

Supporting Information

Highly sulphated cellulose: A versatile, reusable and selective desilylating agent for deprotection of alcoholic TBDMs ethers

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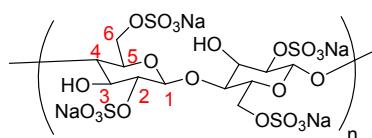
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General Information

Solvents were dried by standard procedures. All commercially available alcohols and phenols were purchased from the Sigma Aldrich Company. Thin-layer chromatography was performed on silica gel plates and chromatographic spots were visualized by using UV light and staining with anisaldehyde charring solution. ¹H NMR and ¹³C NMR spectral analyses were recorded on a Varian 400 MHz spectrometer equipped with a Linux workstation running on vNMRj software and the chemical shifts are based on the internal standard TMS peak at $\delta = 0.00\text{ ppm}$ for proton NMR and the CDCl₃ peak at $\delta = 77.23\text{ ppm}$ (*t*) for carbon NMR. For cellulose sulphate sodium both ¹H NMR and ¹³C NMR were recorded in D₂O. IR spectra were obtained on a Thermo Scientific Nicolet iS 50 FT-IR instrument. Silyl ethers were prepared from phenols\alcohols using a standard procedure¹ (TBDMs chloride, imidazole, DCM). TBDMs ethers **3k**, **5e**, **5f** were synthesized by the Barbier allylation procedure². TBDMs ethers **3a-3j** and **5a-5d** were prepared from commercially available phenols/alcohols. Compound **3l** was prepared by the reduction of methyl 8-(4-chlorophenyl)-1, 4-dioxaspiro[4.5]dec-7-ene-7-carboxylate using lithium aluminum hydride (LAH) as reducing agent to afford **4l**. Compound **4l** was converted to its TBDMs ether by reaction with TBDMs chloride and imidazole in DCM.¹ TBDMs ethers **3a**³, **3b**⁴, **3e**⁵, **5a**⁵, **5b**⁶, **5c**⁵ are known compounds and the characterization data for these ethers complied with the literature reported data. The desilylated products **4a**⁷, **4b**⁸, **4c**⁹, **4d**,¹⁰ **4e**¹¹, **4f**¹², **4g**¹³, **4h**¹⁴, **4i**,¹⁵ **4j**,¹⁶ **4k**¹⁷, **6a**⁶, **6b**¹⁸, **6c**¹⁹, **6d**²⁰, **6e**²¹ are known

compounds, and their characterization data complied with the literature reported data. Characterization data for the new compounds **3c**, **3d**, **3f**, **3g**, **3h**, **3i**, **3j**, **3k**, **3l**, **4l**, **5d**, **5e**, **5f** and **6f** are provided below.

Characterization data for new compounds



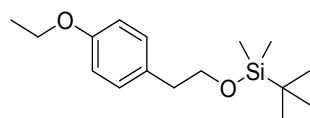
HS-cellulose sulphate sodium salt

HS-Cellulose sulphate (H_2SO_4 method) sodium salt: The compound was obtained as a white solid; 1H -NMR (D_2O , 400 MHz): δ 4.56 (d, $J = 16.0$ Hz, 1H, H_1), 4.37 (m, 1H, H_6), 4.0-3.49 (m, 3H, H_3 , H_6 , H_4), 3.36 (s, 1H, H_5) ppm; ^{13}C -NMR (D_2O , 100 MHz): δ 104.8 (C-1), 80.7, 77.2, 76.4, 75.4 (C-2 to C-5), 62.3 (C-6) ppm. Sulphur combustion analysis calcd for the 2,6-disubstituted cellulose sulphate, empirical formula $C_6H_8S_2O_{11}Na_2$: 17.51%. Found: 15.42% (88.06% of theoretical), FT-IR: 3332, 2896, 1427, 1053, 663, 557 cm $^{-1}$.

NOTE: Sulphur combustion analysis calcd for 2,6-disubstituted cellulose sulphate, empirical formula $C_6H_8S_2O_{11}Na_2$: 17.51%. Found: 15.42% (88.06% of theoretical); sulphur combustion analysis calcd for 6-monosubstituted cellulose sulphate, empirical formula $C_6H_9SO_8Na$: 12.14%. Found: 15.42% (27.01 % greater than theoretical); sulphur combustion analysis calcd for 2,3,6-trisubstituted cellulose sulphate, empirical formula $C_6H_7S_3O_{14}Na_2$: 20.54%. Found: 15.42% (24.92% less than theoretical). These data indicate that the sulphated cellulose product is predominantly the 2,6-disulphated cellulose rather than the 2,3,6-trisubstituted or 6-monosulphated cellulose.

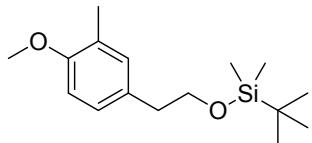
Cellulose sulphate (chlorosulphonic acid method): Sulphur content calcd for the empirical formula $C_6H_7S_3O_{14}Na_3$: 20.54%. Found: 0.42%; sulphur content: 2.04% of theoretical; FT-IR: 3331, 2891, 1315, 1201, 1159, 1023, 895, 558, and 433 cm $^{-1}$.

Tert-butyl(4-ethoxyphenethoxy)dimethylsilane (**3c**)



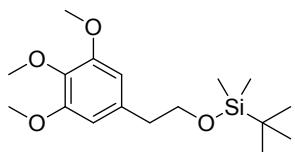
Colourless liquid, Yield: (95%), 1H NMR (400 MHz, $CDCl_3$): δ 7.15 (d, $J = 8.0$ Hz, 2H, Ar-H), 6.82 (t, $J = 8.0, 4.0$ Hz, 2H, Ar-H), 4.01 (q, $J = 8.0$ Hz, 2H, -OCH $_2$), 3.76 (t, $J = 7.43, 7.04$ Hz, 2H, -OCH $_2$), 2.74 (t, $J = 8.0, 4.0$ Hz, 2H, -CH $_2$), 1.40 (t, $J = 8.0$ Hz, 3H, -CH $_3$), 0.88 (s, 9H, t-Bu), -0.01 (s, 6H, $(CH_3)_2Si$) ppm; ^{13}C -NMR ($CDCl_3$, 100 MHz): δ 157.3, 131.0, 130.0, 114.2, 64.8, 63.3, 38.7, 25.9, 18.3, 14.8, -5.4 ppm; ESI-MS (m/z): 281.0447 ($M+H$) $^+$.

Tert-butyl (4-methoxy-3-methylphenethoxy) dimethylsilane (**3d**)



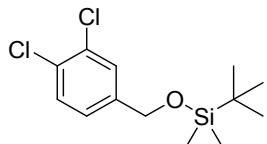
Colourless liquid, Yield: (95%), ^1H NMR (400 MHz, CDCl_3): δ 7.00-6.98 (m, 2H, Ar-H), 6.73 (t, $J = 8.0$ Hz, 1H, Ar-H), 3.80 (s, 3H, -OCH₃), 3.75 (t, $J = 4$ Hz, 2H, -OCH₂), 2.74 (t, $J = 8$ Hz, 2H, -CH₂), 2.20 (s, 3H, Ar-CH₃), 0.89 (s, 9H, t-Bu), 0.01 (s, 6H, (CH₃)₂-Si) ppm; ^{13}C -NMR (CDCl_3 , 100 MHz): δ 156.1, 131.5, 130.6, 127.0, 126.2, 109.7, 64.9, 55.3, 38.7, 25.9, 18.35, 16.1, -5.3 ppm; ESI-MS(m/z): 281.0610 ($\text{M}+\text{H})^+$, 304.0410 ($\text{M}+\text{Na})^+$.

Tert-butyldimethyl (3, 4, 5-trimethoxyphenethoxy)silane (3f)



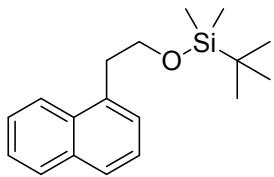
Colourless liquid, Yield: (85%), ^1H NMR (400 MHz, CDCl_3): δ 6.43 (s, 2H, Ar-H), 3.84 (s, 6H, 2 x -OCH₃), 3.82-3.78 (m, 5H, -OCH₃ and -OCH₂), 2.75 (t, $J = 8.0$ Hz, 4.0 Hz, 2H, CH₂), 0.87 (s, 9H, t-Bu), -0.01 (s, 6H, (CH₃)₂-Si) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 152.9, 136.3, 135.1, 106.0, 64.4, 60.7, 55.9, 39.8, 25.8, 18.2, -5.4 ppm; ESI-MS (m/z): 327.0410 ($\text{M}+\text{H})^+$, 349.0256 ($\text{M}+\text{Na})^+$.

Tert-butyl ((3, 4-dichlorobenzyl) oxy)dimethylsilane (3g)



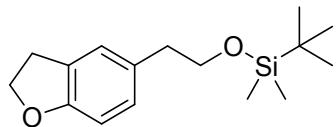
Colourless liquid, Yield: (85%), ^1H NMR (400 MHz, CDCl_3): δ 7.42 -7.41 (m, 1H, Ar-H), 7.39 (d, $J = 8.2$ Hz, 1H, Ar-H), 7.16-7.13 (m, 1H, Ar-H), 4.68 (s, 2H, -OCH₂), 0.95 (s, 9H, t-Bu), 0.11 (s, 6H, (CH₃)₂-Si) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 141.7, 132.2, 130.6, 130, 127.8, 125.1, 63.6, 25.8, 18.3, -5.3 ppm; ESI-MS(m/z): 291.022 ($\text{M}+\text{H})^+$.

Tert-butyldimethyl(2-(naphthalen-1-yl)ethoxy)silane (3h)



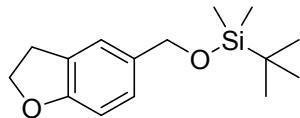
Colourless liquid; Yield: (88%), ^1H NMR (400 MHz, CDCl_3): δ 8.09-8.07 (m, 1H, Ar-H), 7.87-7.84 (m, 1H, Ar-H), 7.74 (d, J = 8.0 Hz, 1H, Ar-H), 7.54-7.45 (m, 2H, Ar-H), 7.43-7.35 (m, 2H, Ar-H), 3.97-3.93 (m, 2H, -OCH₂), 3.33 (t, J = 8.0 Hz, 2H, CH₂), 0.88 (s, 9H, t-Bu), -0.03 (s, 6H, (CH₃)₂-Si) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 135.0, 133.7, 132.2, 128.6, 127.1, 126.9, 125.7, 125.4, 125.3, 123.8, 63.9, 36.6, 25.9, 18.3, -5.4 ppm; ESI-MS (m/z): 287.0406 (M+H)⁺, 310.294 (M+Na)⁺.

Tert-butyl (2-(2, 3-dihydrobenzofuran-5-yl)ethoxy)dimethylsilane (3i)



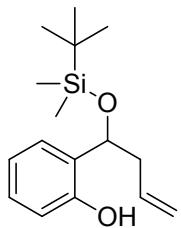
Colourless liquid, Yield: (92%), ^1H NMR (400 MHz, CDCl_3): δ 7.04 (s, 1H, Ar-H), 6.94-6.92 (m, 1H, Ar-H), 6.70 (d, J = 8.0 Hz, 1H, Ar-H), 4.54 (t, J = 8.99, 8.66 Hz, 2H, -OCH₂), 3.75 (t, J = 8.0 Hz, 2H, -OCH₂), 3.17 (t, J = 8.6 Hz, 2H, CH₂), 2.75 (t, J = 12.0, 8.0 Hz, 7.04 Hz, 2H, CH₂), 0.89 (s, 9H, t-Bu), 0.01 (s, 6H, (CH₃)₂-Si) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 158.4, 130.9, 128.4, 126.7, 125.6, 108.8, 71.1, 64.9, 39.01, 29.7, 25.9, 18.3, -5.3 ppm; ESI-MS (m/z): 279.0549 (M+H)⁺, 301.0227 (M+Na)⁺.

Tert-butyl ((2, 3-dihydrobenzofuran-5-yl) methoxy)dimethylsilane (3j)



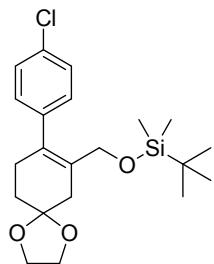
Colourless liquid, Yield: (75%), ^1H NMR (400 MHz, CDCl_3): δ 7.17 (s, 1H, Ar-H), 7.06-7.03 (m, 1H, Ar-H), 6.74 (d, J = 8.0 Hz, 1H, Ar-H), 4.65 (s, 2H, -OCH₂), 4.56 (t, J = 8.0 Hz, 2H, -OCH₂), 3.20 (t, J = 8.0 Hz, 2H, CH₂), 0.94 (s, 9H, t-Bu), 0.10 (s, 6H, (CH₃)₂-Si) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 159.1, 133.4, 126.9, 126.2, 123.2, 108.78, 71.2, 65.0, 29.7, 26.0, 18.4, -5.1 ppm; ESI-MS (m/z): 287.0778 (M+Na)⁺.

2-((Tert-butyldimethylsilyl) oxy) but-3-en-1-ylphenol (3k)



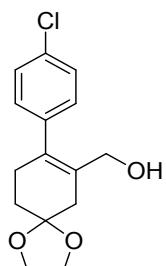
Colourless liquid, Yield: (82%), ¹H NMR (400 MHz, CDCl₃): δ 8.18 (s, 1H, Ar-OH), 7.17-7.13 (m, 1H, Ar-H), 6.89-6.85 (m, 2H, Ar-H), 6.81-6.77 (m, 1H, Ar-H), 5.78-5.67 (m, 1H, -OCH), 5.08 (d, *J* = 1.17 Hz, 1H, Vinyl-CH), 5.05-5.03 (m, 1H, Vinyl-CH), 4.76 (dd, *J* = 8.0 Hz, 1H, Vinyl-CH), 2.65-2.45 (m, 2H, Allyl-CH), 0.90 (s, 9H, t-Bu), 0.14 (s, 3H, CH₃-Si), 0.02 (s, 3H, CH₃-Si) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 155.7, 134.1, 128.6, 127.0, 126.8, 119.2, 117.9, 117.1, 77.7, 43.2, 25.6, 18.1, -5.0, -5.1 ppm; ESI-MS(*m/z*): 279.0549 (M+H)⁺, 301.0227 (M+Na)⁺.

Tert-Butyl((8-(4-chlorophenyl)-1,4-dioxaspiro[4.5]dec-7-en-7-yl)methoxy)dimethylsilane (3l)



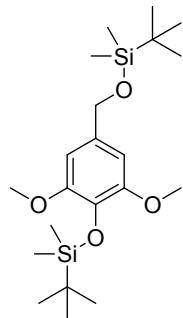
Colourless liquid, Yield: 95%, ¹H-NMR (CDCl₃, 400MHz): δ 7.28 (t, *J* = 4.0 Hz, 1H, Ar-H), 7.26 (t, *J* = 4.0 Hz, 1H, Ar-H), 7.12 (t, *J* = 4.0 Hz, 1H, Ar-H), 7.10 (t, *J* = 4.0 Hz, 1H, Ar-H), 4.04 – 4.01 (m, 4H, -(OCH₂)₂), 3.94 (s, 2H, -OCH₂), 2.48 (dd, *J* = 8.0, 4.0 Hz, 4H, 2x CH₂), 1.86 (t, *J* = 4.0 Hz, 2H, -CH₂), 0.85 (s, 9H, t-Bu), -0.05 (s, 6H, (CH₃)₂-Si) ppm; ¹³C-NMR (CDCl₃, 100 MHz): δ 140.3, 132.6, 132.4, 131.2, 129.6, 128.1, 108.1, 64.4, 63.9, 37.0, 31.4, 31.1, 25.8, 18.2, -5.3 ppm; ESI-MS (*m/z*): 394.9949 (M+H)⁺, 416.9845 (M+Na)⁺.

(8-(4-Chlorophenyl)-1,4-dioxaspiro[4.5]dec-7-en-7-yl)methanol (4l)



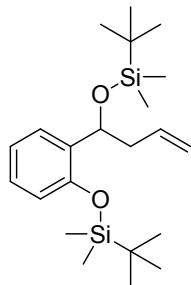
Colourless liquid, Yield: 91%, ¹H-NMR (CDCl₃, 400MHz): δ 7.29 (d, J = 8.0 Hz, 2H, Ar-H), 7.11 (d, J = 8.0 Hz, 2H, Ar-H), 4.05-4.01 (m, 4H, -(OCH₂)₂), 3.93 (s, 2H, -OCH₂), 2.50 (t, J = 12.0, 8Hz, 4H, Allyl-CH₂), 1.87 (t, J = 4.0 Hz, 1H, -CH₂) ppm; ¹³C-NMR (CDCl₃, 100 MHz): δ 140.0, 134.6, 132.6, 130.7, 129.5, 128.3, 107.9, 64.4, 63.4, 37.0, 31.4, 31.3 ppm; ESI-MS (m/z): 281.9937 (M+2H)⁺, 302.9126 (M+Na)⁺.

