

Supporting Information

Concise Large-Scale Synthesis of Tomatidine, a Potent Antibiotic Natural Product

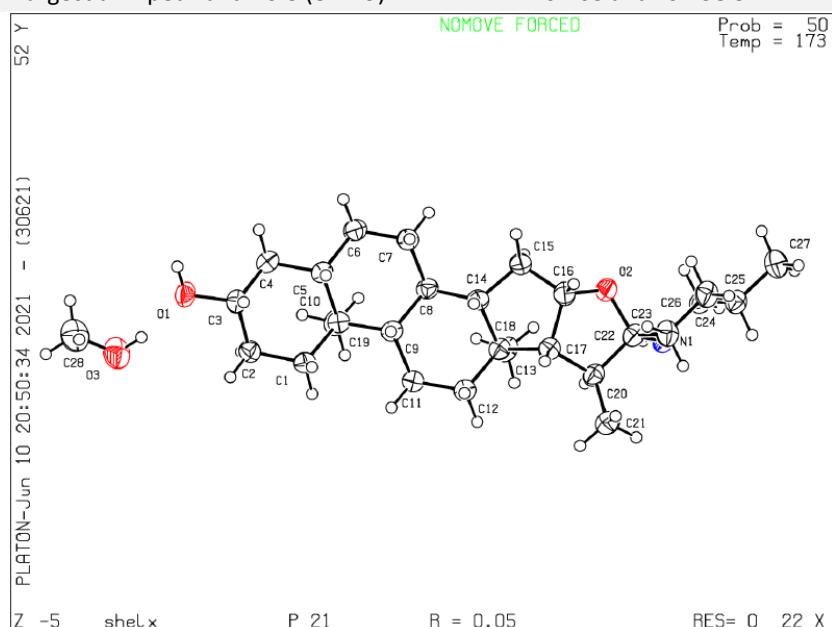
Chad Normandin and Pierre-Luc Boudreault*

Table of Contents of the Supporting information

Table S1. Crystal data and structure refinement for 1	2
Figure S1. ^1H NMR (400 MHz, CDCl_3) of 15	3
Figure S2. ^{13}C NMR (400 MHz, CDCl_3) of 15	3
Figure S3. ^1H NMR (400 MHz, CDCl_3) of 8	4
Figure S4. ^{13}C NMR (400 MHz, CDCl_3) of 8	4
Figure S5. ^1H NMR (400 MHz, CDCl_3) of 9a	5
Figure S6. ^{13}C NMR (400 MHz, CDCl_3) of 9a	5
Figure S7. ^1H NMR (400 MHz, CDCl_3) of 11	6
Figure S8. ^{13}C NMR (400 MHz, CDCl_3) of 11	6
Figure S9. ^1H NMR (400 MHz, CDCl_3) of 12	7
Figure S10. ^{13}C NMR (400 MHz, CDCl_3) of 12	7
Figure S11. ^1H NMR (400 MHz, CDCl_3) of 13	8
Figure S12. ^{13}C NMR (100 MHz, CDCl_3) of 13	8
Figure S13. ^1H NMR (400 MHz, CDCl_3) of 7	9
Figure S14. ^{13}C NMR (100 MHz, CDCl_3) of 7	9
Figure S15. ^1H NMR (400 MHz, CDCl_3) of 2	10
Figure S16. ^{13}C NMR (100 MHz, CDCl_3) of 2	10
Figure S17. ^1H NMR (400 MHz, CDCl_3) of OAc- 2	11
Figure S18. ^{13}C NMR (100 MHz, CDCl_3) of OAc- 2	11
Figure S19. ^1H NMR (400 MHz, CDCl_3) of 16	12
Figure S20. ^{13}C NMR (100 MHz, CDCl_3) of 16	12
Figure S21. ^1H NMR (400 MHz, CDCl_3) of 1	13
Figure S22. ^{13}C NMR (100 MHz, CDCl_3) of 1	13
Figure S23. Vapor Diffusion Apparatus for the Crystallization of 1	14

Table S1. Crystal Data and Structure Refinement for **1**

Empirical formula	C ₂₇ H ₄₅ NO ₂ • CH ₃ OH
Formula weight	447.68 g / mol
Temperature	173 K
Wavelength	1.54178 Å
Crystal system	Monoclinic
a	13.2306(5) Å
b	7.4199(2) Å
c	14.8312(5) Å
α	90 °
β	115.7860(10) °
γ	90 °
Volume	1311.00(8) Å ³
Z, Calculated density	1.134 g/cm ³
Absorption coefficient	0.554 mm ⁻¹
F(000)	496
Crystal size	0.430 x 0.592 x 0.750 mm
Theta Range for data collection	3.31 to 71.87°
Index ranges	-15<=h<=16, -9<=k<=8, -17<=l<=18
Reflections collected/ unique	13514
Completeness	97.1 %
Absorption correction	Multi-Scan
Max. and min. transmission	0.7530 and 0.4570
Refinement method	Full-matrix least-squares on F ²
Data/ restraints/ parameter	4556 / 2 / 301
Goodness-of-fit on F ²	1.056
Final R (I > 2σ (I))	R1 = 0.0544, wR2 = 0.1483
R indices (all data)	R1 = 0.0579, wR2 = 0.1534
Absolute structure parameter	0.02(16)
Largest diff. peak and hole (e. Å ⁻³)	0.265 and -0.238 eÅ ⁻³



The structure was deposited at the Cambridge Crystallographic Data Centre with the deposition number #2090407.

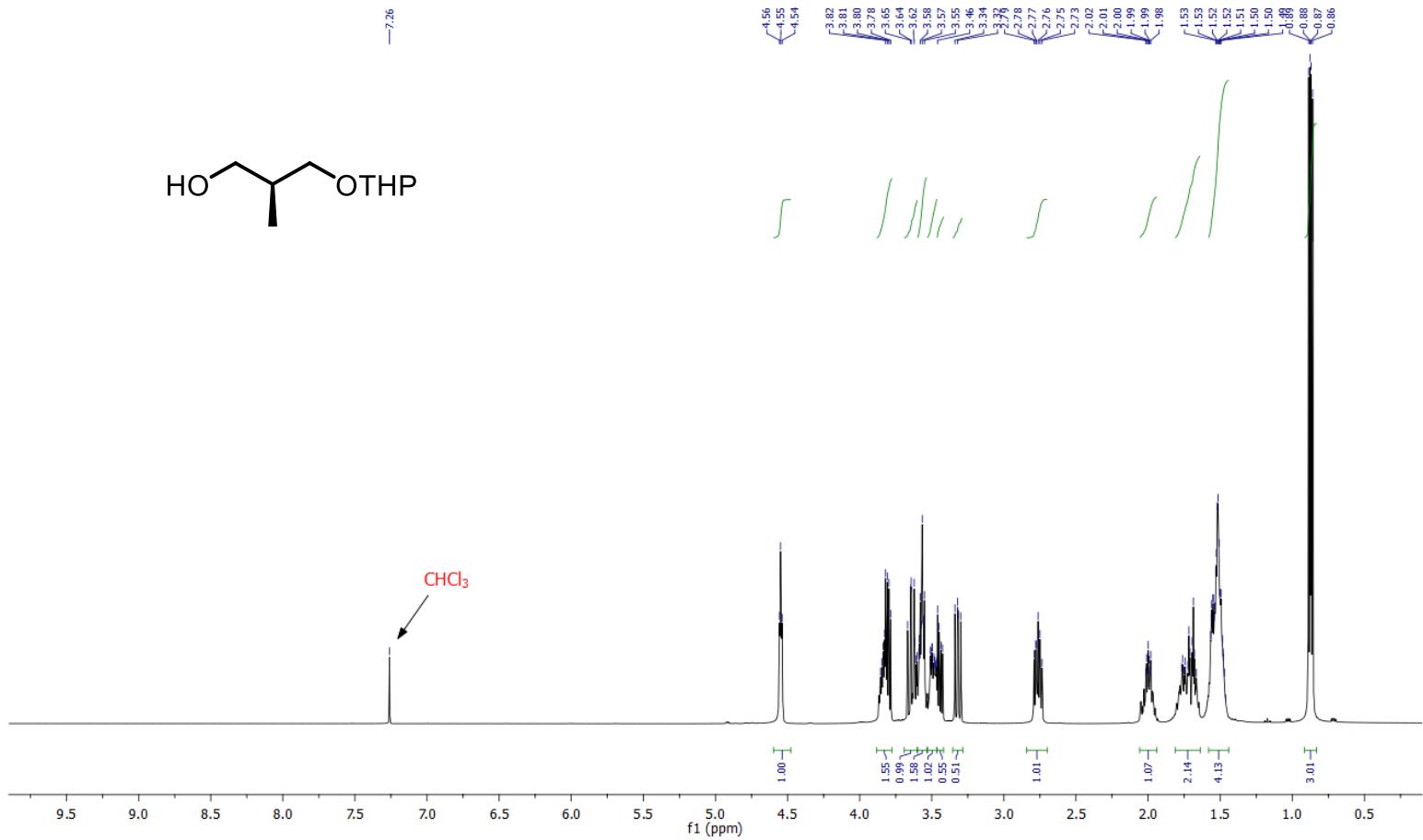
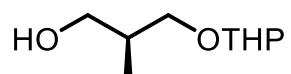


Figure S1. ^1H NMR (400 MHz, CDCl_3) of **15**

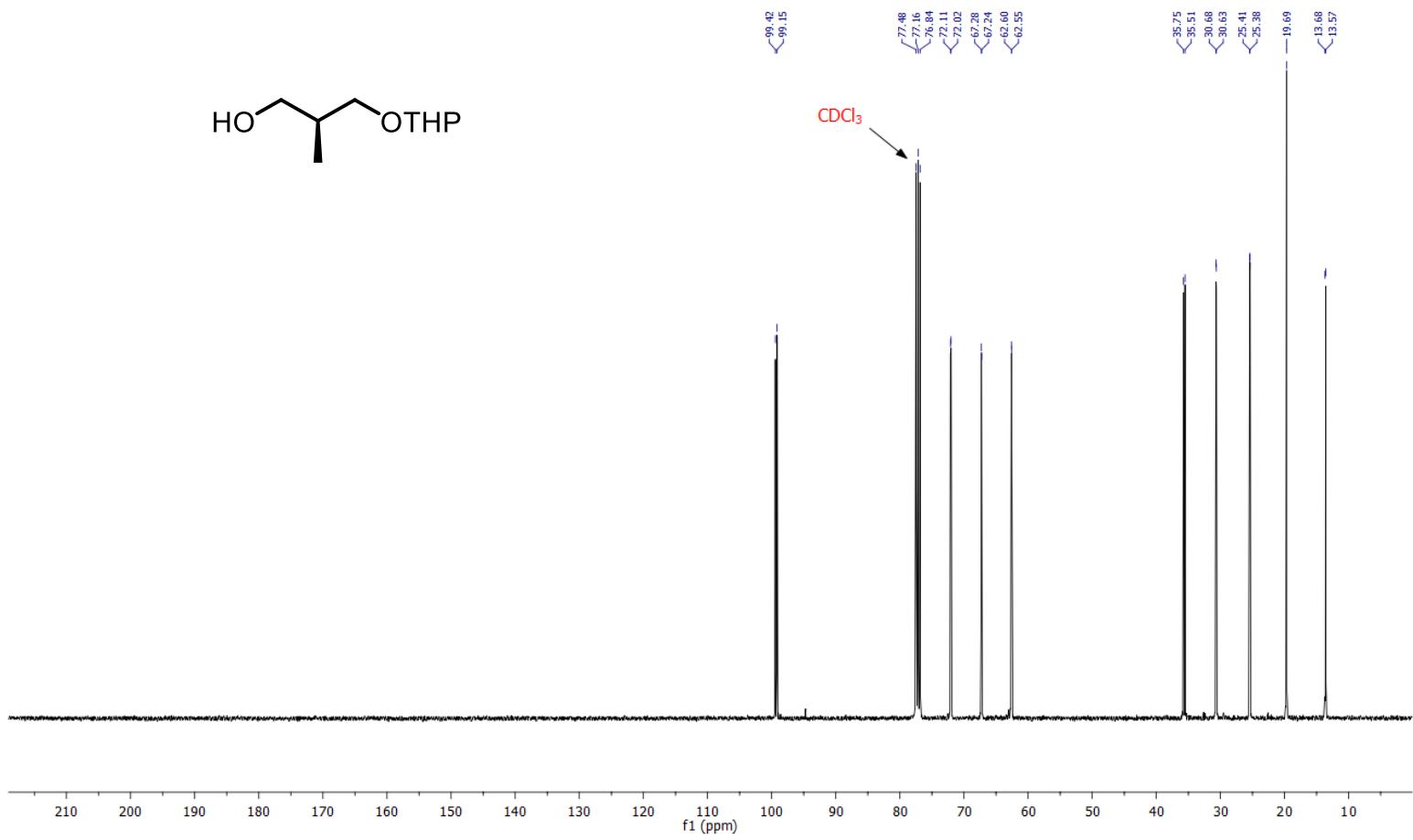
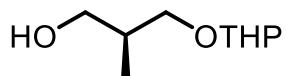


