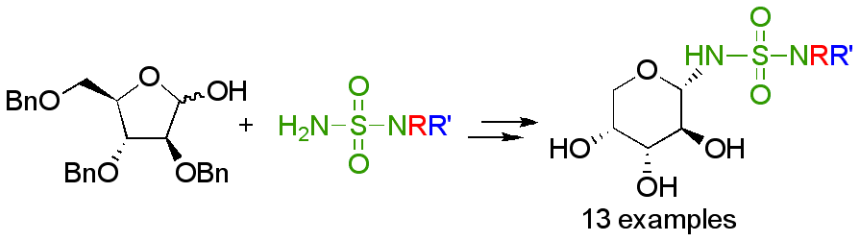

anti-mycobacterial activity at 62 μg/mL for R=$(CH_2)_9CH_3$, R' = H

Synthesis of arabinose glycosyl sulfamides as potential inhibitors of mycobacterial cell wall biosynthesis

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Abstract - A series of arabinose glycosyl sulfamides with varying alkyl chain types and lengths were synthesised as mimics of decaprenolphosphoarabinose (DPA), and as potential inhibitors of mycobacterial cell wall biosynthesis. Unprecedented conversion of the desired furanose to the thermodynamically more stable pyranose form occurred during final de-protection. Biological testing against *Mycobacterium smegmatis* revealed low to moderate anti-mycobacterial activity with marked dependence on alkyl chain length, which in the case of mono-substituted sulfamides was maximal for a C-10 chain.

Keywords: carbohydrates, sulfamides, furanosides, pyranosides, arabinose, mycobacteria, tuberculosis

1. Introduction

Mycobacterium tuberculosis, the bacterium responsible for tuberculosis[1] (TB), is probably the most prevalent of the pathogenic strains of mycobacteria. Tuberculosis has recently re-emerged as a significant threat to human health,[2] and although there are a number of antibiotics available to fight TB infection, some strains have already developed resistance.[3] The development of new therapeutic agents against TB has therefore become the objective of multiple drug discovery programs.

The cell walls of mycobacteria are complex, and contain two polysaccharides, lipoarabinomannan (LAM) and arabinogalactan (AG), the structures of which are unique to mycobacteria, and which are crucial to mycobacterial viability. Disruption of mycobacterial cell wall biosynthesis[4] by inhibition of the assembly of these polysaccharides represents a therapeutic opportunity for the development of novel and selective anti-TB agents. Previous approaches have included attempted inhibition of various glycosyl transferases,[5] and of the Gal*p* / Gal*f* mutase enzyme responsible for a crucial pyranose / furanose isomerisation during assembly of the galactan cell wall component.[6] Attention has also focussed on attempted inhibition of the biosynthesis of mycobacterial arabinan,[7] which is assembled stepwise by arabinosyl transferases[8] that use decaprenol phophoarabinose **1** (DPA, Figure 1) as their donor substrate. Metabolically stable analogues of DPA may inhibit arabinan biosynthesis, and therefore may compromise mycobacterial viability.

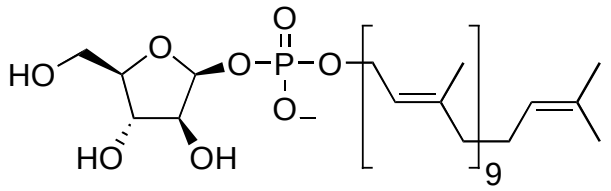

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Figure 1. Structure of decaprenolphophoarabinose **1** (DPA).

Besides the arabinose monosaccharide, DPA contains both a highly polar and labile glycosyl phosphate and a long hydrophobic polyprenol side chain. One may postulate that effective and stable mimics of DPA should therefore contain both a suitable isosteric replacement for the phosphate,[9] and a hydrophobic side chain. Significant work on the synthesis of mimics of DPA has already been reported, for example by Lowary,[7c-e] including the synthesis of β-*C*-glycosyl sulfones[10] and *C*-phosphonates of arabinofuranose. In a related vein Von Itzstein reported the synthesis and bioactivity of a variety of galactofuranosyl alkyl thioglycosides, glycosyl sulfones, sulfenamides and sulfonamides[11] which were presumed to act as inhibitors of the biosynthesis of galactofuranose components of the mycobacterial cell wall. More recent studies have also revealed that 1,2-*trans* alkyl galactofuranosides also display anti-mycobacterial activity.[12] Previous studies from this laboratory have included the synthesis of a series of glycosyl sulfones[13] and triazoles[14] of arabinofuranose, which were decorated with various hydrophobic side chains. In the former study systematic variation of the alkyl chain length revealed that the C-12 compound was the most active, as demonstrated by an MIC against *M. Bovis BCG* of 62 μg/mL. In continuation of this initial study we sought to augment the moderate biological activity previously observed by the synthesis of a

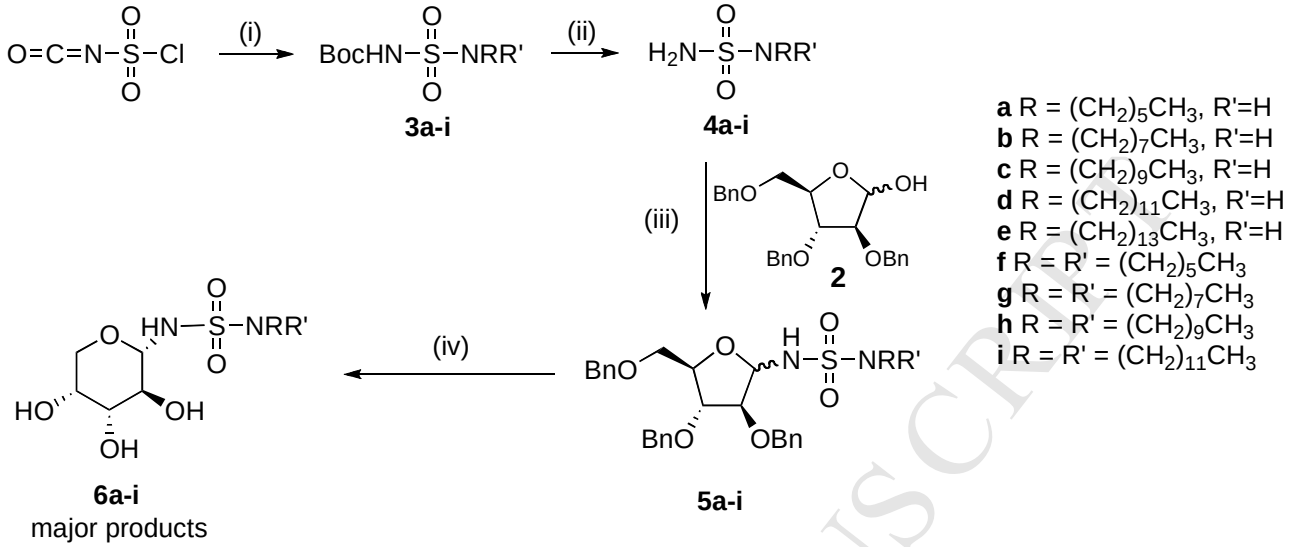
better isostere of phosphate. Although the synthesis and biological activity of sulfonamides[15] and sulfamates[16] have been widely reported, there is only a single report on the use of sulfamide as an isosteric replacement for phosphate in the search for anti-tubercular agents.[17] In contrast the sulfamide functional group has previously found several other applications as an isostere in medicinal chemistry.[18] We considered that the polarity, the availability of heteroatoms for hydrogen bonding and the tetrahedral nature of the central sulfur atom all reinforced our idea that glycosyl sulfamides[19] may be useful mimics of glycosyl phosphates. This paper reports the synthesis of a series of arabinose glycosyl sulfamides as putative mimics of DPA, together with their anti-bacterial activity as observed in an Alamar Blue microplate assay against *Mycobacterium smegmatis.*

2. Results and discussion

2.1 Synthesis

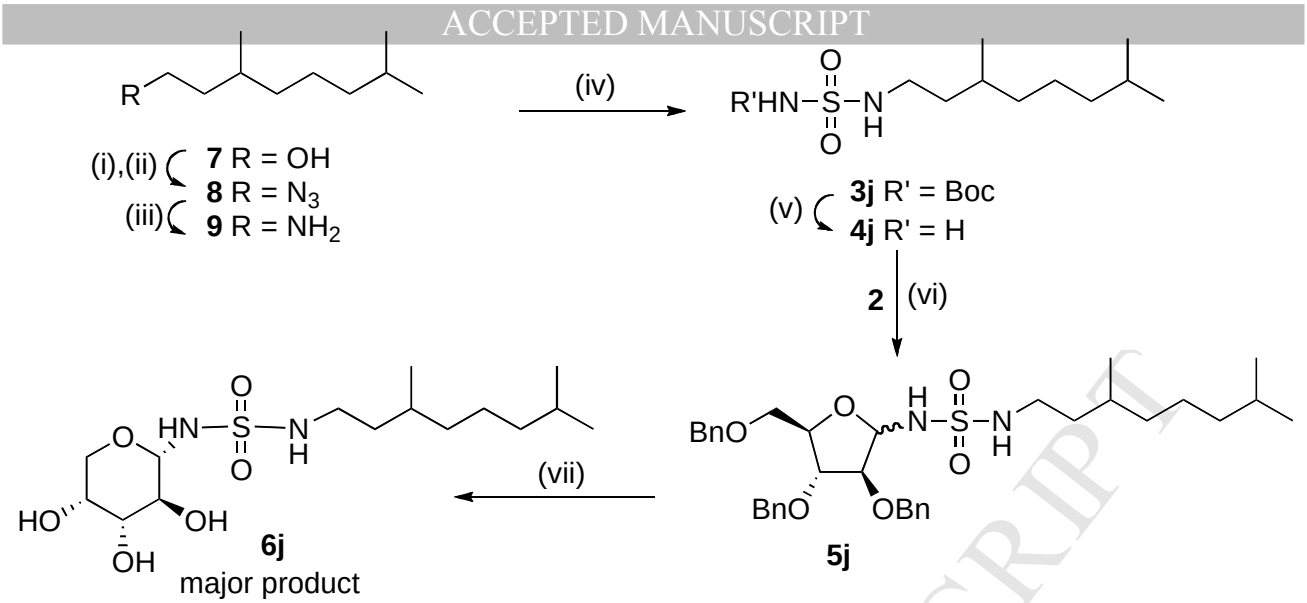
The convergent assembly of a series of arabinose glycosyl sulfamides was envisaged by glycosylation of the protected arabinofuranose donor **2** with a series of sulfamide acceptors bearing a variety of hydrophobic side chains. Sulfamides bearing linear alkyl side chains of varying length were synthesised as follows (Scheme 1). Reaction of chlorosulfonyl isocyanate with *tert*-butanol produced an intermediate sulfonyl chloride that was then directly reacted with a variety of commercially available straight chain primary amines, to produce the Boc-protected sulfamides **3a-e**, containing side chains with 6-14 carbon atoms. Similarly the use of secondary amines allowed the synthesis of the di-substituted sulfamides **3f-i**, containing side chains with 6-12 carbon atoms. Acid catalysed Boc removal by treatment with trifluoroacetic acid (TFA) then furnished the required sulfamides **4a-i**. Trimethylsilyl trifluoromethanesulfonate (TMSOTf) catalysed condensation of these sulfamides with the furanose hemiacetal **2**, synthesised as previously described,[20] produced furanose sulfamides **5a-i**, in each case as an inseparable mixture of anomers. Variation of the glycosylation conditions and the use of other donors was attempted in order to try and improve the stereoselectivity of this process until it was eventually realised that the product sulfamides **5a-i** in fact underwent mutarotation.[21] The liability of glycosyl sulfamides to ring-opening processes became obvious when the furanose sulfamides **5a-i** were de-protected by catalytic hydrogenation in the presence of Pd on carbon in MeOH. Surprisingly the major products from these reactions were identified as the α-pyranose anomers **6a-i**. Subsequent investigations[21] revealed that glycosyl sulfamides, along with *N*-glycosyl sulfonamides and sulfamates, all undergo mutarotation and furanose-pyranose equilibration in aqueous solution. In the cases of the arabinose materials investigated the α-pyranose isomers were demonstrated to be the thermodynamically

preferred products, and were shown to adopt $^1C_4$ conformations, with the anomeric nitrogen and OH-2 and OH-3 were equatorial, and in which the operation of an exo anomeric effect was evident.



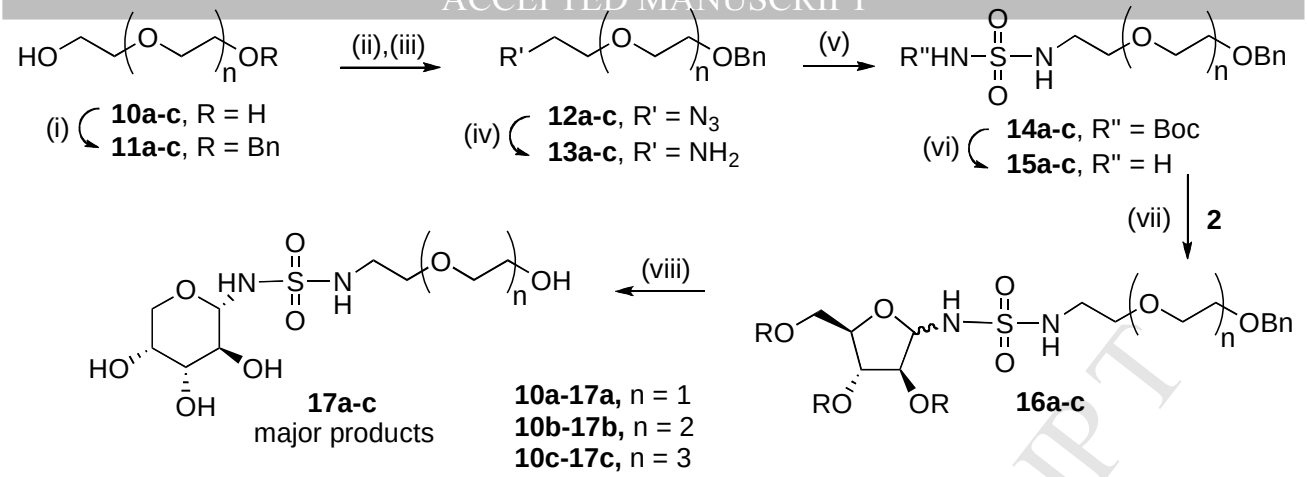
Scheme 1. *Reagents and conditions:* (i) ^{t}BuOH, CH_2Cl_2, 0 °C, 30 min., then add RR'NH, rt, 16 h; **3a** 80%, **3b** 42%, **3c** 41%, **3d** 28%, **3e** 33%, **3f** 70%, **3g** 60%, **3h** 50%, **3i** 53%; (ii) TFA, CH_2Cl_2, rt, 16 h, **4a** 86%, **4b** 69%, **4c** 76%, **4d** 68%, **4e** 75%, **4f** 92%, **4g** 76%, **4h** 66%, **4i** 78%; (iii) **2**, TMSOTf, CH_2Cl_2, rt, 16 h; **5a** 52%, **5b** 54%, **5c** 77%, **5d** 71%, **5e** 54%, **5f** 78%, **5g** 68%, **5h** 59%, **5i** 47%; (iv) H_2, Pd/C, MeOH, rt, 16 h; **6a** 42%, **6b** 36%, **6c** 45%, **6d** 40%, **6e** 47%, **6f** 41%, **6g** 45%, **6h** 41%, **6i** 39%.

In order to access glycosyl sulfamides that possessed a side chains with methyl branch points, which may better mimic the polyprenol chain of DPA, various possibilities were investigated. Although the use of geraniol as a substrate was initially investigated, difficulties encountered with both sigmatropic rearrangement[22] of the intermediate allylic azide and the chemoselective removal of benzyl protecting groups in the presence of the side chain alkenes meant that attention shifted towards the production of a mimic containing a fully saturated side chain. Thus 3,7-dimethyl octanol **7** was mesylated (Scheme 2), and reacted with sodium azide to give **8**, which was then reduced to the amine **9** by a Staudinger reaction. Conversion to the Boc-protected sulfamide **3j**, Boc removal with TFA, and TMSOTf mediated condensation with donor **2** furnished the furanose sulfamide **5j**, again as an inseparable α/β mixture of anomers. Finally removal of the benzyl protecting groups by catalytic hydrogenation produced a mixture of compounds, from which the α-pyranose anomer **6j** was isolated as the major component.



Scheme 2. *Reagents and conditions:* (i) MsCl, Et_3N, CH_2Cl_2, rt, 2 h; (ii) NaN_3, Bu_4NBr, THF, 40 °C, 48 h, 75% over two steps; (iii) Ph_3P, H_2O, THF, rt, 16 h, 63%; (iv) chlorosulfonyl isocyanate, ^{t}BuOH, CH_2Cl_2, 0 °C, 30 min., then add **9**, rt, 16 h, 73%; (v) TFA, CH_2Cl_2, rt, 16 h, 98%; (vi) **2**, TMSOTf, CH_2Cl_2, rt, 16 h, 80%; (vii) H_2, Pd/C, MeOH, rt, 16 h, 43%.

In order to more produce putative mimics containing side chains of differing polarity, a variety of materials containing polyethylene glycol (PEG) side-chains were also synthesised. Three amine-terminated polyethers derived from di-, tri-, and tetra-ethylene glycol were synthesised (Scheme 3) by a sequence of monobenzylation mesylation, azide displacement and Staudinger reduction to yield amines **13a-c**. Addition of these amines to chlorosulfonyl isocyanate, pre-reacted with *tert*-butanol, produced the Boc-protected sulfamides **14a-c**. Removal of the Boc groups with TFA and TMSOTf mediated condensation with donor **2** gave the furanose glycosyl sulfamides **16a-c**, as inseparable mixtures of anomers. Finally removal of all benzyl protecting groups by catalytic hydrogenation was again accompanied by interconversion to the pyranose form, and following purification by RP-HPLC, the α-pyranose isomers **17a-c** were isolated as the major reaction products (Scheme 3).



Scheme 3. *Reagents and conditions:* (i) NaH, BnBr, THF, rt, 16 h; **11a** 25%, **11b** 36%, **11c** 65%; (ii) MsCl, Et_3N, CH_2Cl_2, rt, 2 h; (iii) NaN_3, Bu_4NBr, THF, 40 °C, 48 h; **12a** 94% over two steps, **12b** 98% over two steps, **11c** 91% over two steps; (iv) Ph_3P, H_2O, THF, rt, 16 h; **13a** 71%, **13b** 80%, **13c** 67%; (v) chlorosulfonyl isocyanate, ^tBuOH, CH_2Cl_2, 0 °C, 30 min., then add **9**, rt, 16 h; **14a** 93%, **14b** 39%, **14c** 91%; (vi) TFA, CH_2Cl_2, rt, 16 h; **15a** 88%, **15b** 76%, **15c** 65%; (vii) **2**, TMSOTf, CH_2Cl_2, rt, 16 h; **16a** 74%, **16b** 83%, **16c** 62%; (viii) H_2, Pd/C, MeOH, rt, 16 h; **17a** 46%, **17b** 42%, **17c** 46%.

2.2. Biological testing

Although the major isomers produced by the reaction sequences detailed above were all identified as the α-pyranose isomers, studies had shown that mutarotation and pyranose/furanose equilibration occurred under aqueous conditions.[21] The biological activity of the purified α-pyranose anomers was therefore assessed using the Alamar Blue microplate assay. The minimum inhibitory concentrations (MIC) of sulfamides **6a-j** and **17a-c** were measured in a series of tests on *M. smegmatis*; in these assays the Alamar Blue dye changes from the oxidized indigo blue (and non-fluorescent) form to the reduced pink (and fluorescent) form in the presence of growing bacteria (Fig. 1).[23] The results are shown in Table 1 and Figure 2.

Table 1. Inhibitory effects of deprotected glycosyl sulfamides **6a-i** and **17a-c** against *M. smegmatis*.

Compound	R	MIC[a] (μg/mL)
INH	-	4
4c	-	500
6a	$-(CH_2)_5CH_3$	> 1000
6b	$-(CH_2)_7CH_3$	500

6c	$-(CH_2)_9CH_3$	62
6d	$-(CH_2)_{11}CH_3$	250
6e	$-(CH_2)_{13}CH_3$	> 1000
6f	$-[(CH_2)_5CH_3]_2$	125
6g	$-[(CH_2)_7CH_3]_2$	500
6h	$-[(CH_2)_9CH_3]_2$	> 1000
6i	$-[(CH_2)_{11}CH_3]_2$	> 1000
6j	$-(CH_2)_2CH(CH_3)(CH_2)_3CH(CH_3)_2$	125
17a	$-CH_2CH_2OCH_2CH_2OH$	>1000
17b	$-(CH_2CH_2)_2OCH_2CH_2OH$	>1000
17c	$-(CH_2CH_2)_3OCH_2CH_2OH$	>1000

[a] MIC = minimum inhibitory concentration; the lowest concentration of the compound which inhibited the growth of *M. smegmatis* >90% in the Alamar Blue assay. Isoniazid (**INH,** MIC 4 μg/mL) and alkyl sulfamide **4c** were used as controls.

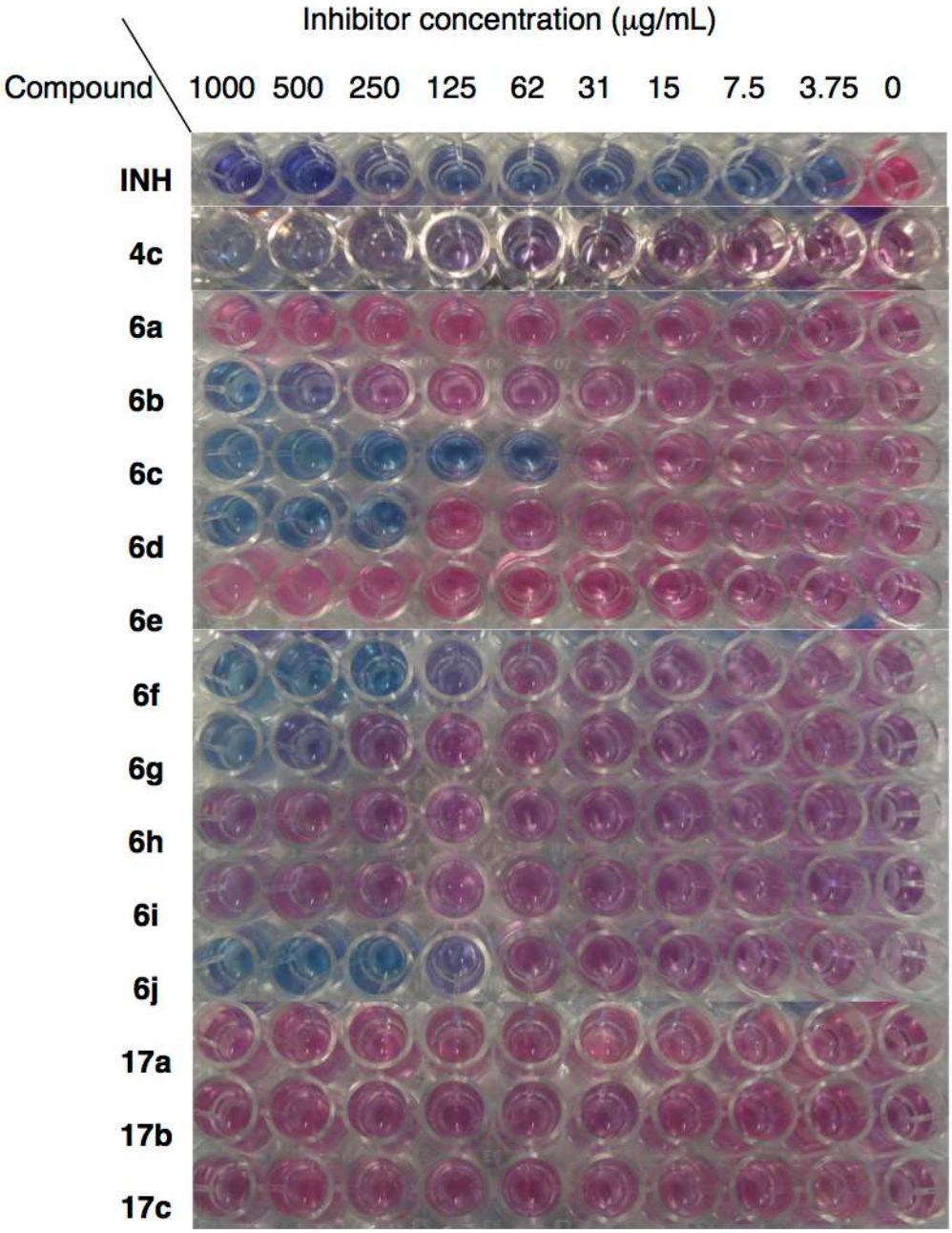


Figure 1. Alamar blue assay of *M. smegmatis* with de-protected glycosyl sulfamides **6a-j** and **17a-c**, and controls with isoniazid (**INH**), and alkyl sulfamide **4c**.

For sulfamides **6a-e**, which comprise a single alkyl chain of increasing length, the biological activity increased as side chain hydrophobicity was increased to reach a maximum for **6c** (MIC 62 μg/mL), which contains a chain of 10 carbon atoms. As the length of the carbon chain was further increased activity then decreased. The observed activity correlated with that of a series of glycosyl sulfones,[13] for which maximal activity was observed for a sulfone with a side chain containing 12 carbon atoms, i.e. a material with a total chain length of 13 atoms from the anomeric centre, as is

the case for **6c**. The activity of the di-alkylated sulfamides **6f-i** was significantly different. In these cases sulfamide **6f**, bearing two alkyl chains of 6 carbon atoms, was the most active (MIC 125 μg/mL), and increasing the chain length resulted in reduced activity (MIC >1000 μg/mL for **6h** and **6i**). Interestingly related work reported by von Itzstein[9] on galactofuranose S-glycosyl sulfonamides and sulfenamides identified the most active compounds as those containing branched side chains comprised two *n*-octyl alkyl units.

In this study the branched 3,7-dimethyloctyl sulfamide **6j** displayed an MIC of 125 μg/mL, and was thus more active than the straight chain octyl sulfamide **6b**. None of the sulfamides **17a-c**, containing PEG side chains, displayed any biological activity at the levels tested (MIC>1000 μg/mL). Finally in order to investigate the importance of the arabinose moiety, alkyl sulfamide **4c** was also assayed; this displayed an MIC of 500 μg/mL, as compared to the observed MIC of 62 μg/mL for the corresponding glycosyl sulfamide **6c**.

Any more precise analysis of these activities at this stage requires caution, though some generalised comments may be stated. Firstly it must be borne in mind that although the compounds in this and related studies were designed as mimics of DPA, their mode of action, for example as putative inhibitors of mycobacterial arabinosyl transferases, is as yet un-demonstrated. The fact that the activity of alkyl sulfamide **4c** was an order of magnitude lower than the corresponding glycosyl sulfamide **6c** does point to the importance of the carbohydrate portion of these compounds, but does not exclude another mode of action. Although it was found that these materials unexpectedly isomerised into the undesired pyranose form during the final de-protection steps, some of them did display moderate anti-mycobacterial activity, though the most potent compound **6c** was two orders of magnitude less active than isoniazid (**INH**). Our very recent studies have demonstrated that arabinose *N*-glycosyl sulfonamides, sulfamides and sulfamates all undergo mutatoration and pyranose equilibration in aqueous solution.[21] Therefore even though the materials submitted to the AB assays here were the pure α-pyranose sulfamides, other isomers would probably be formed during the course of the assay (typical assay length >20 h). In order to quantify the amount of isomerisation, HPLC analyses (see Supporting Information) were performed on compounds **6b** and **6c** under the AB assay conditions. These revealed that in each case, after 24h, only ~7% of the starting α-pyranose sulfamide had converted into a mixture of the β-pyranose and α-furanose forms (inseparable by HPLC); the majority of material (>92%) was still in the α-pyranose form. Although the amount of isomerisation is small, it must therefore be borne in mind that whilst the biological activities reported in Table 1 refer to assays of purified α-pyranose compounds, at the end of these assays minor amounts of the β-pyranose and α-furanose compounds were also present.

The fact that none of the materials possessing PEG side chains (sulfamides **17a-c**) displayed any anti-mycobacterial activity may be due to the increased hydrophilic nature of the side chains. Although both this, and our previous studies, appear to indicate that a chain of 13 atoms extending from the anomeric centre appears to be optimal, it should be borne in mind that the reduced biological activity of longer chain materials may possibly be an effect of the physical properties of the longer chains, which may be prone to aggregation in the testing medium, rather than due to any decreased binding affinity to a biological target.

In conclusion we have synthesised a wide variety of glycosyl sulfamides of arabinose in a convergent fashion, but found that de-protected materials isomerised in aqueous solution to produce mixtures in which the α-pyranose form is predominant. Nonetheless some of the purified α-pyranose sulfamides displayed moderate anti-mycobacterial activity against *M. smegmatis* in an Alamar blue microplate assay.

3. Experimental

3.1. General

Melting points were recorded on a Kofler hot block and are uncorrected. Proton and carbon nuclear magnetic resonance (δ_H, δ_C) spectra were recorded on Bruker AV 400 (400 MHz), or Bruker AMX 500 (500 MHz) spectrometers. All chemical shifts are quoted on the δ-scale in ppm using residual solvent as an internal standard. Low resolution mass spectra were recorded on a Micromass Platform 1 spectrometer using electrospray ionisation in either positive or negative polarity (ES^+ or ES^-), or using a VG Micromass spectrometer. High-resolution mass spectra were recorded on a Walters 2790-Micromass LCT electrospray ionisation mass spectrometer, using either electrospray ionisation (NH_3, Cl) techniques as stated. *M/z* values are reported in Daltons and are followed by their percentage abundance in parentheses. Optical rotations were measured on a Perkin-Elmer 241 polarimeter with a path length of 1 dm. Concentrations are given in g / 100 mL. Microanalyses were performed by the Inorganic Chemistry Laboratory Elemental Analysis service, Oxford University, UK. Thin Layer Chromatography (t.l.c.) was carried out on Merck Kieselgel $60F_{254}$ pre-coated glass-backed plates. Visualisation of the plates was achieved using a U.V. lamp (λ_{max} = 254 or 365 nm), and/or ammonium molybdate (5% in 2 M sulphuric acid), or sulphuric acid (5% in ethanol). Flash column chromatography was carried out using Sorbsil C60 40/60 silica. Reverse phase high performance liquid chromatography (RP-HPLC) was performed on a Dionex P680 HPLC instrument with a Phenomenex Luna C 18(2) 100 A column (5 µm, 10 x 250 mm) at 15 °C. The column was eluted with a gradient of $MeCN/H_2O$ at a flow rate of 1 $mLmin^{-1}$. Alcohol-free dichloromethane was dried on an alumina column. Anhydrous DMF, pyridine, methanol and

toluene were purchased from Sigma Aldrich. 'Petrol' refers to the fraction of light petrol ether boiling in the range of 40-60 °C.

3.2. General Procedures

3.2.1. General Procedure A

Tert-butyl alcohol (1.5 equiv.) was added drop-wise to a stirred solution of chlorosulfonyl isocyanate (1 equiv.) in dry DCM (15 mL) at 0 ˚C under nitrogen. The solution was stirred for a further 30 minutes at 0 ˚C before a solution of the amine (1 equiv.) and dry triethylamine (1.5 equiv.) in dry DCM (15 mL) was added drop-wise. The reaction mixture was allowed to warm to room temperature, and then stirred for 16 hours. The reaction mixture was then diluted with DCM (20 mL), washed with saturated aqueous $NaHCO_3$ (3 x 20 mL) and brine (3 x 20 mL). The combined organic extracts were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo* to give a residue that was then purified by flash chromatography.

3.2.2. General Procedure B:

Trifluoroacetic acid (4.5 equiv) was added to a solution of the sulfamide (1 equiv.) in DCM (25 mL). The solution was stirred at room temperature for 16 hours. The reaction mixture was concentrated *in vacuo*, and the residue then purified by flash chromatography.

3.2.3. General Procedure C:

2,3,5-Tri-*O*-benzyl-α,β-D-arabinofuranose **2** (1 equiv.), and the sulfamide (1.2 equiv.) were stirred at room temperature in dry DCM (15 mL) under nitrogen. TMSOTf (1 equiv) was added drop-wise, and the mixture was then stirred for 16 hours. The reaction was neutralized by the drop-wise addition of excess triethylamine (~0.3 mL). The reaction mixture was then filtered through Celite®, eluting with ethyl acetate, and concentrated *in vacuo* to give a residue which was purified by flash chromatography.

3.2.4. General Procedure D:

Methanesulfonyl chloride (1.5 equiv.) was added drop-wise to a stirred solution of alcohol (1 equiv.) and Et_3N (1.5 equiv.) in anhydrous DCM (30 mL) at 0 ˚C under nitrogen. The reaction was allowed to warm to room temperature, and stirred for 2 hours. The reaction mixture was poured into methanol (10 mL), and concentrated *in vacuo*. The reaction mixture was then diluted with diethyl ether (20 mL), washed with water (3 x 20 mL) and brine (3 x 20 mL). The combined organic extracts were then dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The residue

was dissolved in DMF (25 mL), sodium azide (3 equiv.) was added, and the mixture was stirred at 60 ˚C for 16 hours. The reaction mixture was concentrated *in vacuo*, and The reaction mixture was then diluted with diethyl ether (50 mL), washed with water (3 x 20 mL) and brine (3 x 30 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated in *vacuo,* to give a residue which was purified by flash chromatography.

3.2.5. General Procedure E:

Triphenylphosphine (2 equiv.) and water (1.5 equiv.) were added to a stirred solution of azide (1 equiv.) in THF (25 mL) under nitrogen. The reaction was stirred under nitrogen for 16 hours. The reaction mixture was concentrated *in vacuo*, to afford a yellow oil, which was then purified by flash chromatography.

3.2.6. General Procedure F:

10 % Activated Pd-C (20 mg) was added to a solution of the protected glycosyl sulfamide (0.1 mmol) in methanol (5 mL). The flask was evacuated and purged with nitrogen five times, before it was placed under an atmosphere of hydrogen. The solution was stirred for 16 hours at room temperature. The reaction mixture was filtered through Celite® (eluting with methanol, 20 mL), and concentrated *in vacuo* to give a residue which was purified by flash column chromatography.

3.2.7. General Procedure G:

Sodium hydride (60 % dispersion in mineral oil, 0.5 equiv) was added portion-wise to a solution of triethyleneglycol (1 equiv.) in THF (50 mL) under nitrogen. The reaction was stirred for 1 hour and then cooled to 0 ˚C. Benzyl bromide (0.5 equiv.) was then added drop-wise, and the reaction mixture was warmed to room temperature and then stirred for 16 hours. The reaction mixture was cooled in an ice bath quenched by the addition of methanol (20 mL), and then concentrated *in vacuo*. The residue was dissolved in DCM (30 mL), and washed with water (30 mL). The combined organic extracts were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*, to afford a yellow oil, which was purified by flash chromatography.

3.3. Alamar Blue Assay

The anti-mycobacterial activity of deprotected glycosyl sulfamides and isoniazid were performed using *M. smegmatis*. The purified test compounds and isoniazid were prepared in DMSO at 40 mg/mL, and subsequent 2 fold serial dilutions were performed in 100 µl of LB/T media in 96 well microplates, producing compound concentrations across the plate of 1000, 500, 250, 125, 62, 31, 15, 7.5, and 3.75 µg/mL. Approximately 4.5 x 10^6 cfu/mL of *M. smegmatis* was added to each well

to give a total volume of 200 µl. Control wells contained only bacteria with 2.5 % DMSO in LB/T media. The plates were incubated at 37 °C for 18 hours. After this time, 10 µl of Alamar Blue dye was added to all wells, and the plate was then incubated for another 5 hours. The wells were then observed for a colour change (blue to pink), and the MIC value was determined by visual observation.

3.4.1. *tert*-Butyl *N*-hexylsulfamoylcarbamate 3a

General procedure A, using hexylamine, and purification by flash chromatography (DCM, R_f 0.3) afforded sulfamoylcarbamate **3a** (4.49 g, 80% yield) as a colourless solid; m.p. 106-108 ºC; ν_{max} (neat) 3295 (N-H), 1708 (C=O), 1352 (S=O), 1142 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.87-0.90 (3H, m, $(CH_2)_5C\underline{H}_3$), 1.29-1.36 (6H, m, 3 x $C\underline{H}_2$), 1.50 (9H, s, $C(C\underline{H}_3)_3$), 1.53-1.60 (2H, m, $C\underline{H}_2$), 3.06 (2H, aq, *J* 6.8 Hz, $SO_2NHC\underline{H}_2$), 5.06 (1H, br t, *J* 5.2 Hz, $SO_2N\underline{H}CH_2$), 7.12 (1H, br s, $BocN\underline{H}SO_2NH$); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, $(CH_2)_5\underline{C}H_3$), 22.6 (t, $\underline{C}H_2$), 26.3 (t, $\underline{C}H_2$), 28.1 (3 x q, $C(\underline{C}H_3)_3$), 29.1 (t, $\underline{C}H_2$), 31.4 (t, $\underline{C}H_2$), 44.0 (t, $SO_2NH\underline{C}H_2$), 83.8 (s, $\underline{C}(CH_3)_3$), 150.5 (s, C=O); HRMS (ESI): calculated for $C_{11}H_{25}N_2O_4S$: 281.1530. Found: 281.1528 (MH^+).

3.4.2. *tert*-Butyl *N*-octylsulfamoylcarbamate 3b

General procedure A using octylamine, and purification by flash chromatography (DCM, R_f 0.3), afforded sulfamoylcarbamate **3b** (2.59 g, 42% yield) as a colourless solid; m.p. 96-98 ºC; ν_{max} (neat) 3290 (N-H), 1708 (C=O), 1350 (S=O) 1141 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.86-0.90 (3H, m, $(CH_2)_7C\underline{H}_3$), 1.27-1.36 (10H, m, 5 x $C\underline{H}_2$), 1.50 (9H, s, $C(C\underline{H}_3)_3$), 1.53-1.60 (2H, m, $C\underline{H}_2$), 3.06 (2H, aq, *J* 6.8 Hz, $SO_2NHC\underline{H}_2$), 5.04 (1H, br t, *J* 6.4 Hz, $SO_2N\underline{H}CH_2$), 7.10 (1H, br s, $BocN\underline{H}SO_2NH$); δ_C (100.5 MHz, $CDCl_3$) 14.2 (q, $(CH_2)_7\underline{C}H_3$), 22.7 (t, $\underline{C}H_2$), 26.7 (t, $\underline{C}H_2$), 28.1 (3 x q, $C(\underline{C}H_3)_3$), 29.2 (t, $\underline{C}H_2$), 29.2 (t, $\underline{C}H_2$), 29.2 (t, $\underline{C}H_2$), 31.9 (t, $\underline{C}H_2$), 44.0 (t, $SO_2NH\underline{C}H_2$), 83.8 (s, $\underline{C}(CH_3)_3$), 150.5 (s, C=O); HRMS (ESI): calculated for $C_{13}H_{28}N_2NaO_4S$: 331.1662. Found: 331.1662 (MNa^+).

3.4.3. *tert*-Butyl *N*-decylsulfamoylcarbamate 3c

General procedure A, using decylamine, and purification by flash chromatography (DCM, R_f 0.3), afforded sulfamoylcarbamate **3c** (2.76 g, 41% yield) as a colourless solid; m.p. 94-96 ºC; ν_{max} (neat) 3287 (N-H), 1712 (C=O), 1367 (S=O), 1148 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.86-0.90 (3H, m, $(CH_2)_9C\underline{H}_3$), 1.26-1.36 (14H, m, 7 x $C\underline{H}_2$), 1.50 (9H, s, $C(C\underline{H}_3)_3$), 1.53-1.60 (2H, m, $C\underline{H}_2$), 3.05 (2H, aq, *J* 6.8 Hz, $SO_2NHC\underline{H}_2$), 5.05 (1H, br t, *J* 6.0 Hz, $SO_2N\underline{H}CH_2$), 7.10 (1H, br s, $BocN\underline{H}SO_2NH$); δ_C (100.5 MHz, $CDCl_3$) 14.2 (q, $(CH_2)_9\underline{C}H_3$), 22.8 (t, $\underline{C}H_2$), 26.7 (t, $\underline{C}H_2$), 28.1 (3 x q, $C(\underline{C}H_3)_3$), 29.2 (t, $\underline{C}H_2$), 29.2 (t, $\underline{C}H_2$), 29.4 (t, $\underline{C}H_2$), 29.6 (t, $\underline{C}H_2$), 29.6 (t, $\underline{C}H_2$), 32.0 (t, $\underline{C}H_2$),

44.0 (t, $SO_2NH\underline{C}H_2$), 83.8 (s, $\underline{C}(CH_3)_3$), 150.4 (s, C=O); HRMS (ESI): calculated for $C_{15}H_{32}N_2NaO_4S$: 359.1975. Found: 359.1976 (MNa^+).

3.4.4. *tert*-Butyl *N*-dodecylsulfamoylcarbamate 3d

General procedure A, using dodecylamine, and purification by flash chromatography (DCM, R_f 0.3), afforded sulfamoylcarbamate **3d** (2.04 g, 28% yield) as a colourless solid; m.p. 101-103 ºC; ν_{max} (neat) 3287 (N-H), 1713 (C=O), 1349 (S=O), 1148 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.86-0.90 (3H, m, $(CH_2)_{13}C\underline{H}_3$), 1.26-1.35 (18H, m, 9 x $C\underline{H}_2$), 1.50 (9H, s, $C(C\underline{H}_3)_3$), 1.53-1.59 (2H, m, $C\underline{H}_2$), 3.05 (2H, aq, *J* 6.8 Hz, $SO_2NHC\underline{H}_2$), 5.04 (1H, br t, *J* 6.0 Hz, $SO_2N\underline{H}CH_2$), 7.09 (1H, br s, $BocN\underline{H}SO_2NH$); δ_C (100.5 MHz, $CDCl_3$) 14.2 (q, $(CH_2)_{11}\underline{C}H_3$), 22.8 (t, $\underline{C}H_2$), 26.7 (t, $\underline{C}H_2$), 28.1 (3 x q, $C(\underline{C}H_3)_3$), 29.2 (t, $\underline{C}H_2$), 29.2 (t, $\underline{C}H_2$), 29.5 (t, $\underline{C}H_2$), 29.6 (t, $\underline{C}H_2$), 29.7 (t, $\underline{C}H_2$), 29.7 (t, $\underline{C}H_2$), 29.8 (t, $\underline{C}H_2$), 32.0 (t, $\underline{C}H_2$), 44.0 (t, $SO_2NH\underline{C}H_2$), 83.8 (s, $\underline{C}(CH_3)_3$), 150.4 (s, C=O); HRMS (ESI): calculated for $C_{17}H_{36}N_2NaO_4S$: 387.2288. Found: 387.2285 (MNa^+).

3.4.5. *tert*-Butyl *N*-tetradecylsulfamoylcarbamate 3e

General procedure A, using tetradecylamine and purification by flash chromatography (DCM, R_f 0.3), afforded sulfamoylcarbamate **3e** (2.59 g, 33% yield) as a colourless solid; m.p. 104-106 ºC; ν_{max} (neat) 3288 (N-H), 1713 (C=O), 1349 (S=O), 1148 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.86-0.90 (3H, m, $(CH_2)_{13}C\underline{H}_3$), 1.26-1.36 (22H, m, 11 x $C\underline{H}_2$), 1.50 (9H, s, $C(C\underline{H}_3)_3$), 1.53-1.58 (2H, m, $C\underline{H}_2$), 3.05 (2H, aq, *J* 6.8 Hz, $SO_2NHC\underline{H}_2$), 5.02 (1H, br t, *J* 6.0 Hz, $SO_2N\underline{H}CH_2$), 7.06 (1H, br s, $BocN\underline{H}SO_2NH$); δ_C (100.5 MHz, $CDCl_3$) 14.2 (q, $(CH_2)_{13}\underline{C}H_3$), 22.8 (t, $\underline{C}H_2$), 26.7 (t, $\underline{C}H_2$), 28.2 (3 x q, $C(\underline{C}H_3)_3$), 29.2 (t, $\underline{C}H_2$), 29.3 (t, $\underline{C}H_2$), 29.5 (t, $\underline{C}H_2$), 29.6 (t, $\underline{C}H_2$), 29.7 (t, $\underline{C}H_2$), 29.8 (t, $\underline{C}H_2$), 29.8 (t, $\underline{C}H_2$), 32.1 (t, $\underline{C}H_2$), 44.1 (t, $SO_2NH\underline{C}H_2$), 83.9 (s, $\underline{C}(CH_3)_3$), 150.4 (s, C=O); HRMS (ESI): calculated for $C_{19}H_{40}N_2NaO_4S$: 415.2601. Found: 415.2605 (MNa^+).

3.4.6. *tert*-Butyl *N*,*N*-dihexylsulfamoylcarbamate 3f

General procedure A, using dihexylamine, and purification by flash chromatography (DCM, R_f 0.64), afforded sulfamoylcarbamate **3f** (5.10 g, 70% yield) as a yellow oil; ν_{max} (neat) 3263 (N-H), 1742 (s, C=O), 1352 (S=O), 1132 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.89 (6H, t, *J* 6.7 Hz, 2 x $C\underline{H}_3(CH_2)_5$), 1.30-1.32 (12H, m, $CH_3(C\underline{H}_2)_3$), 1.48 (9H, s, $C(C\underline{H}_3)_3$), 1.54-1.60 (4H, m, $C\underline{H}_2CH_2NSO_2$), 3.30 (4H, t, *J* 7.6 Hz, $C\underline{H}_2NSO_2$), 7.00 (1H, br s, $SO_2N\underline{H}$); δ_C (100.5 MHz, $CDCl_3$) 14.1 (2 x q, $\underline{C}H_3(CH_2)_5$), 22.7 (2 x t, $\underline{C}H_2$), 26.5 (2 x t, $\underline{C}H_2$), 28.2 (2 x t, $\underline{C}H_2$), 28.3 (2 x t, $\underline{C}H_2$), 31.6 (2 x t, $\underline{C}H_2$), 49.1 (2 x t, $SO_2NH\underline{C}H_2$), 83.2 (s, $\underline{C}(CH_3)_3$), 150.1 (s, $\underline{C}$=O); HRMS (ESI): calculated for $C_{17}H_{36}N_2NaO_4S$: 387.2288. Found: 387.2294 (MNa^+). (Found: C, 56.43%; N, 7.77%; H 10.12%. $C_{17}H_{36}N_2O_4S$ requires C, 56.01%; N, 7.68%; H, 9.95%).

3.4.7. *tert*-Butyl *N,N*-dioctylsulfamoylcarbamate 3g

General procedure A, using dioctylamine, and purification by flash chromatography (DCM, R_f 0.45), afforded sulfamoylcarbamate **3g** (5.07 g, 60% yield) as a yellow oil; ν_{max} (neat) 3260, (N-H), 1743 (s, C=O), 1366 (S=O), 1132 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.88 (6H, t, *J* 6.7 Hz, 2 x $C\underline{H}_3(CH_2)_7$), 1.21-1.34 (20H, br m, $CH_3(C\underline{H}_2)_5$), 1.48 (9H, s, $C(C\underline{H}_3)_3$), 1.58 (4H, br s, $C\underline{H}_2CH_2NSO_2$), 3.30 (4H, t, *J* 7.6 Hz, $C\underline{H}_2NSO_2$), 7.00 (1H, br s, $SO_2N\underline{H}$); δ_C (100.5 MHz, $CDCl_3$) 14.2 (q, 2 x $\underline{C}H_3(CH_2)_5$), 22.7 (t, $\underline{C}H_2$), 26.8 (t, $\underline{C}H_2$), 28.2 (t, $\underline{C}H_2$), 28.4 (t, $\underline{C}H_2$), 29.3 (t, $\underline{C}H_2$), 29.4 (t, $\underline{C}H_2$), 31.9 (t, $\underline{C}H_2$), 49.1 (t, $SO_2NH\underline{C}H_2$), 83.2 (s, $\underline{C}(CH_3)_3$), 150.1 (s, $\underline{C}$=O); HRMS (ESI): calculated for $C_{21}H_{44}N_2NaO_4S$: 443.2914. Found: 443.2921 (MNa^+). (Found: C, 60.36%; N, 6.73%; H 10.73%. $C_{21}H_{44}N_2O_4S$ requires C, 59.96%; N, 6.66%; H, 10.54%).

3.4.8. *tert*-Butyl *N,N*-didecylsulfamoylcarbamate 3h

General procedure A, using didecylamine, and purification by flash chromatography (DCM, R_f 0.61), afforded sulfamoylcarbamate **3h** (4.76 g, 50% yield) as a yellow oil; ν_{max} (neat) 3262 (N-H), 1743 (s, C=O), 1366 (S=O), 1133 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.88 (6H, t, *J* 6.7 Hz, 2 x $C\underline{H}_3(CH_2)_9$), 1.26-1.29 (28H, br m, $CH_3(C\underline{H}_2)_7$), 1.48 (9H, s, $C(C\underline{H}_3)_3$), 1.55-1.60 (4H, br s, $C\underline{H}_2CH_2NSO_2$), 3.30 (4H, t, *J* 7.6 Hz, $C\underline{H}_2NSO_2$), 6.95 (1H, br s, $SO_2N\underline{H}$); δ_C (100.5 MHz, $CDCl_3$) 14.2 (q, $\underline{C}H_3(CH_2)_7$), 22.8 (t, $\underline{C}H_2$), 26.8 (t, $\underline{C}H_2$), 28.2 (t, $\underline{C}H_2$), 28.4 (t, $\underline{C}H_2$), 29.4 (t, $\underline{C}H_2$), 29.4 (t, $\underline{C}H_2$), 29.7 (t, $\underline{C}H_2$), 32.0 (t, $\underline{C}H_2$), 49.1 (t, $SO_2NH\underline{C}H_2$), 83.2 (s, $\underline{C}(CH_3)_3$), 150.1 (s, $\underline{C}$=O); HRMS (ESI): calculated for $C_{25}H_{52}N_2NaO_4S$: 499.3529. Found: 499.3552 (MNa^+). (Found: C, 63.04%; N, 5.95%; H 11.12%. $C_{25}H_{52}N_2O_4S$ requires C, 62.98%; N, 5.88%; H, 10.99%).

3.4.9. *tert*-Butyl *N,N*-didodecylsulfamoylcarbamate 3i

General procedure A, using didodecylamine, and purification by flash chromatography (DCM, R_f 0.61), afforded sulfamoylcarbamate **3i** (5.66 g, 53% yield) as a yellow oil; ν_{max} (neat) 3262 (N-H), 1744 (s, C=O), 1367 (S=O), 1133 (S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 0.88 (6H, t, *J* 6.7 Hz, 2 x $C\underline{H}_3(CH_2)_{11}$), 1.26-1.29 (36H, br m, $CH_3(C\underline{H}_2)_9$), 1.48 (9H, s, $C(C\underline{H}_3)_3$), 1.56-1.60 (4H, br s, $C\underline{H}_2CH_2NHSO_2$), 3.30 (4H, t, *J* 7.8 Hz, $C\underline{H}_2NHSO_2$), 6.98 (1H, br s, $SO_2N\underline{H}$); δ_C (100.5 MHz, $CDCl_3$) 14.2 (q, $\underline{C}H_3(CH_2)_{11}$), 22.8 (t, $\underline{C}H_2$), 26.8 (t, $\underline{C}H_2$), 28.2 (t, $\underline{C}H_2$), 28.4 (t, $\underline{C}H_2$), 29.5 (t, $\underline{C}H_2$), 29.5 (t, $\underline{C}H_2$), 29.7 (t, $\underline{C}H_2$), 29.7 (t, $\underline{C}H_2$), 29.8 (t, $\underline{C}H_2$), 29.8 (t, $\underline{C}H_2$), 32.1 (t, $\underline{C}H_2$), 49.1 (t, $SO_2NH\underline{C}H_2$), 83.2 (s, $\underline{C}(CH_3)_3$), 150.1 (s, $\underline{C}$=O); HRMS (ESI): calculated for $C_{29}H_{60}N_2NaO_4S$: 555.4166. Found: 555.4176 (MNa^+). (Found: C, 65.79%; N, 5.33%; H 11.38%. $C_{29}H_{60}N_2O_4S$ requires C, 65.37%; N, 5.26%; H, 11.35%).

3.4.10. *tert*-Butyl *N*-3,7-dimethyloctylsulfamoylcarbamate 3j

General procedure A, using 3,7-dimethyl-1-octanamine **9**, and purification by flash chromatography (DCM, R_f 0.4), afforded sulfamoylcarbamate **3j** (646 mg, 73 %) as a colourless oil; υ_{max} (neat) 3250 (N-H), 1697 (s, C=O), 1345 (s, S=O), 1137 (s, S=O) cm^{-1}; δ_H (500 MHz, $CDCl_3$) 0.85-0.88 (9H, m, 3 x CH_3), 1.11-1.15 (2H, m, C$\underline{H}_2$), 1.21-1.31 (2H, m, C$\underline{H}_2$), 1.35-1.41 (2H, m, C$\underline{H}_2$), 1.49 (9H, s, 3 x CH_3), 1.51-1.64 (4H, m, 2 x C$\underline{H}$, C$\underline{H}_2$), 3.04-3.11 (2H, m, C$\underline{H}_2$NHSO$_2$), 5.19 (1H, br. `s, $CH_2N\underline{H}SO_2$); δ_C (125 MHz, $CDCl_3$) 19.2, 22.6, 22.7 (3 x q, 3 x $\underline{C}H_3$), 24.5 (t, $\underline{C}H_2$), 27.9 (d, $\underline{C}H$), 28.0 (q, (C($\underline{C}H_3$)$_3$), 30.2 (d, $\underline{C}H$), 36.1, 37.0, 39.2 (3 x t, 3 x $\underline{C}H_2$), 42.0 (t, NH$\underline{C}H_2$), 83.7 (s, $\underline{C}(CH_3)_3$), 150.3 (s, C=O); HRMS (ESI) calculated for $C_{15}H_{32}N_2NaO_4S$: 359.1980. Found: 359.1977 (MNa^+).

3.4.11. *N*-(Hexyl)sulfamide 4a

General Procedure B, using sulfamoylcarbamate **3a**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.3), afforded sulfamide **4a** (280 mg, 86 %) as a white solid; m.p. 67-70 °C (DCM); υ_{max} (neat) 3331 (N-H), 3289 (N-H), 1357 (s, S=O), 1130 (s, S=O); δ_H (400 MHz, $CDCl_3$) 0.89 (3H, t, *J* 6.4 Hz, C$\underline{H}_3$), 1.28-1.37 (6H, m, 3 x C$\underline{H}_2$), 1.53-1.61 (2H, m, NHCH$_2$C$\underline{H}_2$), 3.13 (2H, t, *J* 7.2 Hz, C$\underline{H}_2$NHSO$_2$), 4.52 (2H, br s, N$\underline{H}_2$); δ_C (100.5 MHz, $CDCl_3$) 13.9 (q, $\underline{C}H_3$), 22.5, 26.3, 29.4, 31.3, (4 x t, 4 x $\underline{C}H_2$), 43.7 (t, $\underline{C}H_2$NH); HRMS (ESI) calculated for $C_6H_{16}N_2NaO_2S$: 203.0830. Found: 203.0825 (MNa^+).

3.4.12. *N*-(Octyl)sulfamide 4b

General Procedure B, with sulfamoylcarbamate **3b**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.3), afforded sulfamide **4b** (472 mg, 69 %) as a white solid; m.p. 82-85 °C (DCM); υ_{max} (neat) 3330 (N-H), 3283 (N-H), 1345 (s, S=O), 1135 (s, S=O); δ_H (400 MHz, DMSO) 0.85 (3H, t, *J* 6.8 Hz, C$\underline{H}_3$), 1.18-1.30 (10H, m, 5 x C$\underline{H}_2$), 1.41-1.46 (2H, m, NHCH$_2$C$\underline{H}_2$), 2.82 (2H, aq, *J* 6.7 Hz, C$\underline{H}_2$NHSO$_2$), 6.37 (1H, t, *J* 5.5 Hz, SO$_2$N$\underline{H}$), 6.40 (2H, s, N$\underline{H}_2$); δ_C (100.5 MHz, DMSO) 14.4 (q, $\underline{C}H_3$), 22.5, 26.8, 29.1, 29.1, 29.4, 31.7 (6 x t, 6 x $\underline{C}H_2$), 43.0 (t, $\underline{C}H_2$NH); HRMS (ESI) calculated for $C_8H_{20}N_2NaO_2S$: 231.1143. Found: 231.1134 (MNa^+)

3.4.13. *N*-(Decyl)sulfamide 4c

General Procedure B, using sulfamoylcarbamate **3c**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.3), afforded sulfamide **4c** (536 mg, 76 %) as a white solid; m.p. 83-86 °C (DCM); υ_{max} (neat) 3330 (N-H), 3270 (N-H), 1342 (s, S=O), 1135 (s, S=O); δ_H (400 MHz, DMSO) 0.84 (3H, t, *J* 6.4 Hz, C$\underline{H}_3$), 1.18-1.30 (14H, m, 7 x C$\underline{H}_2$), 1.39-1.44 (2H, m, NHCH$_2$C$\underline{H}_2$), 2.82 (2H, aq, *J* 6.7 Hz, C$\underline{H}_2$NH), 6.36 (1H, t, *J* 5.5 Hz, N$\underline{H}$), 6.40 (2H, s, N$\underline{H}_2$); δ_C (100.5 MHz, DMSO)

14.4 (q, $\underline{C}H_3$), 22.6, 26.8, 29.2, 29.4, 29.5, 31.8 (6 x t, 8 x $\underline{C}H_2$), 43.0 (t, $\underline{C}H_2NH$); HRMS (ESI) calculated for $C_{10}H_{24}N_2NaO_2S$: 259.1456. Found: 259.1444 (MNa^+).

3.4.14. *N*-(Dodecyl)sulfamide 4d

General Procedure B**,** using sulfamoylcarbamate **3d** and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.3), sulfamide **4d** (493 mg, 68 %) as a white solid; m.p. 95-96 °C (DCM); υ_{max} (neat) 3338 (N-H), 3278 (N-H), 1345 (s, S=O), 1136 (s, S=O); δ_H (400 MHz, DMSO) 0.84 (3H, t, *J* 6.4 Hz, $C\underline{H}_3$), 1.18-1.30 (18H, m, 9 x $C\underline{H}_2$), 1.39-1.44 (2H, m, $NHCH_2C\underline{H}_2$), 2.82 (2H, aq, *J* 6.7 Hz, $C\underline{H}_2NH$), 6.37 (1H, t, *J* 5.5 Hz, NH), 6.40 (2H, s, $N\underline{H}_2$); δ_C (100.5 MHz, DMSO) 14.4 (q, $\underline{C}H_3$), 22.5, 26.8, 29.2, 29.5, 29.5, 31.8 (6 x t, 10 x $\underline{C}H_2$), 43.0 (t, $\underline{C}H_2NH$); HRMS (ESI) calculated for $C_{12}H_{29}N_2O_2S$: 265.1950. Found: 265.1937 (MH^+).

3.4.15. *N*-(Tetradecyl)sulfamide 4e

General Procedure B, using sulfamoylcarbamate **3e**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.4), afforded sulfamide **4e** (562 mg, 75 %) as a white solid; m.p. 99-101 °C (DCM); υ_{max} (neat) 3338 (N-H), 3276 (N-H), 1340 (s, S=O), 1134 (s, S=O); δ_H (400 MHz, DMSO) 0.84 (3H, t, *J* 6.4 Hz, $C\underline{H}_3$), 1.18-1.32 (22H, m, 11 x $C\underline{H}_2$), 1.37-1.44 (2H, m, $NHCH_2C\underline{H}_2$), 2.82 (2H, aq, *J* 6.7 Hz, $C\underline{H}_2NH$), 6.36 (1H, t, *J* 5.5 Hz, NH), 6.40 (2H, s, $N\underline{H}_2$); δ_C (100.5 MHz, DMSO) 14.4 (q, $\underline{C}H_3$), 22.5, 26.8, 29.2, 29.2, 29.5, 29.5, 31.7 (7 x t, 12 x $\underline{C}H_2$), 43.0 (t, $\underline{C}H_2NH$); HRMS (ESI) calculated for $C_{14}H_{33}N_2O_2S$: 293.2263. Found: 293.2252 (MH^+).

3.4.16. *N,N*-(Dihexyl)sulfamide 4f

General Procedure B, using sulfamoylcarbamate **3f,** and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.5), afforded sulfamide **4f** (667 mg, 92 %) as a pale yellow solid; m.p. 55-58 °C (DCM); υ_{max} (neat) 3381 (N-H), 3264 (N-H), 1350 (s, S=O), 1137 (s, S=O); δ_H (400 MHz, $CDCl_3$) 0.89 (6H, t, *J* 6.4 Hz, 2 x $C\underline{H}_3$), 1.25-1.32 (12H, m, 6 x $C\underline{H}_2$), 1.54-1.63 (4H, m, 2 x $NCH_2C\underline{H}_2$), 3.15 (4H, t, *J* 7.8 Hz, 2 x $C\underline{H}_2N$); δ_C (100.5 MHz, $CDCl_3$) 14.0 (q, 2 x $\underline{C}H_3$), 22.6, 26.4, 28.1, 31.5 (4 x t, 8 x $\underline{C}H_2$), 48.4 (t, 2 x $\underline{C}H_2N$); HRMS (ESI) calculated for $C_{12}H_{29}N_2O_2S$: 265.1950. Found: 265.1937 (MH^+).

3.4.17. *N,N*-(Dioctyl)sulfamide 4g

General Procedure B, using sulfamoylcarbamate **3g**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.5), afforded sulfamide **4g** (537 mg, 76 %) as a pale yellow solid; m.p. 48-50 °C (DCM); υ_{max} (neat) 3381 (N-H), 3264 (N-H), 1350 (s, S=O), 1137 (s, S=O); δ_H (400 MHz, $CDCl_3$) 0.88 (6H, t, *J* 6.4 Hz, 2 x $C\underline{H}_3$), 1.27-1.29 (20H, m, 10 x $C\underline{H}_2$), 1.55-1.63 (4H, m, 2 x

$NCH_2C\underline{H}_2$), 3.15 (4H, t, *J* 7.8 Hz, 2 x $C\underline{H}_2N$); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, 2 x $\underline{C}H_3$), 22.6, 26.8, 28.2, 29.2, 31.8 (5 x t, 12 x $\underline{C}H_2$), 48.4 (t, 2 x $\underline{C}H_2N$); HRMS (ESI) calculated for $C_{16}H_{37}N_2O_2S$: 321.2576. Found: 321.2565 (MH^+).

3.4.18. *N,N*-(Didecyl)sulfamide 4h

General Procedure B, using sulfamoylcarbamate **3h**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.6), afforded sulfamide **4h** (521 mg, 66 %) as a pale yellow solid; m.p. 53-55 °C (DCM); υ_{max} (neat) 3392 (N-H), 3285 (N-H), 1350 (s, S=O), 1137 (s, S=O); δ_H (400 MHz, $CDCl_3$) 0.88 (6H, t, *J* 6.6 Hz, 2 x $C\underline{H}_3$), 1.26-1.29 (28H, m, 14 x $C\underline{H}_2$), 1.54-1.63 (4H, m, 2 x $NCH_2C\underline{H}_2$), 3.15 (4H, t, *J* 7.8 Hz, 2 x $C\underline{H}_2N$); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, 2 x $\underline{C}H_3$), 22.7, 26.8, 28.2, 29.3, 29.6, 31.9 (6 x t, 16 x $\underline{C}H_2$), 48.4 (t, 2 x $\underline{C}H_2N$); HRMS (ESI) calculated for $C_{20}H_{45}N_2O_2S$: 377.3202. Found: 377.3196 (MH^+).

3.4.19. *N,N*-(Didodecyl)sulfamide 4i

General Procedure B, using sulfamoylcarbamate **3i**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.6), afforded sulfamide **4i** (632 mg, 78 %) as a pale yellow solid; m.p. 58-60 °C (DCM); υ_{max} (neat) 3392 (N-H), 3285 (N-H), 1350 (s, S=O), 1137 (s, S=O); δ_H (400 MHz, $CDCl_3$) 0.88 (6H, t, *J* 6.6 Hz, 2 x CH_3), 1.25-1.29 (36H, m, 18 x CH_2), 1.54-1.62 (4H, m, 2 x $NCH_2C\underline{H}_2$), 3.15 (4H, t, *J* 7.2 Hz, 2 x $C\underline{H}_2N$); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, 2 x $\underline{C}H_3$), 22.7, 26.8, 28.2, 29.3, 29.6, 29.6, 31.9 (7 x t, 20 x $\underline{C}H_2$), 48.4 (t, 2 x $\underline{C}H_2N$); HRMS (ESI) calculated for $C_{24}H_{53}N_2O_2S$: 433.3828. Found: 433.3824 (MH^+).

3.4.20. *N*-(3,7-Dimethyloctyl)sulfamide 4j

General Procedure B, using sulfamoylcarbamate **3j,** and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.4), afforded sulfamide **4j** (0.8 g, 98 %) as a yellowish brown waxy solid; υ_{max} (neat) 3275 (N-H), 1324 (w, S=O), 1137 (s, S=O); δ_H (500 MHz, $CDCl_3$) 0.85-0.91 (9H, m, 3 x CH_3), 1.11-1.15 (2H, m, $C\underline{H}_2$), 1.21-1.31 (2H, m, $C\underline{H}_2$), 1.35-1.41 (2H, m, $C\underline{H}_2$), 1.48-1.50 (2H, m, $C\underline{H}_2$), 1.51-1.64 (2H, m, 2 x $C\underline{H}$), 3.10-3.16 (2H, m, $C\underline{H}_2NHSO_2$), 4.40 (1H, s, $N\underline{H}$), 4.67 (2H, s, $N\underline{H}_2$); δ_C (125 MHz, $CDCl_3$) 19.3, 22.6, 22.7 (3 x q, 3 x $(\underline{C}H_3)_2$), 24.6 (t, $\underline{C}H_2$), 27.9, 30.4 (2 x d, 2 x $\underline{C}H$), 36.5, 37.1 (2 x t, 2 x $\underline{C}H_2$), 39.2 (t, $\underline{C}H_2NH$), 41.8 (t, $NH\underline{C}H_2$); HRMS (ESI) calculated for $C_{10}H_{25}N_2O_2S$: 237.1637 . Found: 237.1633 (MH^+).

3.4.21. *N*-(Hexyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5a

General Procedure C, using sulfamide **4a**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.4), afforded glycosylsulfamide **5a** (280 mg, 52 %, α:β, 1:1) as a yellow waxy

solid; υ_{max} (neat) 3285 (NH), 1350 (s, S=O), 1158 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.89 (3H, t, *J* 6.8 Hz, C<u>H</u>$_3$), 1.24-1.36 (6H, m, C<u>H</u>$_2$), 1.48-1.55 (2H, m, NHCH$_2$C<u>H</u>$_2$), 3.00-3.04 (2H, m, C<u>H</u>$_2$NH), 3.48 (1H, d, *J* 7.0 Hz, H-5), 3.55 (1H, d, *J* 5.8 Hz, H-5'), 3.94-4.03 (2H, m, H-2, H-3), 4.34 (1H, d, *J* 7.0 Hz, H-4), 4.47-4.59 (6H, m, Ph-C<u>H</u>$_2$), 5.44 (1H, d, $J_{NH,1}$ 11.8 Hz, H-1), 5.52-5.64 (1H, m, N<u>H</u>), 7.16-7.42 (15H, m, Ar-H); β anomer: 0.89 (3H, t, *J* 6.8 Hz, C<u>H</u>$_3$), 1.24-1.36 (6H, m, C<u>H</u>$_2$), 1.48-1.55 (2H, m, NHCH$_2$C<u>H</u>$_2$), 3.00-3.03 (2H, m, C<u>H</u>$_2$NH), 3.53 (2H, dd, $J_{5,5'}$ 7.0 Hz, $J_{4,5}$ 5.5 Hz, H-5, H-5'), 3.94-4.04 (3H, m, H-2, H-3, H-4), 4.47-4.59 (6H, m, Ph-C<u>H</u>$_2$), 5.37 (1H, dd, $J_{1,NH}$ 6.8 Hz, $J_{1,2}$ 4.5 Hz, H-1), 5.52-5.64 (1H, m, N<u>H</u>), 7.16-7.42 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.0 (q, CH_3), 22.5, 26.3, 29.4, 31.4 (4 x t, 4 x CH_2), 43.4, 43.5 (2 x t, NHCH$_2$α, NHCH$_2$β), 70.0, 70.1(2 x t, C-5α, C-5β), 71.8, 71.8, 72.0, 72.3, 73.4, 73.4 (6 x t, Ph<u>C</u>H$_2$), 80.8 (d, C-4β), 81.2, 81.8 (2 x d, C-2α, C-2β), 82.4 (C-3β), 83.3 (d, C-4α), 84.3 (d, C-1β), 84.8 (d, C-3α), 88.2 (d, C-1α), 127.7, 127.7, 127.8, 127.8, 127.9, 127.9, 128.0, 128.0, 128.2, 128.2, 128.3, 128.4, 128.5, 128.5, 128.6 (15 x d, 15 x Ar-<u>C</u>), 136.7, 136.8, 136.9, 137.4, 137.6, 137.9 (6 x s, 6 x Ar-<u>C</u>); HRMS (ESI) calculated for $C_{32}H_{42}N_2NaO_6S$: 605.2661. Found: 605.2656 (MNa^+).