Tert-butyl ((4-((tert-butyldimethylsilyl) oxy)-3, 5-dimethoxybenzyl) oxy)dimethylsilane (5d)



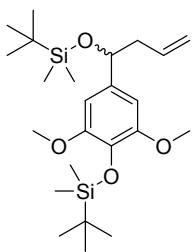
Colourless liquid, Yield: (82%), ¹H NMR (400 MHz, CDCl₃): δ 6.52 (s, 2H, Ar-CH), 4.66 (s, 2H, -OCH₂), 3.78 (s, 6H, -(OCH₃)₂), 1.00 (s, 9H, t-Bu), 0.94 (s, 9H, t-Bu), 0.11 (s, 6H, CH₃)₂-Si), 0.09 (s, 6H, (CH₃)₂-Si) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 151.4, 133.9, 133.0, 103.1, 65.1, 55.6, 25.9, 25.8, 18.7, 18.4, -4.6, -5.1 ppm; ESI-MS (m/z): 413.0653 (M+H)⁺, 435.0308 (M+Na)⁺.

Tert-butyl (2-(1-((tert-butyldimethylsilyl) oxy) but-3-en-1-yl) phenoxy) dimethylsilane (5e)



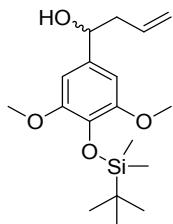
Colourless liquid, Yield: (90%), ¹H NMR (400 MHz, CDCl₃): δ 7.48 (dd, J = 8.0, 4.0 Hz, 1H, Ar-H), 7.10 (td, J = 8.0 Hz, 1H, Ar-H), 6.95 (t, J = 8.0 Hz, 1H, Ar-H), 6.77 (d, J = 8.0Hz, 1H, Ar-H), 5.94-5.84 (m, 1H, Vinyl-CH), 5.16 (dd, J = 4.0 Hz, 1H, Vinyl-CH), 5.05 (dd, J = 4.0 Hz, 1H, Vinyl-CH), 5.02 (s, -OCH), 2.47-2.32 (m, 2H, Allyl-CH₂), 1.06 (s, 9H, t-Bu), 0.92 (s, 9H, t-Bu), 0.31 (s, 3H, CH₃-Si), 0.27 (s, 3H, CH₃-Si), 0.05 (s, 3H, CH₃-Si), -0.08 (s, 3H, CH₃-Si) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 151.2, 135.8, 135.7, 127.3, 127.2, 120.8, 117.4, 116.5, 68.7, 43.8, 25.9, 25.8, 18.3, 18.2, -3.8, -4.2, -4.7, -4.9 ppm; ESI-MS (m/z): 415.0718 (M+Na)⁺.

Tert-butyl ((1-(4-((tert-butyldimethylsilyl) oxy)-3, 5-dimethoxyphenyl) but-3-en-1-yl) oxy) dimethylsilane (5f)

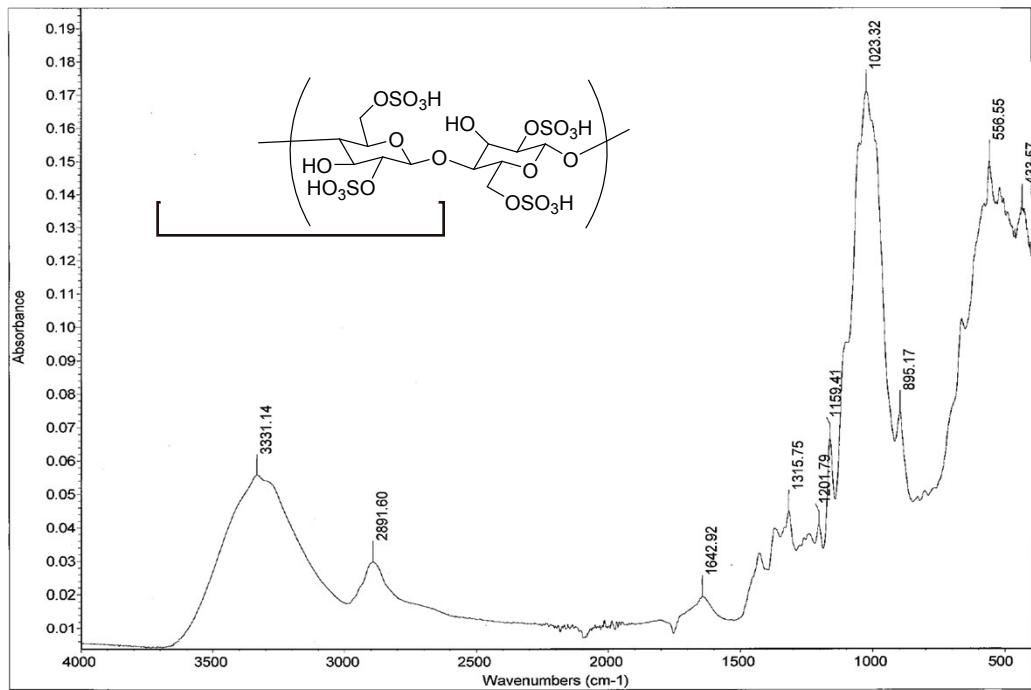


Colourless liquid, Yield: (88%), ¹H NMR (400 MHz, CDCl₃): δ 6.50 (s, 2H, Ar-H), 5.83-5.74 (m, 1H, -OCH), 5.05-5.00 (m, 2H, Vinyl-CH), 4.57 (dd, J = 4.0 Hz, 1H, -OCH), 3.78 (s, 6H, -(OCH₃)₂), 2.48-2.33 (m, 2H, Allyl-CH₂), 1.02 (s, 9H, t-Bu), 0.88 (s, 9H, t-Bu), 0.13 (s, 6H, Si-(CH₃)₂), 0.02 (s, 3H, Si-CH₃), -0.12 (s, 3H, Si-CH₃) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 151.1, 137.9, 135.5, 132.9, 116.5, 102.9, 75.2, 55.6, 44.5, 25.8, 18.7, 18.2, -4.6, -4.69, -4.7, -4.9 ppm; ESI-MS (m/z): 475.0574 (M+Na)⁺.

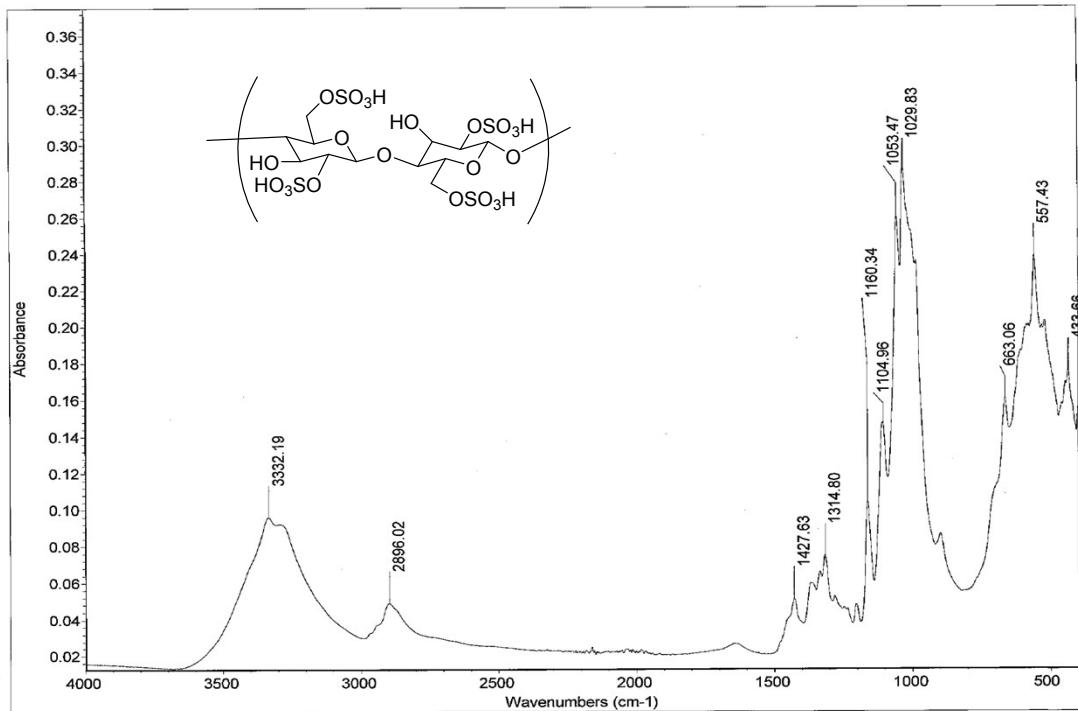
1-(4-((tert-Butyldimethylsilyl)oxy)-3,5-dimethoxyphenyl)but-3-en-1-ol (6f)



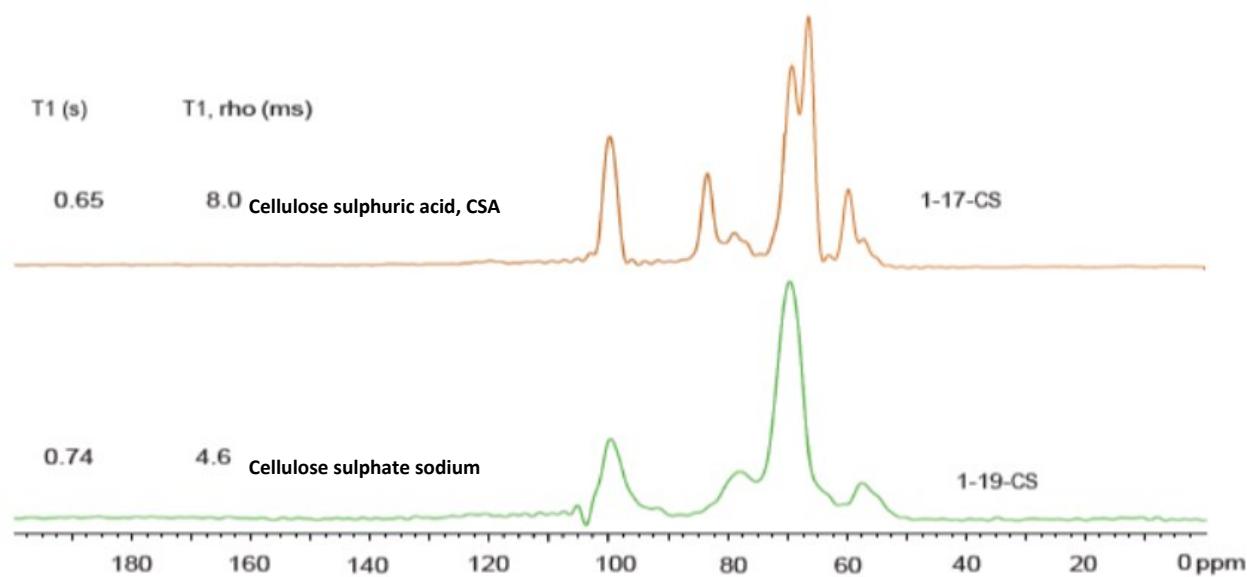
Colourless liquid, Yield: 88%, ¹H-NMR (CDCl₃, 400MHz): δ 6.55 (s, 2H, Ar-H), 5.86-5.76 (m, 1H, -Vinyl-CH), 5.15 (t, , J = 12 Hz, 2H, Vinyl-CH), 4.67-4.63 (m, 1H, -OCH), 3.80 (s, 6H, -(OCH₃)₂), 2.48 (dd, J = 8.0, 4.0 Hz, 2H, Allyl-CH₂), 1.98 (d, J = 4.0 Hz, 1H, -OH), 1.01 (s, 9H, t-Bu), 0.12 (s, 6H, , (CH₃)₂-Si) ppm; ¹³C-NMR (CDCl₃, 100MHz): δ 151.4, 136.4, 134.6, 133.3, 118.0, 102.7, 73.5, 55.6, 43.8, 25.7, 18.6, -4.6 ppm; ESI-MS (m/z): 339.0476 (M+H)⁺, 361.8842 (M+Na)⁺.



