Supplementary Material

Synthesis of [28-^{13}C]-24-methylenecholesterol using 1-*tert*-butyl-1*H*-tetrazol-5-yl [^{13}C]-methyl sulfone to methylenate an isopropyl ketone intermediate

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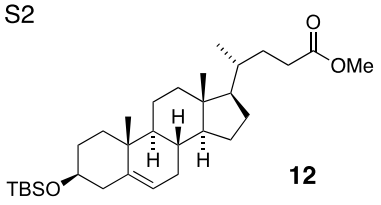
– ^{1}H and ^{13}C NMR Spectra –

^{1}H and ^{13}C NMR spectra were recorded in Fourier transform mode at the field strength specified using standard 5 mm diameter tubes. Chemical shift in ppm is quoted relative to residual solvent signals calibrated as follows:

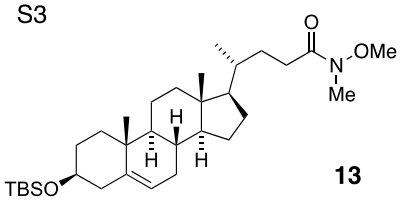
$CDCl_3$ δ_H ($CHCl_3$) = 7.26 ppm, δ_C ($CDCl_3$) = 77.2 ppm

S2



12

S3



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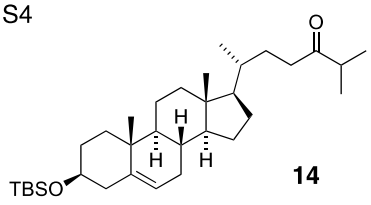

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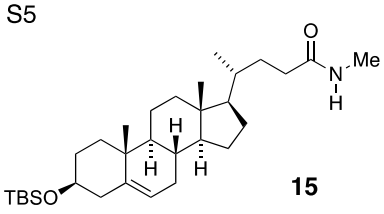
S5



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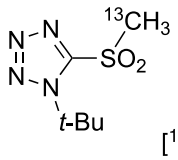
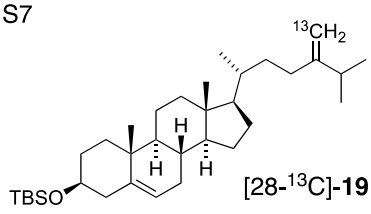
S6



