

Supporting Information

Unusual Vilasinin-Class Limonoids from *Trichilia rubescens*

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Dose Response Curves

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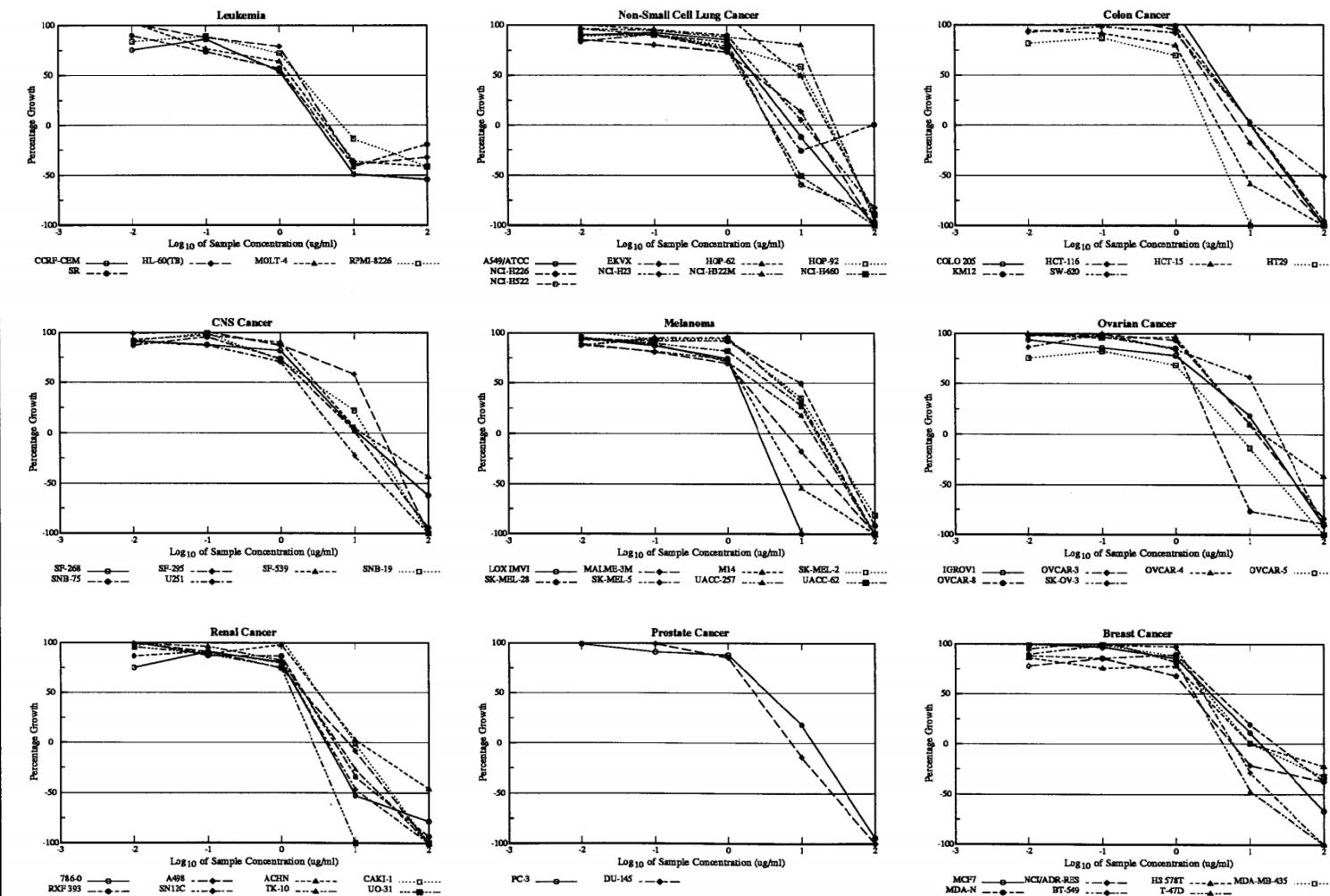


Figure S1. NCI-60 Human tumor cell line assay data for the crude organic extract of *T. rubescens* (N047159)