IR spectrum of HS-cellulose sulphate

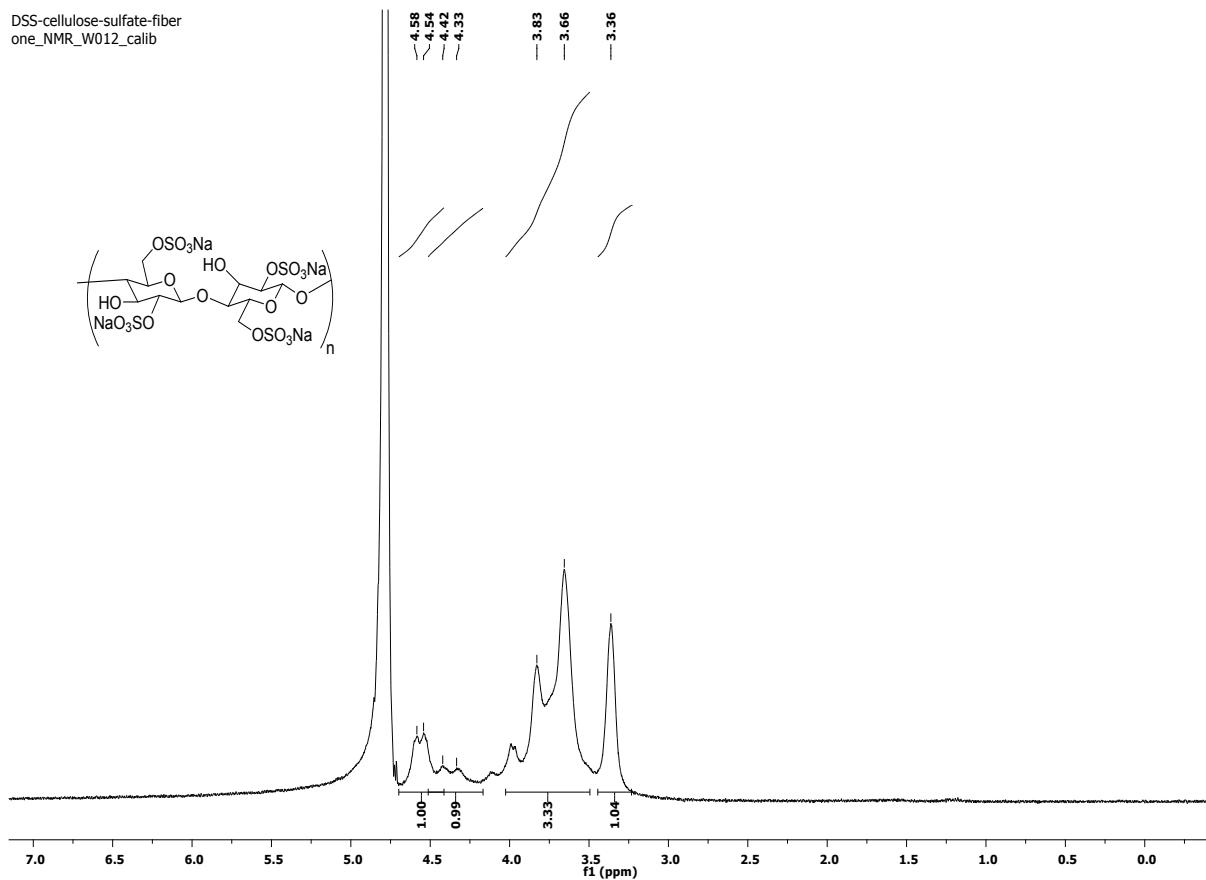


IR spectrum of HS-cellulose sulphate sodium



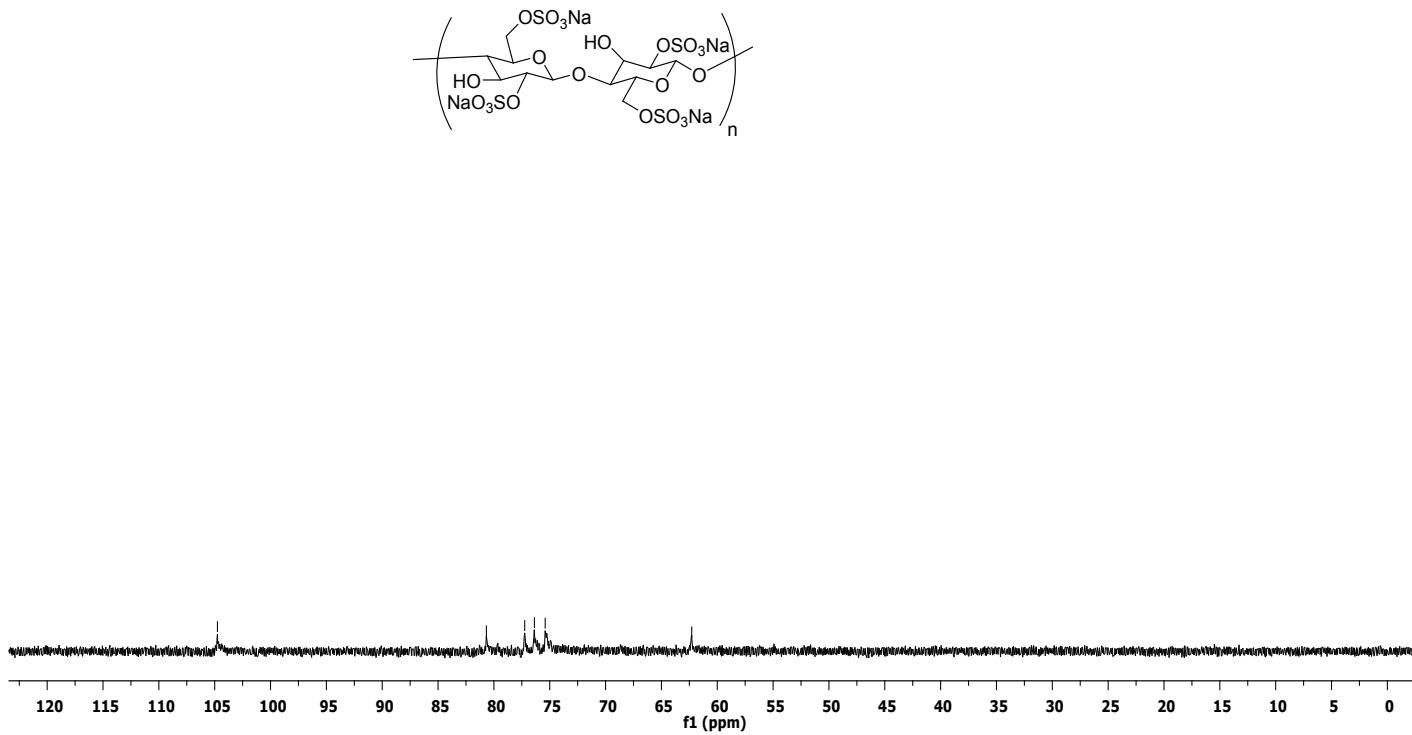
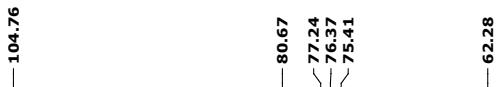
Solid state NMR spectrum of HS-cellulose sulphate sodium

DSS-cellulose-sulfate-fiber
one_NMR_W012_calib



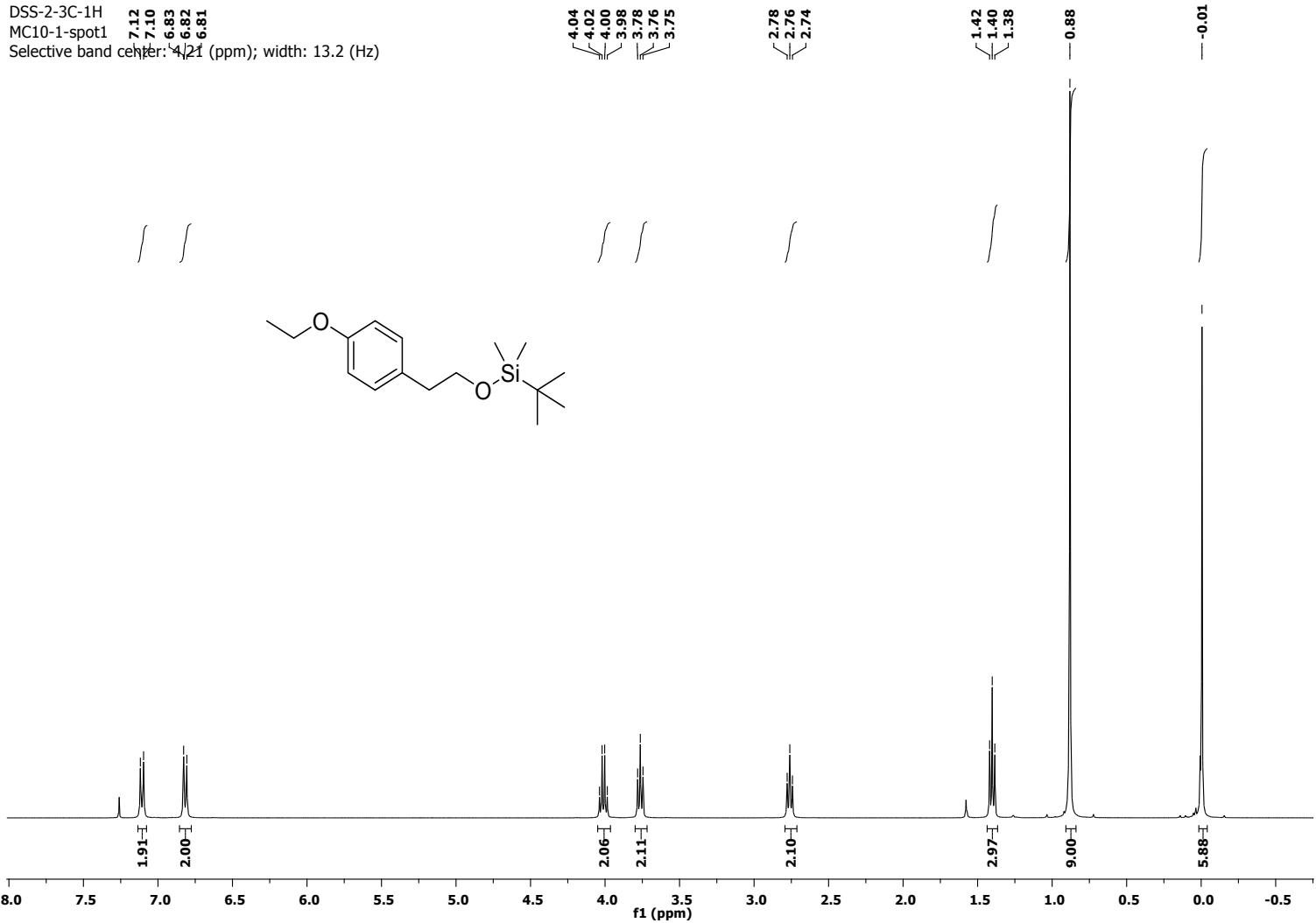
^1H NMR spectrum of HS-cellulose sulphate sodium in D_2O

DSS-cellulosesulfateCMR
one_NMR_W012_calib

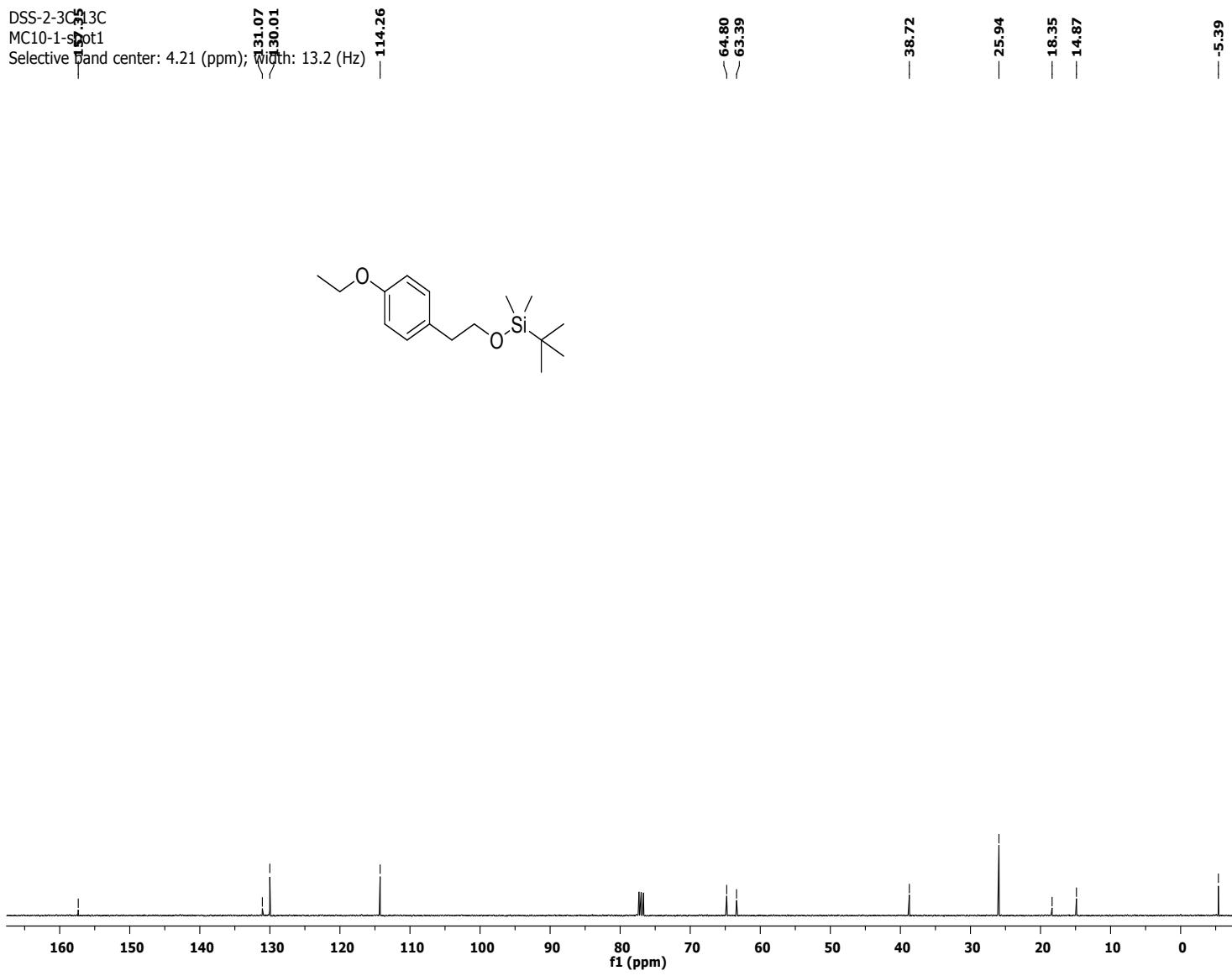


^{13}C NMR spectrum of compound cellulose sulphate sodium in D_2O

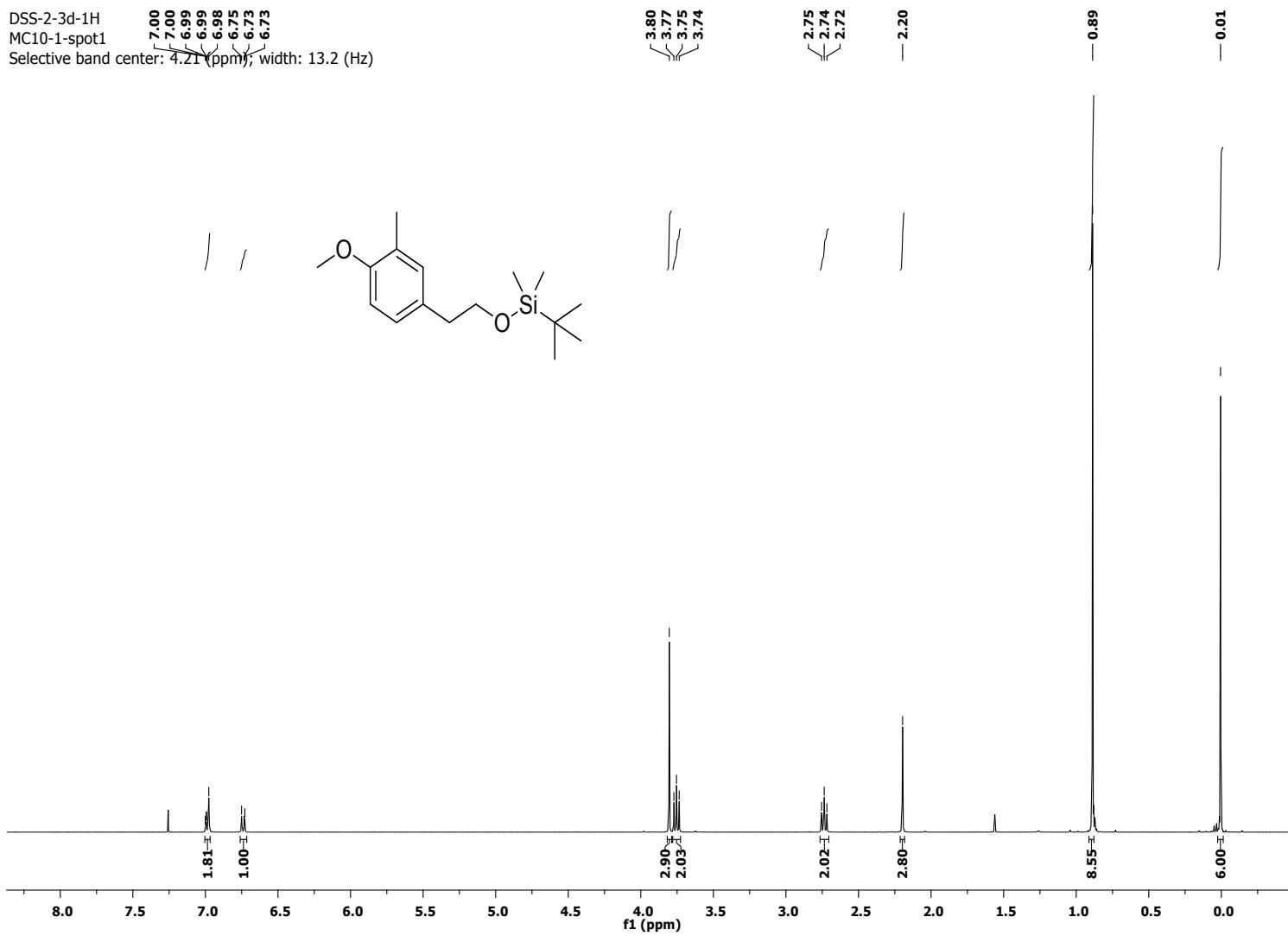
DSS-2-3C-1H
MC10-1-spot1
Selective band center: 4.21 (ppm); width: 13.2 (Hz)