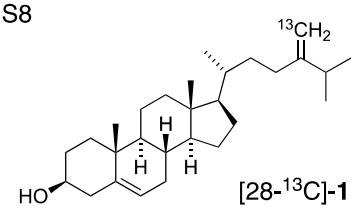
[^{13}C]-**18**

S7



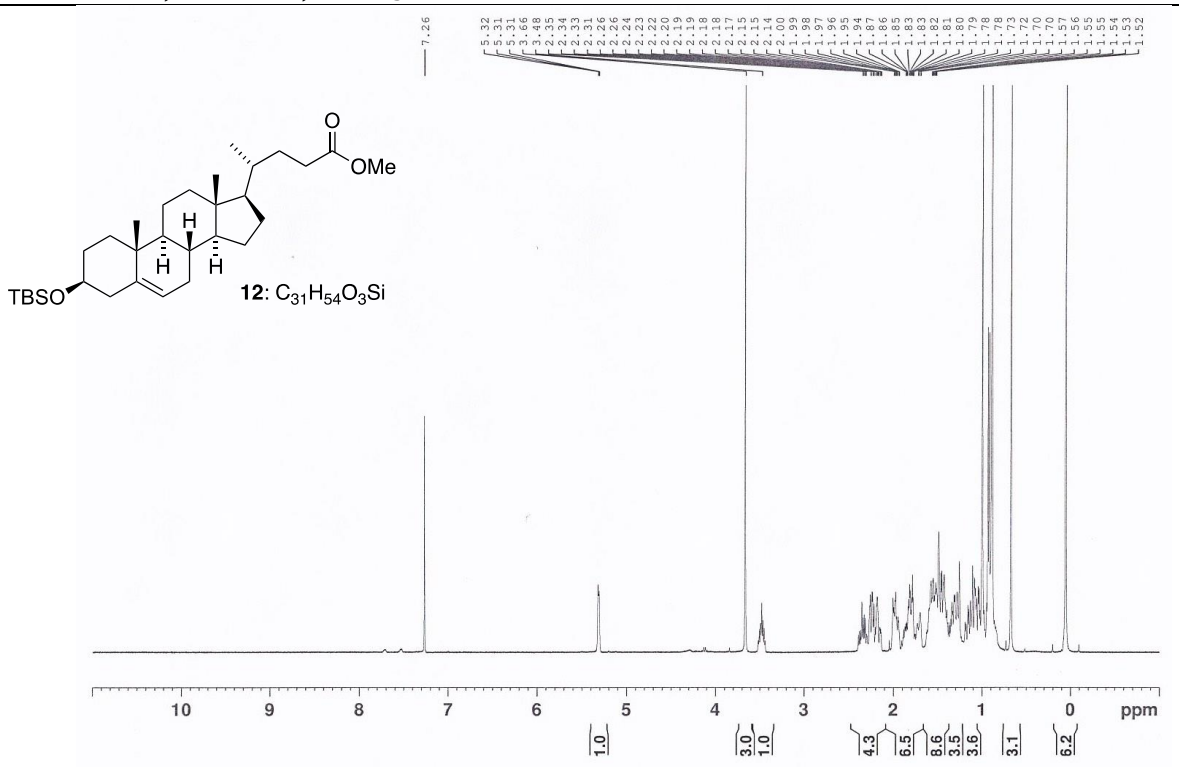
[28-^{13}C]-**19**

S8

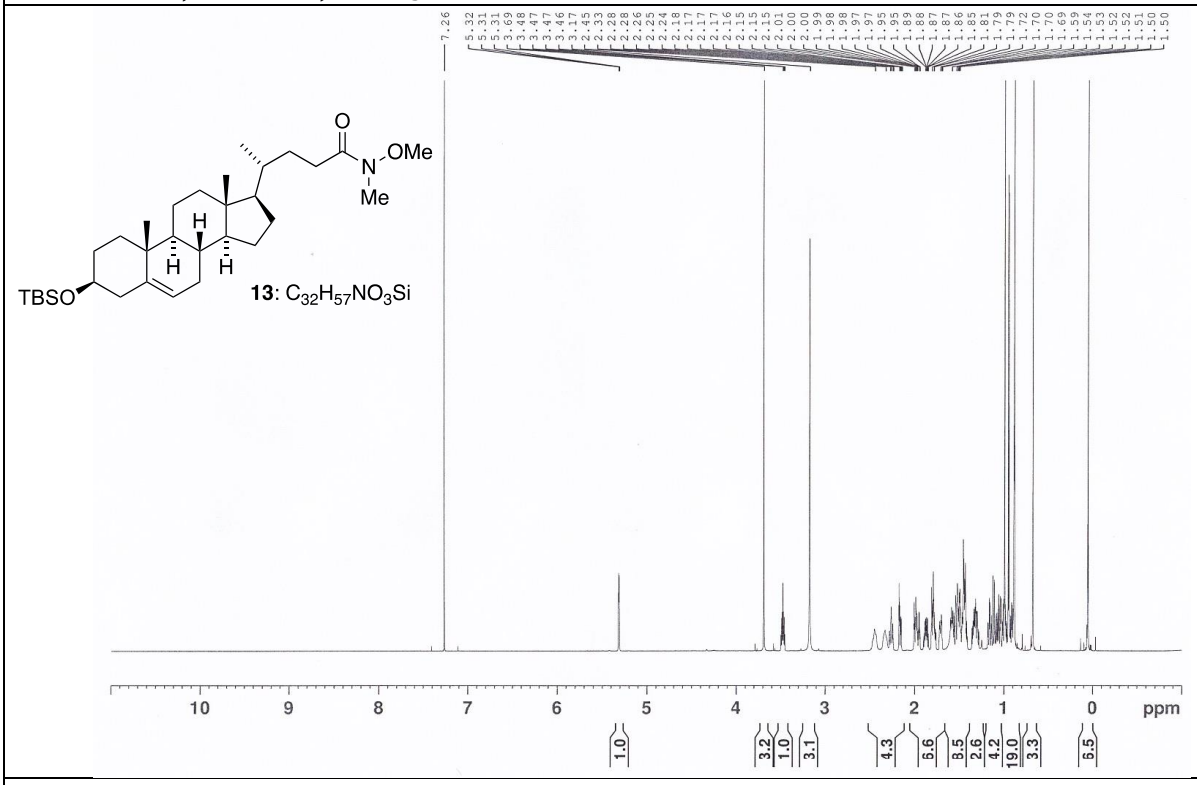