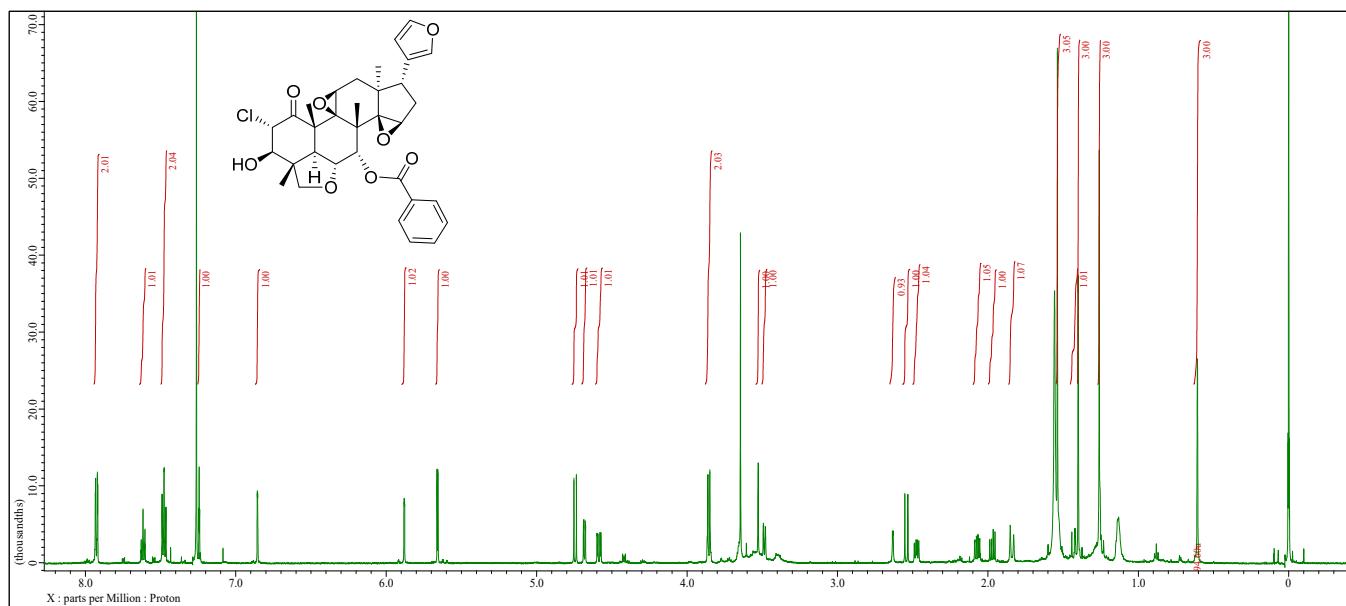


Figure S2. ^1H NMR spectrum of compound 1 in CDCl_3 at 600MHz

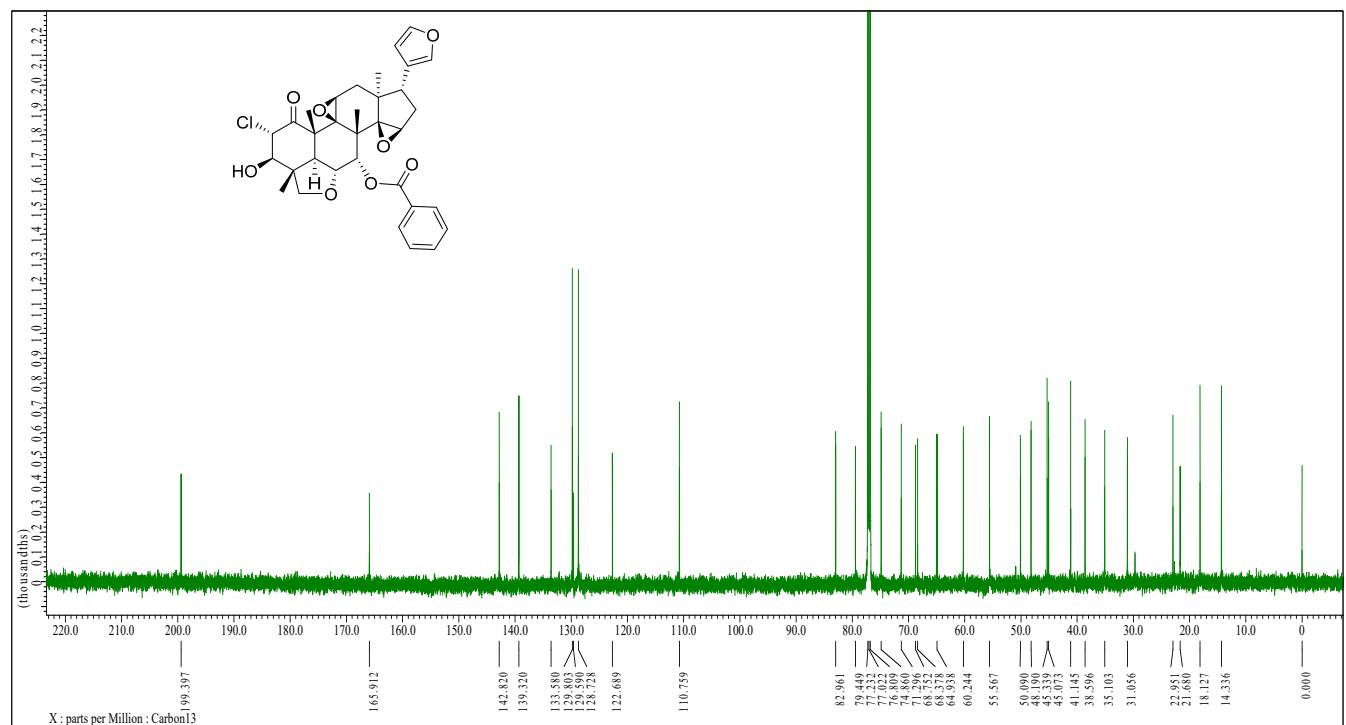


Figure S3. ^{13}C NMR spectrum of compound 1 in CDCl_3 at 150MHz

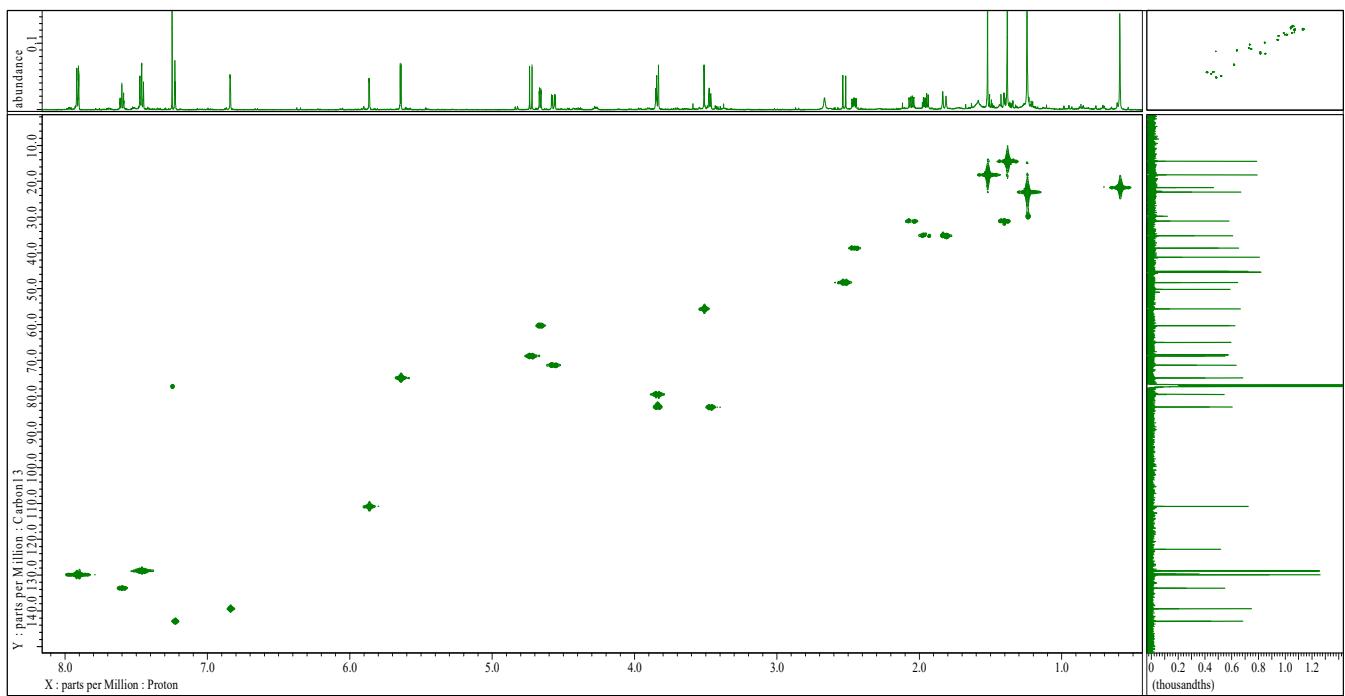


Figure S4. HMQC spectrum of compound **1** in CDCl_3 at 600MHz

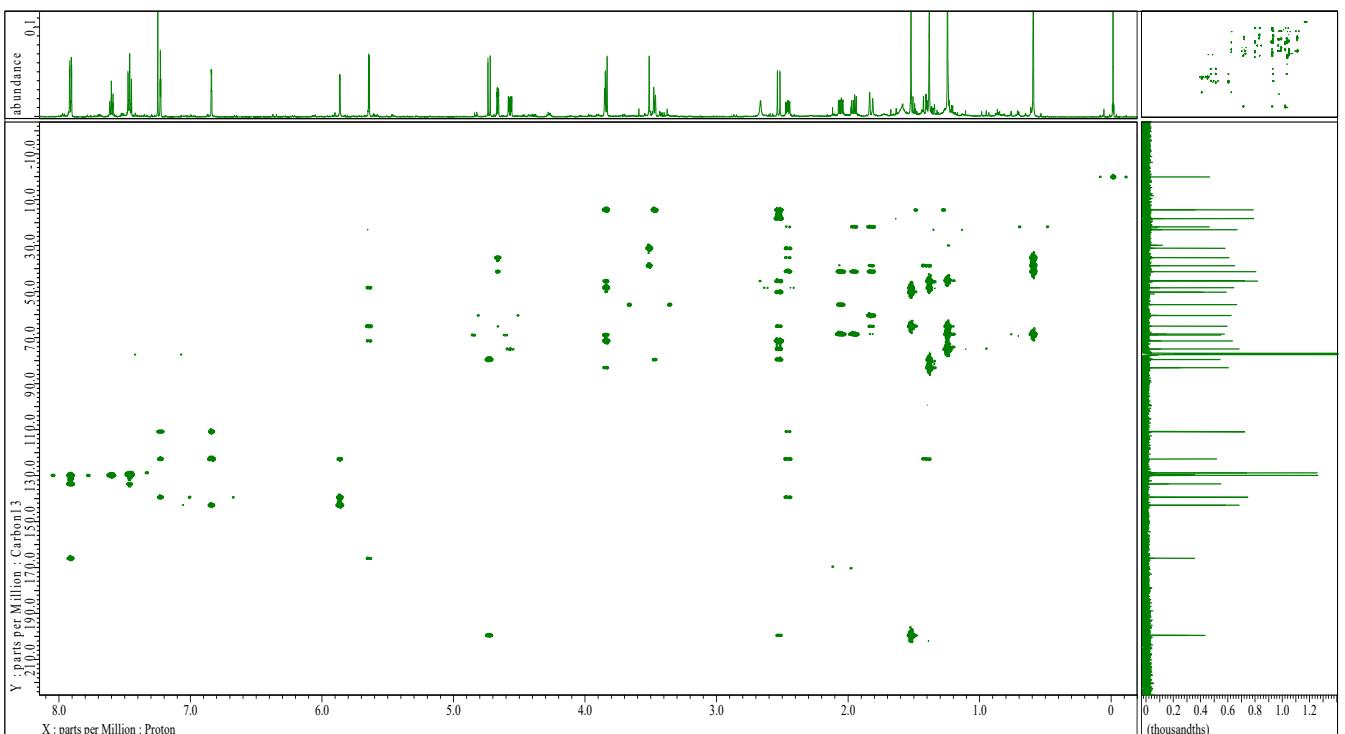


Figure S5. HMBC spectrum of compound **1** in CDCl_3 at 600MHz

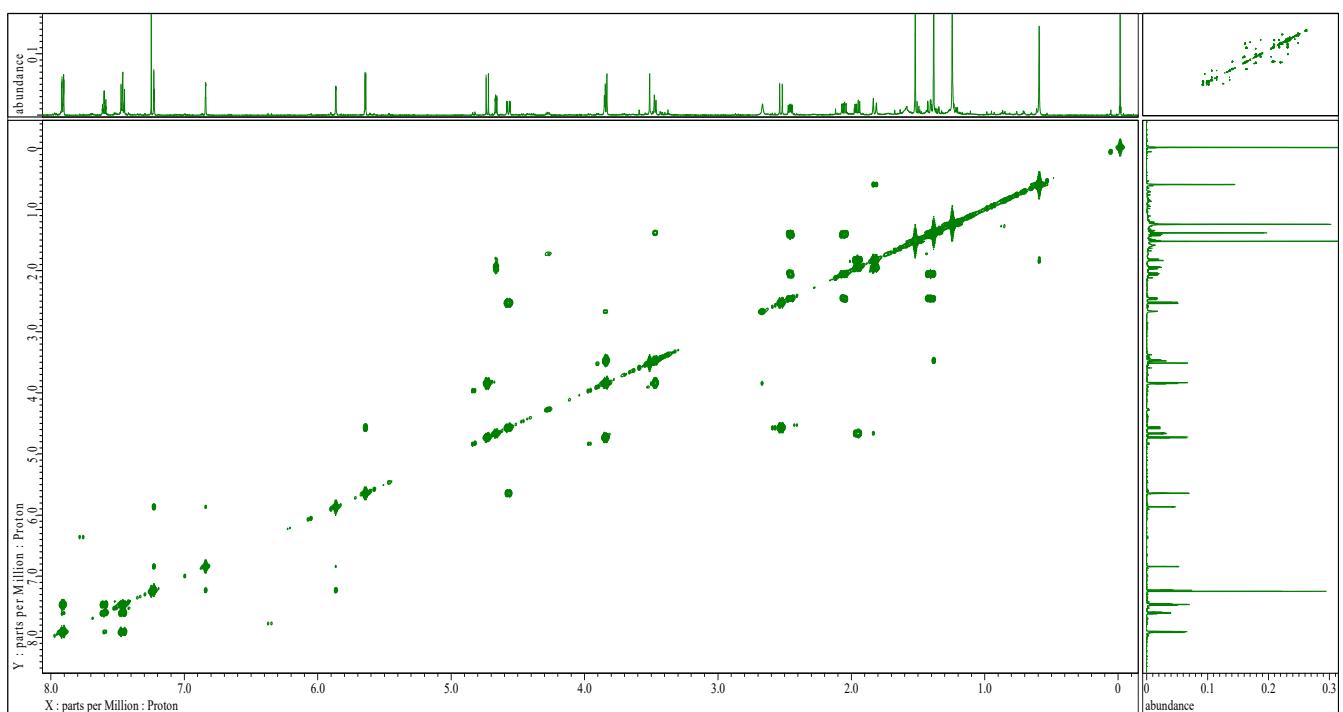