3.4.22. *N*-(Octyl)-*N*'-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5b

General Procedure C, using sulfamide **4b**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.4), afforded glycosylsulfamide sulfamide **5b** (230 mg, 54 %, α:β, 1:1) as a yellow waxy solid; υ_{max} (neat) 3280 (N-H), 1350 (s, S=O), 1158 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.88 (3H, t, *J* 6.6 Hz, C<u>H</u>$_3$), 1.19-1.36 (10H, m, C<u>H</u>$_2$), 1.48-1.55 (2H, m, NHCH$_2$C<u>H</u>$_2$), 2.98-3.03 (2H, m, C<u>H</u>$_2$NH), 3.48 (1H, dd, $J_{5,5'}$ 9.6 Hz, $J_{4,5'}$ 7.6 Hz, H-5), 3.58 (1H, dd, $J_{5,5'}$ 9.8 Hz, $J_{4,5'}$ 5.9 Hz, H-5'), 3.94-3.97 (1H, m, H-3), 3.98 (1H, at, *J* 3.1 Hz, H-2), 4.34 (1H, at, *J* 6.7 Hz, H-4), 4.40-4.61 (6H, m, Ph-C<u>H</u>$_2$), 5.41 (1H, d, $J_{NH,1}$ 10.6 Hz, H-1), 5.52-5.61 (1H, m, N<u>H</u>), 7.19-7.44 (15H, m, Ar-H); β anomer: 0.88 (3H, t, *J* 6.6 Hz, C<u>H</u>$_3$), 1.19-1.36 (10H, m, C<u>H</u>$_2$), 1.48-1.55 (2H, m, NHCH$_2$C<u>H</u>$_2$), 2.98-3.03 (2H, m, C<u>H</u>$_2$NH), 3.52 (2H, d, *J* 5.1 Hz, H-5, H-5'), 3.94-3.97 (1H, m, H-3), 4.01 (1H, at, $J_{1,2}$ 4.3 Hz, H-2), 4.05 (1H, dd, $J_{4,5}$ 5.1 Hz,$J_{3,4}$ 3.5 Hz, H-4), 4.40-4.61 (6H, m, Ph-C<u>H</u>$_2$), 5.37 (1H, dd, $J_{NH,1}$ 10.2 Hz, $J_{1,2}$ 4.3Hz, H-1), 5.52-5.61 (1H, m, N<u>H</u>), 7.19-7.44 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, CH_3), 22.6, 26.7, 29.2, 29.4, 31.8 (5 x t, 6 x CH_2), 43.4, 43.5 (2 x t, NHCH$_2$α, NHCH$_2$β), 70.0, 70.1 (2 x t, C-5α, C-5β), 71.7, 71.8, 72.0, 72.3, 73.3, 73.4 (6 x t, Ph<u>C</u>H$_2$), 80.8 (d, C-4β), 81.2, 81.8 (2 x d, C-2α, C-2β), 82.4 (C-3β), 83.3 (d, C-4α), 84.2 (d, C-1β), 84.8 (d, C-3α), 88.2 (d, C-1α), 127.7, 127.7, 127.8, 127.9,127.9, 127.9, 127.9, 128.1, 128.2, 128.2, 128.3, 128.4, 128.5, 128.5, 128.6 (15 x d, 15 x Ar-<u>C</u>), 136.7, 136.7, 136.8, 137.4, 137.7, 137.9 (6 x s, 6 x Ar-<u>C</u>); HRMS (ESI) calculated for $C_{34}H_{46}N_2NaO_6S$: 633.2974. Found: 633.2976 (MNa^+).

3.4.23. *N*-(Decyl)-*N*'-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5c

General Procedure C, using sulfamide **4c**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.4), afforded glycosylsulfamide **5c** (470 mg, 77 %, α:β, 1:1) as a yellow waxy solid; υ_{max} (neat) 3280 (N-H), 1350 (s, S=O), 1158 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.89 (3H, t, *J* 6.7 Hz, C<u>H</u>$_3$), 1.24-1.29 (14H, m, 7 x C<u>H</u>$_2$), 1.48-1.53 (2H, m, NHCH$_2$C<u>H</u>$_2$), 2.98-3.03 (2H, m, C<u>H</u>$_2$NH), 3.46-3.50 (1H, m, H-5), 3.58 (1H, dd, $J_{5,5'}$ 8.6 Hz, $J_{4,5'}$ 6.7 Hz, H-5'), 3.95-3.97 (1H, m, H-3), 3.98-4.02 (1H, m, H-2), 4.34 (1H, at, *J* 6.6 Hz, H-4), 4.38-4.63 (6H, m, Ph-C<u>H</u>$_2$), 5.42 (1H, d, $J_{NH,1}$ 10.5 Hz, H-1), 5.54 (1H, d, $J_{NH,1}$ 11.0 Hz N<u>H</u>), 7.15-7.40 (15H, m, Ar-H); β anomer: 0.89 (3H, t, *J* 6.7 Hz, C<u>H</u>$_3$), 1.24-1.29 (14H, m, C<u>H</u>$_2$), 1.48-1.53 (2H, m, NHCH$_2$C<u>H</u>$_2$), 2.98-3.03 (2H, m, C<u>H</u>$_2$NH), 3.52-3.54 (1H, m, H-5, H-5'), 3.95-3.97 (1H, m, H-3), 3.98-4.02 (1H, m, H-2), 4.05-4.06 (1H, m, H-4), 4.38-4.63 (6H, m, Ph-C<u>H</u>$_2$), 5.37 (1H, dd, $J_{NH,1}$ 10.2 Hz, $J_{1,2}$ 4.3 Hz, H-1), 5.52 (1H, d, $J_{NH,1}$ 10.8 Hz N<u>H</u>), 7.15-7.40 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, CH_3), 22.7, 26.7, 29.2, 29.3, 29.4, 29.5, 29.5, 31.8, (8 x t, 8 x CH_2), 43.4, 43.5 (2 x t, NHCH$_2$α, NHCH$_2$β), 70.1 (t, C-5α, C-5β), 71.8, 71.8, 72.0, 72.3, 73.3, 73.4 (6 x t, Ph-<u>C</u>H$_2$), 80.8 (d, C-4β), 81.2, 81.8 (2 x d, C-2α, C-2β), 82.4 (d, C-3β), 83.3 (d, C-4α), 84.3 (d, C-1β), 84.8 (d, C-3α), 88.2 (d, C-1α), 127.7, 127.7, 127.7, 127.8, 127.9, 127.9, 128.1, 128.2, 128.2, 128.3, 128.4, 128.5, 128.5, 128.5, 128.6 (15 x d, 15 x Ar-<u>C</u>), 136.7, 136.8, 136.9, 137.4, 137.7, 137.9 (6 x s, 6 x Ar-<u>C</u>); HRMS (ESI) calculated for $C_{36}H_{50}N_2NaO_6S$: 661.3287. Found 661.3288 (MNa^+).

3.4.24. *N*-(Dodecyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5d

General Procedure C, using sulfamide **4d**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.4), afforded glycosylsulfamide **5d** (450 mg, 71 %, α:β, 1:1) as a yellow waxy solid; υ_{max} (neat) 3286 (N-H), 1344 (s, S=O), 1151 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.89 (3H, t, *J* 6.9 Hz, C<u>H</u>$_3$), 1.20-1.38 (18H, m, 9 x C<u>H</u>$_2$), 1.44-1.65 (2H, m, C<u>H</u>$_2$), 3.00 (2H, t, *J* 6.7 Hz, C<u>H</u>$_2$NH), 3.47 (1H, dd, $J_{5,5'}$ 9.8 Hz, $J_{4,5'}$ 7.4 Hz, H-5), 3.58 (1H, dd, $J_{5,5'}$ 9.6 Hz, $J_{4,5'}$ 6.1 Hz, H-5'), 3.94-3.97 (1H, m, H-3), 3.98 (1H, at, *J* 3.1 Hz, H-2), 4.34 (1H, at, *J* 5.9 Hz, H-4), 4.40-4.67 (6H, m, Ph-C<u>H</u>$_2$), 5.42 (1H, d, $J_{NH,1}$ 10.5 Hz, H-1), 5.57 (1H, at, *J* 11.3 Hz, N<u>H</u>), 7.18-7.41 (15H, m, Ar-H); β anomer: 0.89 (3H, t, *J* 6.9 Hz, C<u>H</u>$_3$), 1.20-1.38 (18H, m, C<u>H</u>$_2$), 1.44-1.65 (2H, m, C<u>H</u>$_2$), 3.00 (2H, t, *J* 6.7 Hz, C<u>H</u>$_2$NH), 3.52 (2H, d, *J* 5.1 Hz, H-5, H-5'), 3.94-3.97 (1H, m, H-3), 4.01 (1H, at, $J_{1,2}$ 4.3 Hz, H-2), 4.05 (1H, dd, $J_{4,5}$ 5.1 Hz,$J_{3,4}$ 3.5 Hz, H-4), 4.40-4.67 (6H, m, Ph-C<u>H</u>$_2$), 5.37 (1H, dd, $J_{NH,1}$ 10.2 Hz, $J_{1,2}$ 4.3Hz, H-1), 5.57 (1H, t, *J* 11.3 Hz, N<u>H</u>), 7.18-7.41 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, CH_3), 22.7, 26.7, 26.7, 29.2, 29.3, 29.4, 29.6, 29.6, 29.7, 31.9 (10 x t, 10 x CH_2), 43.4, 43.5 (2 x t, NHCH$_2$α, NHCH$_2$β), 70.0, 70.1(2 x t, C-5α, C-5β), 71.7, 71.8, 72.0, 72.3, 73.3, 73.4 (6 x t, Ph-<u>C</u>H$_2$), 80.8 (d, C-4β), 81.2, 81.8 (2 x d, C-2α, C-2β), 82.4 (d, C-3β), 83.3 (d, C-4α), 84.3 (d, C-1β), 84.8 (d, C-3α), 88.2 (d, C-1α), 127.7, 127.7, 127.8, 127.9, 127.9, 127.9, 128.0, 128.1, 128.2, 128.2, 128.4, 128.5, 128.5, 128.5, 128.6 (15 x d, 15 x Ar-<u>C</u>), 136.7, 136.7,

136.9, 137.4, 137.7, 137.9 (6 x s, 6 x Ar-C); HRMS (ESI) calculated for $C_{38}H_{54}N_2NaO_6S$: 689.3600. Found: 689.3600 (MNa^+).

3.4.25. *N*-(Tetradecyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5e

General Procedure C, sulfamide **4e**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.4), afforded glycosylsulfamide **5e** (360 mg, 54 %, α:β, 1:1) as a yellow waxy solid; υ_{max} (neat) 3280 (N-H), 1350 (s, S=O), 1158 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.90 (3H, t, *J* 6.6 Hz, C$\underline{H}_3$), 1.21-1.38 (22H, m, 11 x C$\underline{H}_2$), 1.44-1.74 (2H, m, C$\underline{H}_2$), 3.01 (2H, t, *J* 7.2 Hz, C$\underline{H}_2$NH), 3.49 (1H, dd, $J_{5,5'}$ 9.8 Hz, $J_{4,5'}$ 7.4 Hz, H-5), 3.59 (1H, dd, $J_{5,5'}$ 9.8 Hz, $J_{4,5'}$ 5.9 Hz, H-5'), 3.94-3.97 (1H, m, H-3), 4.00 (1H, at, *J* 3.1 Hz, H-2), 4.35 (1H, at, *J* 6.6 Hz, H-4), 4.43-4.65 (6H, m, Ph-C$\underline{H}_2$), 5.43 (1H, d, $J_{NH,1}$ 10.6 Hz, H-1), 5.62 (1H, d, $J_{NH,1}$ 10.9, N$\underline{H}$), 7.20-7.46 (15H, m, Ar-H); β anomer: 0.90 (3H, t, *J* 6.6 Hz, C$\underline{H}_3$), 1.21-1.38 (22H, m, 11 x C$\underline{H}_2$), 1.44-1.74 (2H, m, C$\underline{H}_2$), 2.98-3.01 (2H, t, *J* 7.2 Hz, C$\underline{H}_2$NH), 3.54 (2H, d, *J* 5.1 Hz, H-5, H-5'), 3.94-3.97 (1H, m, H-3), 4.03 (1H, at, $J_{1,2}$ 4.3 Hz, H-2), 4.06 (1H, dd, $J_{4,5}$ 5.1 Hz,$J_{3,4}$ 3.5 Hz, H-4), 4.43-4.65 (6H, m, Ph-C$\underline{H}_2$), 5.38 (1H, dd, $J_{NH,1}$ 10.4 Hz, $J_{1,2}$ 4.5Hz, H-1), 5.58 (1H, d, $J_{NH,1}$10.4 Hz N$\underline{H}$), 7.20-7.46 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, CH_3), 22.7, 26.7, 26.7, 29.2, 29.4, 29.5, 29.5, 29.6, 29.7, 29.7, 29.7, 31.9 (12 x t, 12 x CH_2), 43.4, 43.5 (2 x t, $NHCH_2$α, $NHCH_2$β), 70.1 (t, C-5α, C-5β), 71.8, 72.0, 72.2, 72.3, 73.4, 73.5 (6 x t, Ph-C$\underline{H}_2$), 80.8 (d, C-4β), 81.2, 81.8 (2 x d, C-2α, C-2β), 82.4 (d, C-3β), 83.2 (d, C-4α), 84.3 (d, C-1β), 84.9 (d, C-3α), 88.2 (d, C-1α), 127.7, 127.7, 127.8, 127.8, 127.9, 128.0, 128.2, 128.2, 128.3, 128.4, 128.4, 128.5, 128.5,128.5, 128.6 (15 x d, 15 x Ar-C), 136.7, 136.8, 136.9, 137.9, 137.9, 138.1 (6 x s, 6 x Ar-C); HRMS (ESI) calculated for $C_{40}H_{58}N_2NaO_6S$: 717.3913. Found: 717.3910 (MNa^+).

3.4.26. *N*-(Dihexyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5f

General Procedure C, using sulfamide **4f**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.6), afforded glycosylsulfamide **5f** (250 mg, 78 %, α:β, 1:1) as a yellow waxy solid; υ_{max} (neat) 3288 (N-H), 1350 (s, S=O), 1158 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.89 (6H, t, *J* 6.7 Hz, 2 x C$\underline{H}_3$), 1.17-1.37 (12H, m, 6 x C$\underline{H}_2$), 1.51-1.63 (4H, m, 2 x $NCH_2C\underline{H}_2$), 2.97-3.25 (4H, m, 2 x C$\underline{H}_2$NH), 3.50 (1H, dd, $J_{5,5'}$ 9.8 Hz, $J_{4,5'}$ 7.8 Hz, H-5), 3.60 (1H, dd, $J_{5,5'}$ 9.8 Hz, $J_{4,5'}$ 5.9 Hz, H-5'), 3.95-3.97 (1H, m, H-3), 3.99-4.02 (1H, m, H-2), 4.35 (1H, at, *J* 6.7 Hz, H-4), 4.42-4.64 (6H, m, Ph-C$\underline{H}_2$), 5.36 (1H, d, $J_{NH,1}$ 10.5 Hz, H-1), 5.47 (1H, d, $J_{NH,1}$ 8.0 Hz, N$\underline{H}$), 7.18-7.44 (15H, m, Ar-H); β anomer: 0.89 (6H, t, *J* 6.7 Hz, 2 x C$\underline{H}_3$), 1.17-1.37 (12H, m, 6 x C$\underline{H}_2$), 1.51-1.63 (4H, m, 2 x $NHCH_2C\underline{H}_2$), 2.97-3.25 (4H, m, 2 x C$\underline{H}_2$NH), 3.54 (2H, at, *J* 5.1 Hz, H-5, H-5'), 3.95-3.97 (1H, m, H-3), 3.99-4.02 (1H, m, H-2), 4.03-4.07 (1H, m, H-4), 4.42-4.64 (6H, m, Ph-C$\underline{H}_2$), 5.31 (1H, dd, $J_{NH,1}$ 9.8Hz, $J_{1,2}$ 3.1 Hz, H-1), 5.43 (1H, d, $J_{NH,1}$ 8.5 Hz, N$\underline{H}$), 7.18-7.44 (15H,

m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.0 (q, 2 x CH_3), 22.6, 26.5, 28.7, 31.5, (4 x t, 8 x CH_2), 48.8 (t, 2 x $\underline{C}H_2N$), 70.2, 70.2 (t, C-5α, C-5β), 71.7, 71.8, 71.8, 72.3, 73.3, 73.4 (6 x t, Ph-$\underline{C}H_2$), 80.5 (d, C-4β), 81.5, 81.9 (2 x d, C-2α, C-2β), 82.5 (d, C-3β), 82.8 (d, C-4α), 84.0 (d, C-1β), 85.1 (d, C-3α), 87.8 (d, C-1α), 127.7, 127.7, 127.7, 127.8, 127.9, 128.0, 128.1, 128.1, 128.2, 128.4, 128.4, 128.5, 128.5, 128.6 (14 x d, 14 x Ar-$\underline{C}$), 136.9, 136.9, 137.1, 137.6, 137.8, 138.0 (6 x s, 6 x Ar-$\underline{C}$); HRMS (ESI) calculated for $C_{38}H_{55}N_2O_6S$: 667.3781. Found: 667.3790 (MH^+).

3.4.27. *N*-(Dioctyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5g

General Procedure C, using sulfamide **4g**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.6), afforded glycosylsulfamide **5g** (230 mg, 68 %, α:β, 1:1) as a yellow waxy solid; υ_{max} (neat) 3282 (N-H), 1350 (s, S=O), 1158 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.88 (6H, t, *J* 6.7 Hz, 2 x C$\underline{H}_3$), 1.21-1.33 (20H, m, 10 x C$\underline{H}_2$), 1.51-1.60 (4H, m, 2 x NHCH$_2$C$\underline{H}_2$), 2.99-3.25 (4H, m, 2 x C$\underline{H}_2$NH),), 3.48 (1H, dd, $J_{5,5'}$ 9.8 Hz, $J_{4,5'}$ 8.2 Hz, H-5), 3.59 (1H, dd, $J_{5,5'}$ 9.6 Hz, $J_{4,5'}$ 5.7 Hz, H-5'), 3.93-3.96 (1H, m, H-3), 3.97-4.00 (1H, m, H-2), 4.32 (1H, at, *J* 6.5 Hz, H-4), 4.41-4.62 (6H, m, Ph-C$\underline{H}_2$), 5.33 (1H, d, J_{NH-1} 11.9 Hz, H-1), 5.42 (1H, d, *J* 9.8 Hz, NH), 7.20-7.38 (15H, m, Ar-H); β anomer: 0.88 (6H, t, *J* 6.7 Hz, 2 x CH_3), 1.21-1.33 (20H, m, 10 x CH_2), 1.54-1.56 (4H, m, 2 x NCH$_2$C$\underline{H}_2$), 2.99-3.25 (4H, m, 2 x C$\underline{H}_2$NH), 3.53 (2H, at, *J* 4.3 Hz, H-5, H-5'), 3.93-3.96 (1H, m, H-3), 3.97-4.00 (1H, m, H-2), 4.01-4.04 (1H, m, H-4), 4.41-4.62 (6H, m, Ph-CH_2), 5.29 (1H, dd, $J_{NH,1}$ 10.2 Hz, $J_{1,2}$ 3.9 Hz, H-1), 5.42 (1H, d, *J* 9.8 Hz, NH), 7.20-7.38 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, 2 x CH_3), 22.6, 26.8, 28.7, 28.8, 29.2, 29.3, 31.8 (7 x t, 12 x CH_2), 48.8, 48.9 (2 x t, 2 x $\underline{C}H_2N$), 70.1, 70.2 (t, C-5α, C-5β), 71.7, 71.8, 71.8, 72.3, 73.3, 73.4 (6 x t, Ph-$\underline{C}H_2$), 80.5 (d, C-4β), 81.5, 81.9 (2 x d, C-2α, C-2β), 82.5 (d, C-3β), 82.8 (d, C-4α), 84.0 (d, C-1β), 85.1 (d, C-3α), 87.9 (d, C-1α), 127.6, 127.7, 127.7, 127.8, 127.9, 128.0, 128.1, 128.1, 128.1, 128.2, 128.3, 128.4, 128.5, 128.6 (14 x d, 14 x Ar-$\underline{C}$), 136.8, 136.9, 137.0, 137.5, 137.8, 138.0 (6 x s, 6 x Ar-$\underline{C}$); HRMS (ESI) calculated for $C_{42}H_{63}N_2O_6S$: 723.4407. Found: 723.4416 (MH^+).

3.4.28. *N*-(Didecyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5h

General Procedure C, using sulfamide **4h**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.6), afforded glycosylsulfamide **5h** (320 mg, 59 %, α:β, 1:1) as a yellow waxy solid. υ_{max} (neat) 3285 (N-H), 1350 (s, S=O), 1158 (s, S=O) cm^{-1}; 0.88 (6H, t, *J* 6.7 Hz, 2 x C$\underline{H}_3$), 1.18-1.35 (28H, m, 14 x C$\underline{H}_2$), 1.50-1.61 (4H, m, 2 x NHCH$_2$C$\underline{H}_2$), 3.00-3.19 (4H, m, 2 x C$\underline{H}_2$NH), 3.49 (1H, dd, $J_{5,5'}$ 9.7 Hz, $J_{4,5'}$ 7.4 Hz, H-5), 3.58 (1H, dd, $J_{5,5'}$ 10.0 Hz, $J_{4,5'}$ 5.7 Hz, H-5'), 3.94-3.97 (1H, m, H-3), 3.97-4.00 (1H, m, H-2), 4.33 (1H, at, *J* 6.8 Hz, H-4), 4.42-4.58 (6H, m, Ph-C$\underline{H}_2$), 5.34 (1H, d, $J_{NH,1}$ 10.5 Hz, H-1), 5.43 (1H, d, *J* 9.7 Hz, NH), 7.22-7.38 (15H, m, Ar-H); β anomer: 0.88 (6H, t, *J* 6.7 Hz, 2 x C$\underline{H}_3$), 1.18-1.35 (28H, m, 14 x C$\underline{H}_2$), 1.50-1.61 (4H, m, 2 x NHCH$_2$C$\underline{H}_2$),

3.04-3.21 (4H, m, 2 x C$\underline{H}_2$NH), 3.54 (2H, at, *J* 4.7 Hz, H-5, H-5'), 3.94-3.97 (1H, m, H-3), 3.97-4.00 (1H, m, H-2), 4.02-4.04 (1H, m, H-4), 4.39-4.61 (6H, m, Ph-CH_2), 5.29 (1H, dd, $J_{NH,1}$ 9.8 Hz, $J_{1,2}$ 3.9 Hz, H-1), 5.43 (1H, d, *J* 9.7 Hz, NH), 7.22-7.38 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, 2 x CH_3), 22.6, 26.8, 26.8, 28.8, 28.8, 29.3, 29.4, 29.6, 29.6, 31.9 (10 x t, 20 x CH_2), 48.9, 48.9 (2 x t, 2 x $\underline{C}H_2$N), 70.2, 70.2 (2 x t, C-5α, C-5β), 71.7, 71.8, 71.8, 72.3, 73.3, 73.4 (6 x t, Ph-$\underline{C}H_2$), 80.5 (d, C-4β), 81.5, 81.9 (2 x d, C-2α, C-2β), 82.5 (d, C-3β), 82.7 (d, C-4α), 84.0 (d, C-1β), 85.1 (d, C-3α), 87.8 (d, C-1α), 127.7, 127.7, 127.7, 127.8, 127.9, 127.9, 128.1, 128.1, 128.2, 128.3, 128.4, 128.4, 128.5, 128.5, 128.6 (15 x d, 15 x Ar-$\underline{C}$), 136.9, 136.9, 137.1, 137.6, 137.8, 138.0 (6 x s, 6 x Ar-$\underline{C}$); HRMS (ESI) calculated for $C_{46}H_{71}N_2O_6S$ 779.5033. Found 779.5045 (MH^+).

3.4.29. *N*-(Didodecyl)-*N*'-(2,3,5-tri-O-benzyl-α,β-D-arabinofuranosyl)sulfamide 5i

General Procedure C, using sulfamide **4i**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.6), afforded glycosylsulfamide **5i** (280 mg, 47 %, α:β, 1:1) as a yellow waxy solid. υ_{max} (neat) 3265 (N-H), 1350 (s, S=O), 1158 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.89 (6H, t, *J* 6.5 Hz, 2 x C$\underline{H}_3$), 1.12-1.37 (36H, m, 18 x C$\underline{H}_2$), 1.52-1.61 (4H, m, 2 x NHCH_2C$\underline{H}_2$), 3.04-3.21 (4H, m, 2 x C$\underline{H}_2$NH), 3.49 (1H, dd, $J_{5,5'}$ 9.7 Hz, $J_{4,5'}$ 7.4 Hz, H-5), 3.59 (1H, dd, $J_{5,5'}$ 10.0 Hz, $J_{4,5'}$ 5.7 Hz, H-5'), 3.94-3.97 (1H, m, H-3), 3.97-4.00 (1H, m, H-2), 4.33 (1H, at, *J* 6.8 Hz, H-4), 4.42-4.58 (6H, m, Ph-C$\underline{H}_2$), 5.35 (1H, d, $J_{NH,1}$ 10.5 Hz, H-1), 5.43 (1H, d, *J* 9.7 Hz, NH), 7.22-7.38 (15H, m, Ar-H); β anomer: 0.89 (6H, t, *J* 6.5 Hz, 2 x C$\underline{H}_3$), 1.12-1.37 (36H, m, 18 x C$\underline{H}_2$), 1.52-1.61 (4H, m, 2 x NHCH_2C$\underline{H}_2$), 3.04-3.21 (4H, m, 2 x C$\underline{H}_2$NH), 3.54 (2H, at, *J* 4.7 Hz, H-5, H-5'), 3.94-3.97 (1H, m, H-3), 3.97-4.00 (1H, m, H-2), 4.02-4.04 (1H, m, H-4), 4.39-4.61 (6H, m, Ph-CH_2), 5.30 (1H, dd, $J_{NH,1}$ 9.8 Hz, $J_{1,2}$ 3.9 Hz, H-1), 5.43 (1H, d, *J* 9.7 Hz, NH), 7.22-7.38 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 14.1 (q, 2 x CH_3), 22.7, 26.8, 28.8, 28.8, 29.4, 29.6, 29.6, 29.7, 29.7, 31.7 (10 x t, 20 x CH_2), 48.9, 48.9 (2 x t, 2 x $\underline{C}H_2$N), 70.2, 70.2 (2 x t, C-5α, C-5β), 71.7, 71.8, 71.8, 72.3, 73.3, 73.4 (6 x t, Ph-$\underline{C}H_2$), 80.5 (d, C-4β), 81.5, 81.9 (2 x d, C-2α, C-2β), 82.5 (d, C-3β), 82.8 (d, C-4α), 84.0 (d, C-1β), 85.1 (d, C-3α), 87.9 (d, C-1α), 127.6, 127.7,127.7, 127.8, 127.8, 127.9, 127.9, 128.1, 128.2, 128.3, 128.4, 128.4, 128.5, 128.5, 128.6 (15 x d, 15 x Ar-$\underline{C}$), 136.8, 136.9, 137.0, 137.8, 137.9, 138.0 (6 x s, 6 x Ar-$\underline{C}$); HRMS (ESI) calculated for $C_{50}H_{79}N_2O_6S$ 835.5659. Found 835.5667 (MH^+).

3.4.30. *N*-(3,7-Dimethyloctyl)-*N*'-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 5j

General Procedure C, using sulfamide **4j**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.6), afforded *N*-(3,7-dimethyloctyl)-*N*'-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide **5j** (250 mg, 80 %, α:β, 1:1) as a yellow waxy solid. υ_{max} (neat) 3270 (w, NH), 1337 (s, S=O), 1071 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 0.88 (9H, d, *J* 6.7

Hz, 3 x CH_3), 1.10-1.16 (2H, m, C$\underline{H}_2$), 1.20-1.32 (4H, m, 2 x C$\underline{H}_2$), 1.48-1.58 (4H, m, 2 x CH, C$\underline{H}_2$), 3.02-3.07 (2H, m, C$\underline{H}_2$NHSO$_2$), 3.48 (1H, dd, $J_{5,5'}$ 9.6 Hz, $J_{4,5'}$ 7.6 Hz, H-5), 3.58 (1H, dd, $J_{5,5'}$ 9.6 Hz, $J_{4,5'}$ 5.7 Hz, H-5'), 3.94-3.97 (1H, m, H-3), 3.98-4.00 (1H, at, *J* 3.1 Hz, H-2), 4.35 (1H, at, *J* 6.8 Hz, H-4), 4.45-4.57 (6H, m, Ph-C$\underline{H}_2$), 5.42 (1H, d, $J_{1,NH}$ 10.6 Hz, H-1), 5.61 (1H, d, J_{NH-1} 10.6 Hz, NH), 7.22-7.37 (15H, m, Ar-H); β anomer: 0.88 (9H, d, *J* 6.7 Hz, 3 x CH_3), 1.10-1.16 (2H, m, C$\underline{H}_2$), 1.20-1.32 (4H, m, 2 x C$\underline{H}_2$), 1.48-1.58 (4H, m, 2 x CH, C$\underline{H}_2$), 3.02-3.07 (2H, m, C$\underline{H}_2$NHSO$_2$), 3.54 (2H, at, *J* 5.1 Hz, H-5, H-5'), 3.94-3.97 (1H, m, H-3), 4.01-4.02 (1H, at, *J* 4.7 Hz, H-2), 4.04-4.07 (1H, dd, $J_{4,5}$ 5.1 Hz, $J_{3,4}$ 3.5 Hz, H-4), 4.45-4.57 (6H, m, Ph-C$\underline{H}_2$), 5.37 (1H, dd, $J_{1,NH}$ 10.2 Hz, $J_{1,2}$ 4.3 Hz, H-1), 5.57 (1H, d, $J_{1,NH}$ 10.2 Hz, NH), 7.22-7.37 (15H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 19.3, 22.6, 22.7 (3 x q, 3 x $\underline{C}H_3$), 24.6 (t, $\underline{C}H_2$), 27.9, 30.4 (2 x d, 2 x $\underline{C}H$), 36.6, 36.6 (2 x t, 2 x $\underline{C}H_2$), 39.2 (t, $\underline{C}H_2$), 41.5, 41.6 (2 x t, NH$\underline{C}H_2$α, NH$\underline{C}H_2$ β), 70.0 (t, C-5α, C-5β), 71.8, 71.8, 72.0, 72.3, 73.3, 73.4 (6 x t, Ph$\underline{C}H_2$), 80.8 (d, C-4β), 81.2, 81.8 (2 x d, C-2α, C-2β), 82.3 (d, C-3β), 83.3 (d, C-4α,), 84.3 (d, C-1β), 84.8 (d, C-3α,), 88.2 (d, C-1α), 127.7, 127.7, 127.7, 127.8, 127.9, 127.9, 127.9, 128.0, 128.2, 128.2, 128.3, 128.4, 128.5, 128.5, 128.6 (15 x d, 15 x Ar-$\underline{C}$), 136.7, 136.8, 136.9, 137.4, 137.7, 137.9 (6 x s, 6 x Ar-$\underline{C}$); HRMS (ESI) calculated for $C_{36}H_{51}N_2O_6S$ 639.3468. Found 639.3469 (MH^+).

3.4.31. *N-(*Hexyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6a

General Procedure F, using sulfamide **5a**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 35-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6a** (18 mg, 42 %) as white solid. $[\alpha]_D^{20}$ -11.6 (*c*, 0.5 in CH_3OH); m.p 125-128 °C ($MeOH/Et_2O$); υ_{max} (neat) 3310 (br, OH), 1318 (s, S=O), 1134 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.92 (3H, t, *J* 7.3 Hz, C$\underline{H}_3$), 1.28-1.38 (6H, m, 3 x C$\underline{H}_2$), 1.48-1.54 (2H, m, NHCH$_2$C$\underline{H}_2$), 2.93-3.01 (2H, m, C$\underline{H}_2$NH), 3.46 (1H, at, *J* 8.3 Hz, H-2), 3.51-3.56 (2H, m, H-3, H-5), 3.77-3.82 (2H, m, H-4, H-5'), 4.27 (1H, at, $J_{1,2}$ 8.9 Hz, H-1), 5.07-5.13 (1H, m, N$\underline{H}$CH$_2$), 6.12 (1H, d, $J_{1,NH}$ 9.8 Hz, N$\underline{H}$SO$_2$); δ_C (100.5 MHz, CD_3OD) 12.9 (q, CH_3), 22.2, 26.2, 29.1, 31.2 (4 x t, 4 x CH_2), 42.6 (t, CH_2NH), 66.7 (t, C-5), 68.3 (d, C-4), 70.0 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{11}H_{24}N_2NaO_6S$ 335.1253. Found 335.1246 (MNa^+).

3.4.32. *N-(*Octyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6b

General Procedure F, using sulfamide **5b**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 35-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6b** (16 mg, 36 %) as white solid. $[\alpha]_D^{20}$ -15.6 (*c*, 0.5 in MeOH); m.p 115-118 °C

(MeOH/Et_2O); υ_{max} (neat) 3315 (br, OH), 1316 (s, S=O), 1152 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.91 (3H, t, *J* 6.7 Hz, C<u>H</u>$_3$), 1.29-1.36 (10H, m, 5 x C<u>H</u>$_2$), 1.48-1.54 (2H, m, NHCH$_2$C<u>H</u>$_2$), 2.93-3.00 (2H, m, C<u>H</u>$_2$NH), 3.45 (1H, at, *J* 8.3 Hz, H-2), 3.51-3.54 (1H, m, H-3), 3.55-3.56 (1H, m, H-5), 3.78-3.80 (1H, m, H-4), 3.81 (1H, d, *J* 2.9 Hz, H-5'), 4.27 (1H, at, $J_{1,2}$ 8.1 Hz, H-1), 5.07-5.13 (1H, m, N<u>H</u>CH$_2$); δ_C (100.5 MHz, CD_3OD) 13.0 (q, CH_3), 22.3, 26.5, 28.8, 29.0, 29.1, 31.8 (6 x t, 6 x CH_2), 42.6 (t, CH_2NH), 66.7 (t, C-5), 68.3 (d, C-4), 70.0 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{13}H_{28}N_2NaO_6S$ 363.1566. Found 363.1570 (MNa^+).

3.4.33. *N-(*Decyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6c

General Procedure F, using sulfamide **5c** and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 50-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6c** (21 mg, 45 %) as white solid; $[\alpha]_D^{20}$ -14 (*c*, 0.5 in CH_3OH); m.p. 103-105 °C (MeOH/Et_2O); υ_{max} (neat) 3340 (br, OH), 1340 (s, S=O), 1157 (s, S=O) cm^{-1}; δ_H (500 MHz, CD_3CN) 0.90 (3H, t, *J* 6.7 Hz, C<u>H</u>$_3$), 1.29-1.36 (14H, m, 7 x C<u>H</u>$_2$), 1.49-1.53 (2H, m, NHCH$_2$C<u>H</u>$_2$), 2.95-2.99 (2H, t, C<u>H</u>$_2$NH), 3.44 (1H, at, *J* 7.3 Hz, H-2), 3.53-3.56 (2H, m, H-3, H-5), 3.77-3.80 (1H, m, H-4), 3.84 (1H, d, $J_{4,5'}$ 4.0 Hz, H-5'), 4.27 (1H, d, $J_{1,2}$ 7.6 Hz, H-1), 5.03 (1H, t, *J* 5.8 Hz, N<u>H</u>CH$_2$); δ_C (100.5 MHz, CD_3OD) 13.0 (q, CH_3), 22.3, 26.5, 29.0, 29.3, 31.6 (5 x t, 8 x CH_2), 42.6 (t, <u>C</u>H$_2$NH), 66.7 (t, C-5), 68.3 (d, C-4), 70.0 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{15}H_{32}N_2NaO_6S$ 391.1879. Found 391.1881 (MNa^+).

3.4.34. *N-(*Dodecyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6d

General Procedure F, using sulfamide **5d**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 50-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6d** (19 mg, 40 %) as white solid. $[\alpha]_D^{20}$ -12.3 (*c*, 0.5 in CH_3OH); m.p 108-110 °C (MeOH/Et_2O); υ_{max} (neat) 3320 (br, OH), 1330 (s, S=O), 1154 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.91 (3H, t, *J* 7.3 Hz, C<u>H</u>$_3$), 1.27-1.36 (18H, m, 9 x C<u>H</u>$_2$), 1.47-1.54 (2H, m, NHCH$_2$C<u>H</u>$_2$), 2.94-3.00 (2H, m, C<u>H</u>$_2$NH), 3.45 (1H, at, *J* 8.0 Hz, H-2), 3.52-3.54 (1H, m, H-3), 3.54-3.56 (1H, m, H-5), 3.77-3.80 (1H, m, H-4), 3.80-3.82 (1H, m, H-5'), 4.27 (1H, d, $J_{1,2}$ 7.3 Hz, H-1), 5.04 (1H, t, *J* 6.6 Hz, N<u>H</u>CH$_2$); δ_C (100.5 MHz, CD_3OD) 13.0 (q, CH_3), 22.3, 26.5, 29.0, 29.0, 29.1, 29.3, 29.3, 29.3, 29.4, 31.6 (10 x t, 10 x CH_2), 42.6 (t, CH_2NH), 66.7 (t, C-5), 68.3 (d, C-4), 70.0 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{17}H_{36}N_2NaO_6S$ 419.2192. Found 419.2201 (MNa^+).

3.4.35. *N*-(Tetradecyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6e

General Procedure F, using sulfamide **5e**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 50-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6e** (23mg, 47 %) as white solid. $[\alpha]_D^{20}$ -11.4 (*c*, 0.5 in MeOH); m.p 118-128 °C (MeOH/Et_2O); υ_{max} (neat) 3410 (br, OH), 1325 (s, S=O), 1149 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.91 (3H, t, *J* 6.5 Hz, C<u>H</u>$_3$), 1.27-1.37 (22H, m, 11 x C<u>H</u>$_2$), 1.49-1.52 (2H, m, NHCH$_2$C<u>H</u>$_2$), 2.94-3.00 (2H, m, C<u>H</u>$_2$NH),), 3.45 (1H, at, *J* 8.3 Hz, H-2), 3.51-3.54 (1H, m, H-3), 3.54-3.56 (1H, m, H-5), 3.77-3.80 (1H, m, H-4), 3.80-3.82 (1H, m, H-5'), 4.27 (1H, d, $J_{1,2}$ 7.8 Hz, H-1), 5.02-5.07 (1H, m, N<u>H</u>CH$_2$); δ_C (100.5 MHz, CD_3OD) 13.0 (q, CH_3), 22.3, 26.5, 29.0, 29.0, 29.1, 29.3, 29.3, 29.3, 29.4, 31.6 (10 x t, 12 x CH_2), 42.6 (t, CH_2NH), 66.7 (t, C-5), 68.3 (d, C-4), 70.0 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{19}H_{41}N_2O_6S$ 425.2685. Found 425.2699 (MNa^+).

3.4.36. *N,N*-(Dihexyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6f

General Procedure F, using sulfamide **5f**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 50-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6f** (12 mg, 41 %) as yellow waxy solid. $[\alpha]_D^{20}$ -12.1 (*c*, 0.5 in MeOH); υ_{max} (neat) 3366 (br, OH), 1334 (s, S=O), 1135 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.92 (6H, t, *J* 6.5 Hz, 2 x C<u>H</u>$_3$), 1.29-1.37 (12H, m, 6 x C<u>H</u>$_2$), 1.53-1.62 (4H, m, 2 x NHCH$_2$C<u>H</u>$_2$), 3.03-3.15 (4H, t, 2 x C<u>H</u>$_2$NH), 3.43 (1H, at, *J* 8.3 Hz, H-2), 3.50-3.55 (2H, m, H-3, H-5), 3.77-3.79 (1H, m, H-4), 3.80-3.82 (1H, m, H-5'), 4.24 (1H, d, $J_{1,2}$ 7.8 Hz, H-1); δ_C (100.5 MHz, CD_3OD) 12.9 (q, 2 x CH_3), 22.3, 26.2, 28.8, 31.3 (4 x t, 8 x CH_2), 49.1 (t, 2 x CH_2NH), 66.4 (t, C-5), 68.2 (d, C-4), 70.1 (d, C-2), 73.5 (d, C-3), 85.0 (d, C-1); HRMS (ESI) calculated for $C_{17}H_{37}N_2O_6S$ 397.2372. Found 397.2380 (MH^+).

3.4.37. *N,N*-(Dioctyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6g

General Procedure F, using sulfamide **5g**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 50-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6g** (14 mg, 45 %) as yellow waxy solid. $[\alpha]_D^{20}$ -14.8 (*c*, 0.5 in MeOH); υ_{max} (neat) 3396 (br, OH), 1313 (s, S=O), 1140 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.91 (6H, t, *J* 6.2 Hz, 2 x C<u>H</u>$_3$), 1.26-1.37 (20H, m, 10 x C<u>H</u>$_2$), 1.54-1.61 (4H, m, 2 x NHCH$_2$C<u>H</u>$_2$), 3.02-3.14 (4H, m, 2 x C<u>H</u>$_2$NH), 3.43 (1H, at, *J* 8.1 Hz, H-2), 3.49-3.55 (2H, m, H-3, H-5), 3.75-3.80 (2H, m, H-4, H-5'),

4.24 (1H, d, $J_{1,2}$ 7.6 Hz, H-1); δ_C (100.5 MHz, CD_3OD) 13.0 (q, 2 x CH_3), 22.3, 26.5, 28.8, 28.9, 28.9, 31.5 (6 x t, 12 x CH_2), 49.1 (t, 2 x CH_2NH), 66.4 (t, C-5), 68.2 (d, C-4), 70.1 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{21}H_{45}N_2O_6S$ 453.2998. Found 453.3007 (MH^+).

3.4.38. *N,N-(*Didecyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6h

General Procedure F, using sulfamide **5h**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 50-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6h** (16 mg, 41 %) as yellow waxy solid. $[\alpha]_D^{20}$ -13.2 (*c*, 0.5 in MeOH); υ_{max} (neat) 3389 (w, NH), 1337 (s, S=O), 1137 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.91 (6H, t, *J* 6.5 Hz, 2 x C<u>H</u>$_3$), 1.27-1.37 (28H, m, 14 x C<u>H</u>$_2$), 1.54-1.61 (4H, m, 2 x NHCH$_2$C<u>H</u>$_2$), 3.04-3.15 (4H, t, 2 x C<u>H</u>$_2$NH), 3.44 (1H, at, *J* 8.1 Hz, H-2), 3.50-3.57 (2H, m, H-3, H-5), 3.77-3.82 (2H, m, H-4, H-5'), 4.26 (1H, at, *J* 9.0 Hz, H-1), 5.96 (1H, d, $J_{1,NH}$ 9.8 Hz, N<u>H</u>SO$_2$); δ_C (100.5 MHz, CD_3OD) 13.0 (q, 2 x CH_3), 22.3, 26.4, 28.8, 28.9, 29.0, 29.3, 29.3, 31.6 (8 x t, 16 x CH_2), 49.1 (t, 2 x CH_2NH), 66.4 (t, C-5), 68.2 (d, C-4), 70.1 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{25}H_{53}N_2O_6S$ 509.3624. Found 509.3634 (MH^+).

3.4.39. *N,N-(*Didodecyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6i

General Procedure F, using sulfamide **5i**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 50-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6i** (13 mg, 39 %) as yellow waxy solid. $[\alpha]_D^{20}$ -15.2 (*c*, 0.5 in MeOH); υ_{max} (neat) 3385 (br, OH), 1315 (s, S=O), 1132 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.91 (6H, t, *J* 6.5 Hz, 2 x C<u>H</u>$_3$), 1.27-1.37 (36H, m, 18 x C<u>H</u>$_2$), 1.54-1.61 (4H, m, 2 x NHCH$_2$C<u>H</u>$_2$), 3.04-3.15 (4H, t, 2 x C<u>H</u>$_2$NH), 3.44 (1H, at, *J* 8.1 Hz, H-2), 3.50-3.57 (2H, m, H-3, H-5), 3.77-3.82 (2H, m, H-4, H-5'), 4.26 (1H, at, *J* 9.0 Hz, H-1), 5.96 (1H, d, $J_{1,NH}$ 9.8 Hz, N<u>H</u>SO$_2$); δ_C (100.5 MHz, CD_3OD) 13.0 (q, 2 x CH_3), 22.3, 26.4, 28.8, 29.0, 29.1, 29.2, 29.3, 29.3, 29.4, 31.6 (10 x t, 20 x CH_2), 49.1 (t, 2 x CH_2NH), 66.4 (t, C-5), 68.2 (d, C-4), 70.1 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{29}H_{61}N_2O_6S$ 565.4250. Found 565.4255 (MH^+)

3.4.40. *N-(*3,7-Dimethyloctyl)-*N'*-(α-D-arabinopyranosyl)sulfamide 6j

General Procedure F, using sulfamide **5j**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 50-85 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **6j** (10 mg, 43 %) as yellow waxy solid. $[\alpha]_D^{20}$ -11.2 (*c*, 0.5 in MeOH); υ_{max} (neat) 3391

(br, OH), 1314 (s, S=O), 1134 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3CN) 0.88-0.91 (9H, m, 3 x CH_3), 1.16-1.21 (2H, m, CH_2), 1.32-1.36 (6H, m, 3 x CH_2), 1.54-1.59 (2H, m, 2 x CH), 2.98-3.05 (2H, m, $C\underline{H}_2NH$), 3.44 (1H, at, *J* 8.1 Hz, H-2), 3.50-3.57 (2H, m, H-3, H-5), 3.76-3.86 (2H, m, H-4, H-5'), 4.27 (1H, at, *J* 9.0 Hz, H-1), 5.00-5.02 (1H, m, $N\underline{H}CH_2$), 6.11 (1H, d, $J_{1,NH}$ 10.2 Hz, $N\underline{H}SO_2$); δ_C (100.5 MHz, CD_3OD) 18.4, 22.5, 22.6 (3 x q, 3 x $\underline{C}H_3$), 24.4 (t, $\underline{C}H_2$), 27.7, 30.2 (2 x d, 2 x $\underline{C}H$), 36.2 (t, $\underline{C}H_2$), 36.9 (t, $\underline{C}H_2$), 39.0 (t, $\underline{C}H_2CH_2NH$), 40.7 (t, $\underline{C}H_2NH$), 66.7 (t, C-5), 68.3 (d, C-4), 70.1 (d, C-2), 73.5 (d, C-3), 85.1 (d, C-1); HRMS (ESI) calculated for $C_{15}H_{32}N_2NaO_6S$ 391.1879. Found 391.1887 (MNa^+).

3.4.41. 1-Azido-3,7-dimethyloctane 8

General procedure D, using alcohol **7**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.9), afforded azide **8** (4.26 g, 75 %) as a clear oil. υ_{max} (KBr) 2096 (s, N_3) cm^{-1}; δ_H (500 MHz, $CDCl_3$) 0.85-0.89 (9H, m, 3 x CH_3), 1.11-1.15 (2H, m, $C\underline{H}_2$), 1.21-1.31 (2H, m, $C\underline{H}_2$), 1.35-1.41 (2H, m, $C\underline{H}_2$), 1.48-1.50 (2H, m, $C\underline{H}_2$), 1.51-1.64 (2H, m, 2 x $C\underline{H}$), 3.23-3.29 (2H, m, $C\underline{H}_2N_3$); δ_C (125 MHz, $CDCl_3$) 19.3, 22.6, 22.7 (3 x q, 3 x $\underline{C}H_3$), 24.6 (t, $\underline{C}H_2$), 27.9, 30.3 (2 x d, 2 x $\underline{C}H$), 35.7, 37.0, 39.2 (3 x t, 3 x $\underline{C}H_2$), 49.5 (t, $\underline{C}H_2N_3$); HRMS (ESI) calculated for $C_{10}H_{24}NO$ 174.1858. Found 174.1853 ($M\text{-}N_2\text{+}H_3O^+$). Required for $C_{10}H_{21}N_3$: C 65.53 %, H 11.55 %, 22.93 %; Found C 65.74 %, H 11.54 %, 22.72 %.

3.4.42. 3,7-Dimethyl-1-octanamine 9

General procedure E, using azide **8**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.2), afforded amine **9** (1.6 g, 63 %) as a clear oil. υ_{max} (neat) 3260 (w, C-NH_2) cm^{-1}; δ_H (500 MHz, $CDCl_3$) 0.81-0.83 (9H, m, 3 x $C\underline{H}_3$), 1.10-1.27 (8H, m, 4 x $C\underline{H}_2$), 1.40-1.49 (2H, m, 2 x $C\underline{H}$), 1.89 (s,$N\underline{H}_2$), 2.63-2.70 (2H, m, $C\underline{H}_2NH$); δ_C (125 MHz, $CDCl_3$) 19.6, 22.6, 22.7 (3 x q, 3 x $\underline{C}H_3$), 24.6 (t, $\underline{C}H_2$), 27.9, 30.4 (2 x d, 2 x $\underline{C}H$), 37.3, 39.2, 39.9 (3 x t, 3 x $\underline{C}H_2$), 40.7 (t, $\underline{C}H_2NH$); (ESI) calculated for $C_{10}H_{24}N$: 158.1909. Found: 158.1906 (MH^+).

3.4.43. 2-(2-(Benzyloxy)ethoxy)ethanol 11a[24]

General procedure G, using diethylene glycol **10a**, and purification by flash column chromatography (petrol: ethyl acetate, 4:1 to 1:1, R_f 0.2 in 1:1), afforded benzyl ether **11a** (6.20 g, 25% yield) as pale yellow oil. δ_H (400 MHz, $CDCl_3$) 3.61-3.65 (4H, m, 2 x $C\underline{H}_2$), 3.69-3.75 (4H, m, 2 x $C\underline{H}_2$), 4.58 (2H, s, $PhC\underline{H}_2$), 7.28-7.35 (5H, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 61.7 (t, $\underline{C}H_2OH$), 69.4, 70.4, 72.5 (3 x t, 3 x $O\underline{C}H_2$), 73.3 (t, $Ph\underline{C}H_2$), 127.7, 127.9, 128.5 (3 x d, Ar-C), 138.0 (s, Ar-C); HRMS(ESI): calculated for $C_{11}H_{17}O_3$: 197.1172. Found: 197.1169 (MH^+).

3.4.44. 2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethanol 11b[25]

General procedure G, using triethyleneglycol **10b**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.2), afforded benzyl ether **11b** (6.56 g, 36 %) as a pale yellow oil. δ_H (400 MHz, $CDCl_3$) 2.75 (1H, s, O<u>H</u>), 3.58-3.71 (12H, m, 6 x OC<u>H</u>$_2$), 4.55 (2H, s, PhC<u>H</u>$_2$), 7.26-7.33 (5H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 61.7 (t, <u>C</u>H_2OH), 69.4 (t, O<u>C</u>H_2), 70.4 (t, O<u>C</u>H_2), 70.6 (t, O<u>C</u>H_2), 70.7 (t, O<u>C</u>H_2), 72.5 (t, O<u>C</u>H_2), 73.2 (t, Ph<u>C</u>H_2), 127.6, 127.7, 128.3 (3 x d, Ar-C), 138.2 (s, Ar-C); HRMS (ESI) calculated for $C_{13}H_{20}NaO_4$: 263.1259 Found: 263.1250 (MNa^+).

3.4.45. 2-(2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethoxy)ethanol 11c[26]

General procedure G, using tetraethylene glycol **10c**, and purification by flash chromatography (petrol: ethyl acetate, 5:1 to ethyl acetate, R_f 0.2 in 3:1), afforded benzyl ether **11c** (23.13 g, 65% yield) as a yellow oil. δ_H (400 MHz, $CDCl_3$) 3.59-3.72 (16H, m, 8 x OC<u>H</u>$_2$), 4.57 (2H, s, PhC<u>H</u>$_2$), 7.26-7.34 (5H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 61.8 (t, <u>C</u>H_2OH), 69.5 (t, O<u>C</u>H_2), 70.4 (t, O<u>C</u>H_2), 70.6 (t, O<u>C</u>H_2), 70.6 (t, O<u>C</u>H_2), 70.7 (t, O<u>C</u>H_2), 70.7 (t, O<u>C</u>H_2), 72.7 (t, O<u>C</u>H_2), 73.3 (t, Ph<u>C</u>H_2), 127.7, 127.8, 128.4 (3 x d, Ar-C), 138.2(s, Ar-C); HRMS (ESI): calculated for $C_{15}H_{25}O_5$: 285.1697. Found: 285.1702 (MH^+).

3.4.46. ((2-(2-Azidoethoxy)ethoxy)methyl)benzene 12a

General procedure D, using alcohol **11a**, and purification by flash chromatography (petrol: ethyl acetate, 1:1, R_f 0.8), afforded azide **12a** (0.45 g, 94 %) as a clear oil. υ_{max} (neat) 2099 (s, N_3) cm^{-1}; δ_H (500 MHz, $CDCl_3$) 3.39 (2H, t, *J* 5.0 Hz, C<u>H</u>$_2$N$_3$), 3.64-3.69 (6H, m, 3 x C<u>H</u>$_2$), 4.58 (2H, s, PhC<u>H</u>$_2$), 7.27-7.35 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 50.7 (t, <u>C</u>H_2N$_3$), 69.4, 70.0, 70.7 (3 x t, 3 x <u>C</u>H_2), 73.3 (t, Ph<u>C</u>H_2), 127.6, 127.7, 128.4 (3 x d, 5 x Ar-C), 138.1 (s, Ar-C); HRMS (ESI) calculated for $C_{11}H_{15}N_3NaO_2$ 244.1062. Found 244.1074 (MNa^+).

3.4.47. ((2-(2-(2-Azidoethoxy)ethoxy)ethoxy)methyl)benzene 12b

General procedure D, using alcohol **11b**, and purification by flash chromatography (petrol: ethyl acetate, 1:1, R_f 0.4), afforded azide **12b** (4.38 g, 98 %) as a clear oil. υ_{max} (neat) 2097 (s, N_3) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 3.36 (2H, t, *J* 4.0 Hz, C<u>H</u>$_2$N$_3$), 3.63-3.68 (10H, m, 5 x C<u>H</u>$_2$), 4.56 (2H, s, PhC<u>H</u>$_2$), 7.26-7.34 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 50.6 (t, <u>C</u>H_2N$_3$), 69.4, 70.0, 70.6, (3 x t, 5 x <u>C</u>H_2), 73.2 (t, Ph<u>C</u>H_2), 127.5, 127.7, 128.3 (3 x d, 5 x Ar-C), 138.2 (s, Ar-C); HRMS (ESI) calculated for $C_{13}H_{19}N_3NaO_3$ 288.1324. Found 288.1319 (MNa^+).

3.4.48. ((2-(2-(2-(2-Azidoethoxy)ethoxy)ethoxy)ethoxy)methyl)benzene 12c

General procedure D, using alcohol **11c,** and purification by flash chromatography (petrol: ethyl acetate, 1:1, R_f 0.5), afforded azide **12c** (0.54 g, 91 %) as a clear oil. υ_{max} (neat) 2097 (s, N_3) cm-1; δ_H (500 MHz, $CDCl_3$) 3.37 (2H, t, *J* 5.0 Hz, $C\underline{H}_2N_3$), 3.63-3.66 (14H, m, 7 x CH_2), 4.56 (2H, s, $PhC\underline{H}_2$), 7.26-7.33 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 50.7 (t, $\underline{C}H_2N_3$), 69.5, 70.1, 70.5, 70.6, 70.7, 70.8, 70.9 (7 x t, 7 x $\underline{C}H_2$), 73.3 (t, Ph-$\underline{C}H_2$), 127.9, 128.3, 128.6 (3 x d, 3 x Ar-C, 138.3 (s, Ar-C); HRMS (ESI) calculated for $C_{115}H_{24}N_3O_4$ 310.1767. Found 310.1764 (MH^+).

3.4.49. 2-(2-(Benzyloxy)ethoxy)ethanamine 13a

General procedure E, using azide **12a**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.1), afforded amine **13a** (0.25 g, 71 %) as a clear oil. υ_{max} (neat) 3260 (w, NH_2) cm^{-1} ; δ_H (500 MHz, $CDCl_3$) 2.09 (2H, s, $N\underline{H}_2$), 2.85 (2H, t, *J* 5.0 Hz, $OC\underline{H}_2$), 3.50 (2H, t, *J* 5.0 Hz, $C\underline{H}_2NH_2$), 3.62-3.64 (4H, m, 2 x $C\underline{H}_2$) 4.56 (2H, s, $PhC\underline{H}_2$), 7.25-7.34 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 41.3 (t, $\underline{C}H_2NH_2$), 69.4,70.3 (2 x t, 2 x $\underline{C}H_2$), 72.3 (t, $O\underline{C}H_2CH_2NH_2$), 73.3 (t, Ph-$\underline{C}H_2$), 127.7, 127.8, 128.3, 128.3, 128.4 (5 x d, 5 x Ar-C), 138.0 (s, Ar-C); HRMS (ESI) calculated for $C_{11}H_{18}NO_2$ 196.1338. Found 196.1336 (MH^+).

3.4.50. 2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethanamine 13b

General procedure E, using azide **12b**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.1), afforded amine **13b** (3.10 g, 80 %) as a clear oil. υ_{max} (neat) 3260 (w, NH_2) cm^{-1} ; δ_H (400 MHz, $CDCl_3$)[3] 1.81 (2H, s, $N\underline{H}_2$), 2.84 (2H, t, *J* 6.0 Hz, $OC\underline{H}_2CH_2NH_2$), 3.41 (2H, t, *J* 8.0 Hz, $C\underline{H}_2NH_2$), 3.61-3.67 (8H, m, 4 x $C\underline{H}_2$) 4.55 (2H, s, $PhC\underline{H}_2$), 7.24-7.32 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 41.7 (t, $\underline{C}H_2NH_2$), 69.4, 70.3, 70.56 (3 x t, 5 x $\underline{C}H_2$), 73.2 (t, $Ph\underline{C}H_2$), 127.6, 127.7, 128.3 (3 x d, 5 x Ar-C), 138.2 (s, Ar-C); HRMS (ESI) calculated for $C_{13}H_{21}NNaO_3$ 262.1419. Found 262.1414 (MNa^+).

3.4.51. 2-(2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethoxy)ethanamine 13c

General procedure E, using azide **12c**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.1), afforded amine **13c** (0.23 g, 67 %) as a clear oil. υ_{max} (neat) 3264 (w, NH_2) cm^{-1}; δ_H (500 MHz, $CDCl_3$) 2.23 (2H, s, -$N\underline{H}_2$), 2.79 (2H, t, *J* 5.0 Hz, $OC\underline{H}_2CH_2NH_2$), 3.52 (2H, t, *J* 5.0 Hz, $C\underline{H}_2NH_2$), 3.62-3.68 (12H, m, 6 x $C\underline{H}_2$) 4.56 (2H, s, $PhC\underline{H}_2$), 7.25-7.34 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 40.9 (t, $\underline{C}H_2NH_2$), 69.4, 69.6, 70.3, 70.4, 70.4, 70.6, 71.0 (7 x t, 7 x CH_2), 73.2 (t, $Ph\underline{C}H_2$), 127.8, 128.0, 128.3 (3 x d, 5 x Ar-C), 137.8 (s, Ar-C); HRMS (ESI) calculated for $C_{15}H_{26}NO_4$ 284.1862. Found 284.1863 (MH^+).

3.4.52. *T*ert-butyl *N*-2-(2-(Benzyloxy)ethoxy)ethylsulfamoylcarbamate 14a

General procedure A, using amine **13a**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.5), afforded sulfamoylcarbamate **14a** (0.36 g, 93 %) as a white solid. m.p. 68-70 °C (petrol/ ethyl acetate); υ_{max} (neat) 1708 (s, C=O), 1351 (s, S=O), 1139 (s, S=O) cm^{-1}; δ_H (500 MHz, $CDCl_3$) 1.46-1.48 (9H, s, C($C\underline{H}_3$)$_3$), 3.29 (2H, q, *J* 5.5 Hz, $C\underline{H}_2NH$), 3.61-3.64 (6H, m, 3 x $C\underline{H}_2$), 4.57 (2H, s, Ph$C\underline{H}_2$), 5.63(1H, t, *J* 10 Hz, $CH_2N\underline{H}$), 7.26-7.35 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 27.9 (q, C($\underline{C}H_3$)$_3$), 43.6 (t, $\underline{C}H_2NH$), 69.3, 70.2 (2 x t, 3 x $\underline{C}H_2$), 73.3 (t, Ph$\underline{C}H_2$), 83.5 (s, $\underline{C}(CH_3)_3$), 127.7, 127.9, 128.4 (3 x d, 3 x Ar-$\underline{C}$), 138.0 (s, Ar-$\underline{C}$), 150.4 (s, C=O); HRMS (ESI) calculated for $C_{16}H_{26}N_2NaO_6S$ 397.1409. Found 397.1410 (MNa^+).

3.4.53. *Tert*-butyl *N*-2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethylsulfamoylcarbamate 14b

General procedure A, using amine **13b**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.4), afforded sulfamoylcarbamate **14b** (1.35 g, 39 %) as a clear oil. υ_{max} (neat) 1715 (s, C=O), 1350 (s, S=O), 1150 (s, S=O) cm^{-1}; δ_H (500 MHz, $CDCl_3$) 1.44-1.47 (9H, s, C($C\underline{H}_3$)$_3$), 3.27-3.30 (2H, m, $C\underline{H}_2NH$), 3.61-3.67 (10H, m, 5 x $C\underline{H}_2$), 4.57 (2H, s, Ph-$C\underline{H}_2$), 7.26-7.34 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 28.0 (q, C($\underline{C}H_3$)$_3$), 43.6 (t, $\underline{C}H_2NH$), 69.3, 70.1, 70.6 (3 x t, 5 x CH_2), 73.2 (t, Ph$\underline{C}H_2$), 83.2 (s, $\underline{C}(CH_3)_3$), 127.6, 127.8, 128.3 (3 x d, 5 x Ar-$\underline{C}$), 138.1 (s, Ar-$\underline{C}$), 150.4 (s, C=O); HRMS (ESI) calculated for $C_{18}H_{30}N_2NaO_7S$ 441.1671. Found 441.1666 (MNa^+).

3.4.54. *Tert*-butyl *N*-2-(2-(2-(2-(benzyloxy)ethoxy)ethoxy)ethoxy)ethylsulfamoylcarbamate 14c

General procedure A, using amine **13c**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.3), afforded sulfamoylcarbamate **14c** (0.33 g, 91 %) as a clear oil. υ_{max} (neat) 1729 (s, C=O), 1350 (s, S=O), 1090 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 1.47 (9H, s, C($\underline{C}H_3$)$_3$), 3.29-3.31 (2H, m, $C\underline{H}_2NH$), 3.59-3.68 (14H, m, 7 x $C\underline{H}_2$), 4.57 (2H, s, Ph$C\underline{H}_2$), 7.25-7.34 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 28.1 (q, C($\underline{C}H_3$)$_3$), 43.7 (t, $\underline{C}H_2NH$), 69.4, 70.1, 70.6 (3 x t, 7 x CH_2), 73.2 (t, Ph$\underline{C}H_2$), 83.2 (s, $\underline{C}(CH_3)_3$), 127.6, 127.8, 128.3 (3 x d, 3 x Ar-$\underline{C}$), 138.2 (s, Ar-$\underline{C}$), 150.5 (s, C=O); HRMS (ESI) calculated for $C_{20}H_{34}N_2NaO_8S$ 485.1934. Found 485.1921 (MNa^+).

3.4.55. *N*-(2-(2-(Benzyloxy)ethoxy)ethyl)sulfamide 15a

General procedure B, using sulfamoylcarbamate **14a**, and purification by flash chromatography (DCM: MeOH, 19:1, R_f 0.2), afforded sulfamide **15a** (1.1 g, 88 %) as a yellowish brown waxy solid. υ_{max} (neat) 3253 (N-H), 1333 (s, S=O), 1127 (s, S=O); δ_H (400 MHz, $CDCl_3$) 3.34 (2H, t, *J* 4.0 Hz, $C\underline{H}_2NH$), 3.61-3.65 (6H, m, 3 x $C\underline{H}_2$), 4.54 (2H, s, Ph$C\underline{H}_2$), 7.26-7.38 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 43.1 (t, $\underline{C}H_2NH$), 68.6, 69.7 (2 x t, 3 x CH_2), 72.8 (t, Ph-$\underline{C}H_2$), 127.8, 127.9, 128.4 (3 x d, 5 x Ar-$\underline{C}$), 137.1 (s, Ar-$\underline{C}$); HRMS (ESI) calculated for $C_{11}H_{18}N_2NaO_4S$ 297.0855. Found 297.0870 (MNa^+).

3.4.56. *N*-2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethyl)sulfamide 15b

General procedure B, using sulfamoylcarbamate **14b**, and purification by flash chromatography (petrol: ethyl acetate, 1:2, R_f 0.3), afforded sulfamide **15b** (0.76 g, 76 %) as a yellow waxy solid. υ_{max} (neat) 3277 (N-H), 1335 (s, S=O), 1132 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 3.32 (2H, t, *J* 4.7 Hz, C$\underline{H}_2$NH), 3.62-3.68 (10H, m, 5 x C$\underline{H}_2$), 4.57 (2H, s, Ph-C$\underline{H}_2$), 7.26-7.36 (5H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 43.4 (t, $\underline{C}H_2$NH), 69.1, 69.7, 70.2, 70.3 (4 x t, 5 x $\underline{C}H_2$), 73.1 (t, Ph$\underline{C}H_2$), 127.8, 128.0, 128.4 (3 x d, 5 x Ar-$\underline{C}$), 137.7 (s, Ar-$\underline{C}$); HRMS (ESI) calculated for $C_{13}H_{22}N_2NaO_5S$ 341.1147. Found 341.1142 (MNa^+).

3.4.57. *N*-2-(2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethoxy)ethyl)sulfamide 15c

General procedure B, using sulfamoylcarbamate **14c**, and purification by flash chromatography (petrol: ethyl acetate, 1:2, R_f 0.2), afforded sulfamide **15c** (0.15 g, 65 %) as a yellow waxy solid. υ_{max} (neat) 3251 (N-H), 1332 (s, S=O), 1082 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) 3.29 (2H, t, *J* 4.0 Hz, C$\underline{H}_2$NH), 3.59-3.65 (14H, m, 7 x C$\underline{H}_2$), 4.58 (2H, s, PhC$\underline{H}_2$), 7.26-7.35 (5H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 43.6 (t, $\underline{C}H_2$NH), 69.2, 69.6, 69.9, 70.2 (4 x t, 7 x $\underline{C}H_2$), 73.2 (t, Ph$\underline{C}H_2$), 127.7, 128.1, 128.4 (3 x d, 3 x Ar-$\underline{C}$), 137.9 (s, Ar-$\underline{C}$); HRMS (ESI) calculated for $C_{15}H_{26}N_2NaO_6S$ 385.1409. Found 385.1400 (MNa^+).

3.4.58. *N*-(2-(2-(Benzyloxy)ethoxy)ethyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 16a

General procedure C, using sulfamide **15a**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.2), afforded glycosylsulfamide **16a** (0.36 g, 74 %, α:β, 1:1) as a yellow waxy solid. υ_{max} (neat) 3267 (N-H), 1348 (s, S=O), 1074 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 3.18-3.27 (2H, m, NHC$\underline{H}_2$), 3.47-3.49 (1H, m, H-5), 3.55-3.63 (7H, m, 3 x CH_2, H-5'), 3.93-3.97 (1H, m, H-3), 4.01 (1H, at, *J* 3.5 Hz, H-2), 4.36 (1H, t, *J* 5.5 Hz, H-4), 4.45-4.56 (8H, m, PhC$\underline{H}_2$), 5.40 (1H, d, $J_{1,NH}$ 10.2 Hz, H-1), 5.78 (1H, d, $J_{NH,1}$ 10.6 Hz, NH), 7.22-7.36 (20H, m, Ar-H); β anomer: 3.18-3.27 (2H, m, NHC$\underline{H}_2$), 3.52 (2H, d, *J* 5.5 Hz, H-5, H-5'), 3.55-3.63 (6H, m, 3 x CH_2), 3.93-3.97 (1H, m, H-3), 3.99 (1H, at, *J* 4.3 Hz, H-2), 4.03-4.05 (1H, m, H-4), 4.45-4.56 (8H, m, PhC$\underline{H}_2$), 5.36 (1H, dd, $J_{1,2}$ 4.3 Hz, $J_{NH,1}$ 10.2 Hz, H-1), 5.62 (1H, d, $J_{NH,1}$ 10.2 Hz, NH), 7.22-7.36 (20H, m, Ar-H); δ_C (100.5 MHz, $CDCl_3$) 43.1, 43.3 (2 x t, NH$\underline{C}H_2$α, NH$\underline{C}H_2$β), 69.3, 69.6, 69.7 (3 x t, 3x CH_2), 70.1, 70.2 (2 x t, C-5α, C-5β), 71.7, 71.8, 71.9, 72.3,73.2, 73.2, 73.3, 73.4 (8 x t, 8 x Ph$\underline{C}H_2$), 80.7 (d, C-4β), 81.2, 81.8 (2 x d, C-2α, C-2β), 82.4 (d, C-3β), 82.6 (d, C-4α), 84.2 (d, C-1β), 85.2 (d, C-3α), 88.3 (d, C-1α), 127.7, 127.7, 127.7, 127.8, 127.8, 127.8, 127.9, 128.0, 128.1, 128.2, 128.2, 128.4, 128.4, 128.5, 128.5, 128.5, 128.6, 128.6 (18 x d, Ar-$\underline{C}$), 136.8, 136.9, 137.0,

137.5, 137.7, 137.9, 137.9, 138.0 (8 x s, Ar-C); HRMS (ESI) calculated for $C_{37}H_{44}N_2NaO_8S$ 699.2716. Found 699.2714 (MNa^+).