[28-^{13}C]-**1**

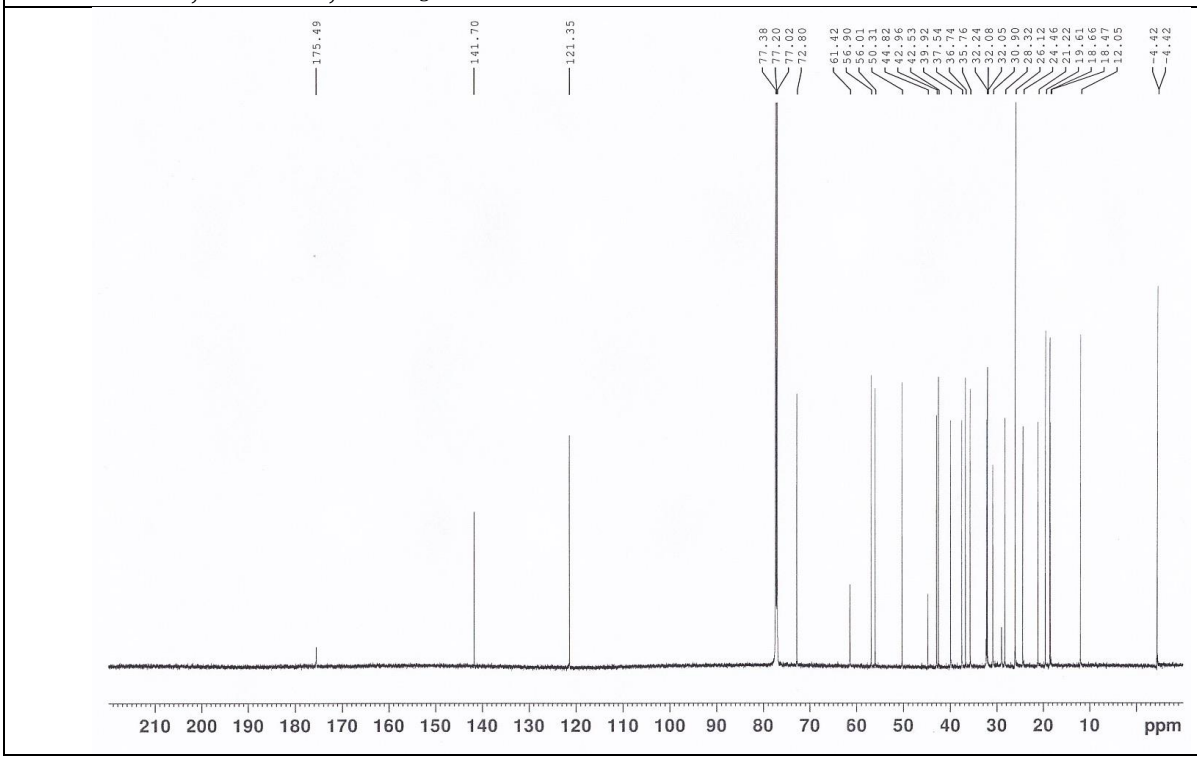
12: ^{1}H NMR, 400 MHz, $CDCl_3$



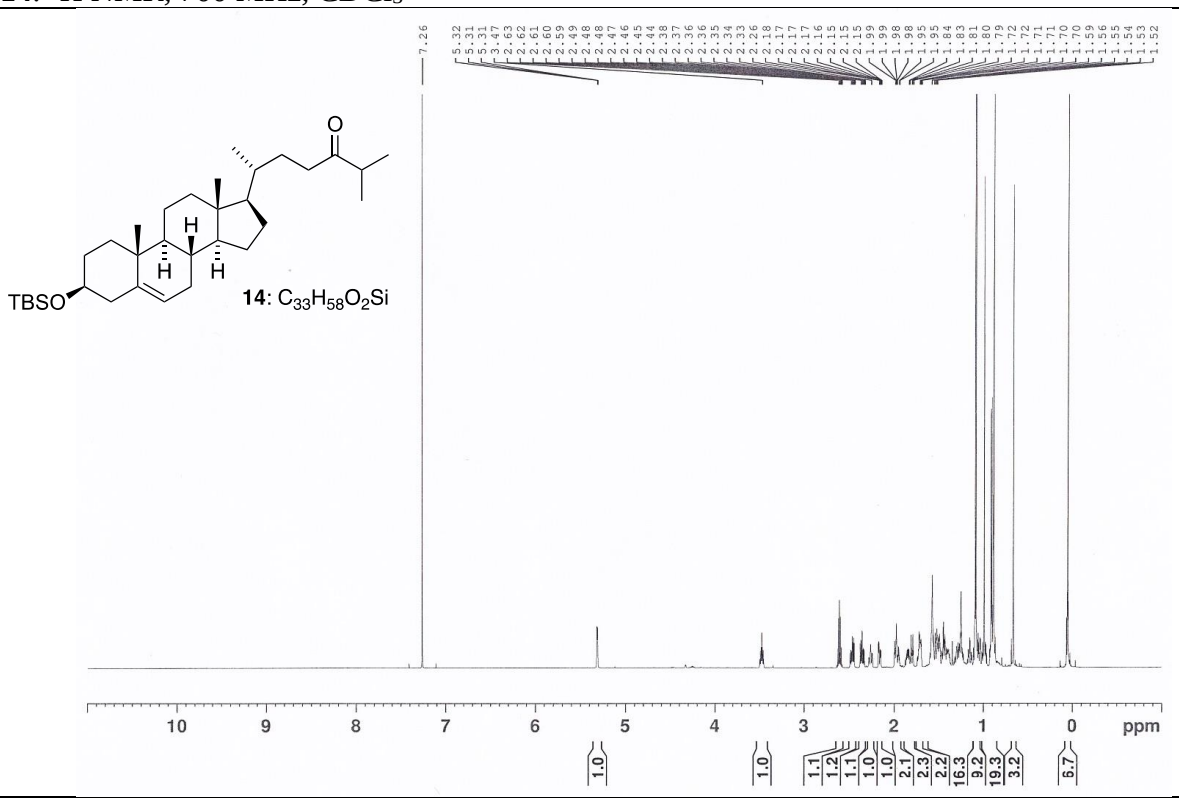
13: ^{1}H NMR, 700 MHz, $CDCl_3$