Figure S2. ^{13}C NMR (400 MHz, CDCl_3) of **15**

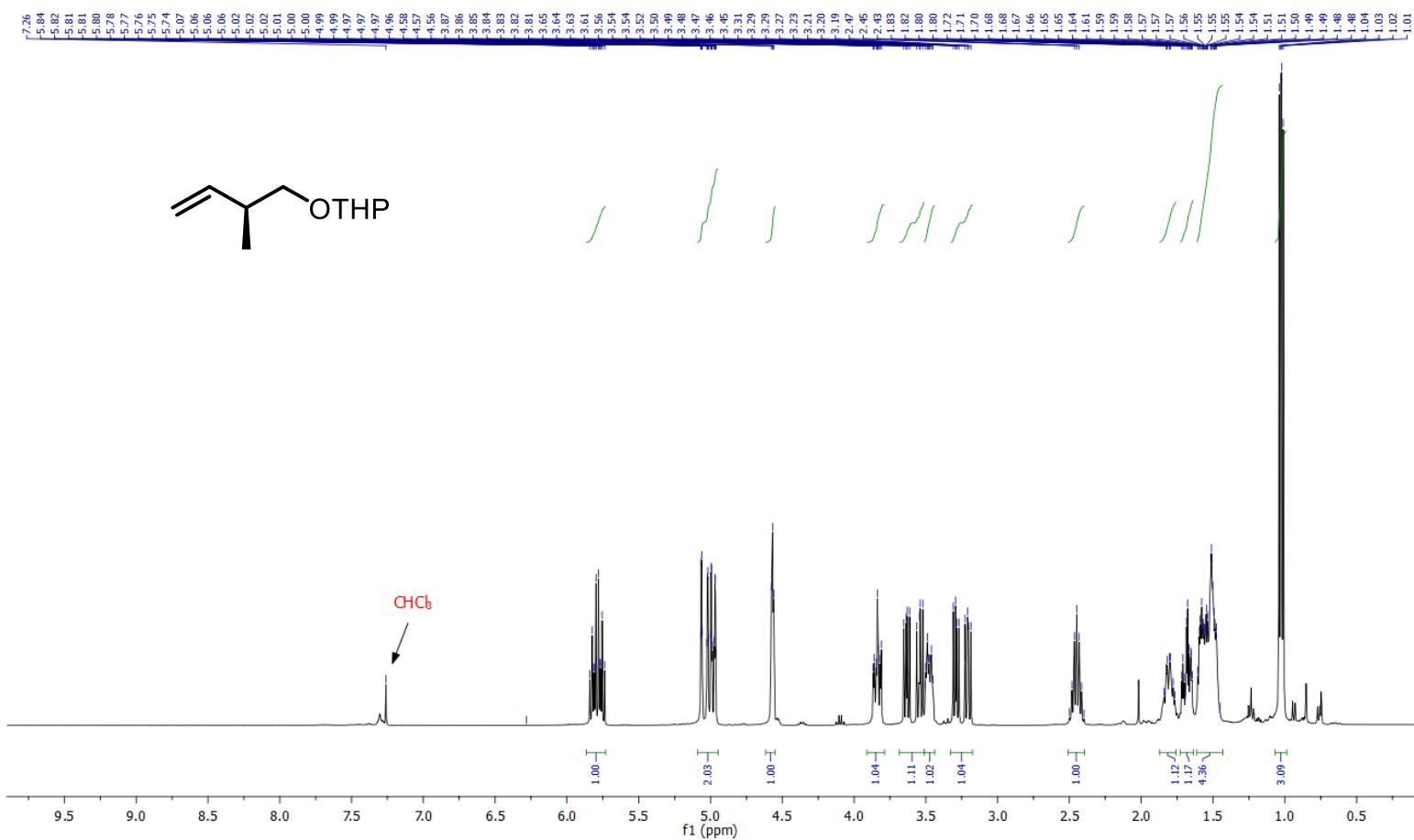


Figure S3. ^1H NMR (400 MHz, CDCl_3) of **8**

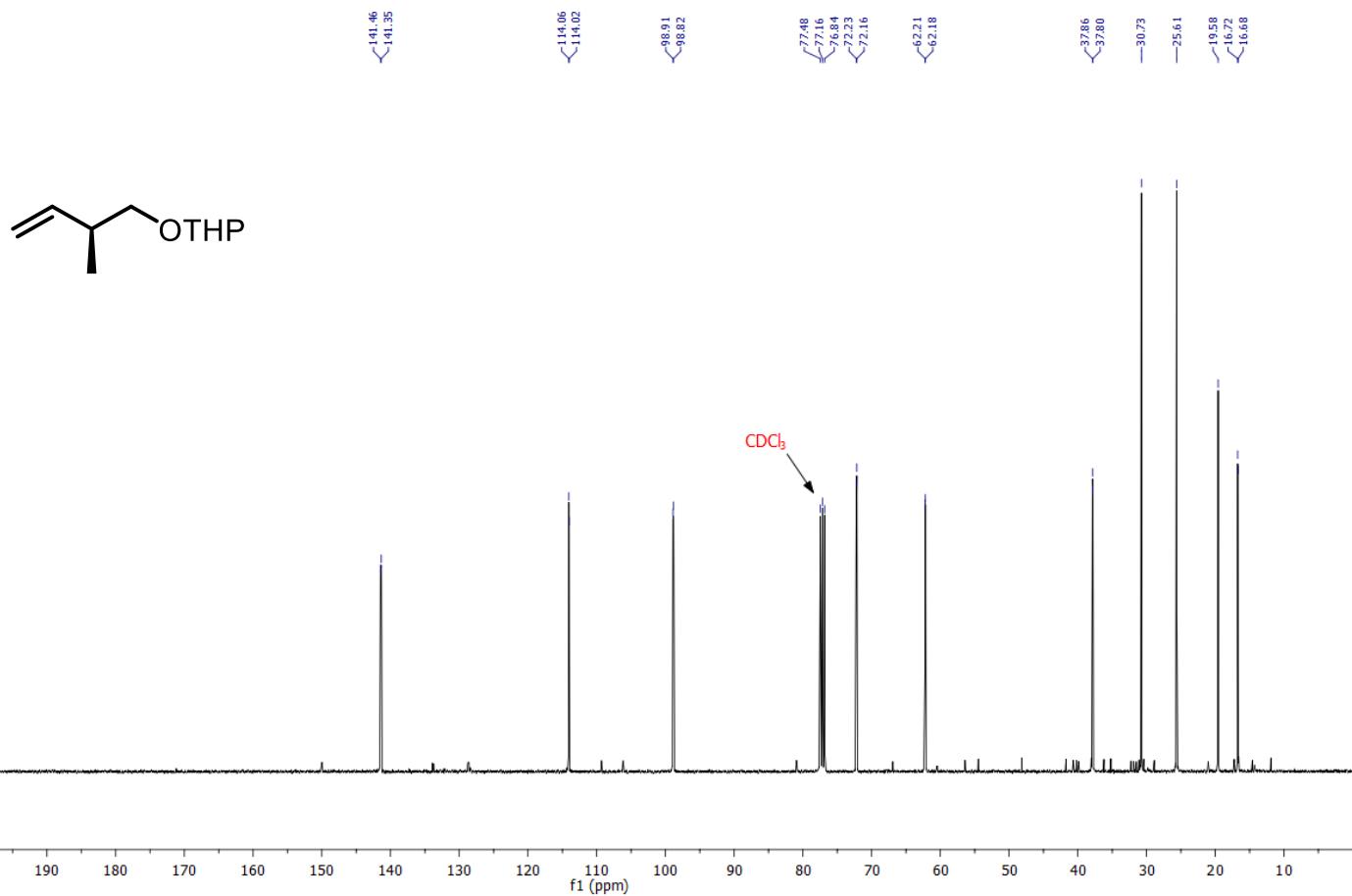


Figure S4. ^{13}C NMR (400 MHz, CDCl_3) of **8**

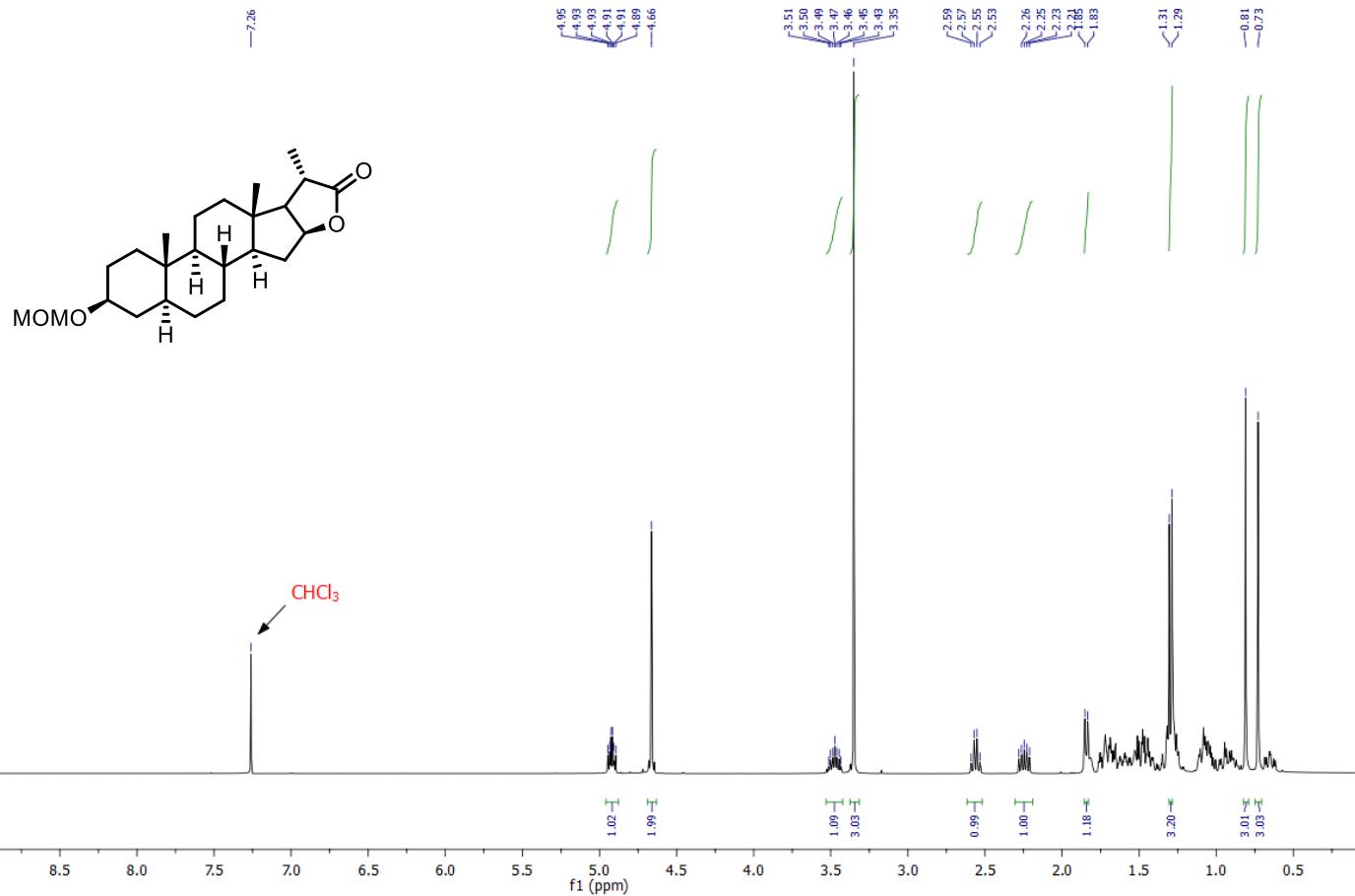


Figure S5. ^1H NMR (400 MHz, CDCl_3) of **9a**