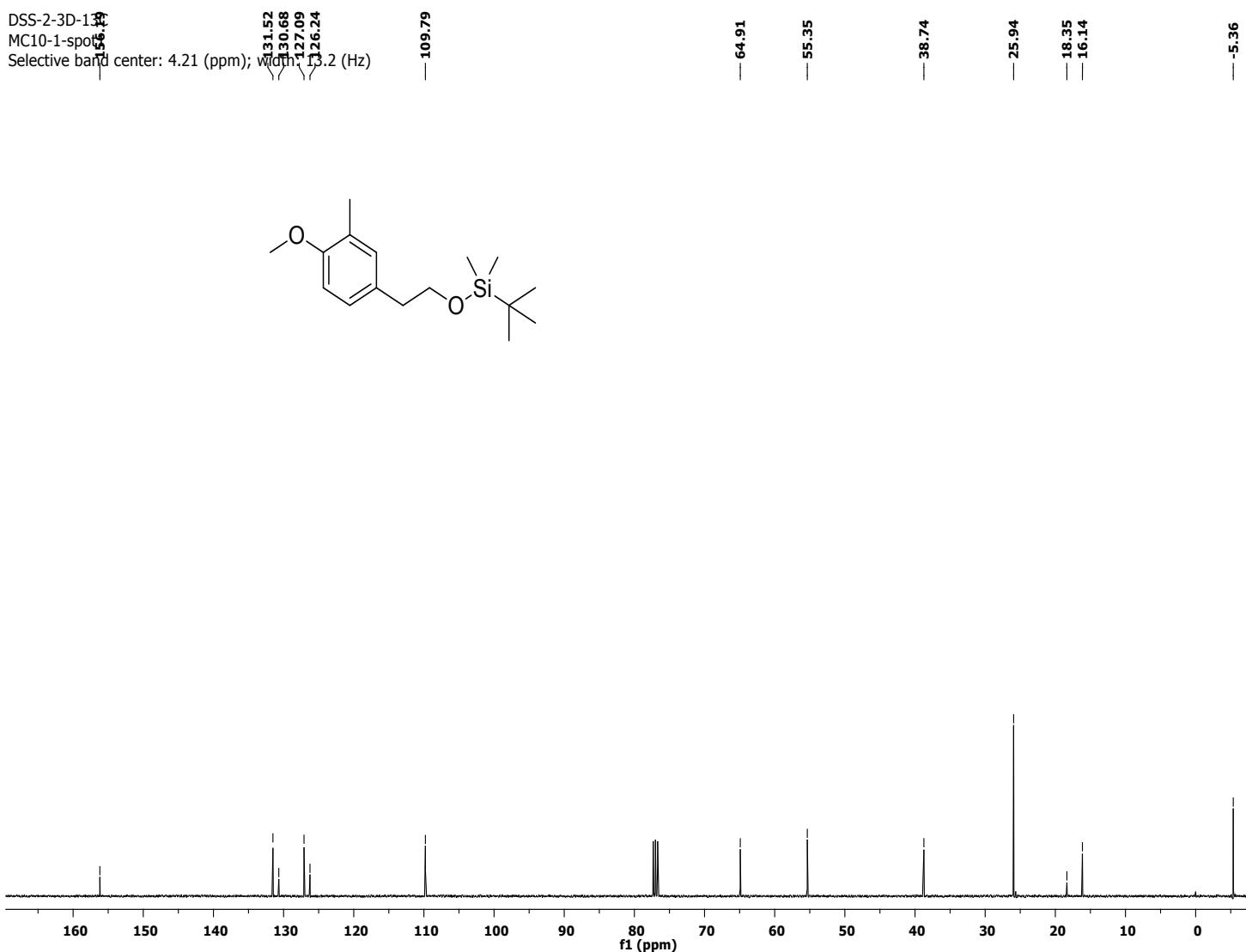
¹H NMR spectrum of compound 3c in CDCl₃



¹³C NMR spectrum of compound 3c in CDCl₃

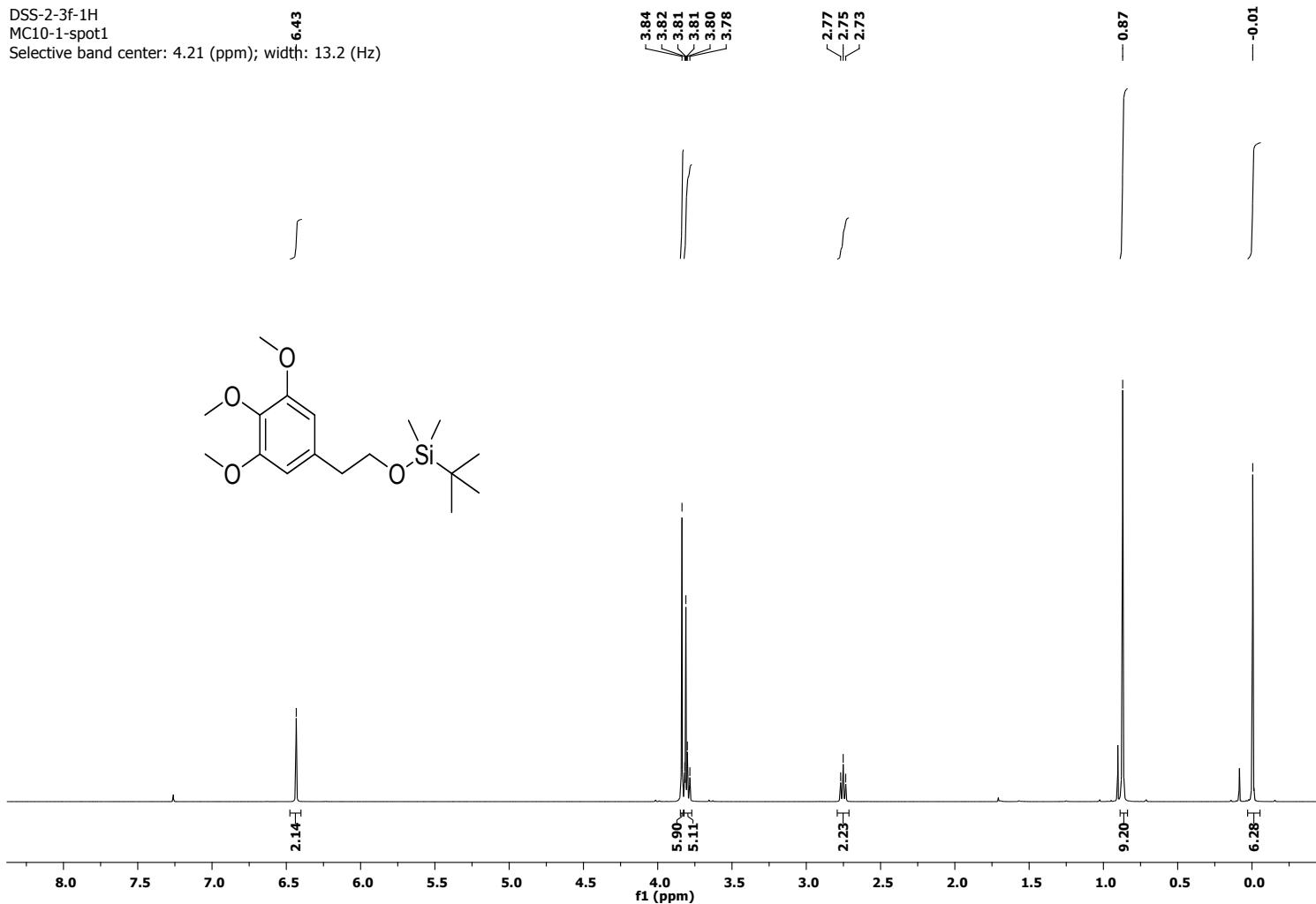


¹H NMR spectrum of compound 3d in CDCl₃



¹³C NMR spectrum of compound 3d in CDCl₃

DSS-2-3f-1H
MC10-1-spot1
Selective band center: 4.21 (ppm); width: 13.2 (Hz)



^1H NMR spectrum of compound 3f in CDCl_3

DSS-2-3f-13C
MC10-1-spot1
Selective band center: 4.21 (ppm); Width: 13.2 (Hz)

152.91

136.33

135.10

106.04

64.40

60.78

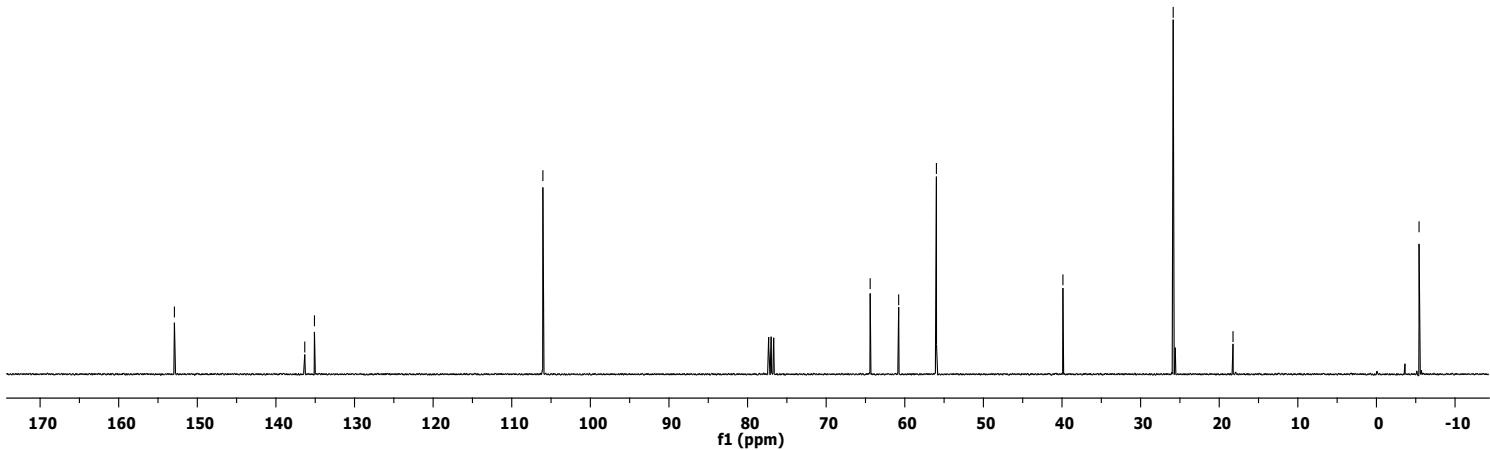
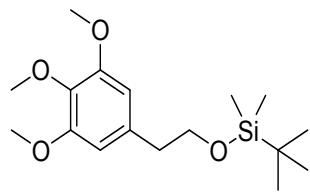
55.98