3.4.59. *N*-2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 16b

General procedure C, sulfamide **15b**, and purification by flash chromatography (petrol: ethyl acetate, 2:1, R_f 0.2), afforded glycosylsulfamide **16b** (0.29 g, 83 %, α:β, 1.1) as a yellow waxy solid. υ_{max} (neat) 3262 (N-H), 1343 (s, S=O), 1068(s, S=O) cm^{-1} δ_H (400 MHz, $CDCl_3$) α anomer: 3.18-3.22 (2H, m, NHC$\underline{H}_2$), 3.48 (1H, dd, *J* 9.6 Hz, *J* 7.2 Hz, H-5), 3.55-3.65 (11H, m, 5 x CH_2, H-5'), 3.93-3.97 (1H, m, H-3), 4.00 (1H, at, *J* 2.7 Hz, H-2), 4.36 (1H, t, *J* 6.6 Hz, H-4), 4.41-4.58 (8H, m, PhC$\underline{H}_2$), 5.41 (1H, d, $J_{1,NH}$ 8.6 Hz, H-1), 5.72 (1H, d, $J_{NH,1}$ 10.2 Hz, NH) , 7.22-7.35 (20H, m, Ar-H); β anomer: 3.18-3.22 (2H, m, NHC$\underline{H}_2$), 3.52 (2H, d, *J* 5.5 Hz, H-5, H-5'), 3.55-3.65 (10H, m, 5 x CH_2), 3.93-3.97 (1H, m, H-3), 3.98 (1H, at, *J* 3.5 Hz, H-2), 4.01-4.05 (1H, m, H-4), 4.41-4.58 (8H, m, $PhCH_2$), 5.36 (1H, dd, $J_{1,2}$ 4.5 Hz, $J_{NH,1}$ 10.0 Hz, H-1), 5.62 (1H, d, $J_{NH,1}$ 9.8 Hz, NH), 7.22-7.35 (20H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 43.0, 43.1 (2 x t, $NHCH_2$α, $NHCH_2$β), 69.4, 69.4, 69.5, 70.0, 70.1 (5 x t, 5 x CH_2), 70.5, 70.6 (2 x t, C-5α, C-5β), 71.6, 71.7, 71.8, 71.9, 72.2, 73.1, 73.3, 73.4 (8 x t, 8 x $PhCH_2$), 80.6 (d, C-4β), 81.2, 81.8 (2 x d, C-2α, C-2β), 82.4 (d, C-3β), 82.6 (d, C-4α), 84.2 (d, C-1β), 85.2 (d, C-3α), 88.3 (d, C-1α), 127.6, 127.7, 127.7, 127.8, 127.8, 127.8, 127.9, 127.9, 128.0, 128.1, 128.2, 128.2, 128.4, 128.4, 128.5, 128.5, 128.6, 128.6 (18 x d, Ar-C), 136.8, 136.9, 137.0, 137.5, 137.7, 138.0, 138.2, 138.2 (8 x s, Ar-C); HRMS (ESI) calculated for $C_{39}H_{48}N_2NaO_9S$ 743.2978. Found 743.2980 (MNa^+).

3.4.60. *N*-2-(2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethoxy)ethyl)-*N'*-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 16c

General procedure C, using sulfamide **15c**, and purification by flash chromatography (petrol: ethyl acetate, 1:1, R_f 0.2), afforded glycosylsulfamide **16c** (0.23 g, 62 %, α:β, 1:1) as a yellow oil. υ_{max} (neat) 3254 (N-H), 1346 (s, S=O), 1089 (s, S=O) cm^{-1}; δ_H (400 MHz, $CDCl_3$) α anomer: 3.19-3.23 (2H, m, NHC$\underline{H}_2$), 3.47-3.49 (1H, m, H-5), 3.55-3.65 (15H, m, 7 x CH_2, H-5'), 3.93-3.97 (1H, m, H-3), 3.99 (1H, at, *J* 2.0 Hz, H-2), 4.36 (1H, t, *J* 6.2 Hz, H-4), 4.42-4.59 (8H, m, $PhCH_2$), 5.41 (1H, d, $J_{NH,1}$ 9.8 Hz, H-1), 5.73 (1H, d, $J_{NH,1}$ 10.9 Hz, NH), 7.23-7.35 (20H, m, Ar-H); β anomer: 3.19-3.23 (2H, m, $NHCH_2$), 3.52 (2H, d, *J* 5.1 Hz, H-5, H-5'), 3.55-3.65 (14H, m, 7 x CH_2), 3.93-3.97 (1H, m, H-3), 4.01 (1H, at, *J* 3.5 Hz, H-2), 4.03-4.05 (1H, m, H-4), 4.42-4.59 (8H, m, $PhCH_2$), 5.37 (1H, dd, $J_{1,2}$ 4.5 Hz, $J_{NH,1}$ 10.4 Hz, H-1), 5.63 (1H, d, $J_{NH,1}$ 10.2 Hz, NH), 7.23-7.35 (20H, m, Ar-H); δ_C (125 MHz, $CDCl_3$) 43.0, 43.1 (2 x t, $NHCH_2$α, $NHCH_2$β), 69.4, 69.5, 69.5, 70.0, 70.1, 70.1, 70.2 (7 x t, 7x CH_2), 70.5, 70.6 (2 x t, C-5α, C-5β), 71.7, 71.8, 71.9, 72.3, 73.2, 73.2, 73.3, 73.4 (8 x t, 8 x

$PhCH_2$), 80.6 (d, C-4β), 81.3, 81.8 (2 x d, C-2α, C-2β), 82.5 (d, C-3β), 82.6 (d, C-4α), 84.2 (d, C-1β), 85.2 (d, C-3α), 88.2 (d, C-1α), 127.6, 127.7, 127.7, 127.8, 127.8, 127.8, 127.9, 127.9, 128.1, 128.2, 128.2, 128.3, 128.4, 128.5, 128.5, 128.5, 128.6, 128.6 (18 x d, Ar-C), 136.8, 136.9, 137.0, 137.5, 137.7, 138.0, 138.2, 138.2 (8 x s, Ar-C); HRMS (ESI) calculated for $C_{41}H_{52}N_2NaO_{10}S$ 787.3240. Found 787.3247 (MNa^+).

3.4.61. *N*-(2-(2-Ethoxy)ethanol-*N'*-(α-D-arabinopyranosyl)sulfamide 17a

General Procedure F, using sulfamide **16a**, and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 10-35 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **17a** (6 mg, 46 %) as yellow waxy solid. $[\alpha]_D^{20}$ -11.7 (*c*, 0.5 in MeOH); υ_{max} (neat) 3328 (br, OH), 1324 (s, S=O), 1130 (s, S=O) cm^{-1}; δ_H (500 MHz, CD_3OD) 3.19-3.24 (2H, m, $NHCH_2$), 3.52-3.56 (4H, m, CH_2, H-2, H-3), 3.57-3.59 (1H, m, H-5), 3.60-3.61 (2H, m, CH_2), 3.67 (2H, t, *J* 5.0 Hz, CH_2), 3.82-3.84 (1H, m, H-4), 3.85-3.87 (1H, m, H-5'), 4.32 (1H, d, $J_{1,2}$ 7.8 Hz, H-1); δ_C (125 MHz, CD_3OD) 42.4 (t, CH_2NH), 60.8 (t, CH_2), 66.8 (t, C-5), 68.3 (d, C-4), 69.4 (t, CH_2), 70.0 (d, C-2), 71.9 (t, CH_2), 73.5 (d, C-3), 85.2 (d, C-1); HRMS (ESI) calculated for $C_9H_{20}N_2NaO_8S$ 339.0838. Found 339.0830 (MNa^+).

3.4.62. *N*-(2-(2-(2-(Ethoxy)ethoxy)ethanol-*N'*-(α-D-arabinopyranosyl)sulfamide 17b

General Procedure F, using sulfamide **16b,** and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 10-35 % B; column oven: 15 °C; detection: CAD), afforded de-protected sulfamide **17b** (6 mg, 42 %) as yellow waxy solid. $[\alpha]_D^{20}$ -12.4 (*c*, 0.5 in MeOH); υ_{max} (neat) 3330 (br, OH), 1322 (s, S=O), 1131 (s, S=O) cm^{-1}; δ_H (500 MHz, CD_3OD) 3.19-3.24 (2H, m, $NHCH_2$), 3.53-3.56 (2H, m, H-2, H-3), 3.57-3.59 (3H, m, CH_2, H-5), 3.60-3.63 (2H, m, CH_2), 3.64-3.68 (4H, m, 2 x CH_2), 3.68- 3.70 (2H, m, CH_2), 3.83-3.86 (2H, m, H-4, H-5'), 4.33 (1H, t, $J_{1,2}$ 7.6 Hz, H-1); δ_C (125 MHz, CD_3OD) 42.4 (t, CH_2NH), 60.7 (t, CH_2), 66.8 (t, C-5), 68.3 (d, C-4), 69.5, 69.7, 69.9 (3 x t, 3 x CH_2), 70.1 (d, C-2), 72.3 (t, CH_2), 73.5 (d, C-3), 85.3 (d, C-1); HRMS (ESI) calculated for $C_{11}H_{24}N_2NaO_9S$ 383.1100. Found 383.1105 (MNa^+).

3.4.63. *N*-(2-(2-(2-(2-Ethoxy)ethoxy)ethoxy)ethyl)ethanol-*N'*-(β-D-arabinopyranosyl)sulfamide 17c

General Procedure F, using sulfamide **16c,** and purification by RP-HPLC (Luna C-18 column (Phenomenex); eluent: A (0.05 % TFA in H_2O) and B MeCN; gradient: the sample was run at 1 mL/min with a gradient of 10-35 % B; column oven: 15 °C; detection: CAD), afforded de-protected

sulfamide **17c** (7 mg, 46 %) as yellow waxy solid. $[\alpha]_D^{20}$ -10.6 (*c*, 0.5 in MeOH); υ_{max} (neat) 3324 (br, OH), 1326 (s, S=O), 1131 (s, S=O) cm^{-1}; δ_H (400 MHz, CD_3OD) 3.12-3.24 (2H, m, NHC$\underline{H}_2$), 3.55-3.68 (17H, m, 7 x CH_2, H-2, H-3, H-5), 3.83-3.86 (2H, m, H-4, H-5'), 4.32 (1H, d, $J_{1,2}$ 7.0 Hz, H-1); δ_C (100.5 MHz, CD_3OD) 42.4 (t, CH_2NH), 60.8 (t, CH_2), 66.8 (t, C-5), 68.3 (d, C-4), 69.5, 69.6, 69.7, 69.9 (4 x t, 4 x CH_2), 70.0 (d, C-2), 72.0, 72.1 (2 x t, 2 x CH_2), 73.5 (d, C-3), 85.2 (d, C-1); HRMS (ESI) calculated for $C_{13}H_{28}N_2NaO_{10}S$ 427.1362. Found 427.1365 (MNa^+).

Acknowledgements

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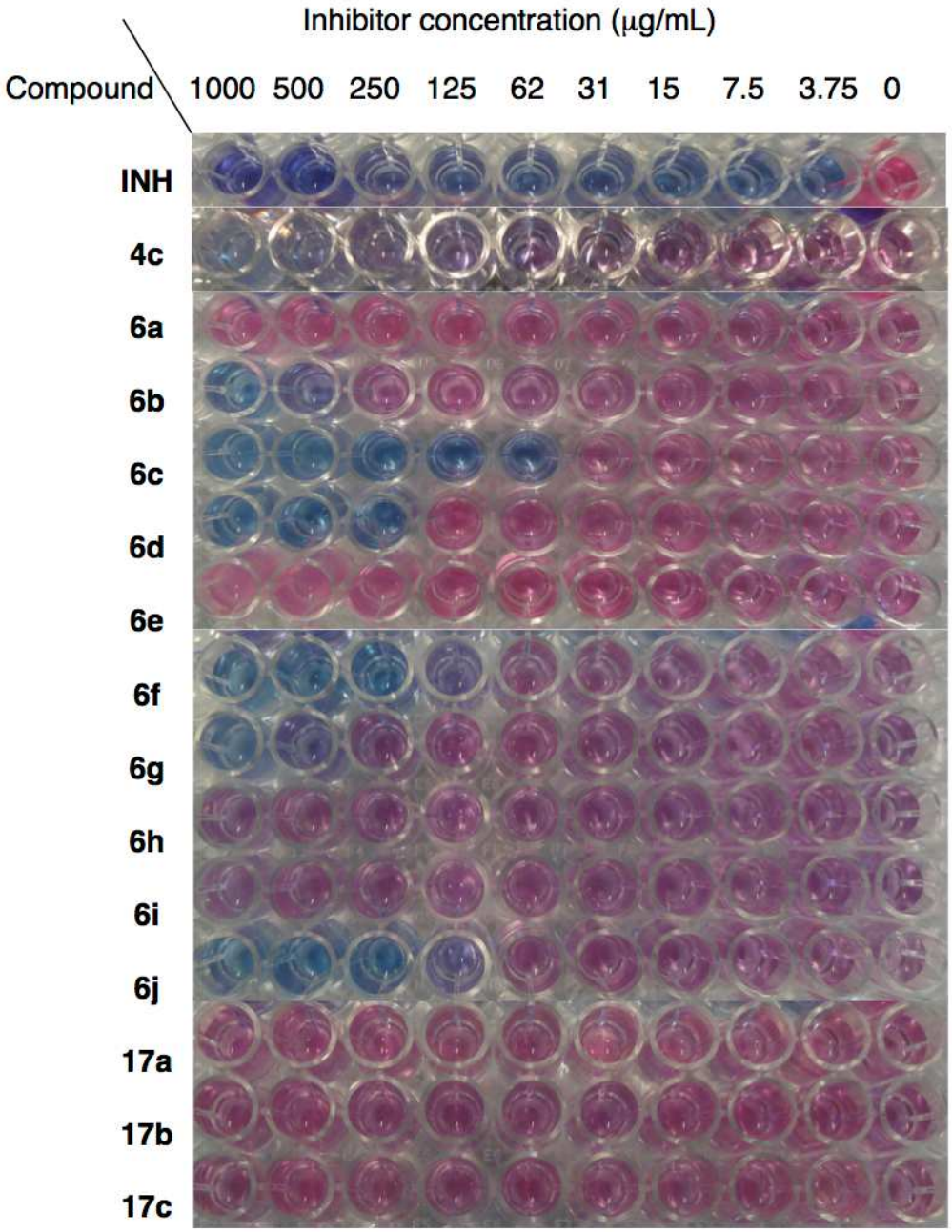
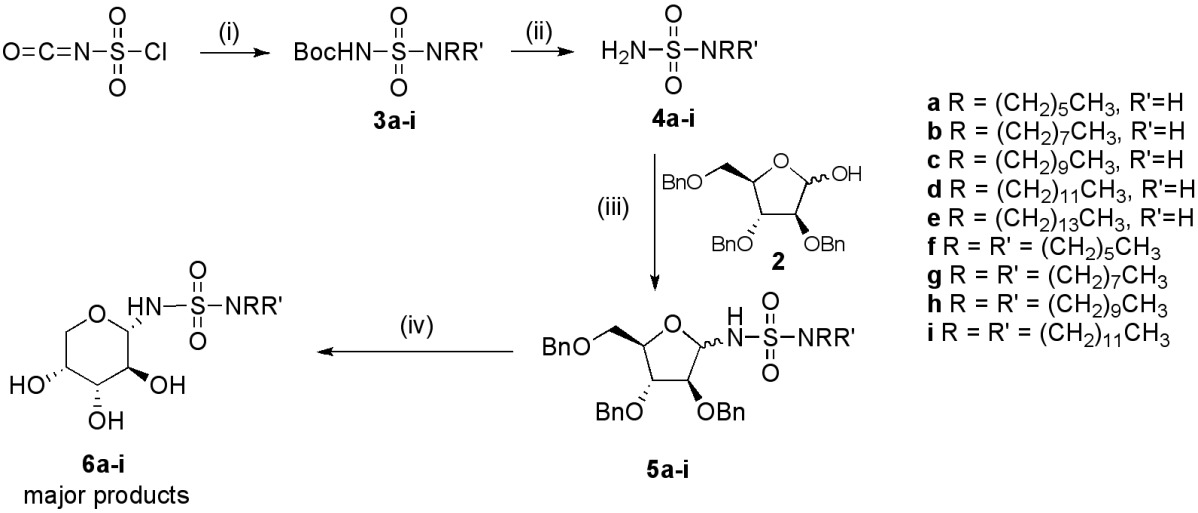
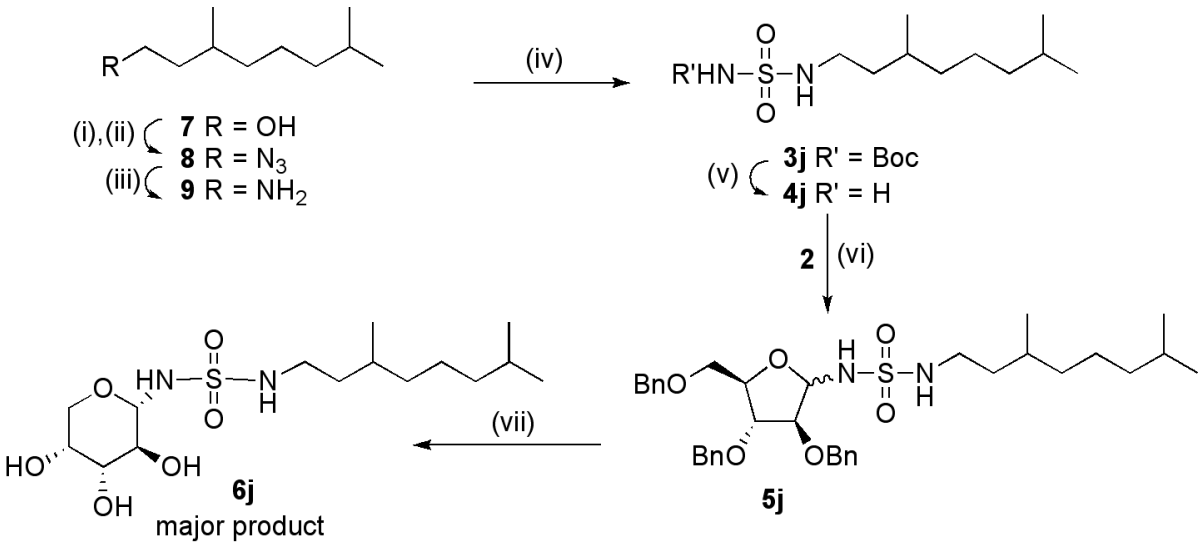
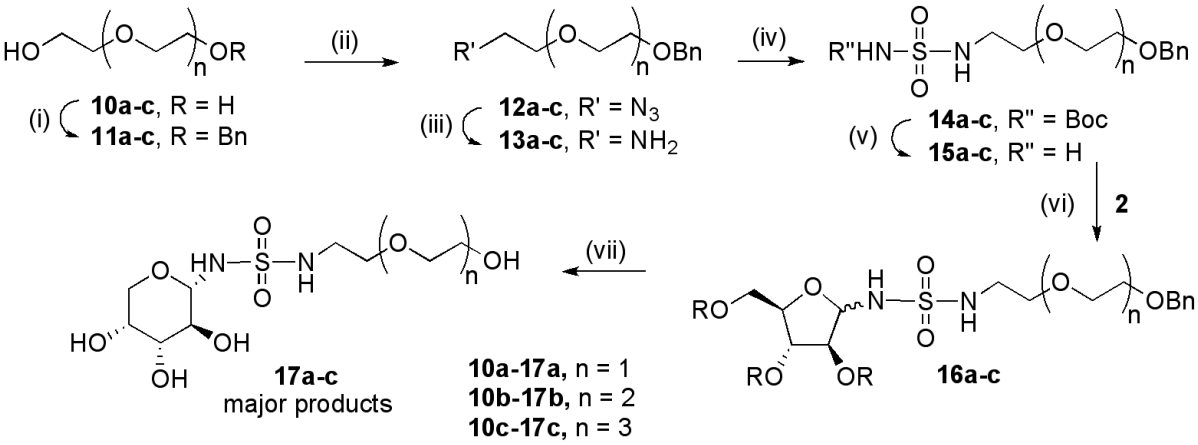

Inhibitor concentration (μg/mL)
Compound
1000 500 250 125 62 31 15 7.5 3.75 0
INH
4c
6a
6b
6c
6d
6e
6f
6g
6h
6i
6j
17a
17b
17c



a R = $(CH_2)_5CH_3$, R'=H
b R = $(CH_2)_7CH_3$, R'=H
c R = $(CH_2)_9CH_3$, R'=H
d R = $(CH_2)_{11}CH_3$, R'=H
e R = $(CH_2)_{13}CH_3$, R'=H
f R = R' = $(CH_2)_5CH_3$
g R = R' = $(CH_2)_7CH_3$
h R = R' = $(CH_2)_9CH_3$
i R = R' = $(CH_2)_{11}CH_3$





- synthesis of a variety of glycosyl sulfamides of arabinose
- assays of anti-mycobacterial activity using *M. smegmatis*
- optimal activity (MIC 62 μg/mL) observed for a sulfamide with a C10 alkyl chain

SUPPORTING INFORMATION

Synthesis of arabinose glycosyl sulfamides as potential inhibitors of mycobacterial cell wall biosynthesis

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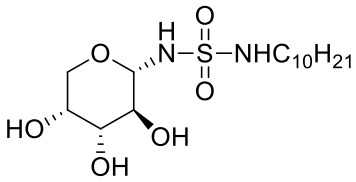
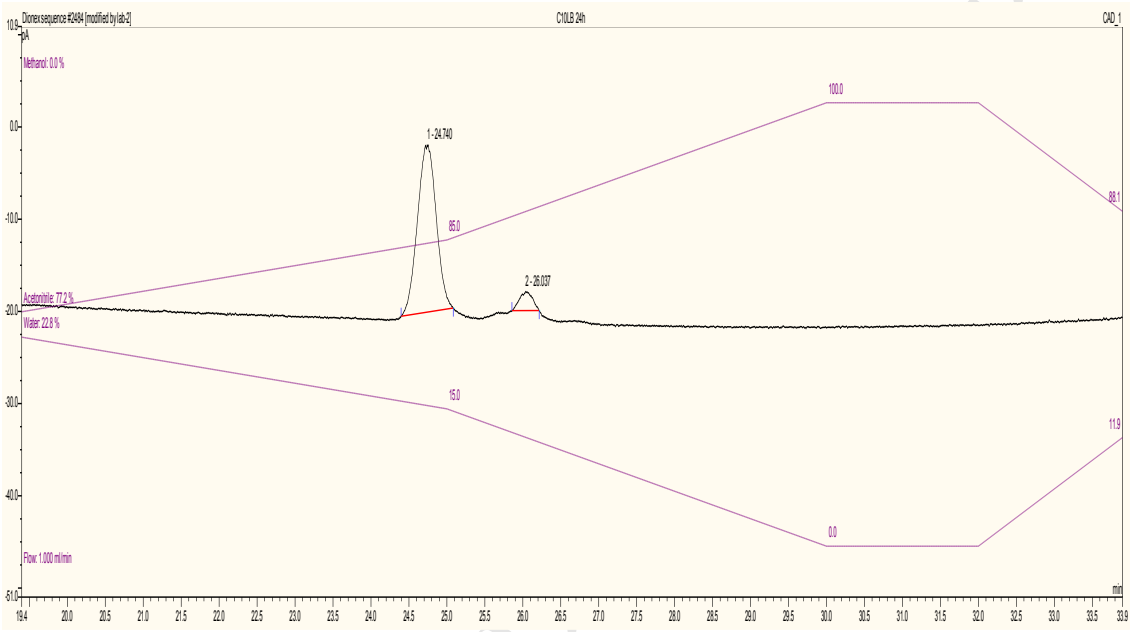
c) School of Chemistry, Monash University, Box 23, Victoria 3800 Australia

Mutarotation and pyranose / furanose equilibration in LB medium after 24 h

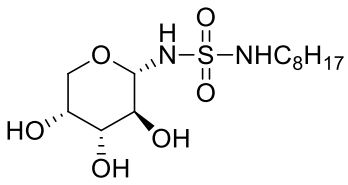
Compound 6c

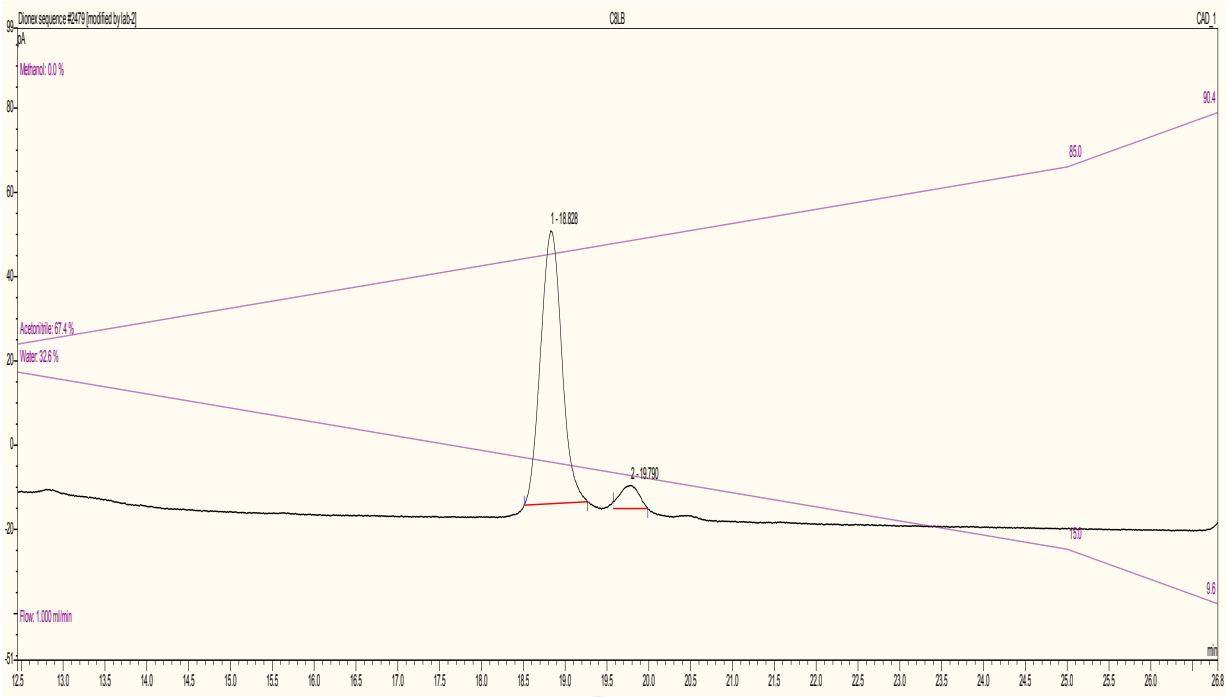




Peak	Ret.Time / min	Height pA	Area pA*min	Rel.Area / %	Identity
1	24.74	18.136	5.215	92.75	α-pyranose
2	26.04	2.062	0.408	7.25	β-pyranose and α-furanose
Total:		20.198	5.622	100.00	

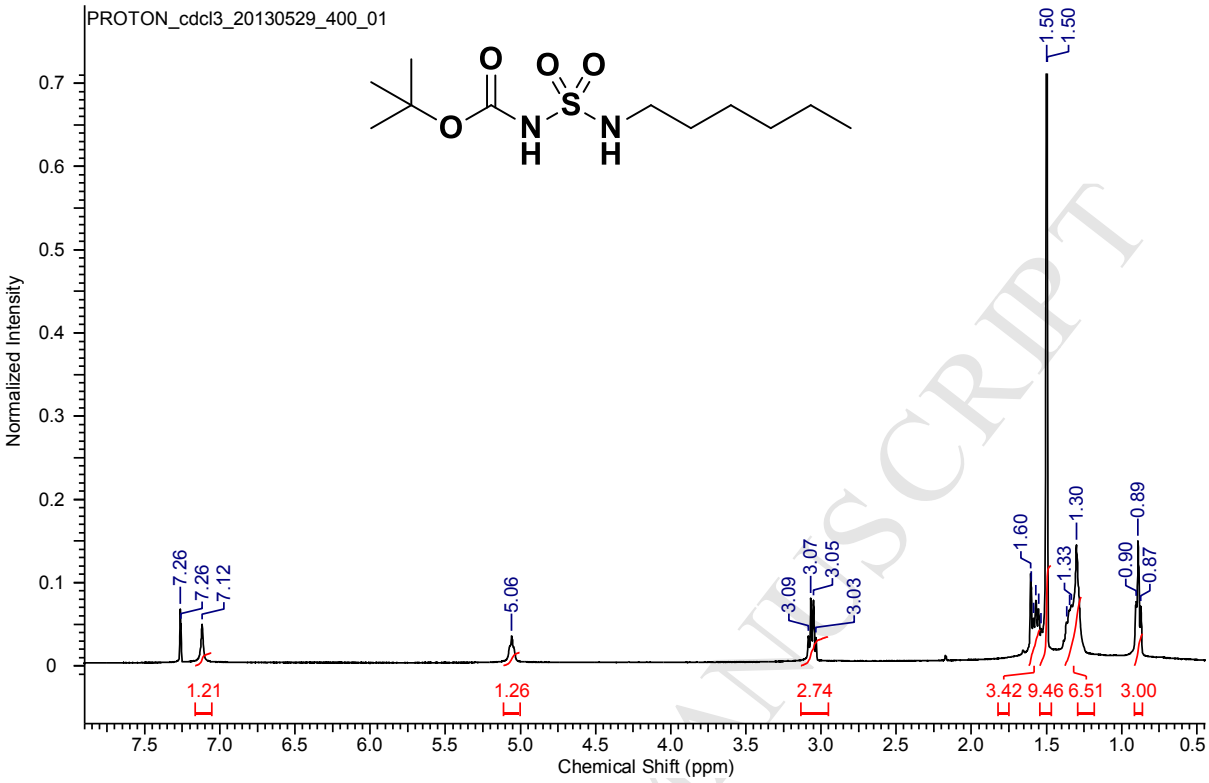
Compound 6b

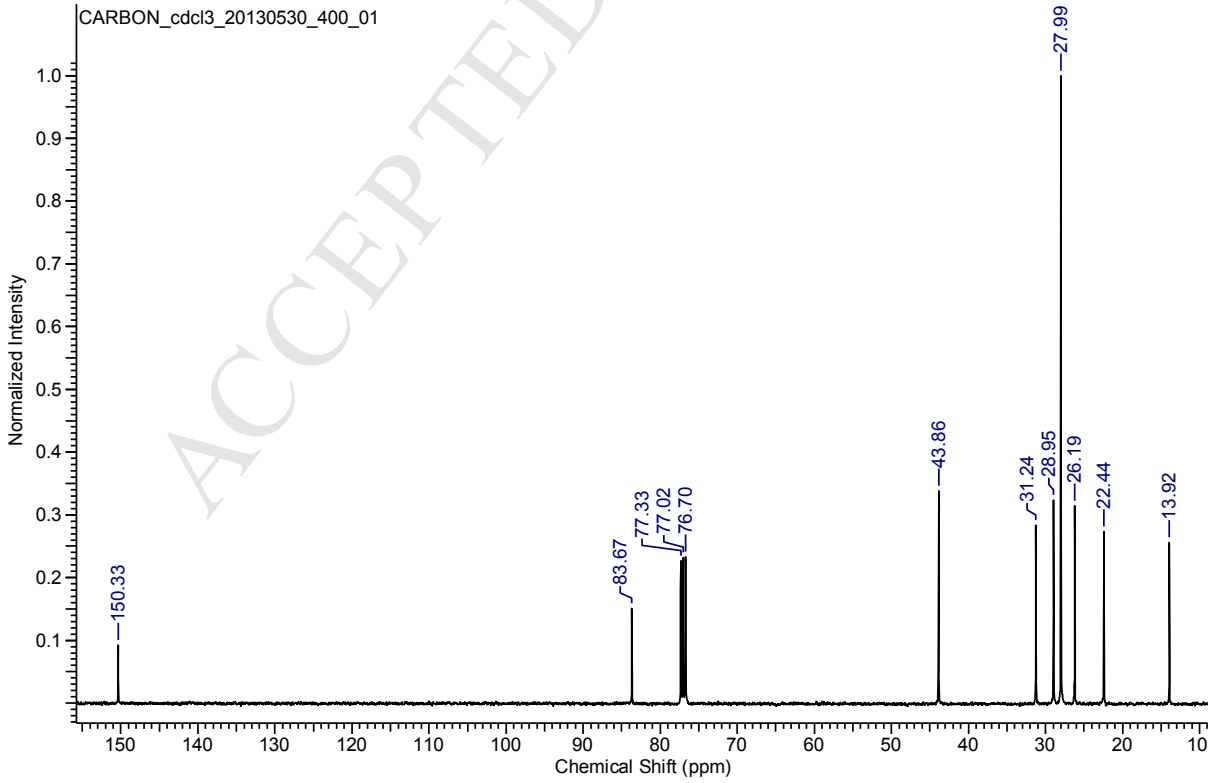




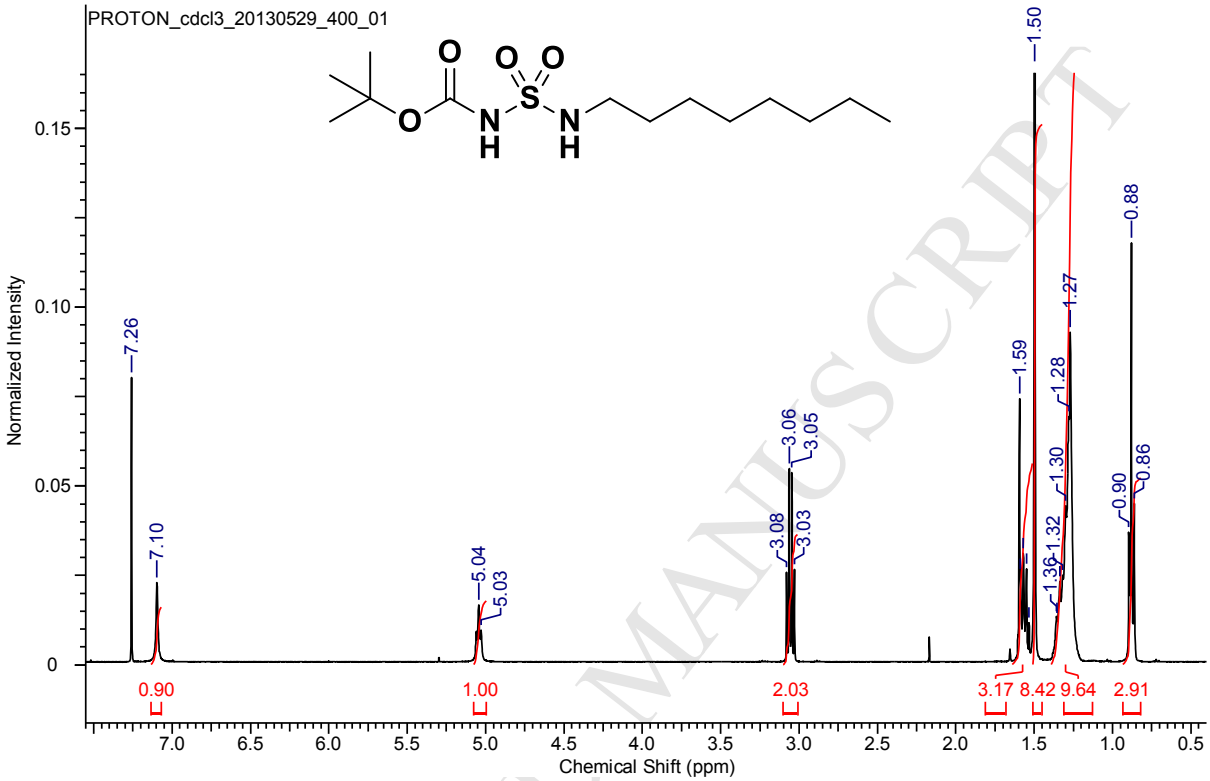
Peak	Ret.Time / min	Height pA	Area pA*min	Rel.Area %	Identity
1	18.83	64.732	19.020	93.00	α-pyranose
2	19.79	5.509	1.432	7.00	β-pyranose and α-furanose
Total:		70.240	20.452	100.00	

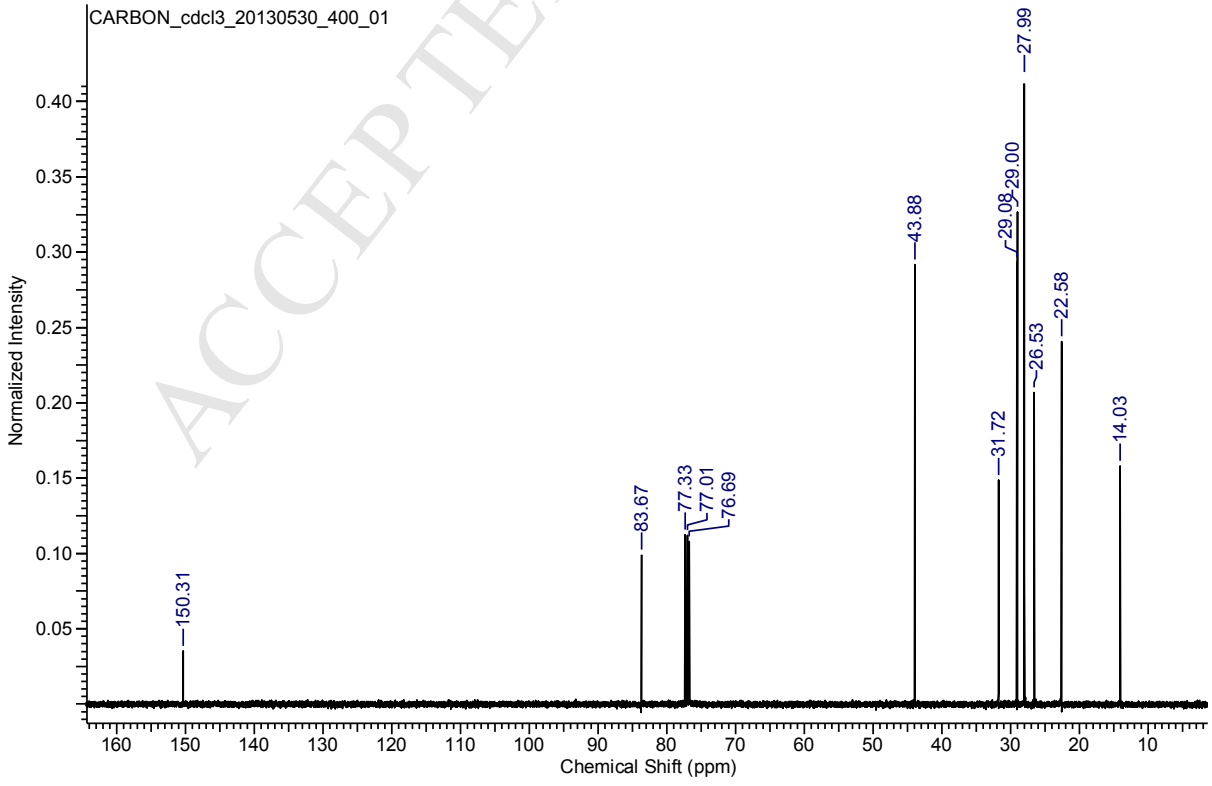
Tert-butyl _N_-hexylsulfamoylcarbamate 3a



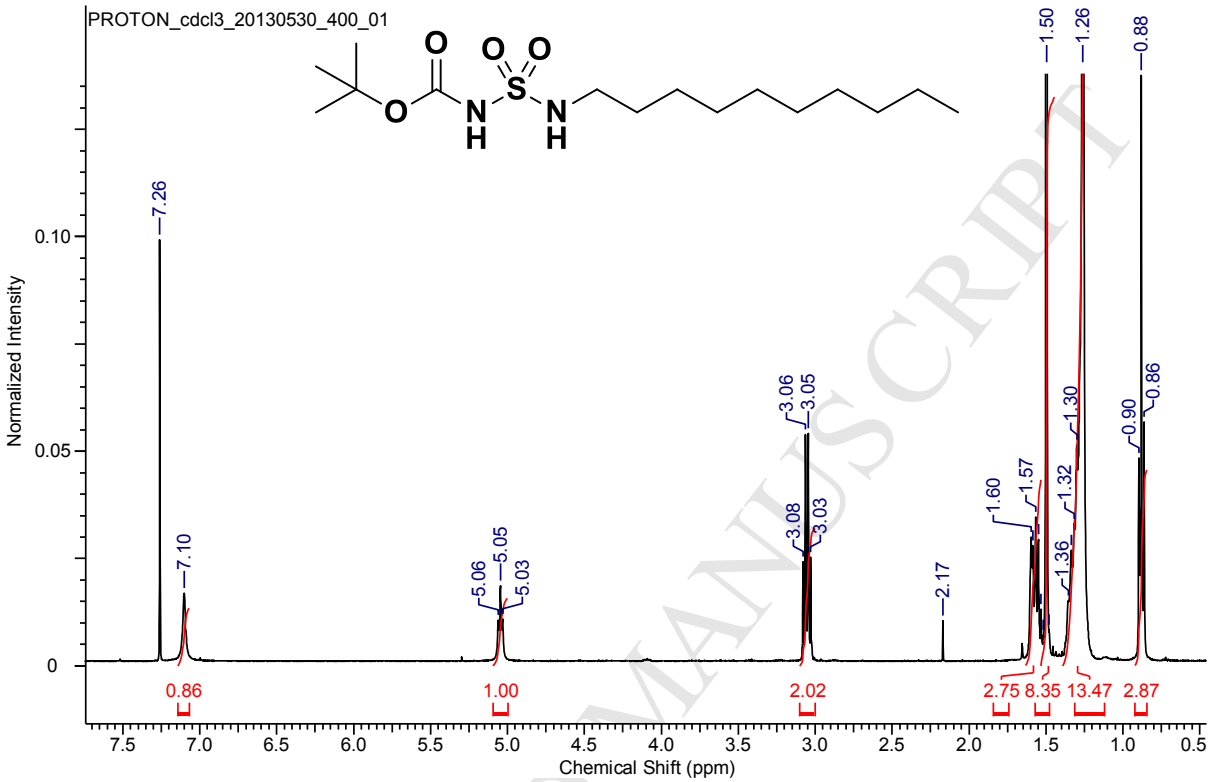


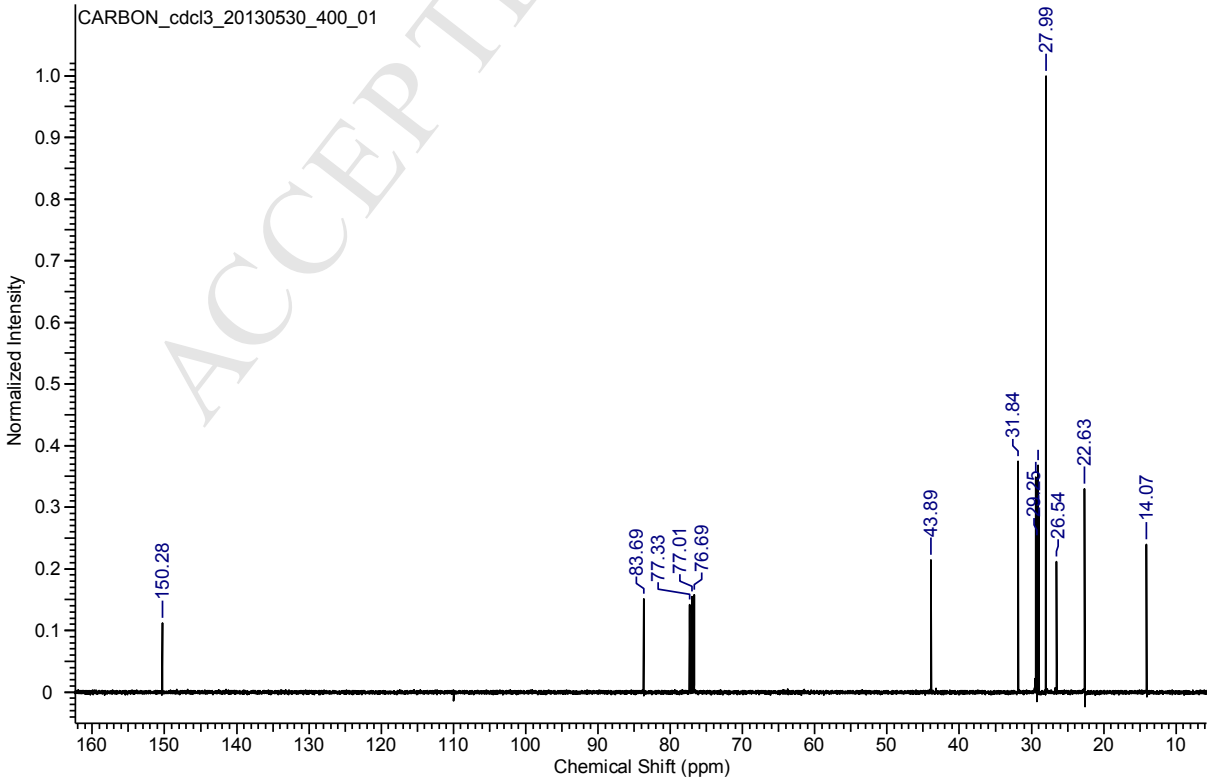
Tert-butyl _N_-octylsulfamoylcarbamate 3b



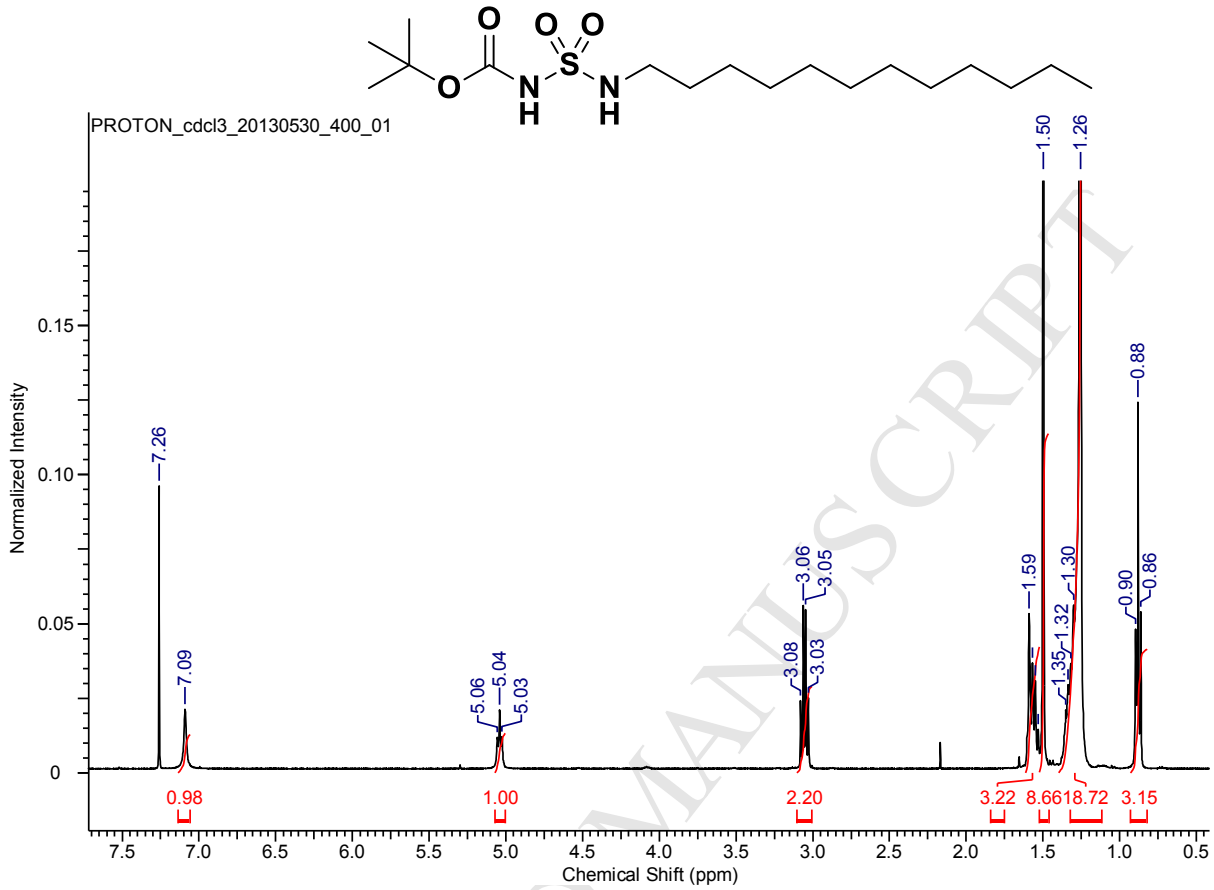


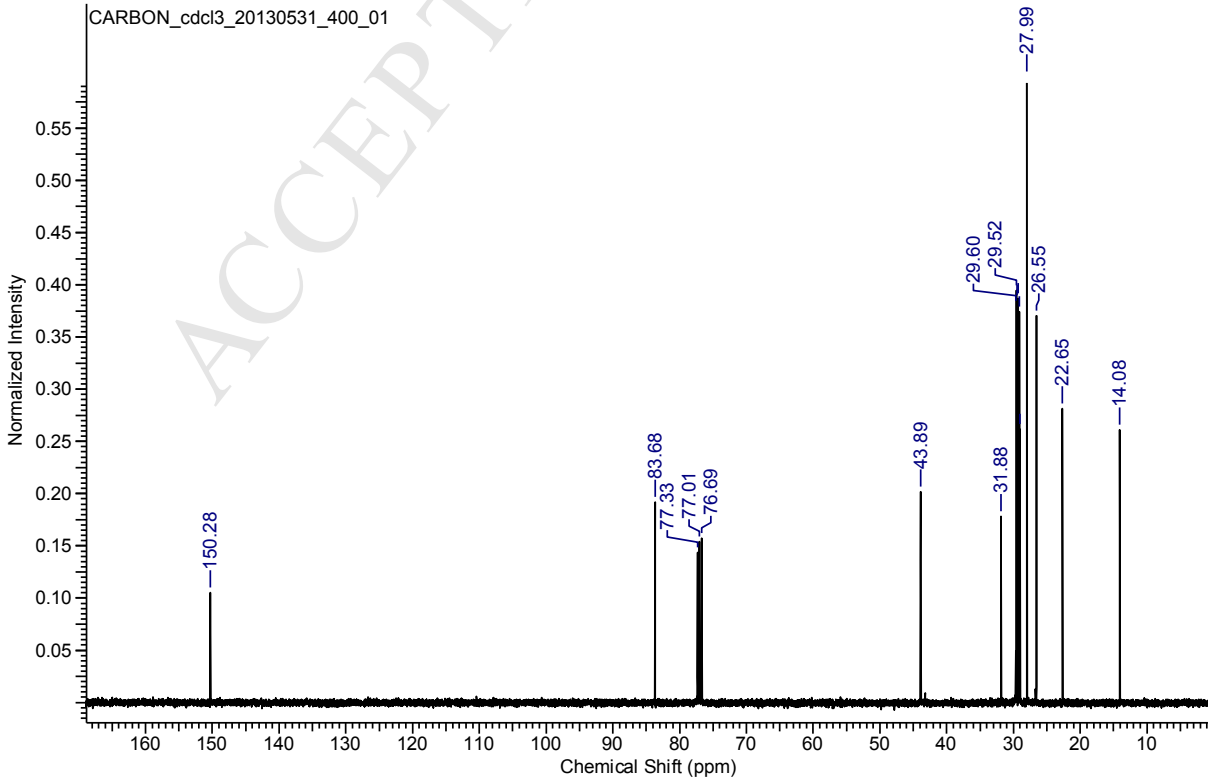
Tert-butyl _N_-decylsulfamoylcarbamate 3c



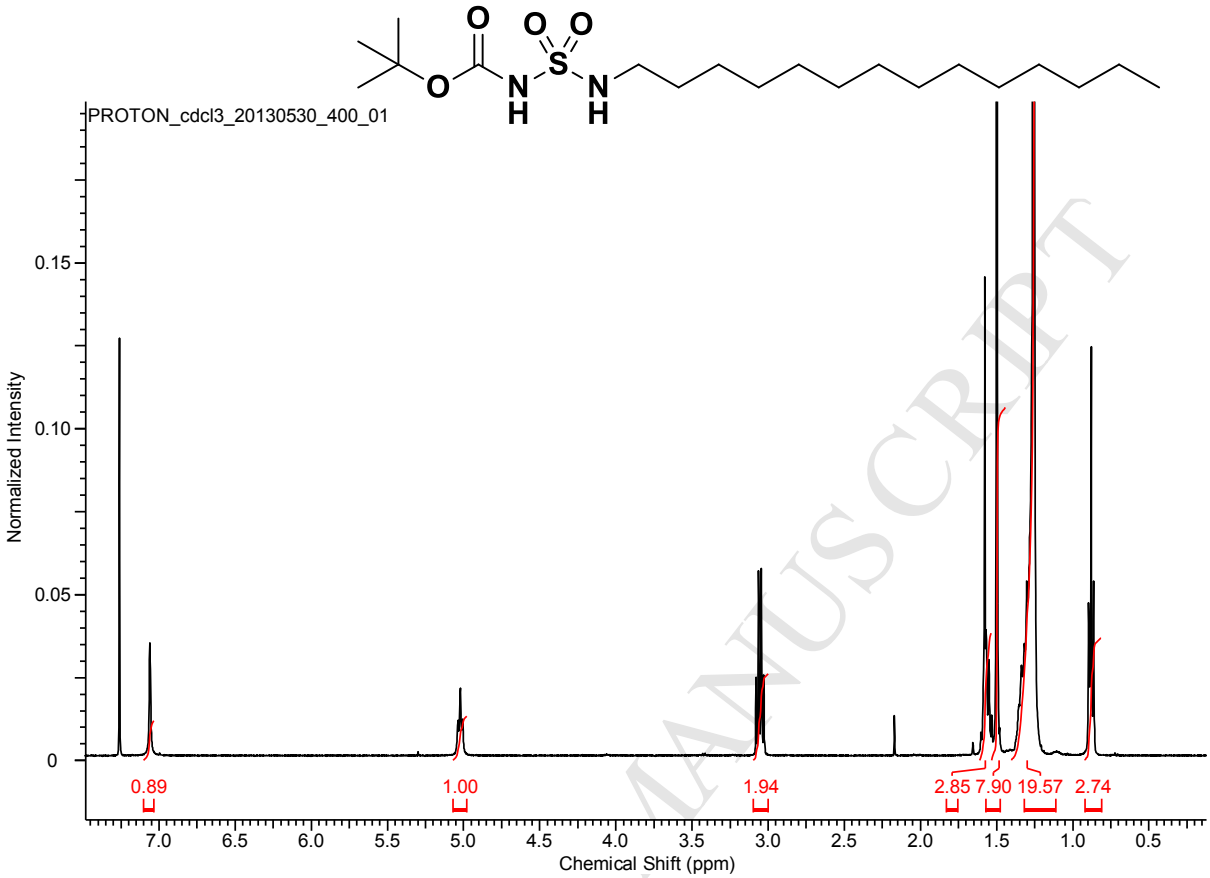


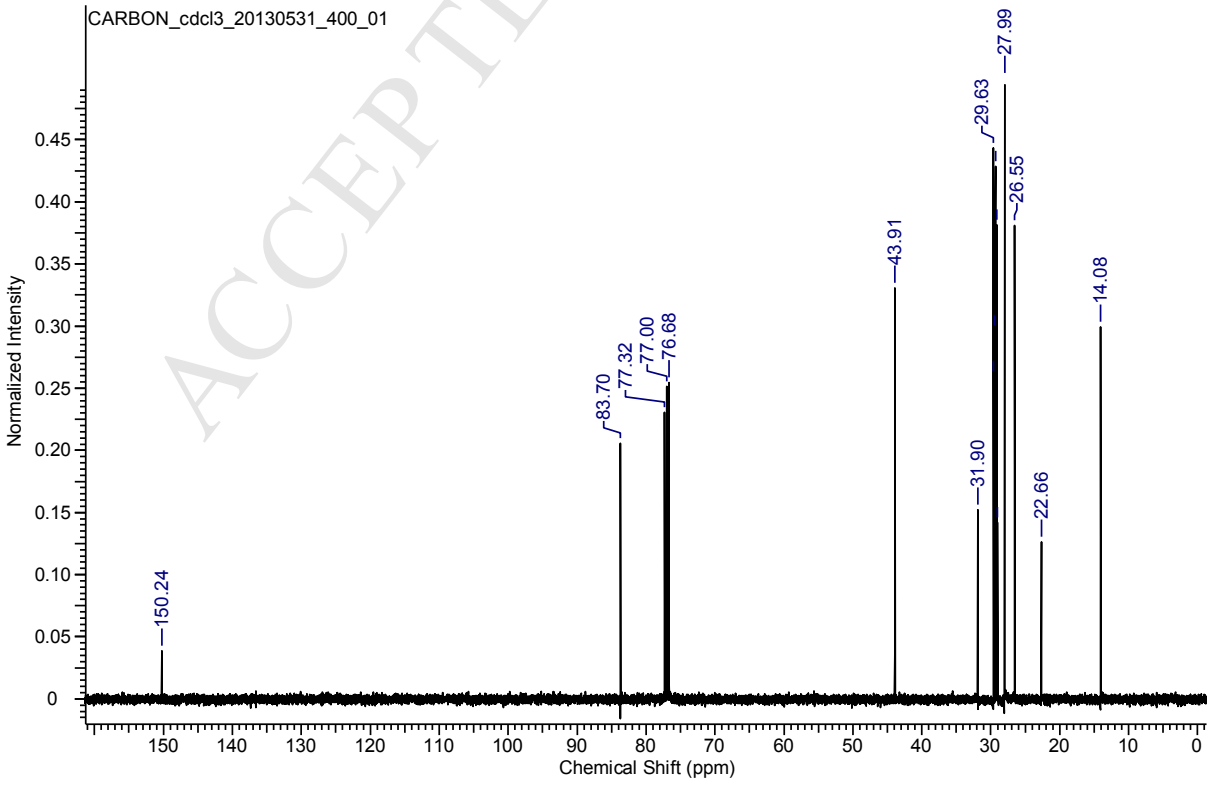
Tert-butyl _N_-dodecylsulfamoylcarbamate 3d



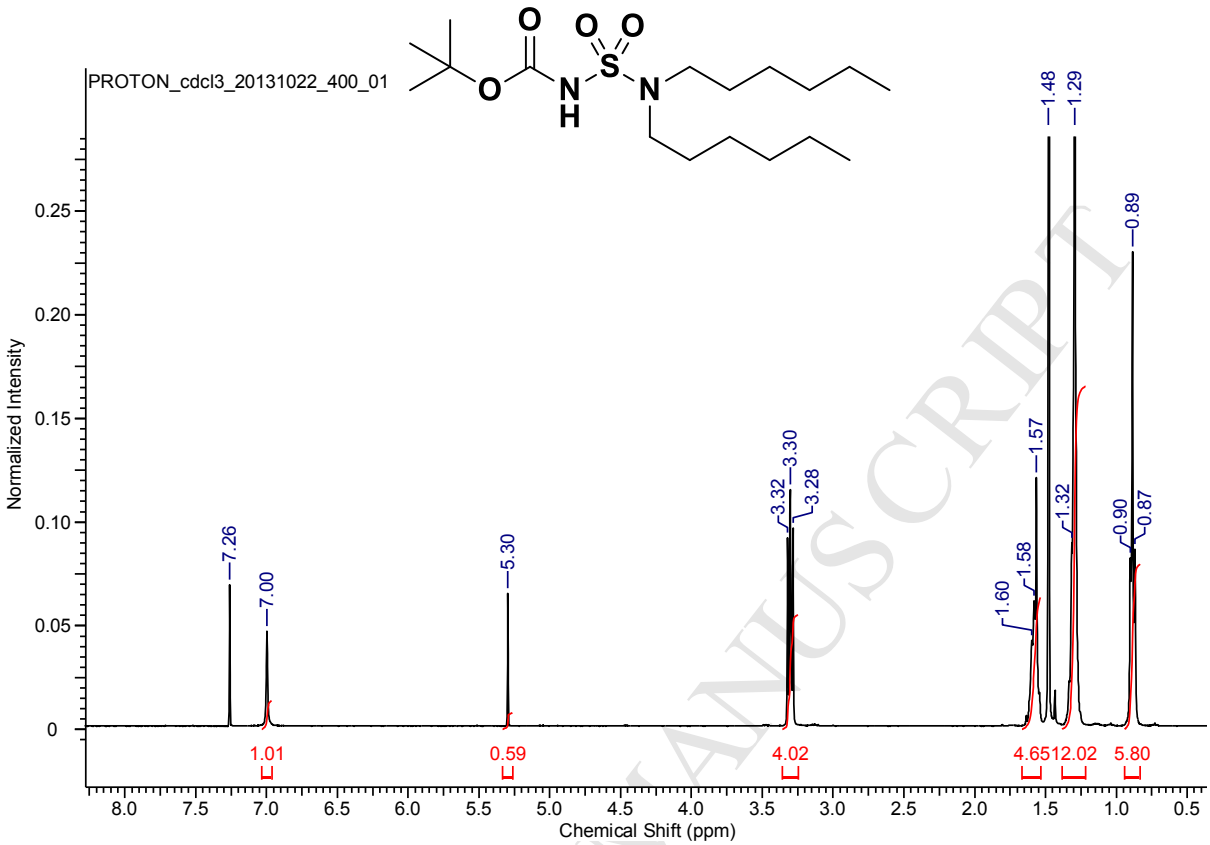


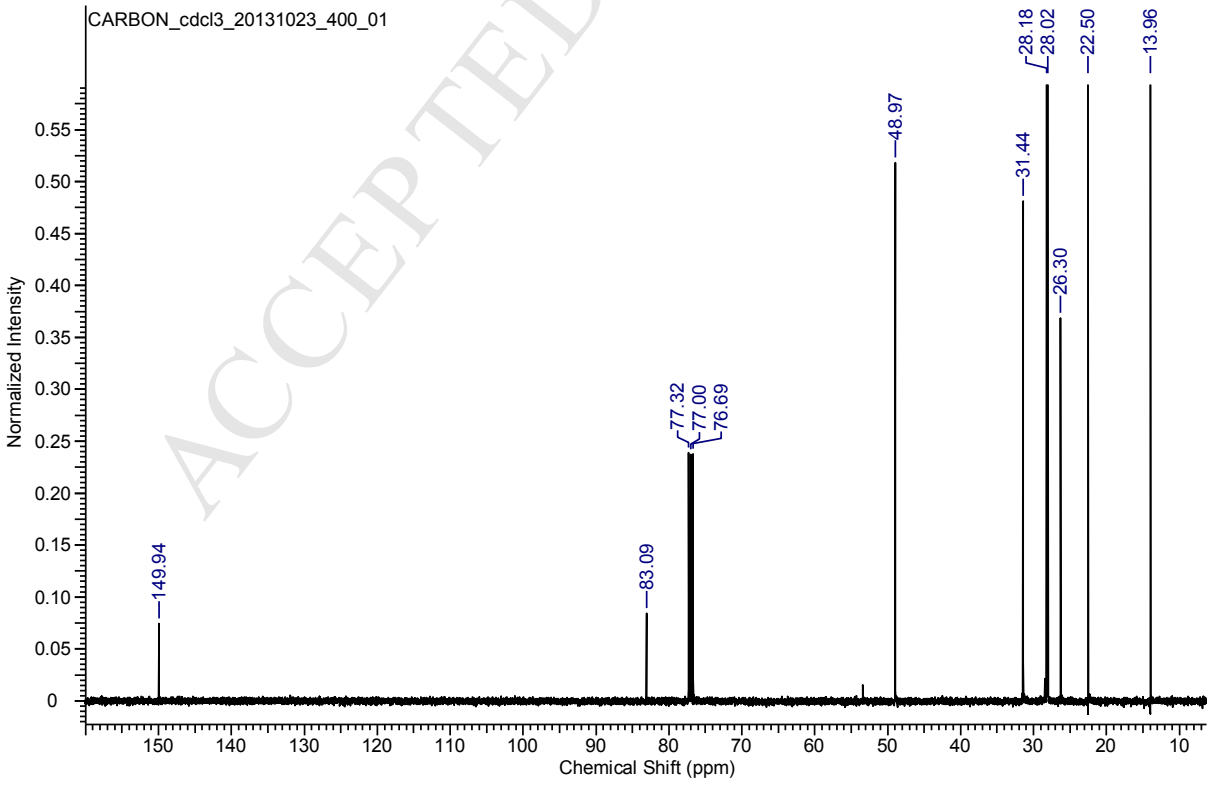
Tert-butyl _N_-tetradecylsulfamoylcarbamate 3e



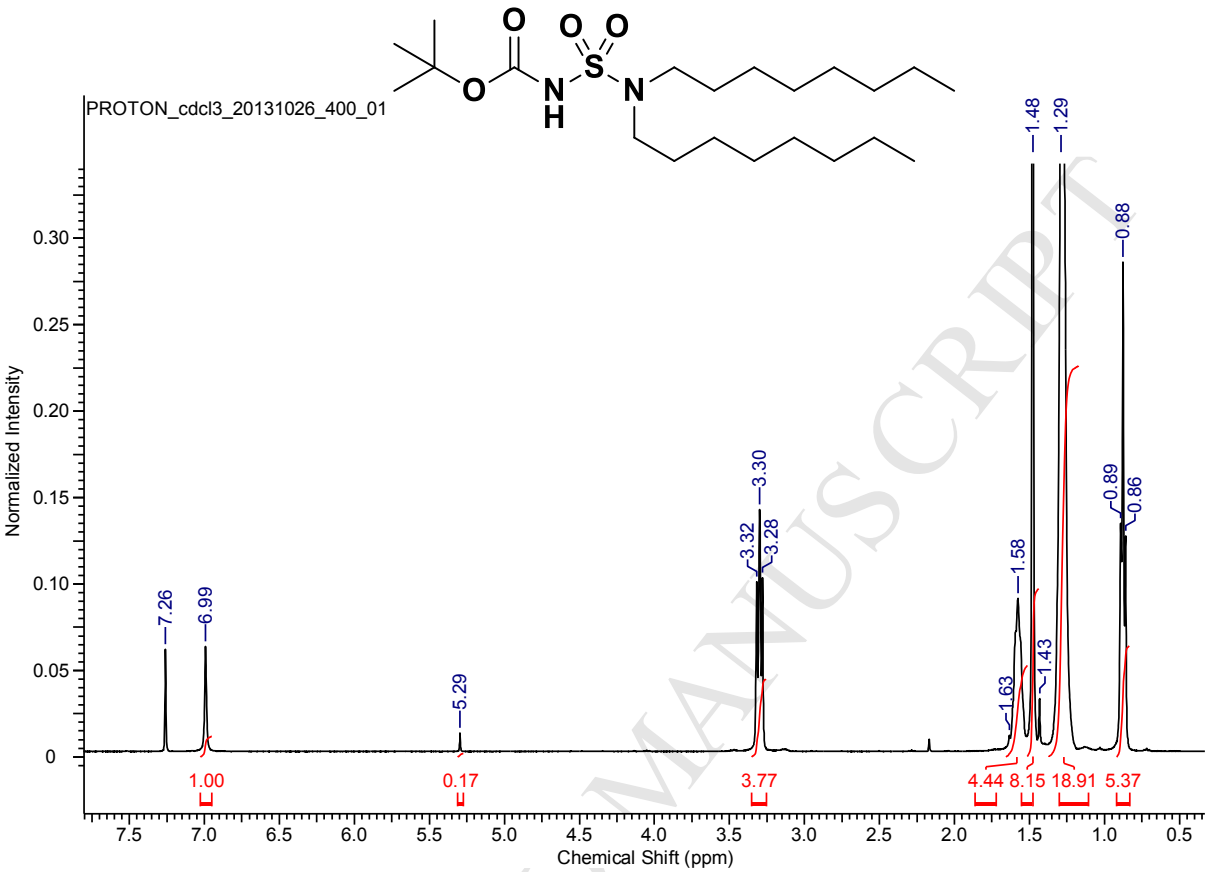


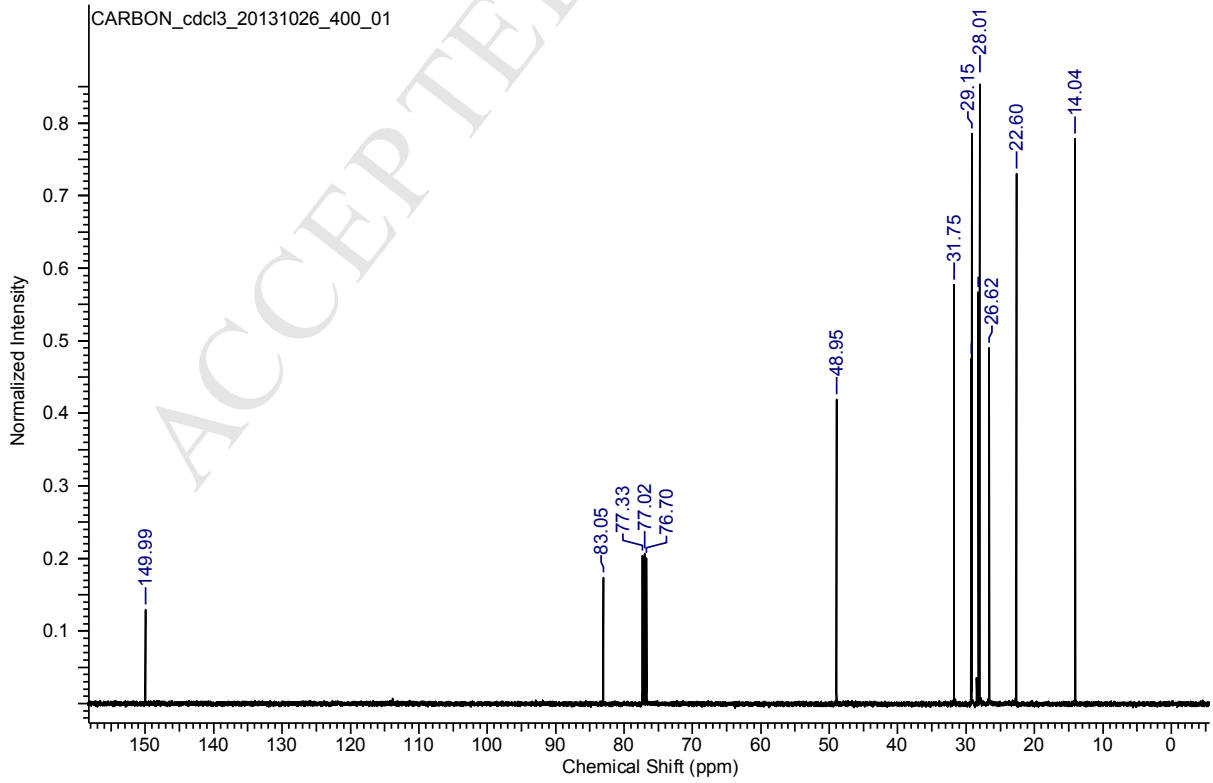
Tert-butyl _N,N_-dihexylsulfamoylcarbamate 3f