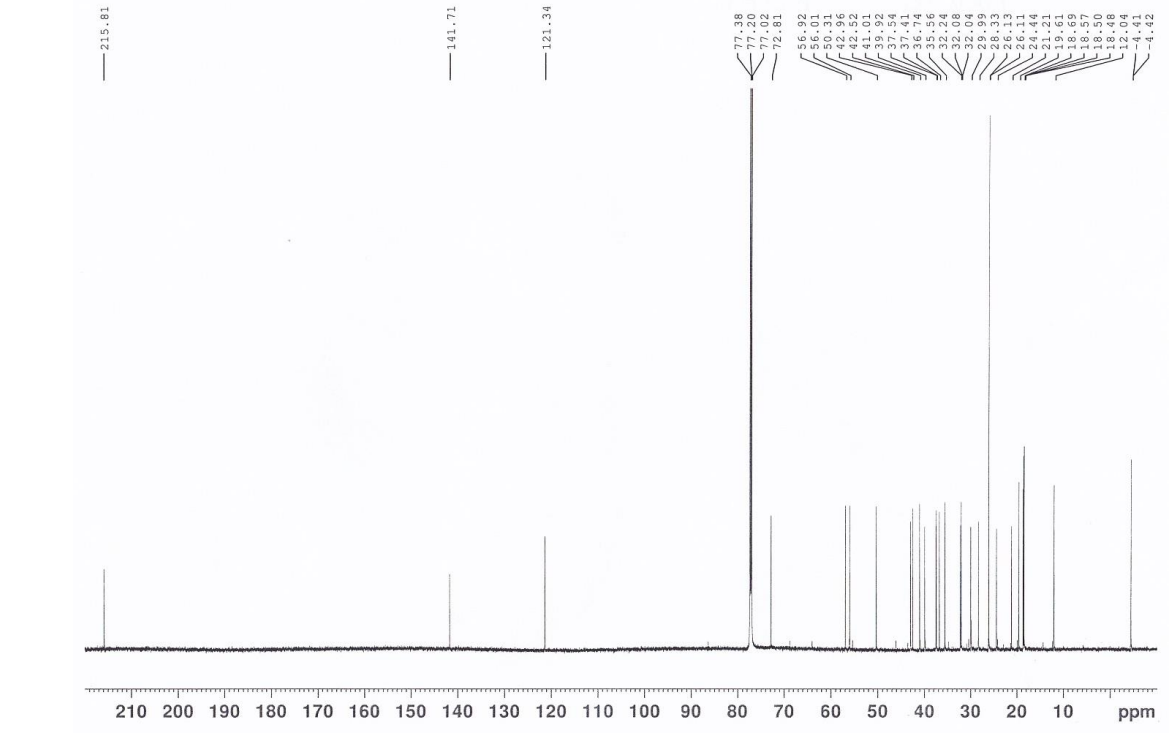
13: ^{13}C NMR, 175 MHz, $CDCl_3$



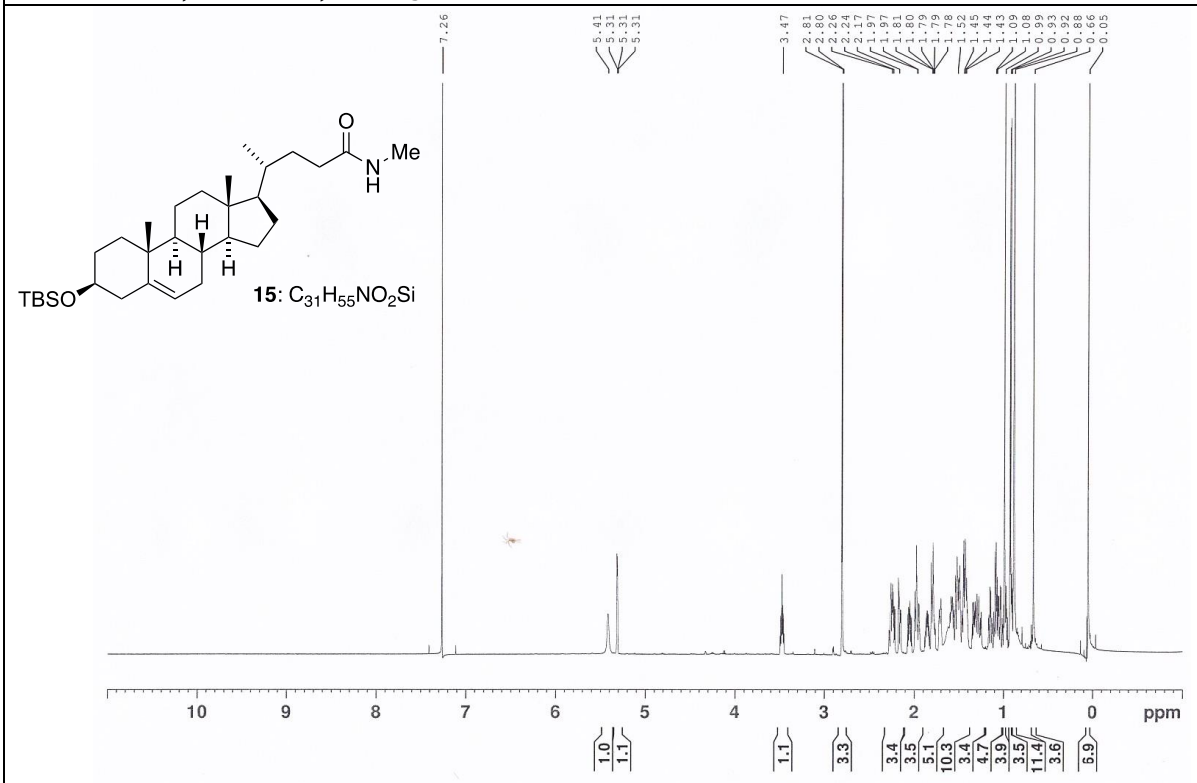
14: ^{1}H NMR, 700 MHz, $CDCl_3$