39.88

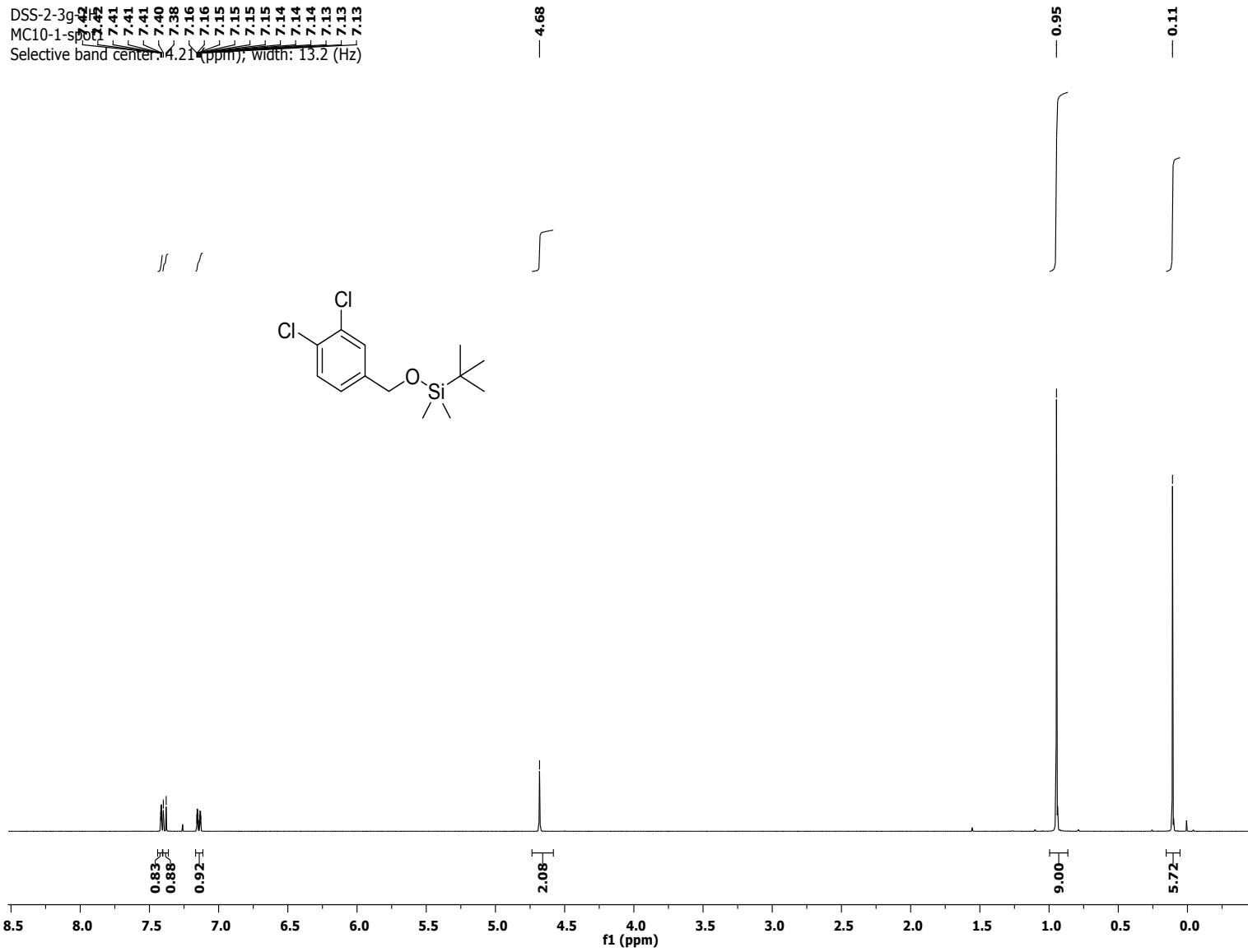
25.86

18.25

-5.42

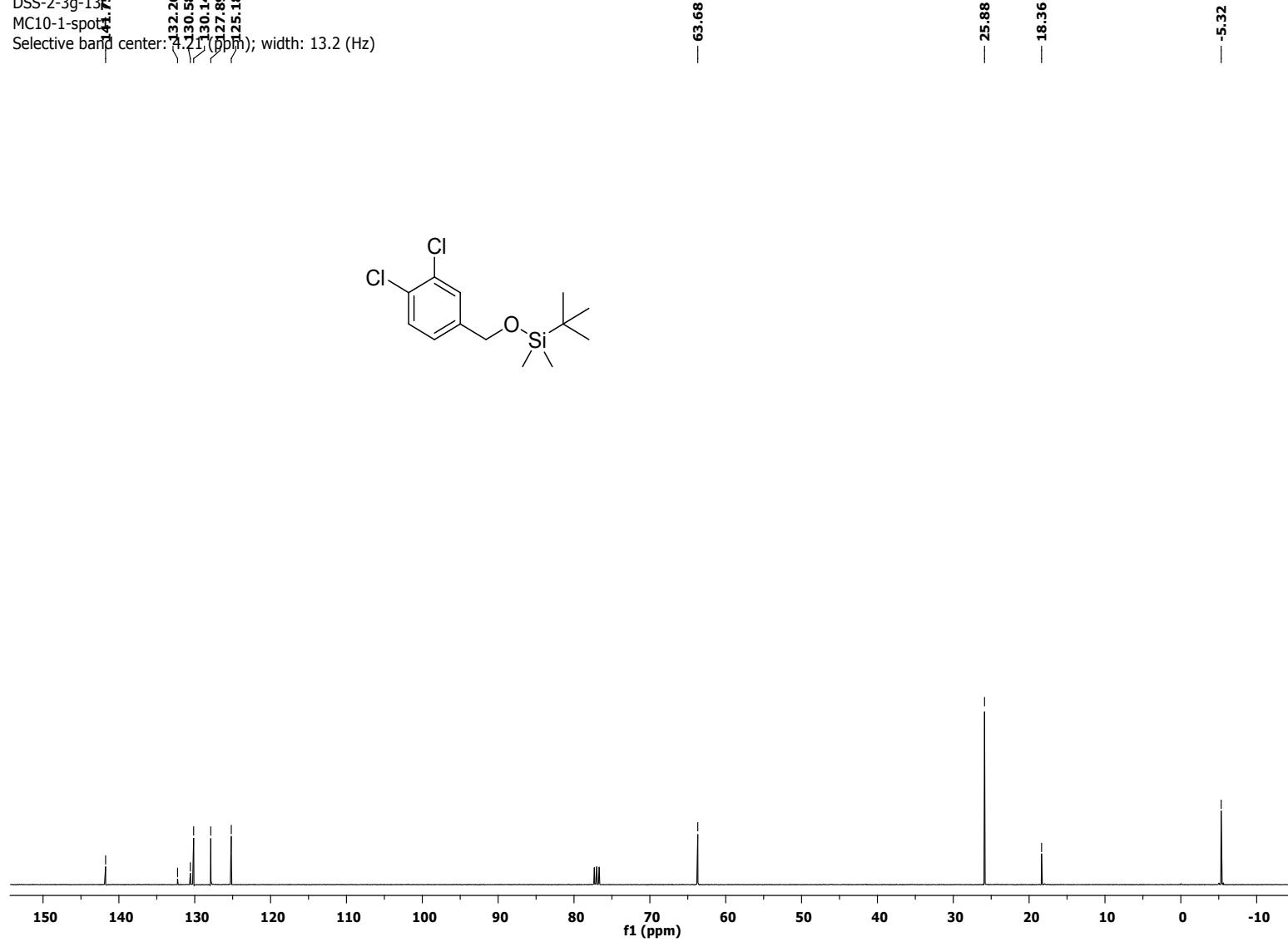


¹³C NMR spectrum of compound 3f in CDCl₃

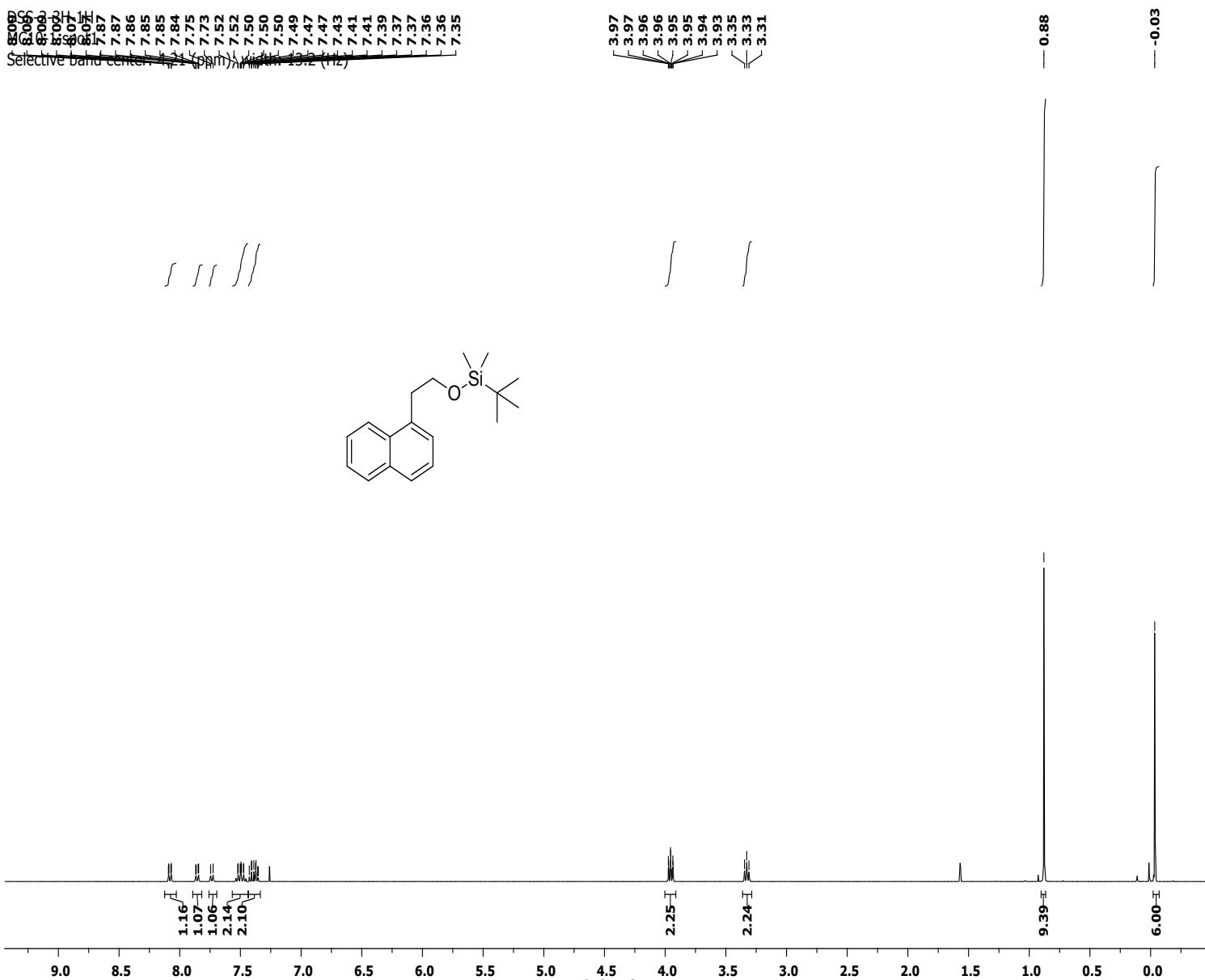


¹H NMR spectrum of compound 3g in CDCl₃

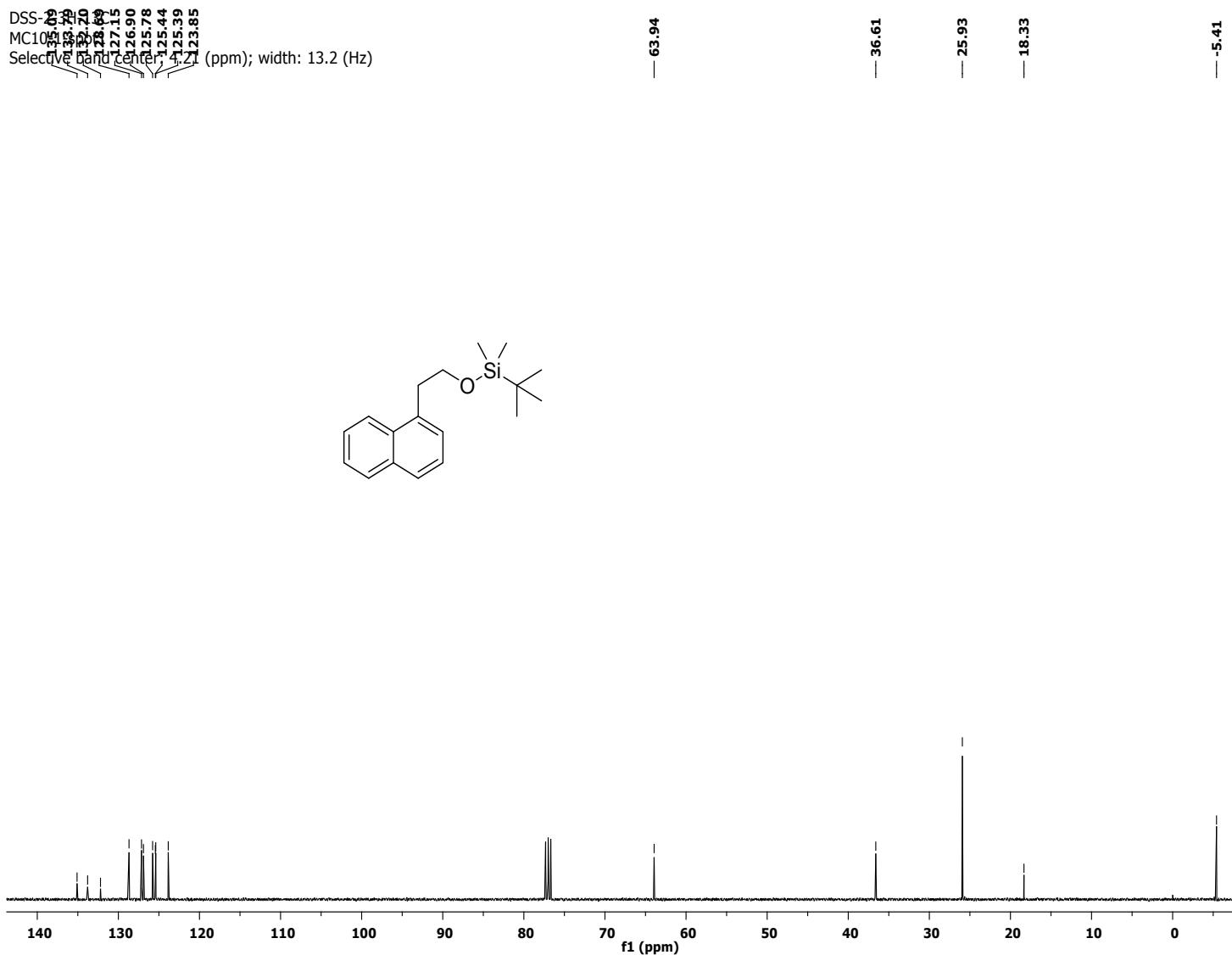
DSS-2-3g-13⁷⁵
MC10-1-spott
Selective band center: 4.21 (ppm); width: 13.2 (Hz)



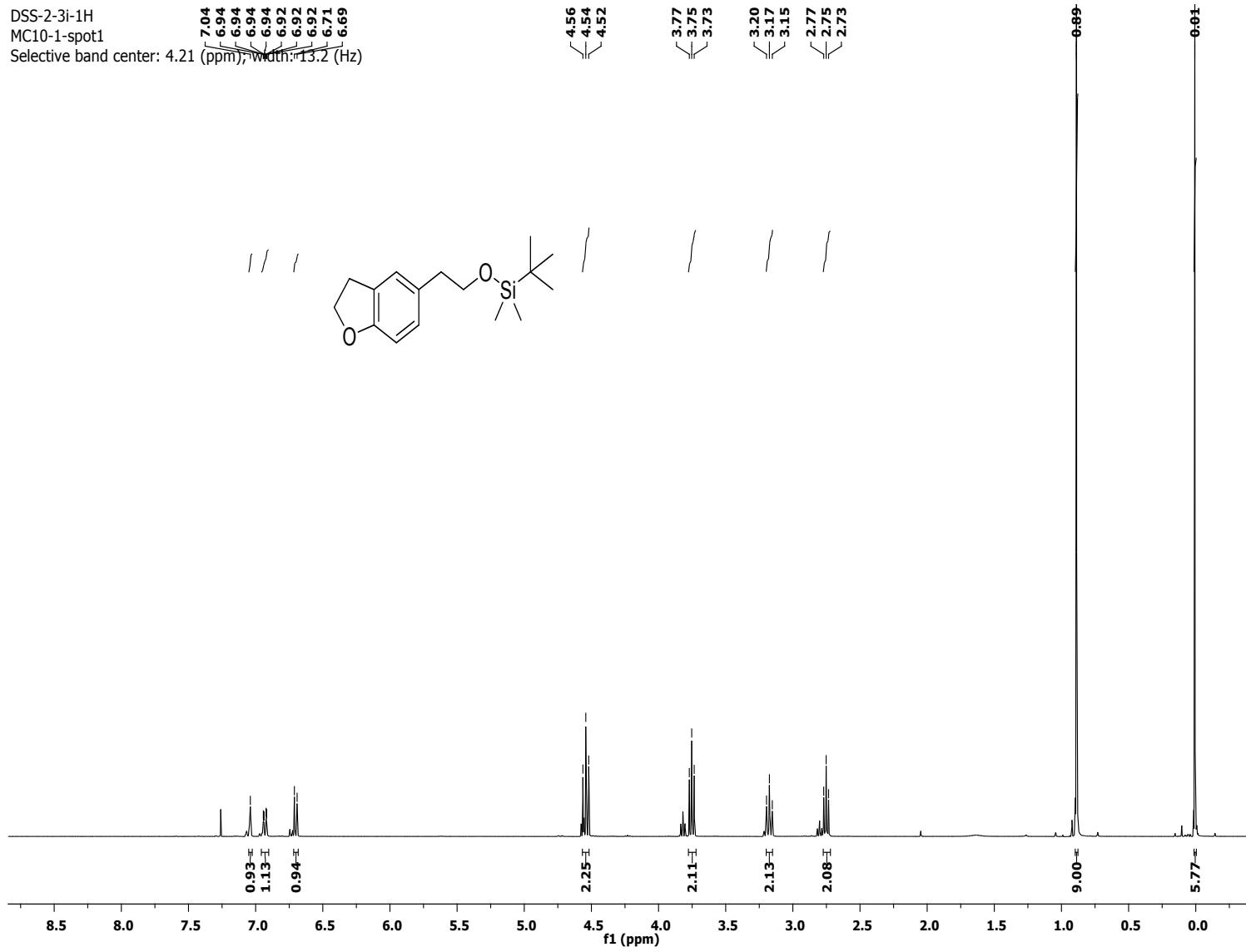
¹³C NMR spectrum of compound 3g in CDCl₃



¹H NMR spectrum of compound 3h in CDCl₃

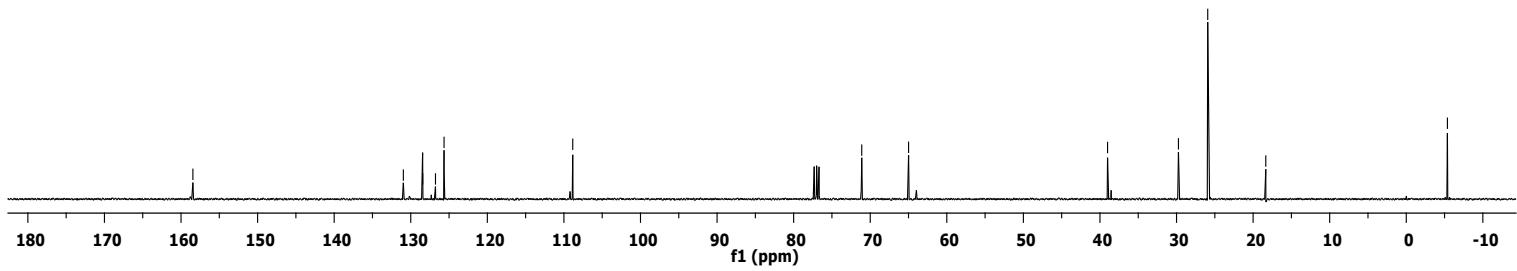
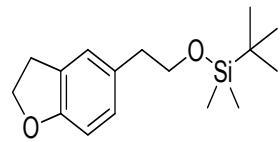


¹³C NMR spectrum of compound 3h in CDCl₃



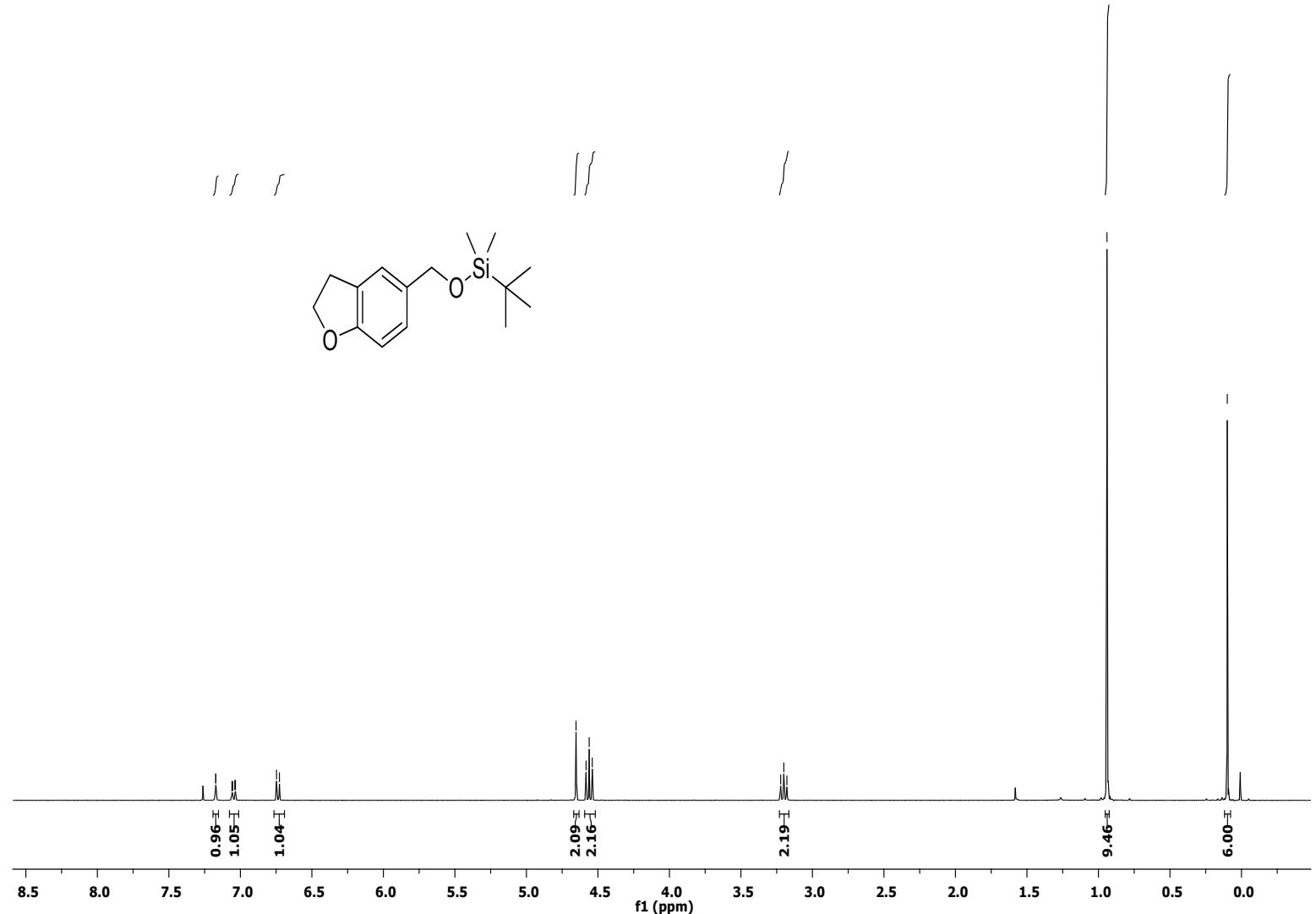
¹H NMR spectrum of compound 3i in CDCl₃

DSS-2-3i-13C
MC10-1-spot1
Selective band center: 14.21 (ppm); width: 13.158.45
130.97
128.44
126.77
125.65
108.85
71.11
64.99
39.01
29.75
25.93
18.35
-5.37