Figure S6. COSY spectrum of compound **1** in CDCl_3 at 600MHz

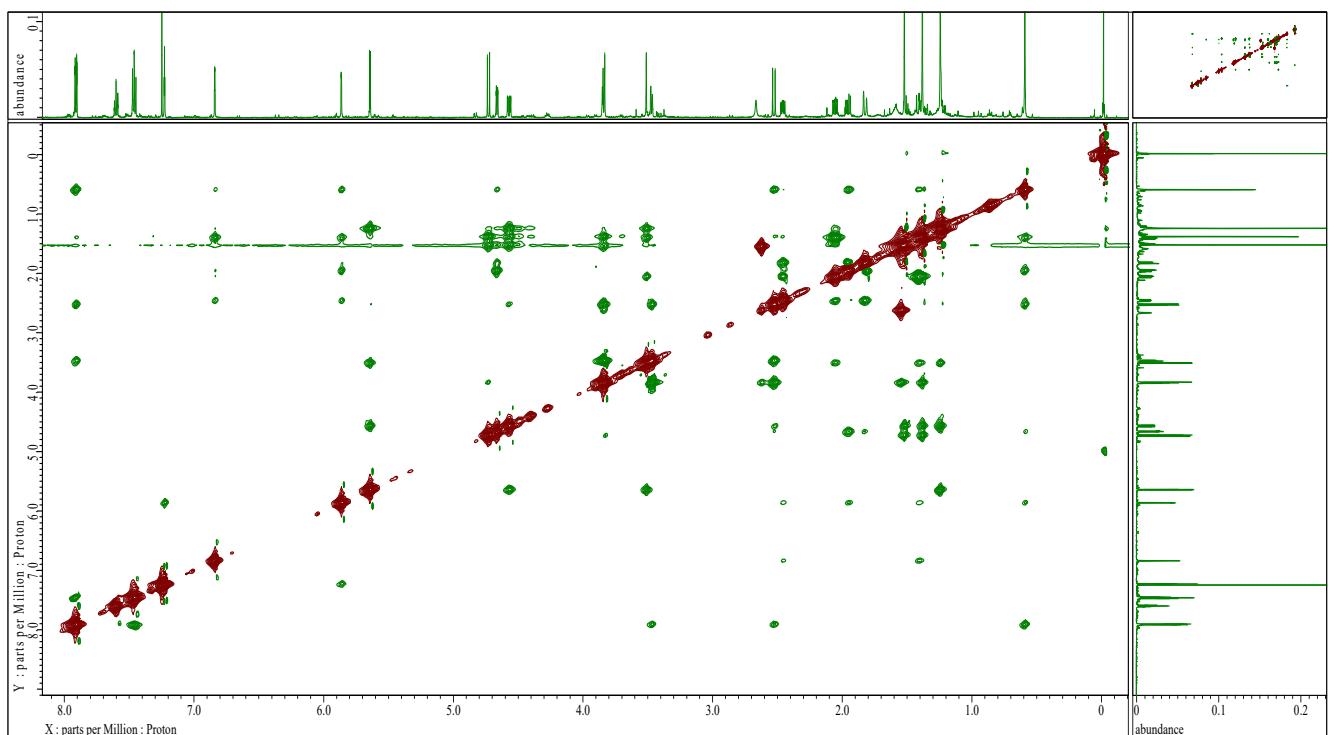


Figure S7. NOESY spectrum of compound **1** in CDCl_3 at 600MHz

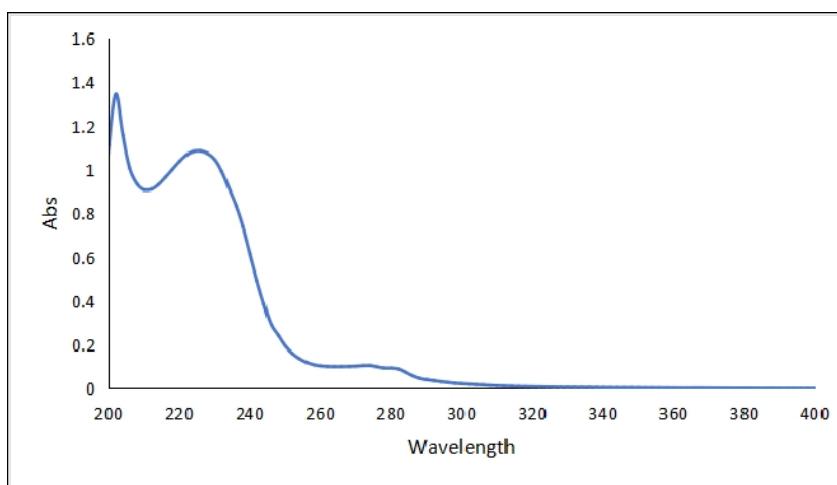


Figure S8. UV spectrum of compound **1** in MeOH

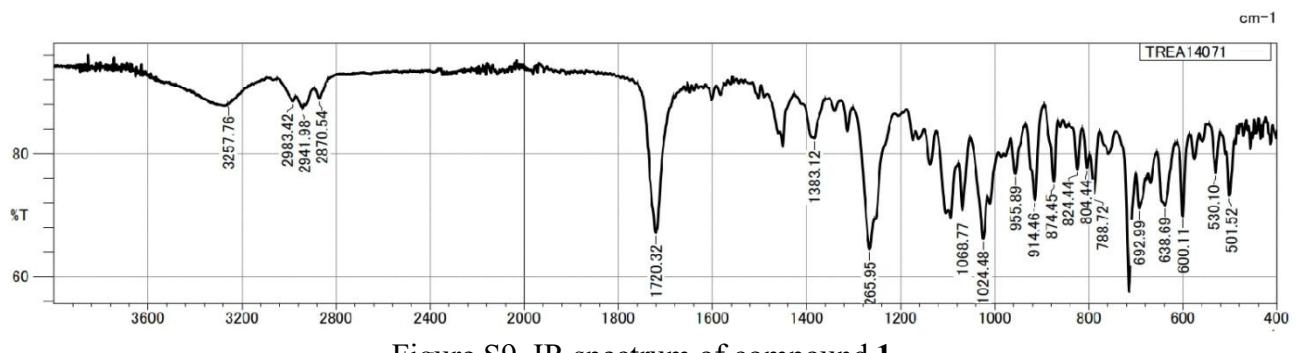


Figure S9. IR spectrum of compound **1**

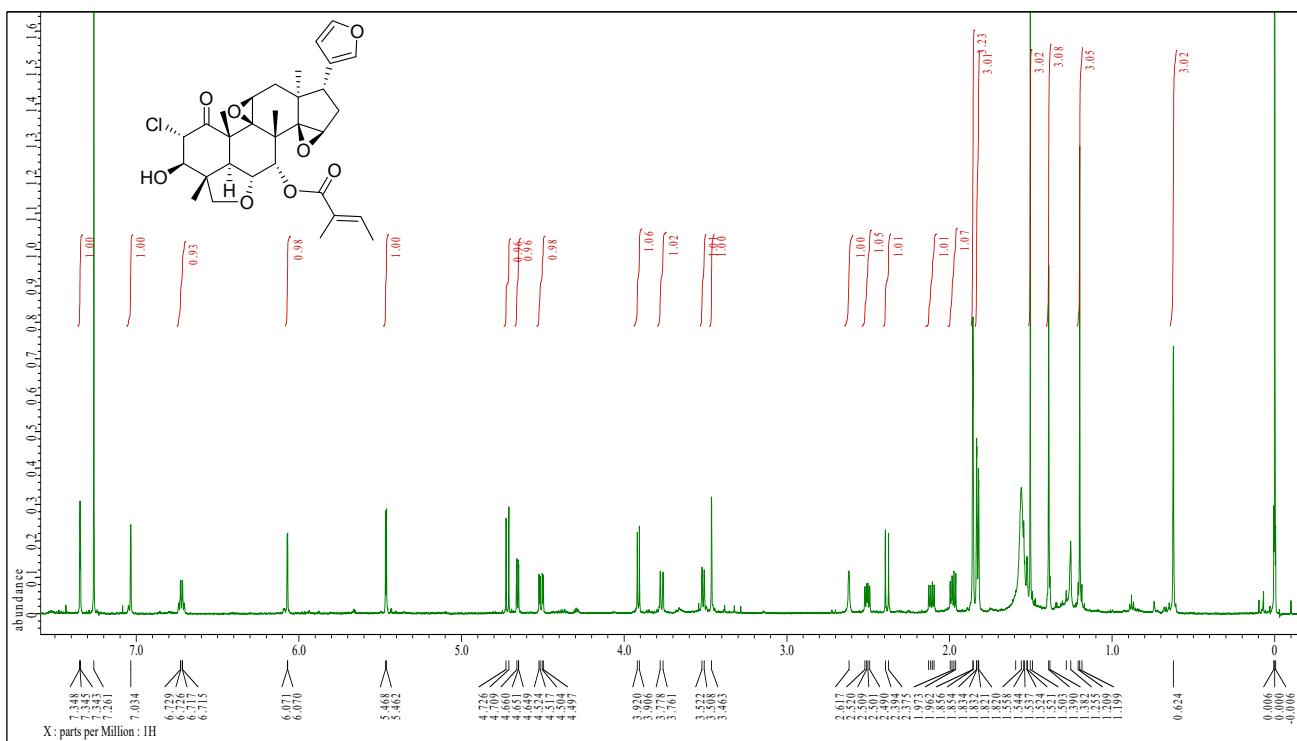


Figure S10. ^1H NMR spectrum of compound **2** in CDCl_3 at 600MHz

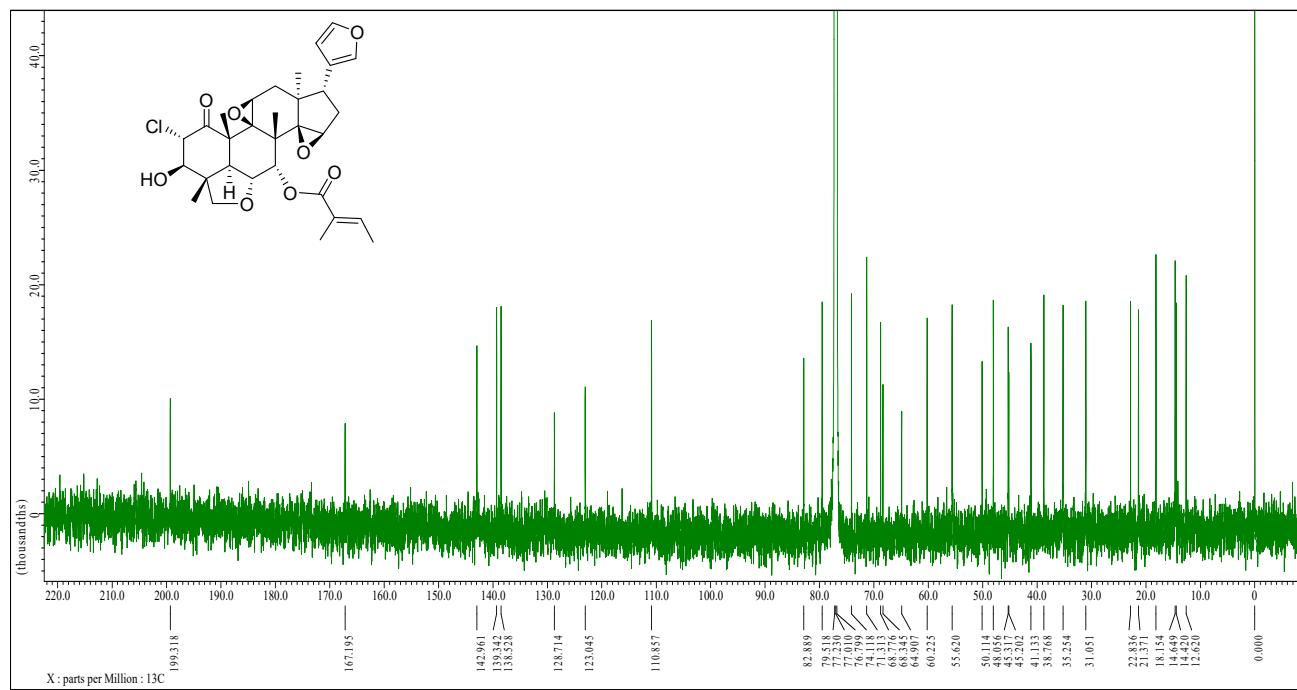