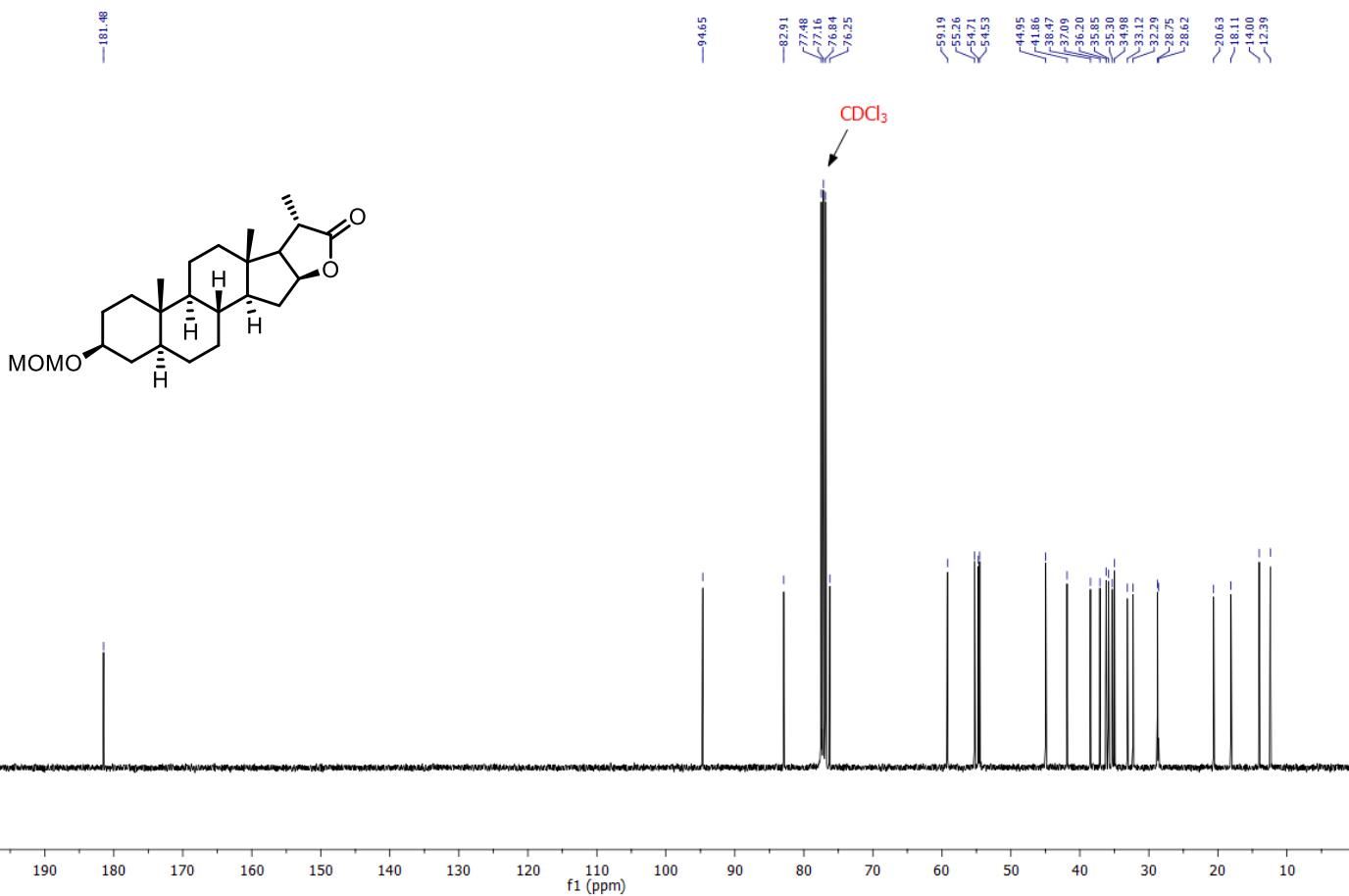


Figure S6. ^{13}C NMR (400 MHz, CDCl_3) of **9a**

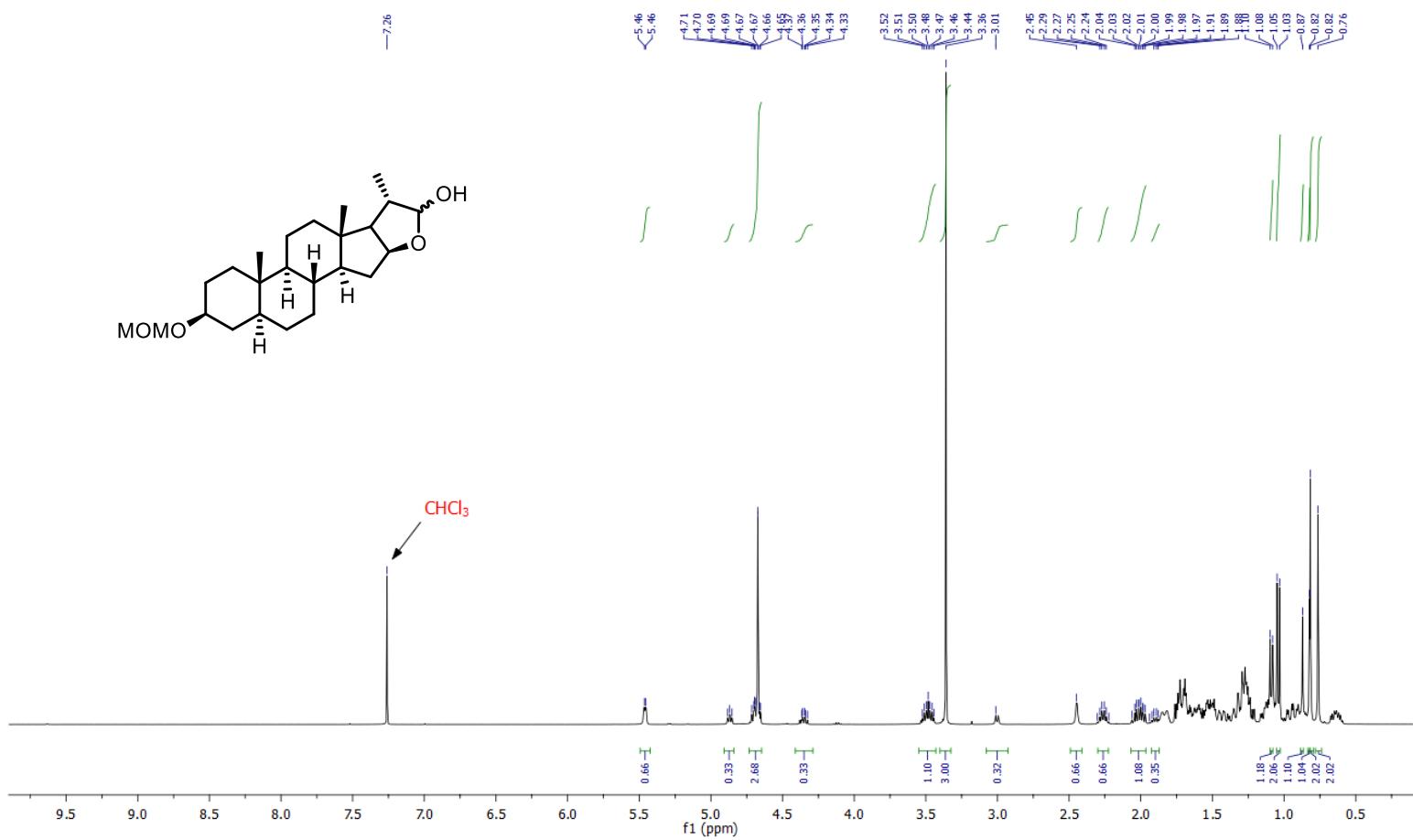
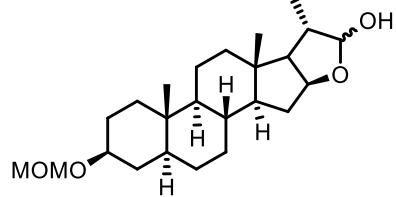


Figure S7. ^1H NMR (400 MHz, CDCl_3) of **11**

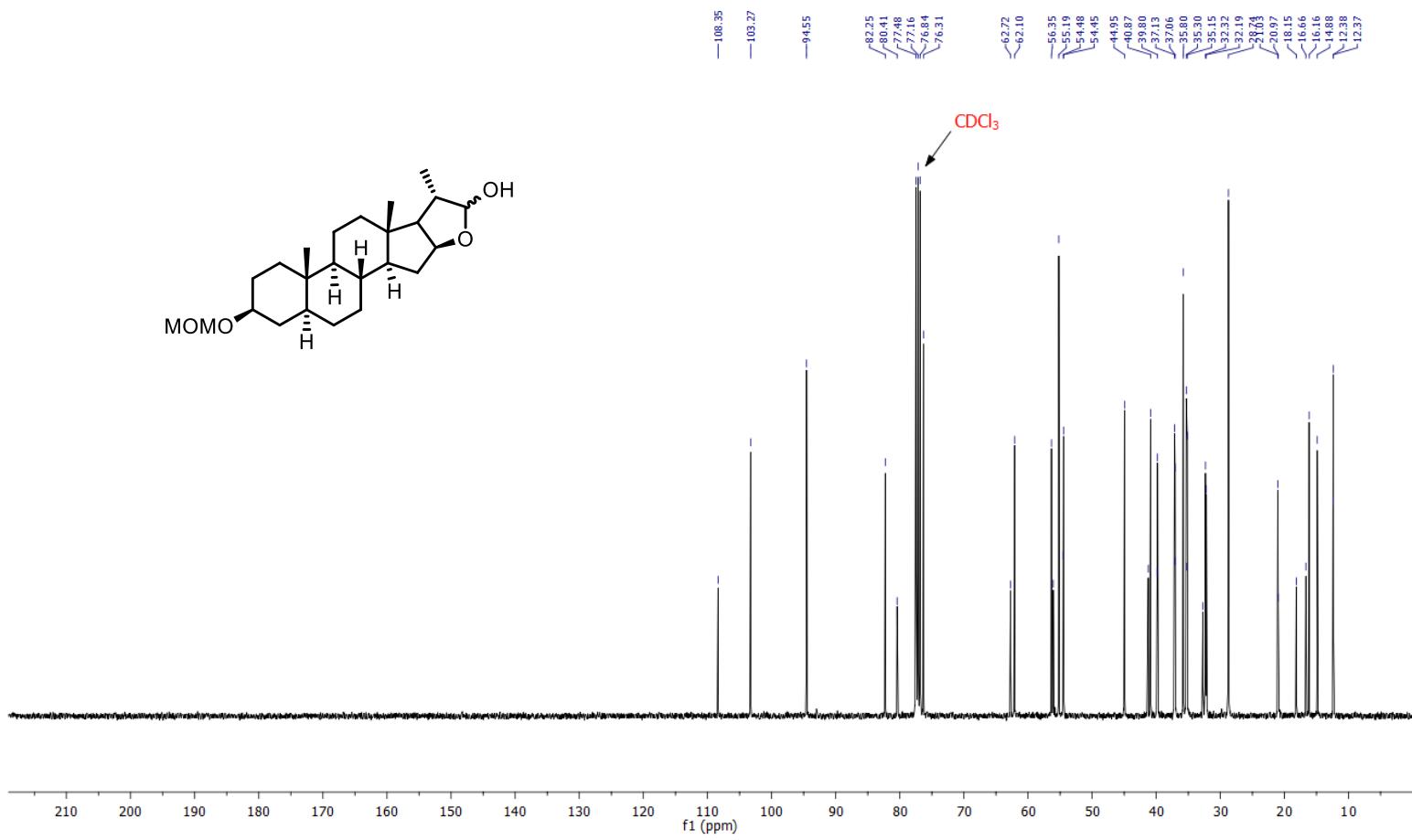
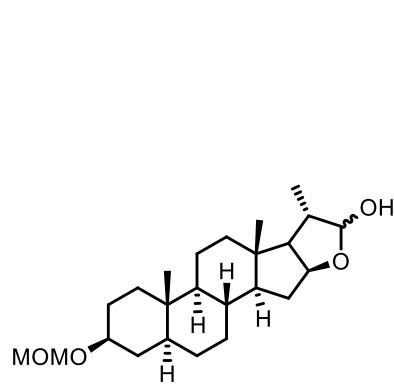