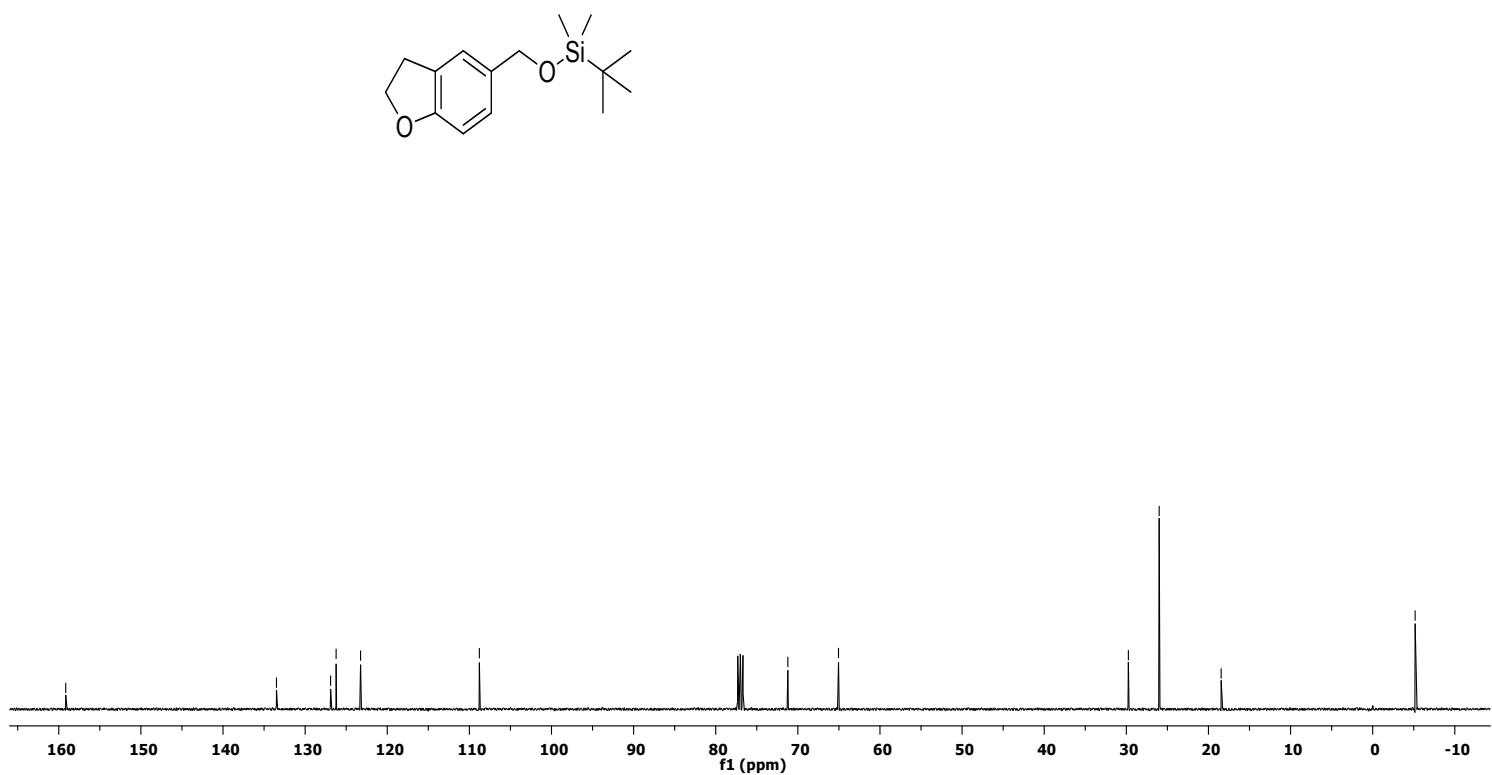
¹³C NMR spectrum of compound 3i in CDCl₃

DSS-2-3J-1H
MC10-1-spot1
Selective band center: 4.21 (ppm); width: 13.2 (Hz)

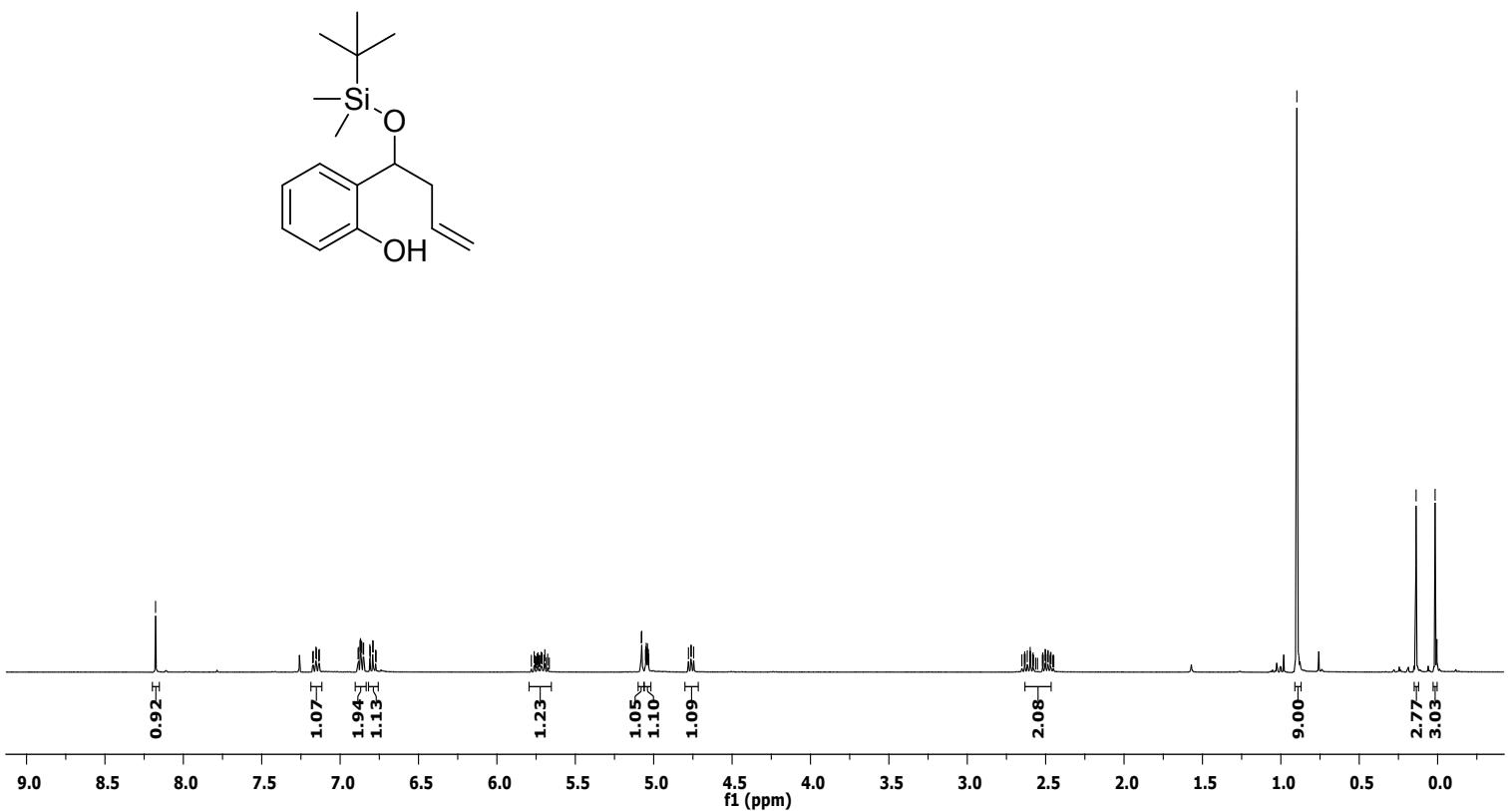


^1H NMR spectrum of compound 3j in CDCl_3

DSS-²⁹
MC10²⁹-spot1
Selective band center: 4.21 (ppm); width: 13.2 (Hz)



¹³C NMR spectrum of compound 3j in CDCl₃



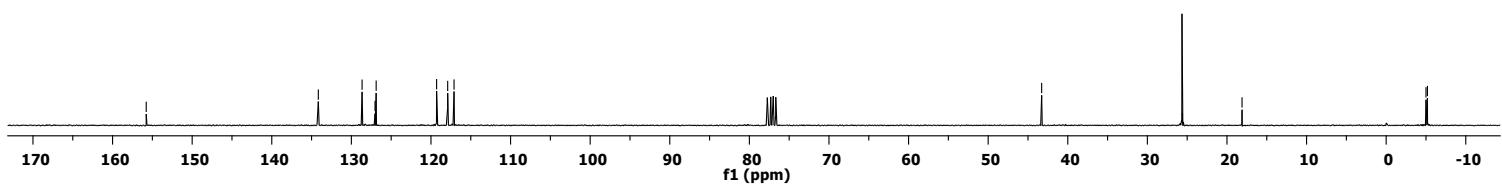
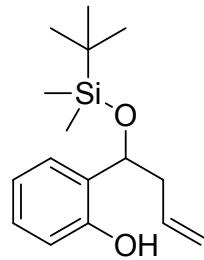
¹H NMR spectrum of compound 3k in CDCl₃

DSS-2-3k-13C
MC10-1-spot1
Selective band center: 4.21 (ppm); width: 55.77

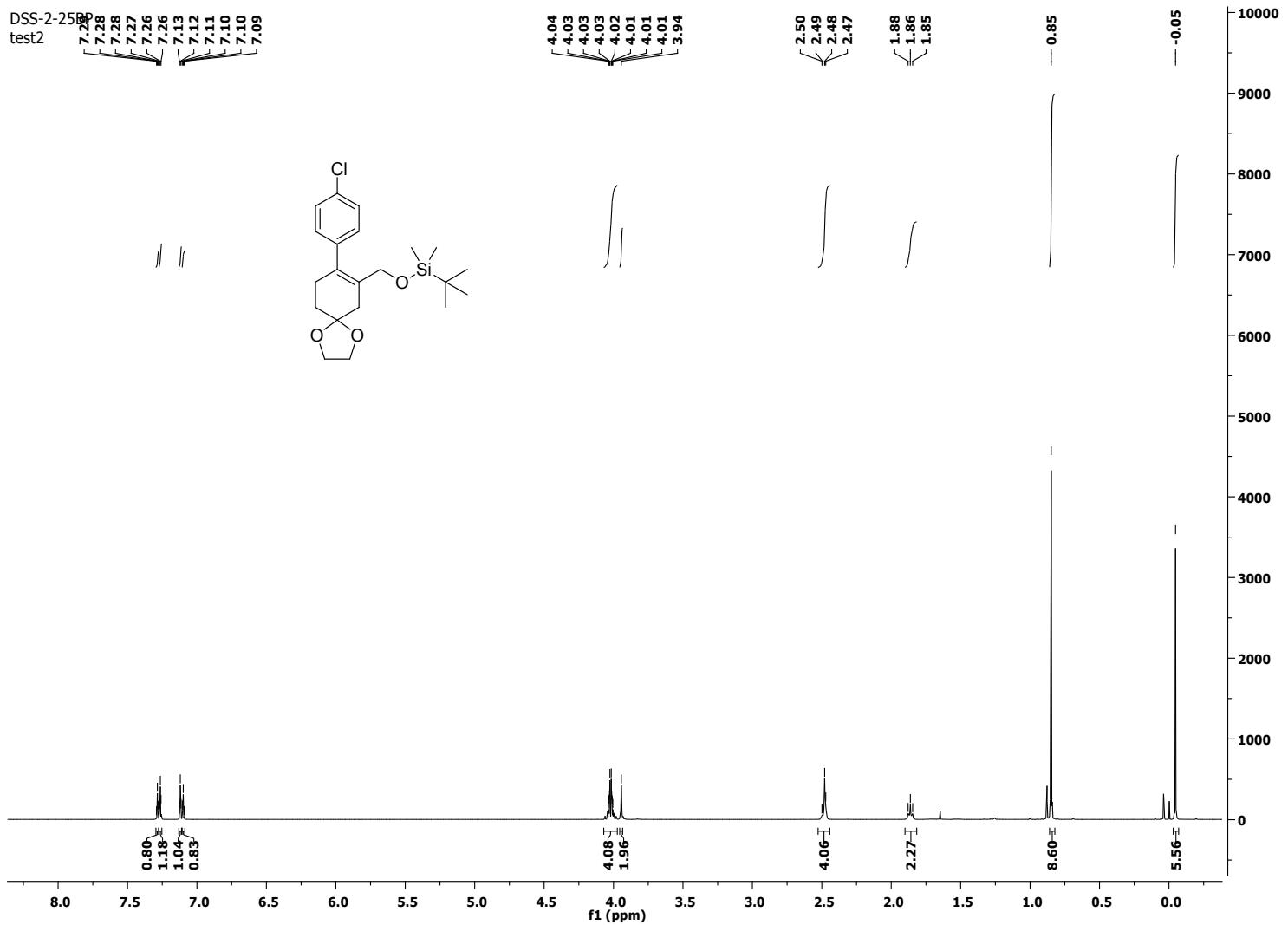
134.15
128.65
127.03
126.88
(H) 19.29
117.90
117.10

-43.28
-25.60
-18.11

< -5.00
< -5.18



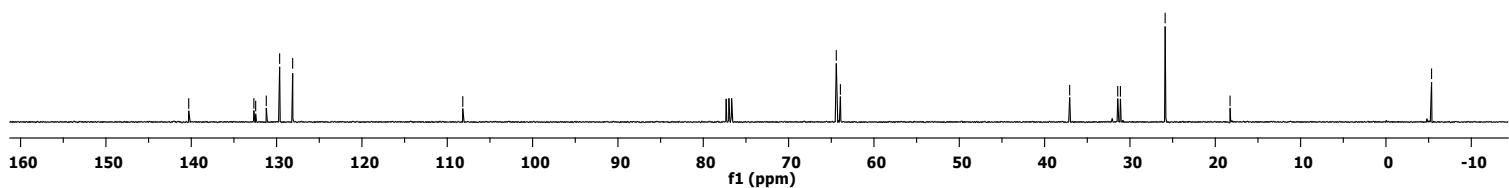
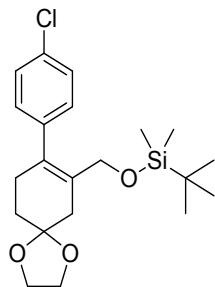
^{13}C NMR spectrum of compound 3k in CDCl_3



¹H NMR spectrum of compound 3l in CDCl₃

DSS-2-25BC
test2

— 140.30
— 132.66
— 132.45
— 131.21
— 129.65
— 128.12
— 108.18
— 64.41
— 63.93
— 37.06
— 31.43
— 31.12
— 25.88
— 18.27
— -5.34



¹³C NMR spectrum of compound 3l in CDCl_3

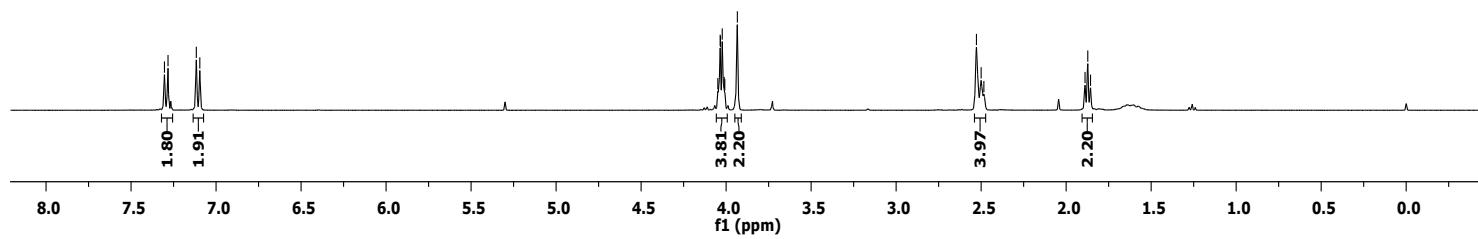
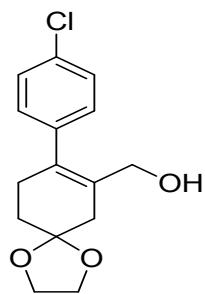
pp-165-38p