Figure S11.¹³C NMR spectrum of compound **2** in CDCl₃ at 150MHz

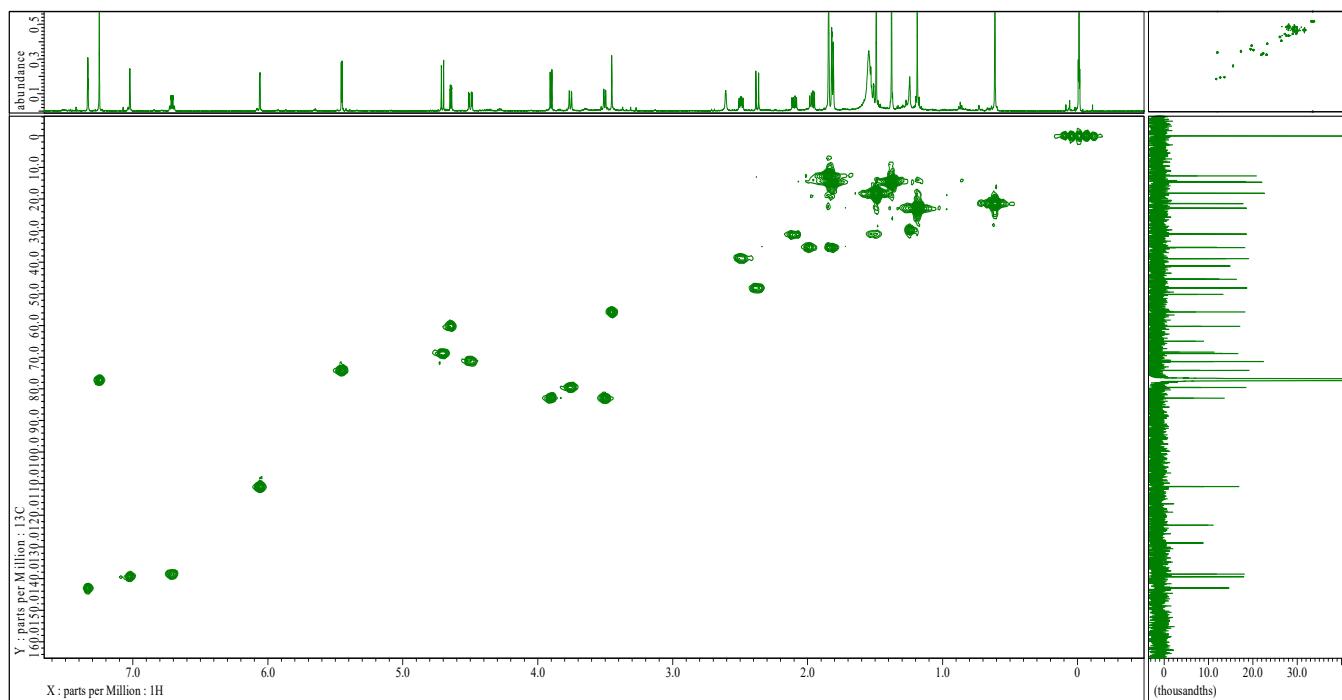


Figure S12. HMQC spectrum of compound **2** in CDCl_3 at 600MHz

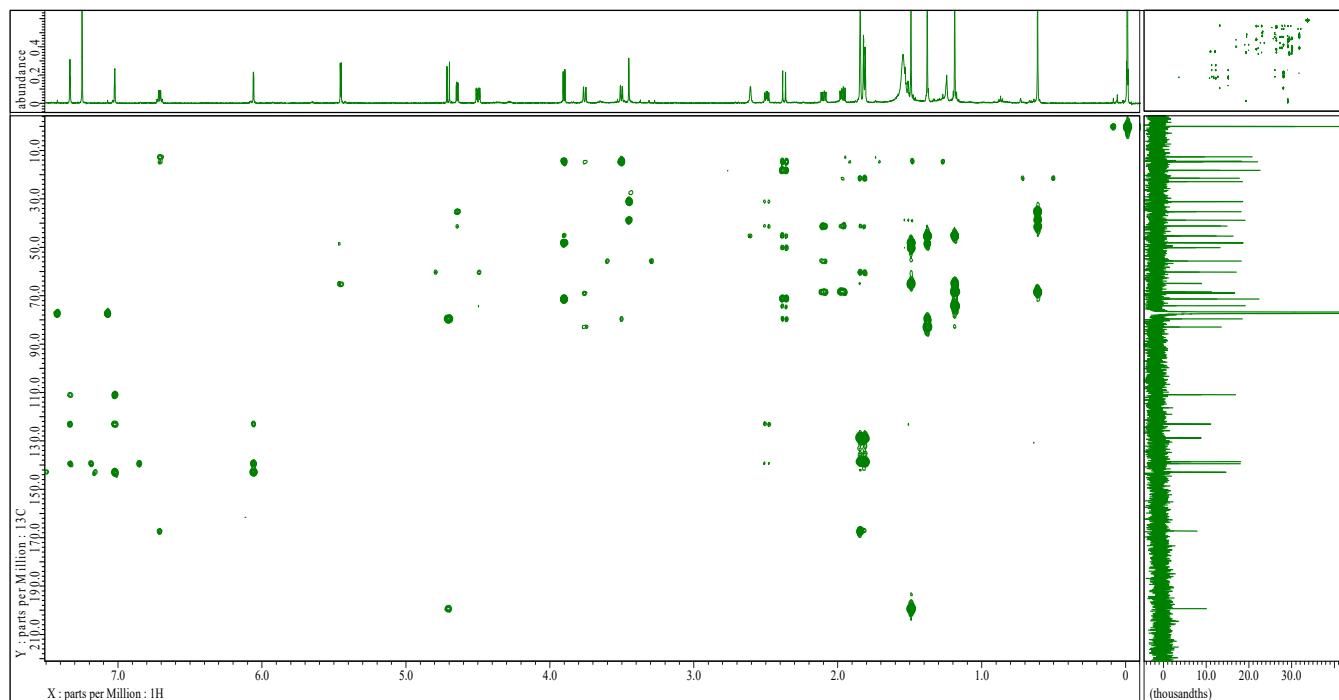


Figure S13. HMBC spectrum of compound **2** in CDCl_3 at 600MHz

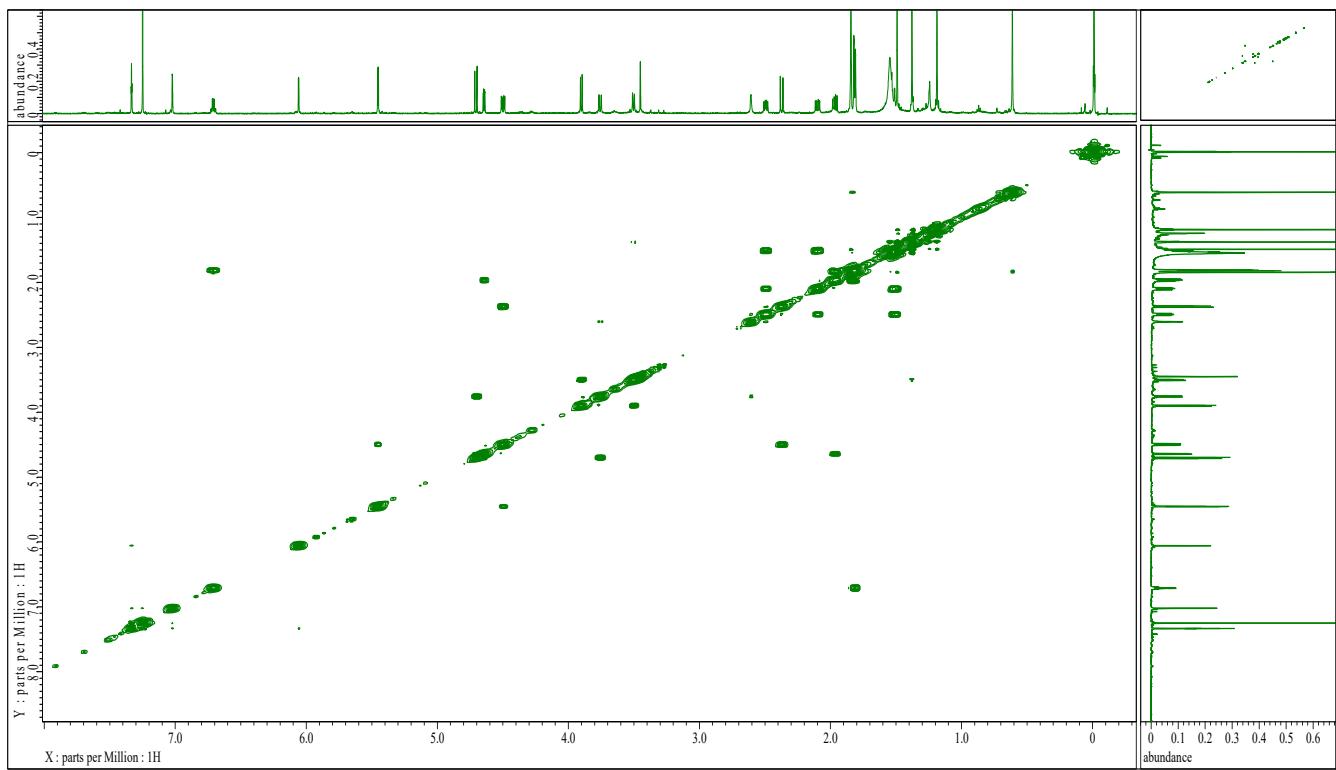


Figure S14. COSY spectrum of compound **2** in CDCl_3 at 600MHz

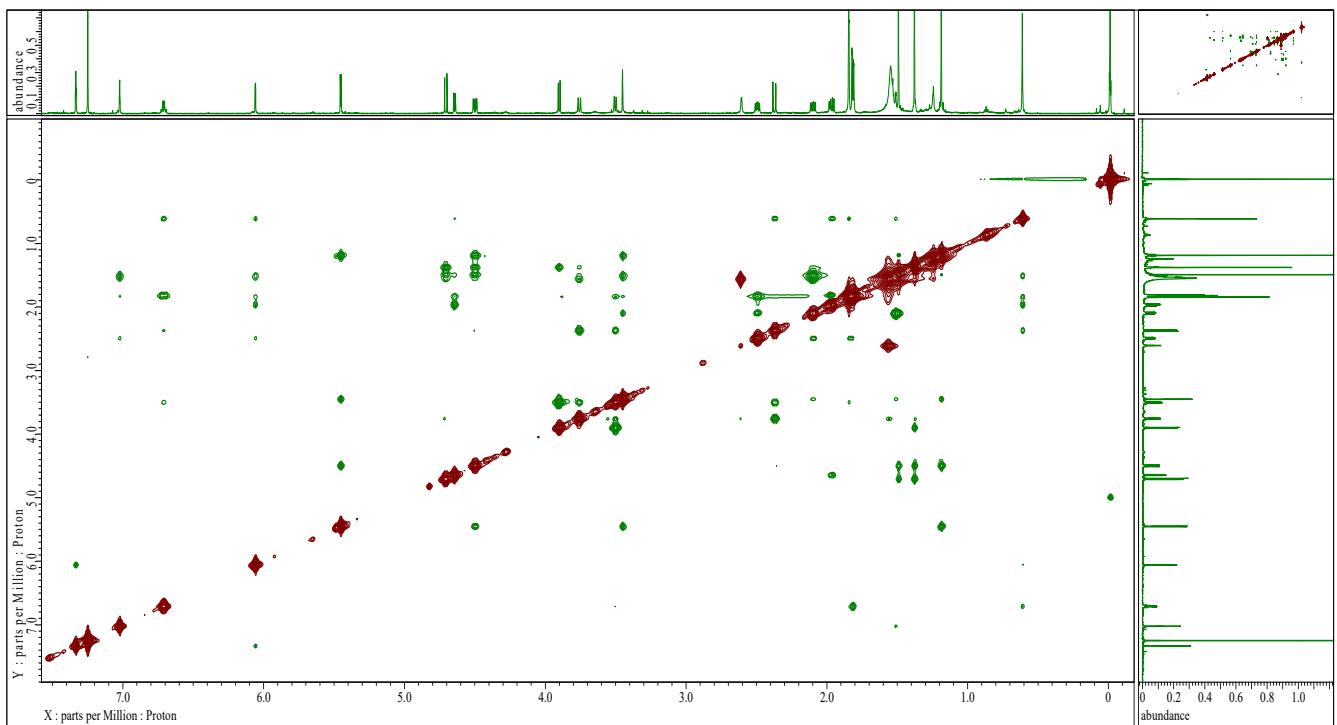


Figure S15. NOESY spectrum of compound **2** in CDCl_3 at 600MHz