Figure S8. ^{13}C NMR (400 MHz, CDCl_3) of **11**

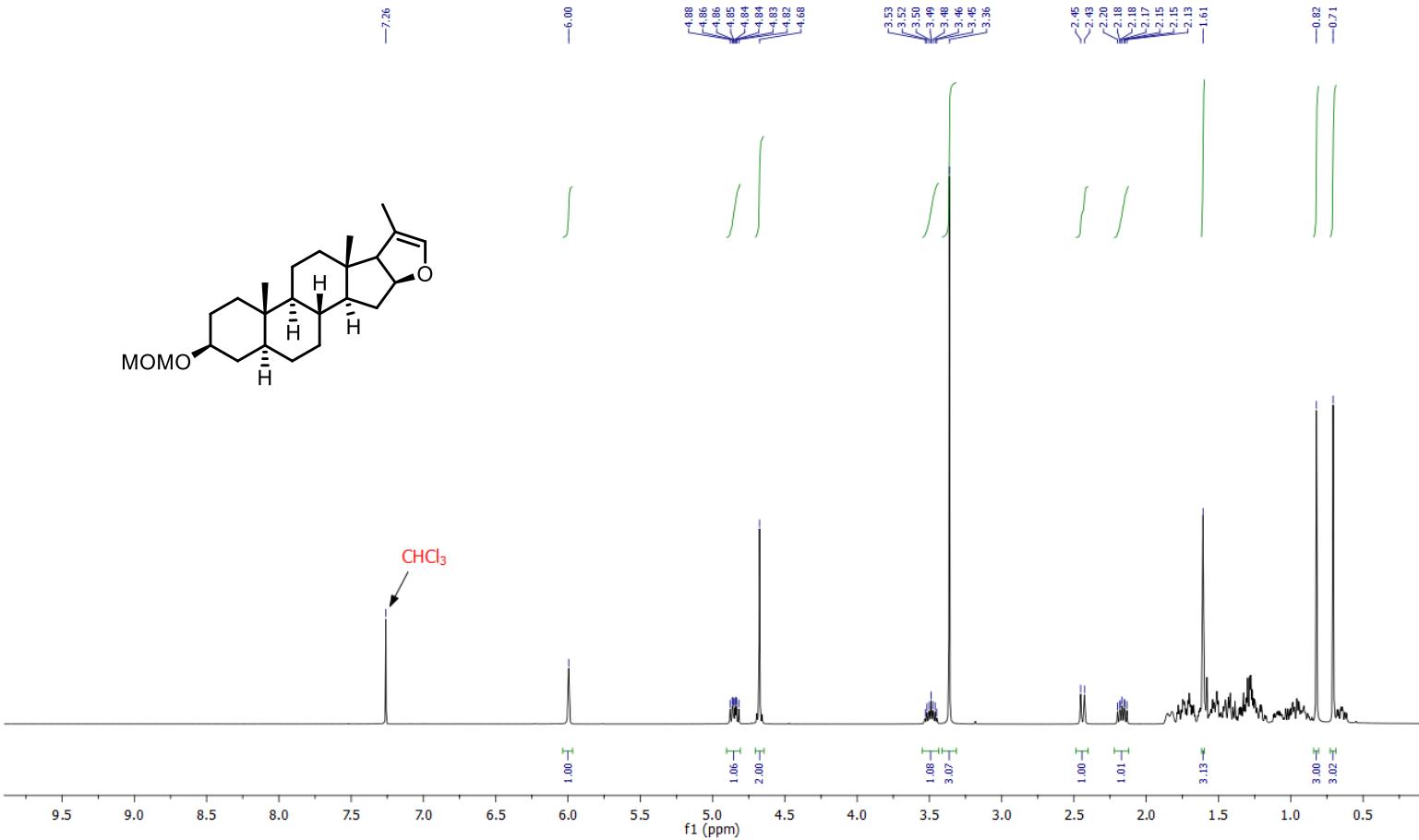
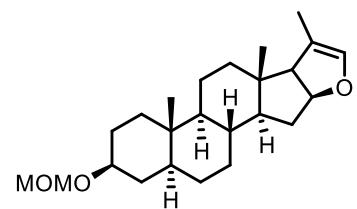


Figure S9. ^1H NMR (400 MHz, CDCl_3) of **12**

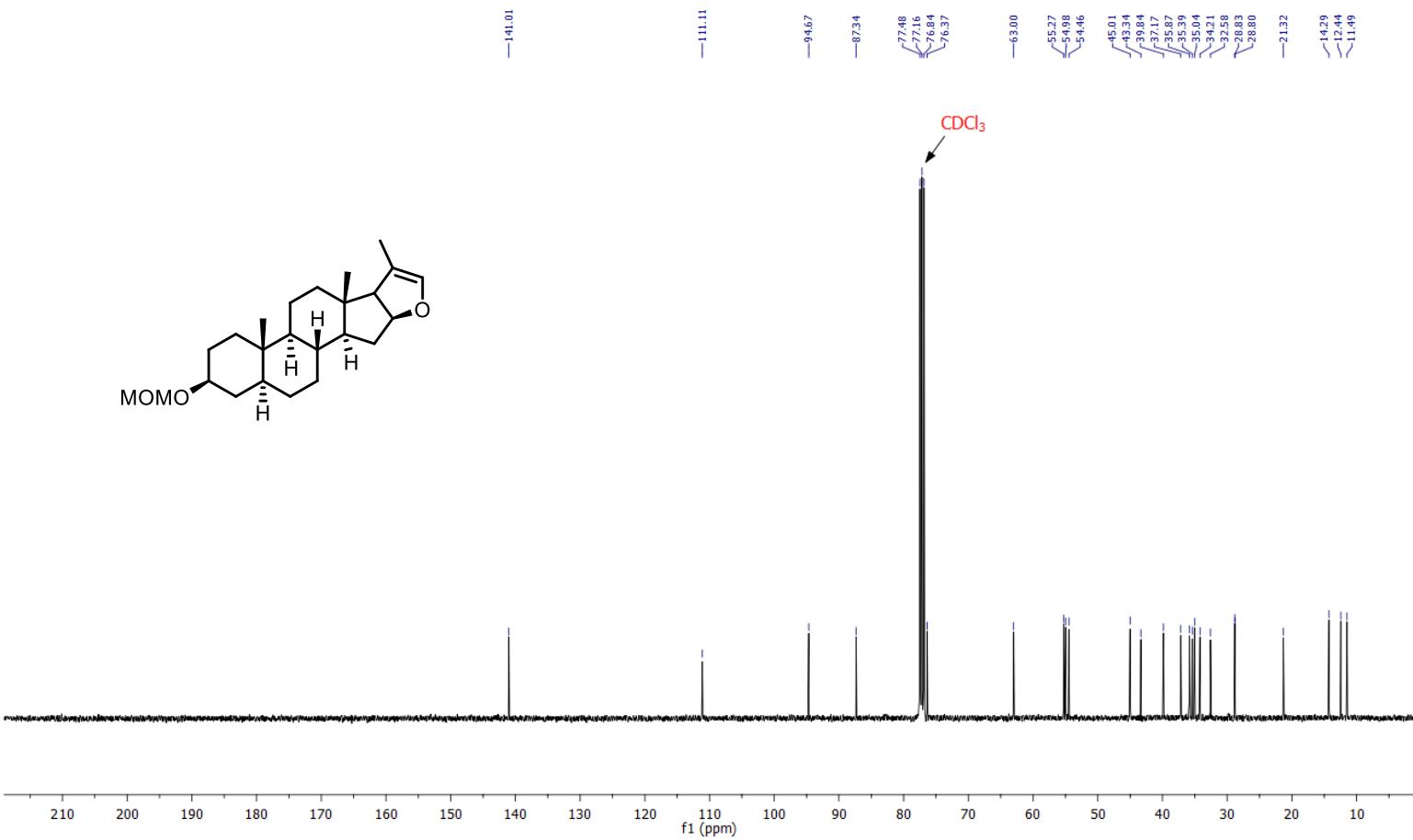
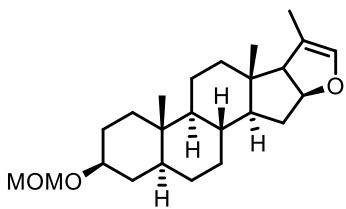


