Supplementary Material

Synthesis of [28-13C]-24-methylenecholesterol using 1-tert-butyl-1H-tetrazol-5-yl [13C]-methyl sulfone to methyleneate 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:

$$\text{CDCl}_3 \delta_H (\text{CHCl}_3) = 7.26 \text{ ppm}, \delta_C (\text{CDCl}_3) = 77.2 \text{ ppm}$$
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$_3$

![NMR spectrum](image)

15: $^{13}$C NMR, 175 MHz, CDCl$_3$

![NMR spectrum](image)
[\textsuperscript{13}C]-18: \textsuperscript{1}H NMR, 700 MHz, CDCl\textsubscript{3}

[\textsuperscript{13}C]-18: \textsuperscript{13}C NMR, 175 MHz, CDCl\textsubscript{3}
[28,13C]-19: 1H NMR, 700 MHz, CDCl3

[28-13C]-19: C3913C H92OSi

[28,13C]-19: 13C NMR, 175 MHz, CDCl3
[28,\textsuperscript{13}C]-I: \textsuperscript{1}H NMR, 700 MHz, CDCl\textsubscript{3}

[28,\textsuperscript{13}C]-I: C\textsubscript{27}\textsuperscript{13}C H\textsubscript{46}O

[28,\textsuperscript{13}C]-I: \textsuperscript{13}C NMR, 175 MHz, CDCl\textsubscript{3}