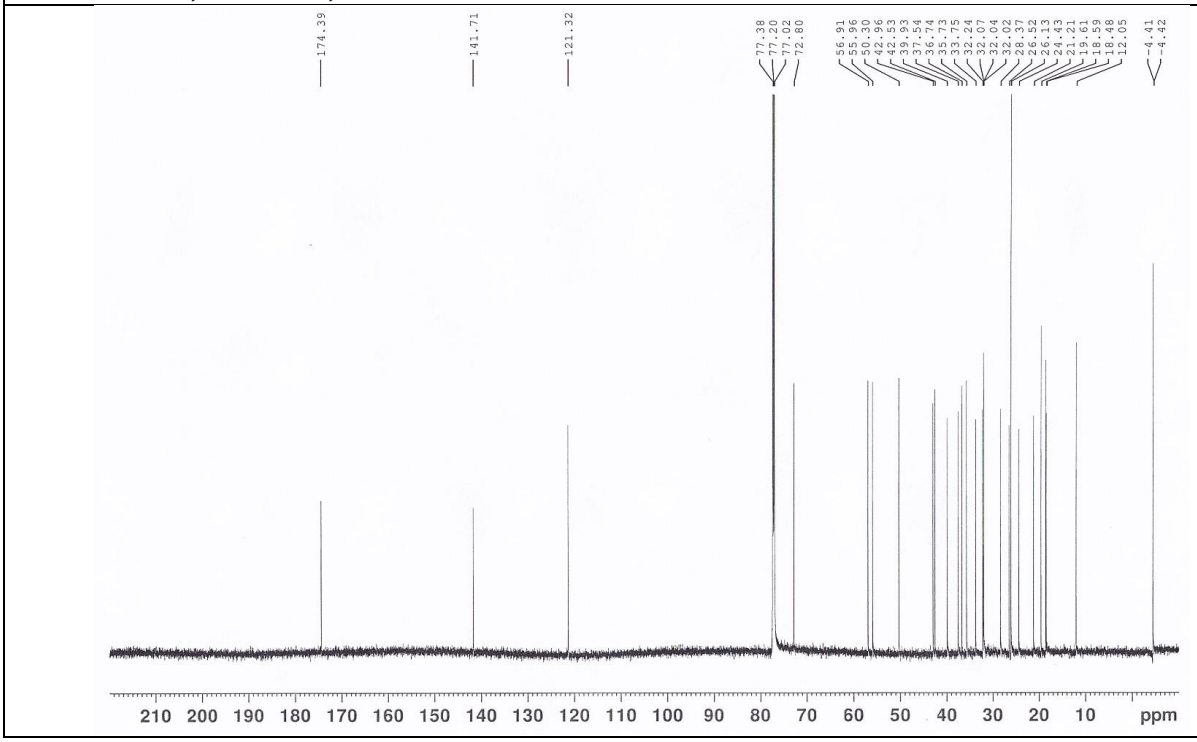
14: ^{13}C NMR, 175 MHz, $CDCl_3$



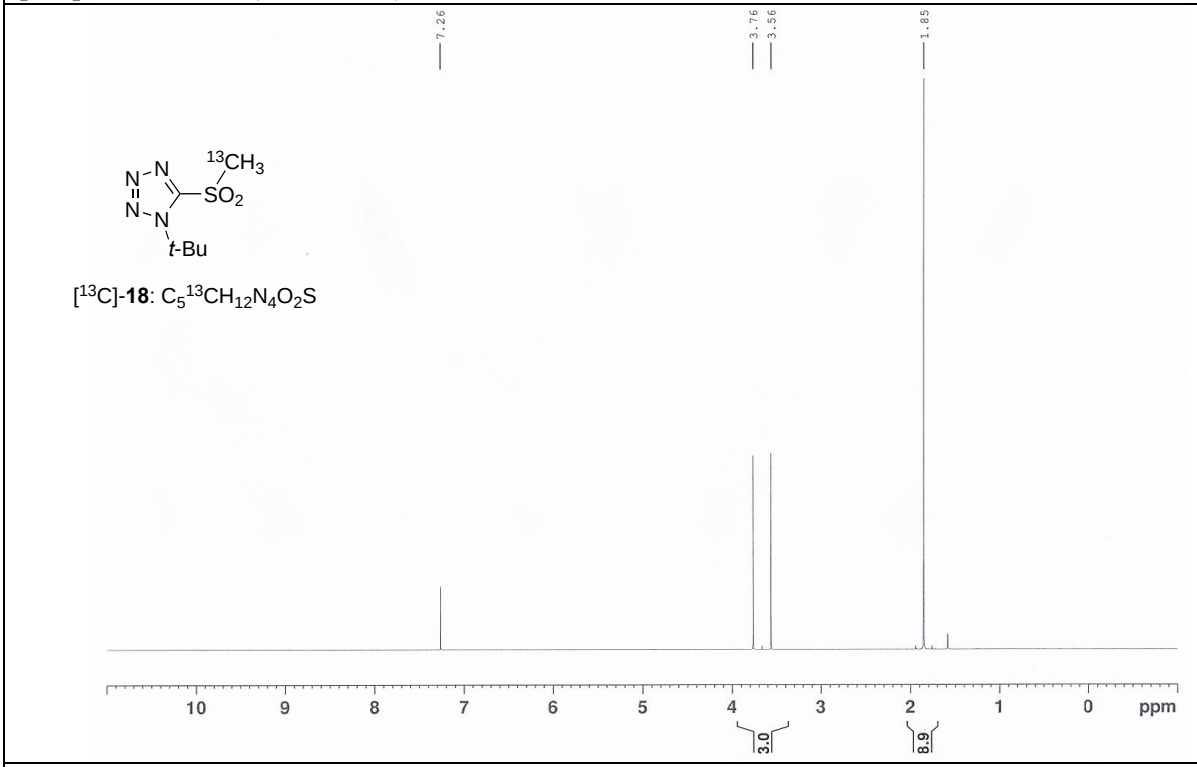
15: ^{1}H NMR, 700 MHz, $CDCl_3$



15: ^{13}C NMR, 175 MHz, $CDCl_3$