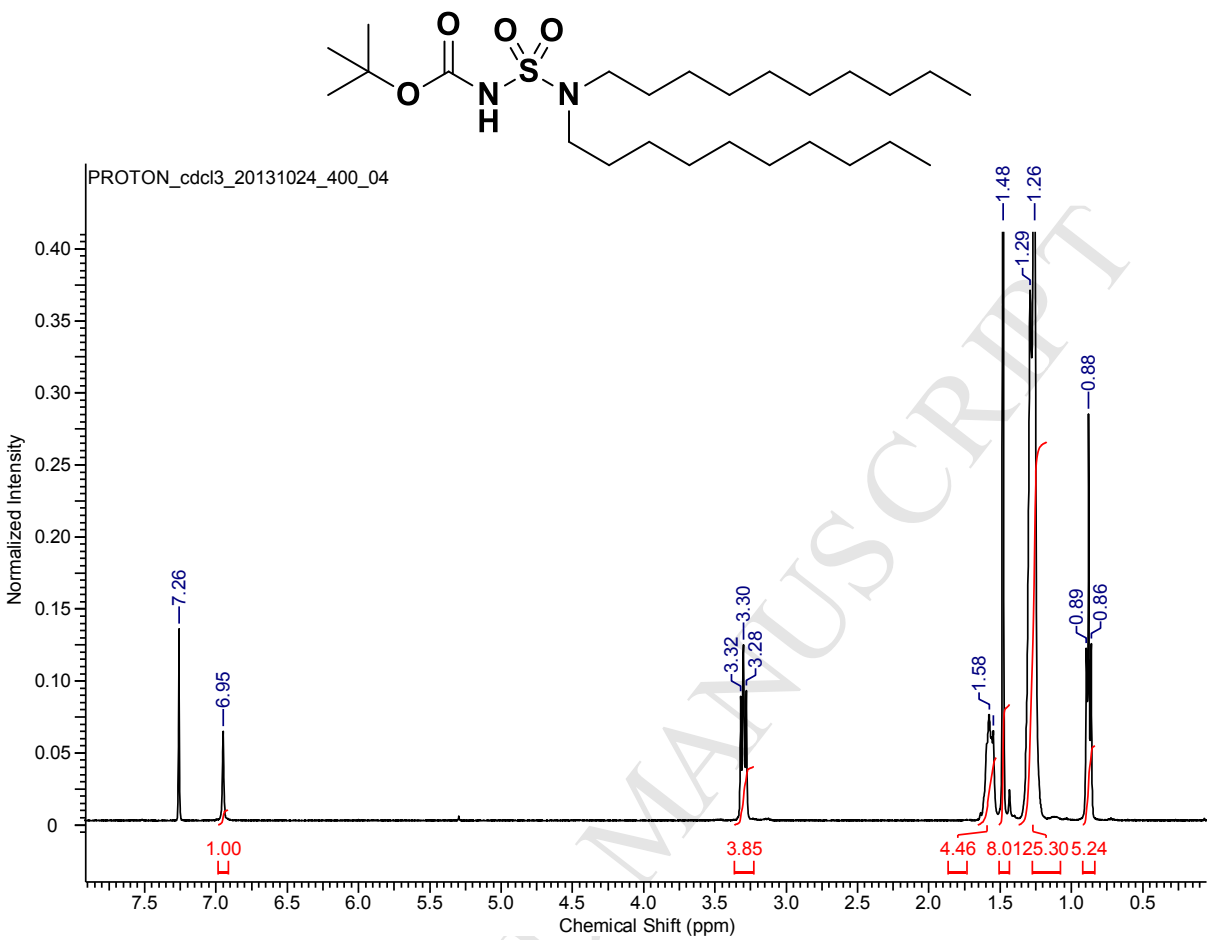


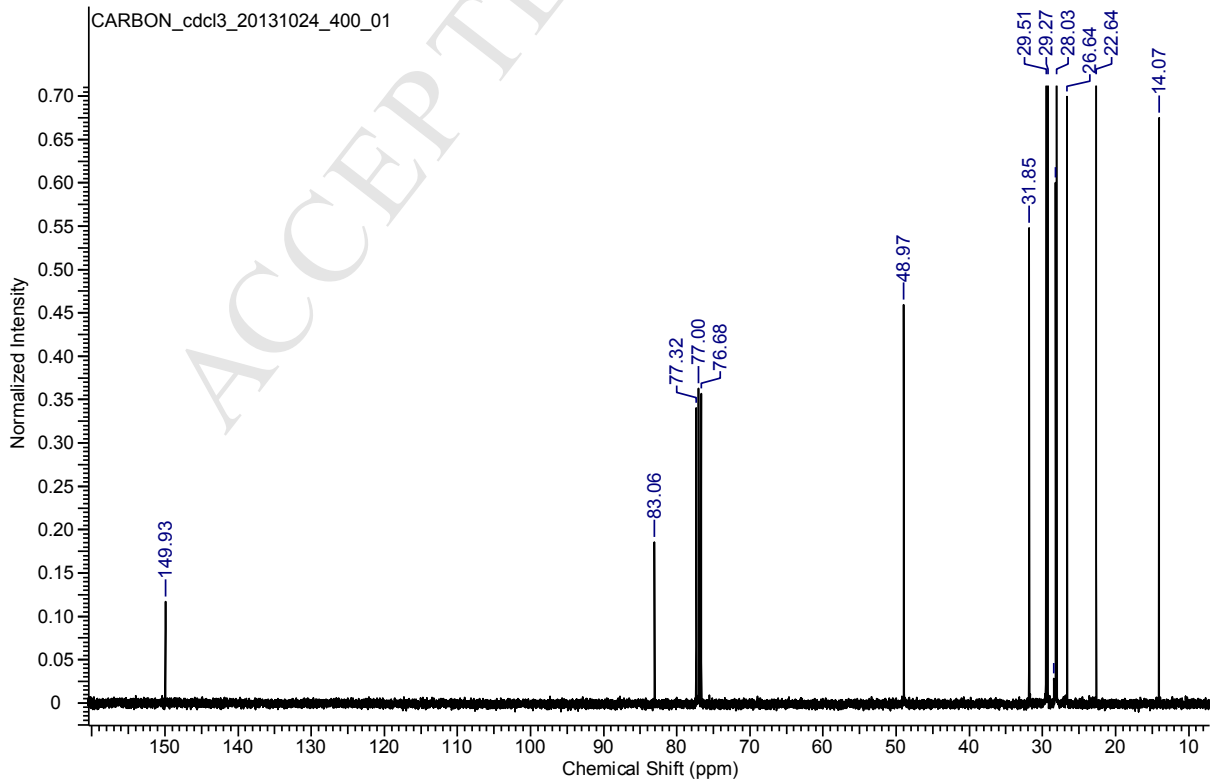
Tert-butyl _N,N_-dioctylsulfamoylcarbamate 3g



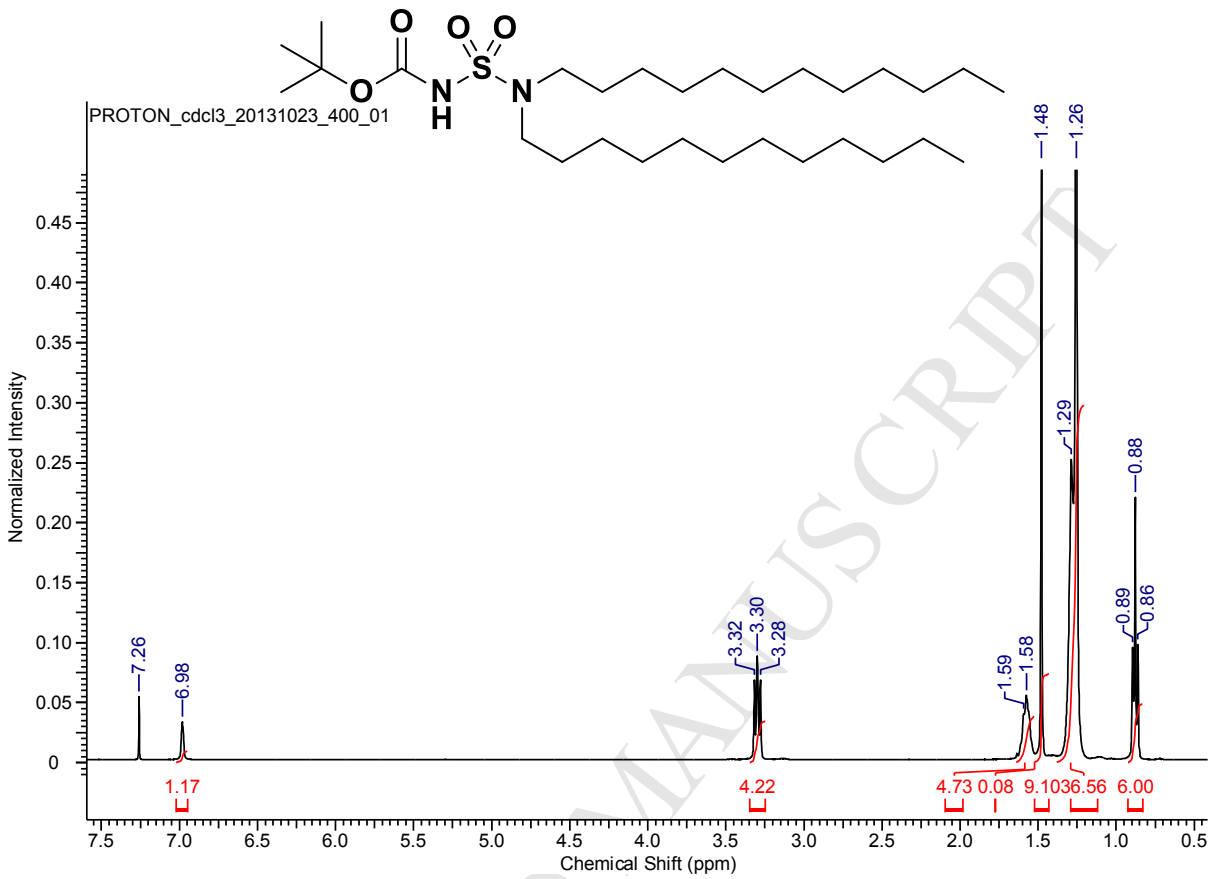


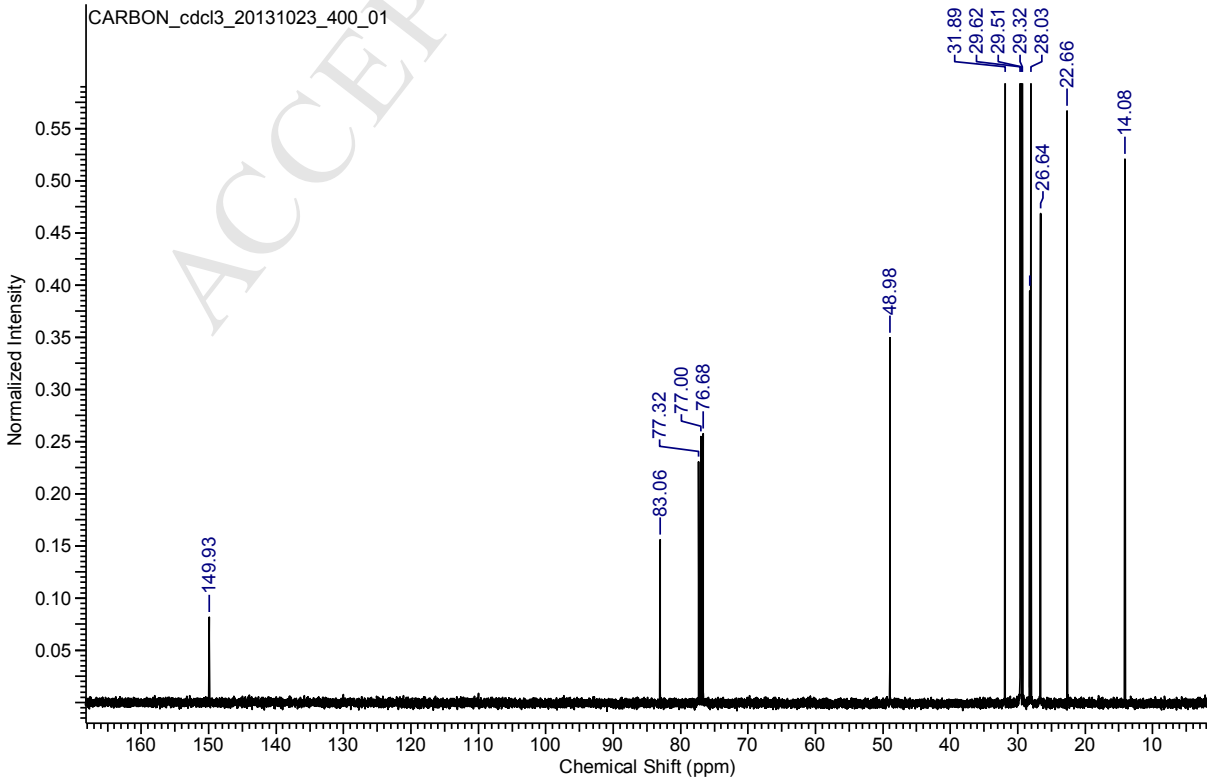
Tert-butyl _N,N_-didecylsulfamoylcarbamate 3h



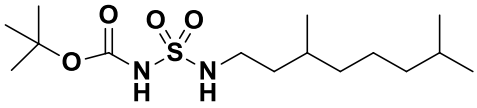


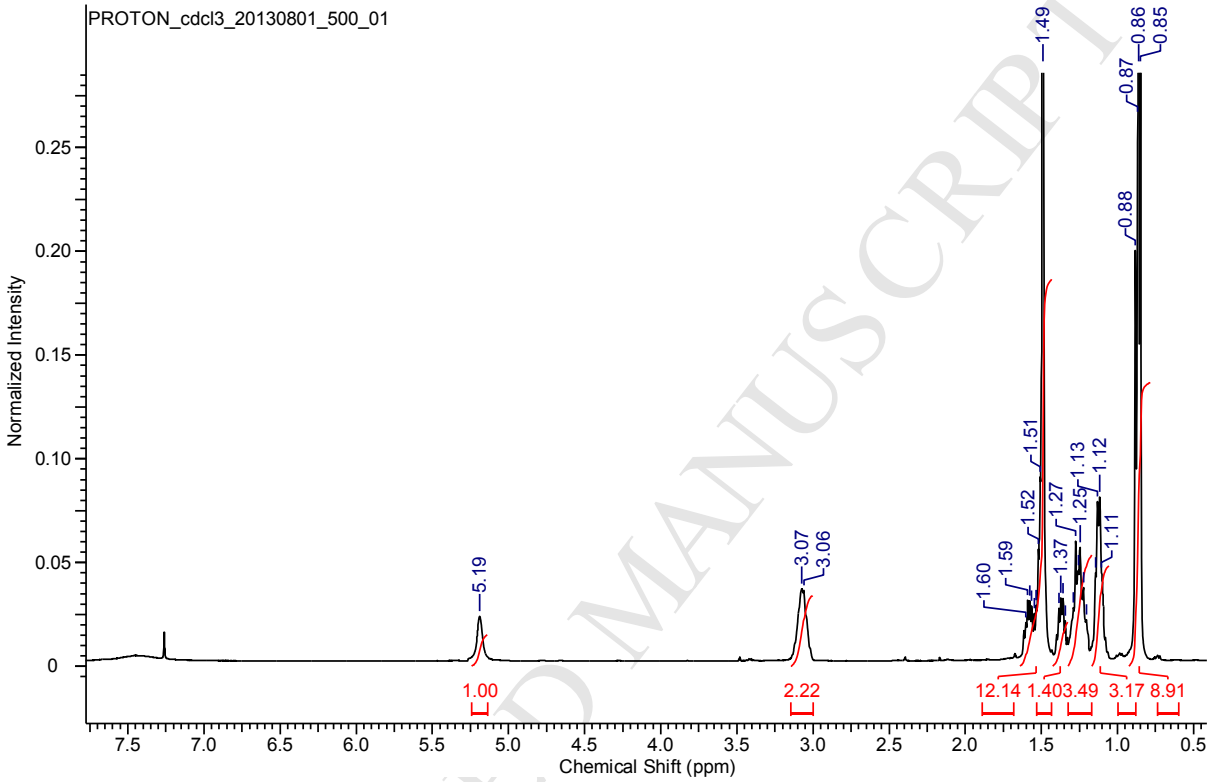
Tert-butyl _N,N_-didodecylsulfamoylcarbamate 3i

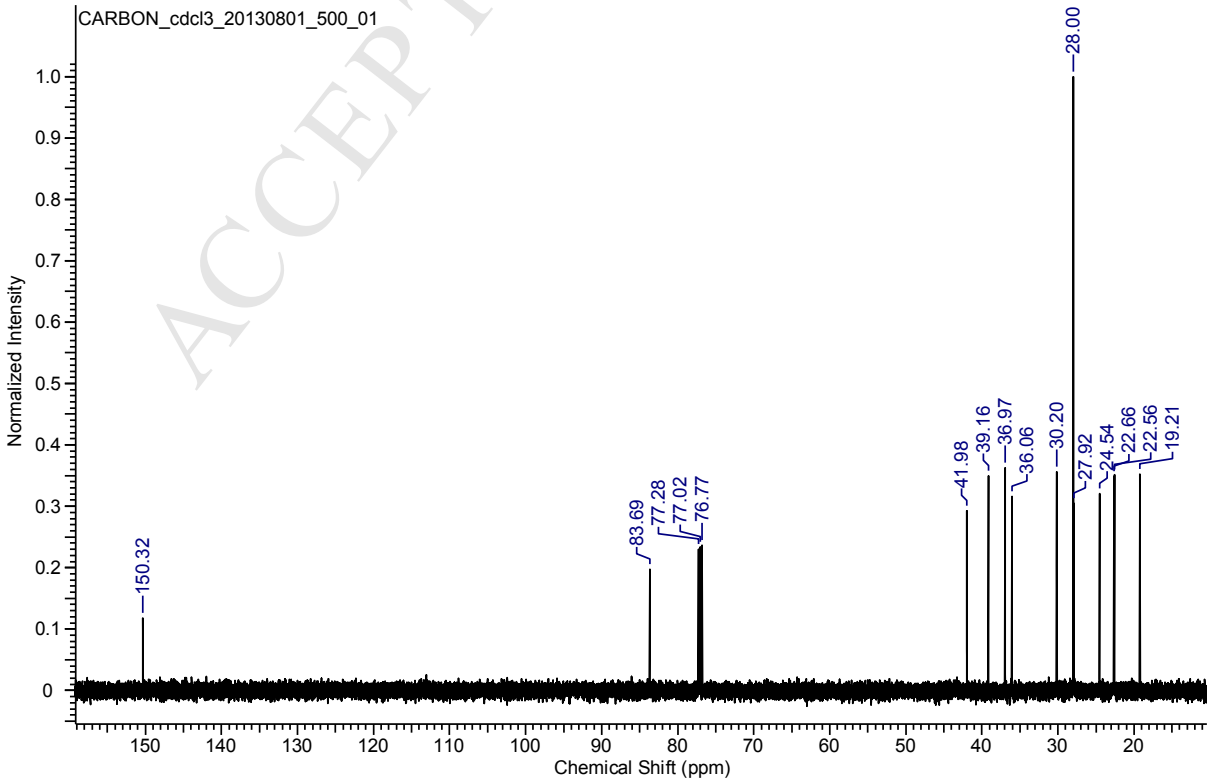




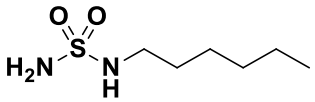
Tert-butyl _N_-3,7-dimethyloctylsulfamoylcarbamate 3j