Figure S10. ^{13}C NMR (400 MHz, CDCl_3) of **12**

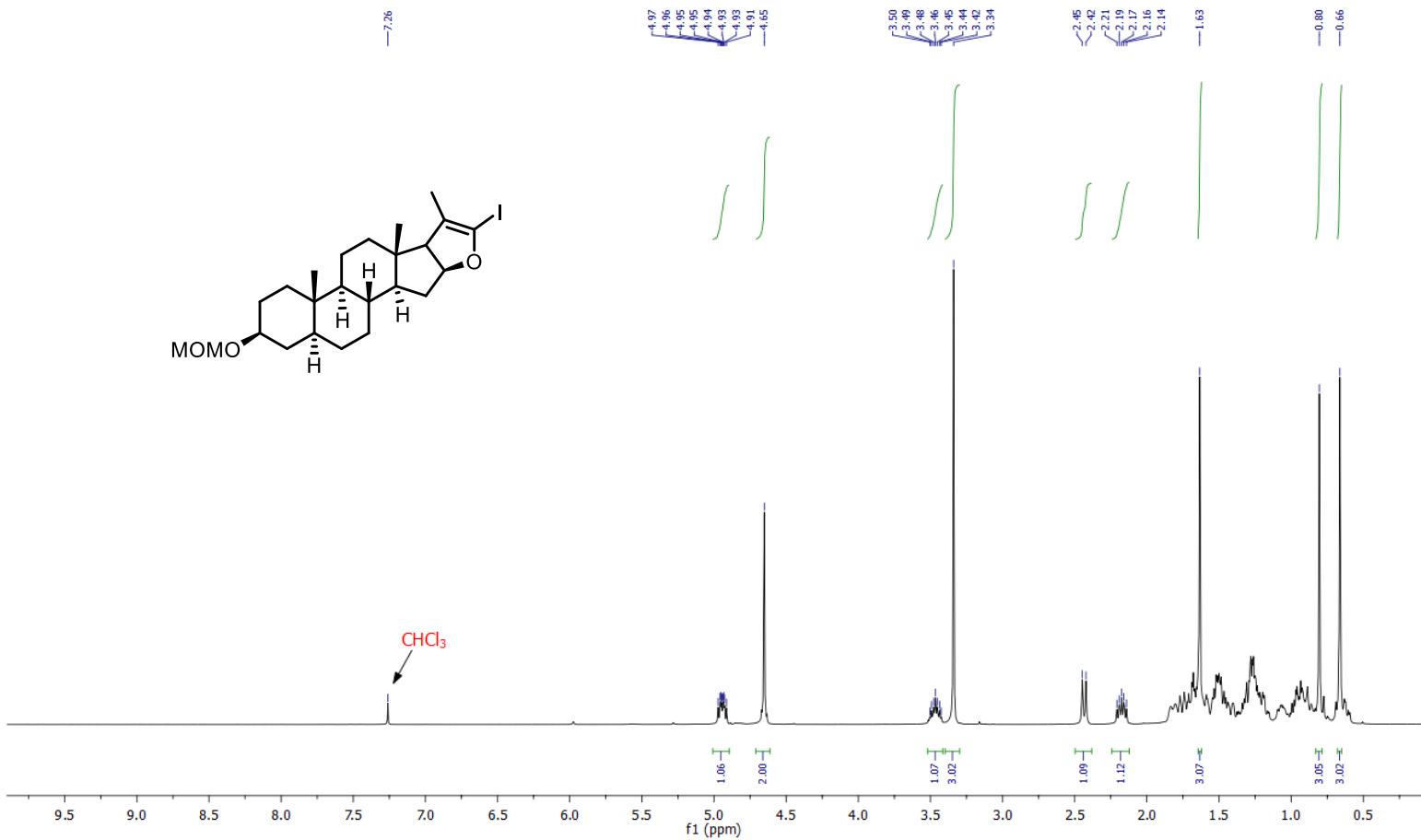


Figure S11. ^1H NMR (400 MHz, CDCl_3) of **13**

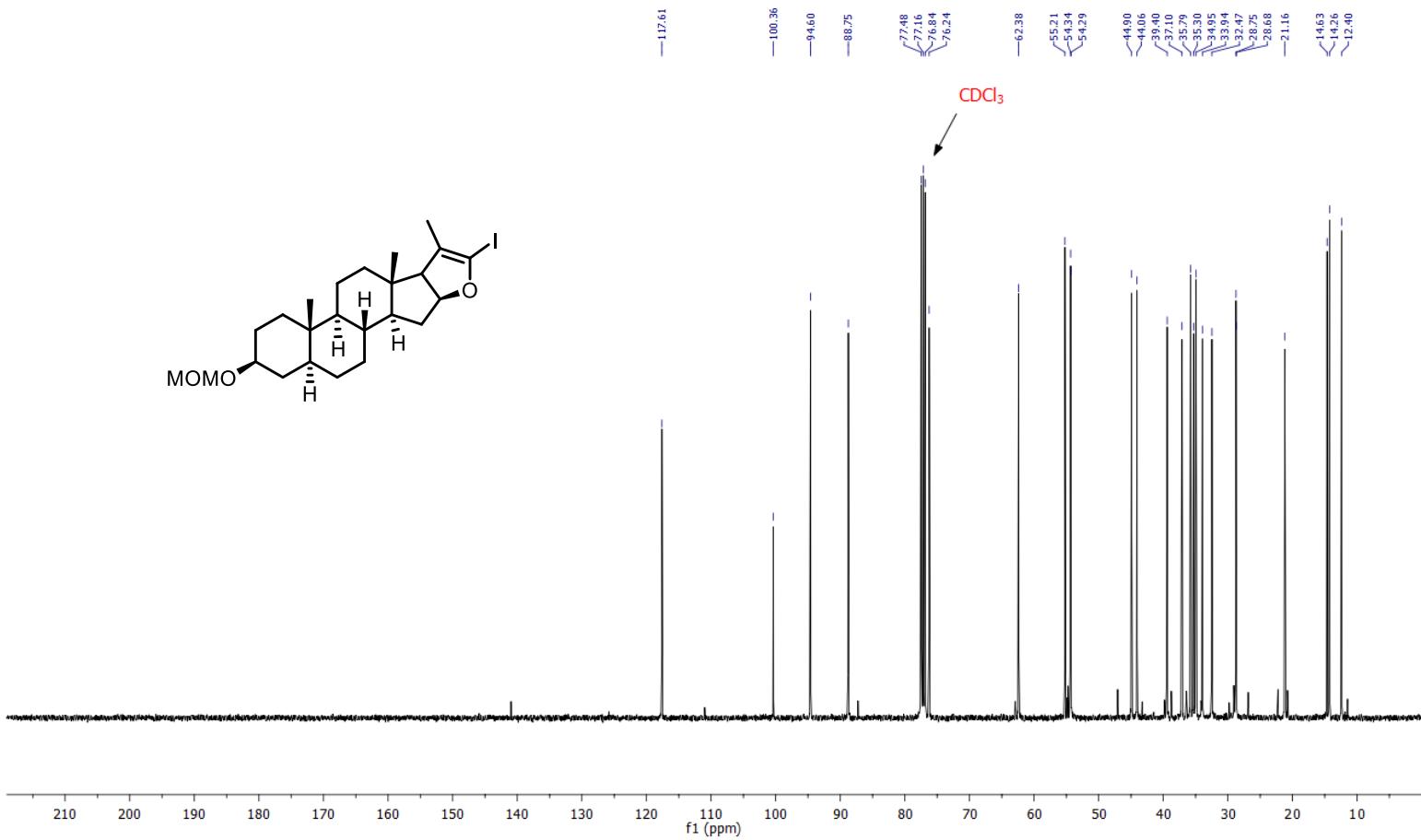


Figure S12. ^{13}C NMR (100 MHz, CDCl_3) of **13**

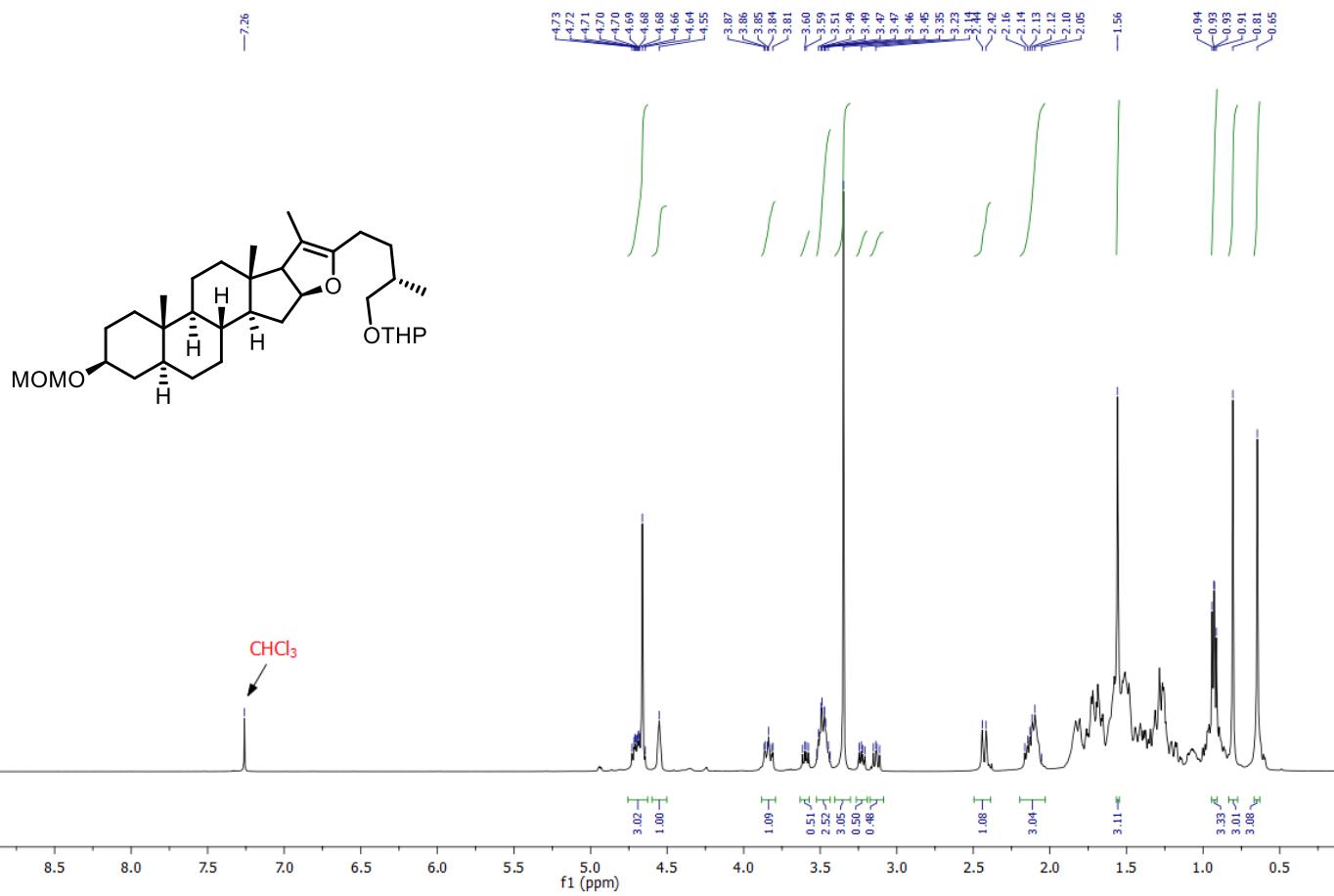


Figure S13. ^1H NMR (400 MHz, CDCl_3) of **7**