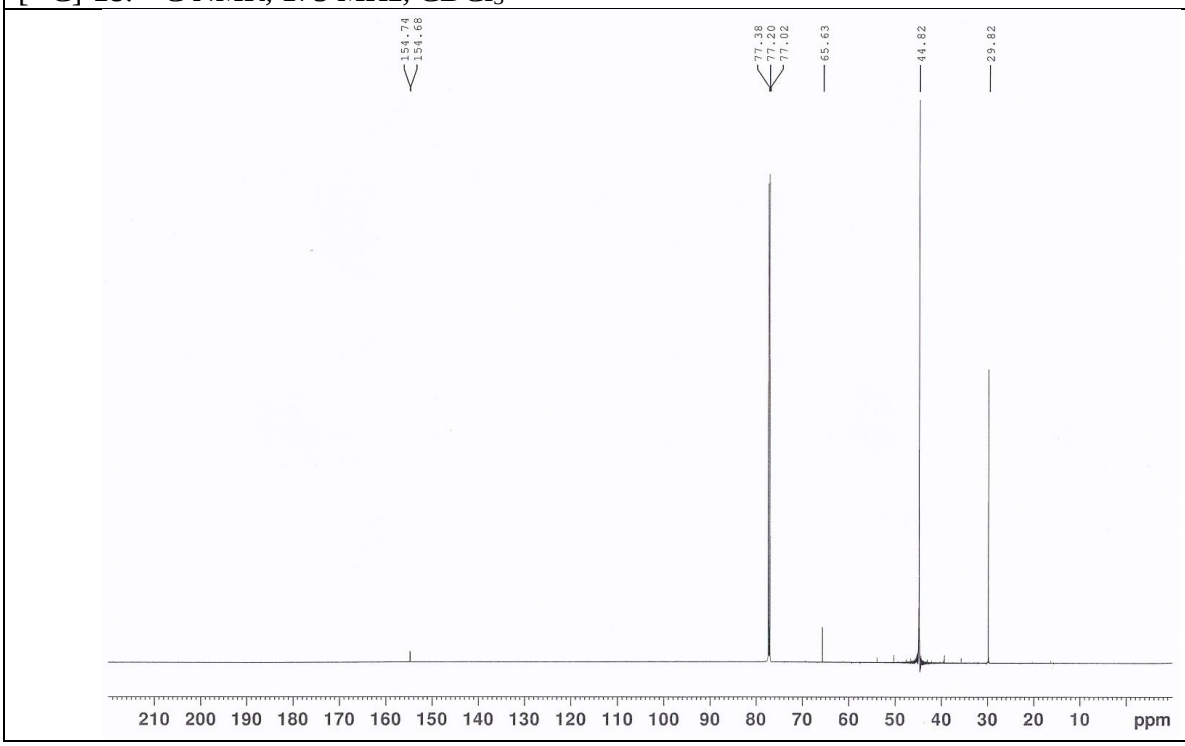


[^{13}C]-**18**: ^{1}H NMR, 700 MHz, $CDCl_3$

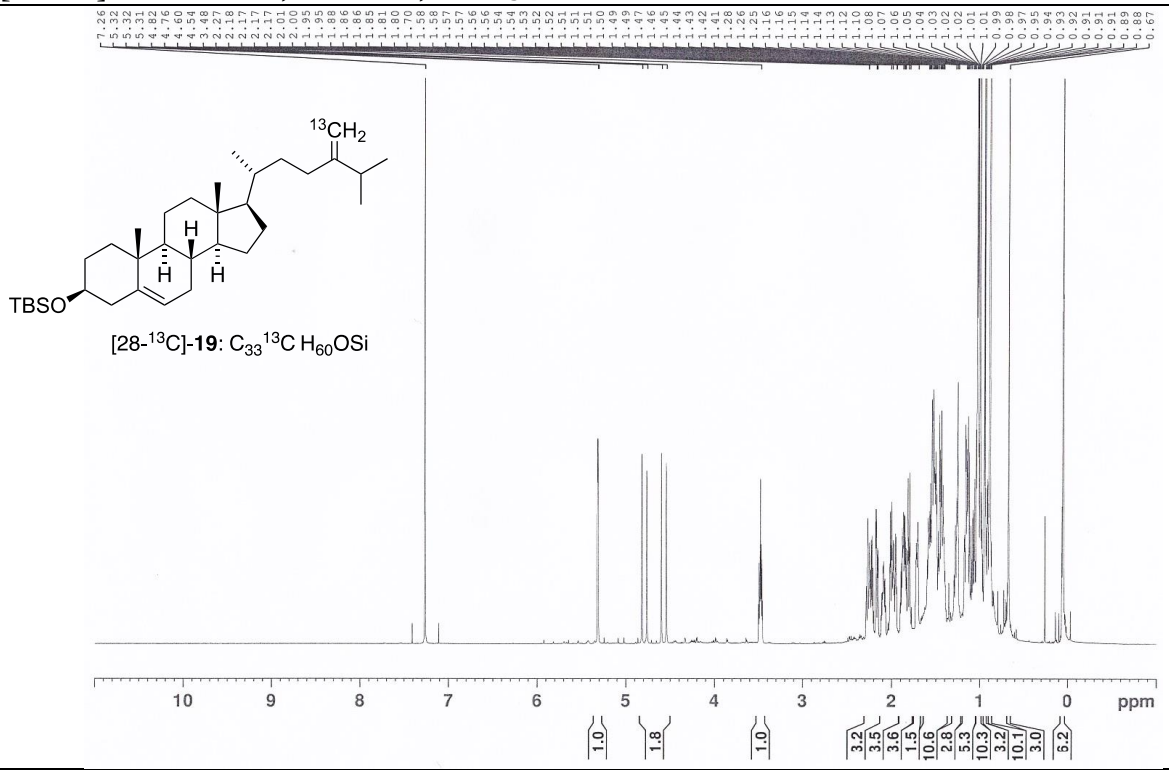


[^{13}C]-**18**: $C_5{}^{13}CH_{12}N_4O_2S$