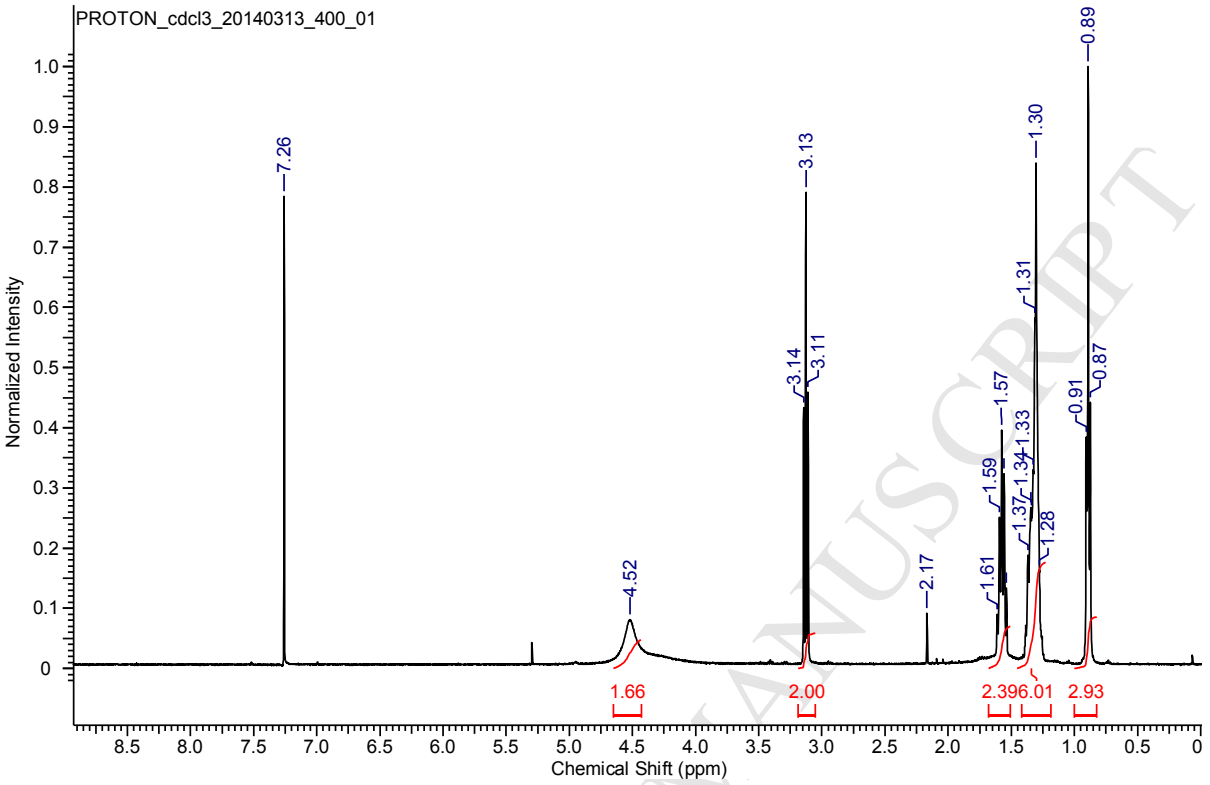


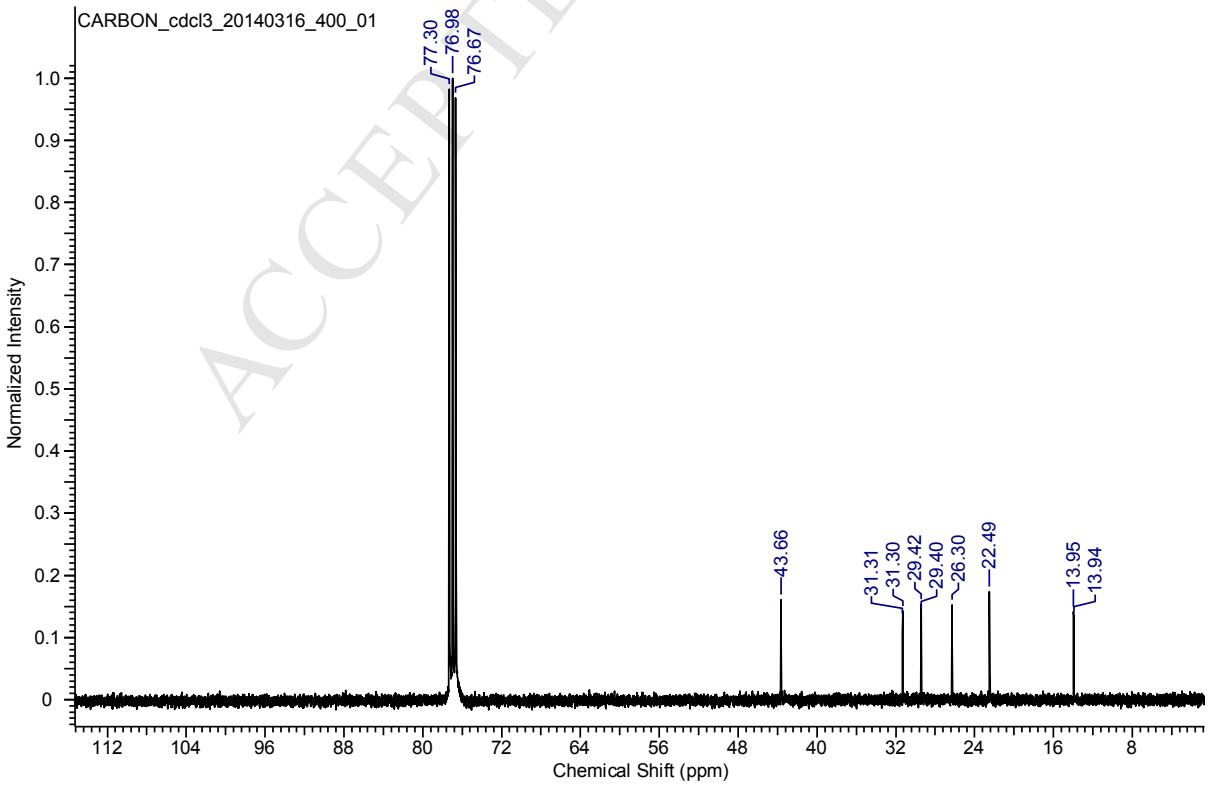




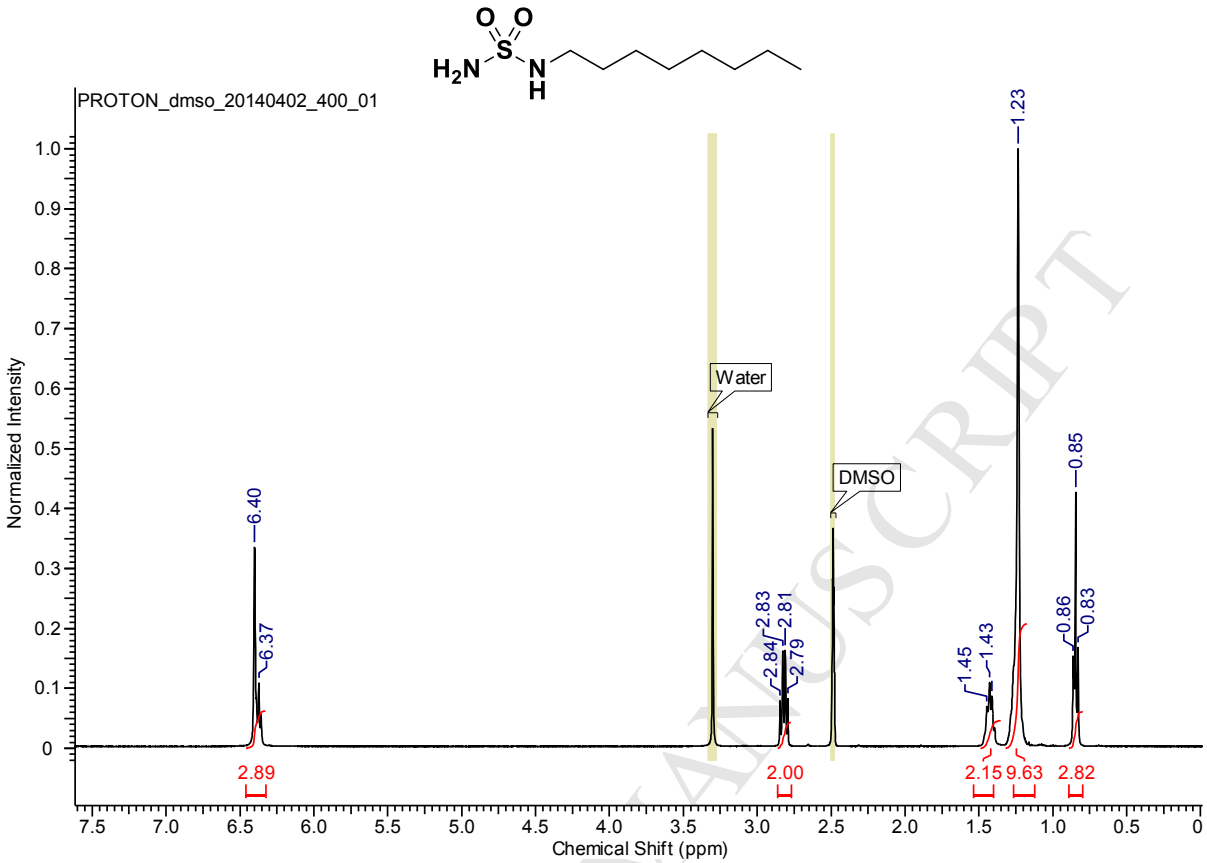
_N_-(Hexyl)sulfamide 4a

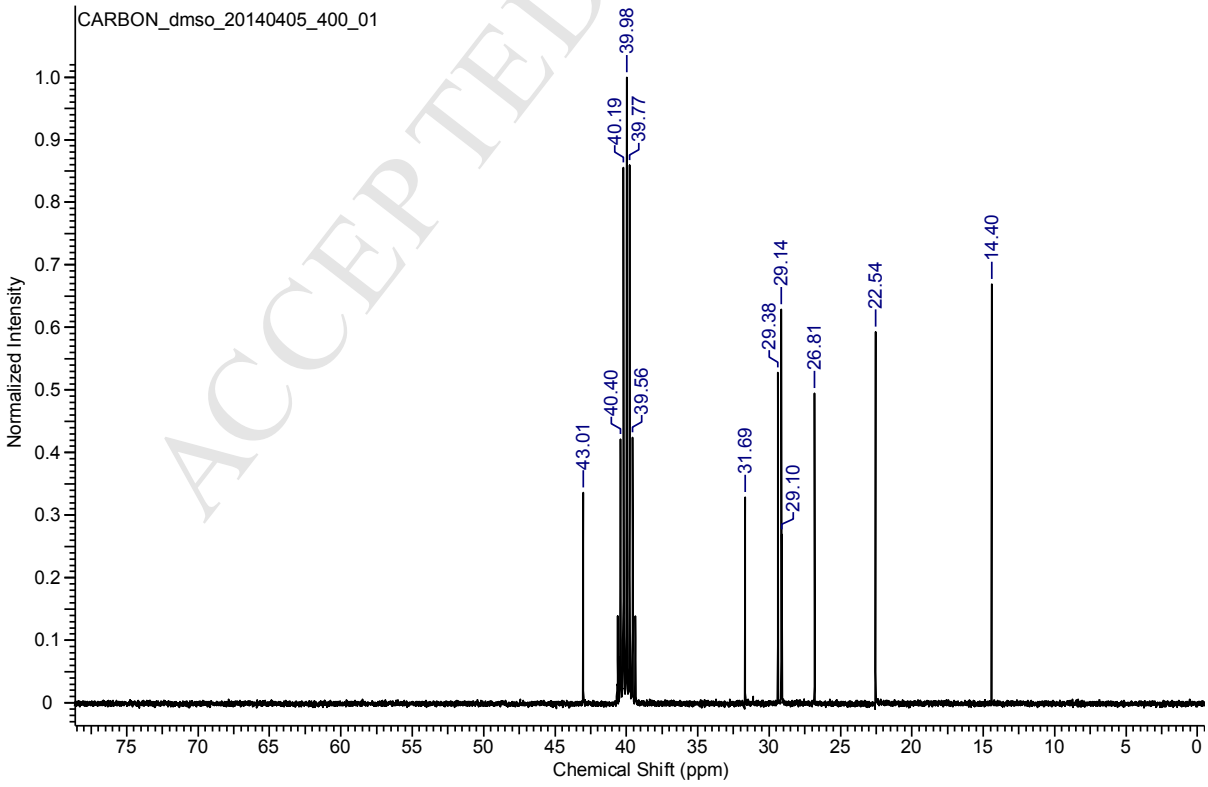




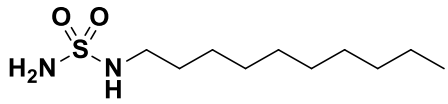


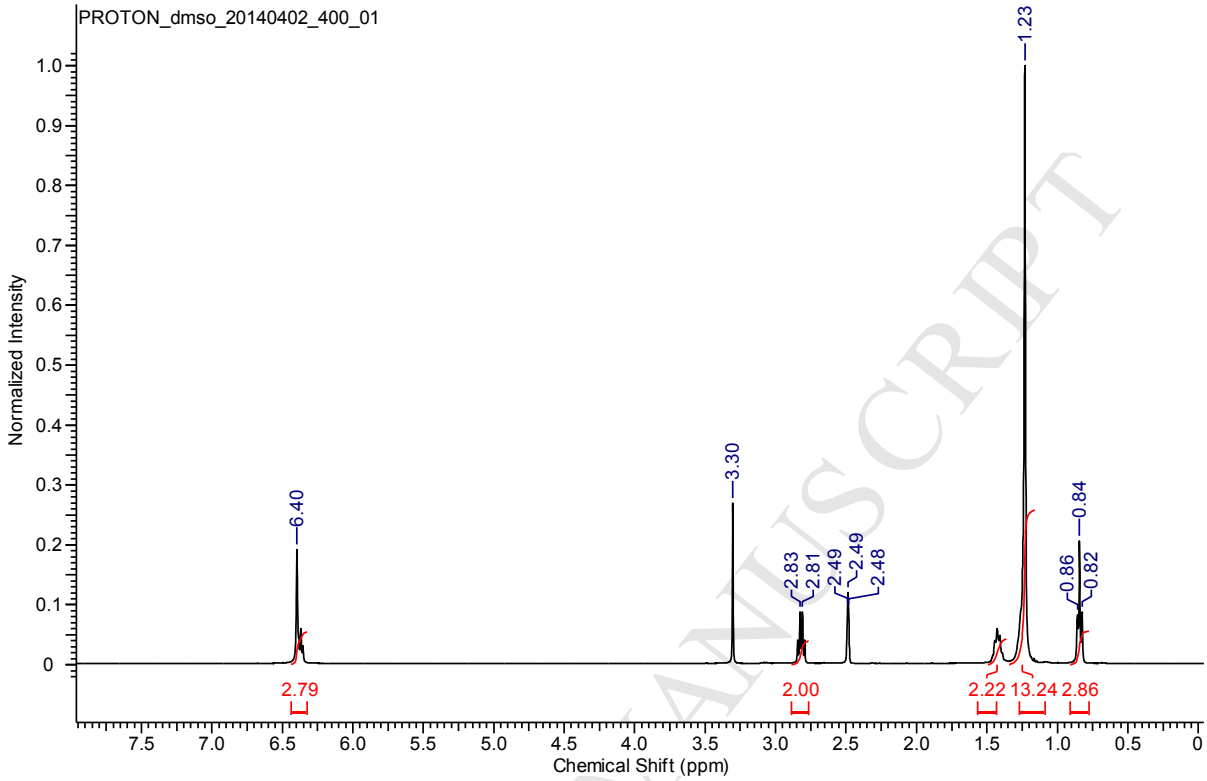
_N_-(Octyl)sulfamide 4b

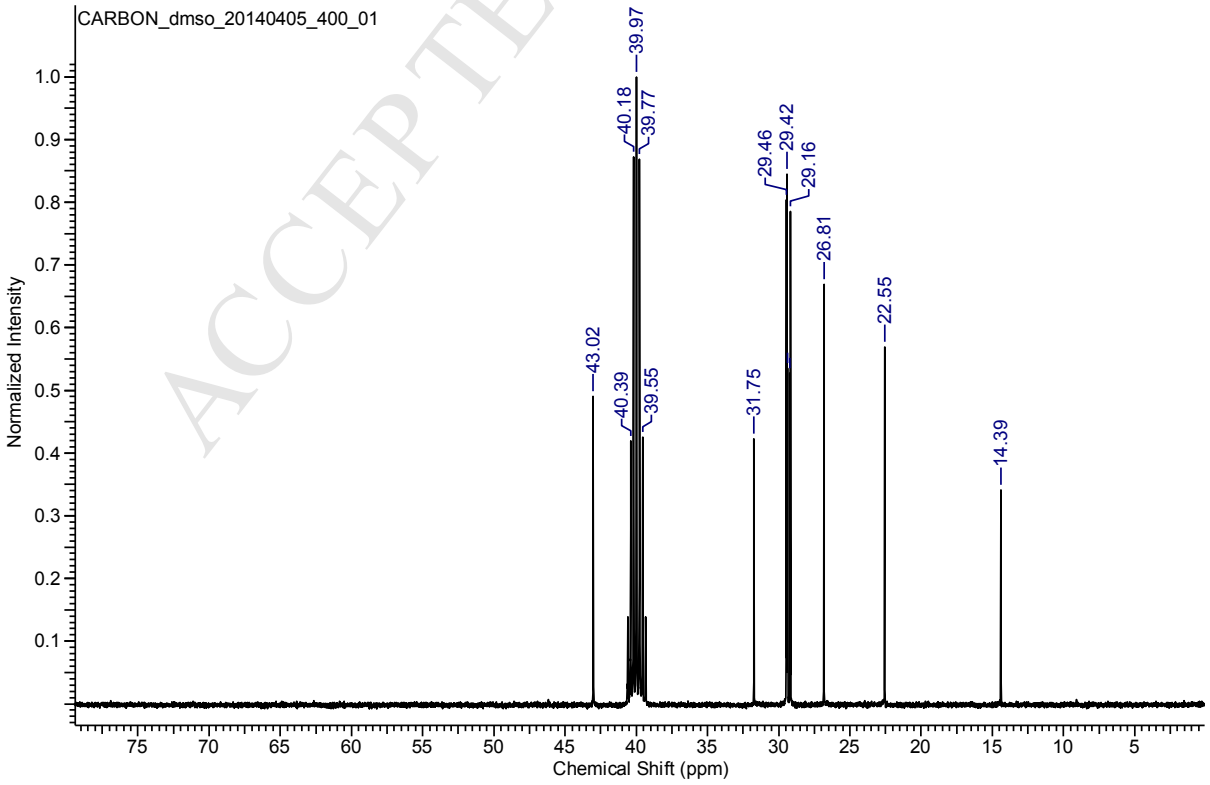




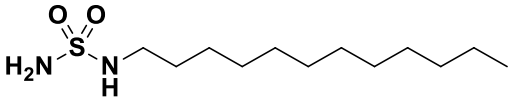
_N_-(Decyl)sulfamide 4c

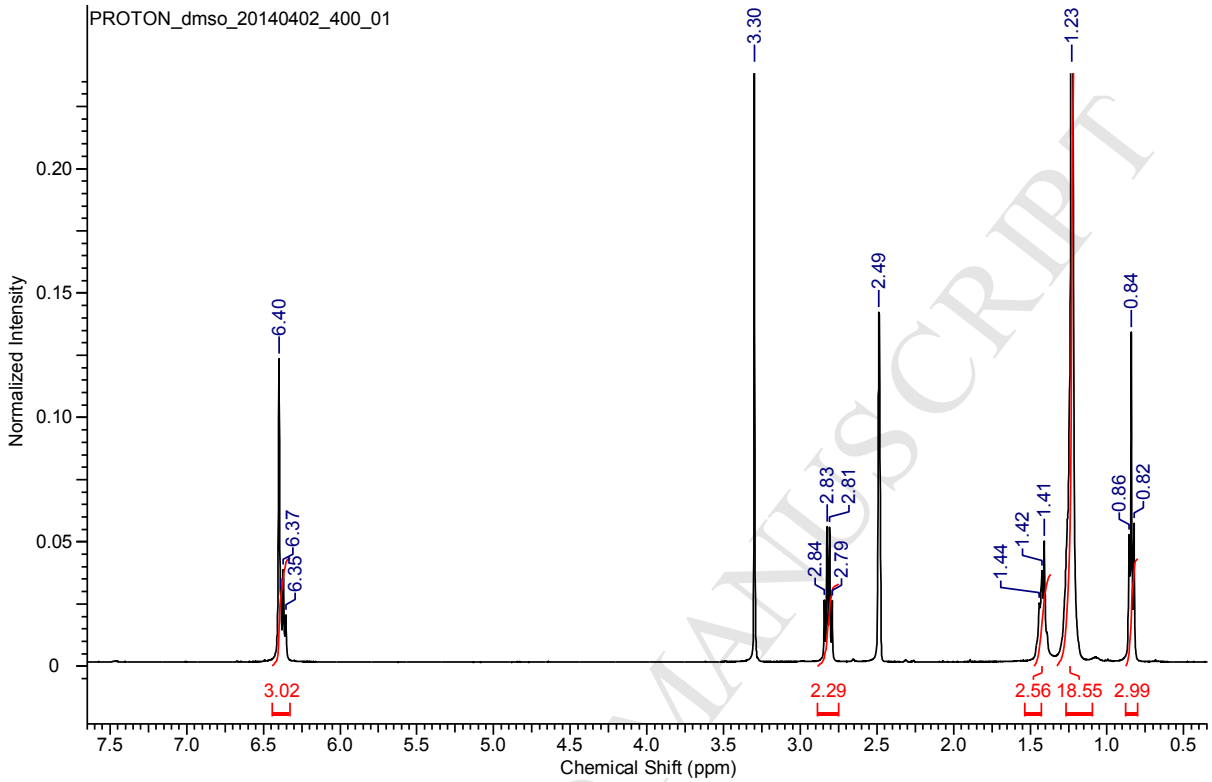


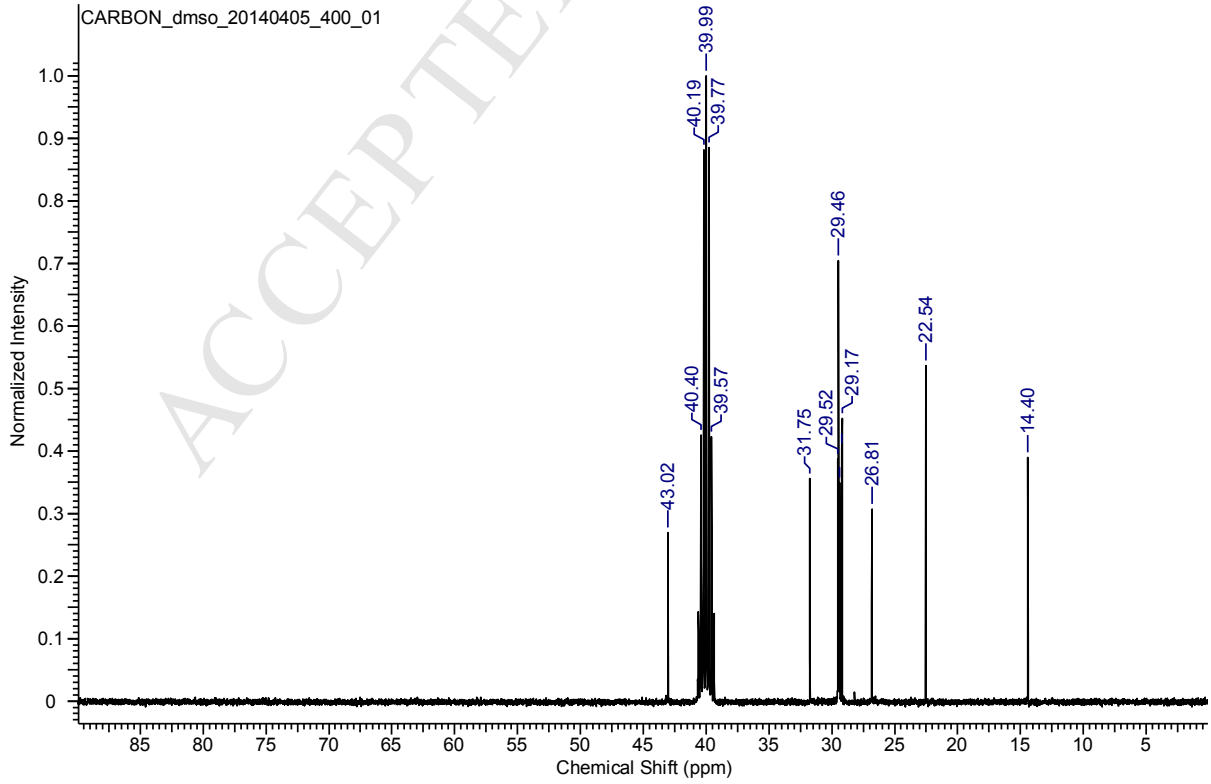




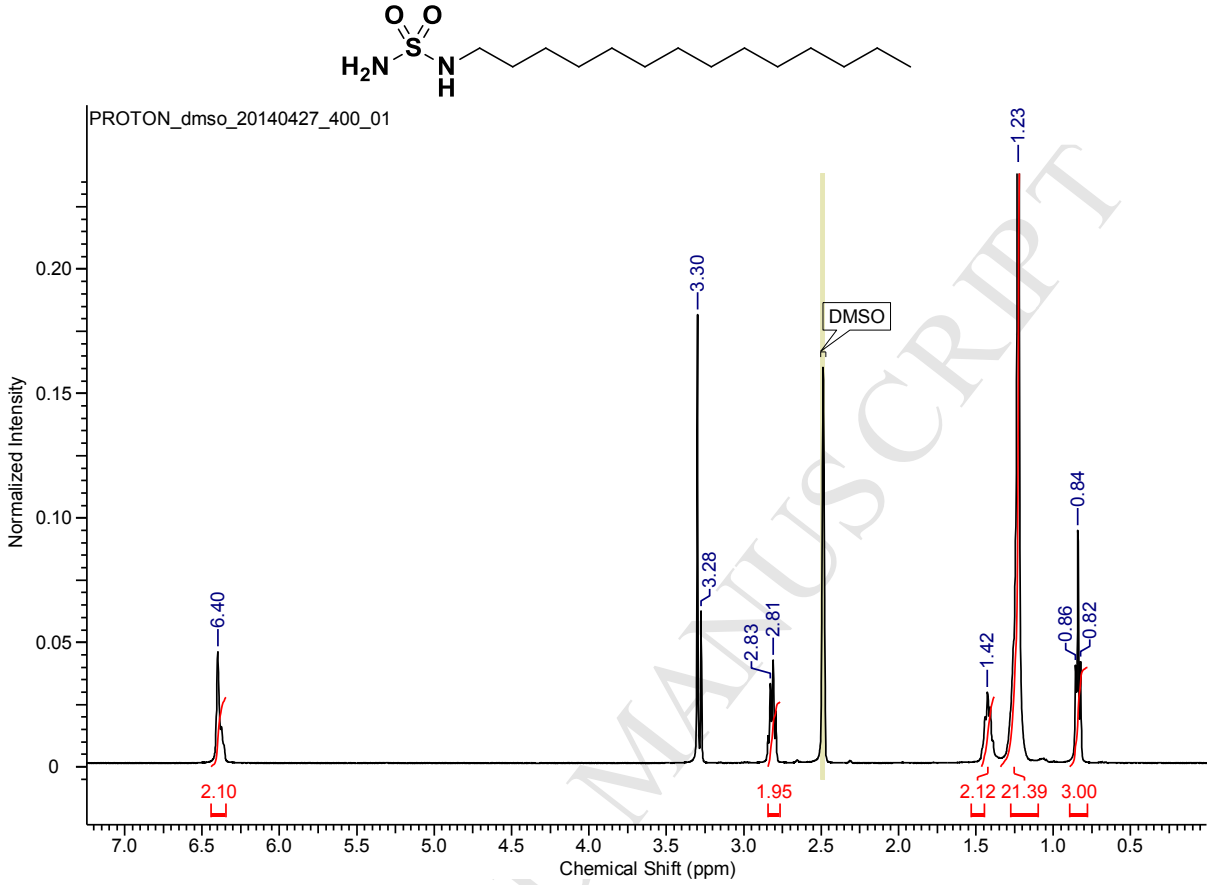
N-(Dodecyl)sulfamide 4d

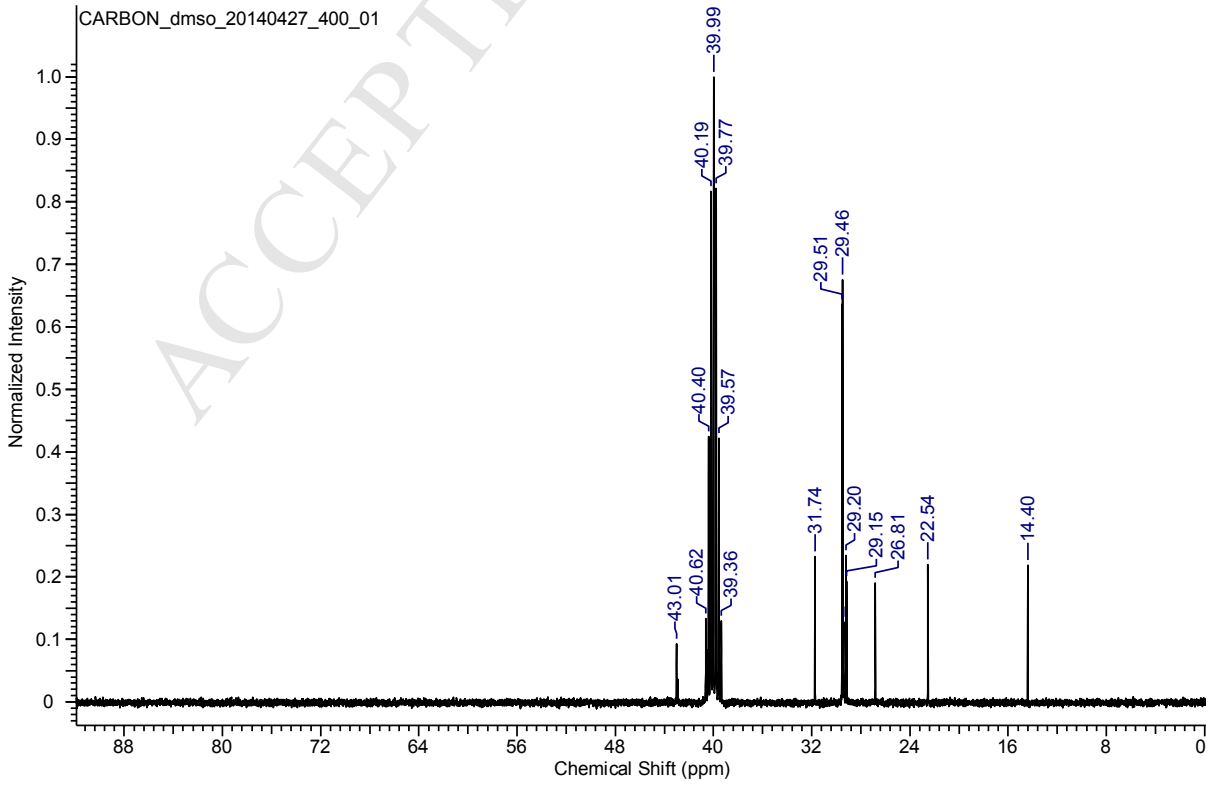




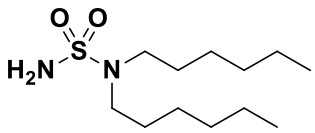


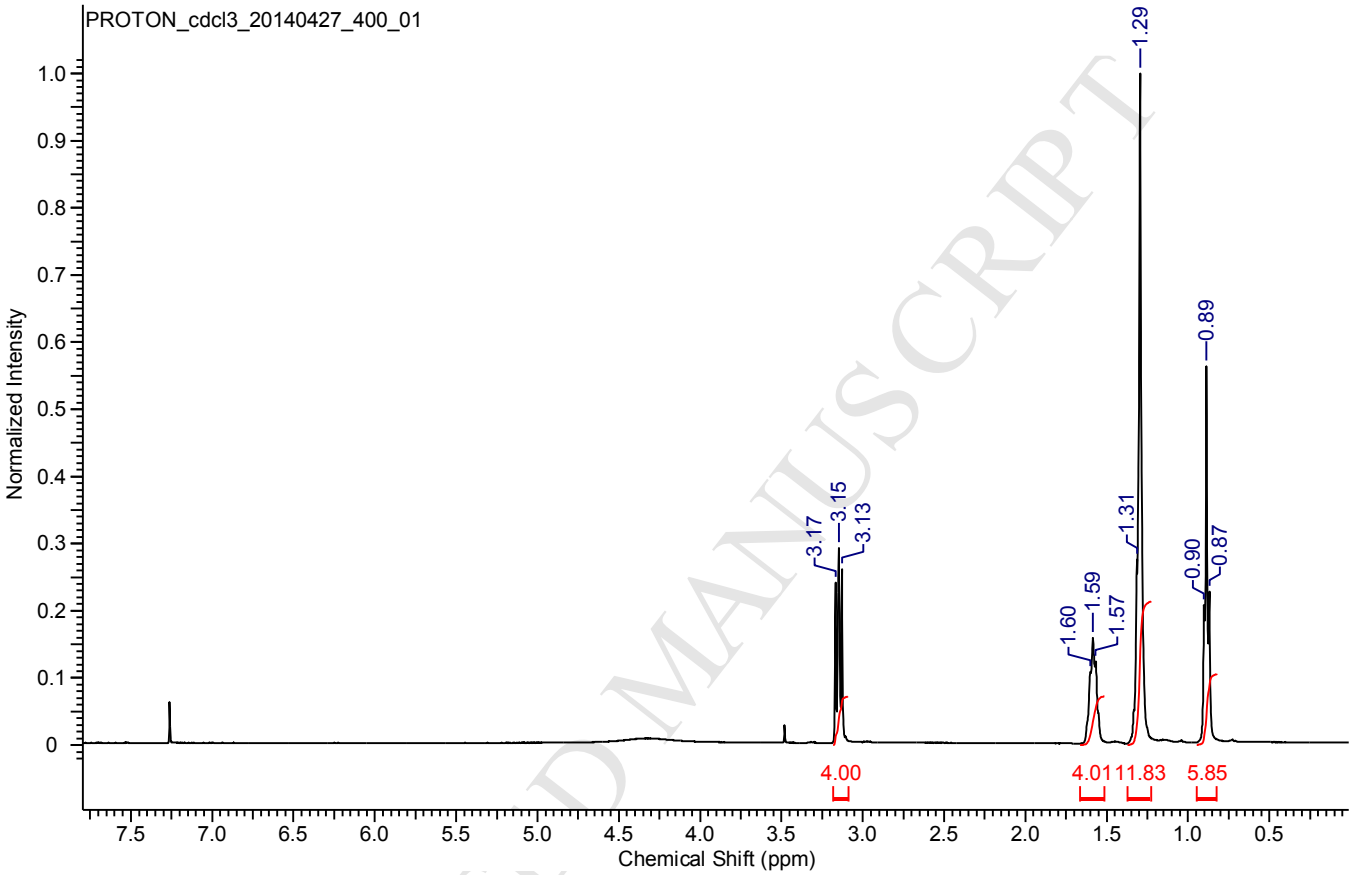
N-(Tetradecyl)sulfamide 4e

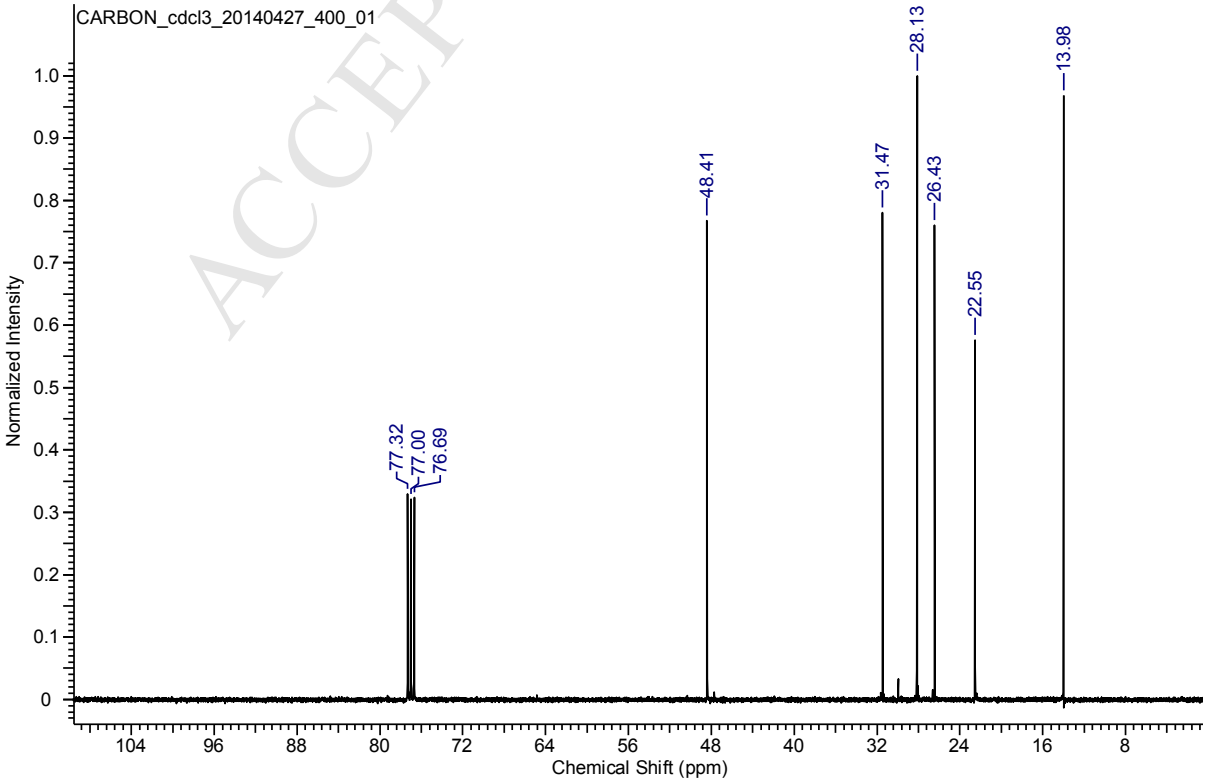




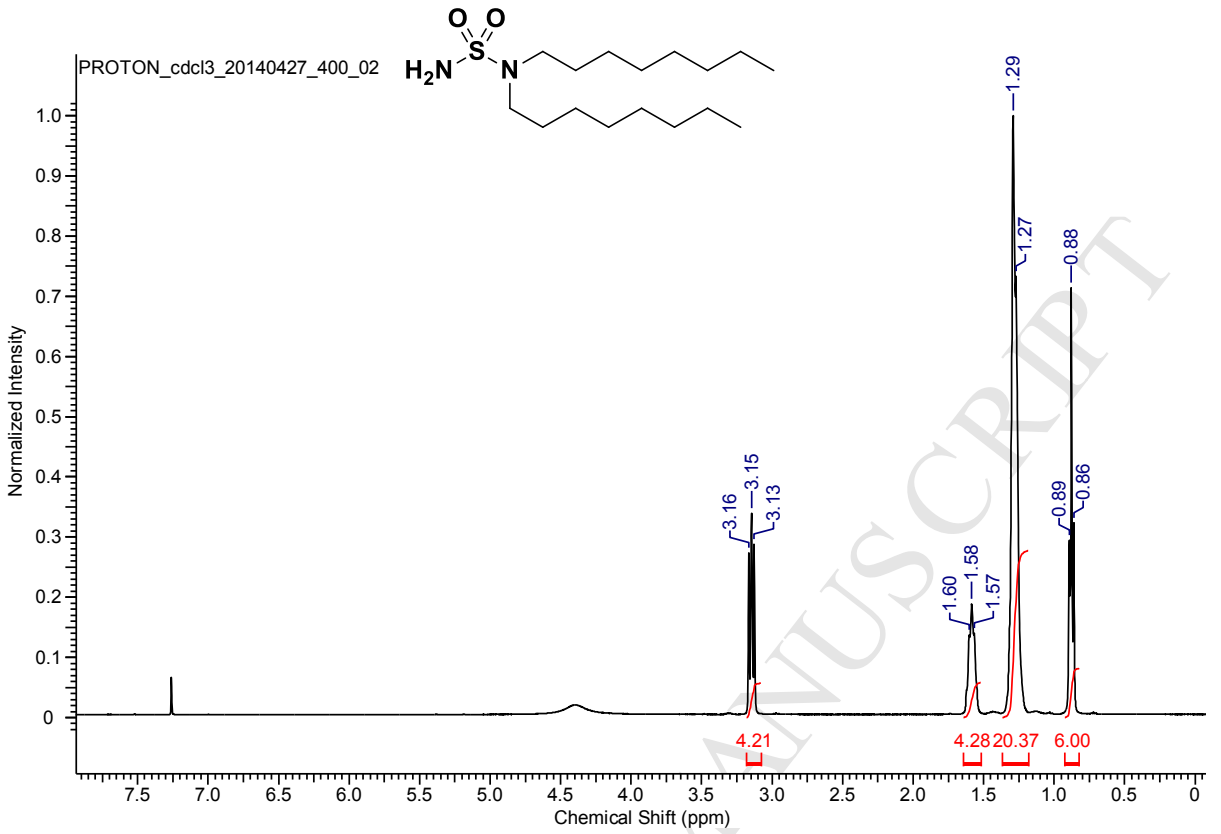
_N,N_-(Dihexyl)sulfamide 4f

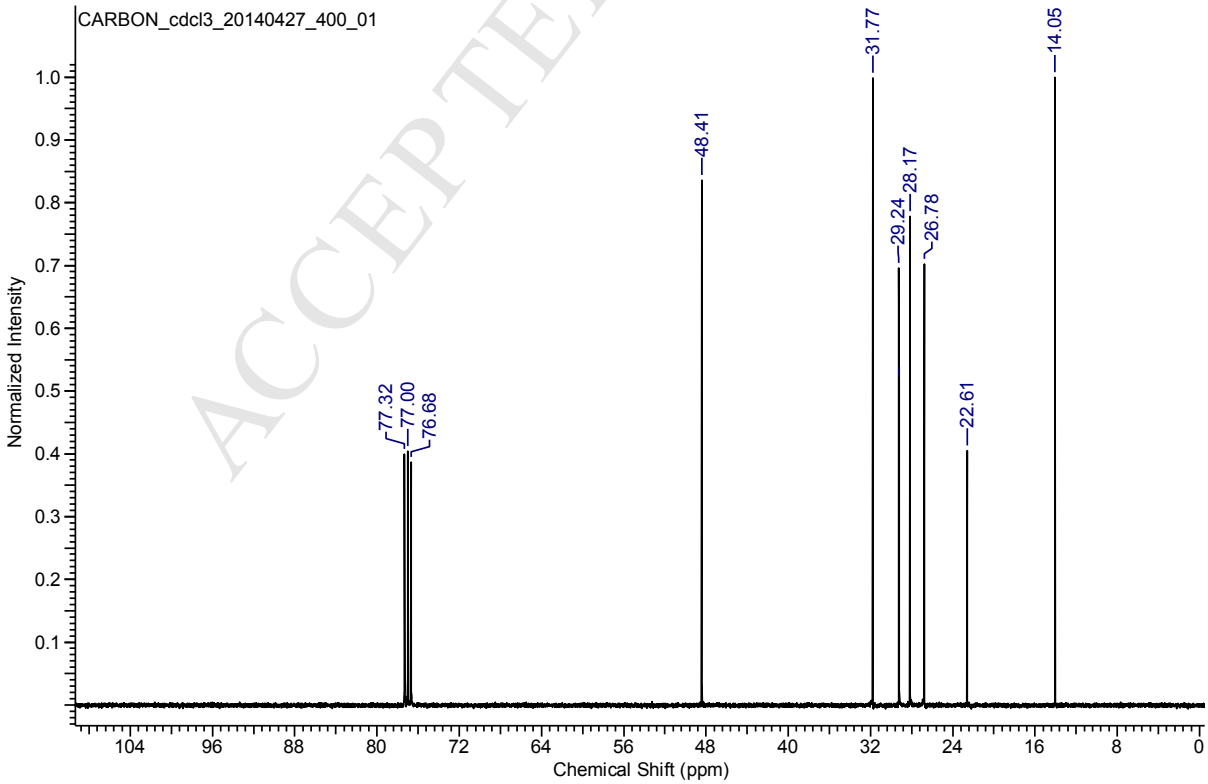




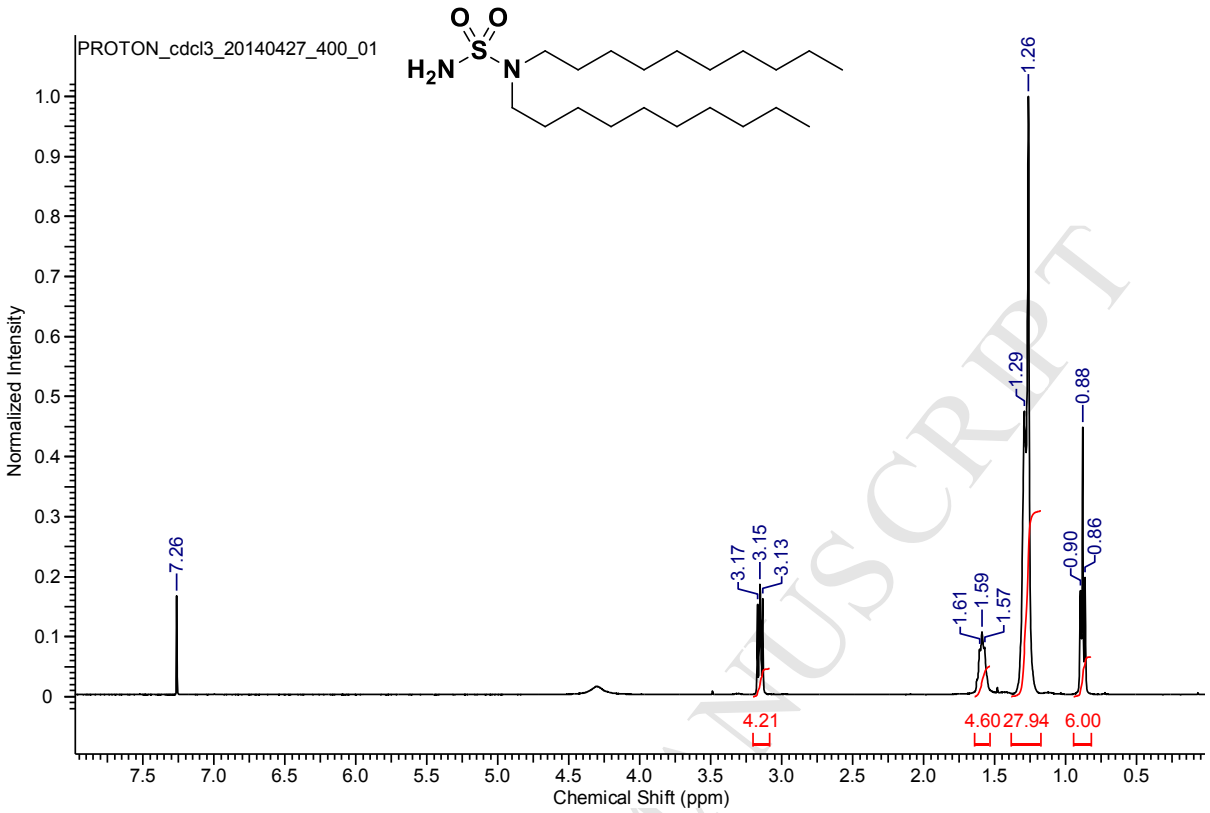


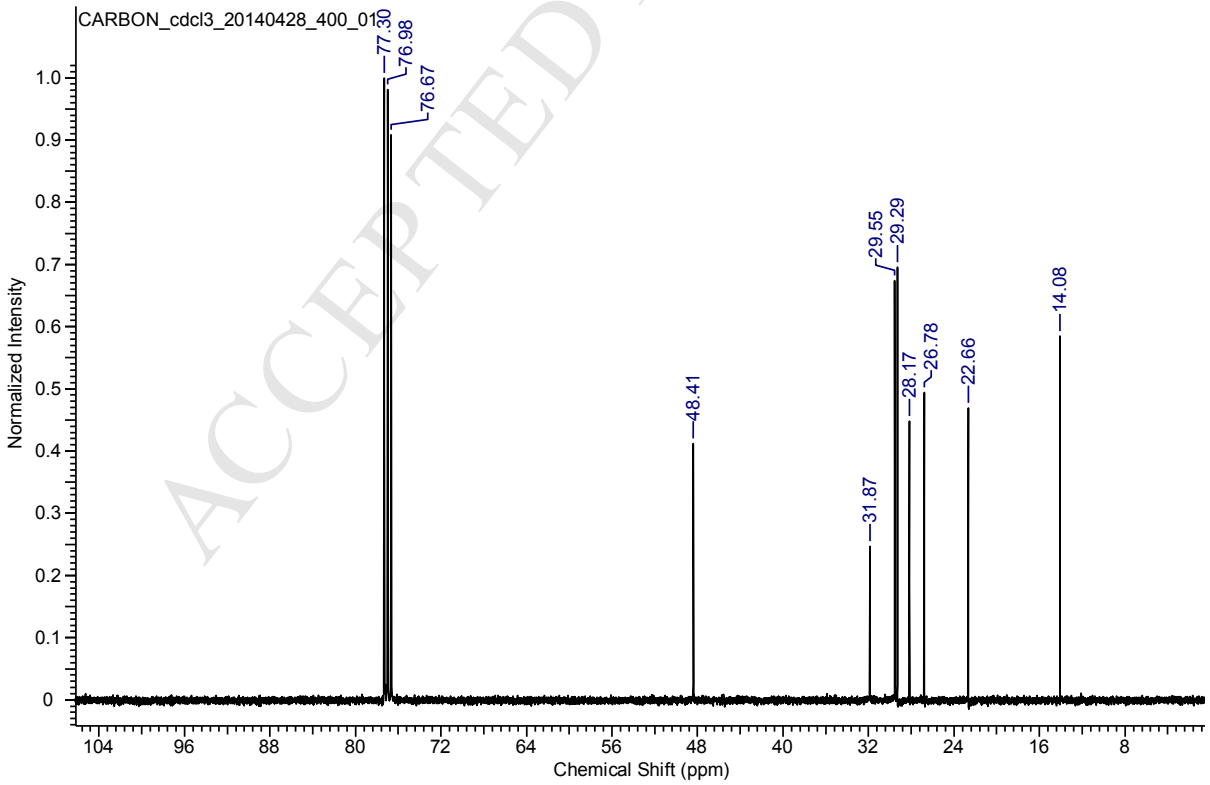
_N,N_-(Dioctyl)sulfamide 4g



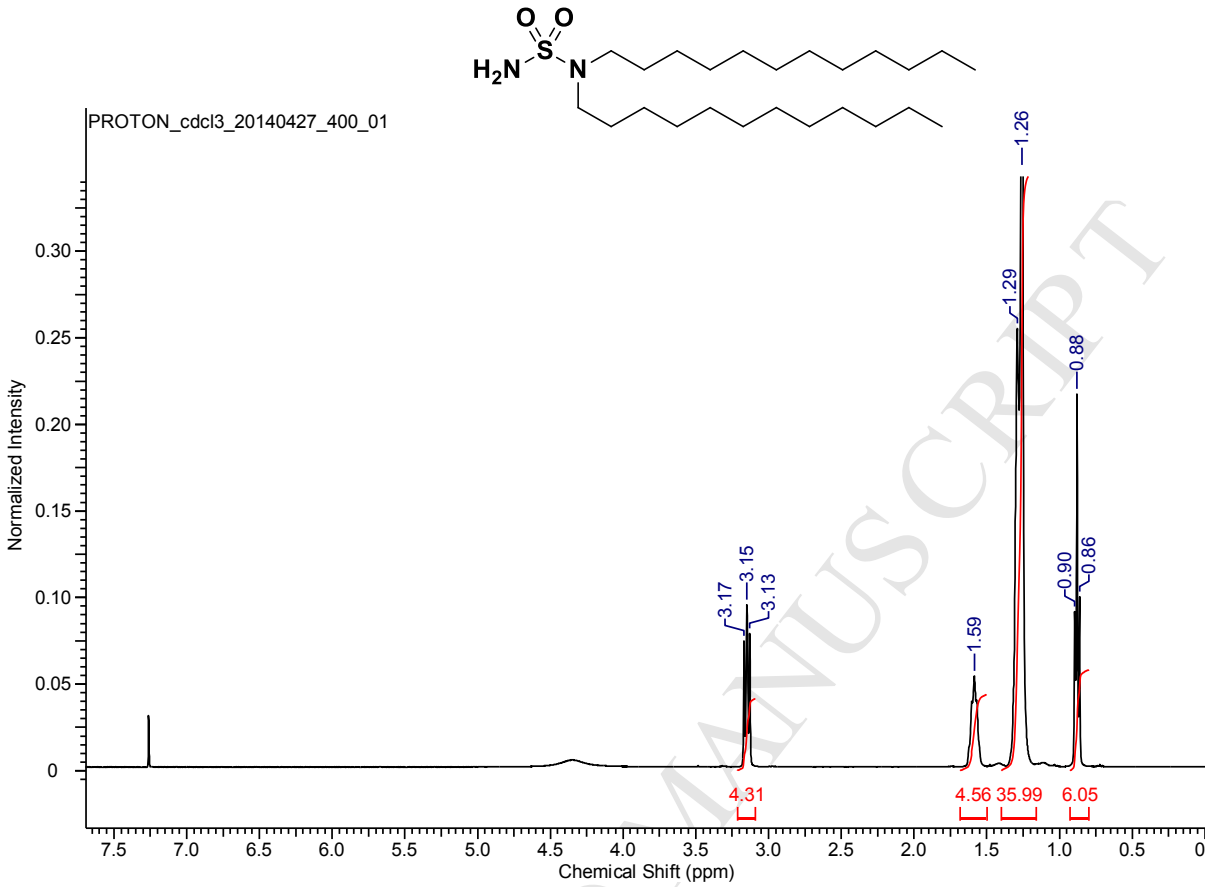


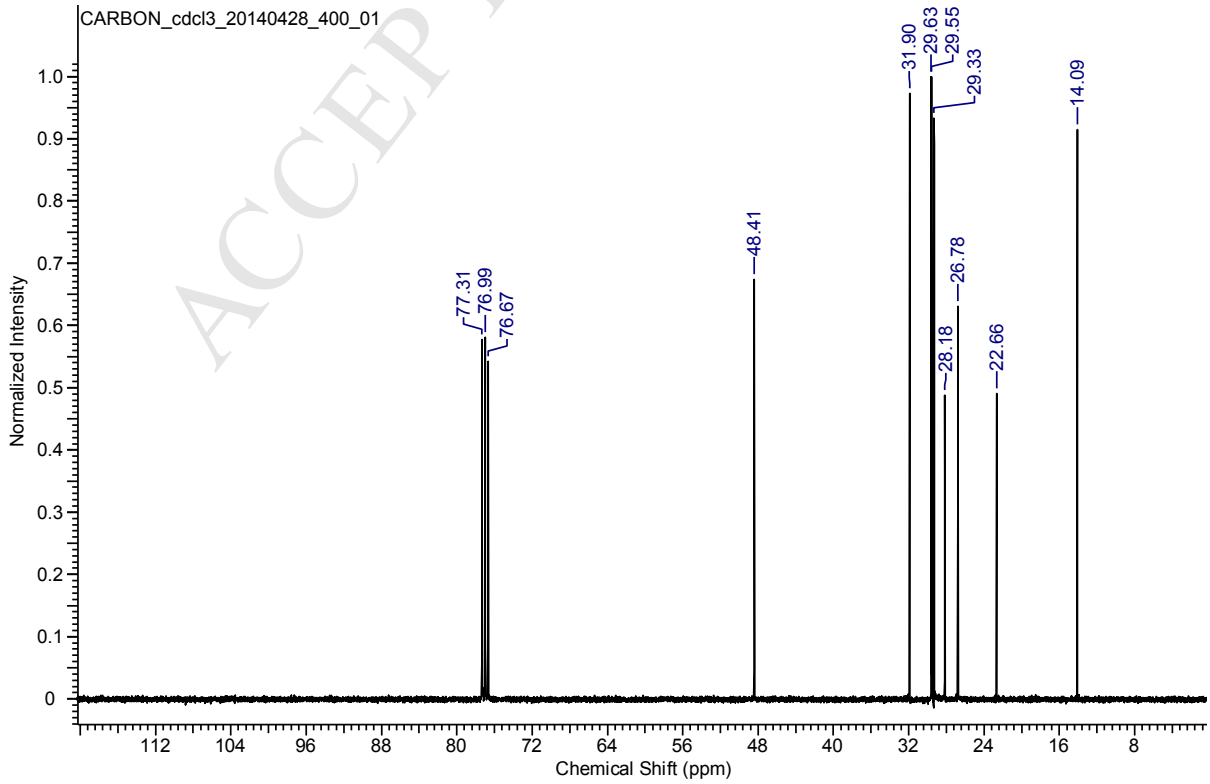
N,N-(Didecyl)sulfamide 4h



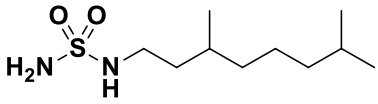


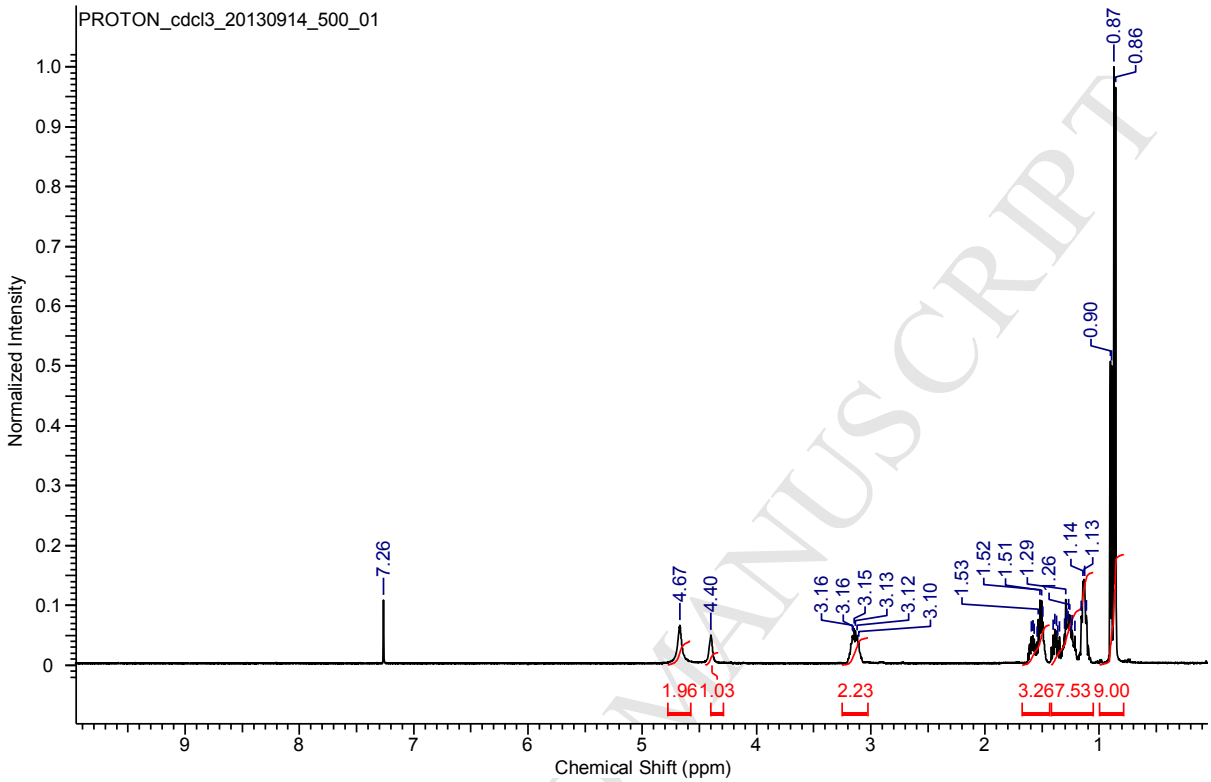
***N,N*-(Didodecyl)sulfamide 4i**

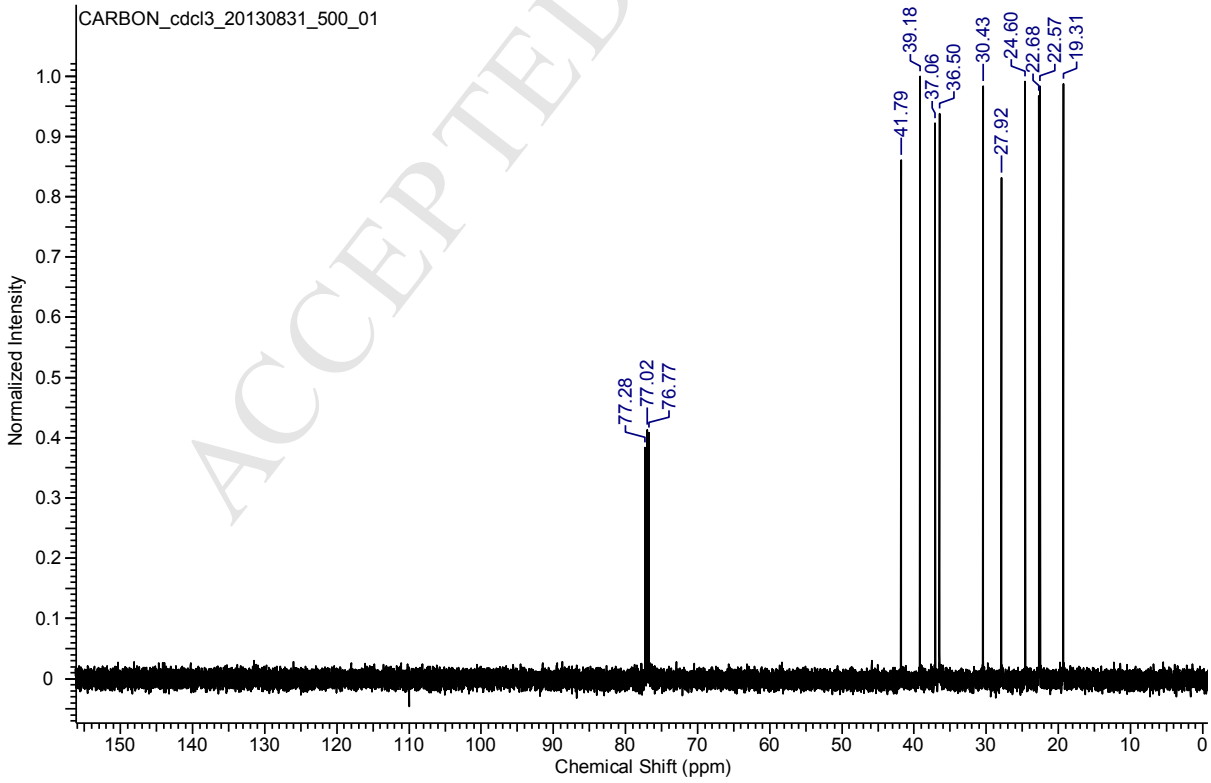




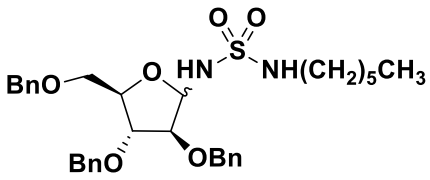
_N_-(3,7-Dimethyloctyl)sulfamide 4j

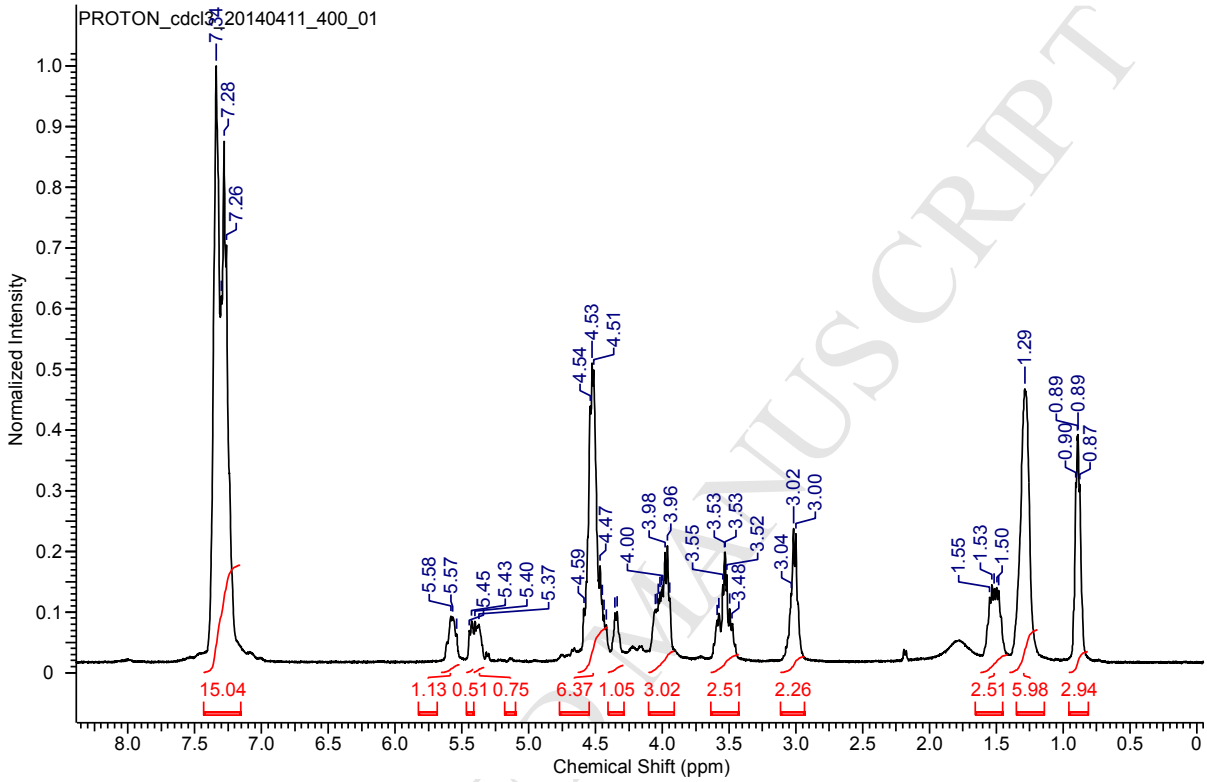


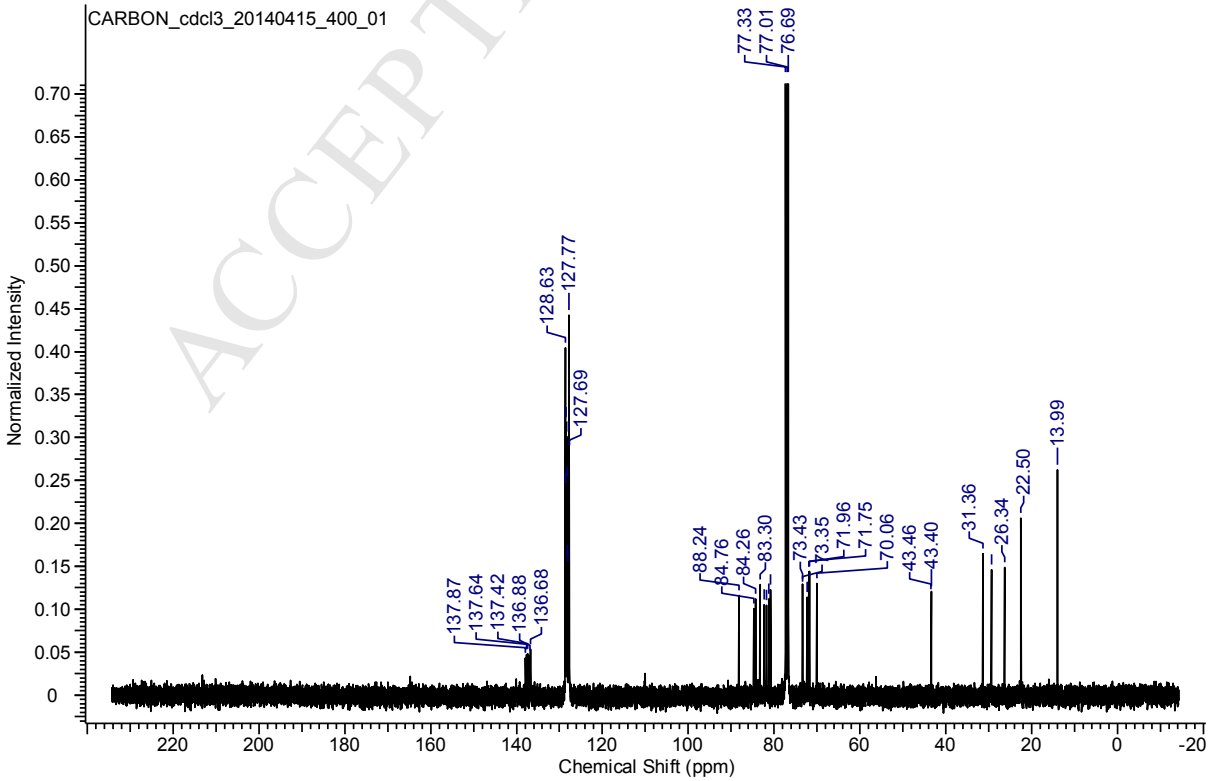




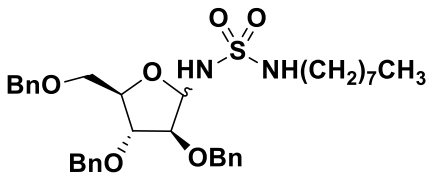
_N_-(Hexyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5a

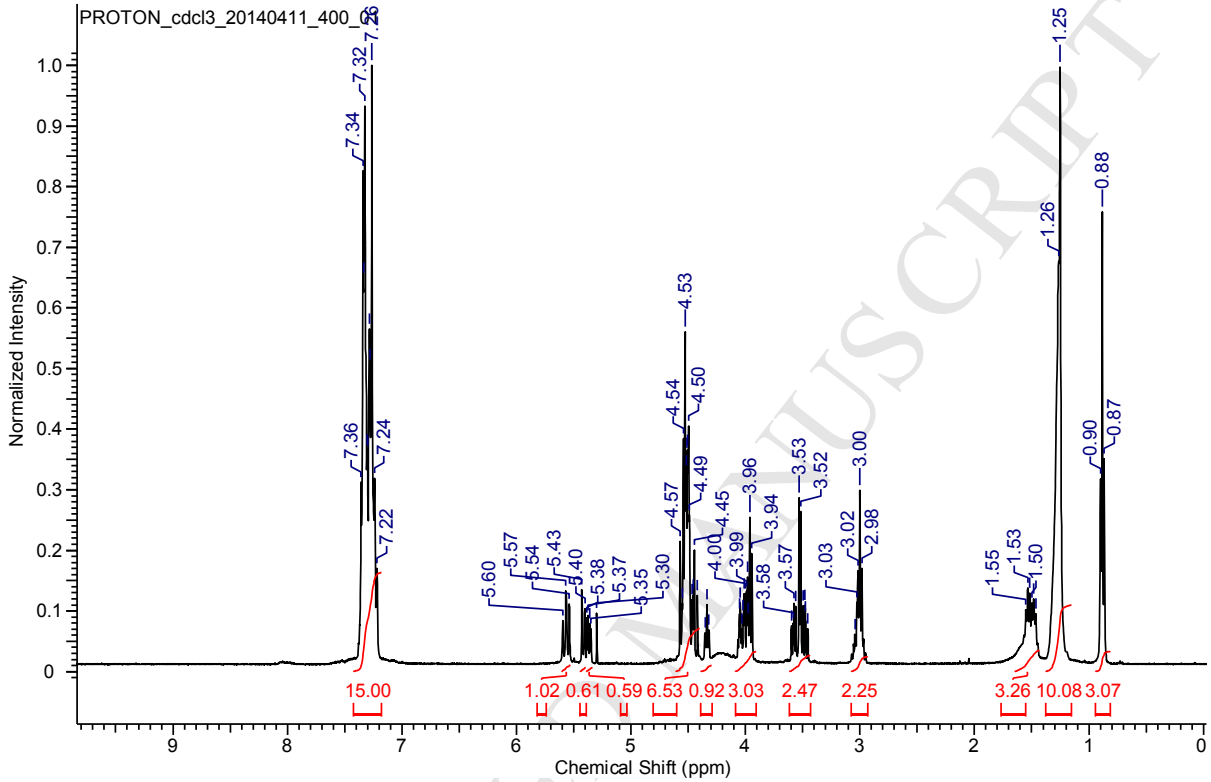


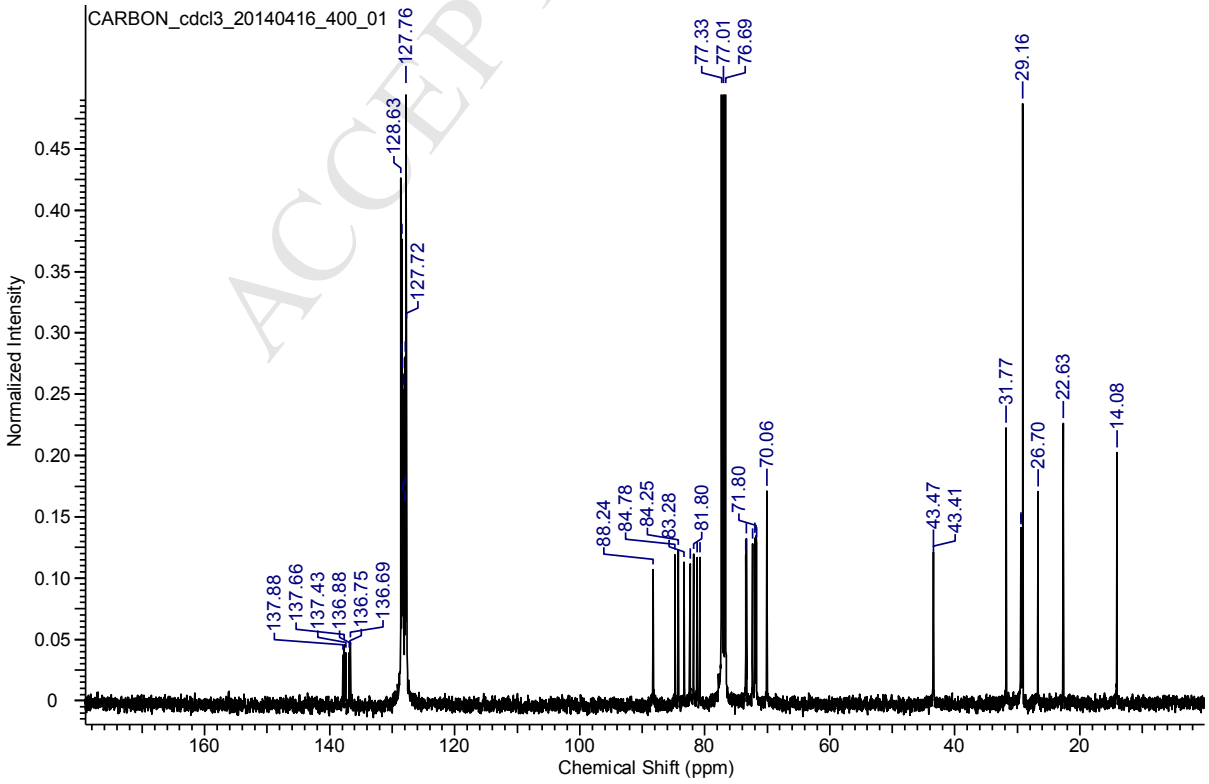




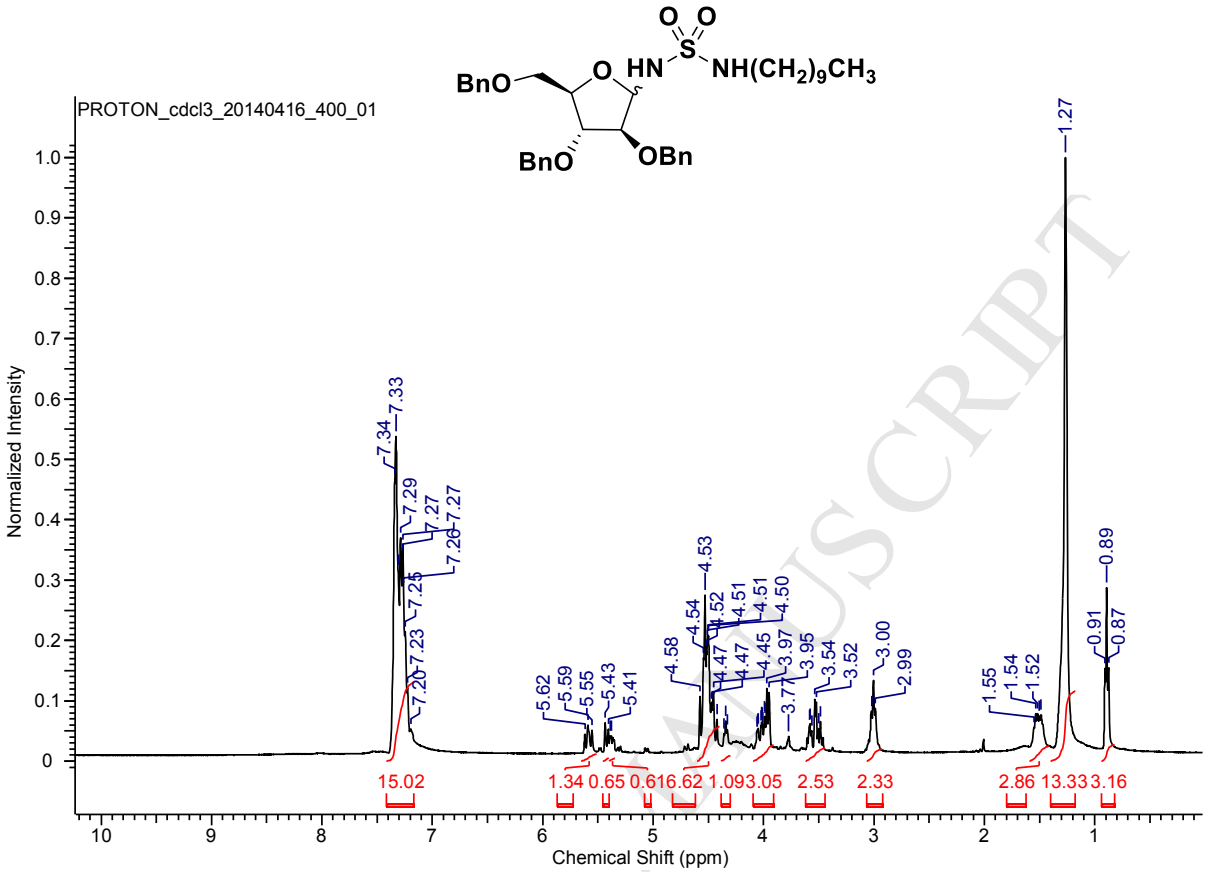
_N_-(Octyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5b

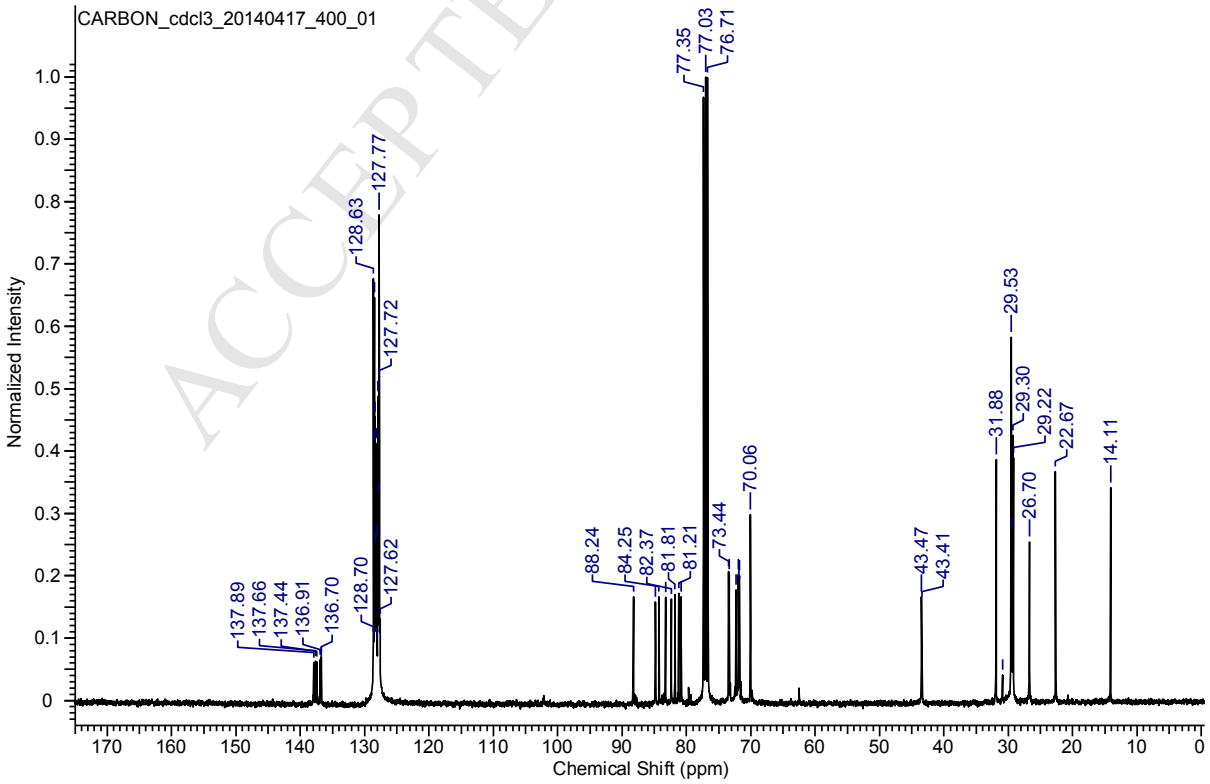




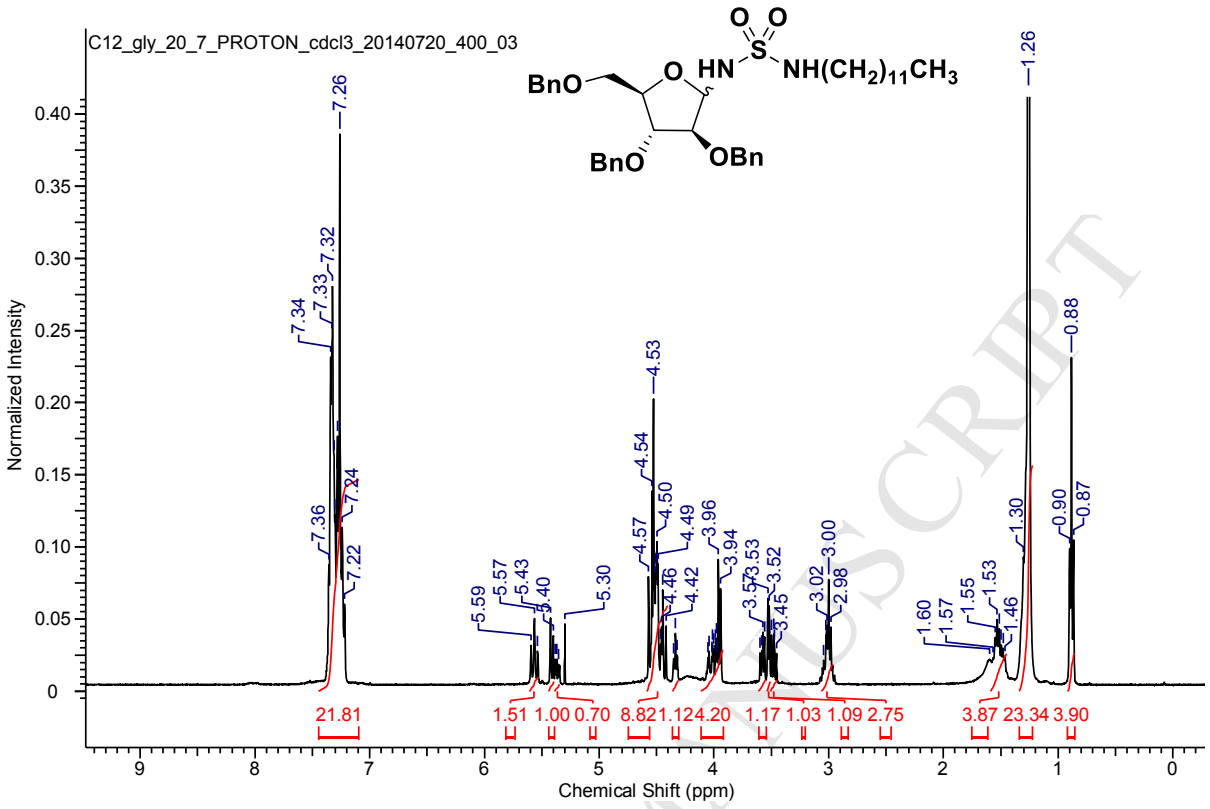


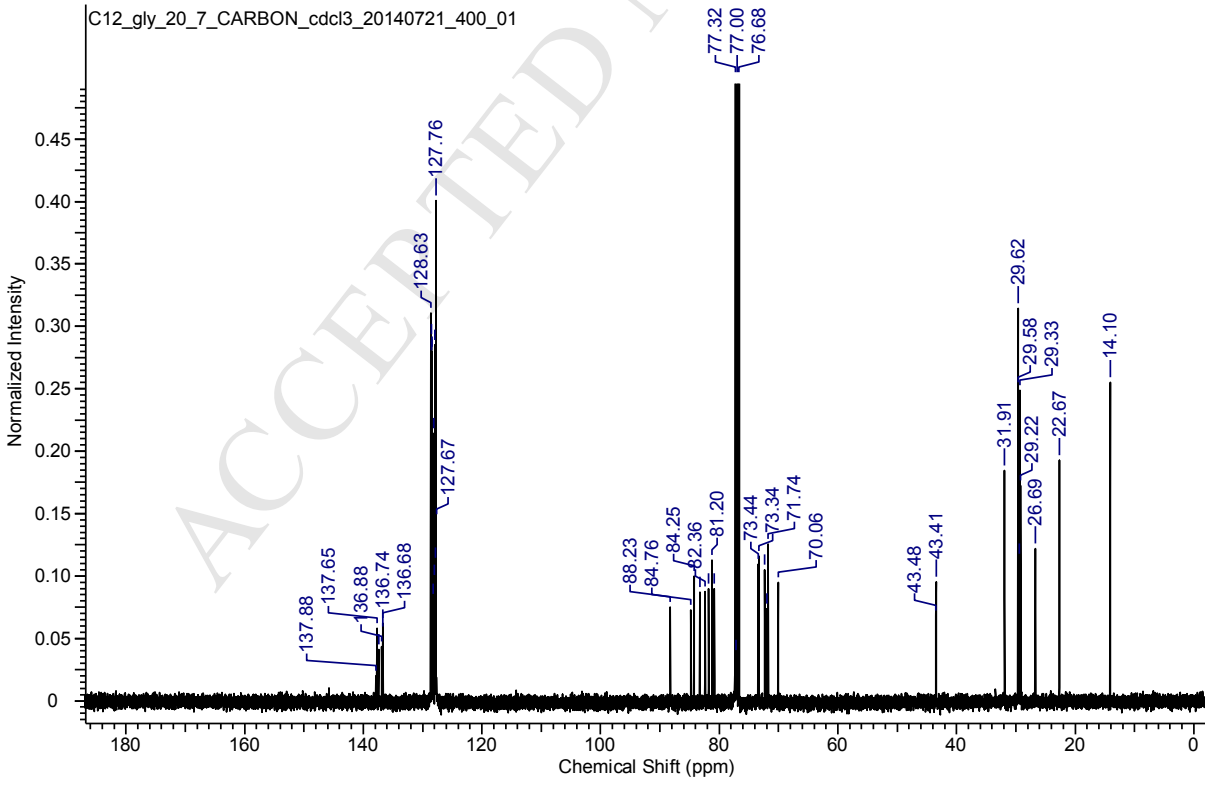
_N_-(Decyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5c



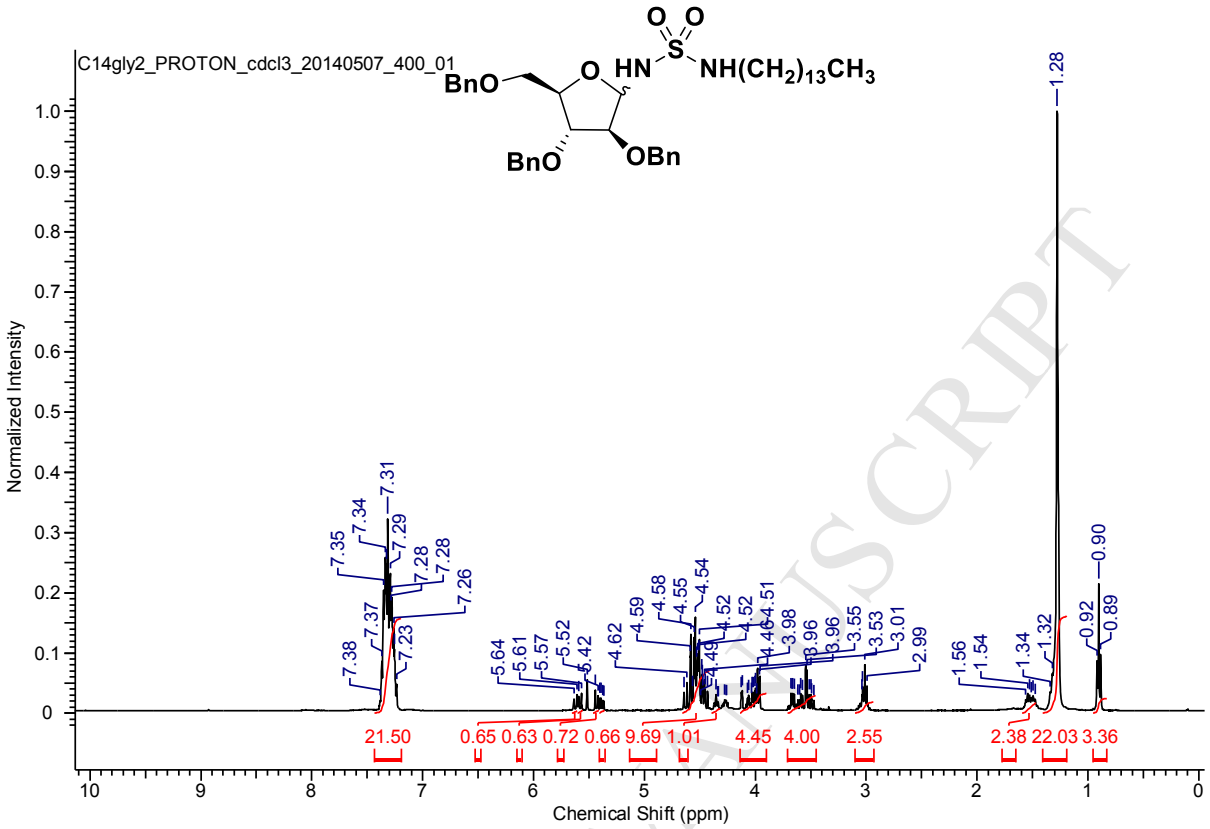


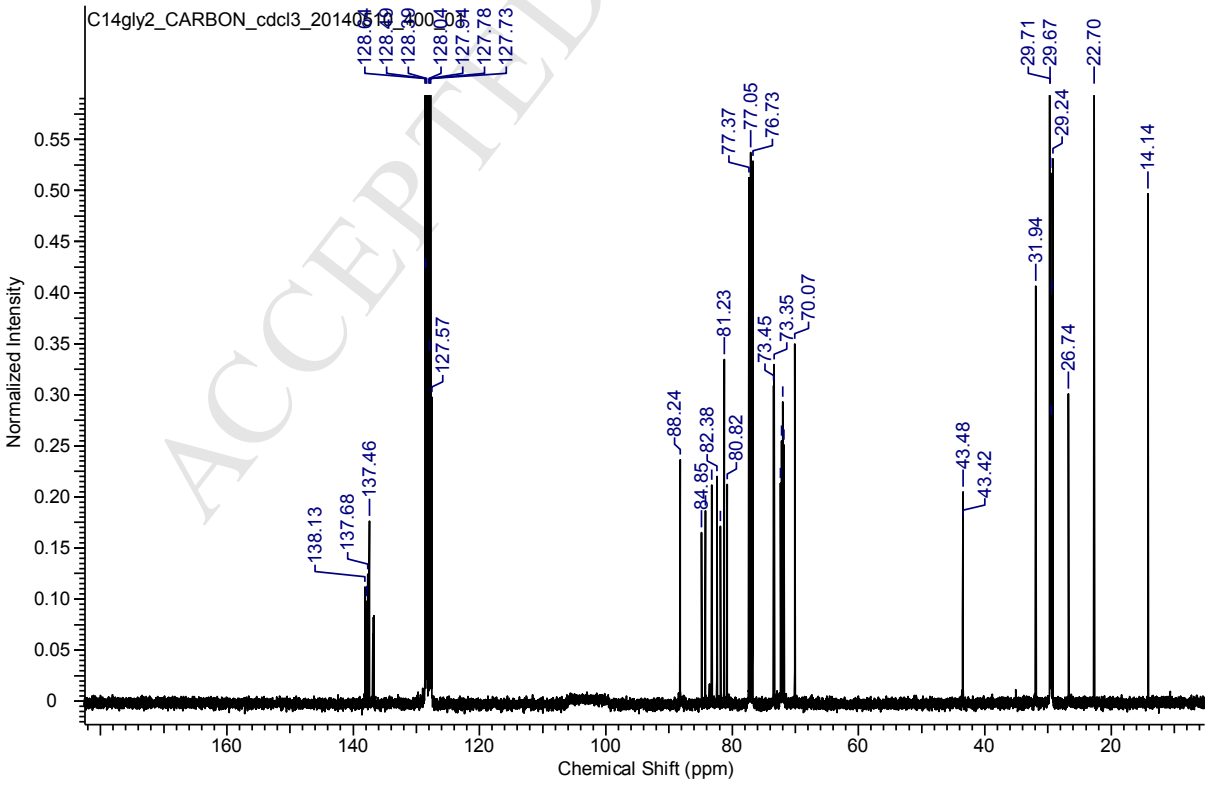
_N_-(Dodecyl)-_N'_-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5d



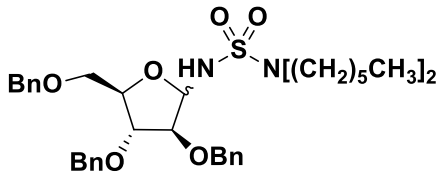


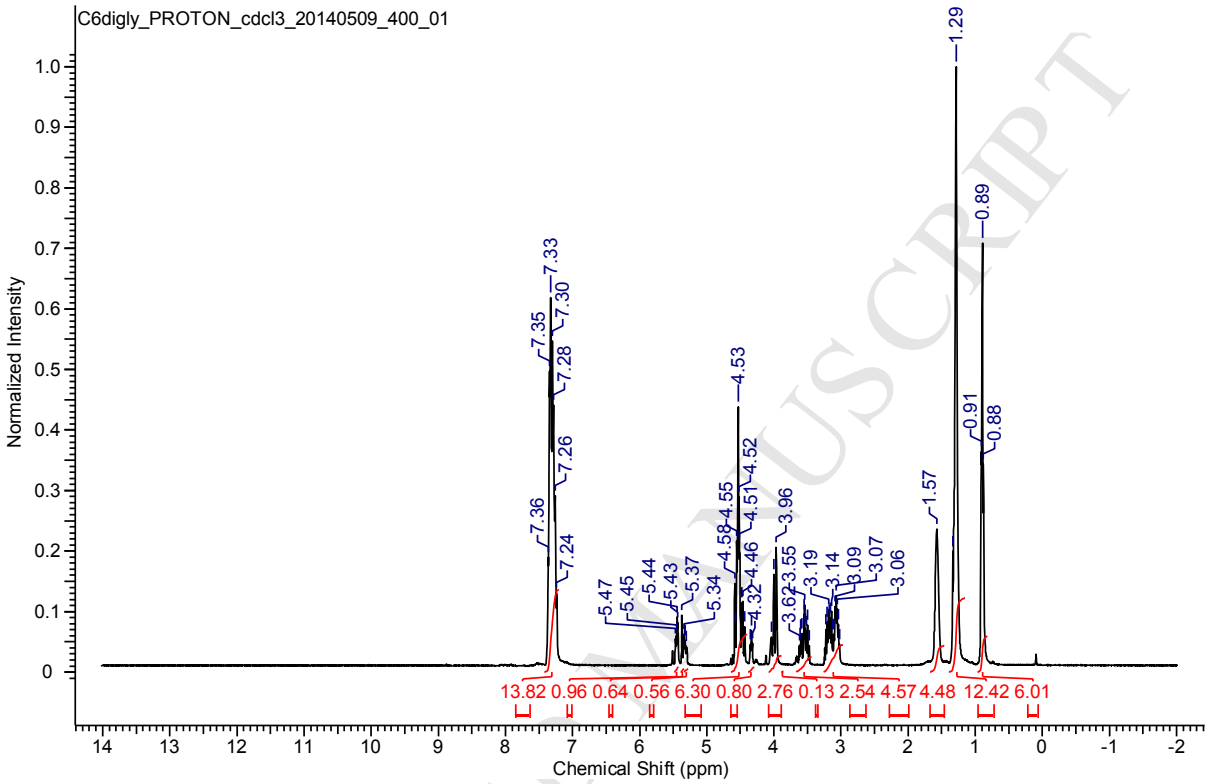
_N_-(Tetradecyl)-_N'_-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5e

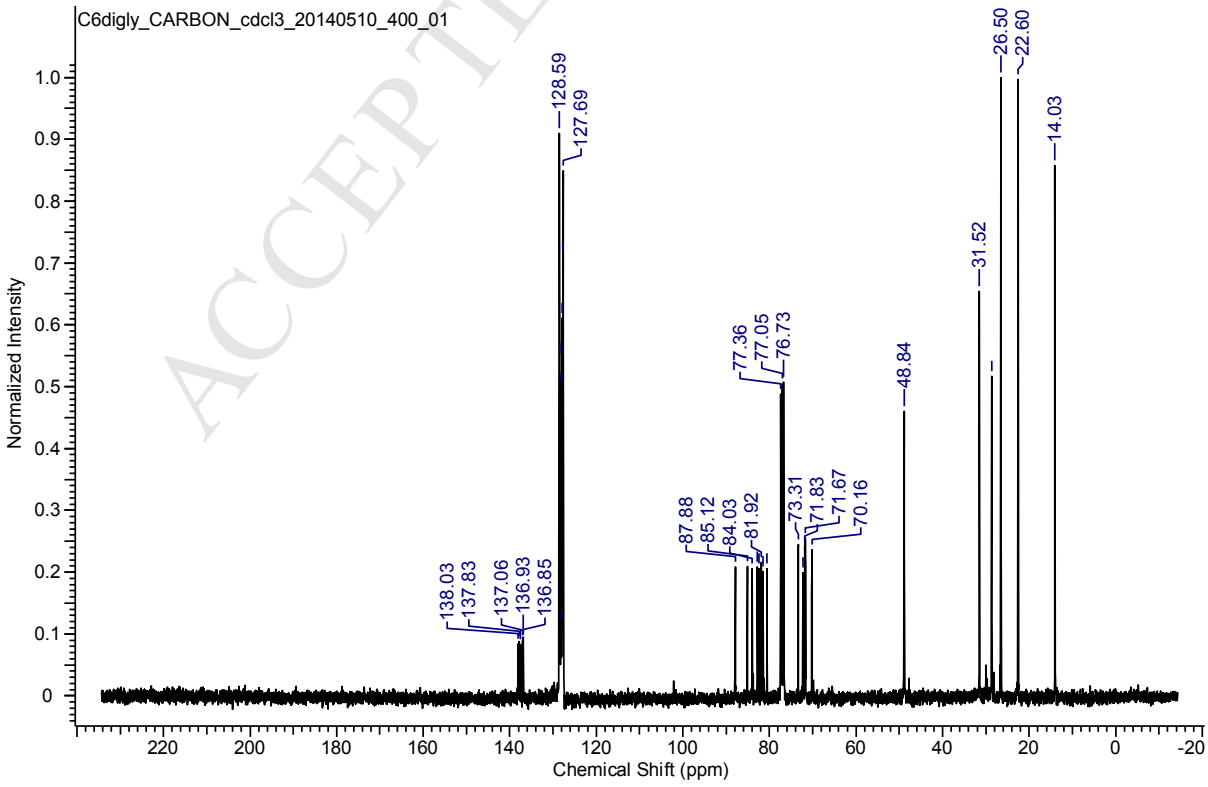




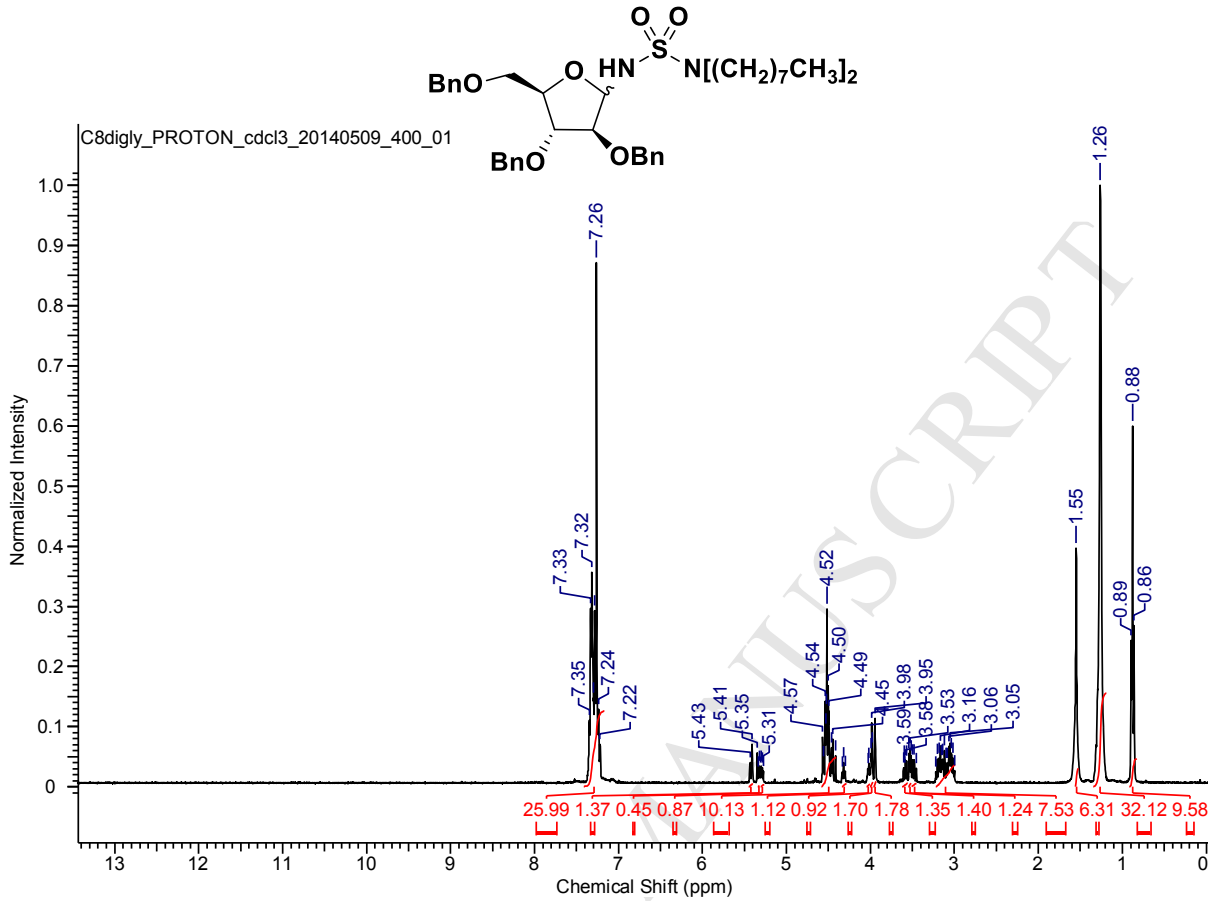
_N_-(Dihexyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5f

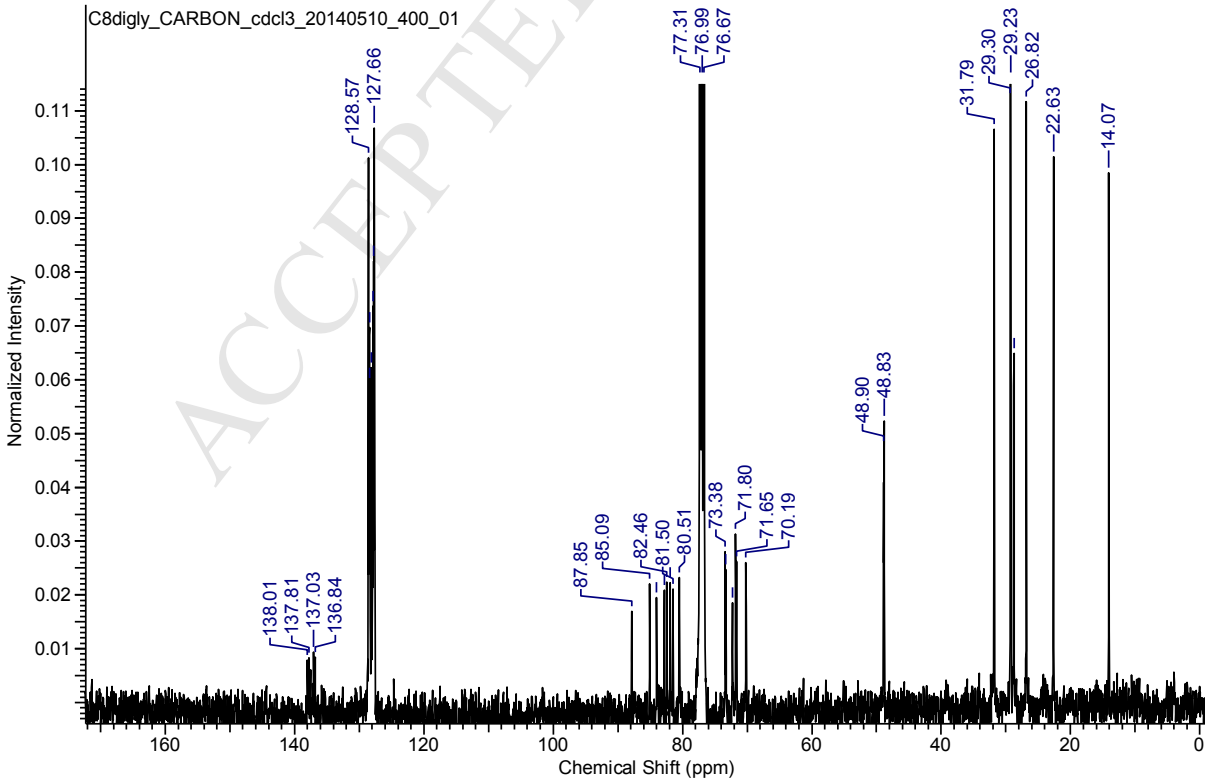




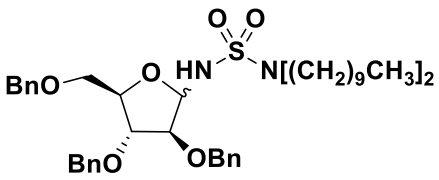


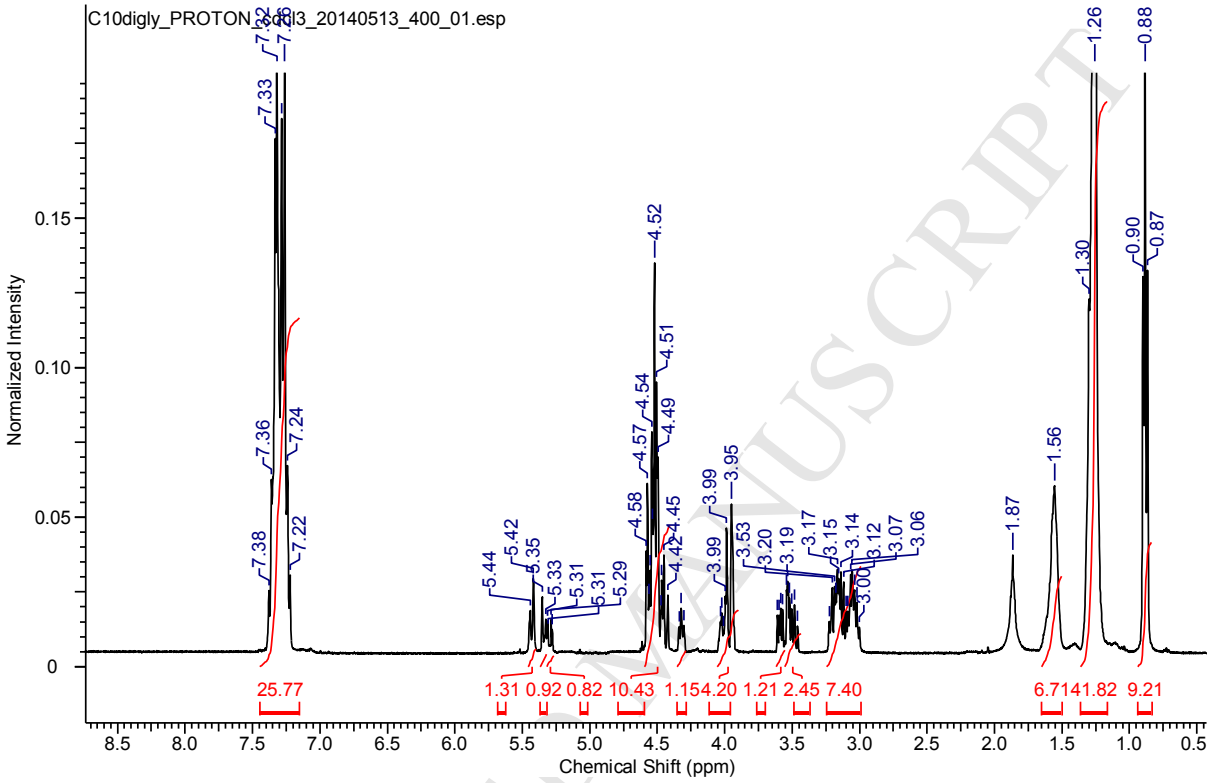
_N_-(Dioctyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5g

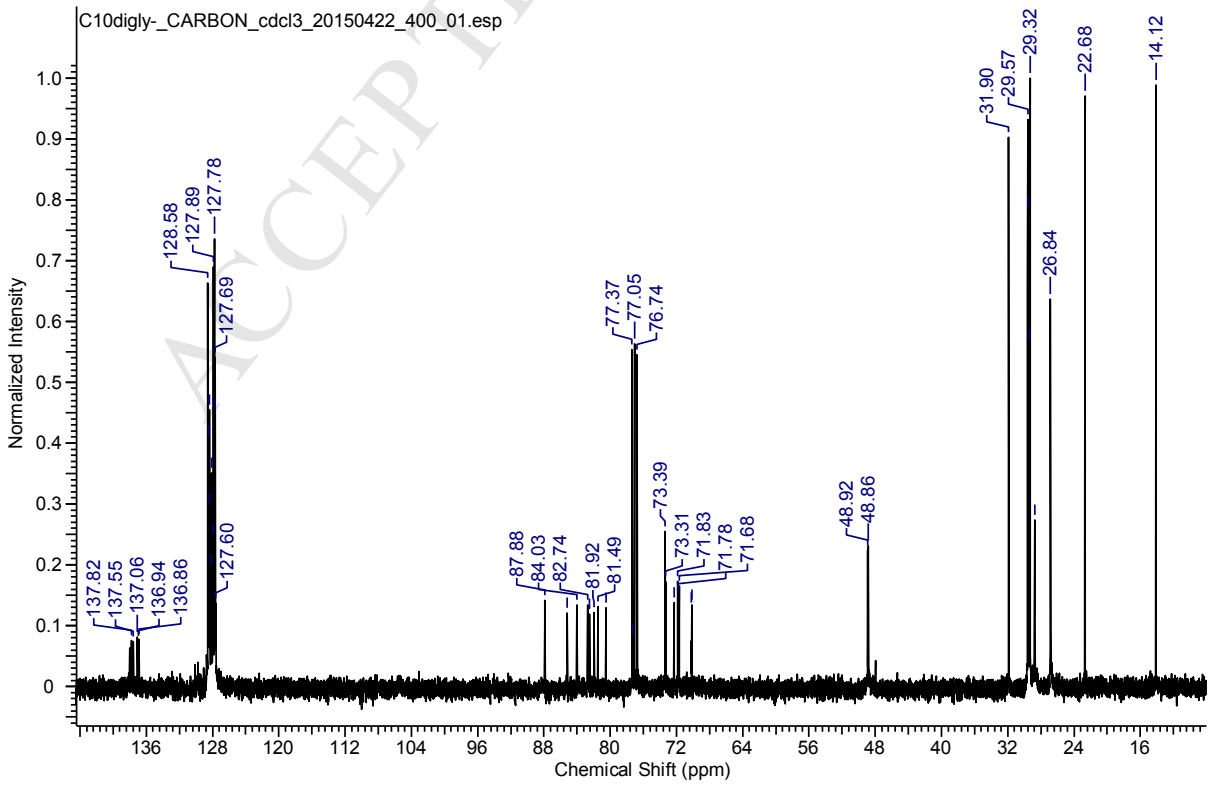




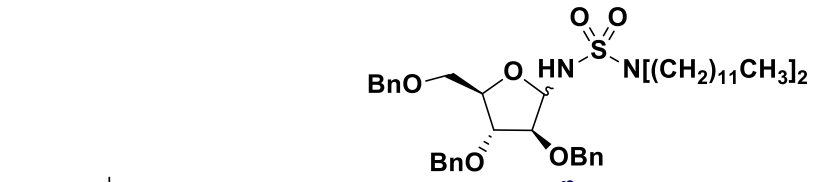
_N_-(Didecyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5h

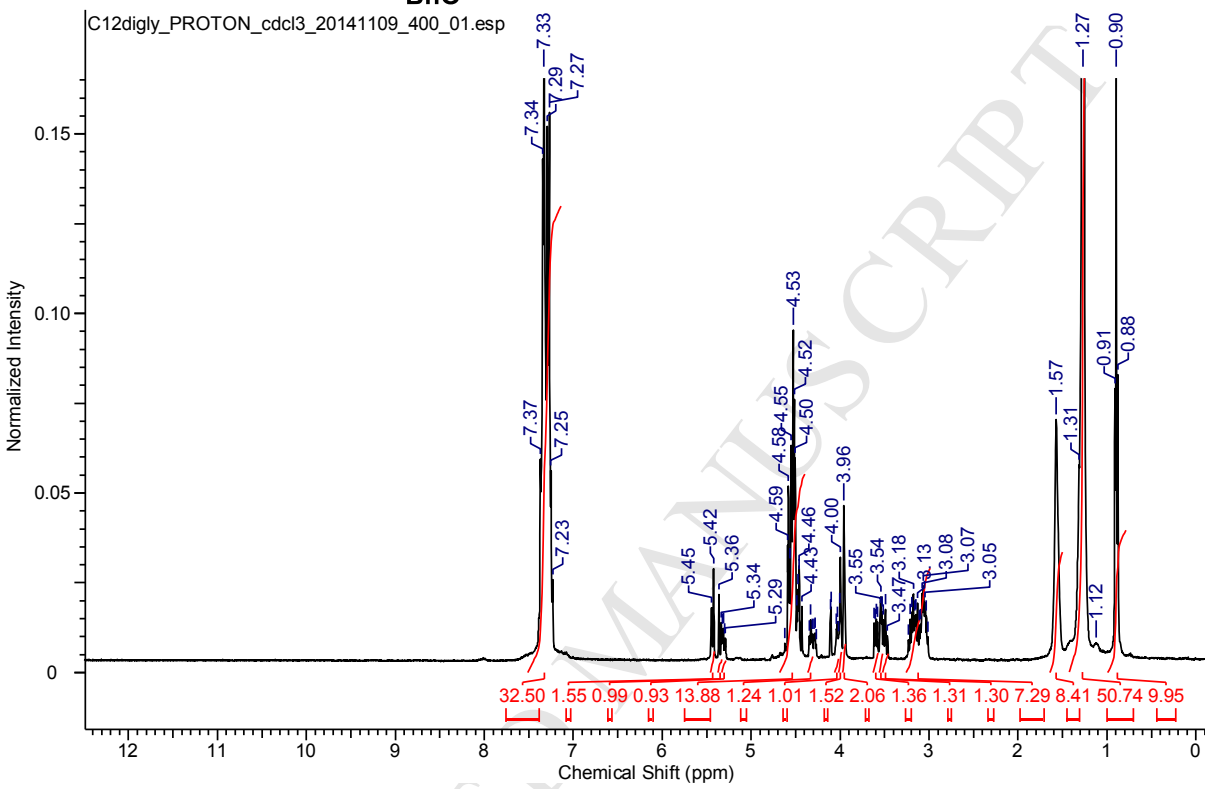


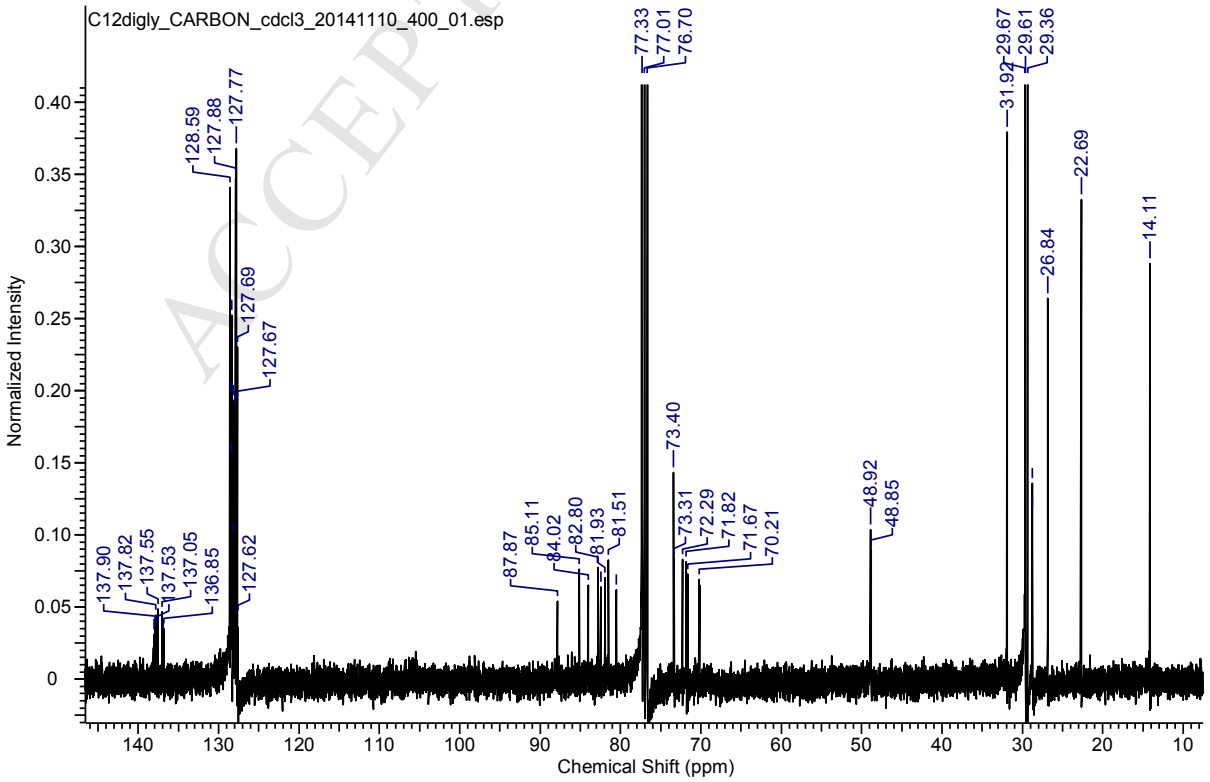




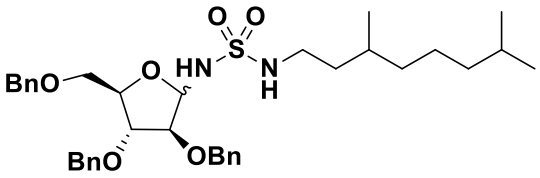
N-(Didodecyl)-N'-(2,3,5-tri-O-benzyl-α,β-D-arabinofuranosyl)sulfamide 5i