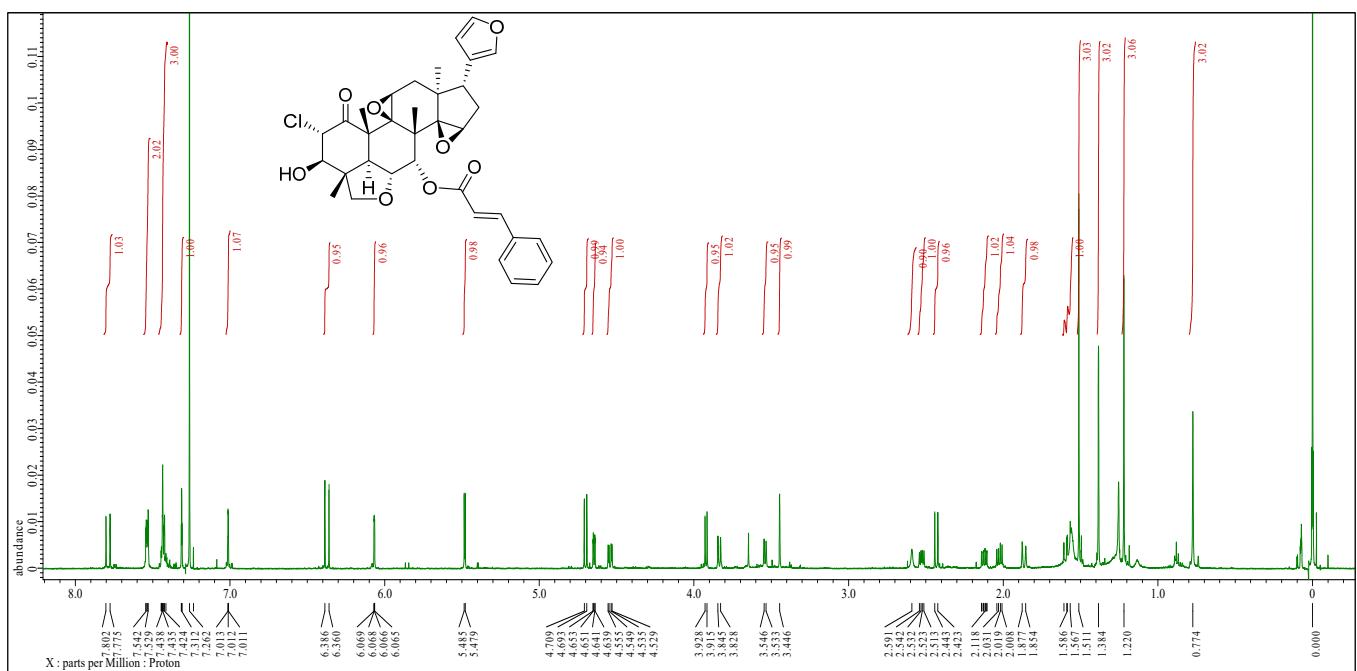


Figure S16. ^1H NMR spectrum of compound 3 in CDCl_3 at 600MHz

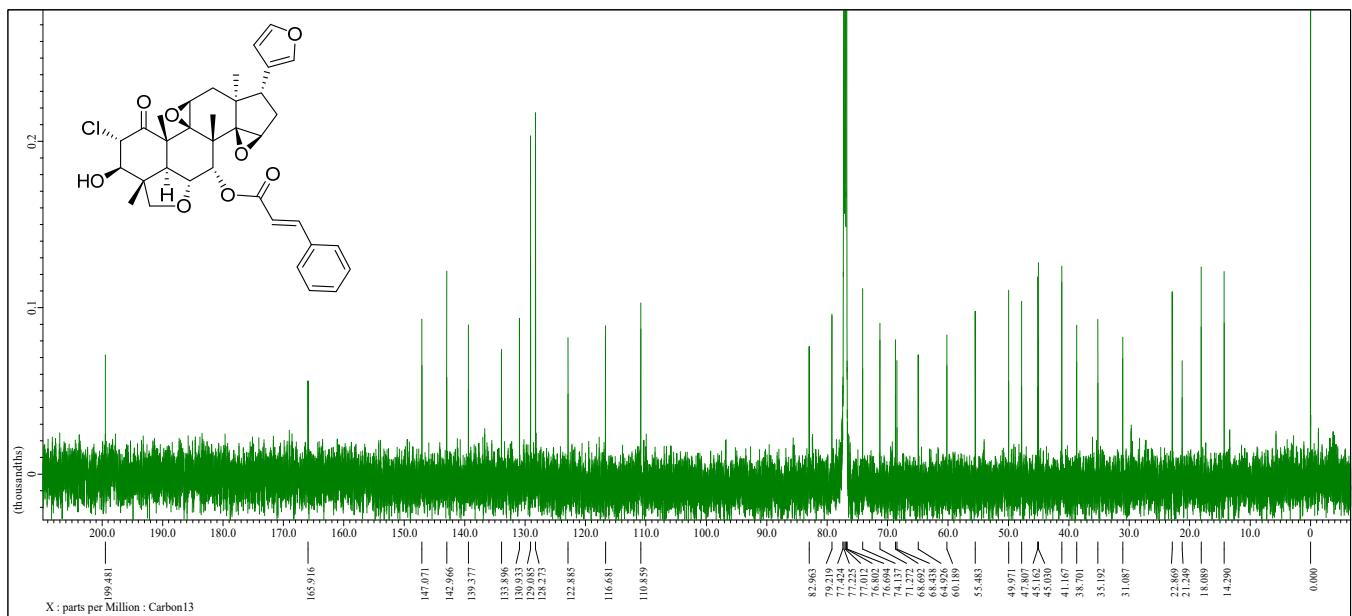


Figure S17. ^{13}C NMR spectrum of compound 3 in CDCl_3 at 150MHz

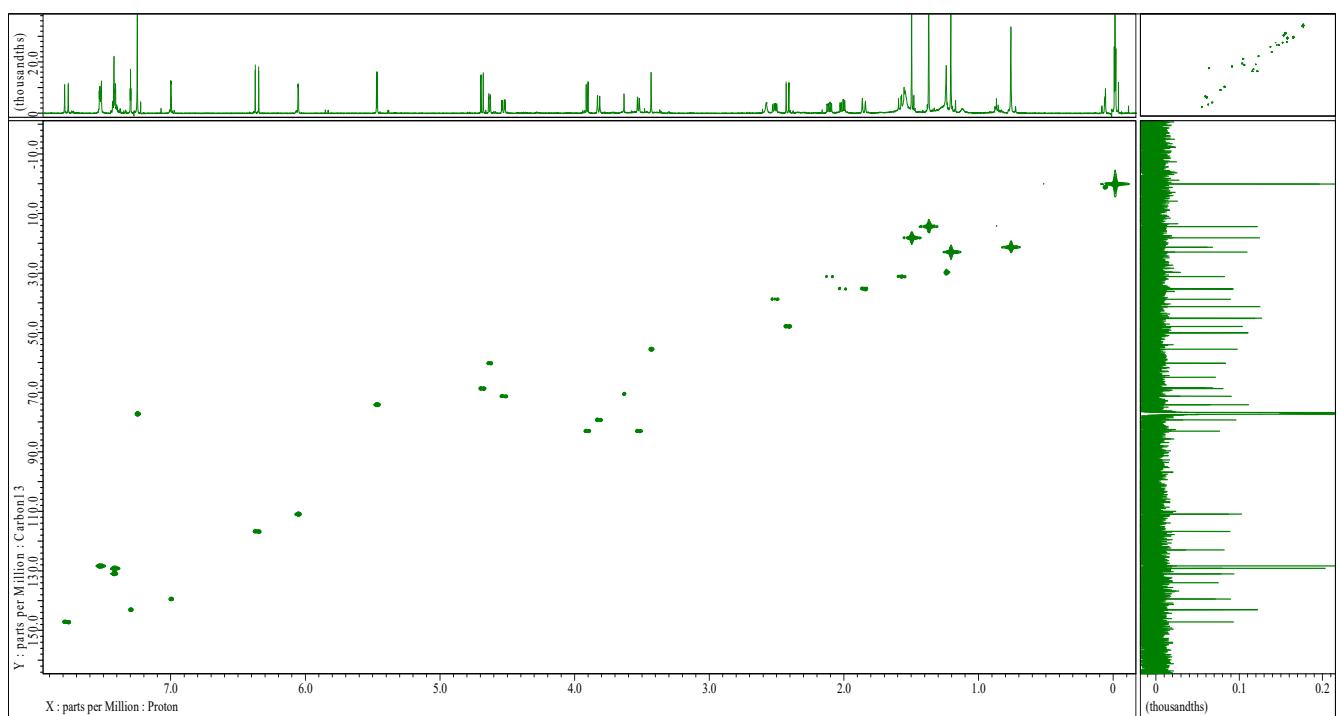


Figure S18. HMQC spectrum of compound **3** in CDCl_3 at 600MHz

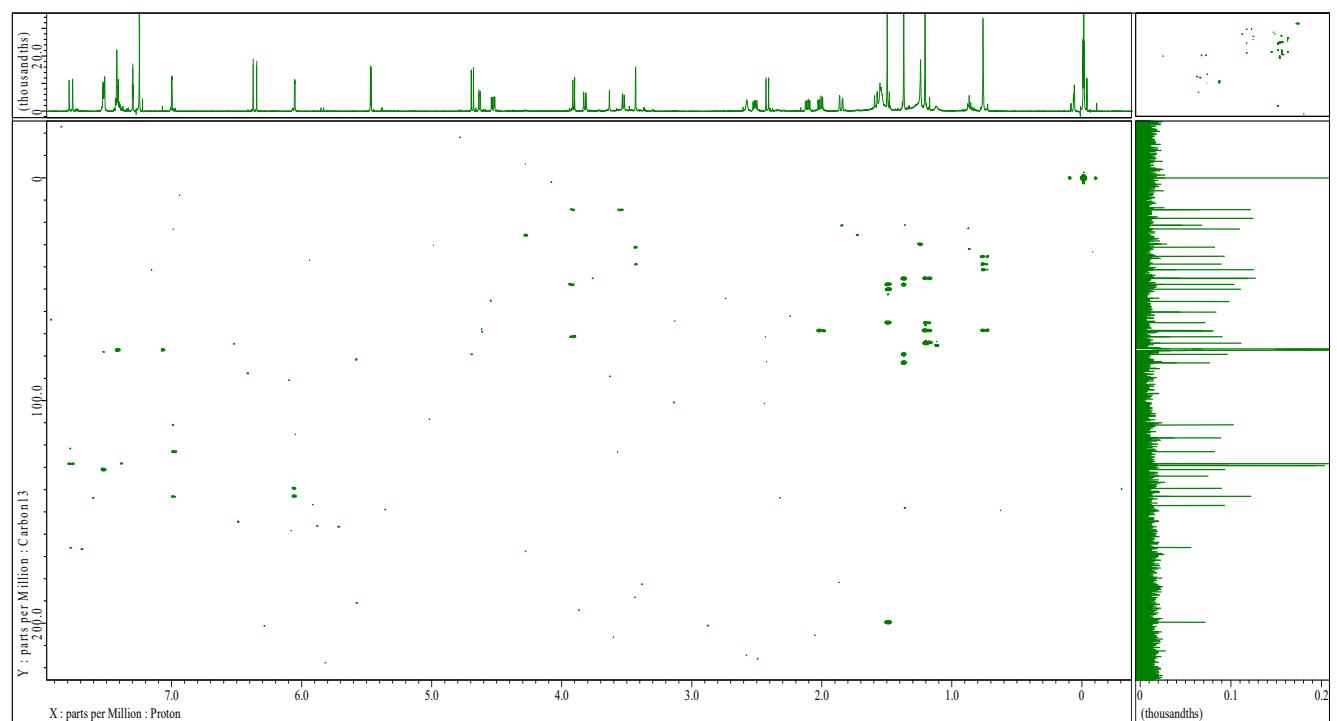


Figure S19. HMBC spectrum of compound **3** in CDCl_3 at 600MHz

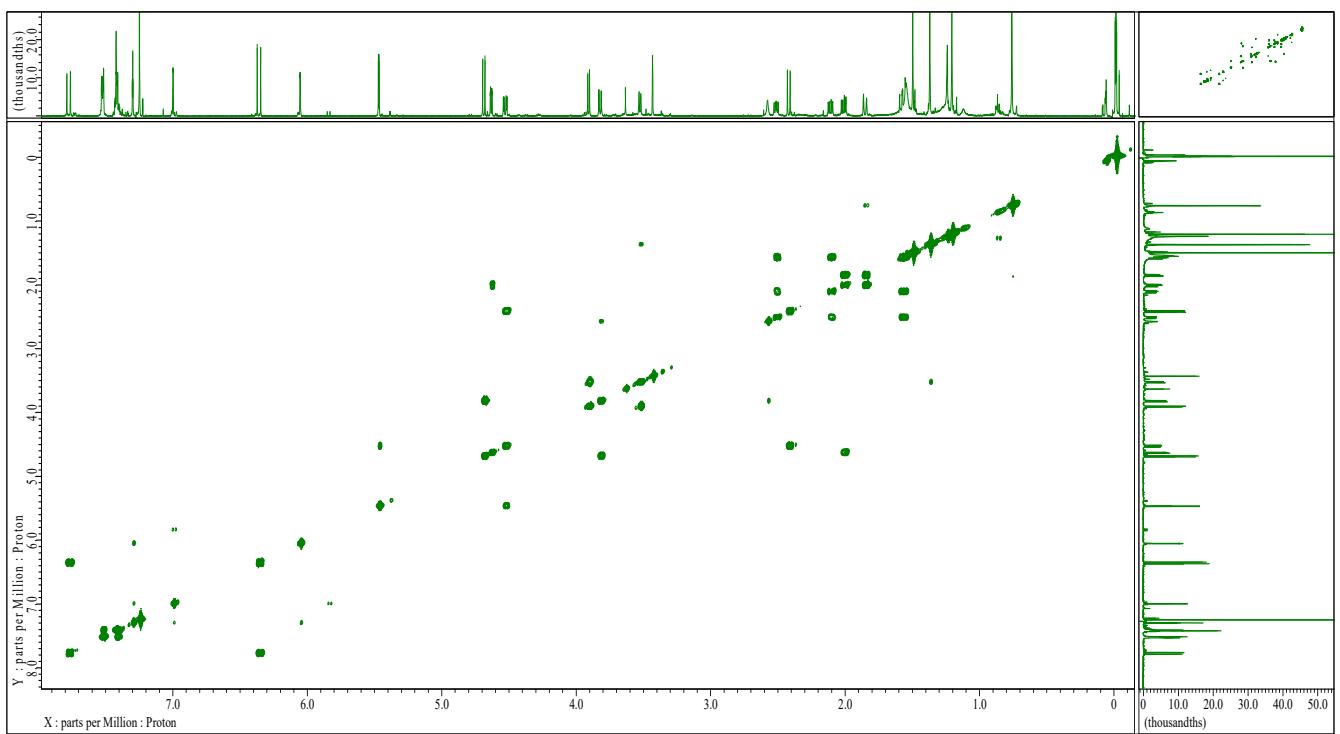


Figure S20. COSY spectrum of compound 3 in CDCl_3 at 600MHz