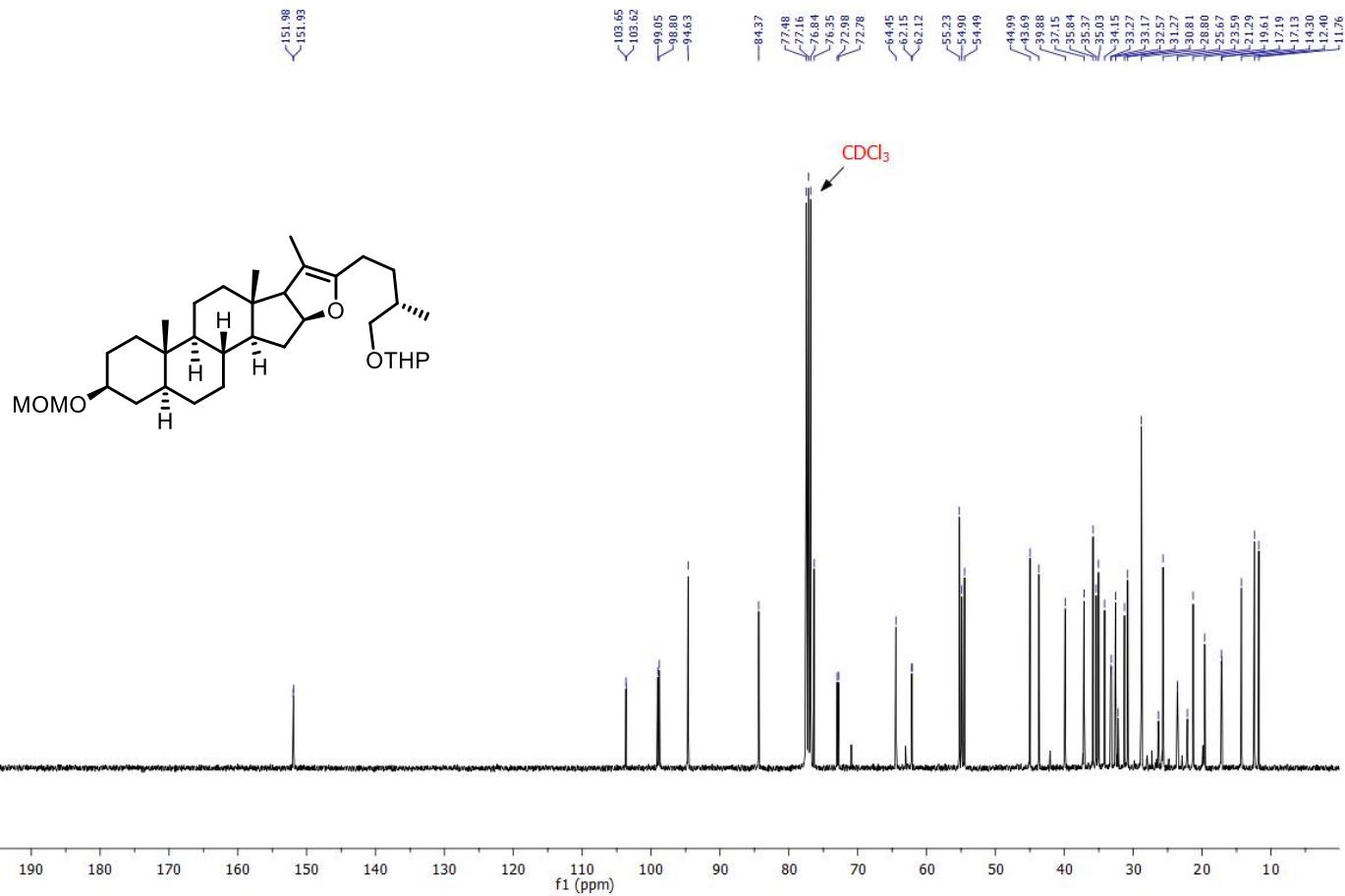


Figure S14. ^{13}C NMR (100 MHz, CDCl_3) of **7**

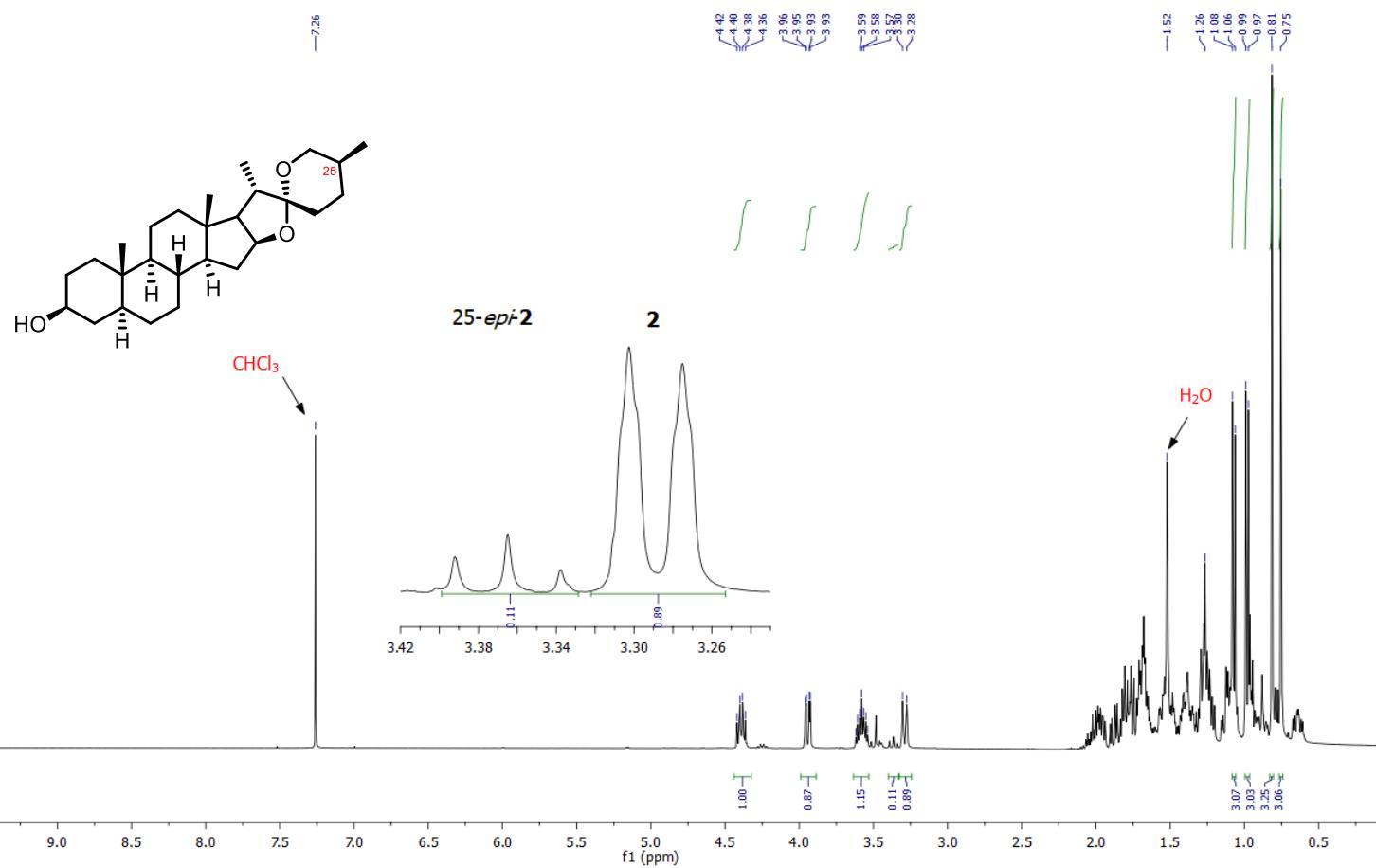


Figure S15. ^1H NMR (400 MHz, CDCl_3) of **2**

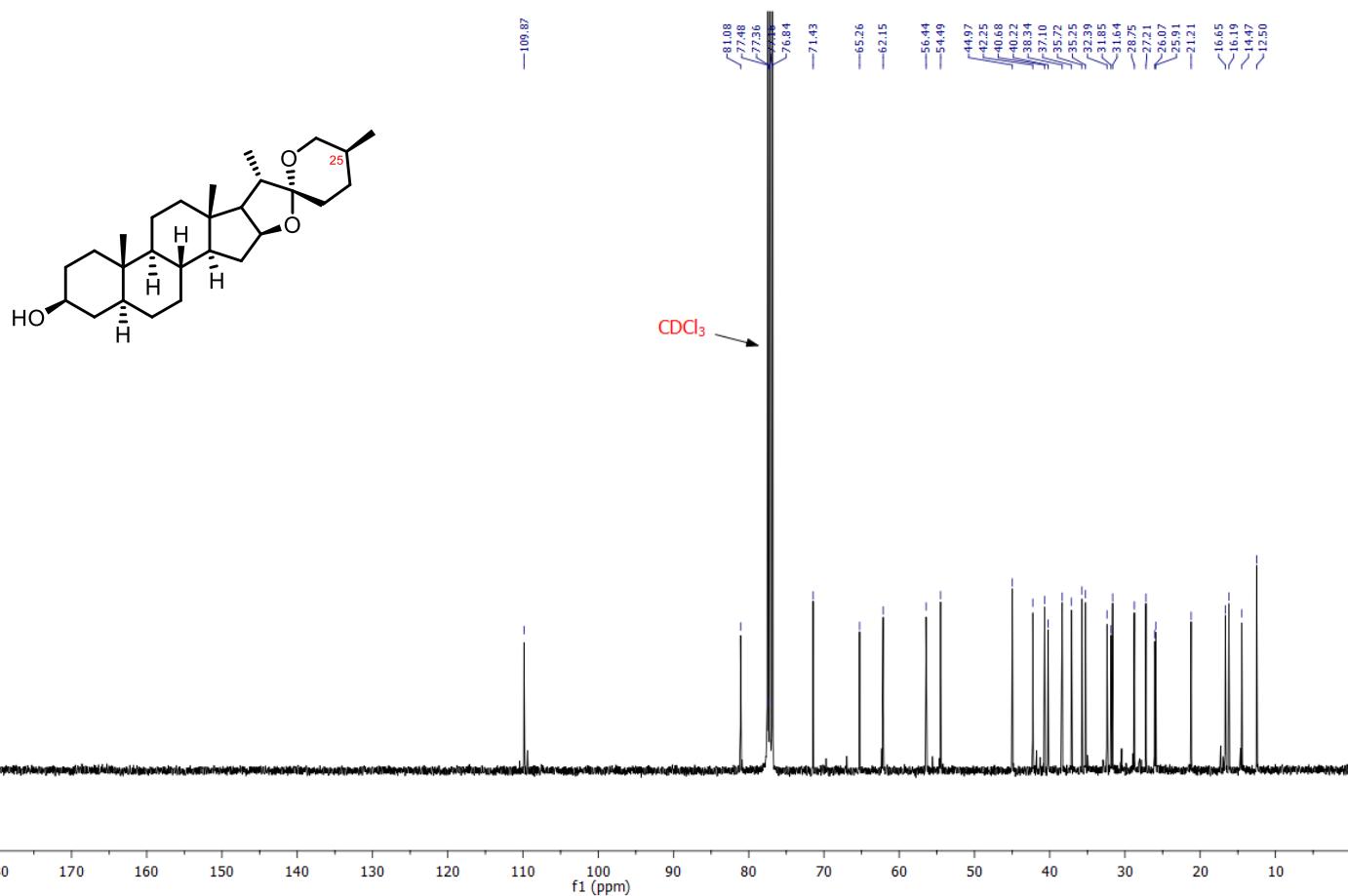


Figure S16. ^{13}C NMR (100 MHz, CDCl_3) of **2**

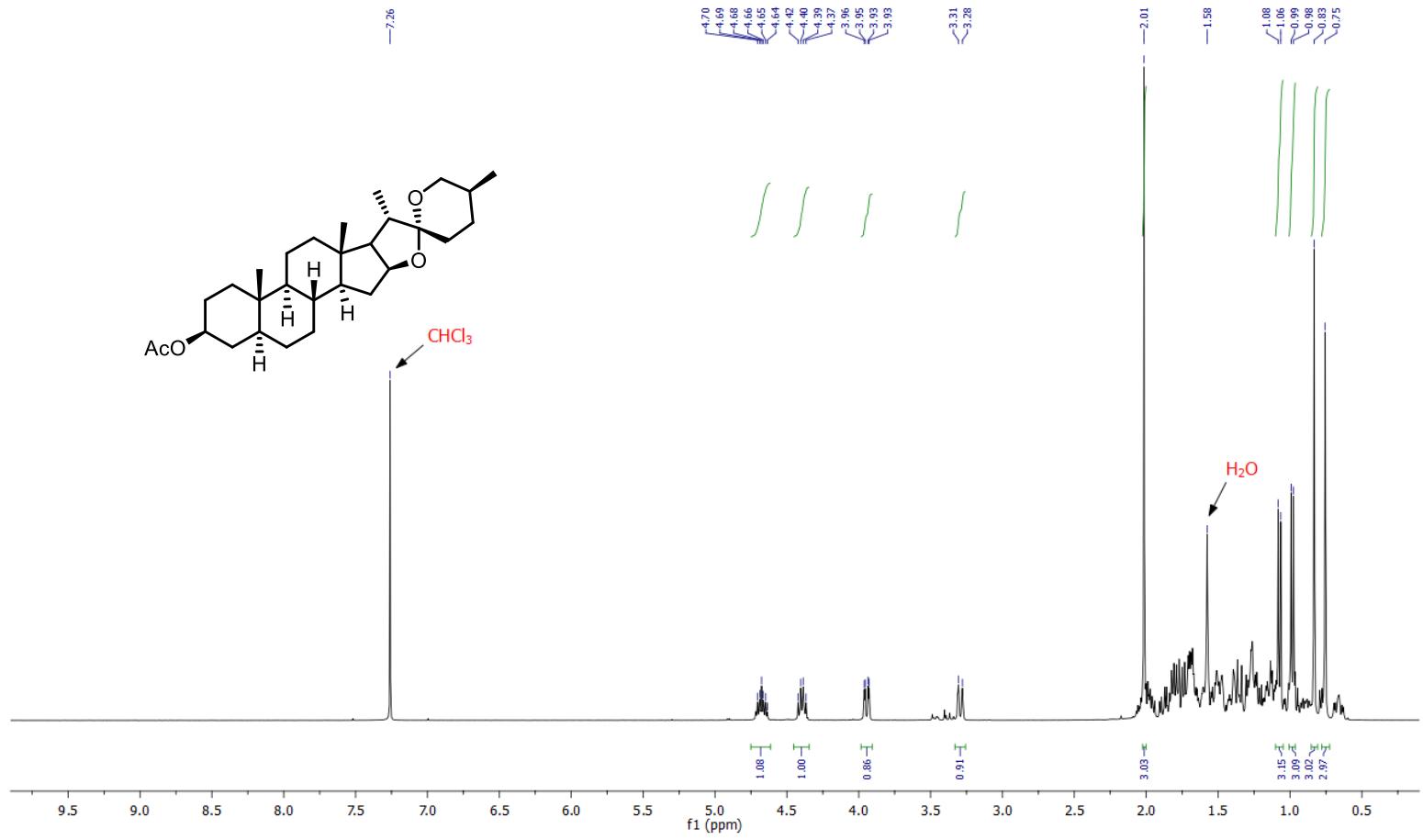
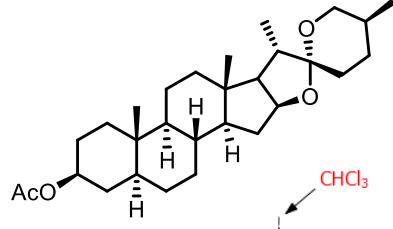