7.30
7.28
7.12
7.10

4.05
4.03
4.02
4.01
4.01
3.93

2.53
2.50
2.48

1.89
1.87
1.86

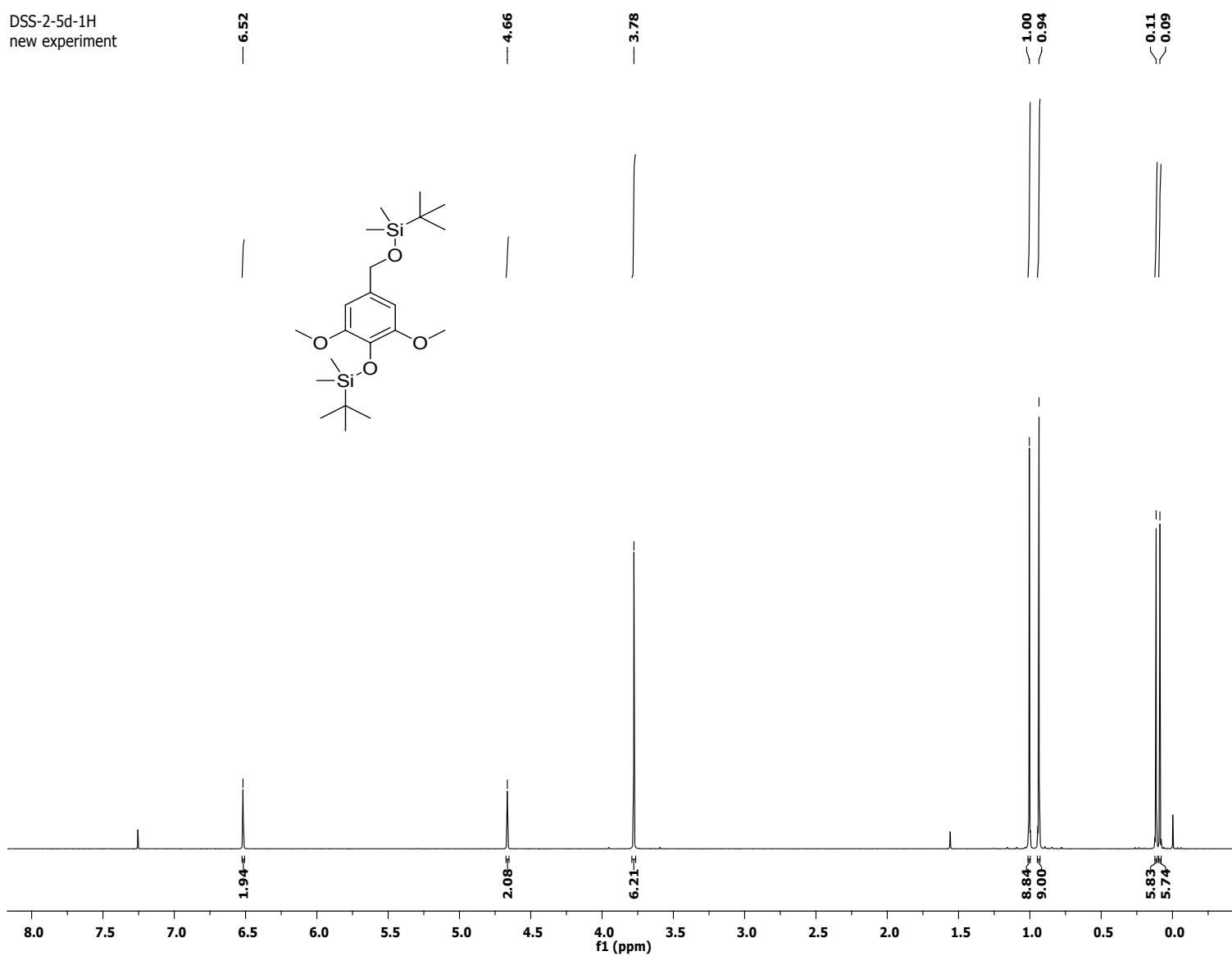


¹H NMR spectrum of compound 4l in CDCl₃

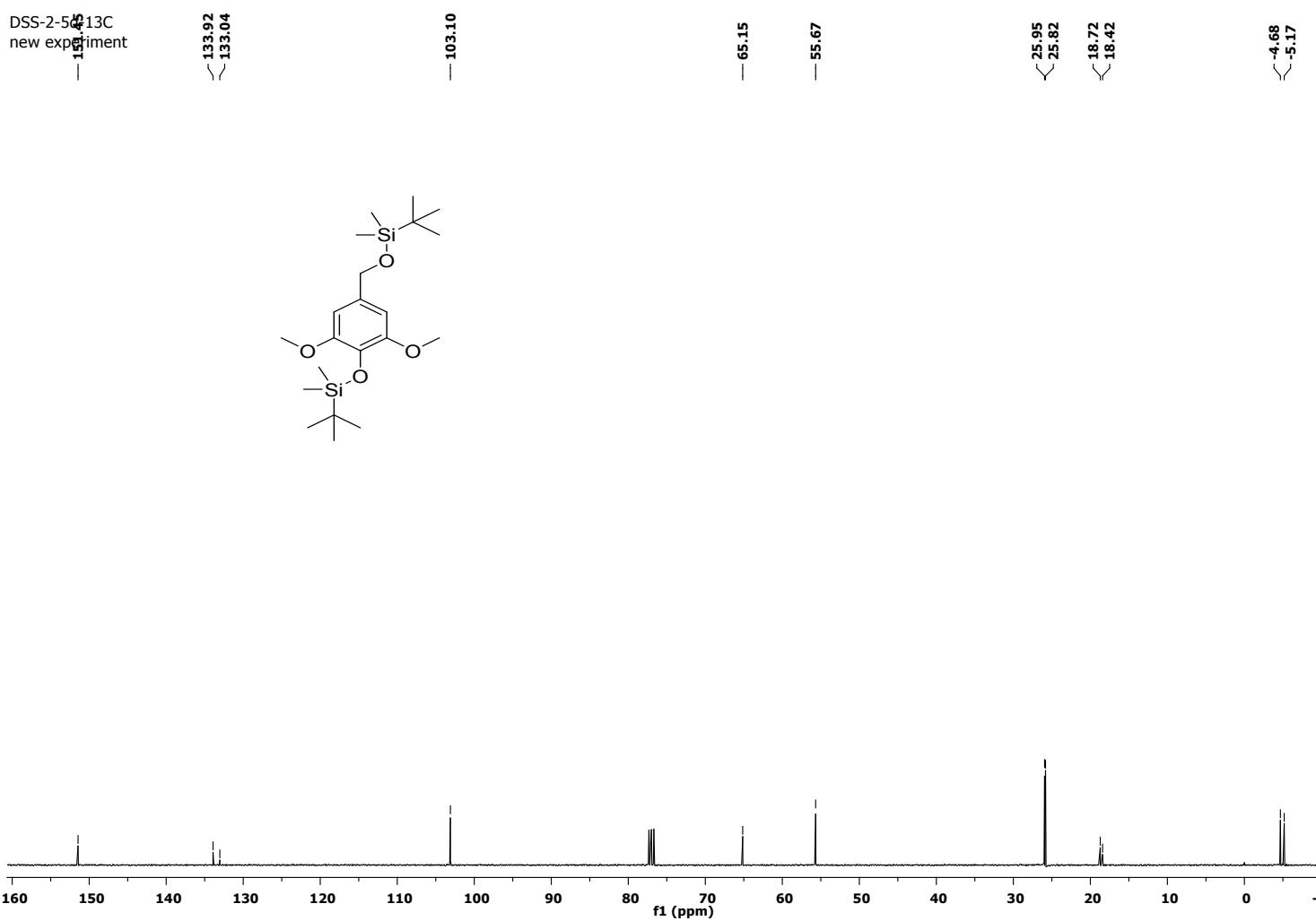


^{13}C NMR spectrum of compound 4l in CDCl_3

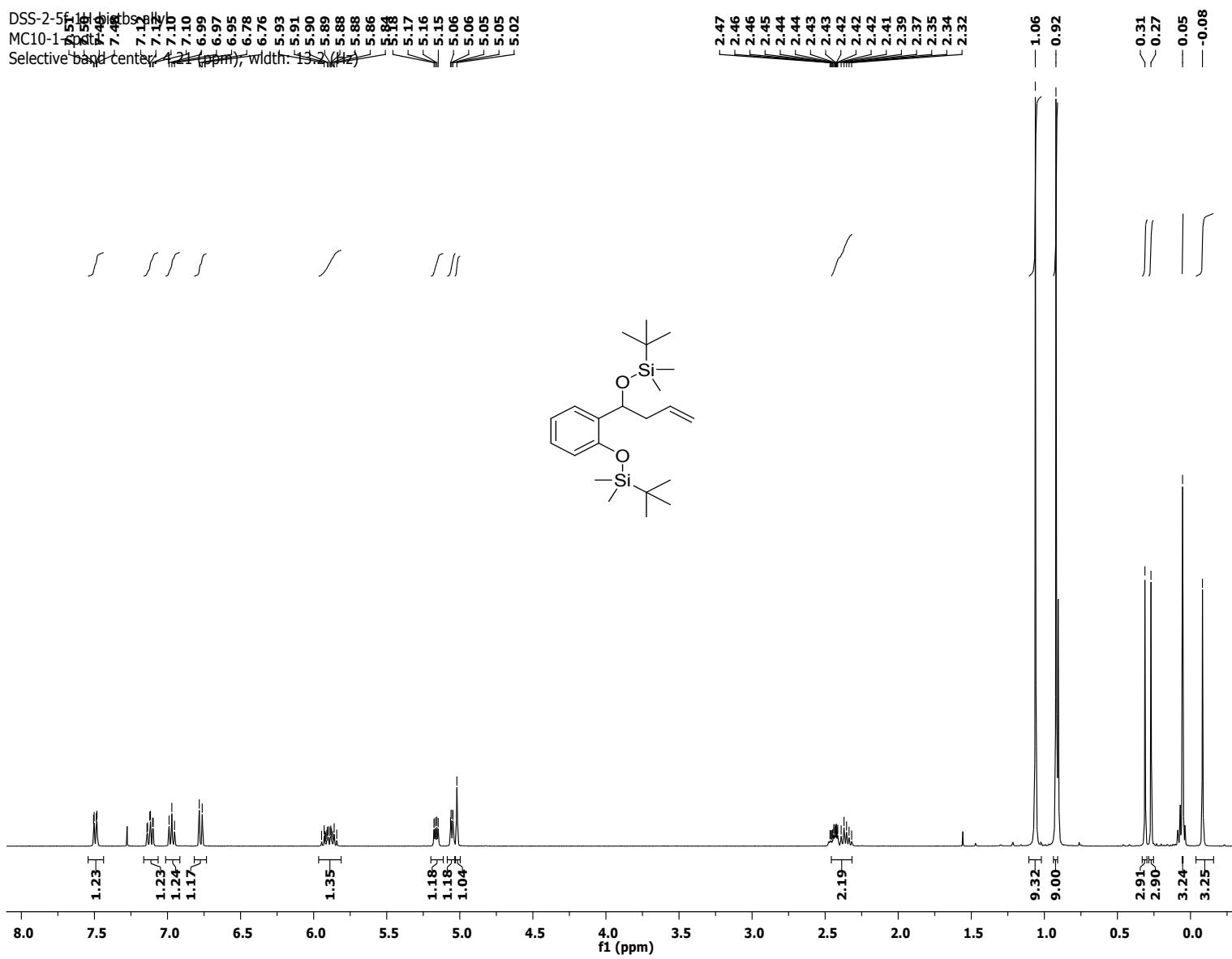
DSS-2-5d-1H
new experiment



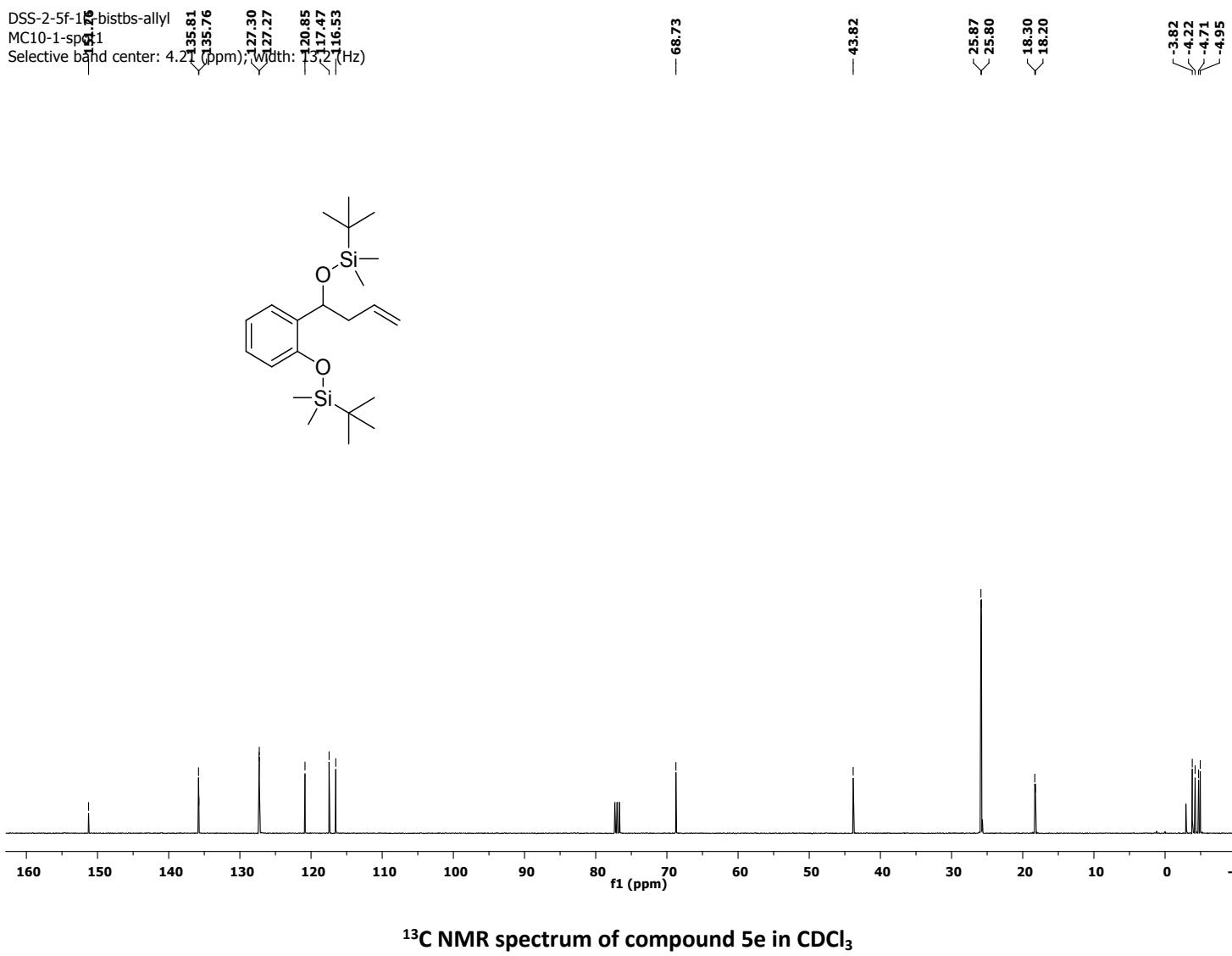
¹H NMR spectrum of compound 5d in CDCl_3