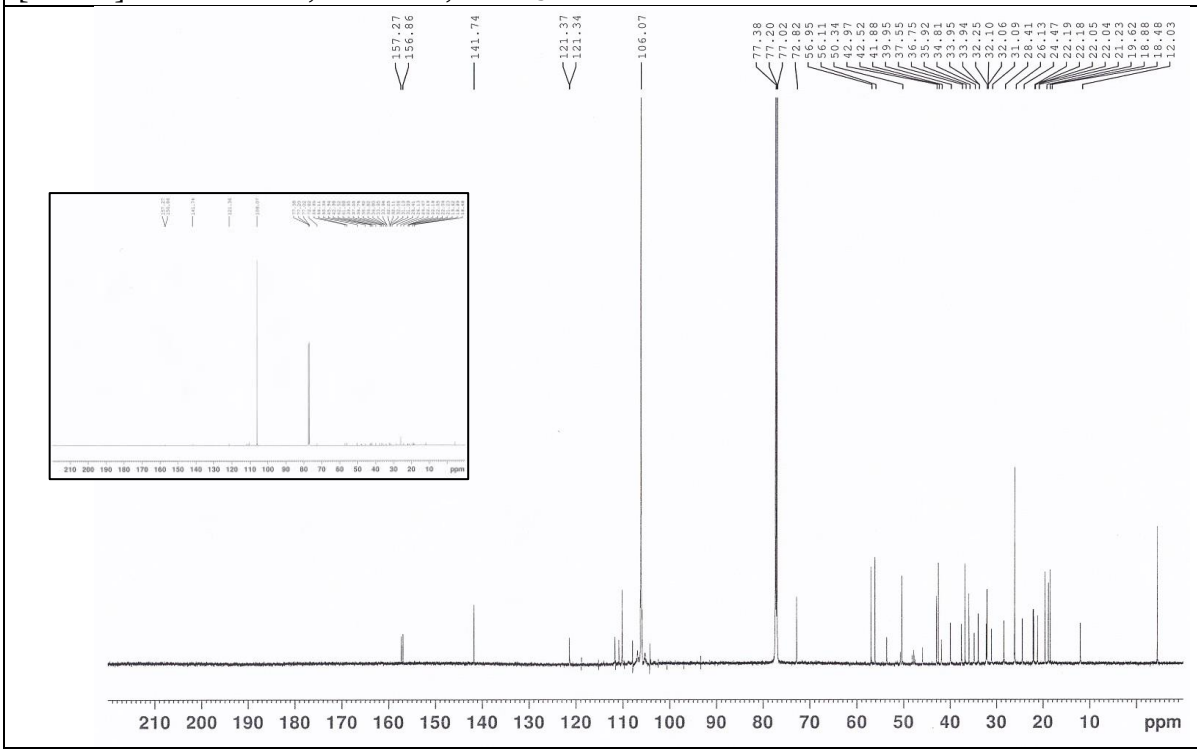
[^{13}C]-**18**: ^{13}C NMR, 175 MHz, $CDCl_3$



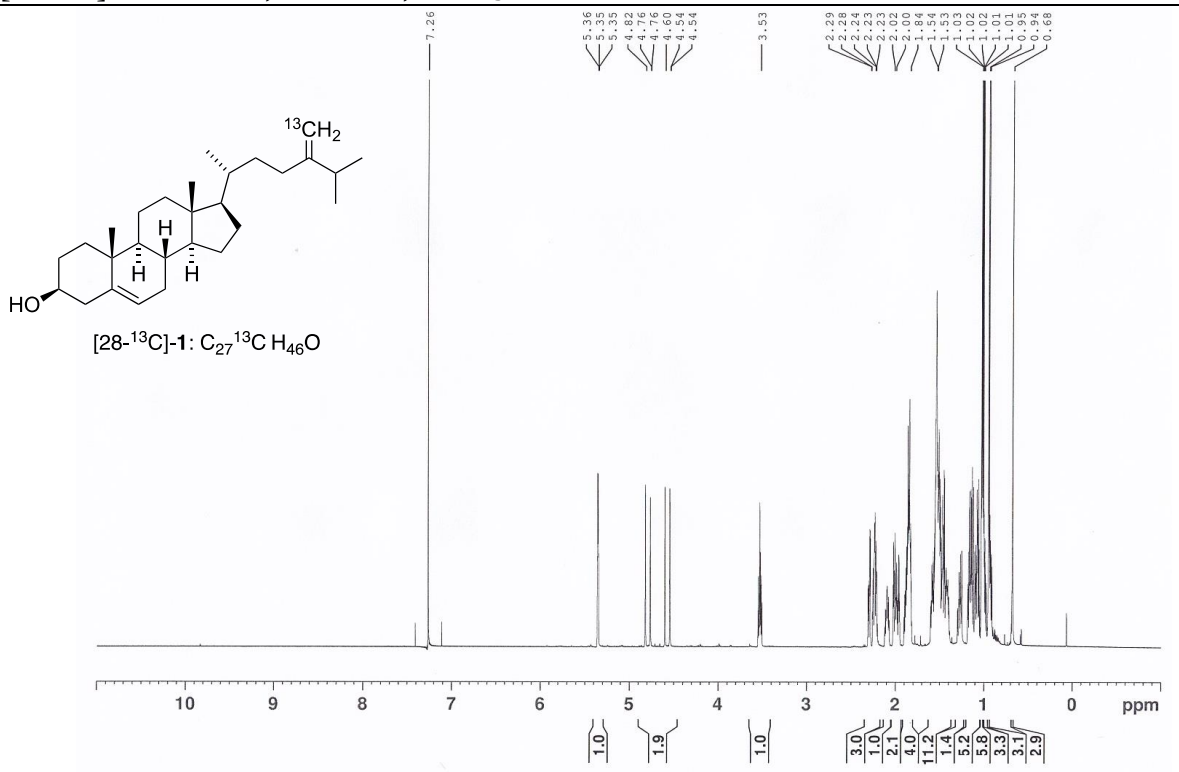
[28-^{13}C]-**19**: ^{1}H NMR, 700 MHz, $CDCl_3$



[28-^{13}C]-**19**: ^{13}C NMR, 175 MHz, $CDCl_3$



[28-^{13}C]-**1**: ^{1}H NMR, 700 MHz, $CDCl_3$



[28-^{13}C]-**1**: ^{13}C NMR, 175 MHz, $CDCl_3$