Figure S17. ^1H NMR (400 MHz, CDCl_3) of OAc-2

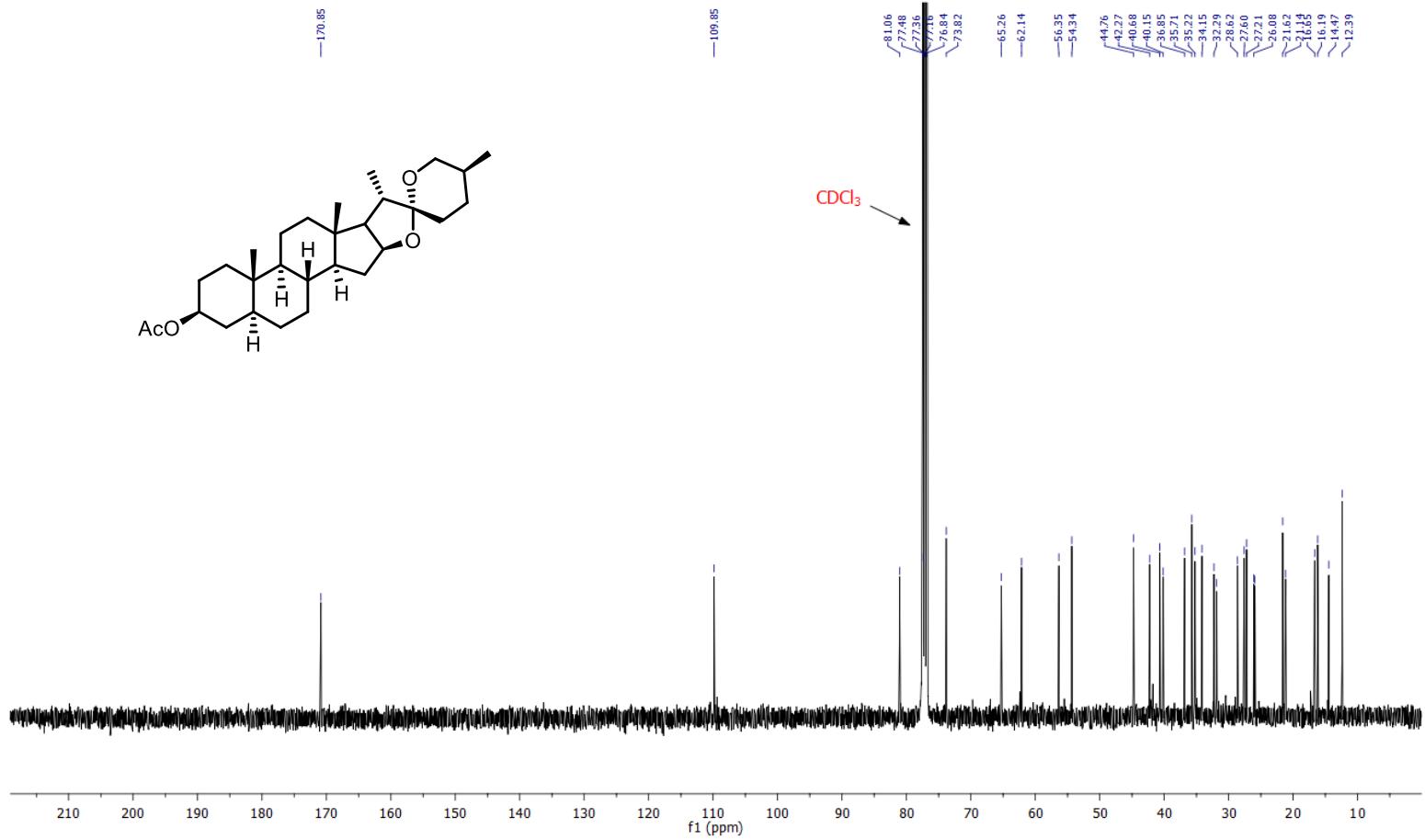
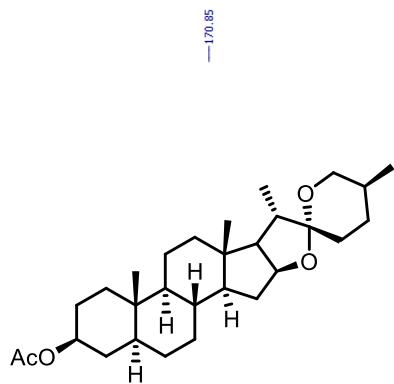


Figure S18. ^{13}C NMR (100 MHz, CDCl_3) of OAc-**2**

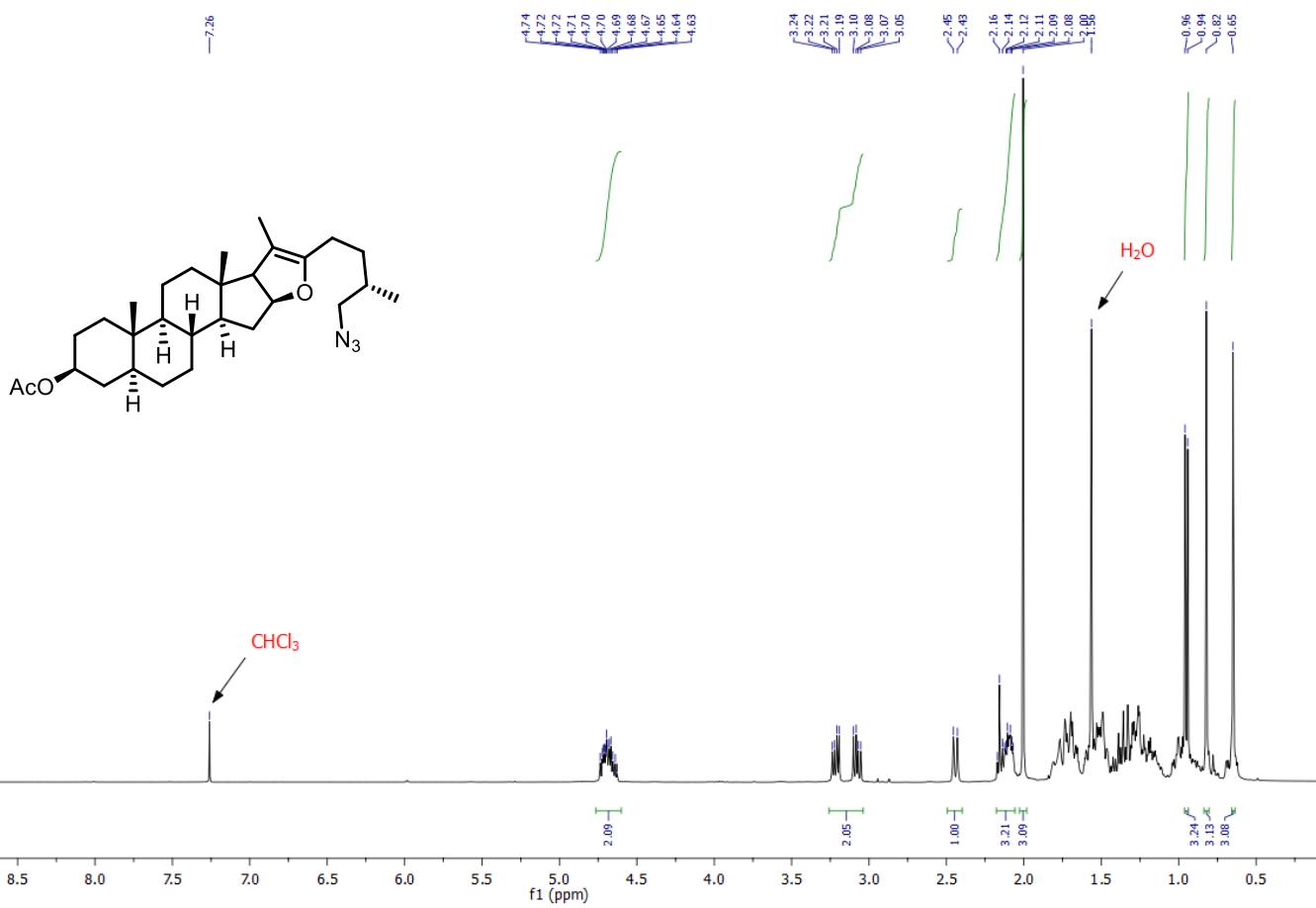


Figure S19. ¹H NMR (400 MHz, CDCl₃) of **16**

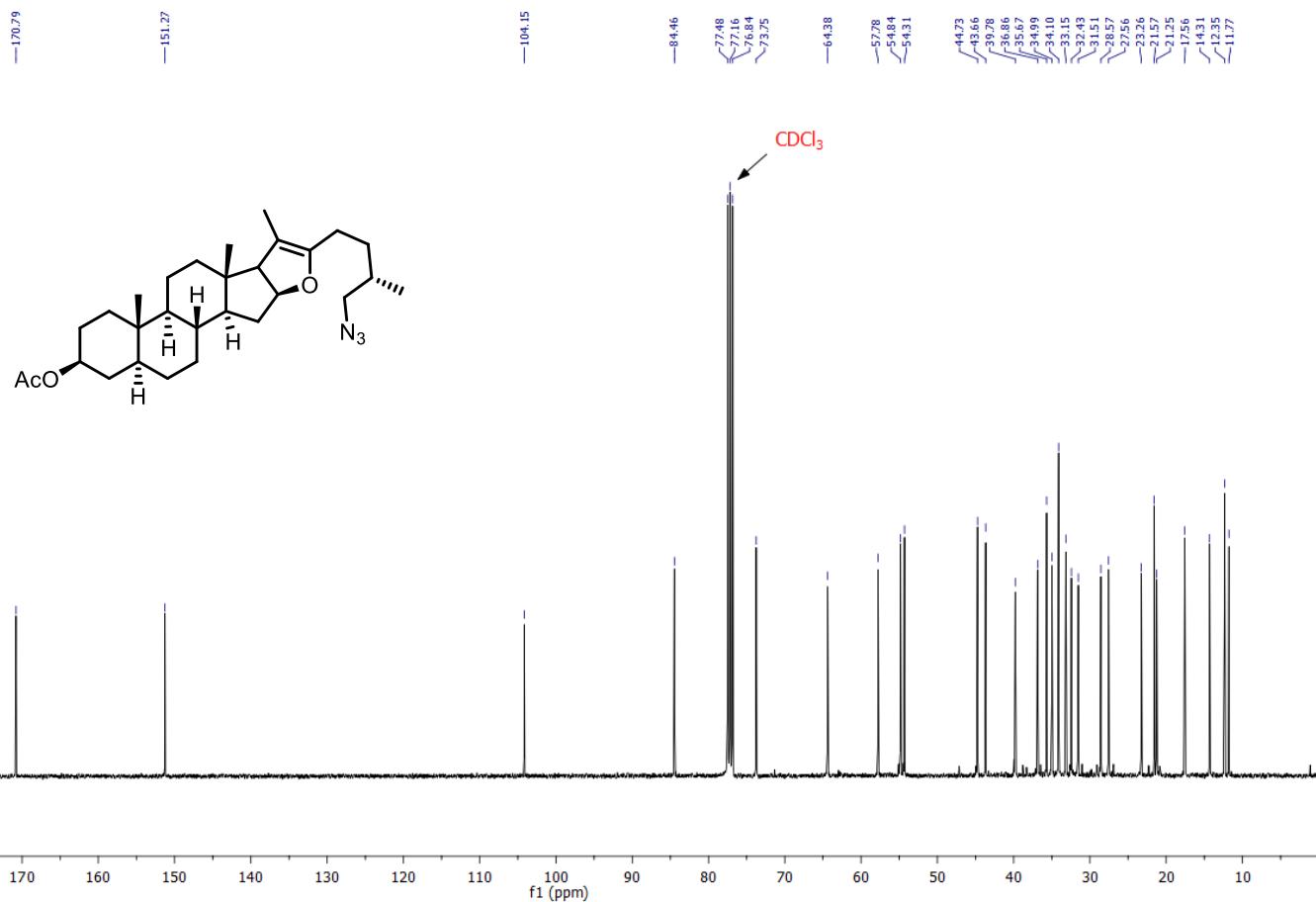
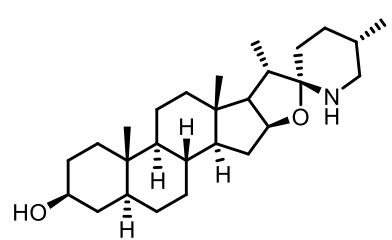