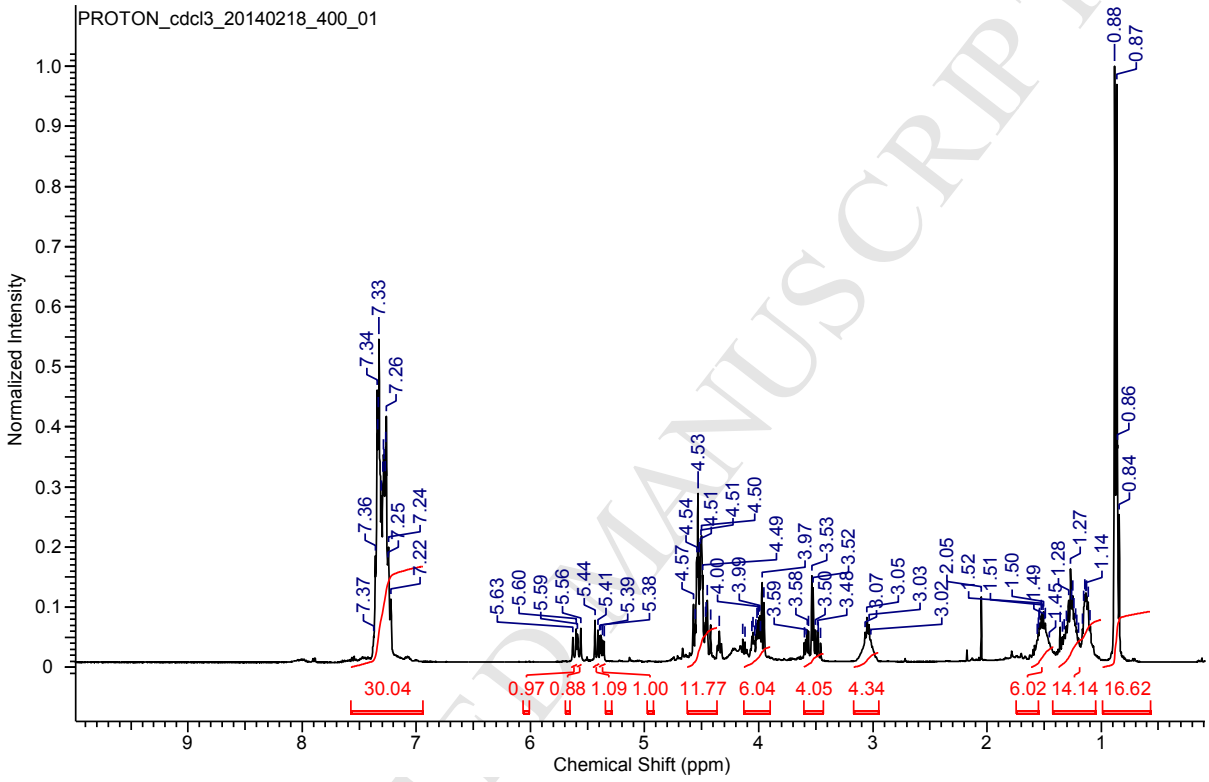


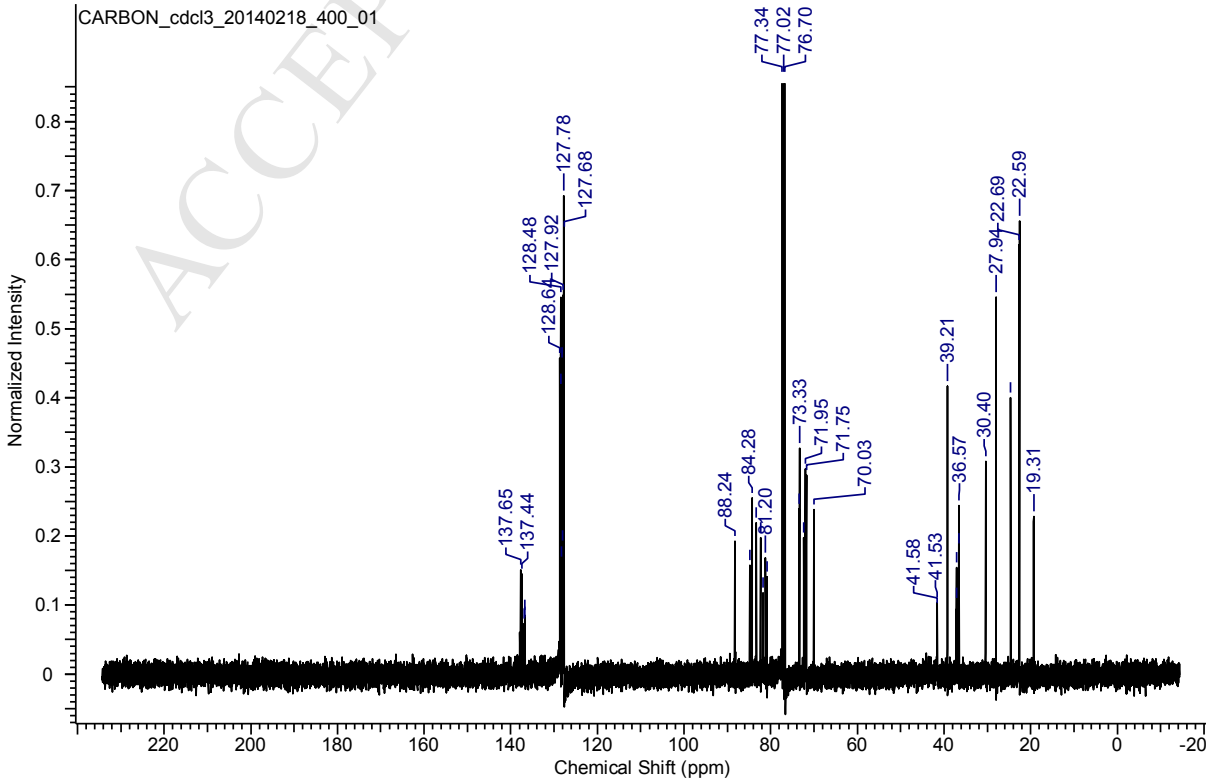




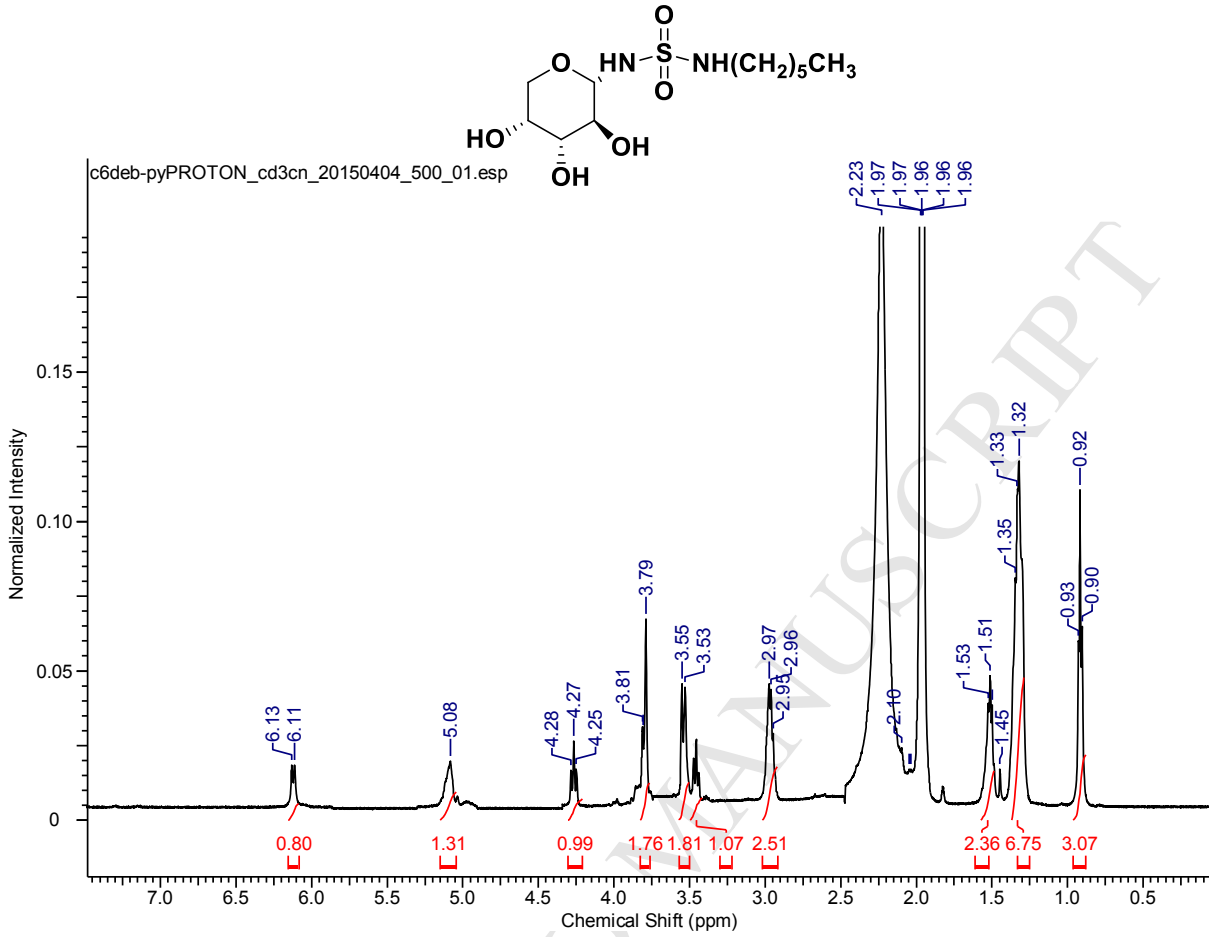
_N_-(3,7-dimethyloctyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 5j

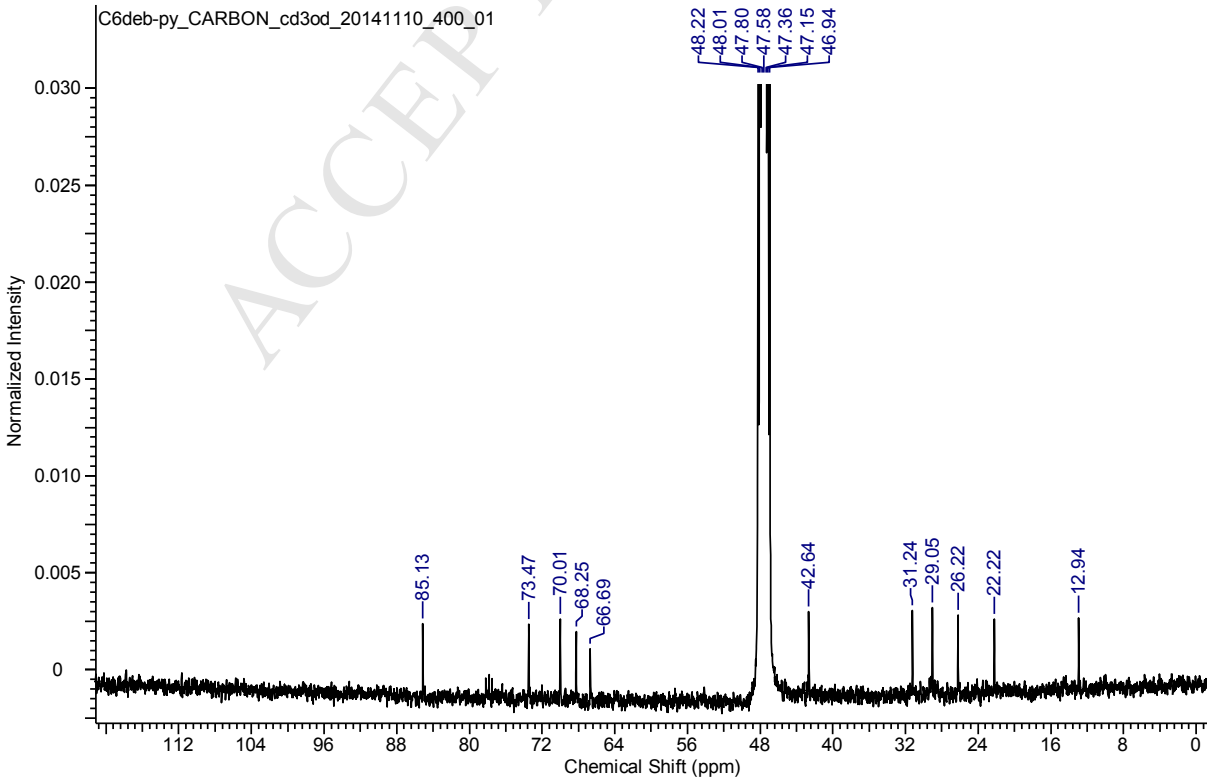




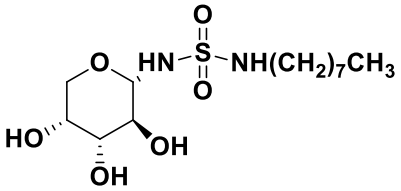


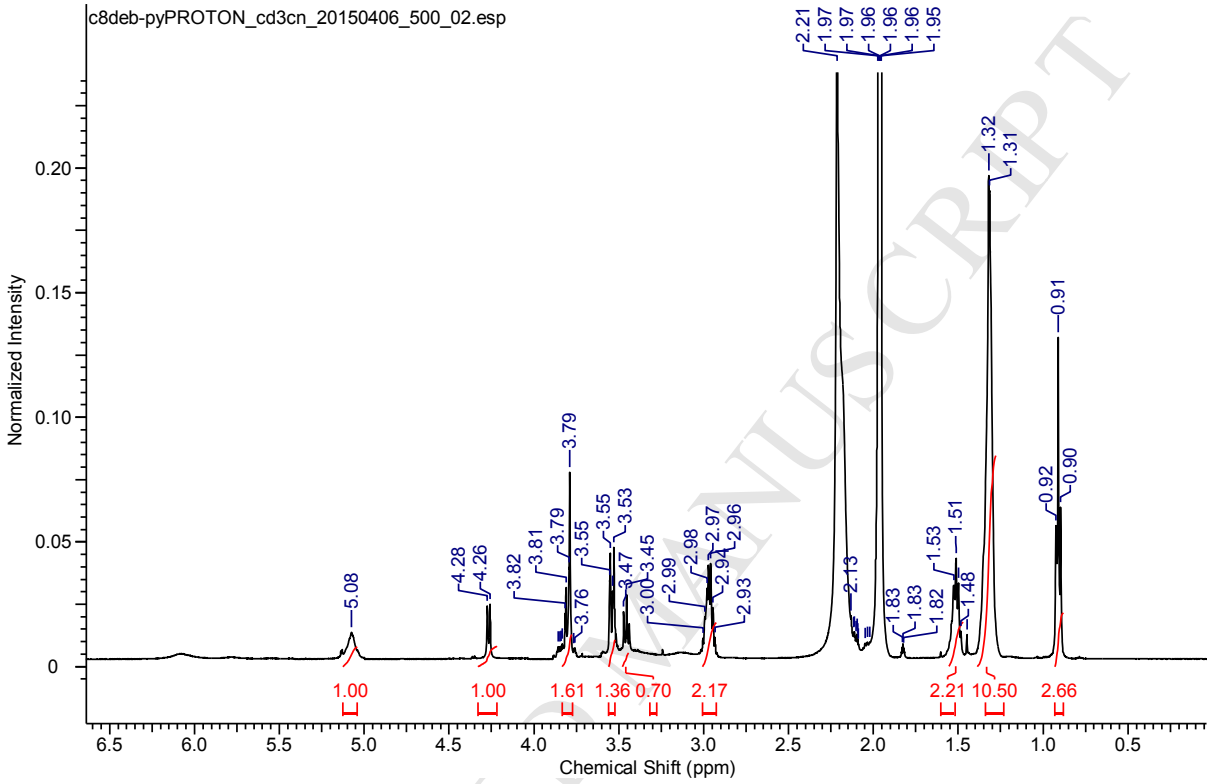
_N-(_Hexyl)-_N'_-(α-D-arabinopyranosyl)sulfamide 6a

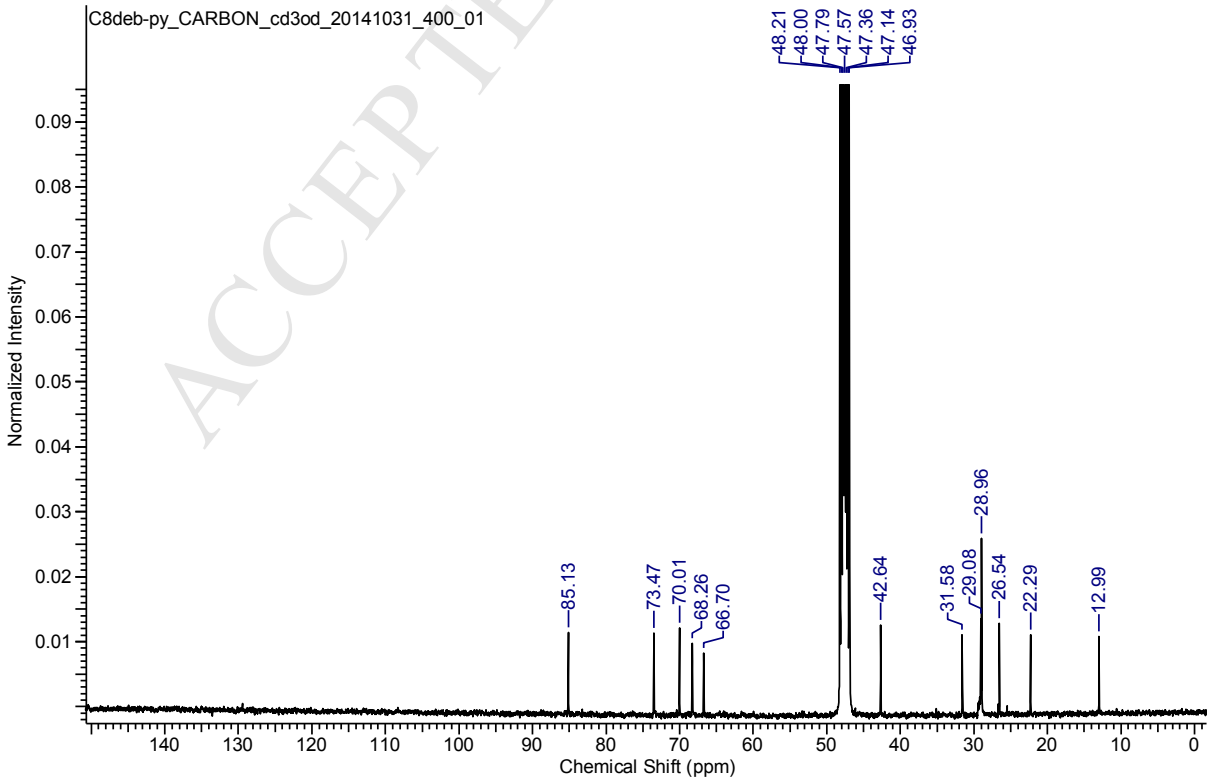




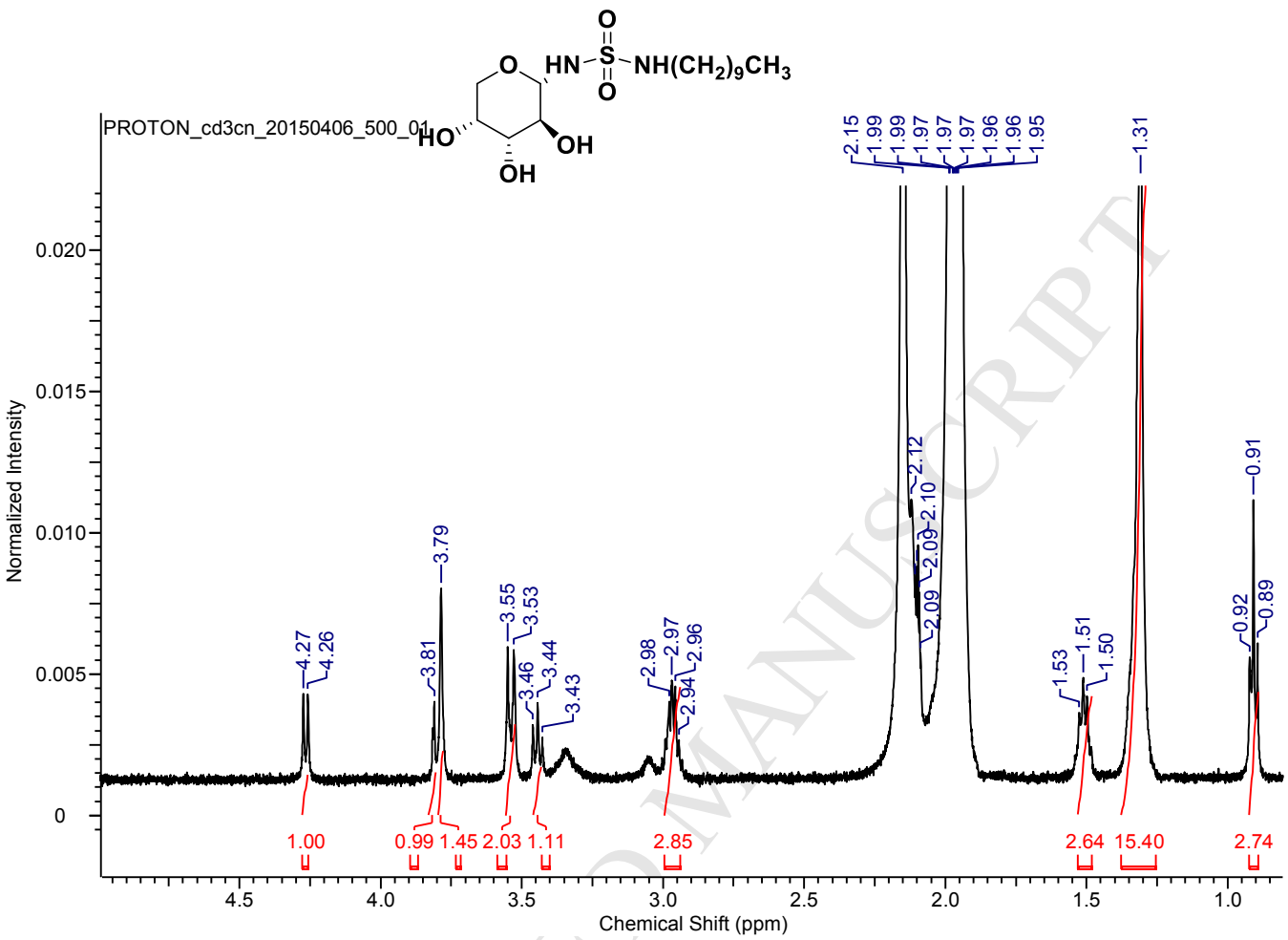
_N_-(Octyl)-_N'_-(α-D-arabinopyranosyl)sulfamide 6b

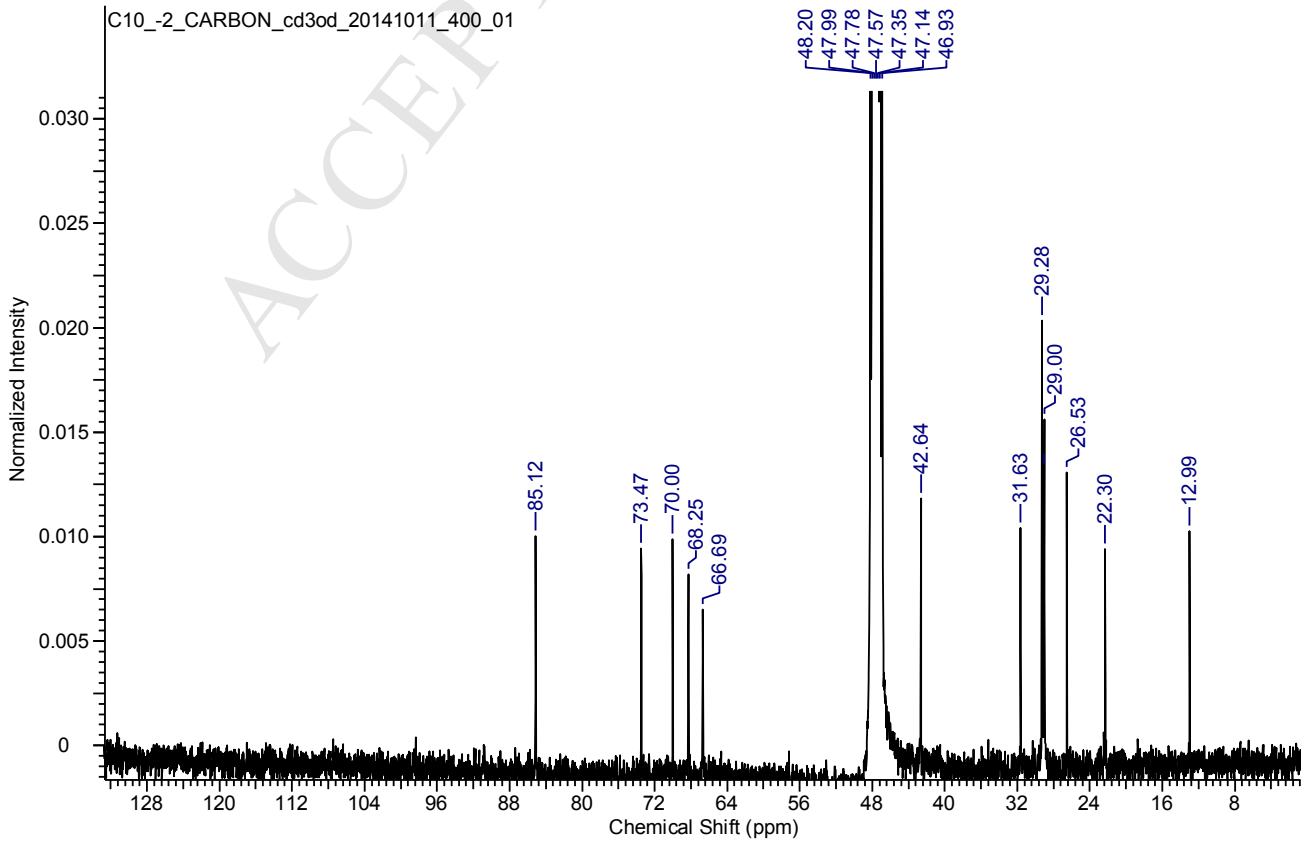




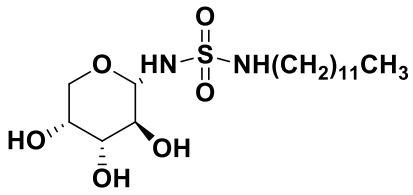


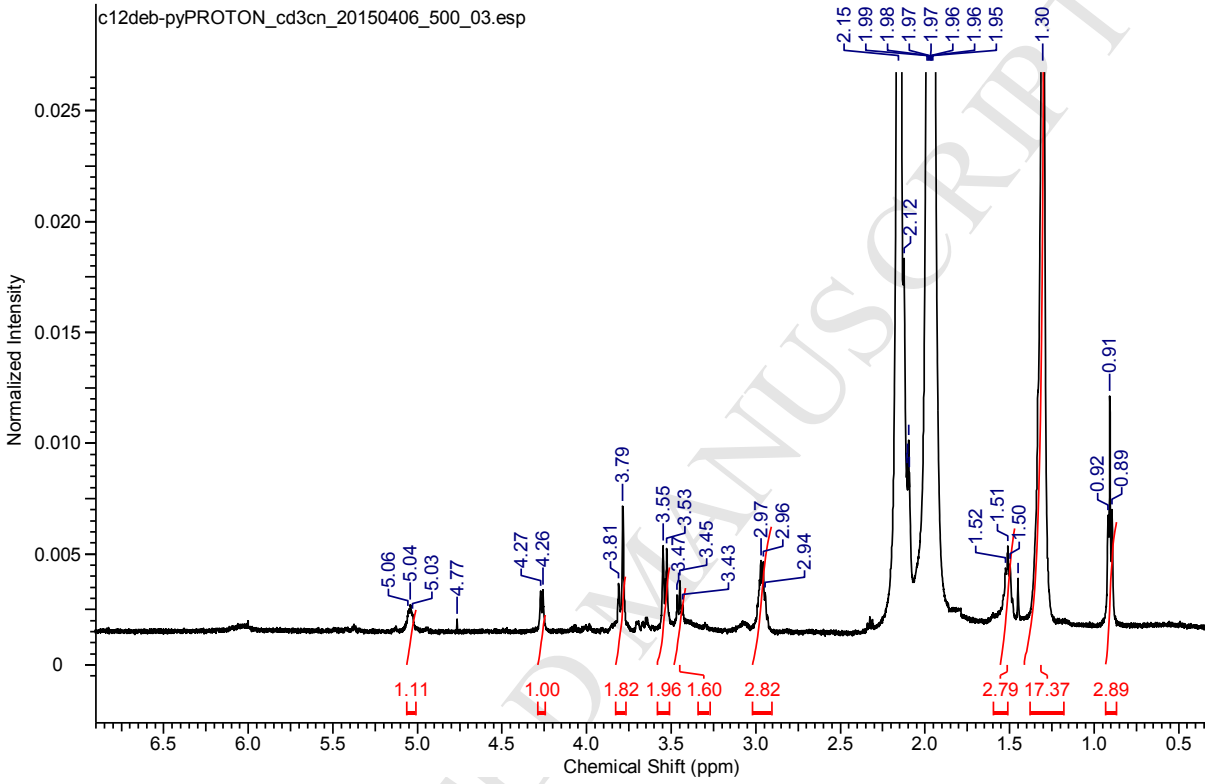
***N*-(Decyl)-*N*'-(α-D-arabinopyranosyl)sulfamide 6c**

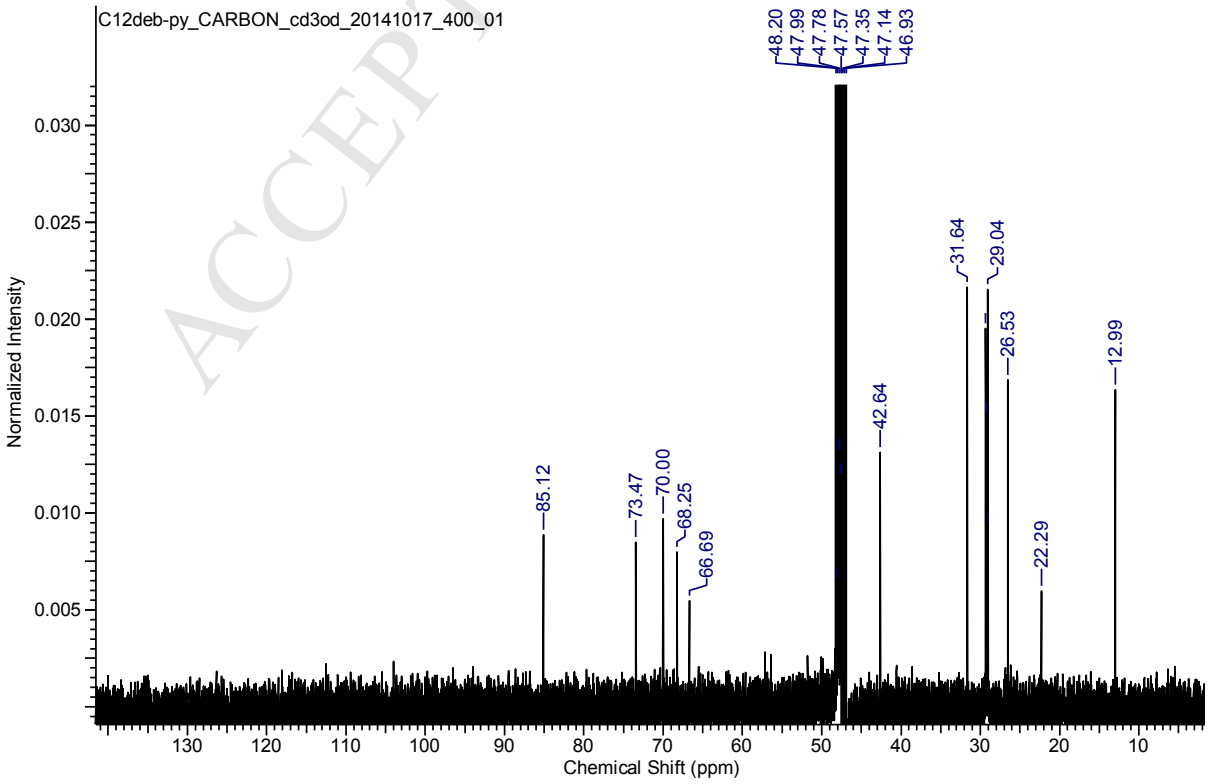




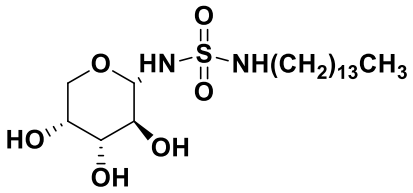
_N_-(Dodecyl)-_N'_-(α-D-arabinopyranosyl)sulfamide 6d

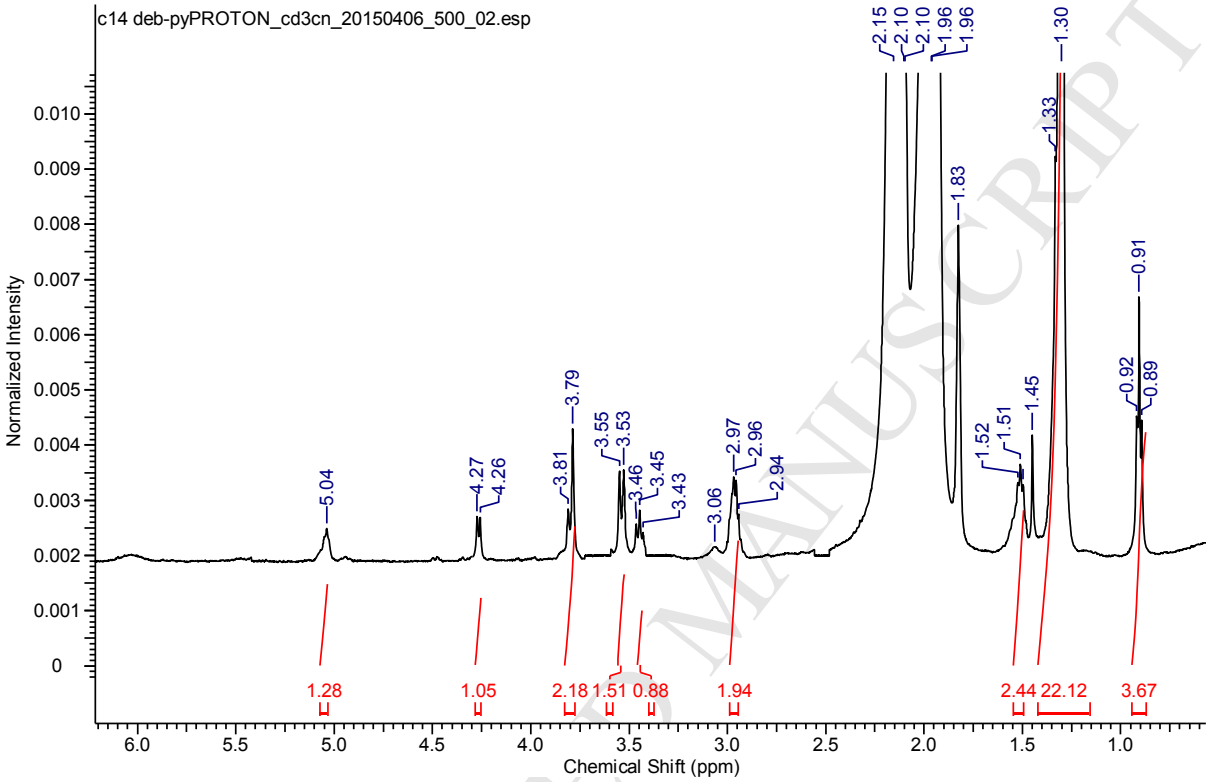


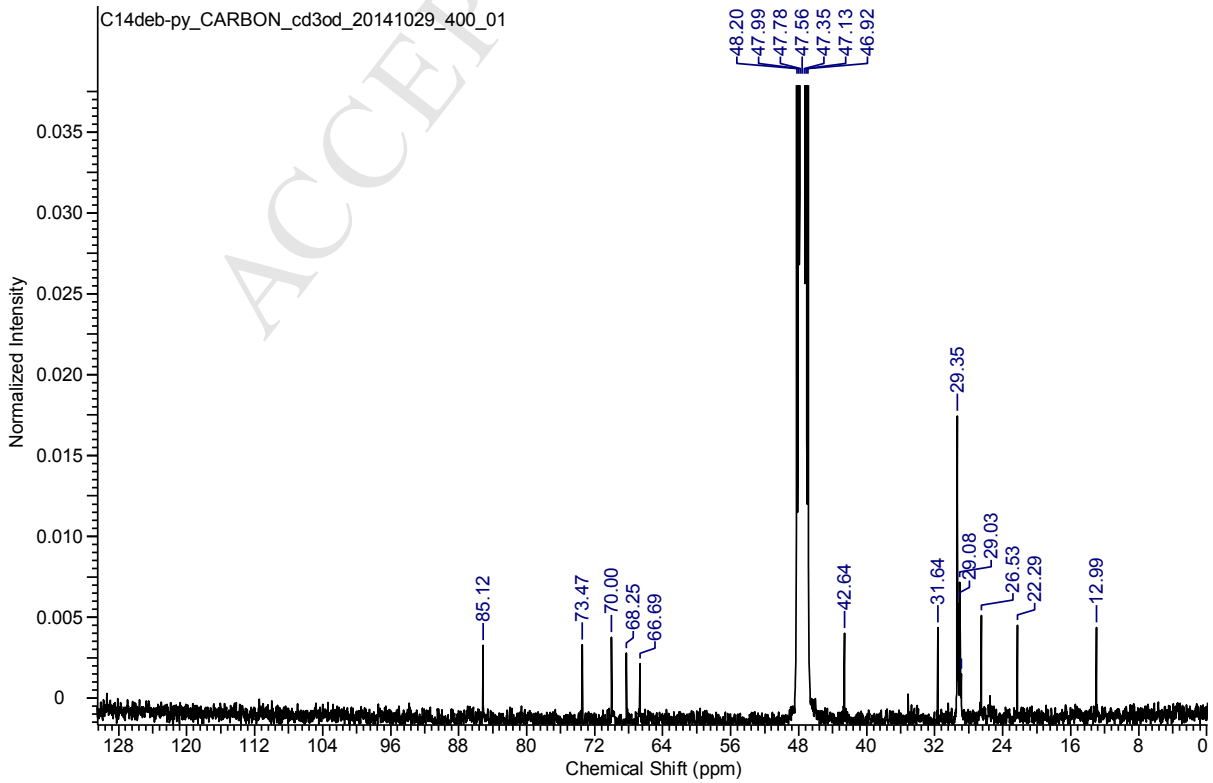




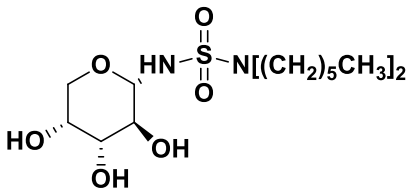
_N_-(Tetradecyl)-_N'_-(α-D-arabinopyranosyl)sulfamide 6e

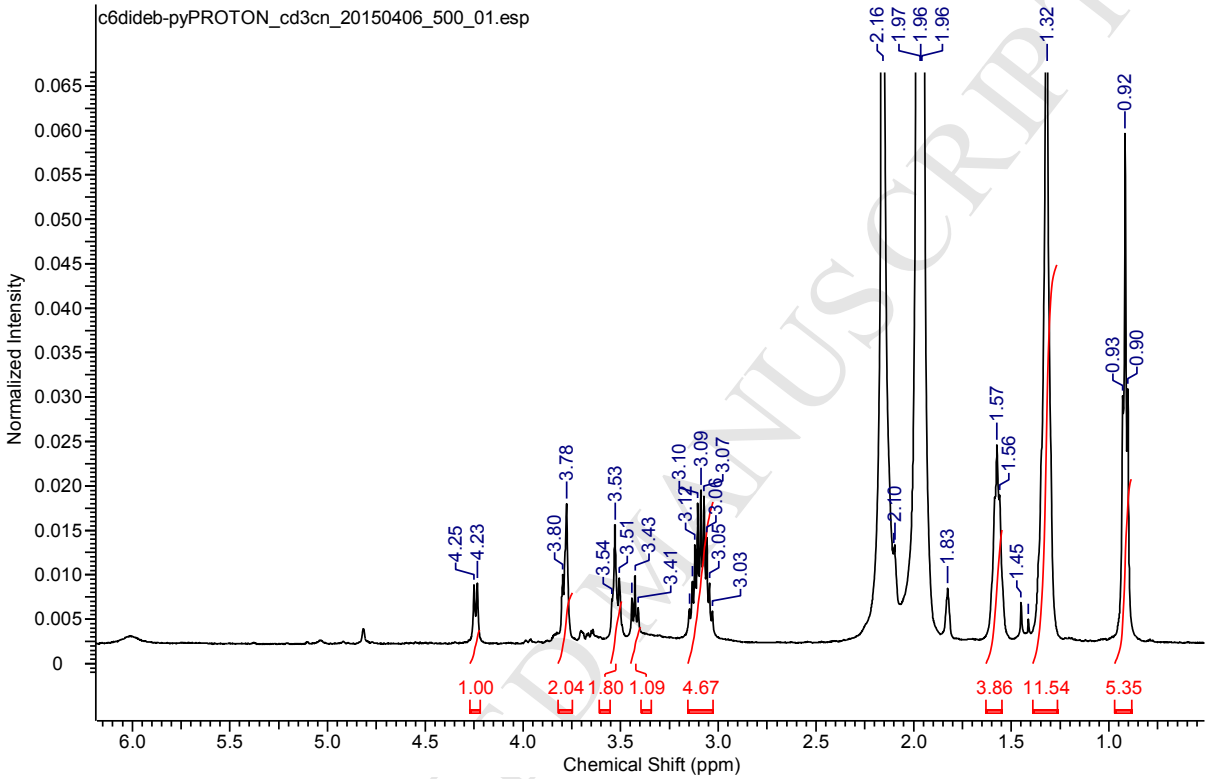


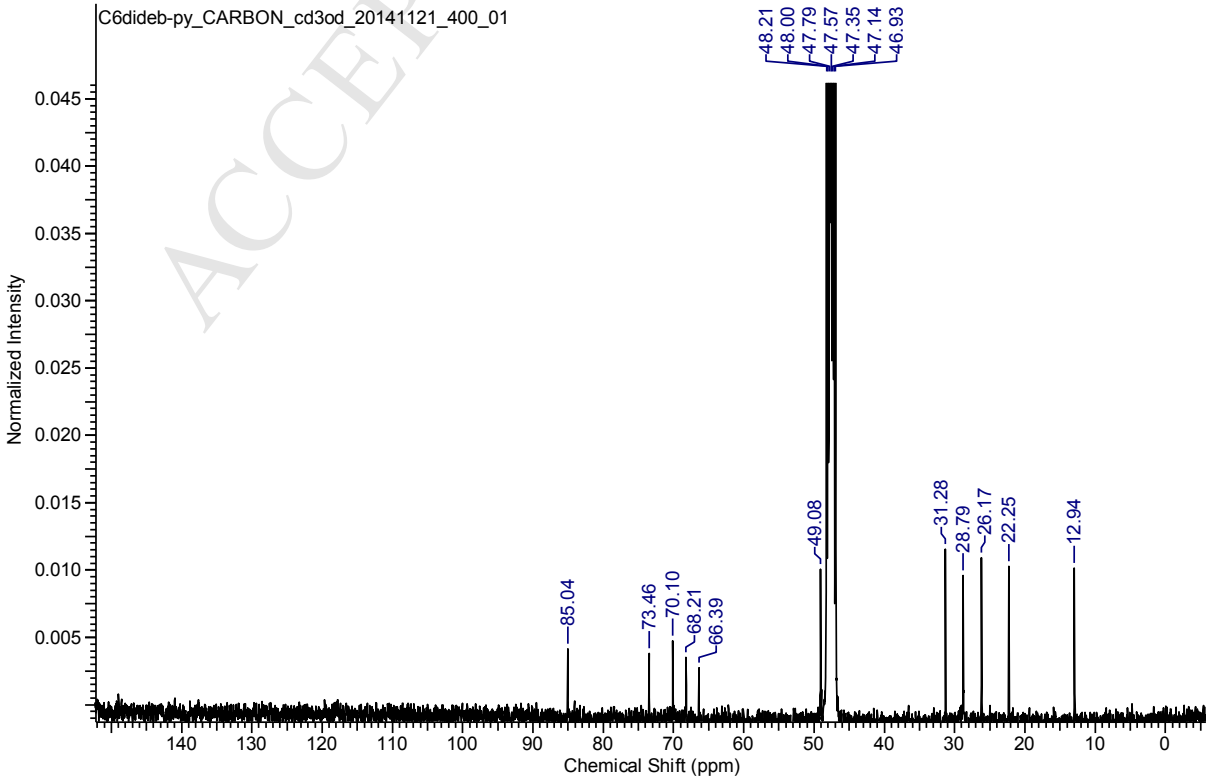




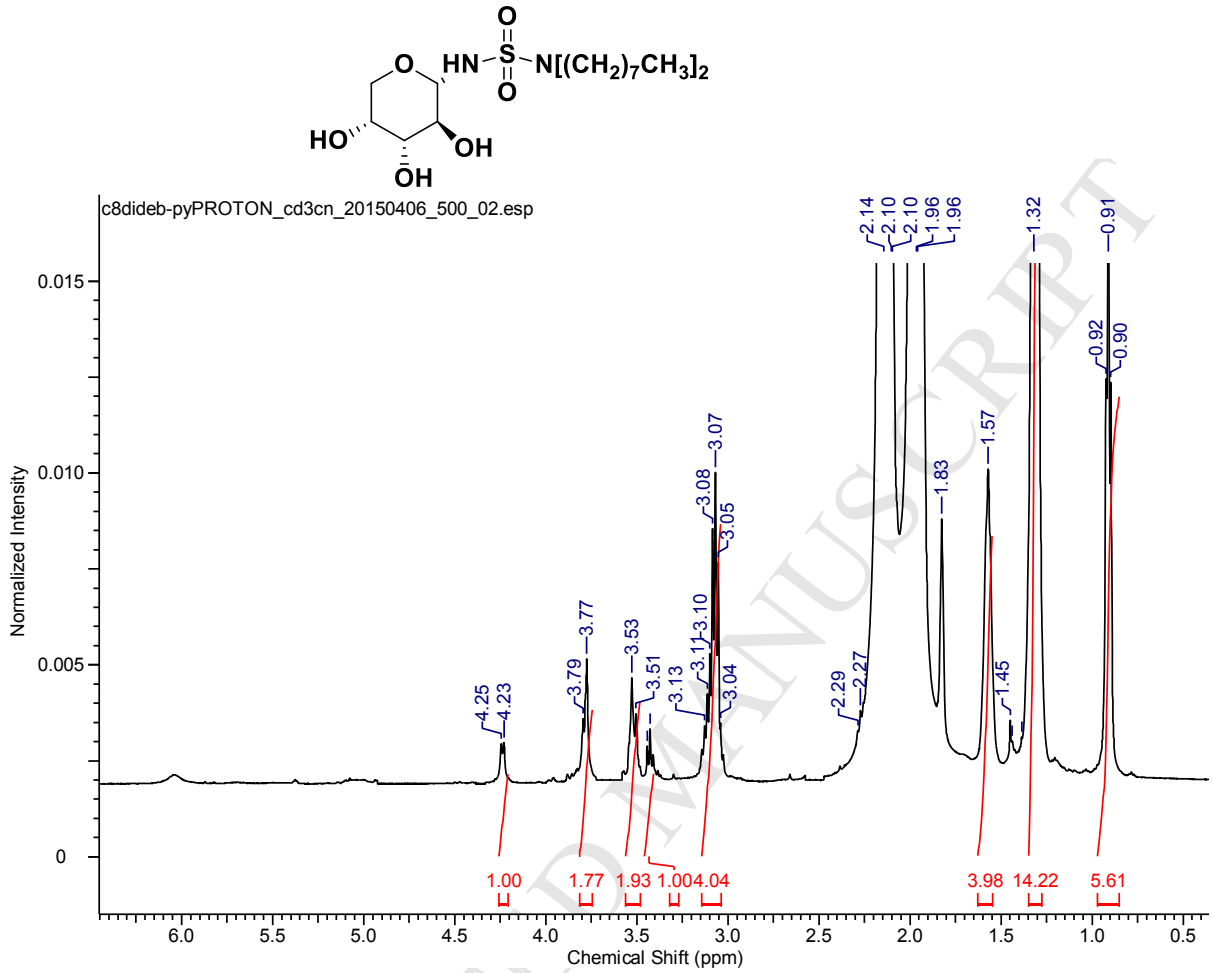
_N,N-(_Dihexyl)-_N'_-(α-D-arabinopyranosyl)sulfamide 6f

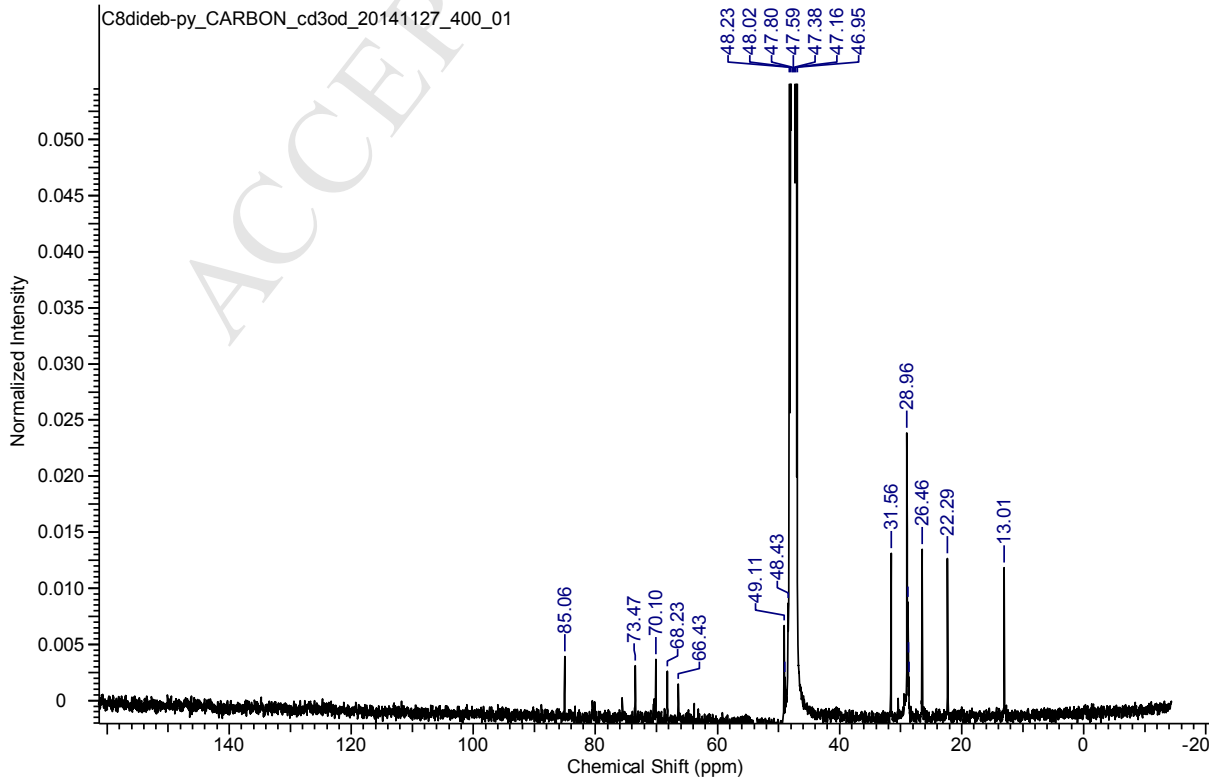




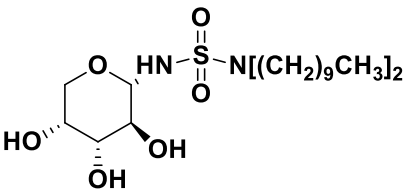


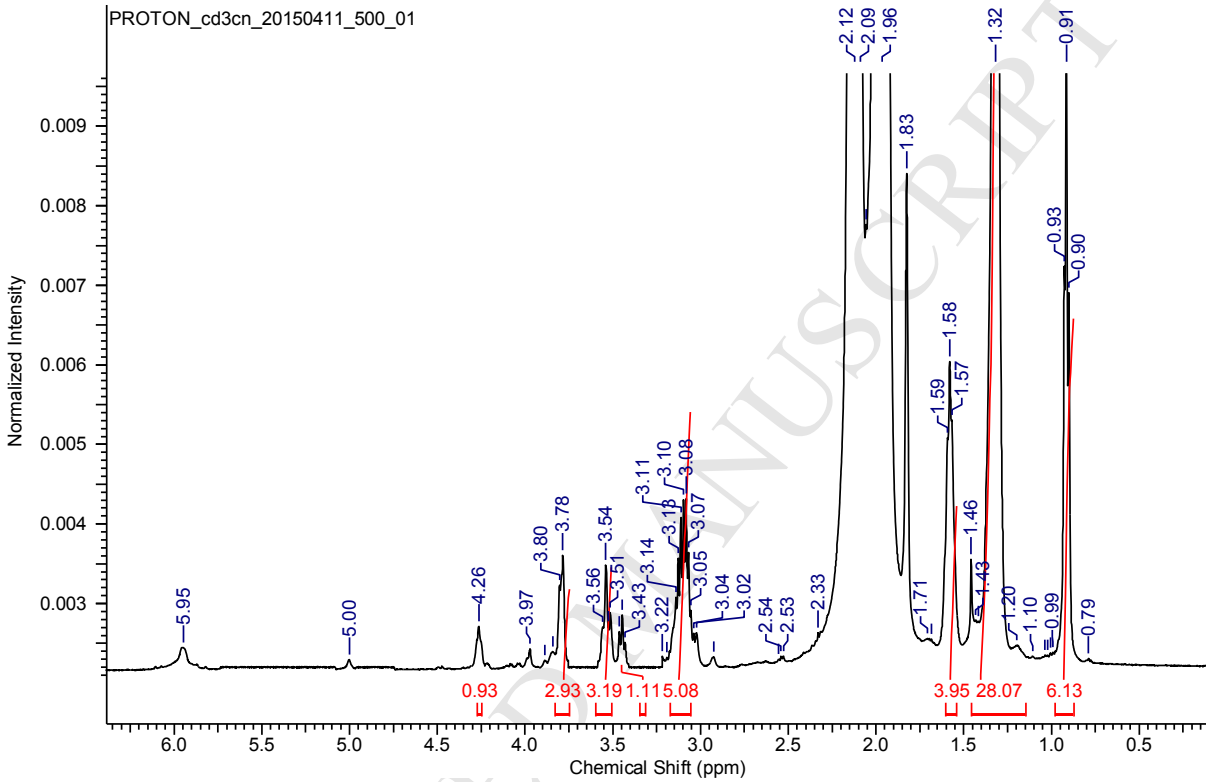
_N,N-(_Dioctyl)-_N'_-(α-D-arabinopyranosyl)sulfamide 6g

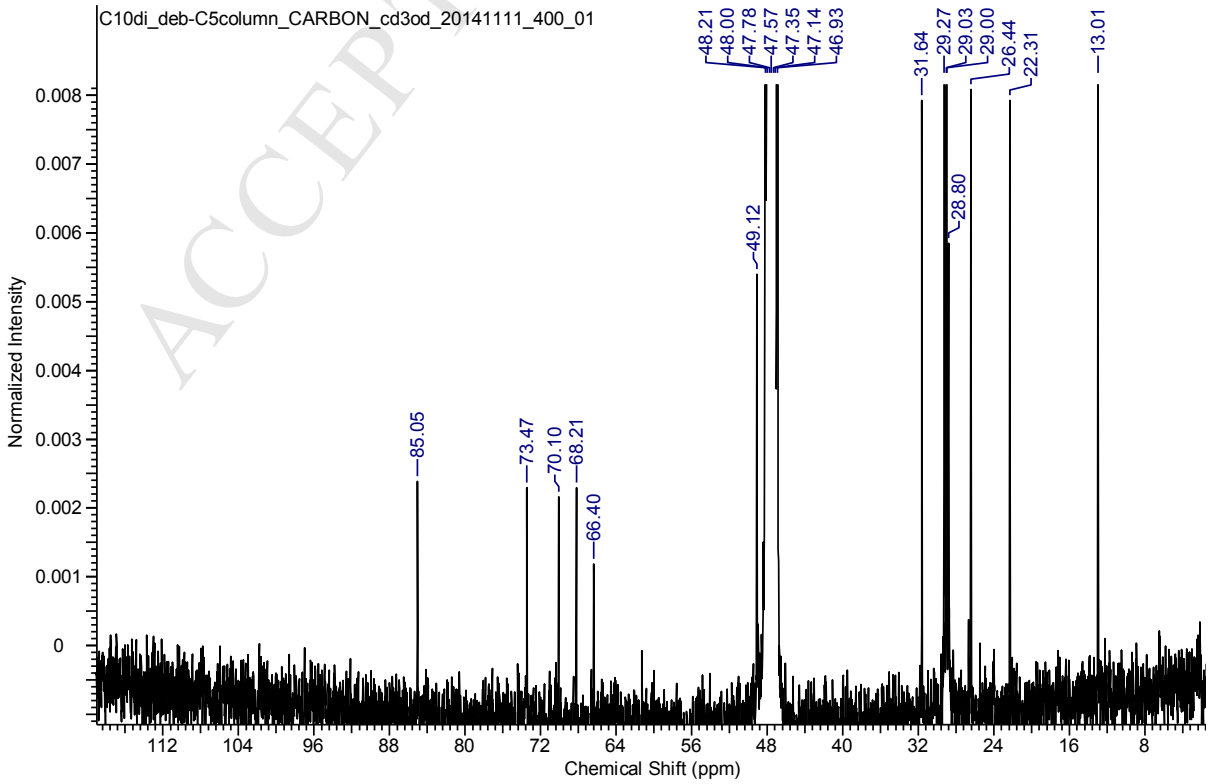




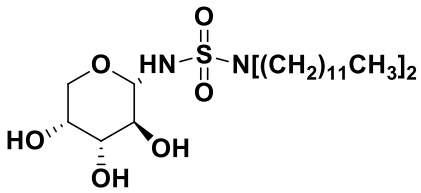
_N,N-(_Didecyl)-_N'_-(α-D-arabinopyranosyl)sulfamide 6h

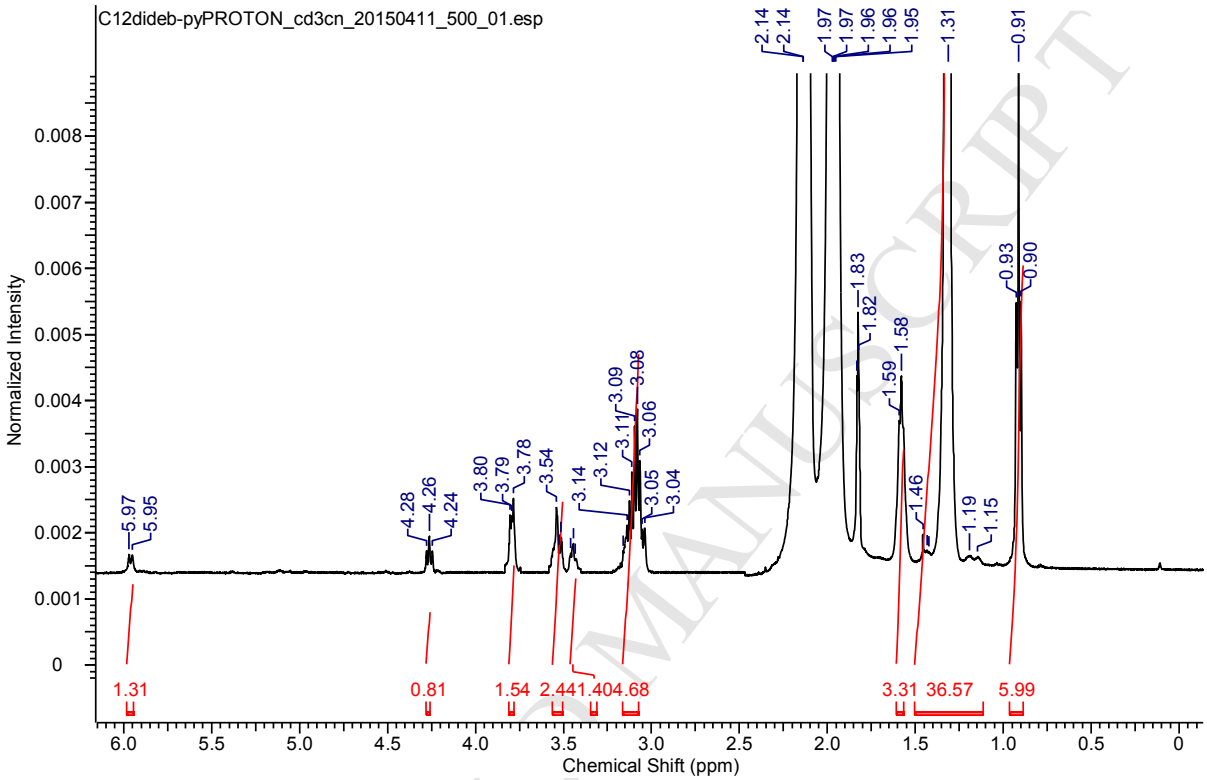


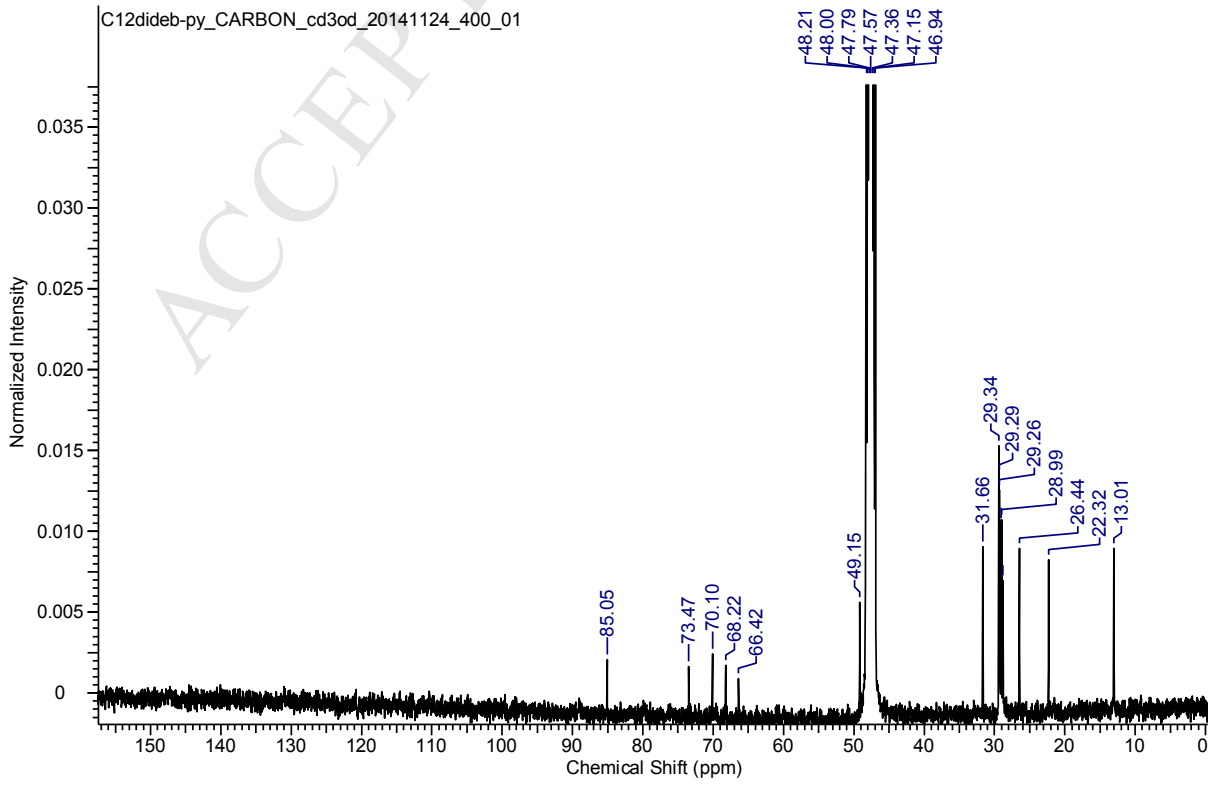




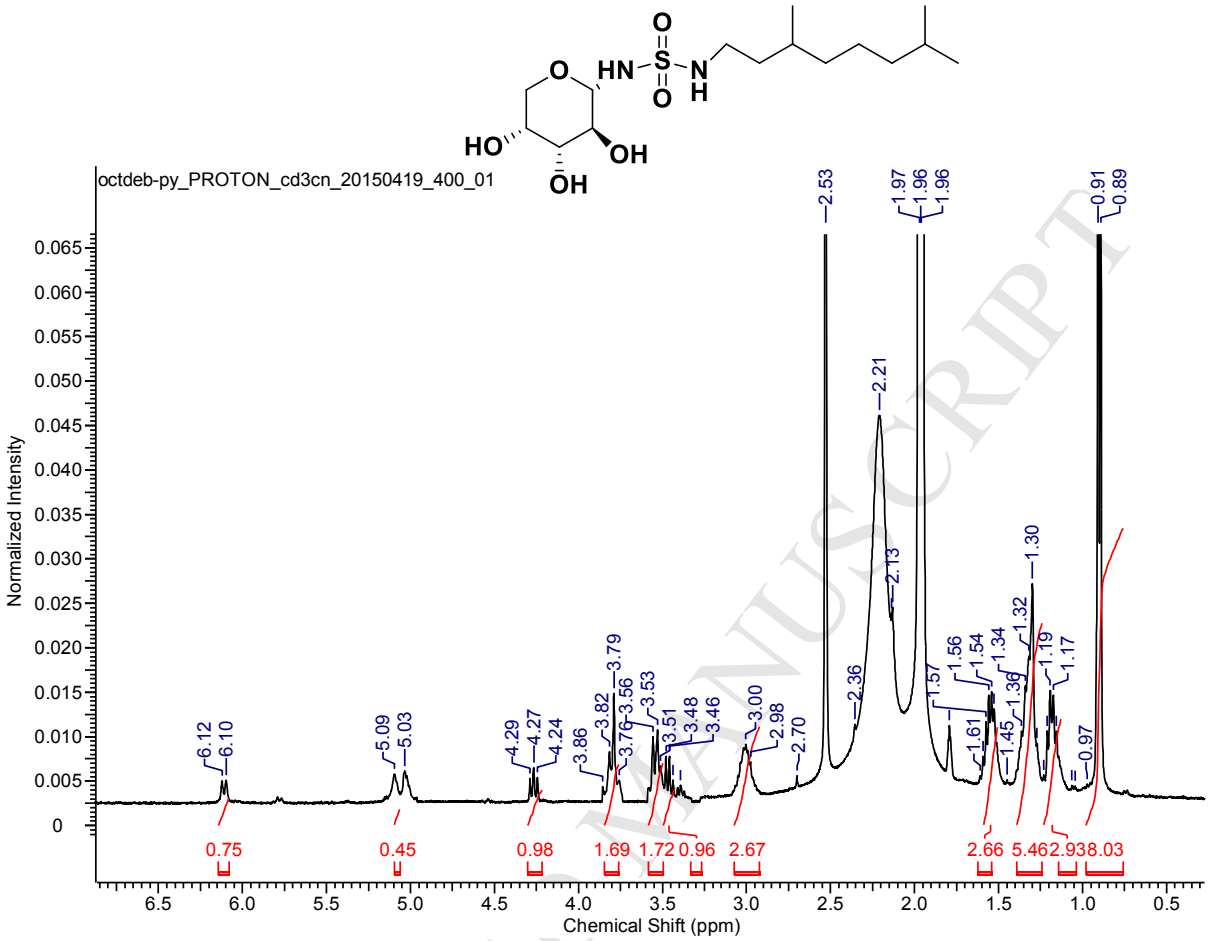
_N,N-(_Didodecyl)-_N'_-(α-D-arabinopyranosyl)sulfamide 6i