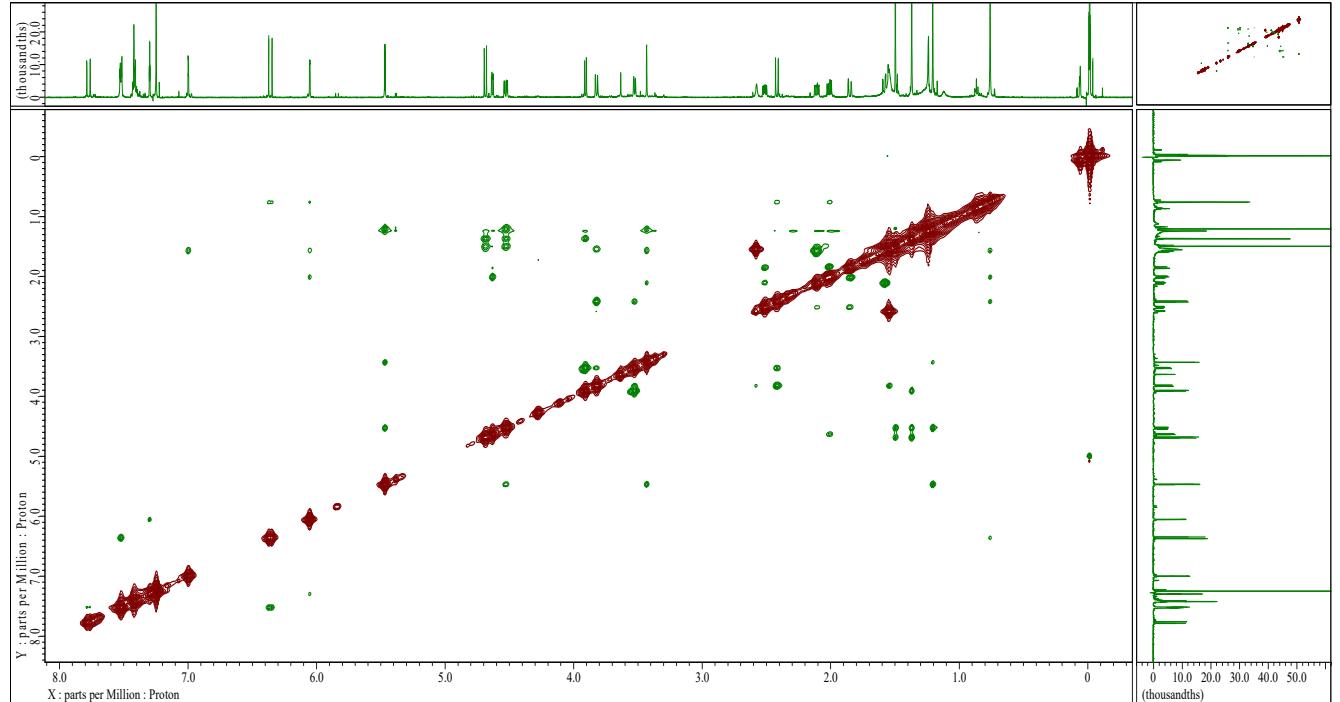


Figure S21. NOESY spectrum of compound 3 in CDCl_3 at 600MHz

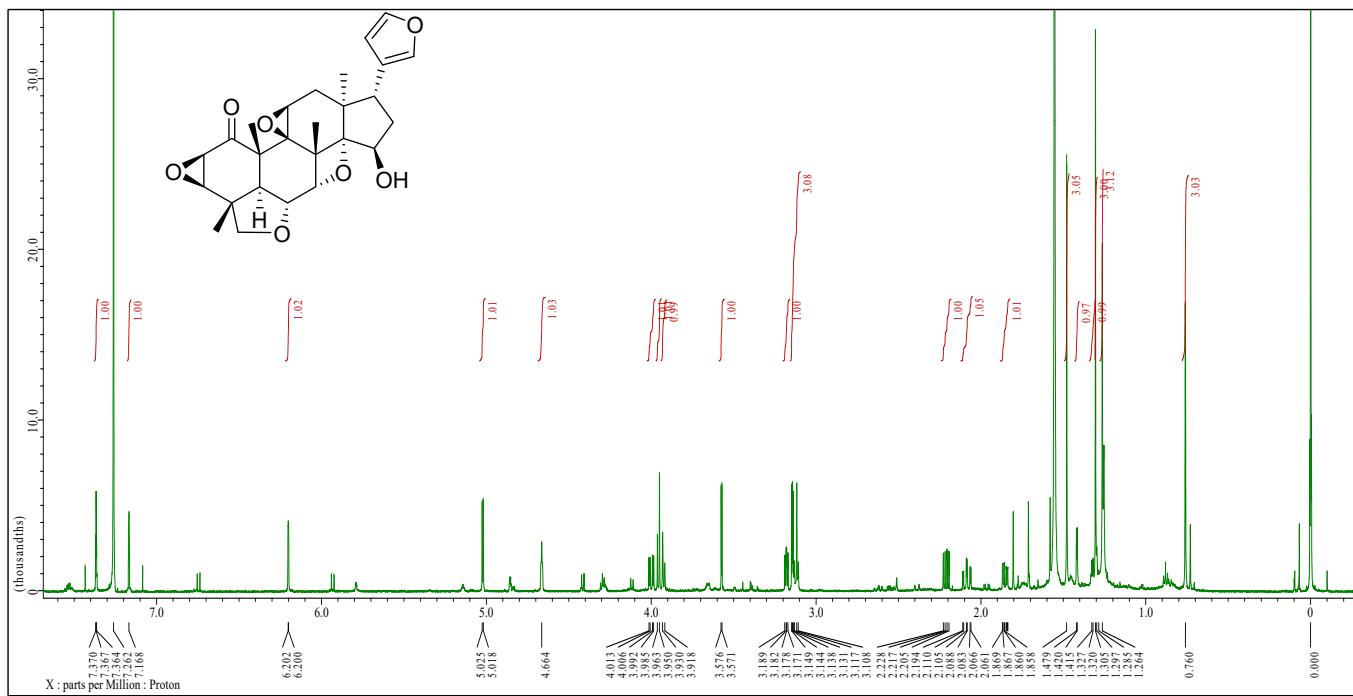


Figure S22. ^1H NMR spectrum of compound 4 in CDCl_3 at 600MHz

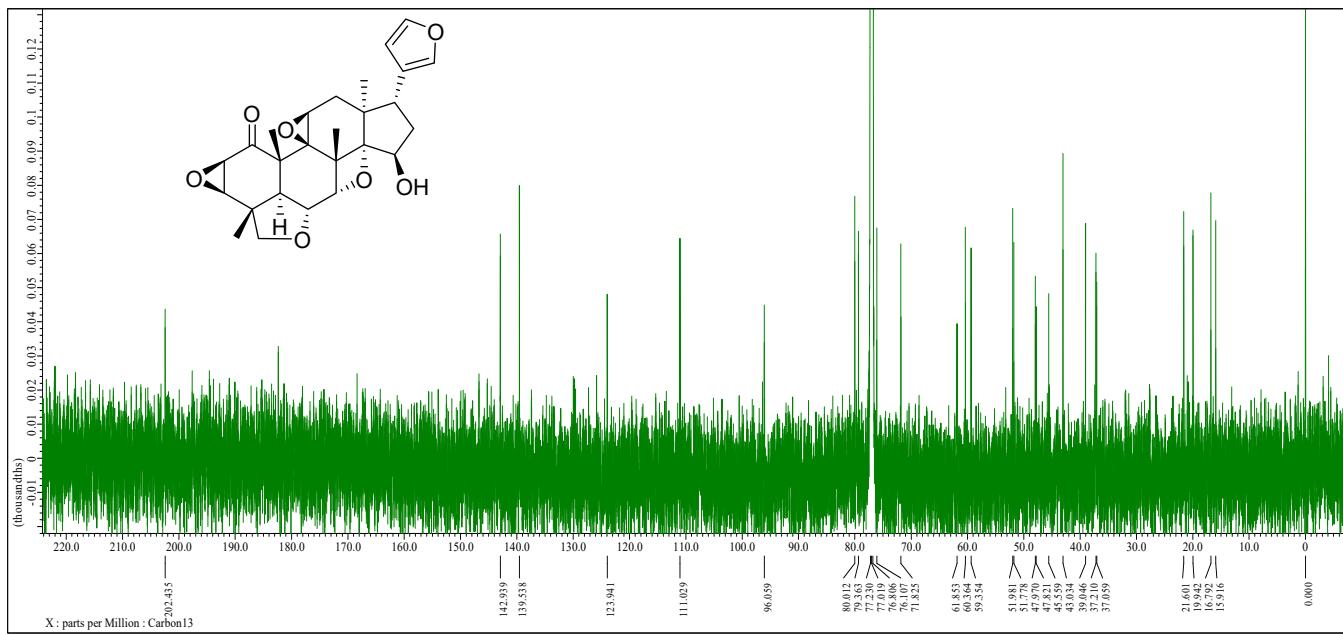


Figure S23. ^{13}C NMR spectrum of compound 4 in CDCl_3 at 150MHz

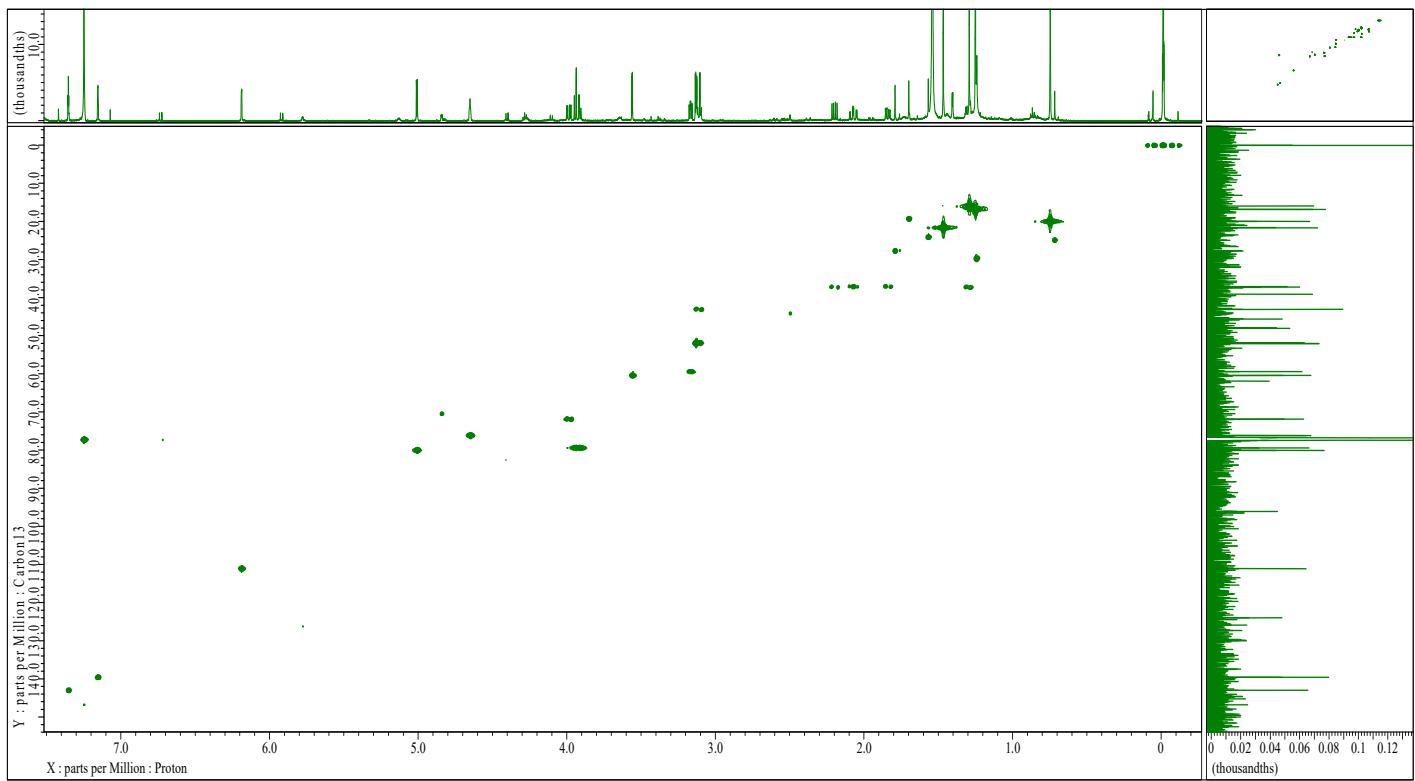


Figure S24. HMQC spectrum of compound 4 in CDCl_3 at 600MHz

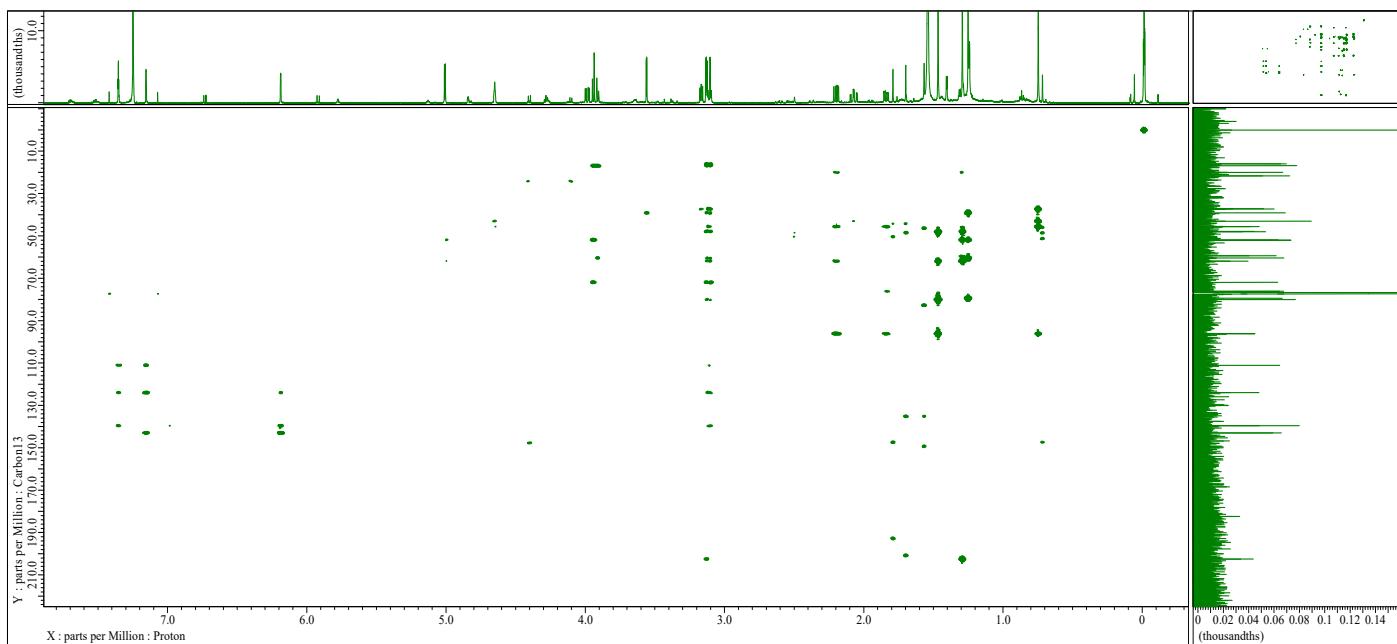