Figure S20. ¹³C NMR (100 MHz, CDCl₃) of **16**



1
17

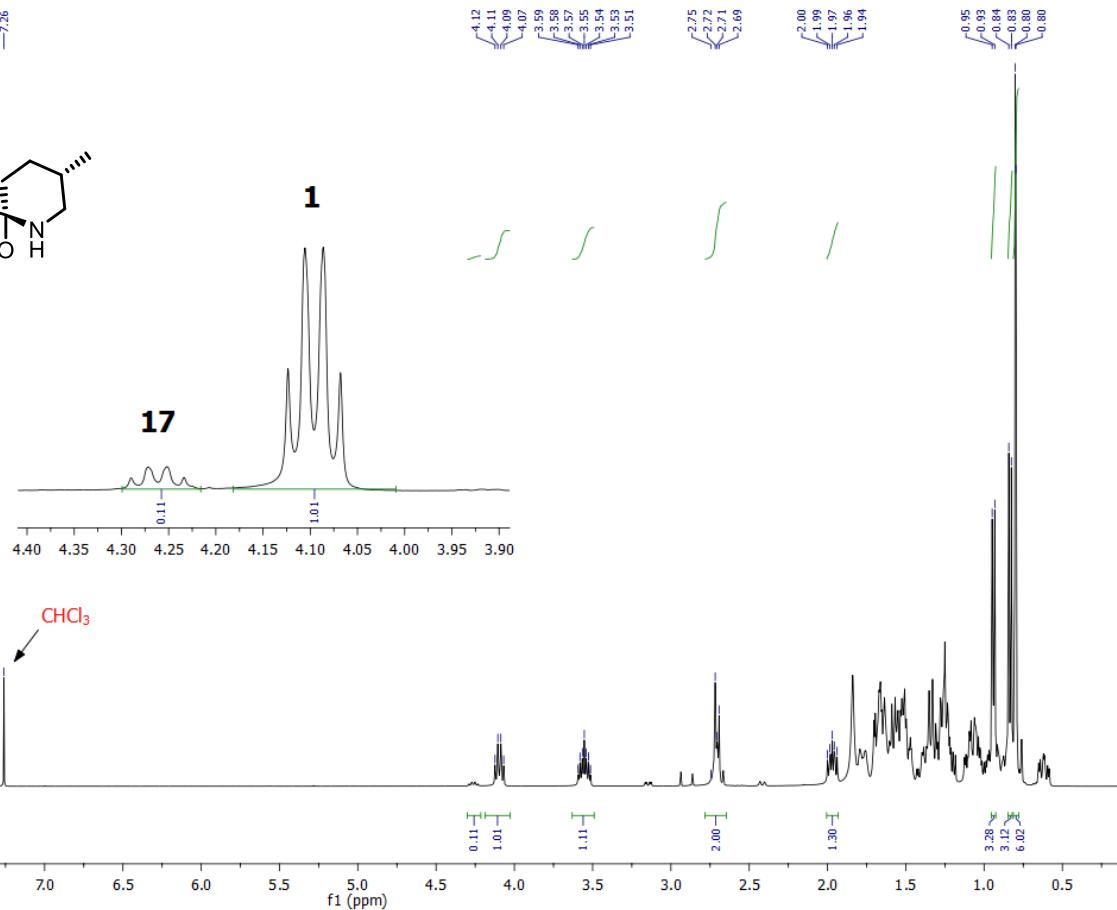
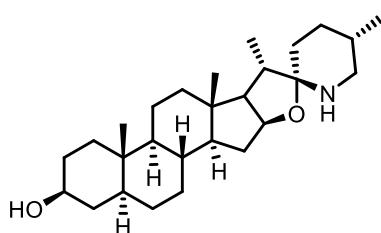


Figure S21. ¹H NMR (400 MHz, CDCl₃) of **1**



—99.14
—78.64
—77.48
—77.16
—76.84
—71.29
—62.10
—55.89
—54.53
—50.32
—44.96
—45.12
—40.99
—40.32
—38.31
—37.08
—35.69
—35.16
—32.41
—31.60
—20.71
—20.65
—21.19
—19.46
—17.06
—15.99
—0.95
—0.93
—0.84
—0.83
—0.80
—0.80

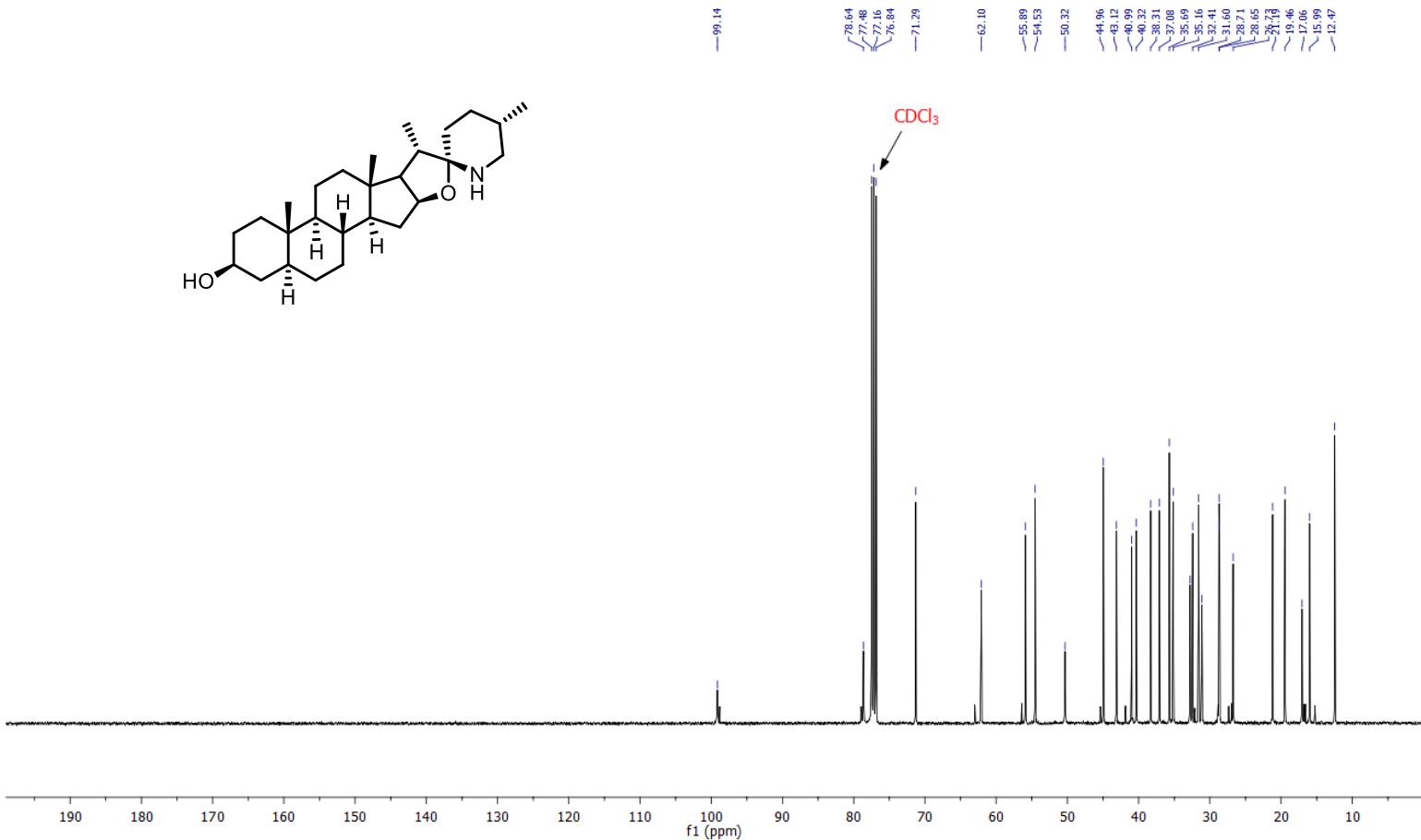
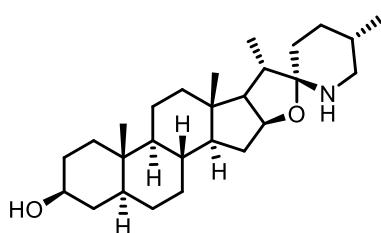


Figure S22. ¹³C NMR (100 MHz, CDCl₃) of **1**

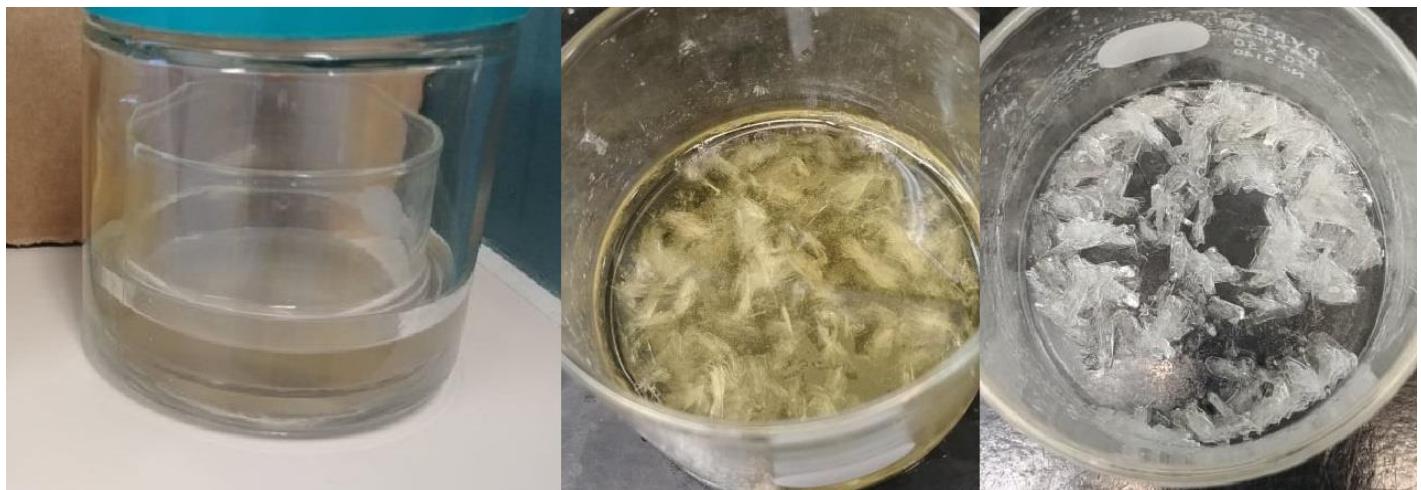


Figure S23. Vapor Diffusion Apparatus for the Crystallization of 1