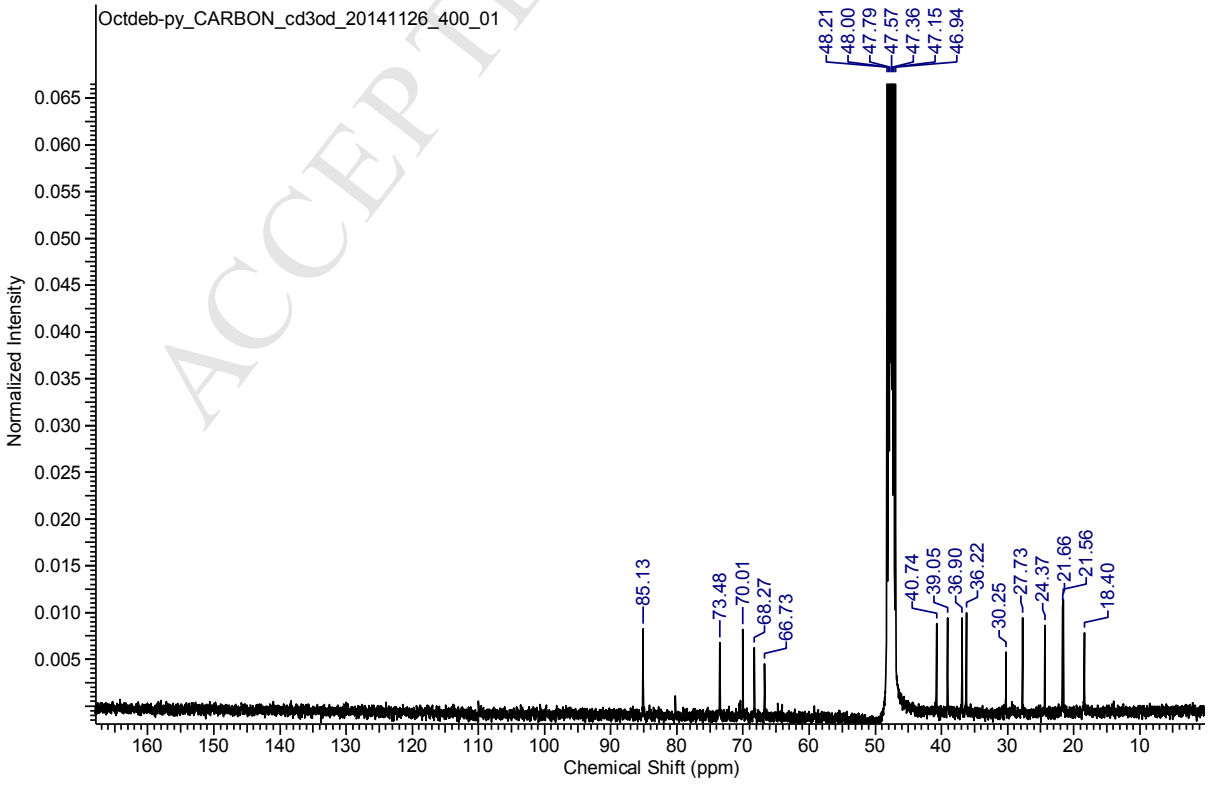




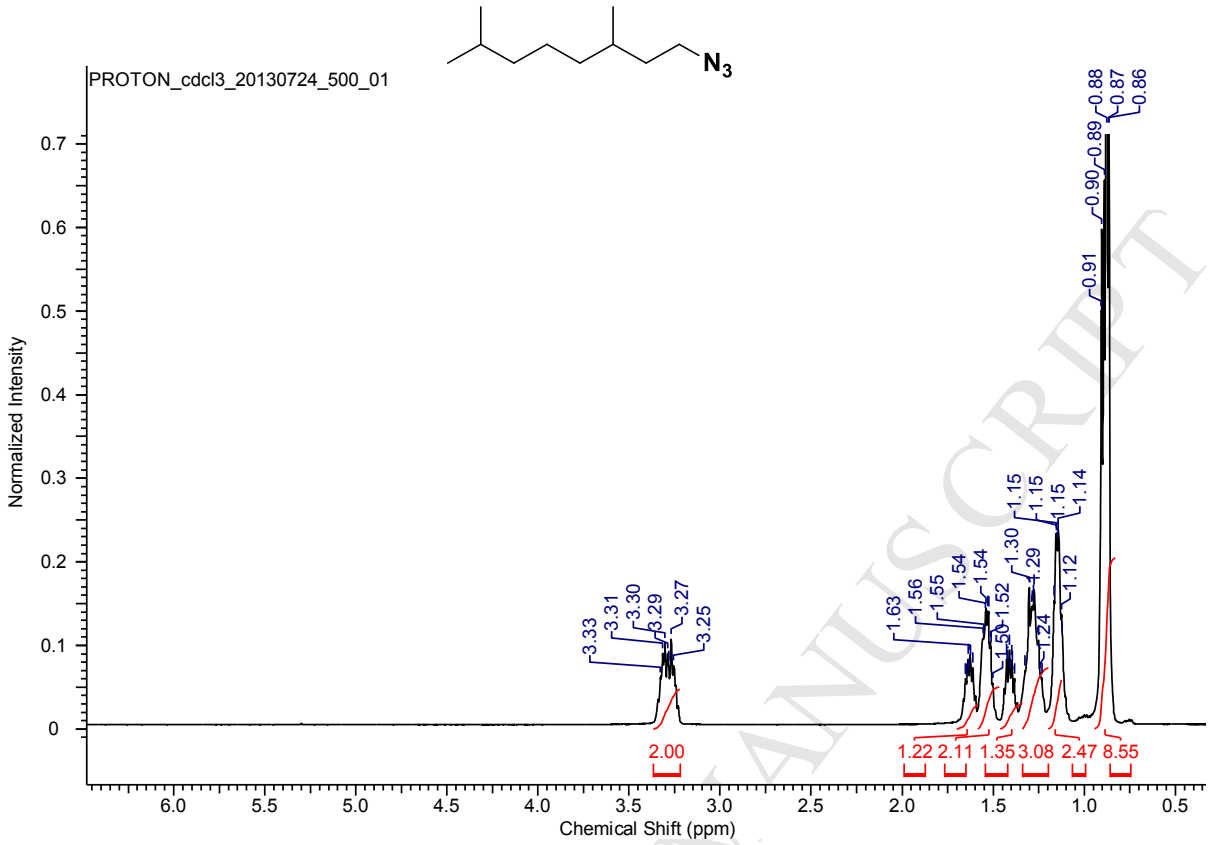


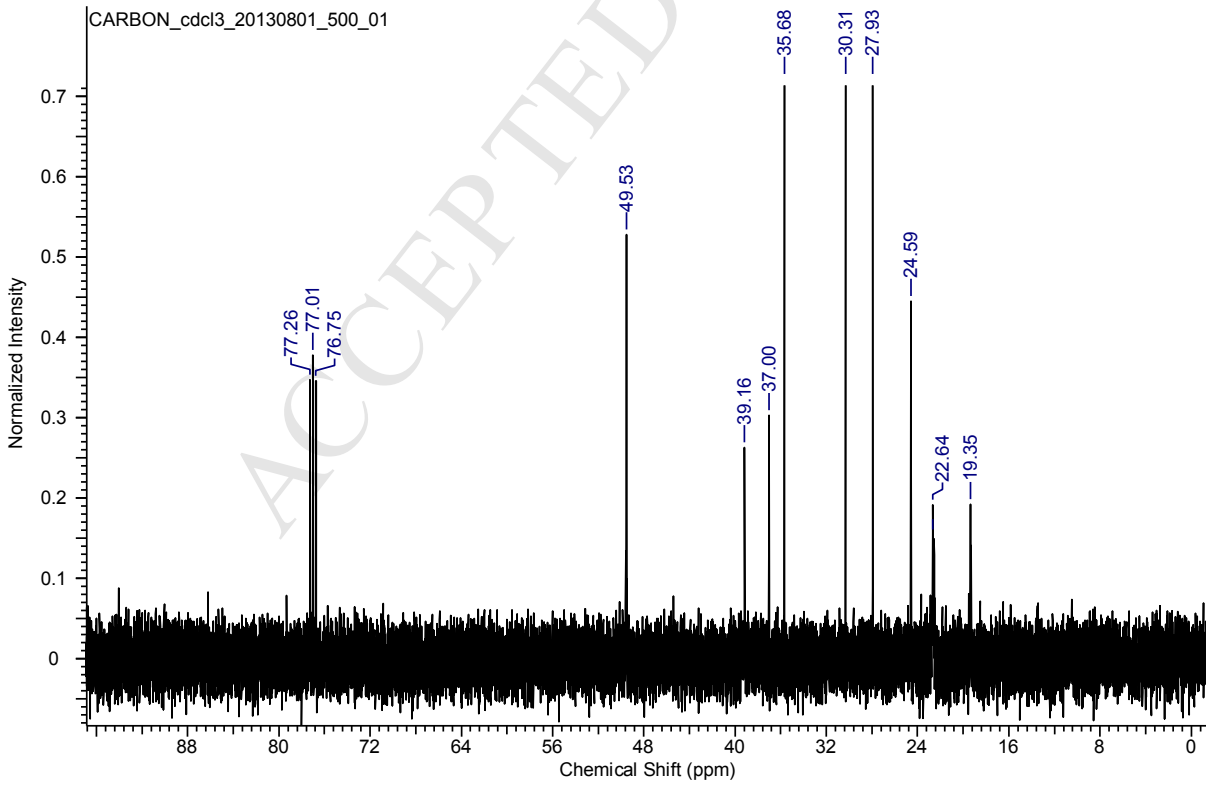
_N_-(3,7-Dimethyloctyl)-_N_'-(α-D-arabinopyranosyl)sulfamide 6j



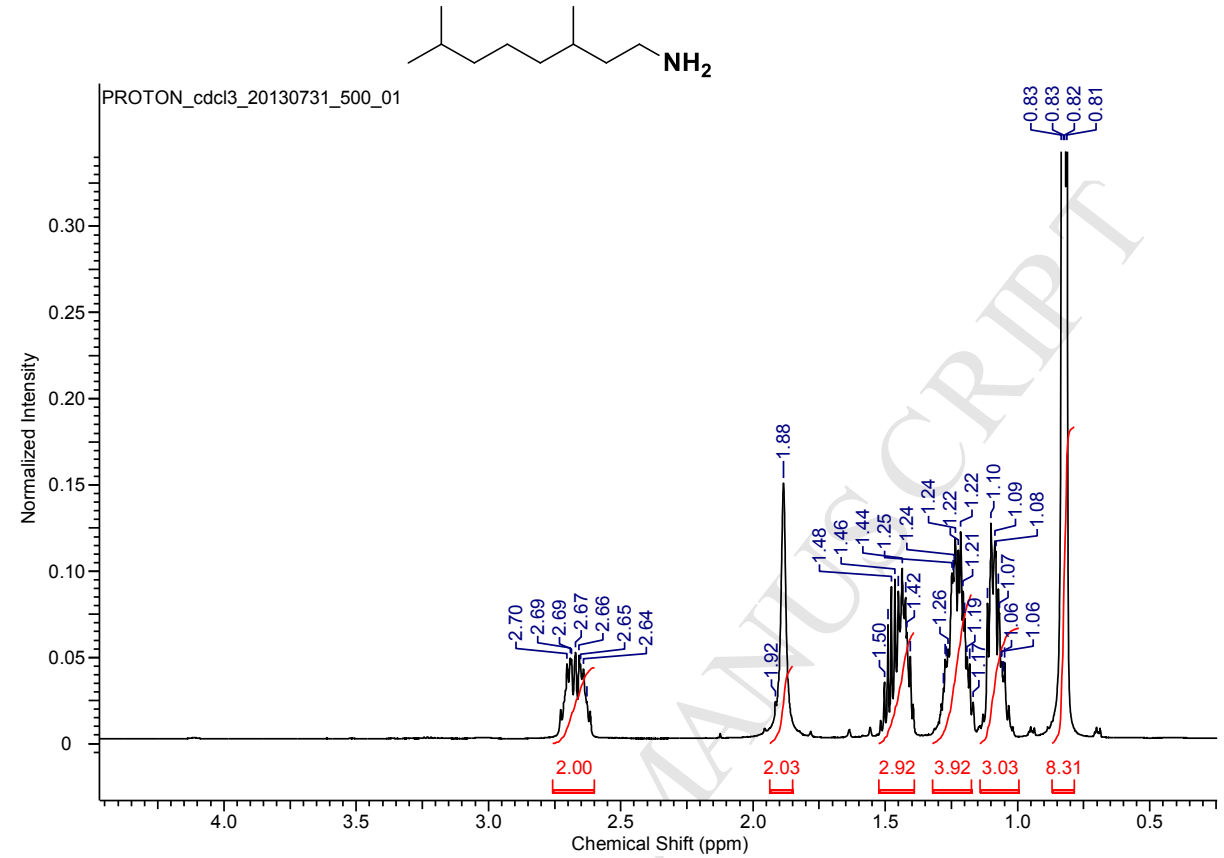


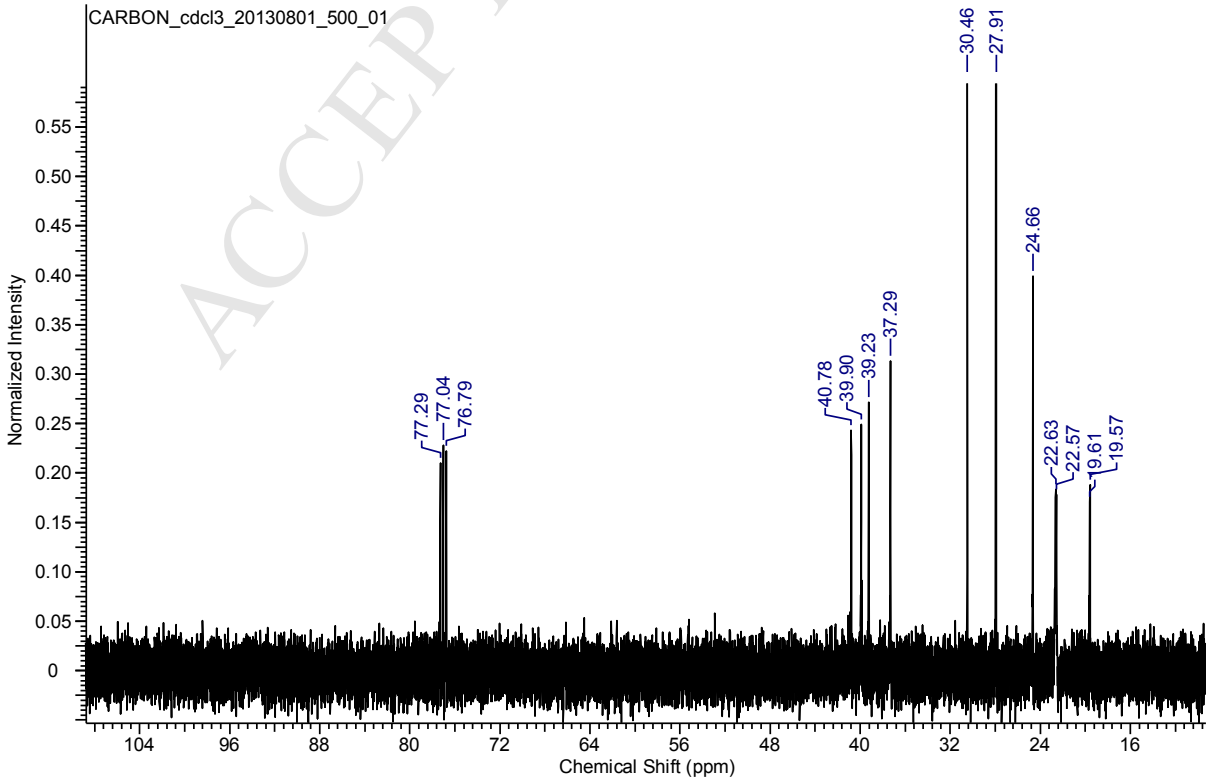
1-Azido-3,7-dimethyloctane 8



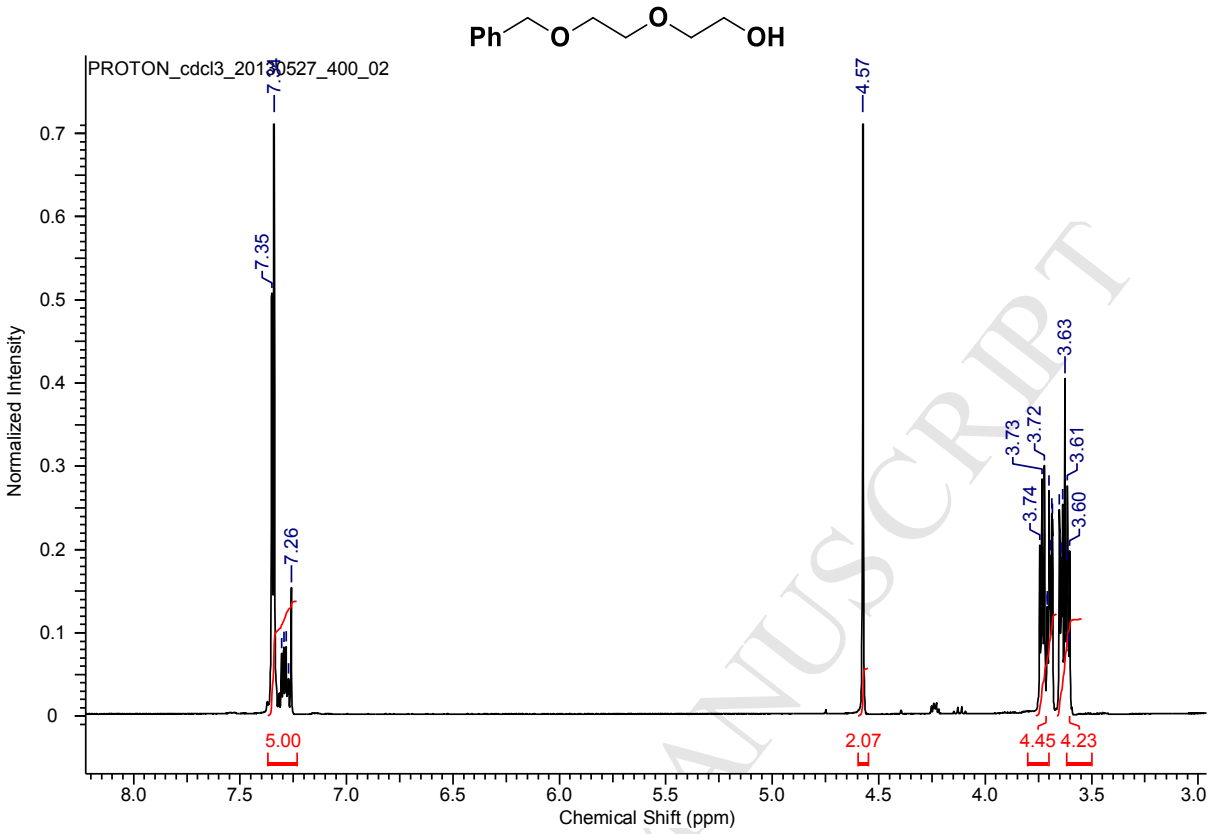


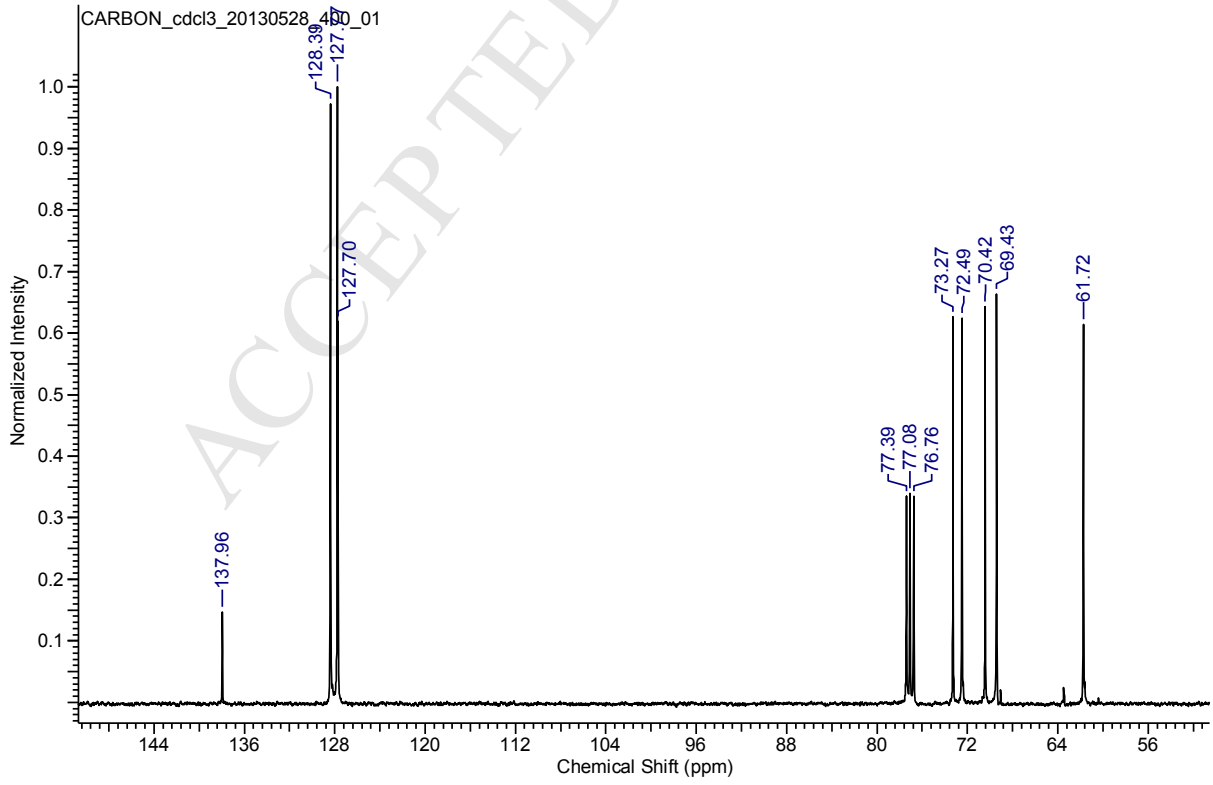
3,7-Dimethyl-1-octanamine 9



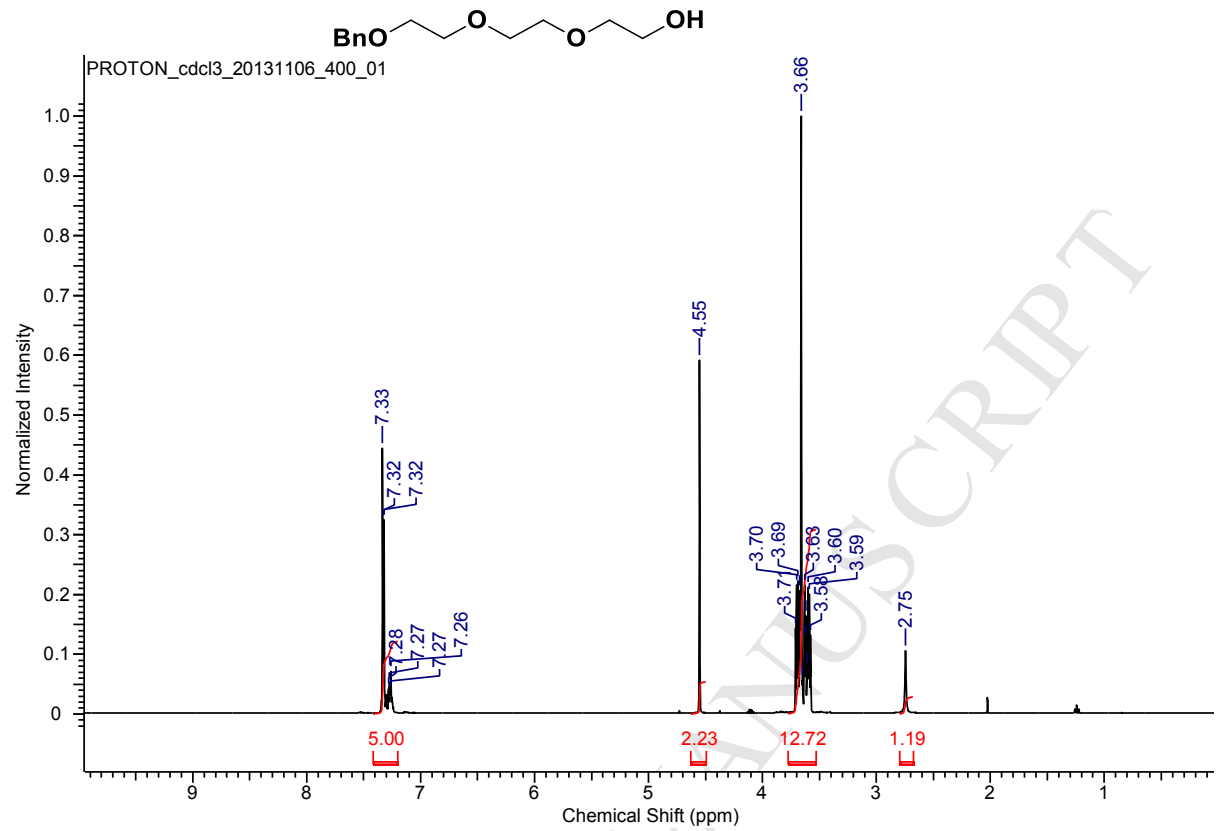


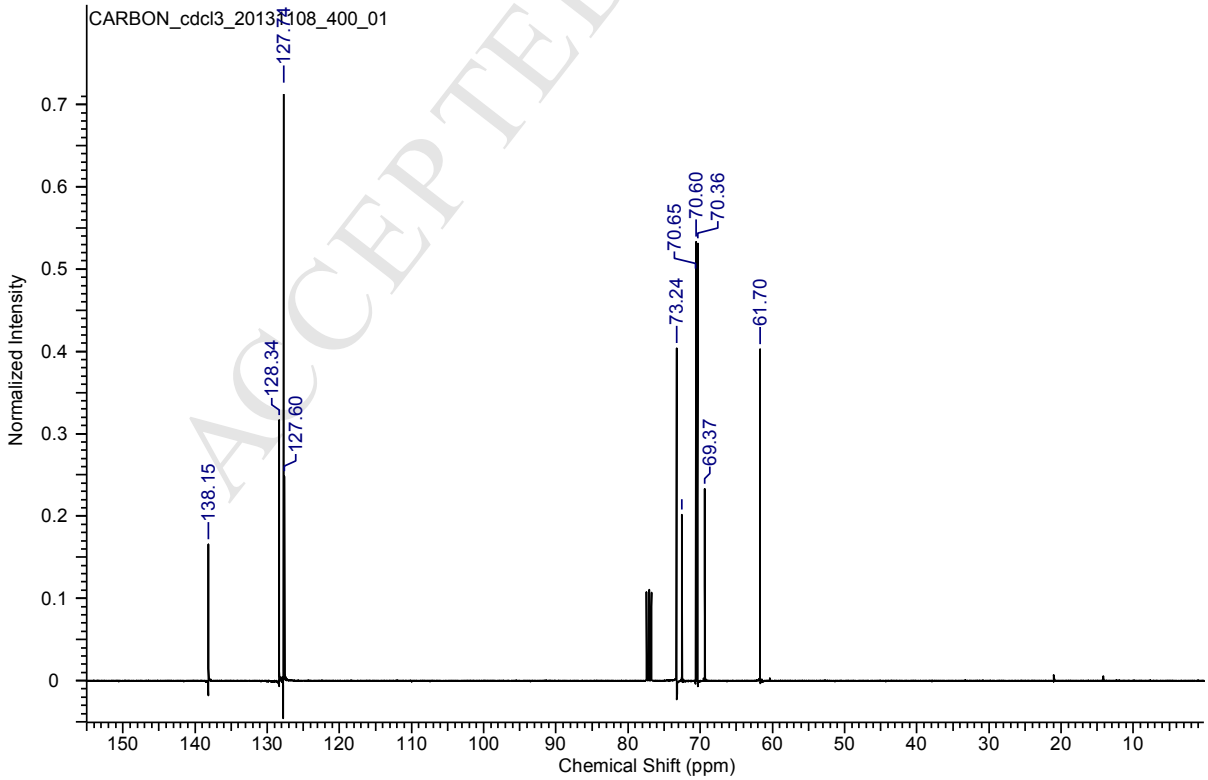
2-(2-(Benzyloxy)ethoxy)ethanol 11a



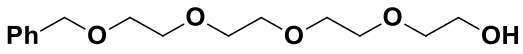


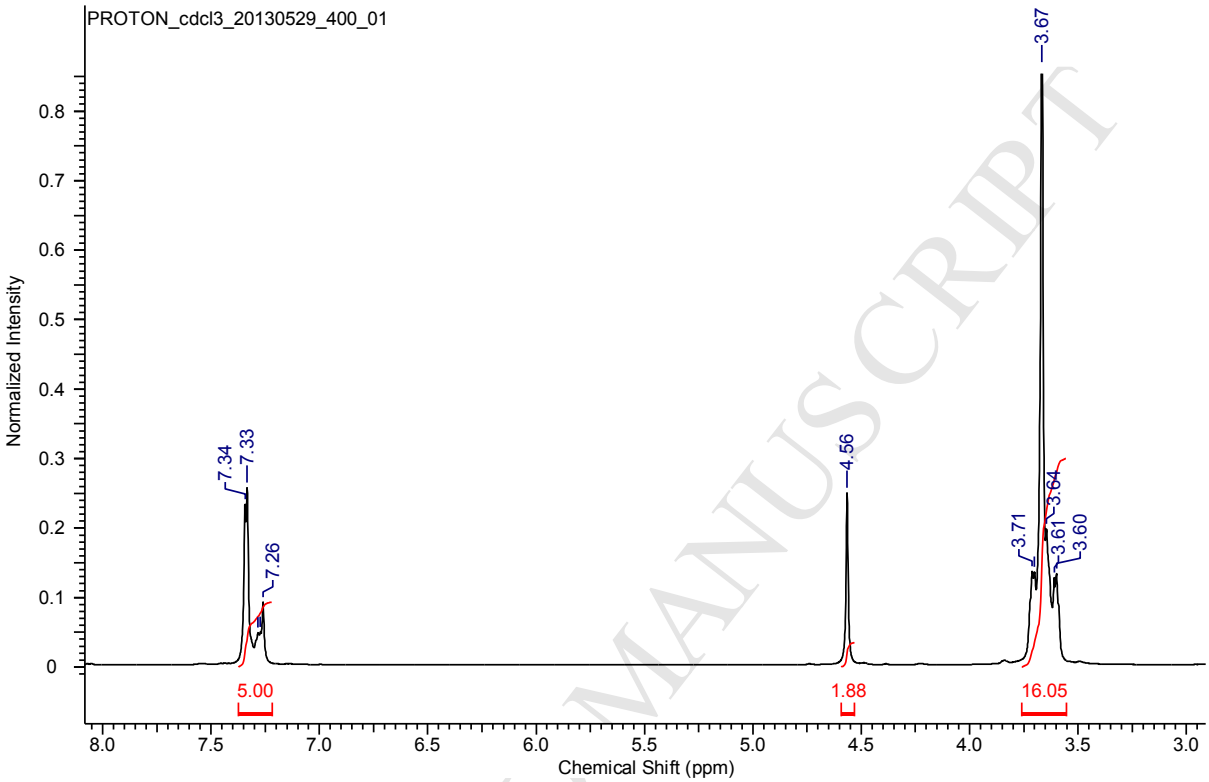
2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethanol 11b

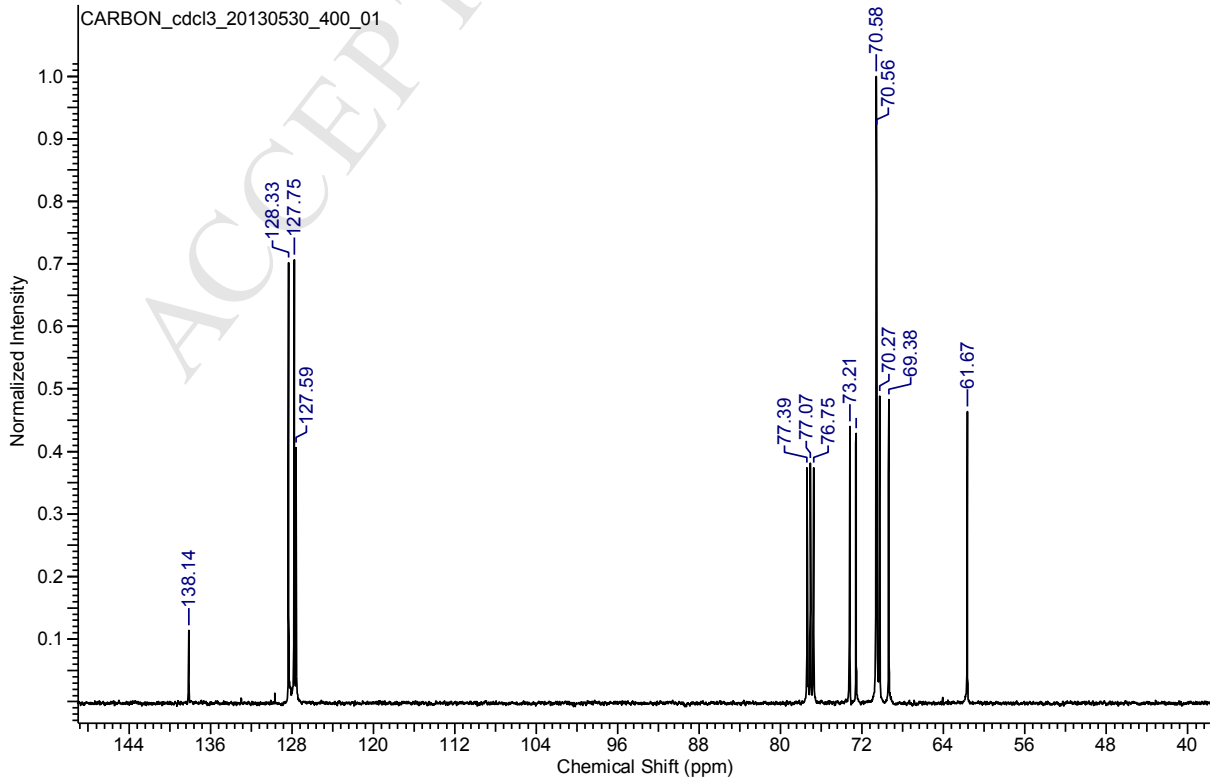




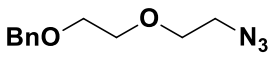
2-(2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethoxy)ethanol 11c

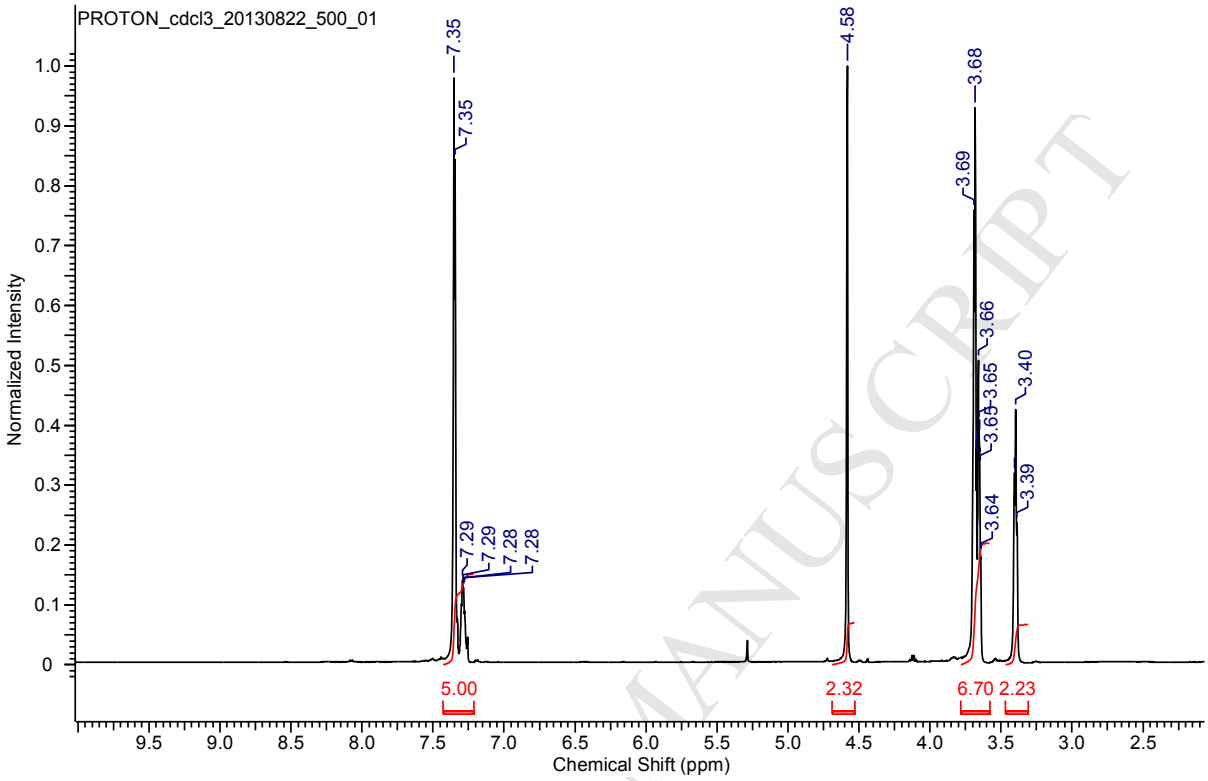


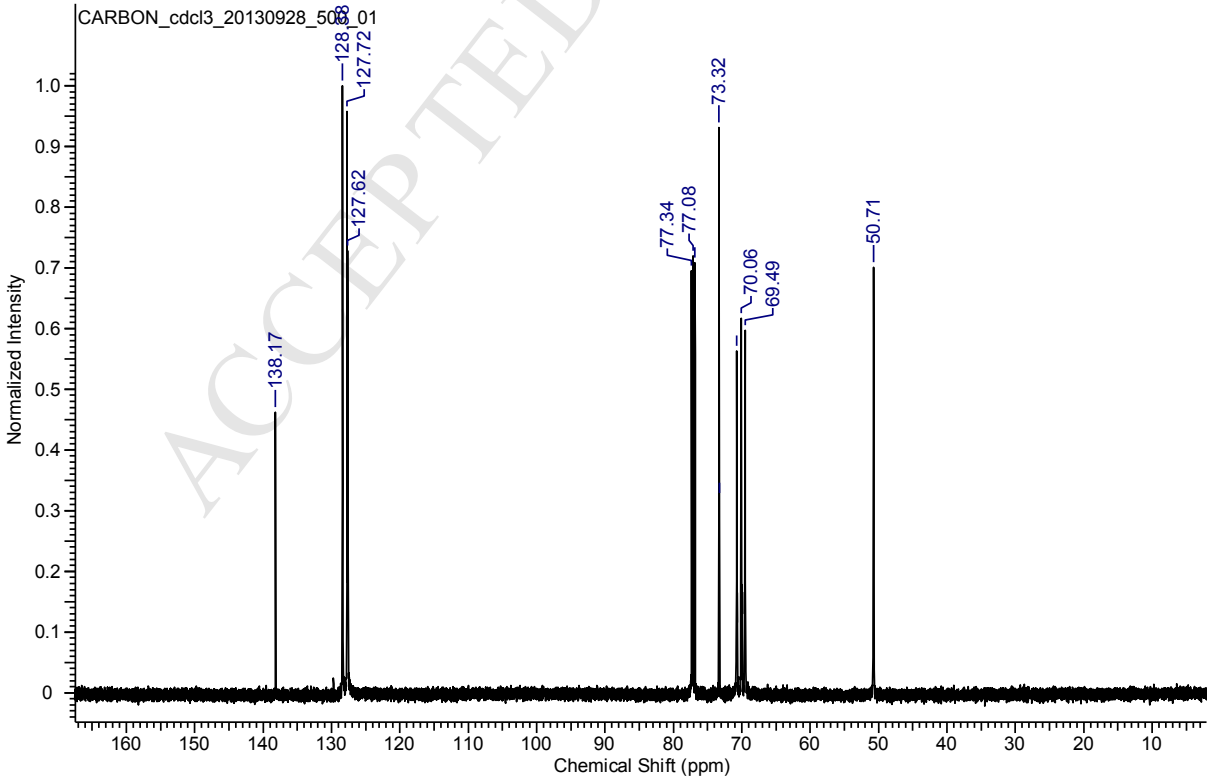




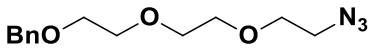
((2-(2-Azidoethoxy)ethoxy)methyl)benzene 12a

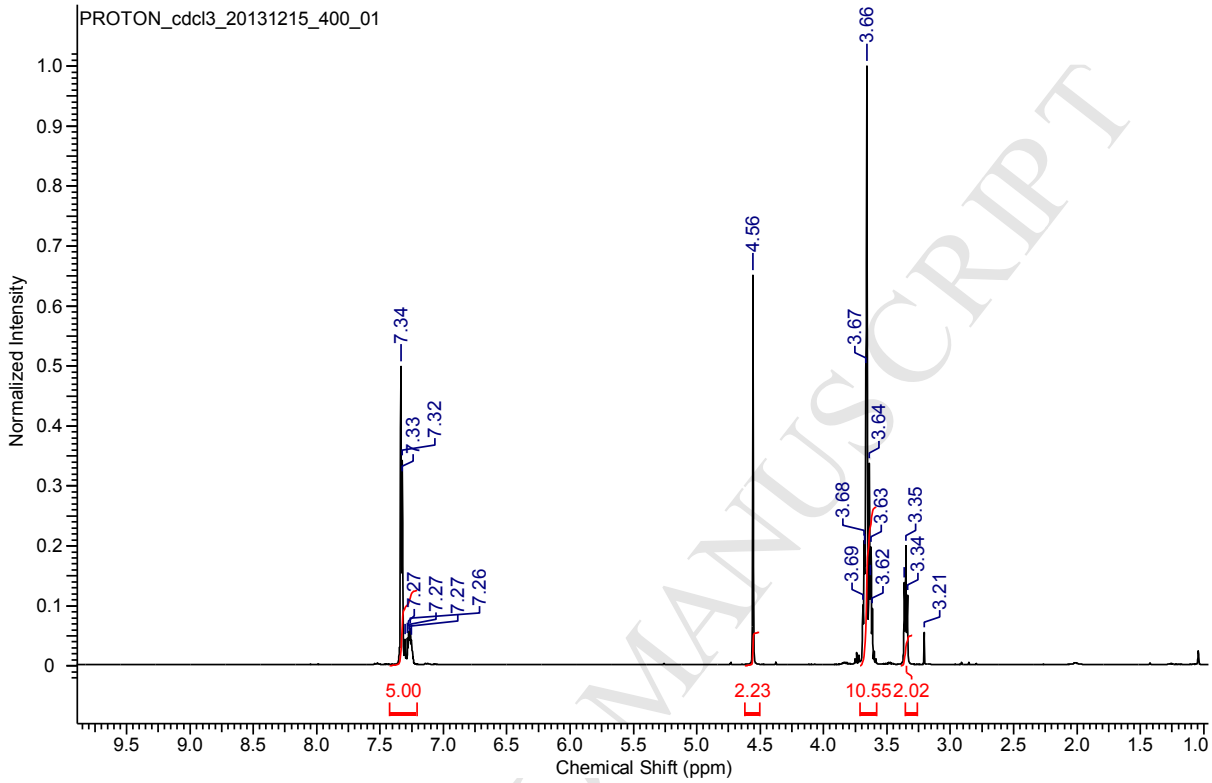


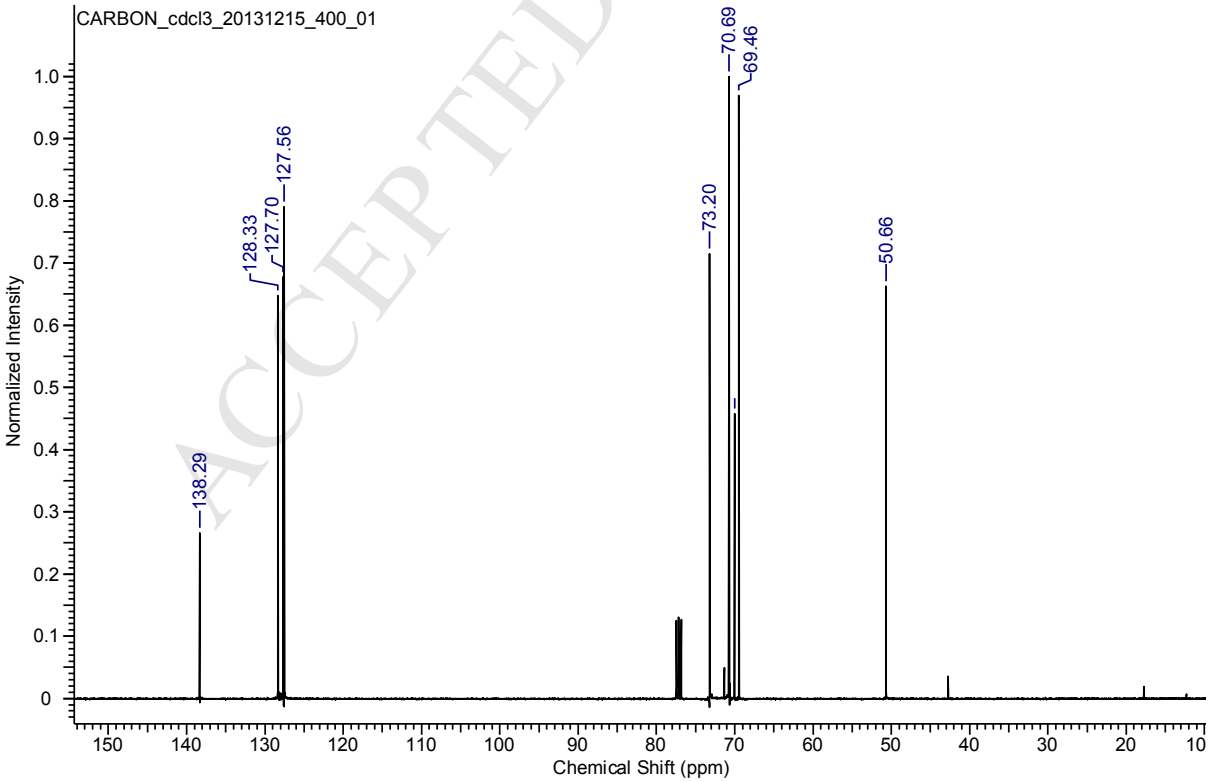




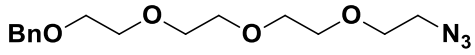
((2-(2-(2-Azidoethoxy)ethoxy)ethoxy)methyl)benzene 12b

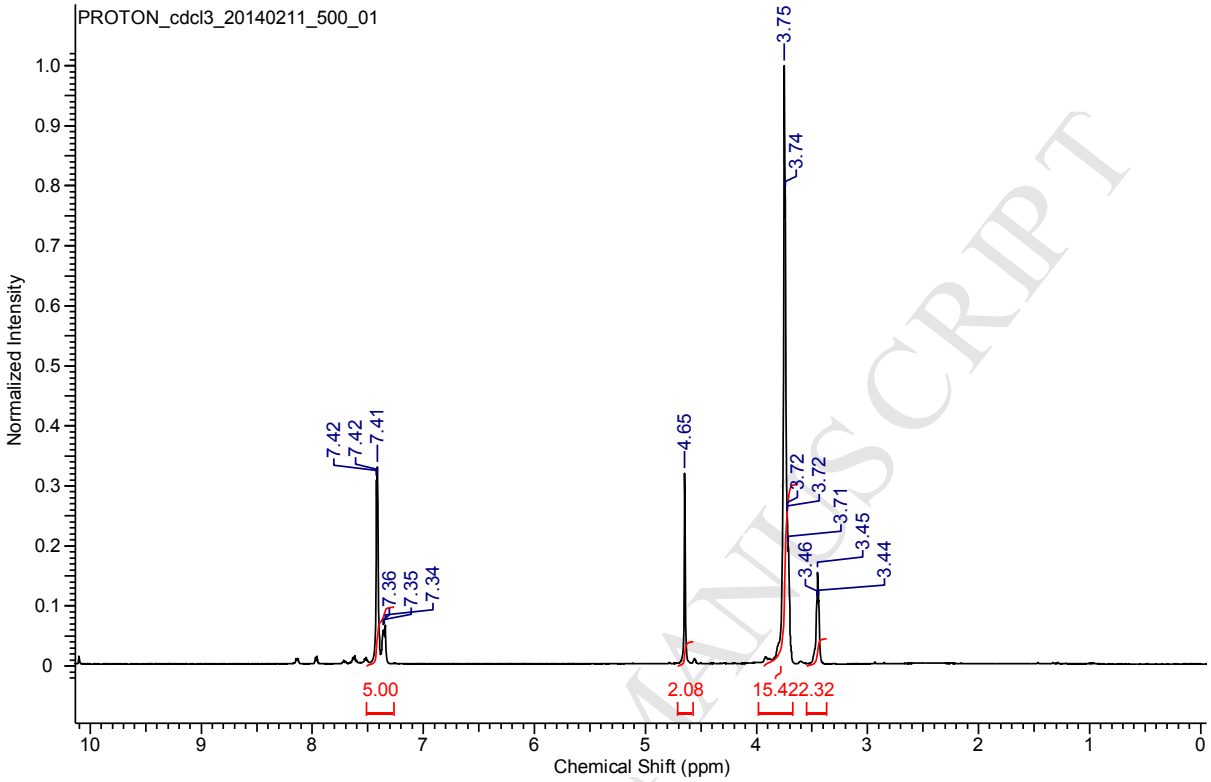


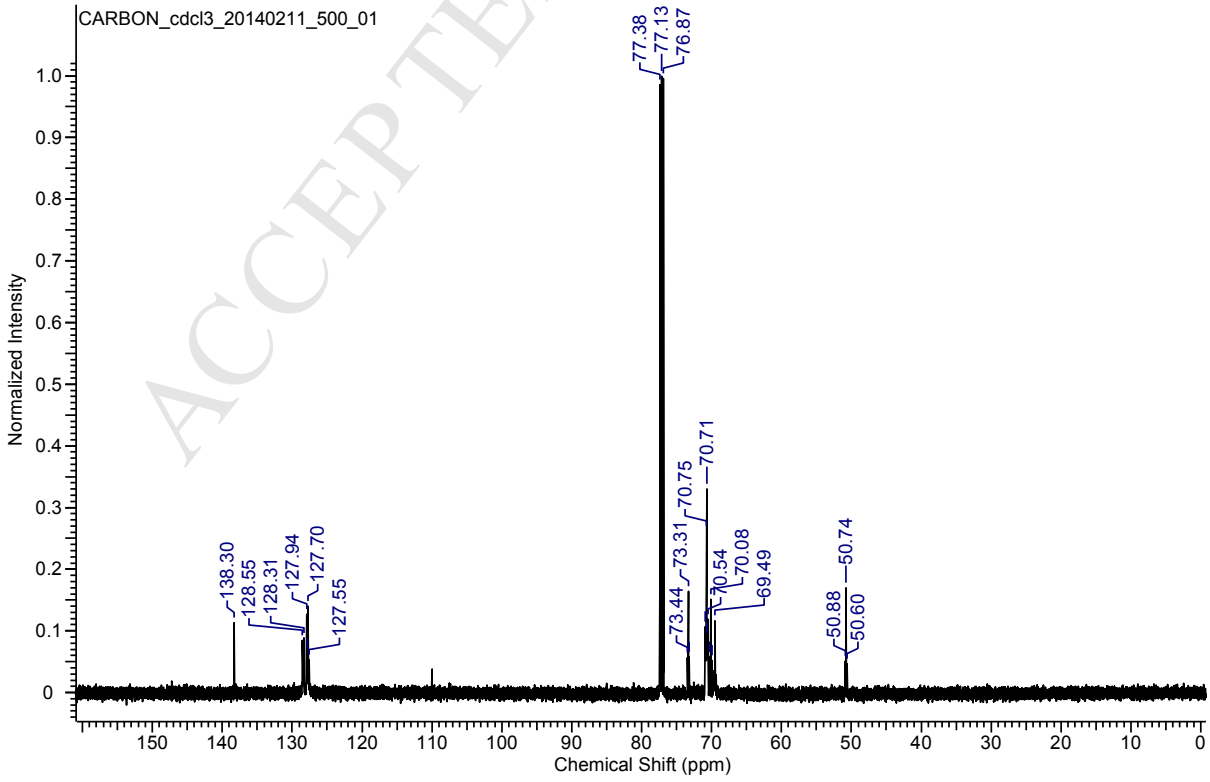




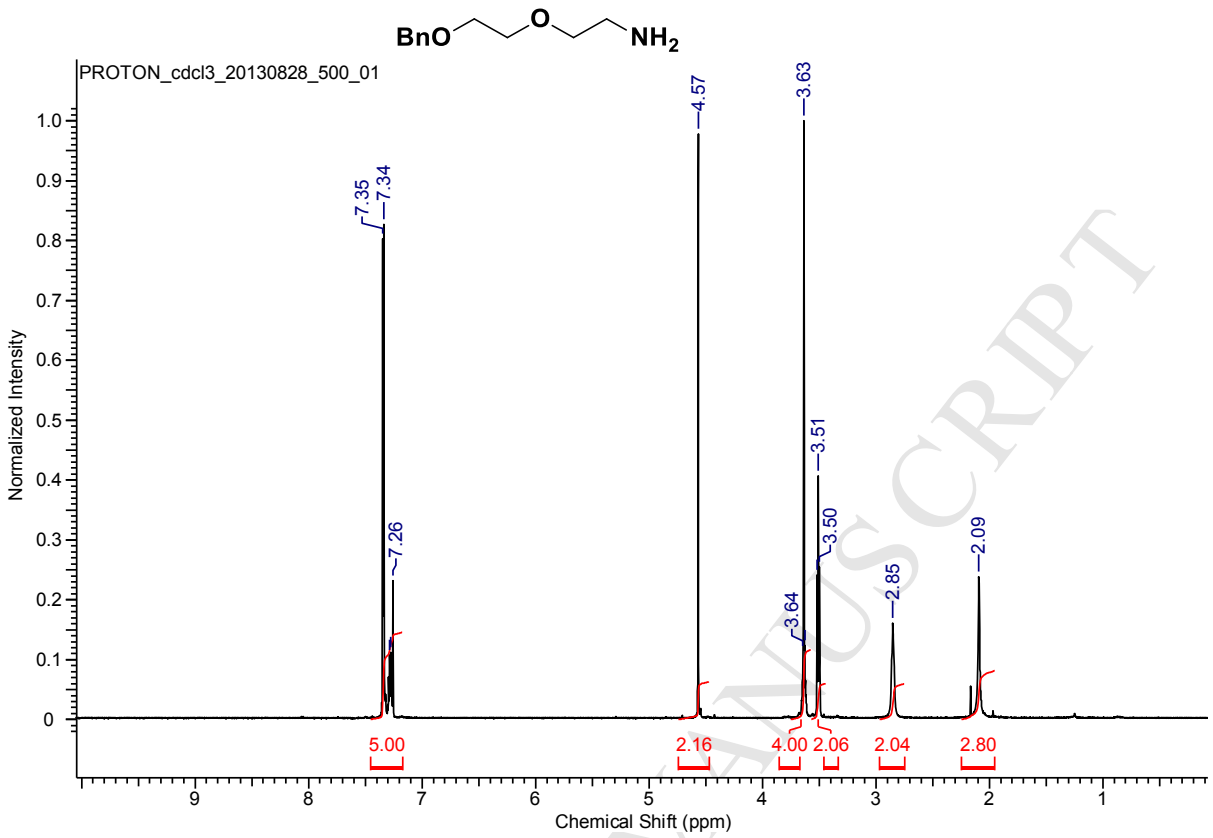
((2-(2-(2-(2-Azidoethoxy)ethoxy)ethoxy)ethoxy)methyl)benzene 12c

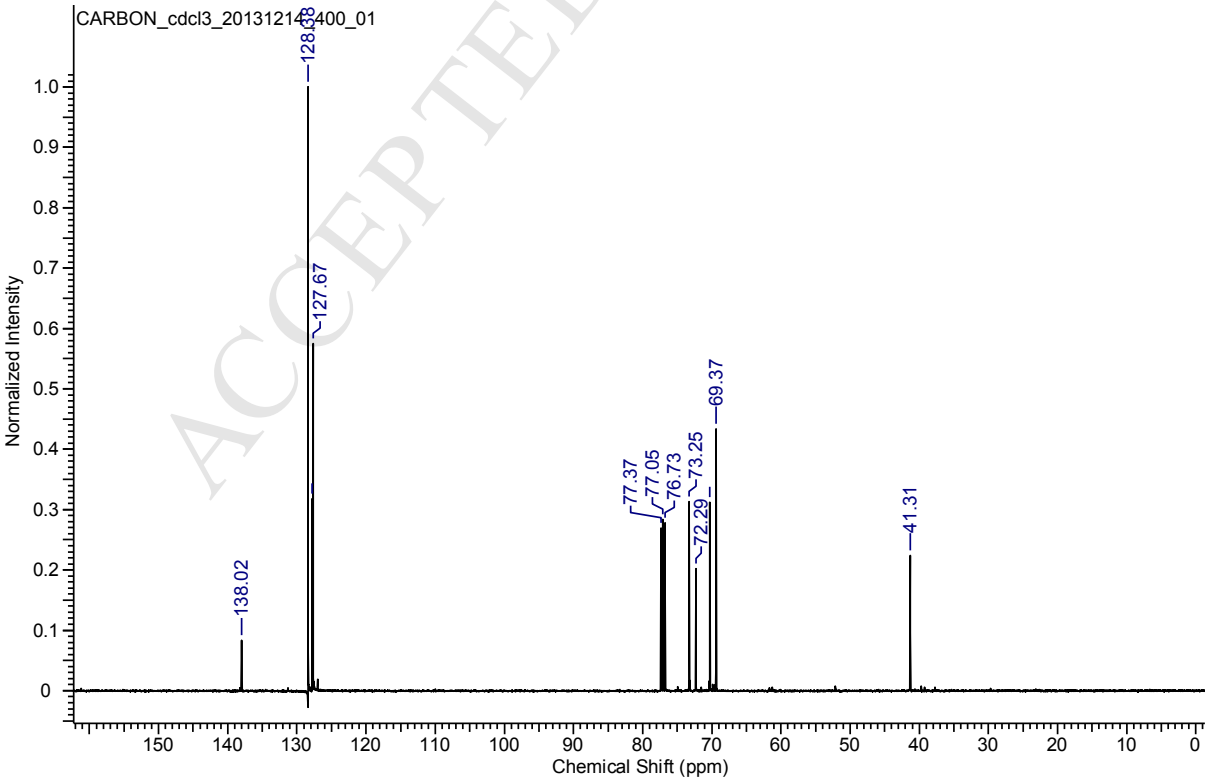




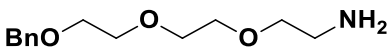


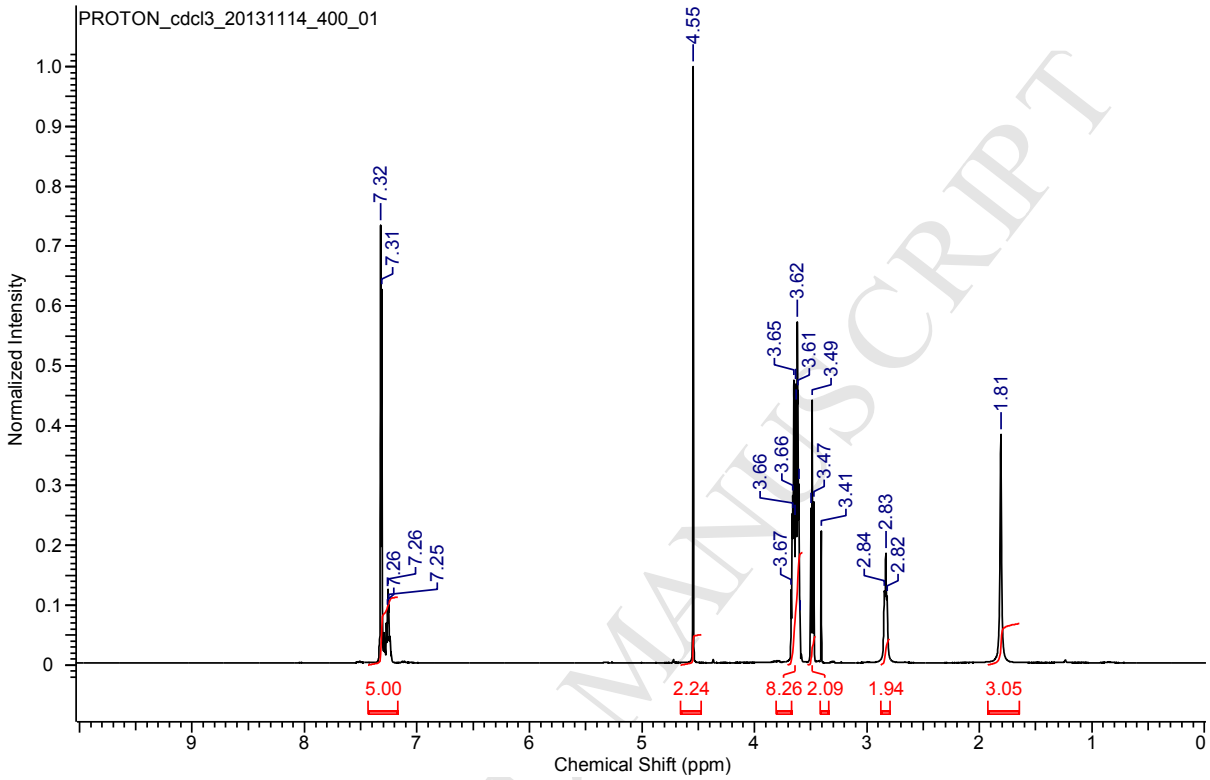
2-(2-(Benzyloxy)ethoxy)ethanamine 13a

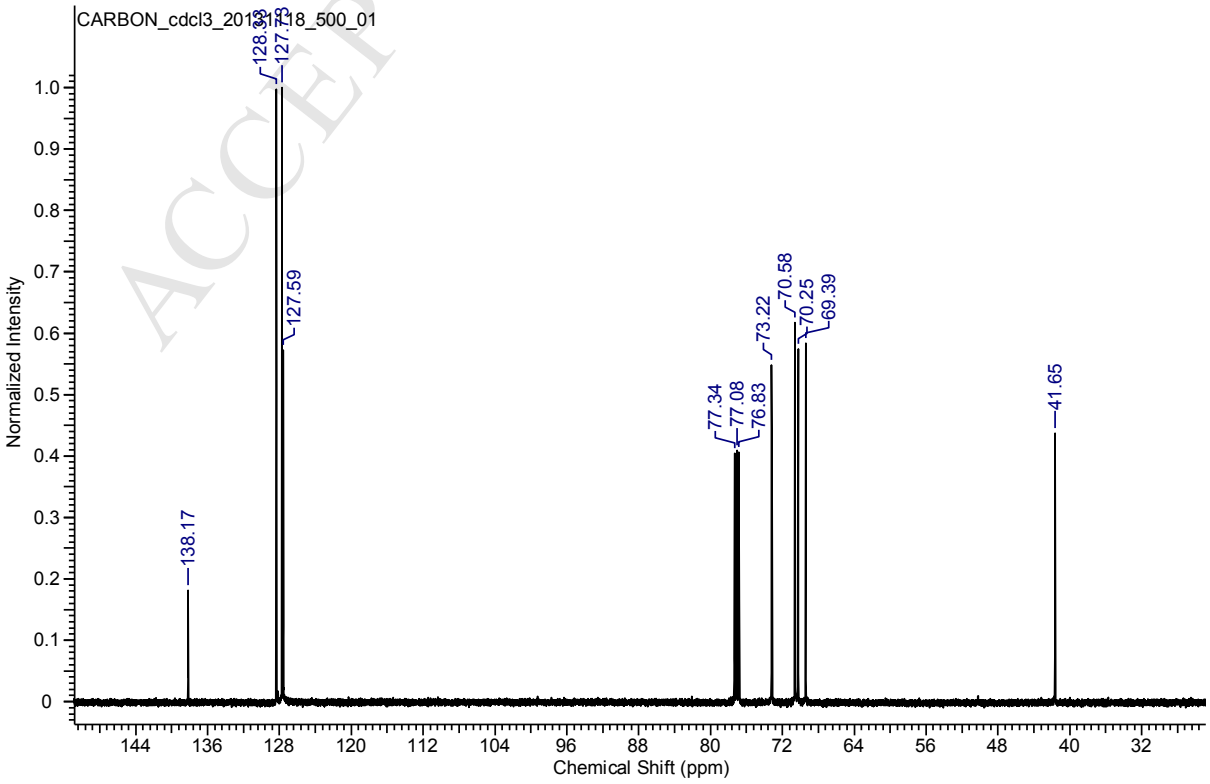




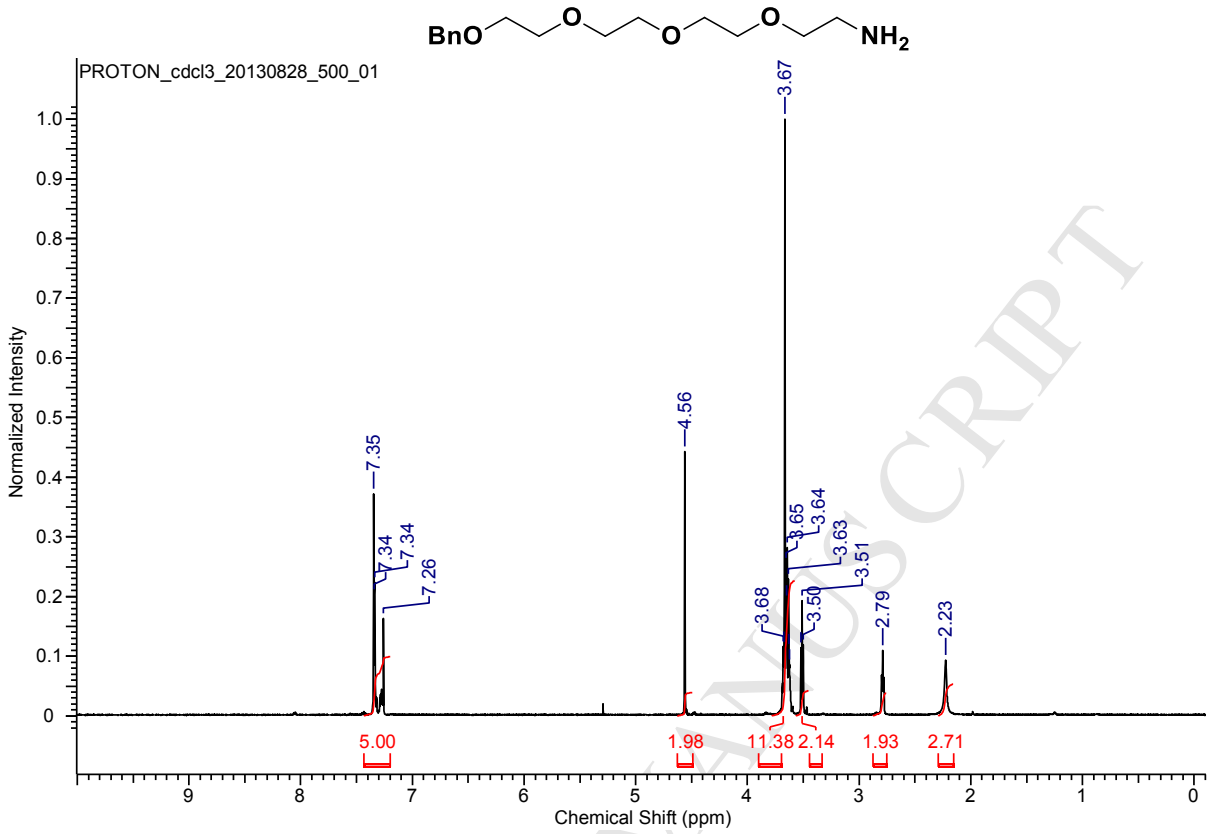
2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethanamine 13b