Figure S25. HMBC spectrum of compound 4 in CDCl_3 at 600MHz

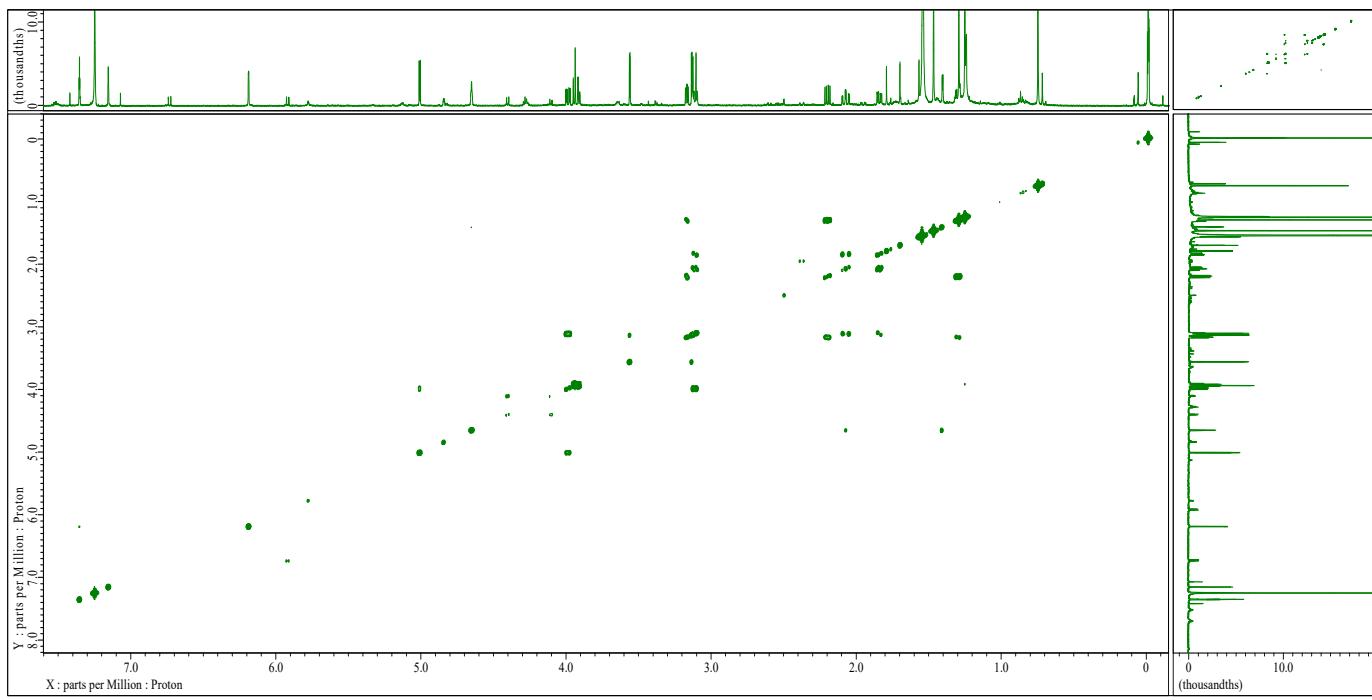


Figure S26. COSY spectrum of compound 4 in CDCl_3 at 600MHz

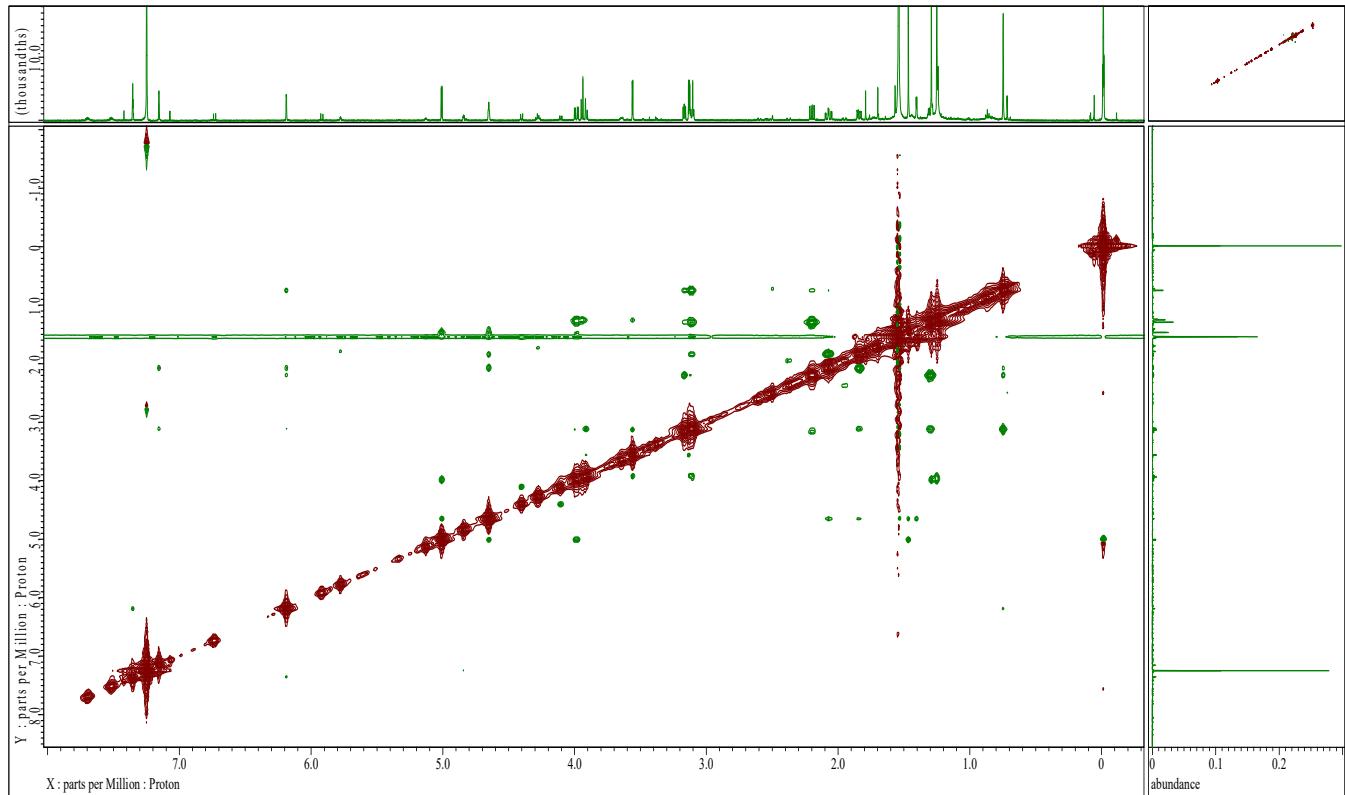


Figure S27. NOESY spectrum of compound 4 in CDCl_3 at 600MHz

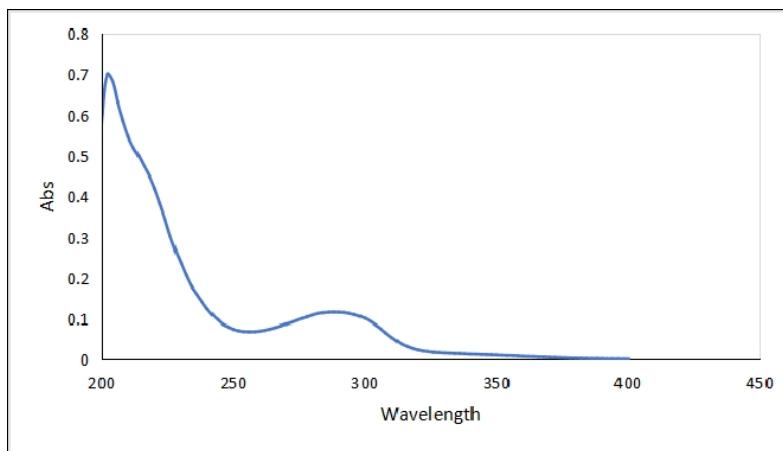


Figure S28. UV spectrum of compound **4** in MeOH

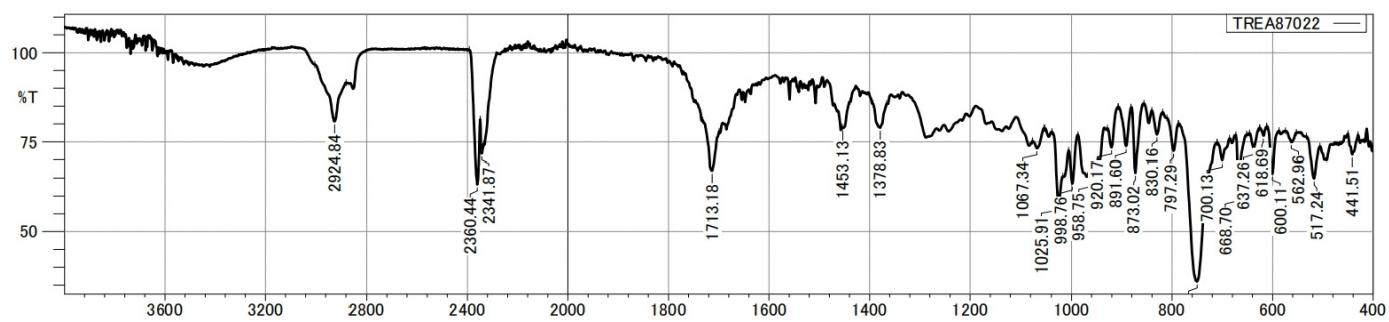


Figure S29. IR spectrum of compound **4**

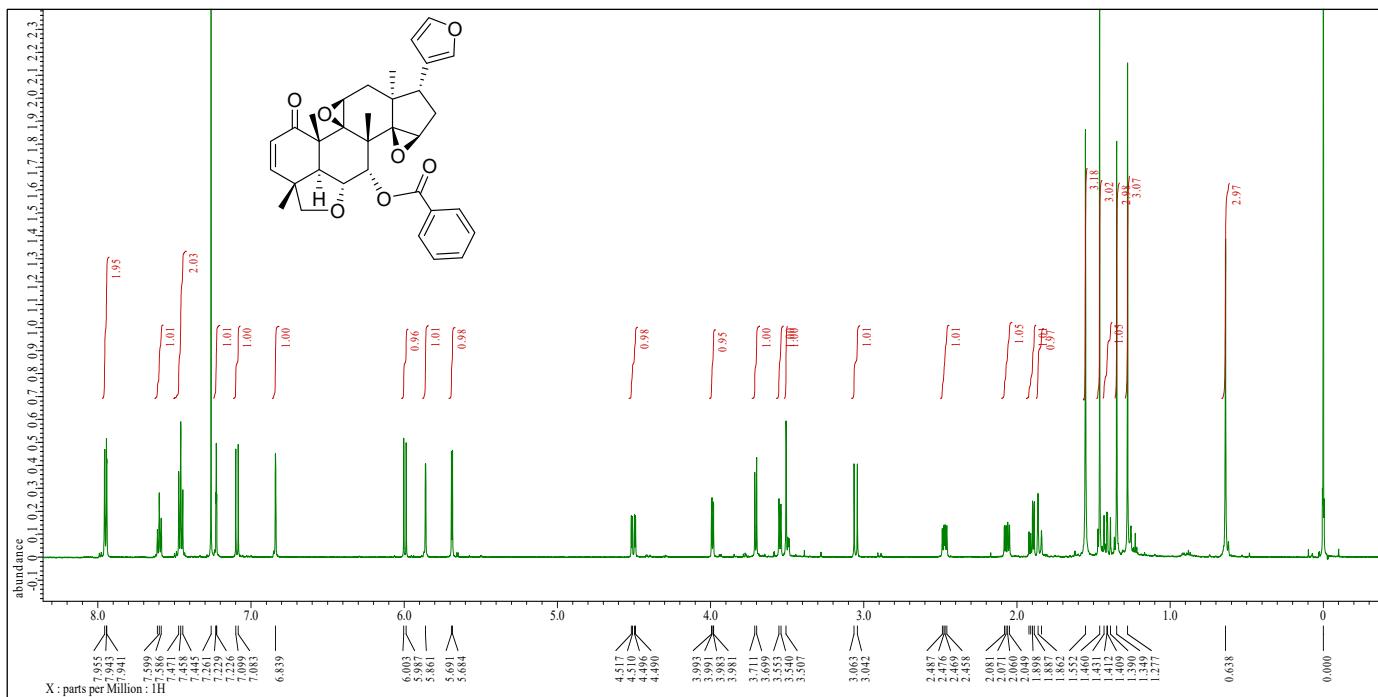