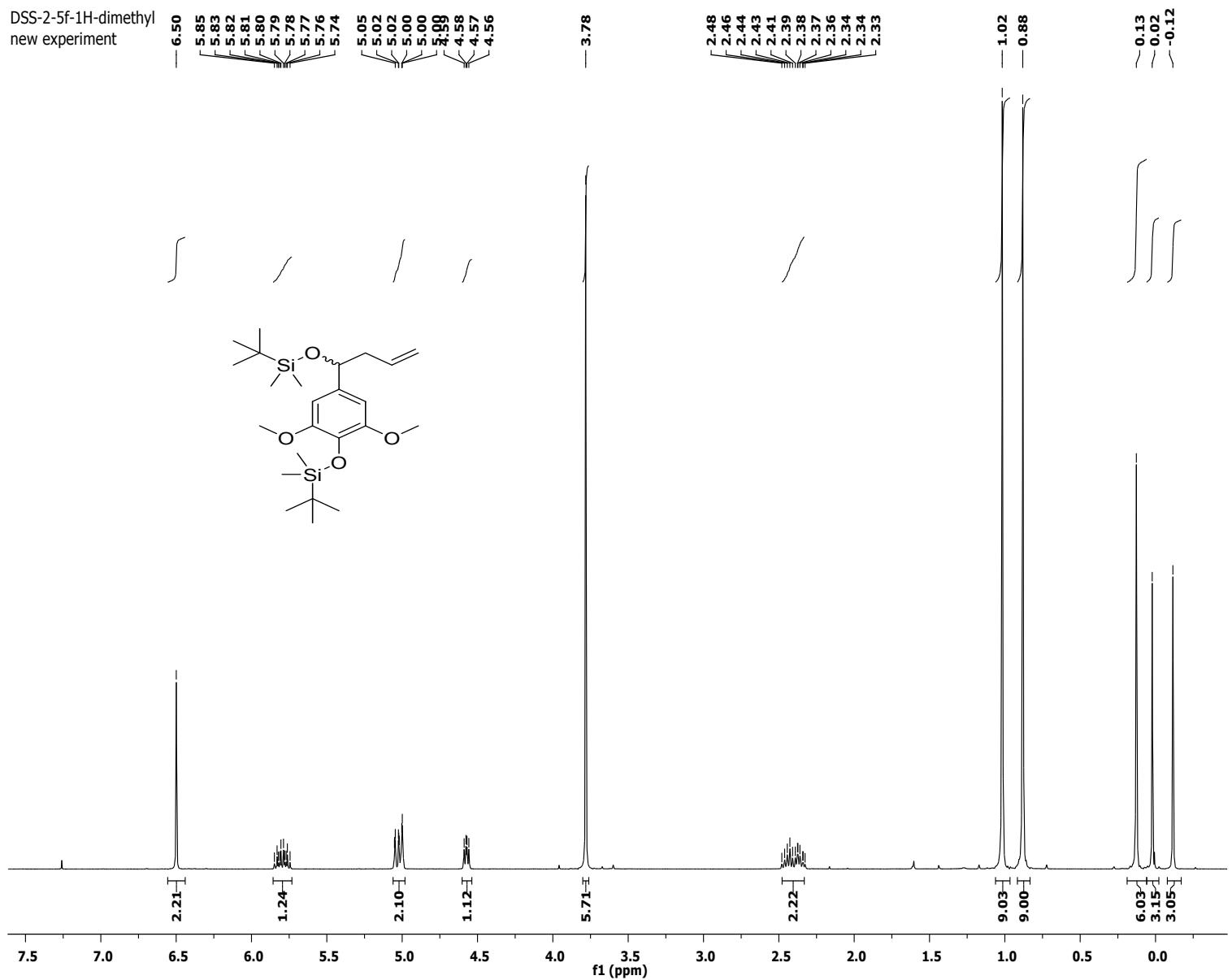
^{13}C NMR spectrum of compound 5d in CDCl_3



¹H NMR spectrum of compound 5e in CDCl₃



DSS-2-5f-1H-dimethyl
new experiment



¹H NMR spectrum of compound 5f in CDCl₃

DSS-2-5f-13C-dimethyl
new experiment

— 151.0

— 137.92
— 135.56
— 132.96

— 116.59

— 102.90

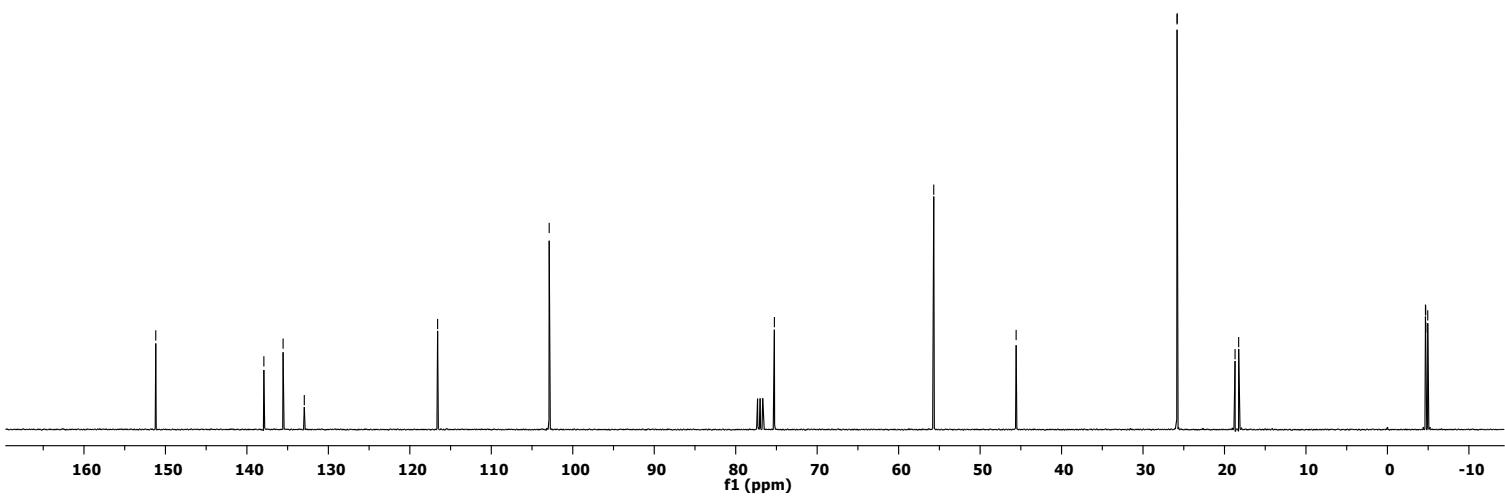
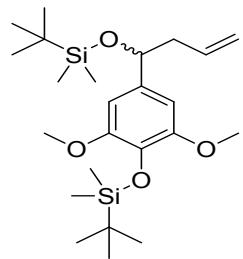
— 75.25

— 55.69

— 45.58

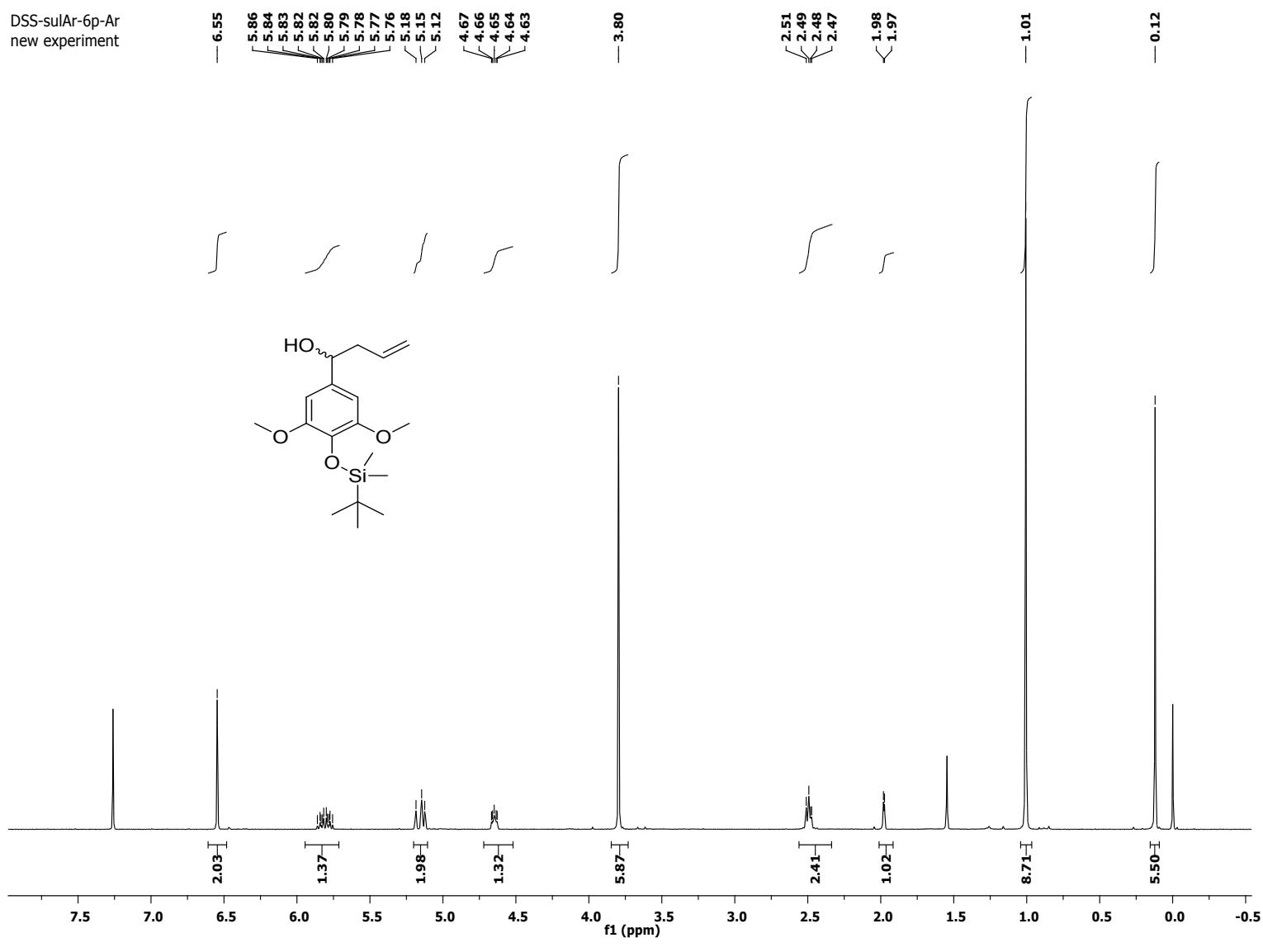
— 25.82
— 25.80
— 18.70
— 18.26

— -4.67
— -4.69
— -4.71
— -4.95



^{13}C NMR spectrum of compound 5f in CDCl_3

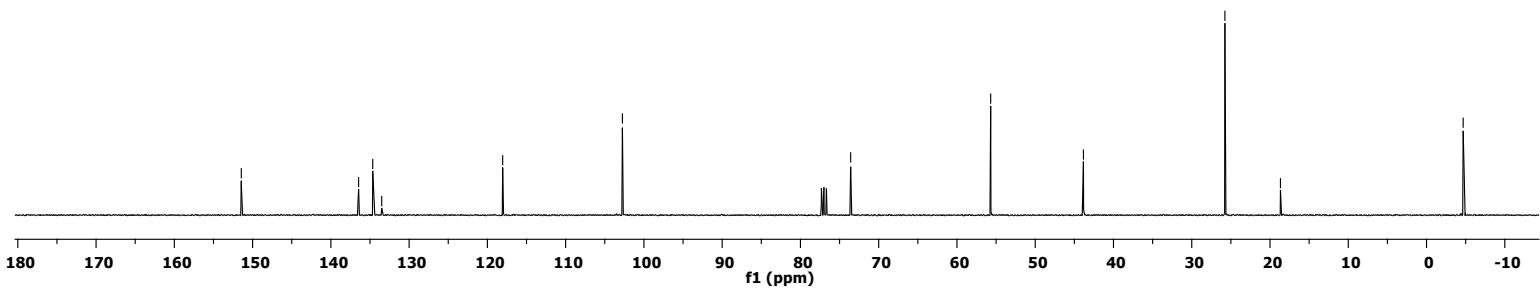
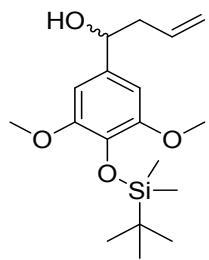
DSS-sulAr-6p-Ar
new experiment



^1H NMR spectrum of compound 6f in CDCl_3

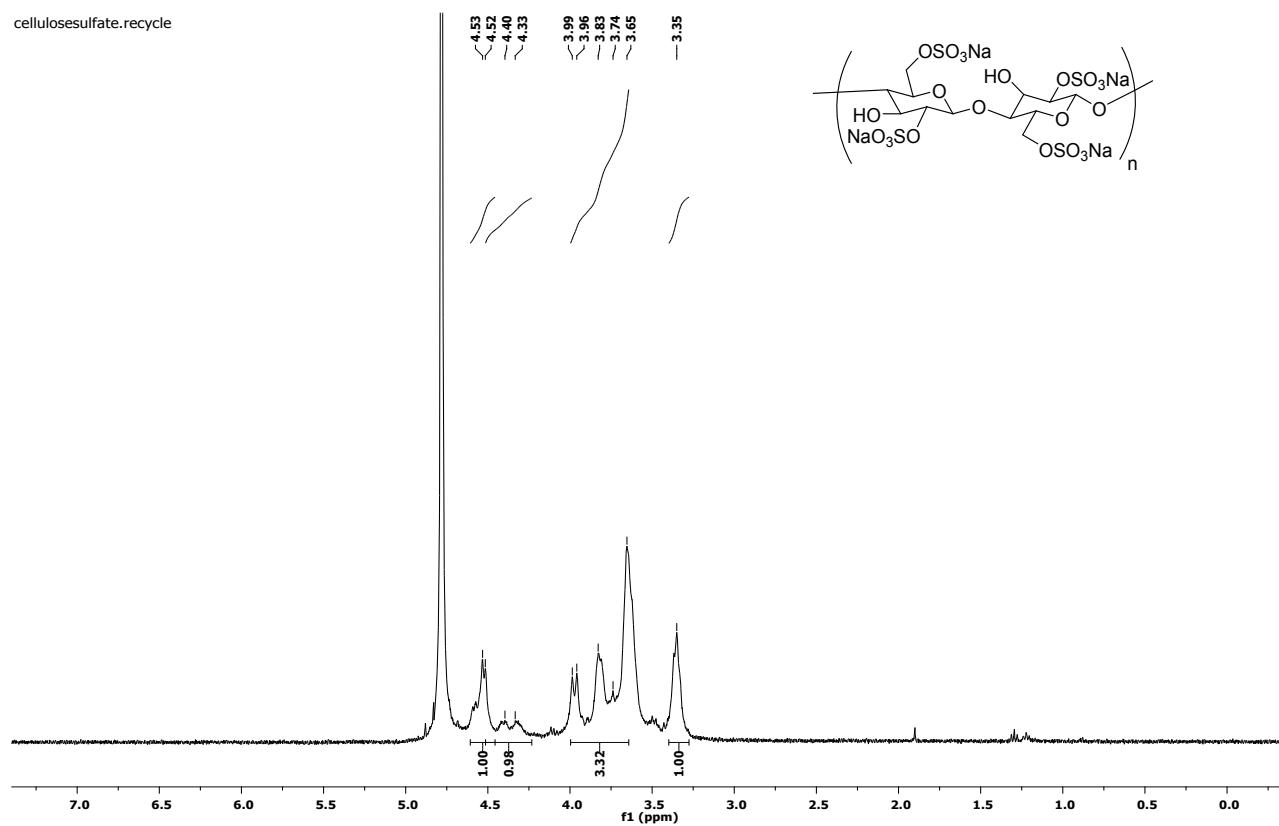
DSS-sulAr-6C-Ar
new experiment

— 151.44
— 136.45
— 134.65
— 133.50
— 118.05
— 102.75
— 73.58
— 55.69
— 43.84
— 25.76
— 18.66
— 4.68



¹³C NMR spectrum of compound 6f in CDCl_3

cellulosulfate.recycle



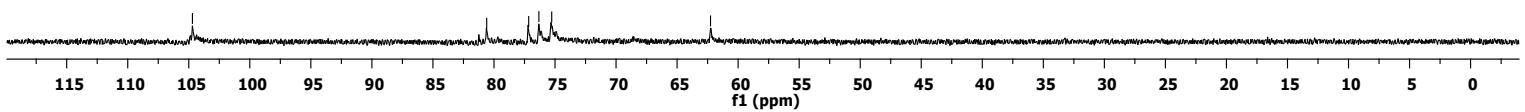
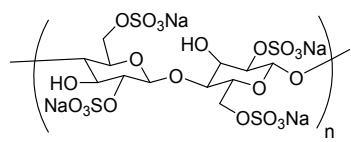
^1H NMR spectrum of recovered (after 3rd cycle) HS-cellulose sulphate sodium in D_2O

cellulose.sulfate.1st.recycle.fid.CMR

— 104.89

— 80.60
77.16
76.32
75.27

— 62.26



¹³C NMR spectrum of recovered (after 3rd cycle) HS-cellulose sulphate sodium in D₂O

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