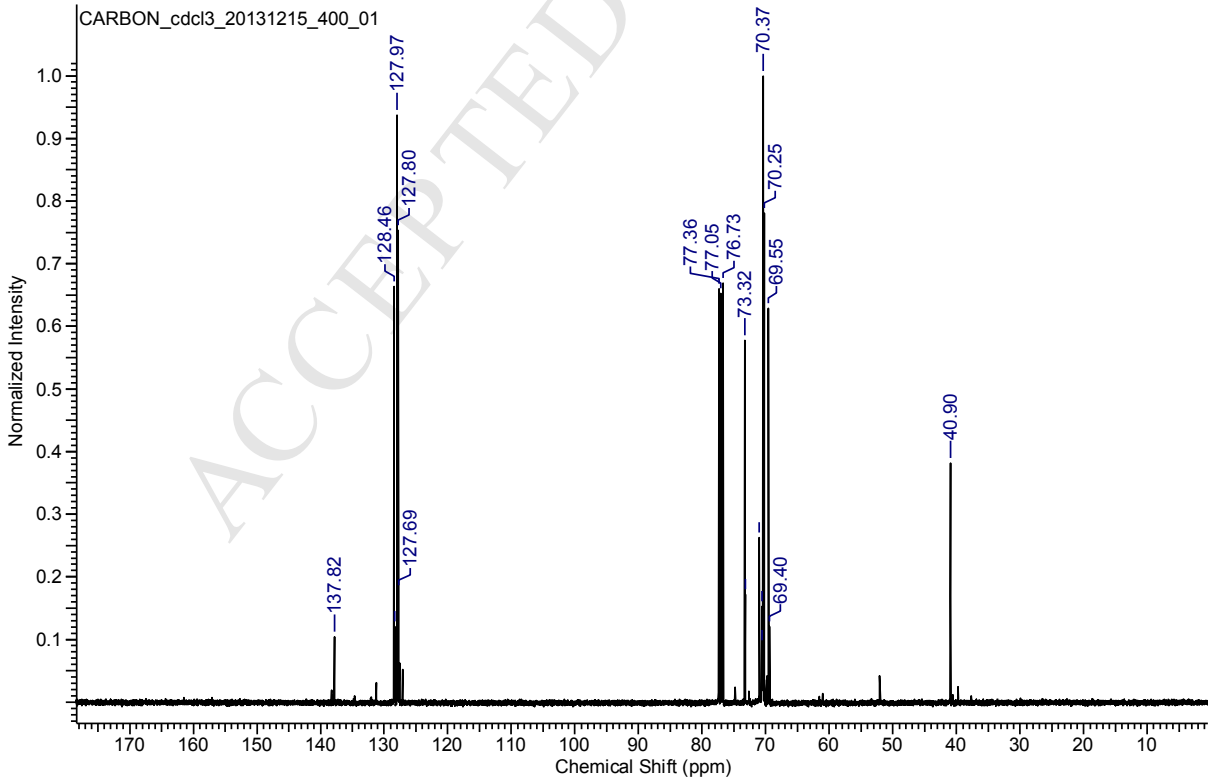




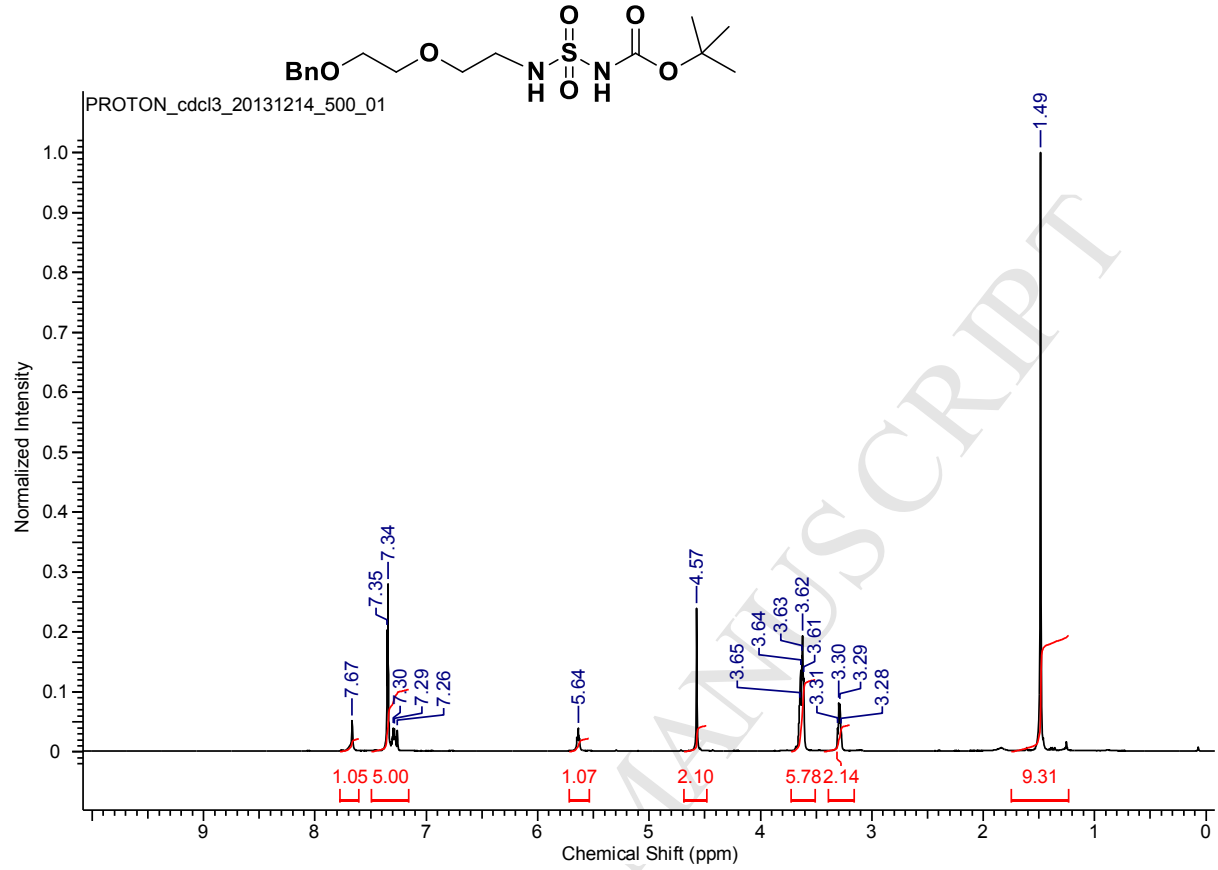


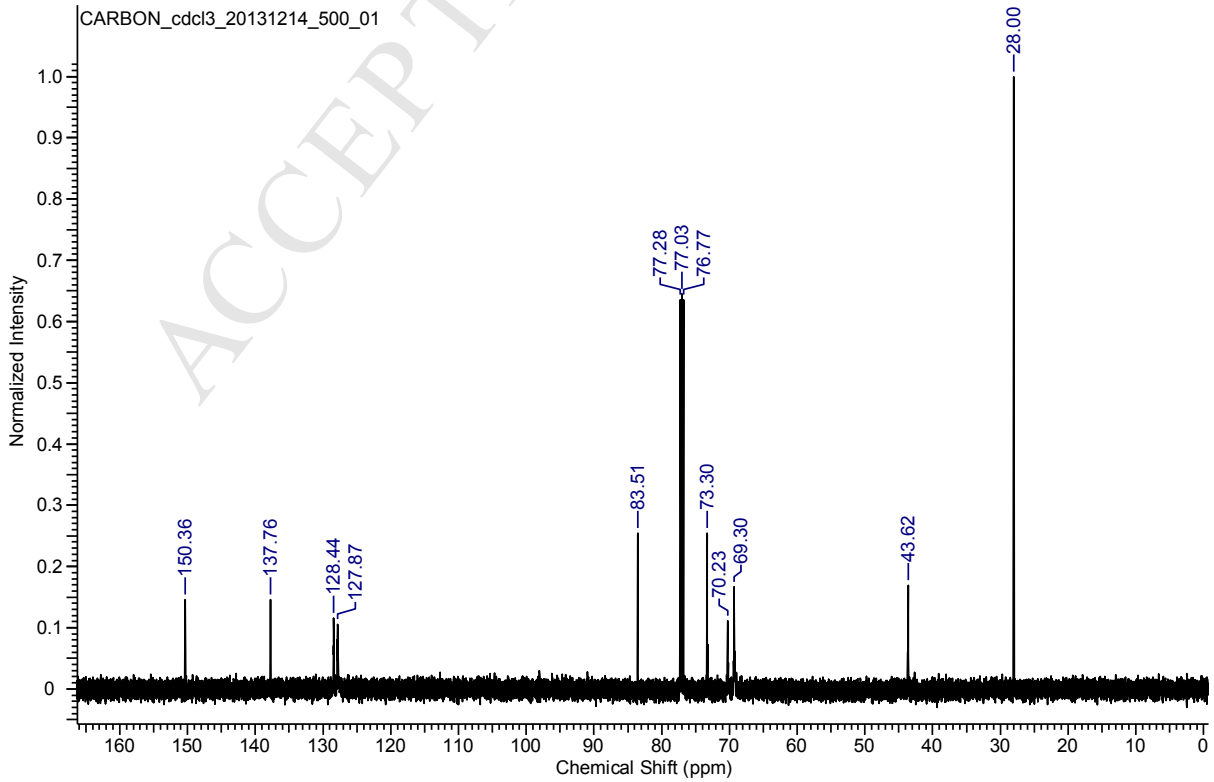
2-(2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethoxy)ethanamine 13c



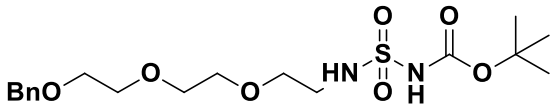


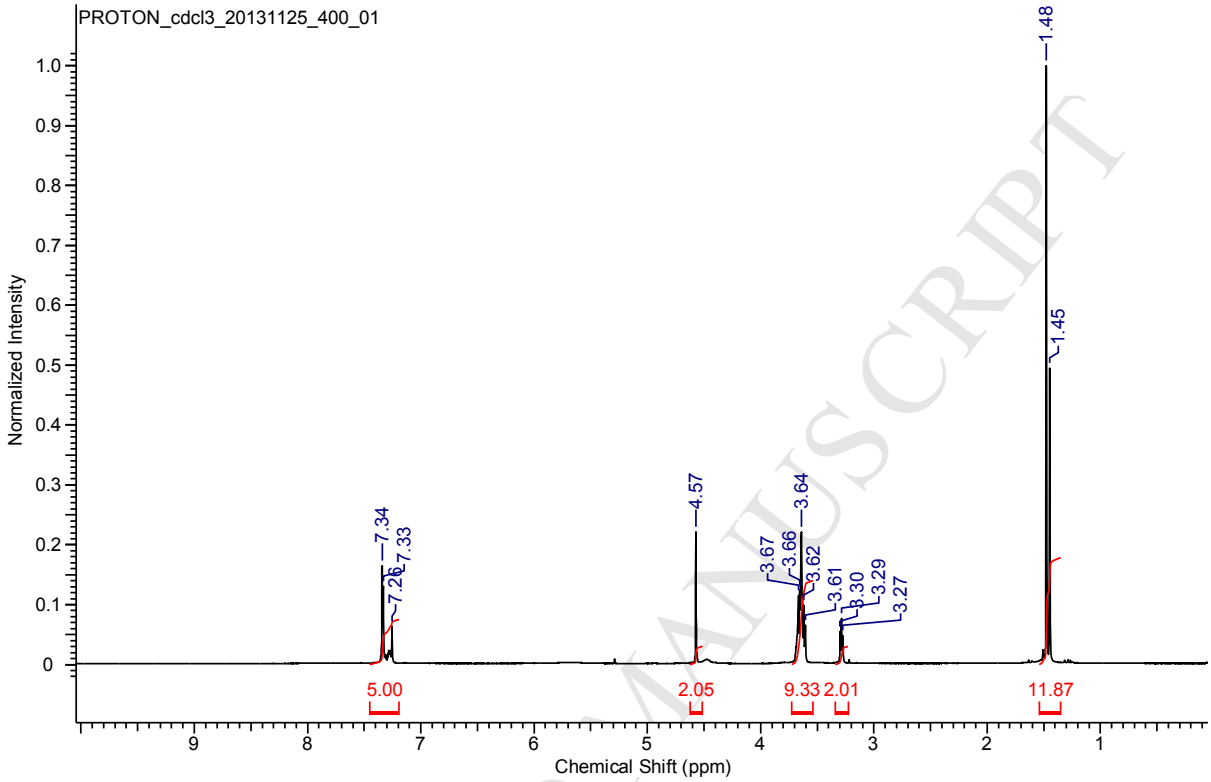
Tert-butyl *N*-2-(2-(Benzyloxy)ethoxy)ethylsulfamoylcarbamate 14a

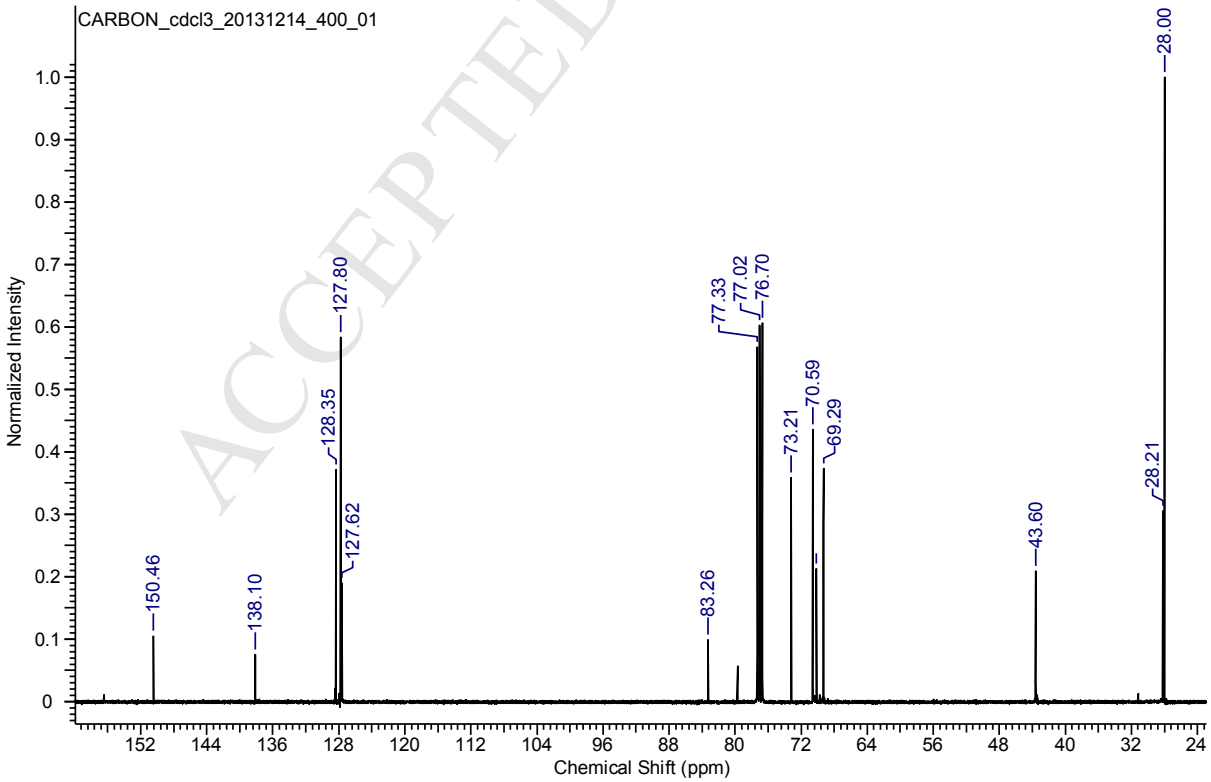




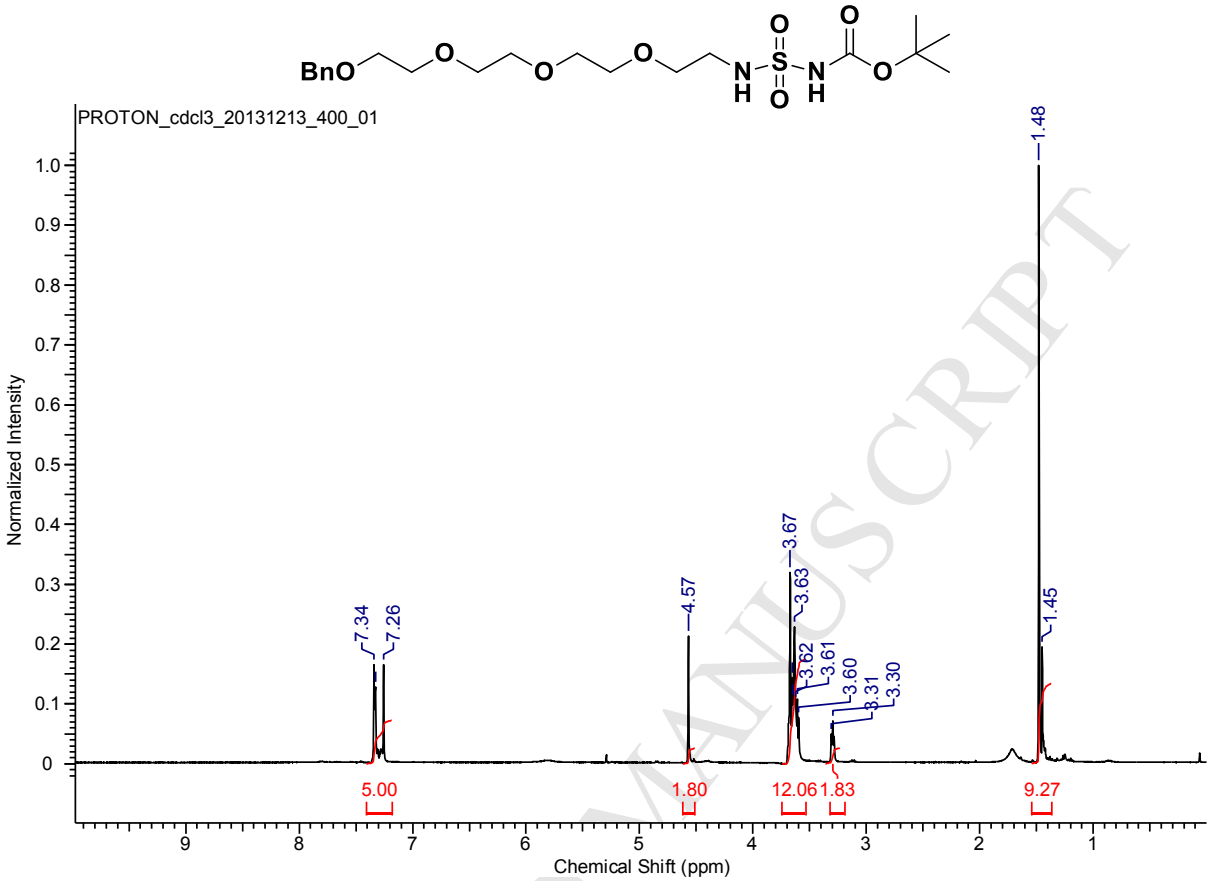
Tert-butyl _N_-2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethylsulfamoylcarbamate 14b

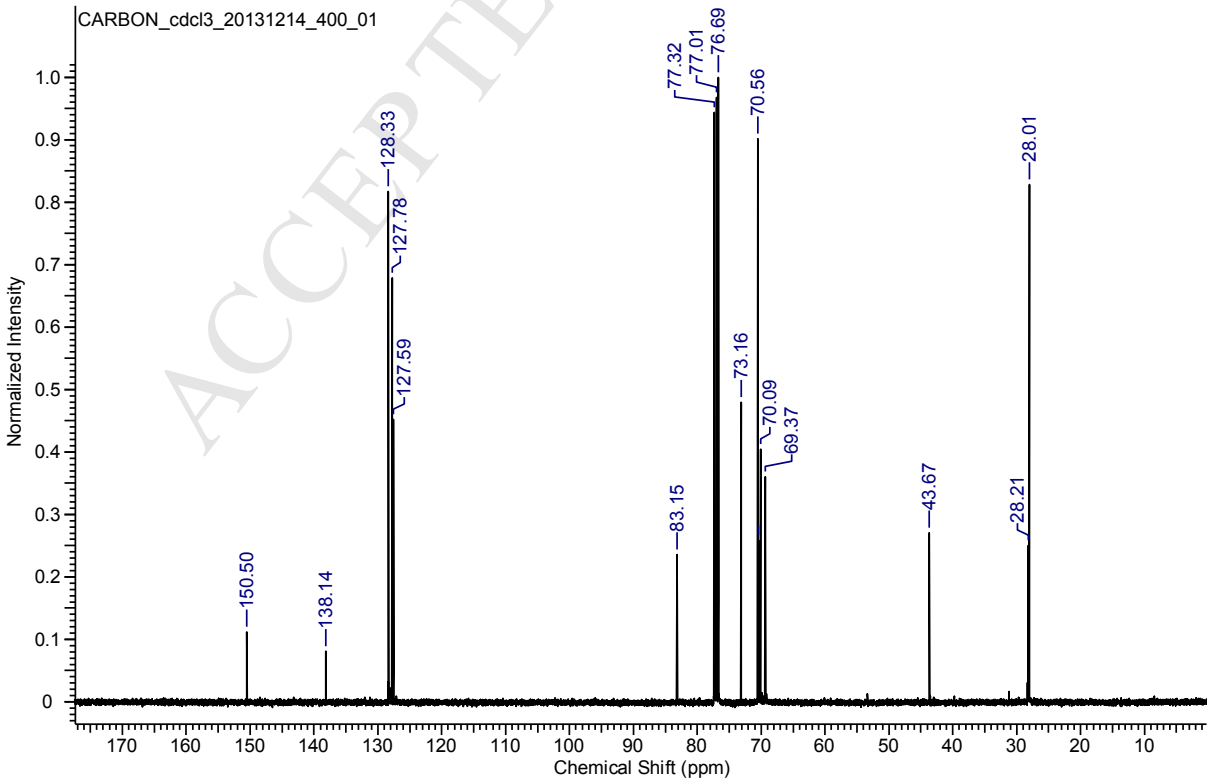




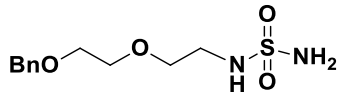


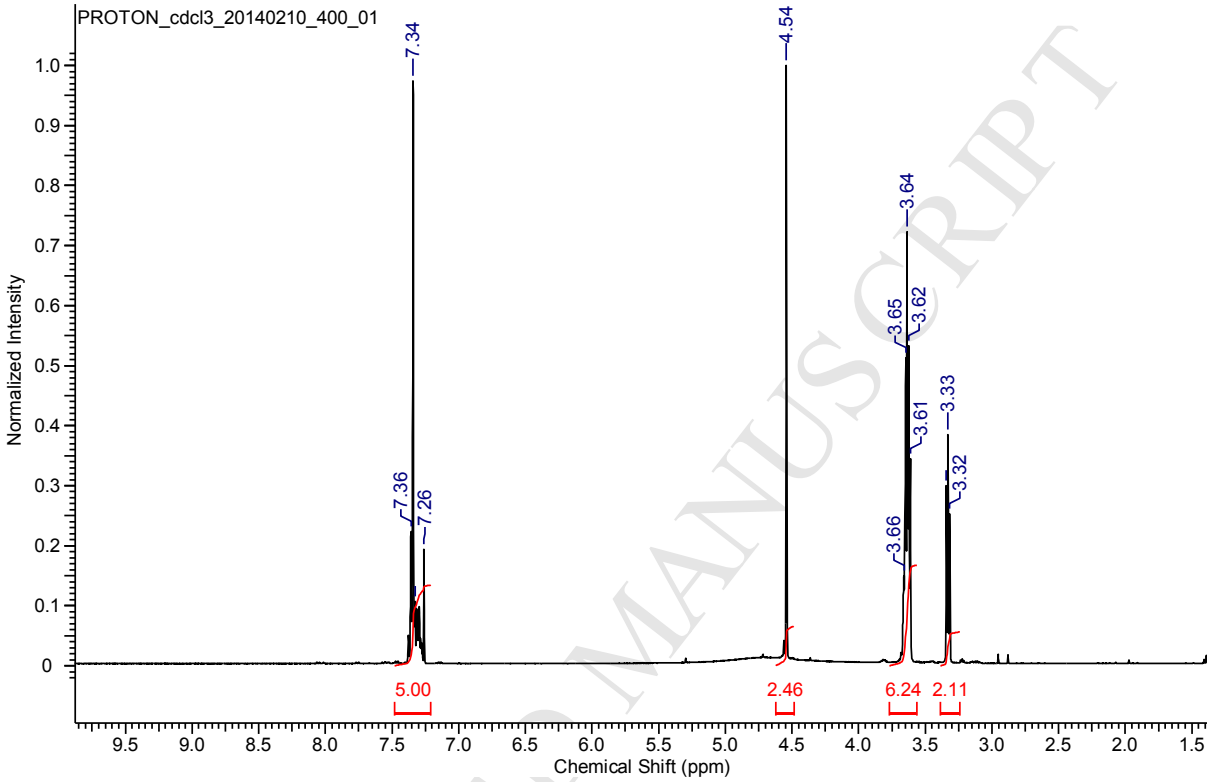
Tert-butyl _N_-2-(2-(2-(2-(benzyloxy)ethoxy)ethoxy)ethoxy)ethylsulfamoylcarbamate 14c

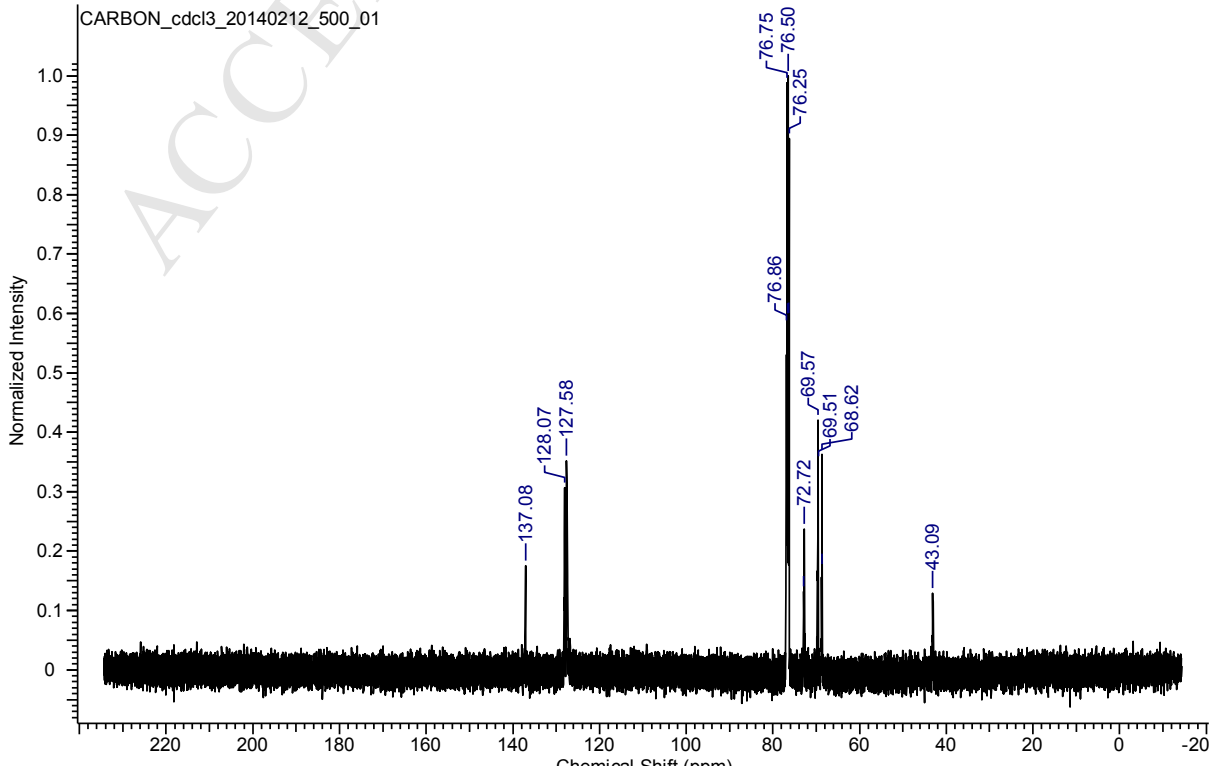




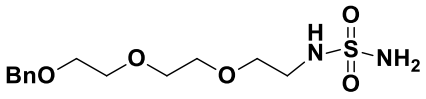
_N_-(2-(2-(Benzyloxy)ethoxy)ethyl)sulfamide 15a

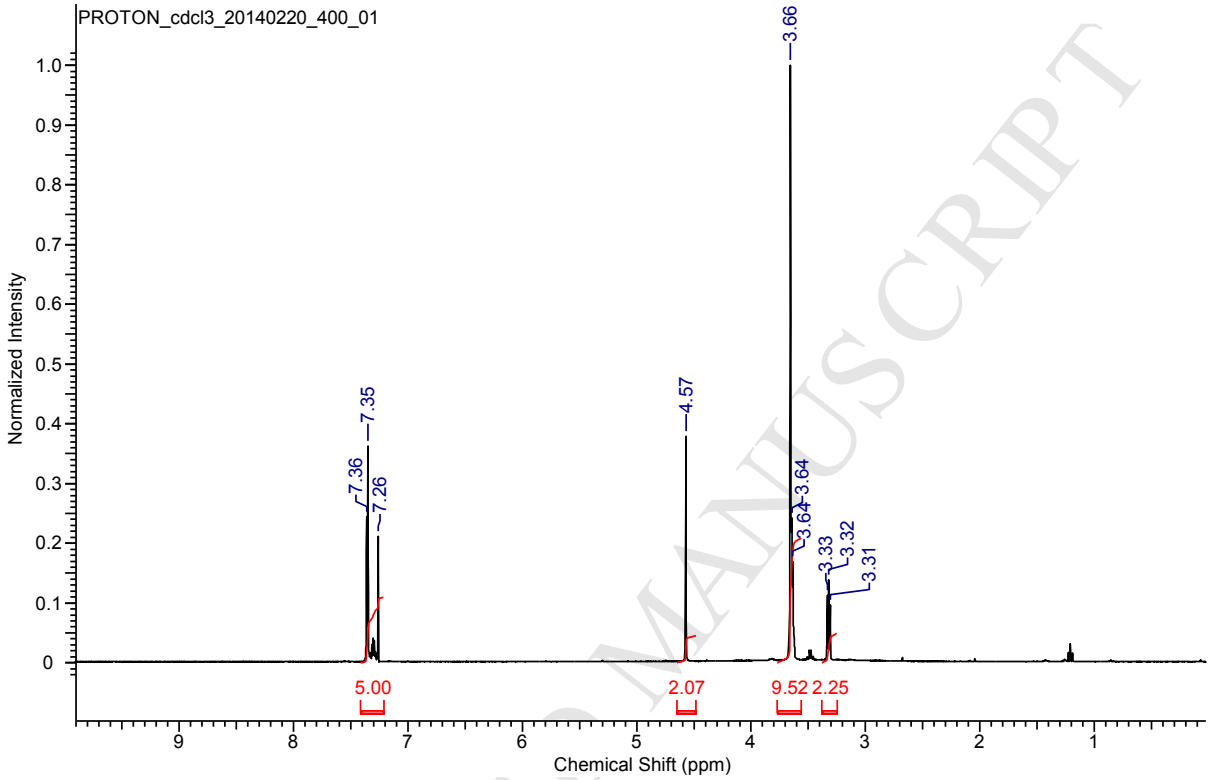


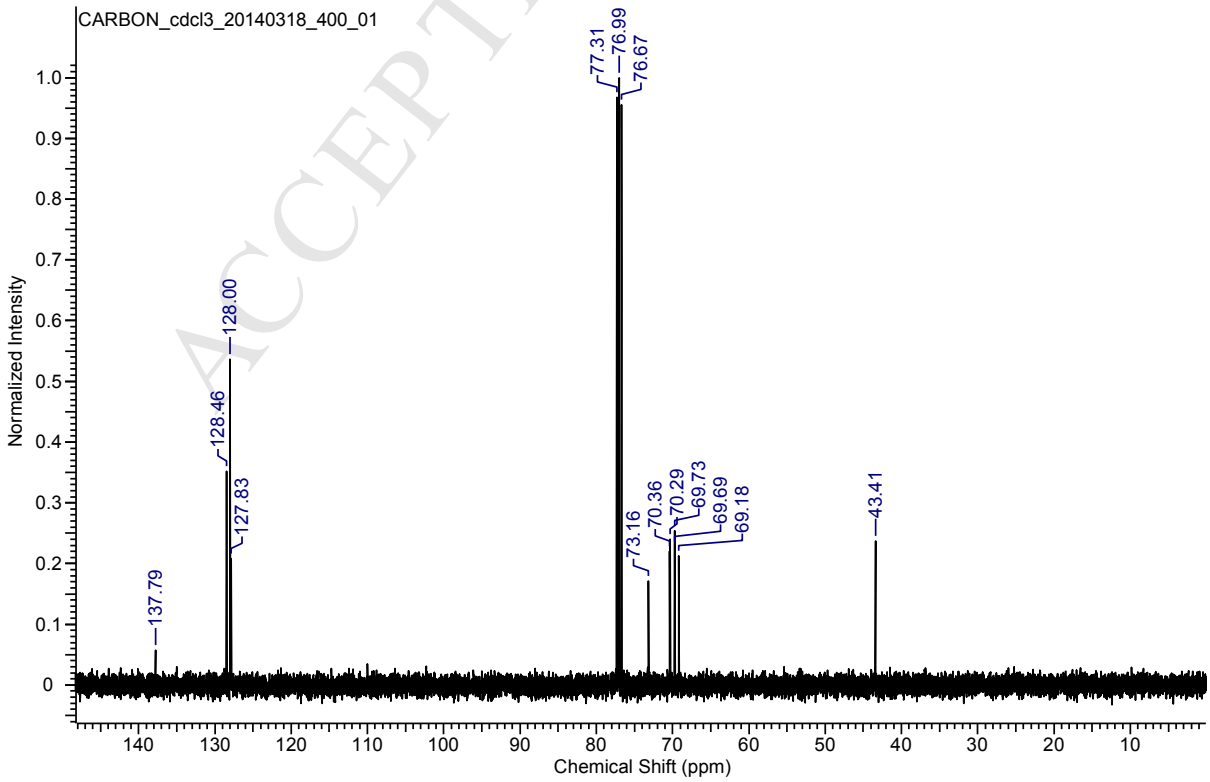




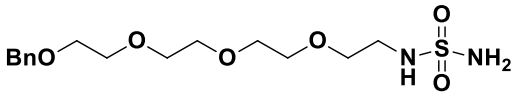
_N_-2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethyl)sulfamide 15b

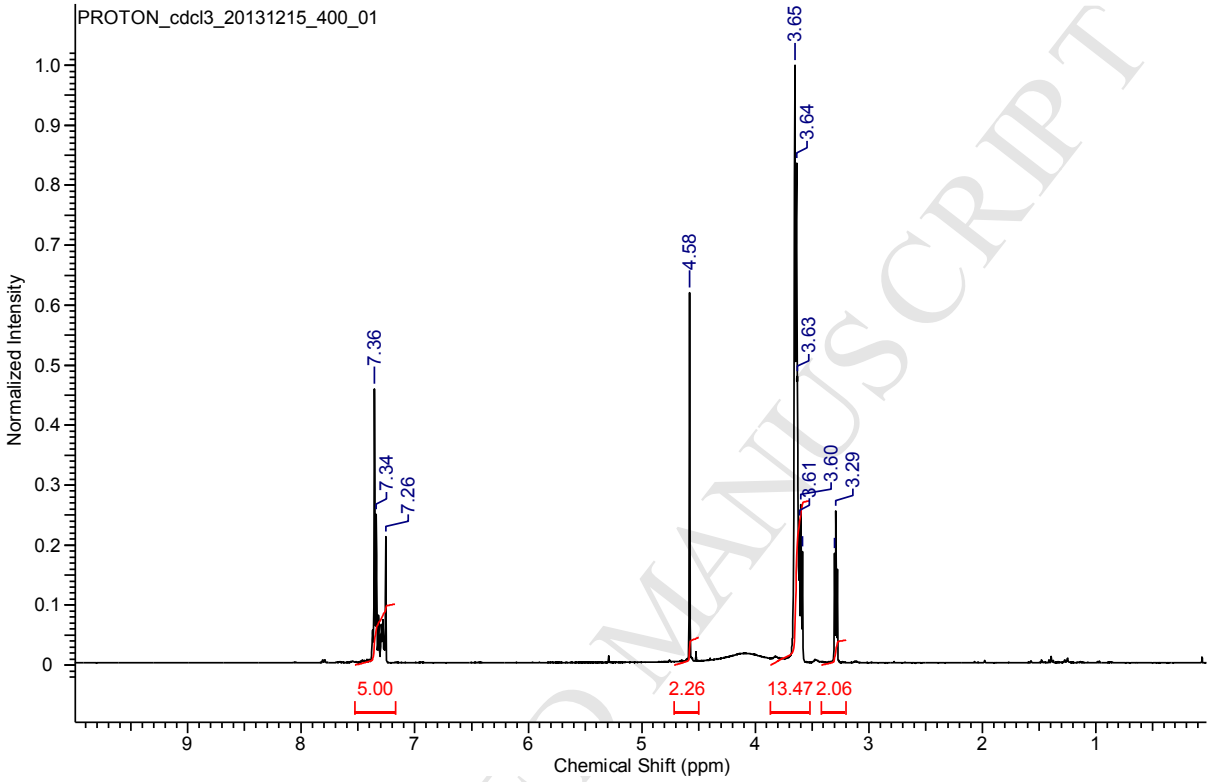


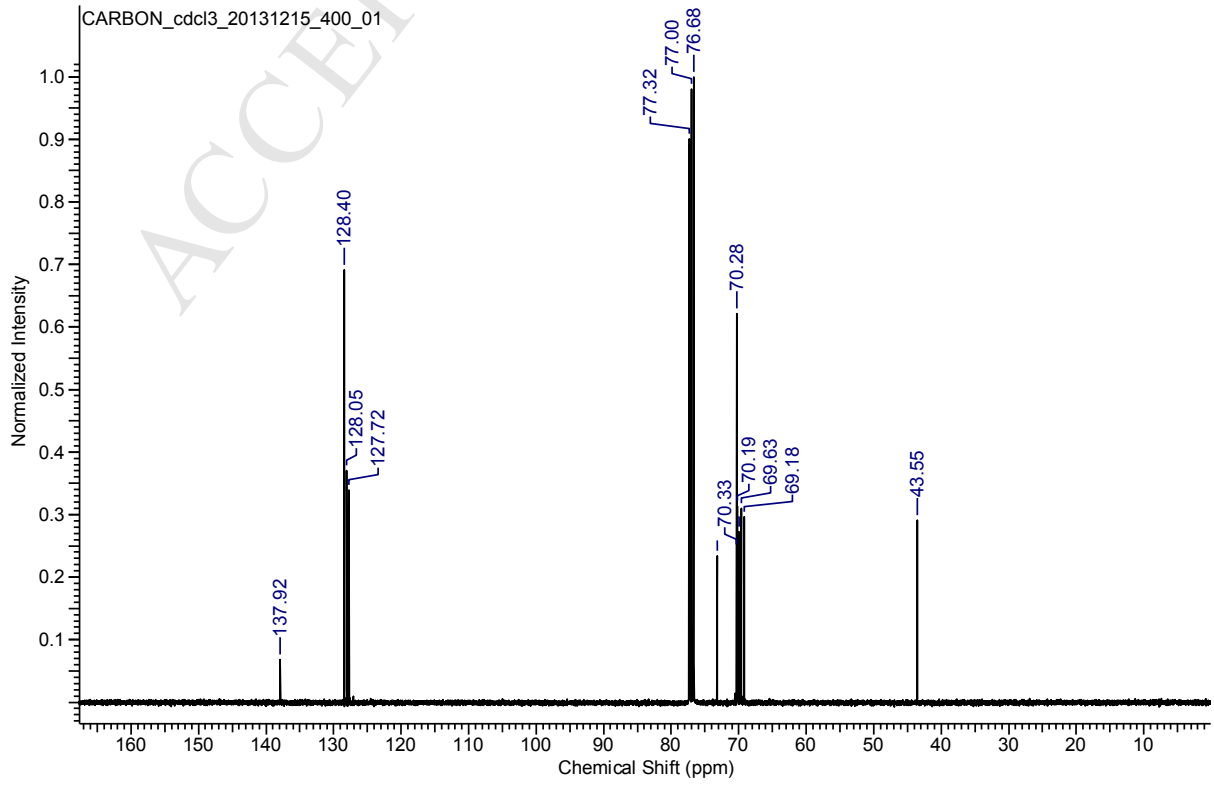




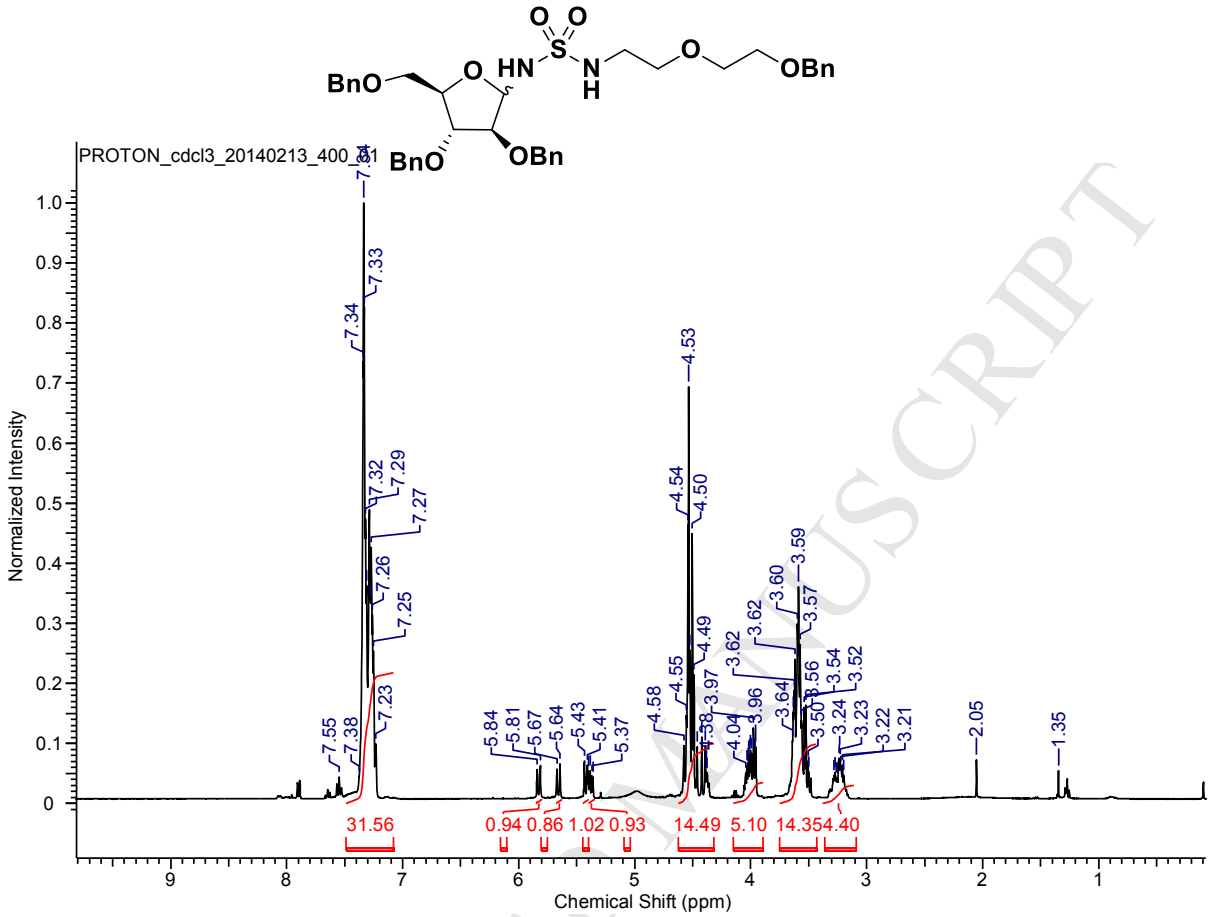
_N_-2-(2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethoxy)ethyl)sulfamide 15c

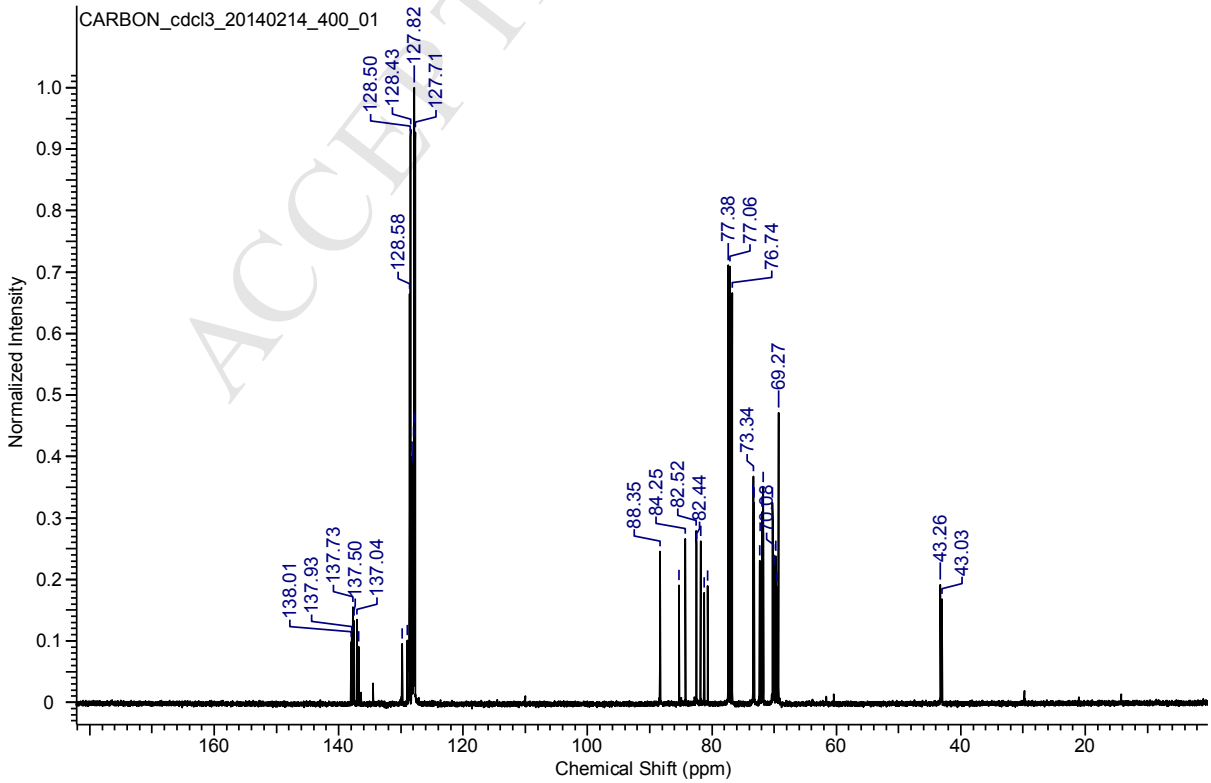




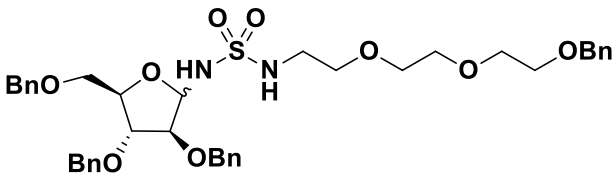


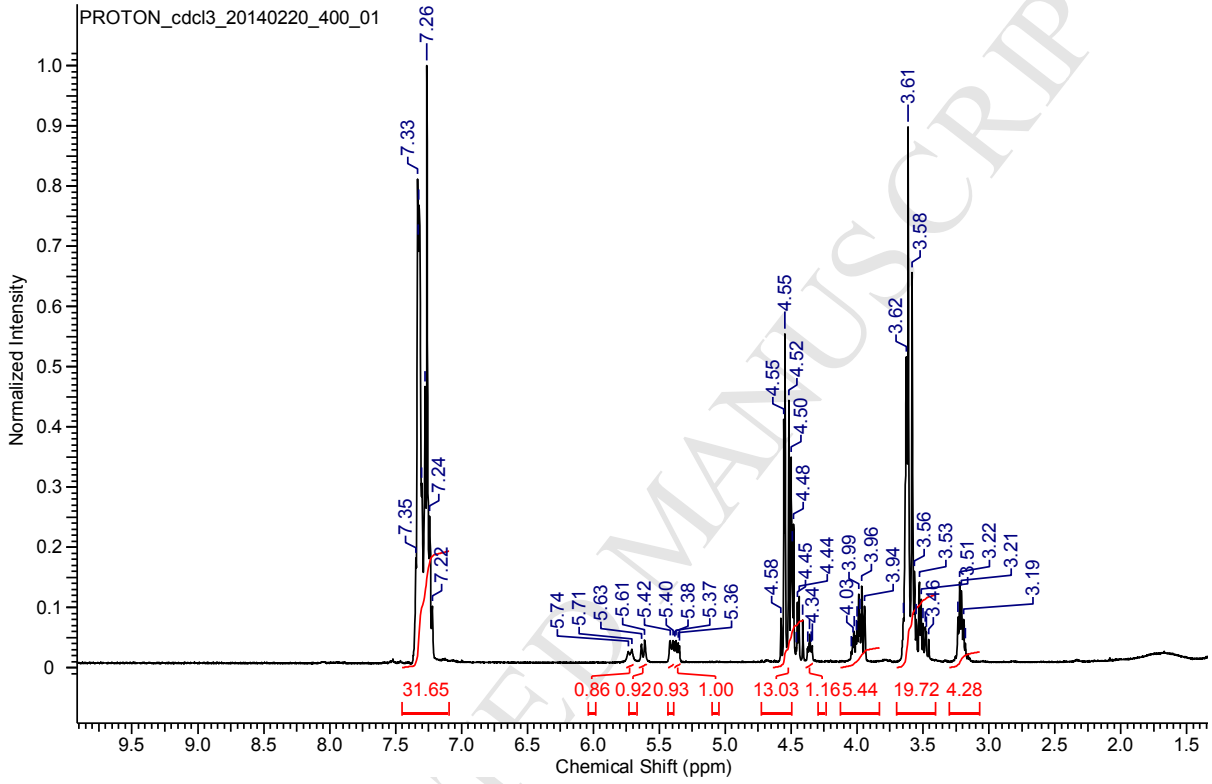
N-(2-(2-(Benzyloxy)ethoxy)ethyl)-*N*'-(2,3,5-tri-*O*-benzyl-α,β-D-arabinofuranosyl)sulfamide 16a

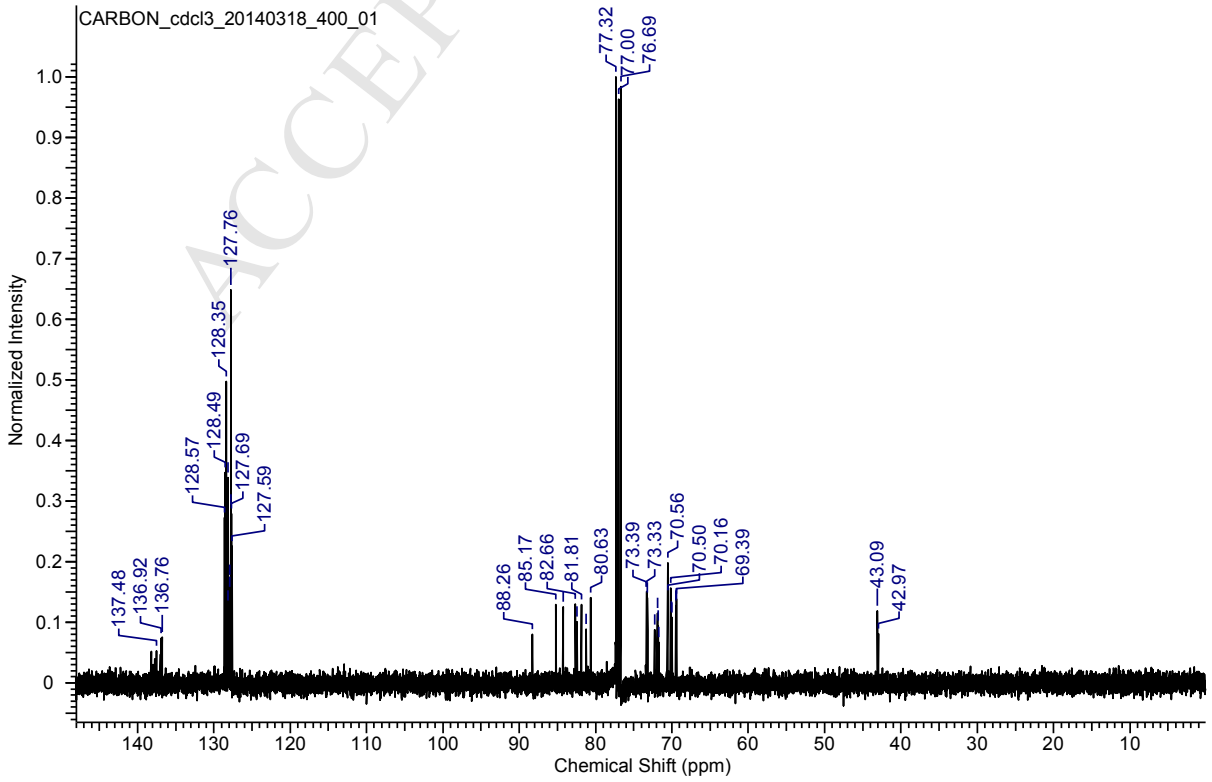




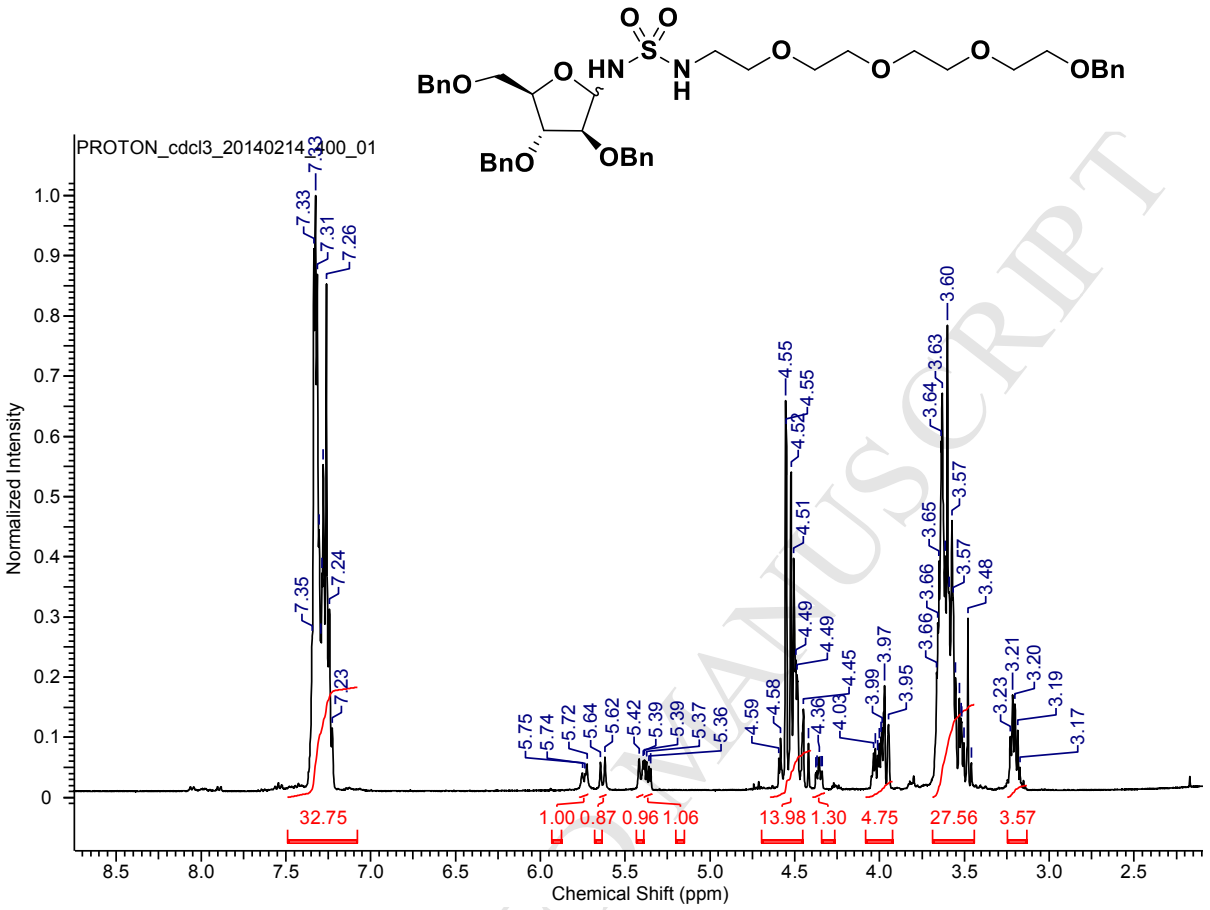
_N_-2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 16b

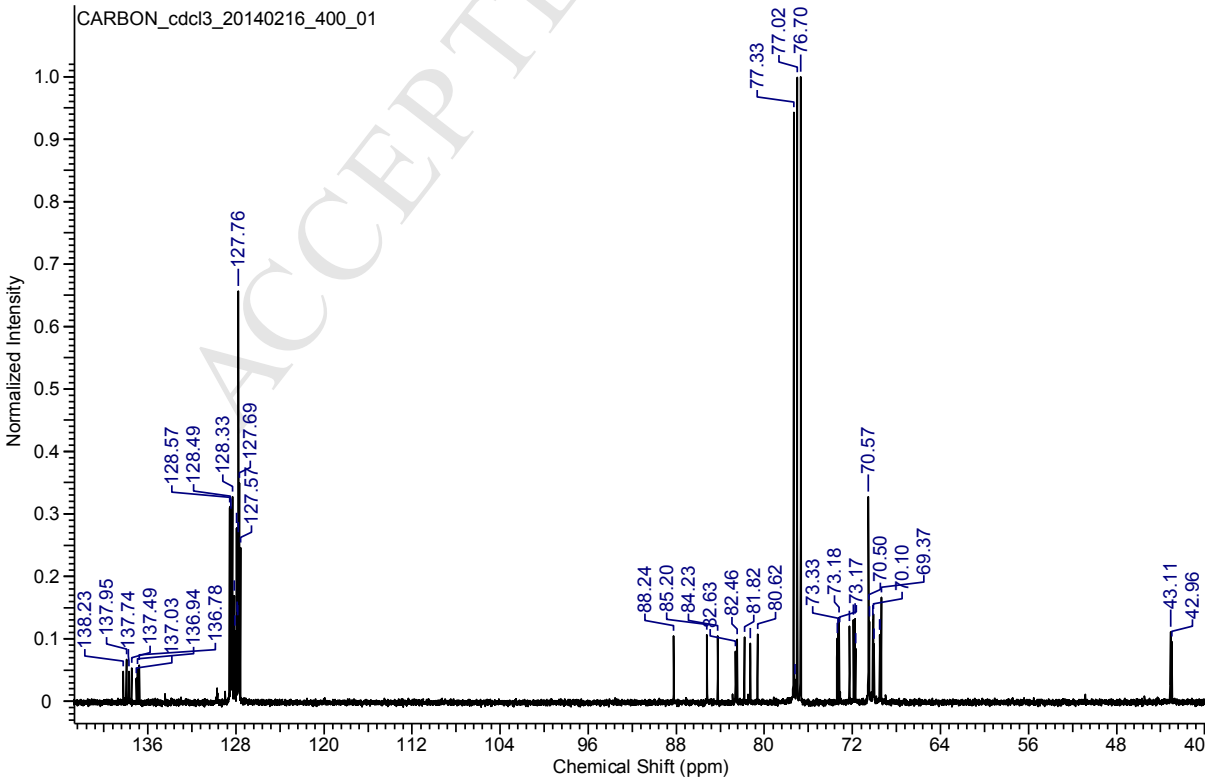




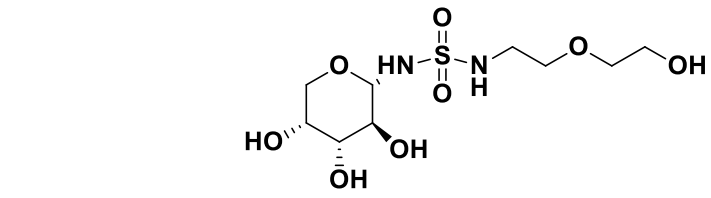


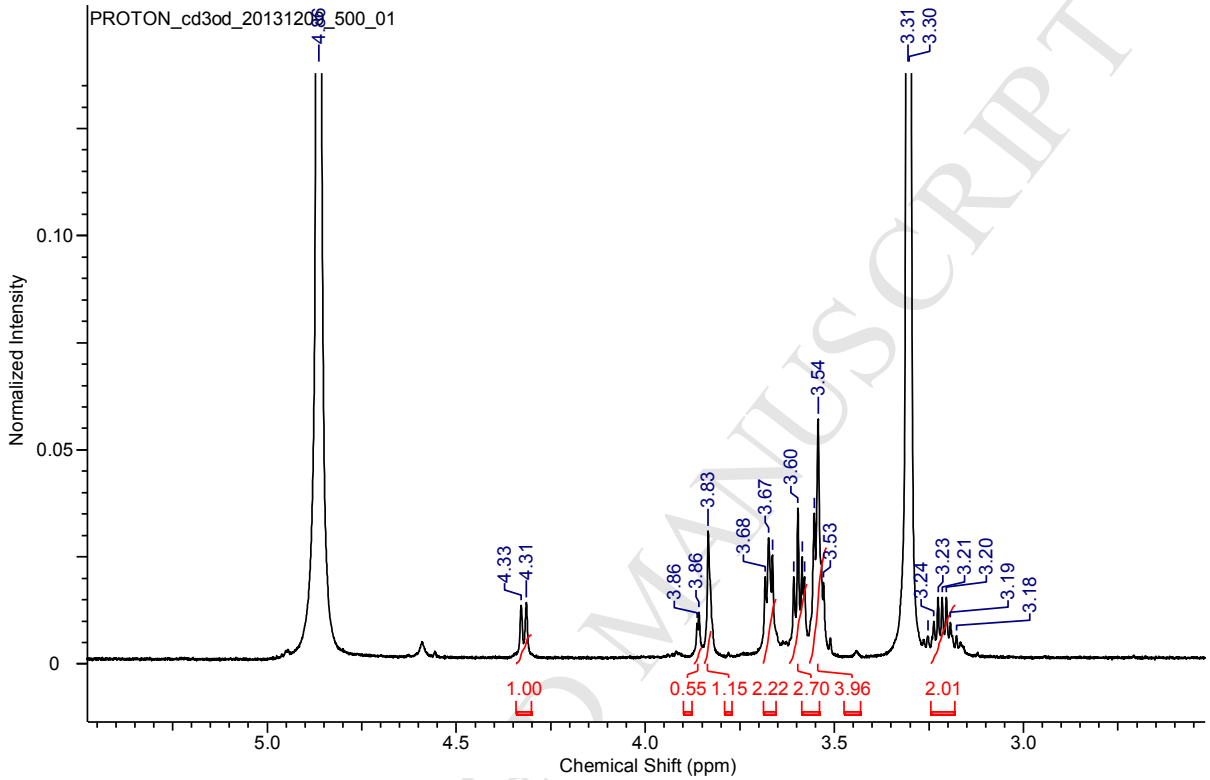
_N_-2-(2-(2-(2-(Benzyloxy)ethoxy)ethoxy)ethoxy)ethyl)-_N_'-(2,3,5-tri-_O_-benzyl-α,β-D-arabinofuranosyl)sulfamide 16c

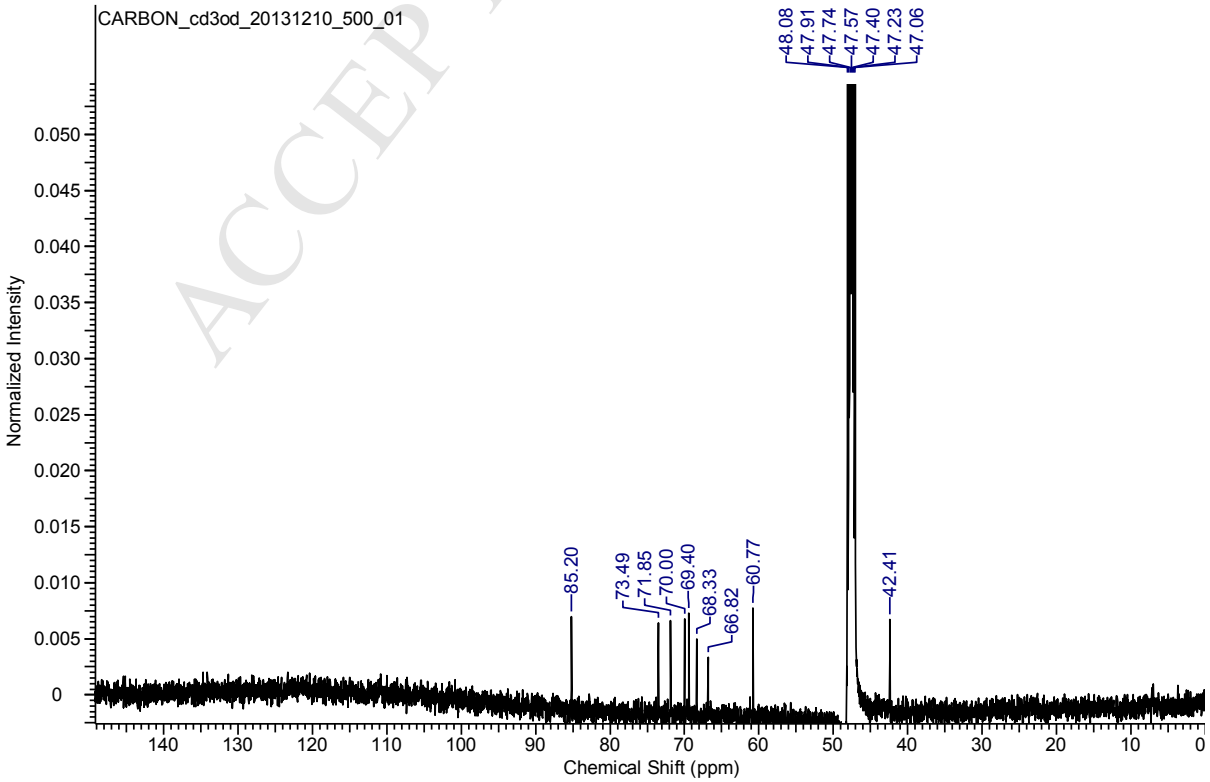




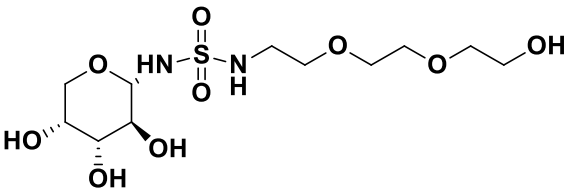
_N_-(2-(2-ethoxy)ethanol-_N_'-(α-D-arabinopyranosyl)sulfamide 17a

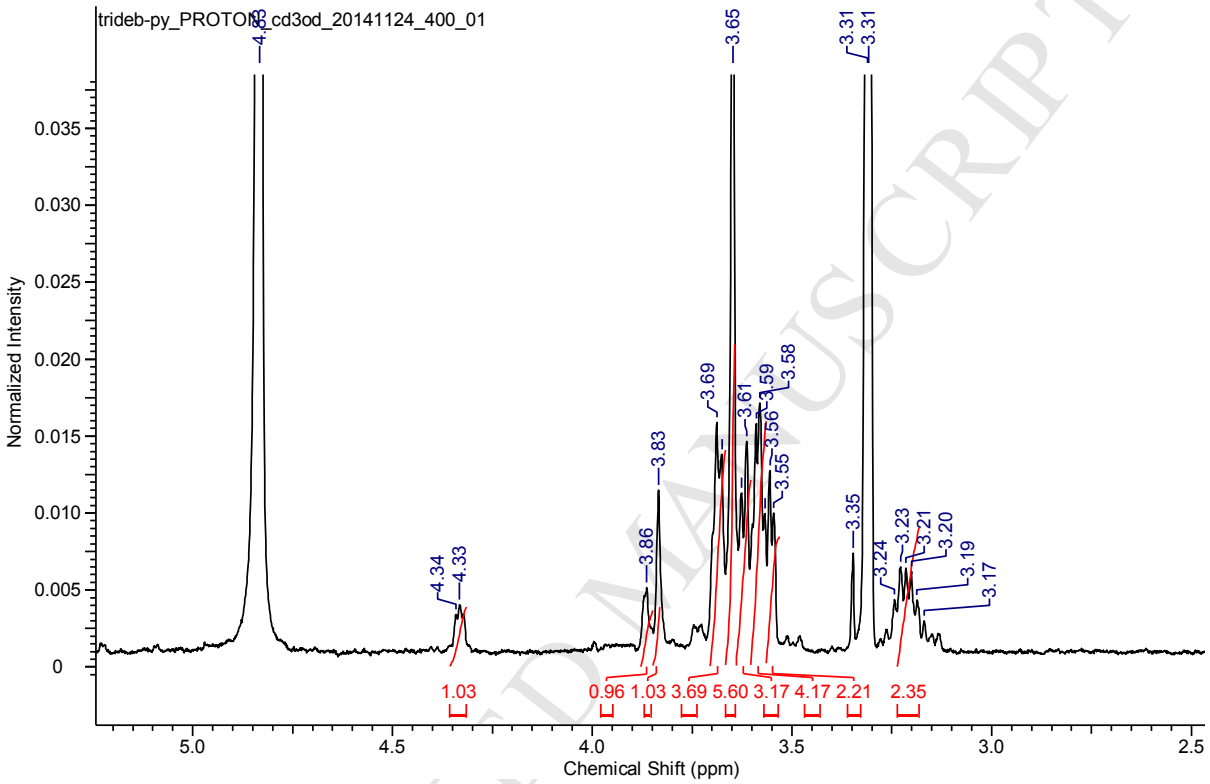


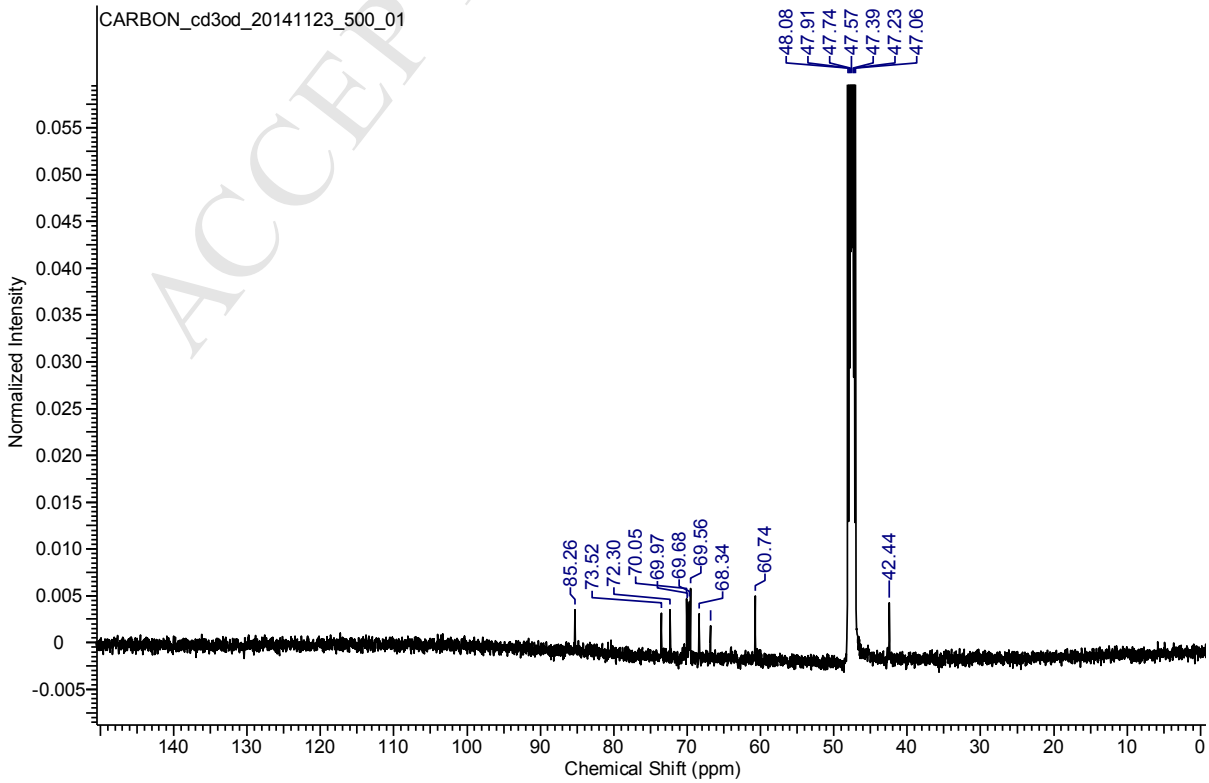




_N_-(2-(2-(2-(ethoxy)ethoxy)ethanol-_N_'-(α-D-arabinopyranosyl)sulfamide 17b







_N_-(2-(2-(2-(2-ethoxy)ethoxy)ethoxy)ethyl)ethanol-_N_'-(β-D-arabinopyranosyl)sulfamide

17c