Figure S30. ^1H NMR spectrum of compound **5** in CDCl_3 at 600MHz

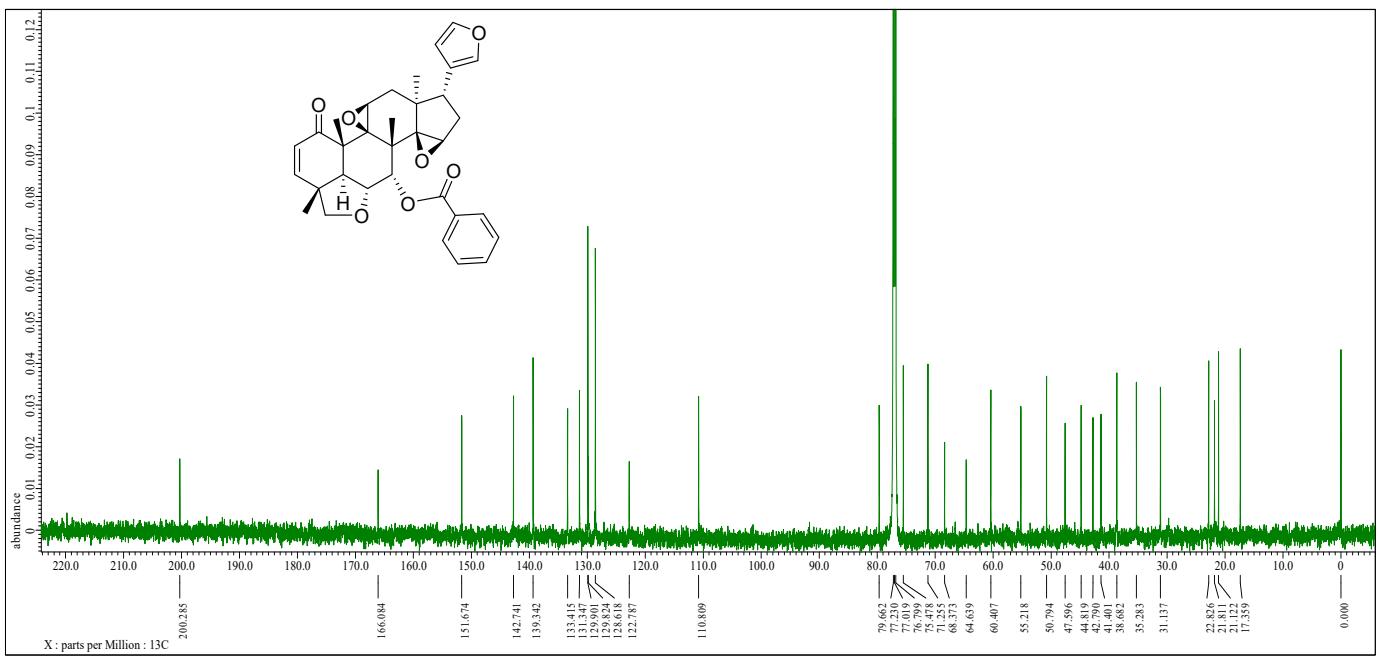


Figure S31. ^{13}C NMR spectrum of compound **5** in CDCl_3 at 150MHz

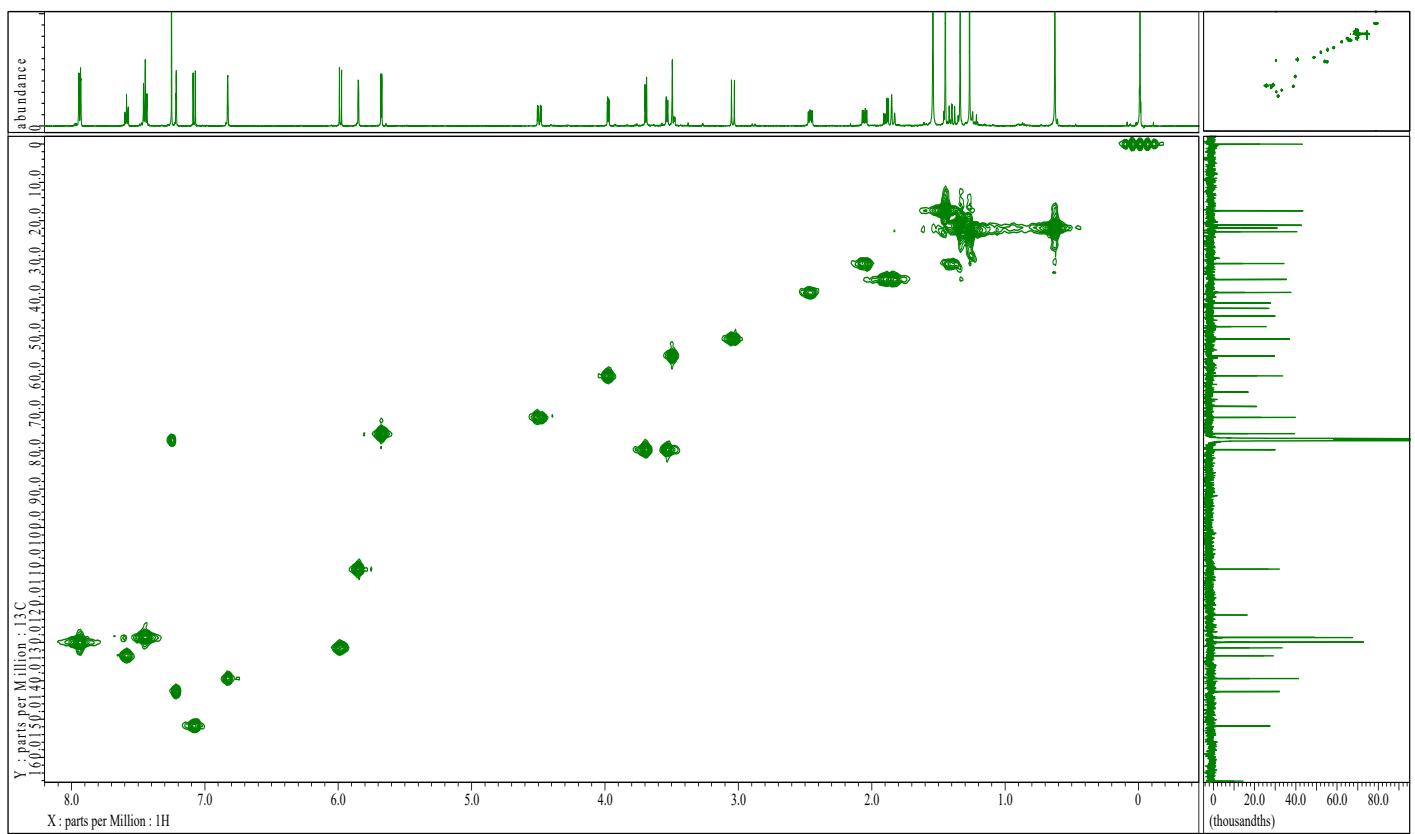


Figure S32. HMQC spectrum of compound **5** in CDCl_3 at 600MHz

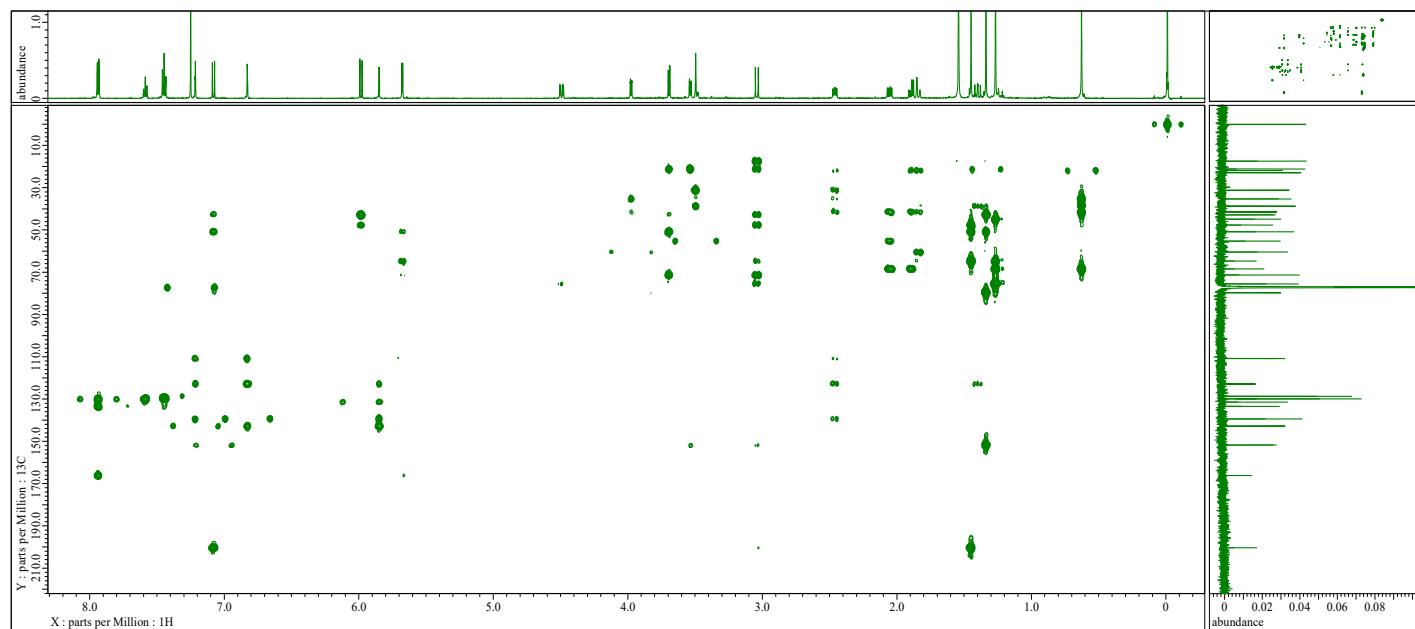


Figure S33. HMBC spectrum of compound **5** in CDCl_3 at 600MHz

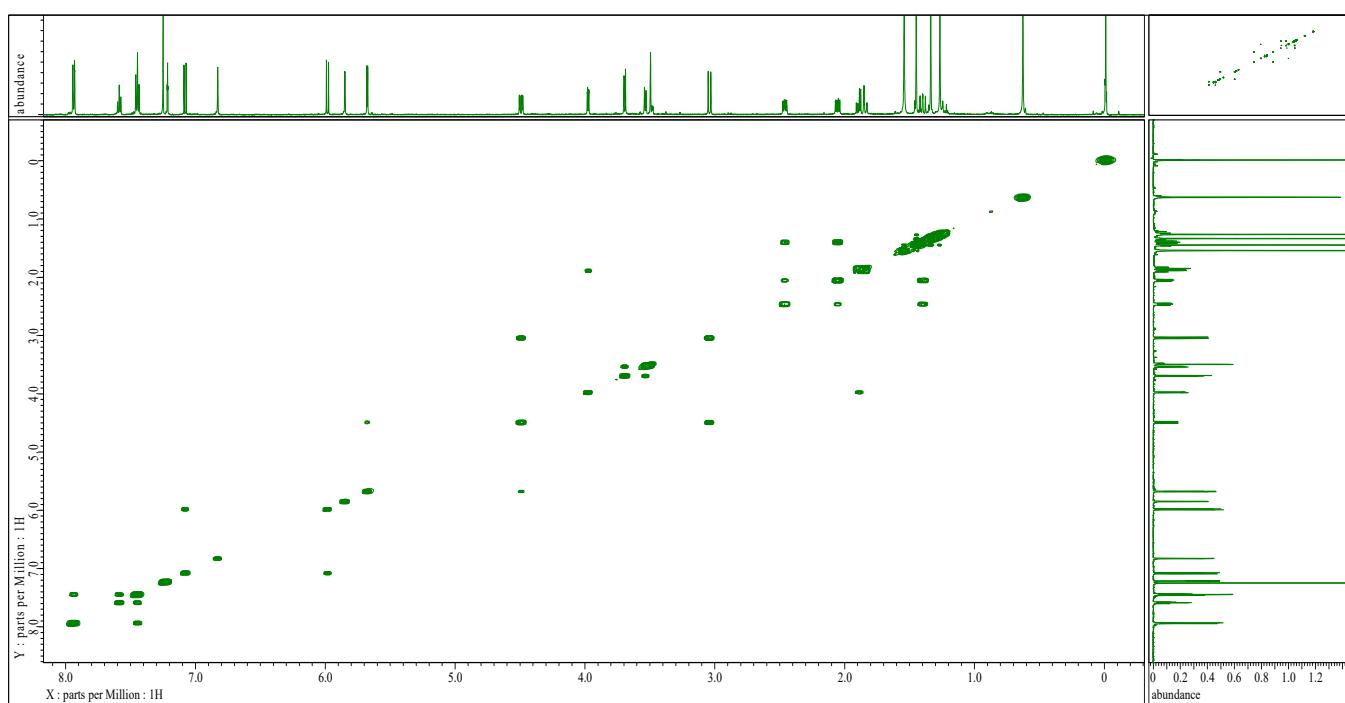


Figure S34. COSY spectrum of compound **5** in CDCl_3 at 600MHz

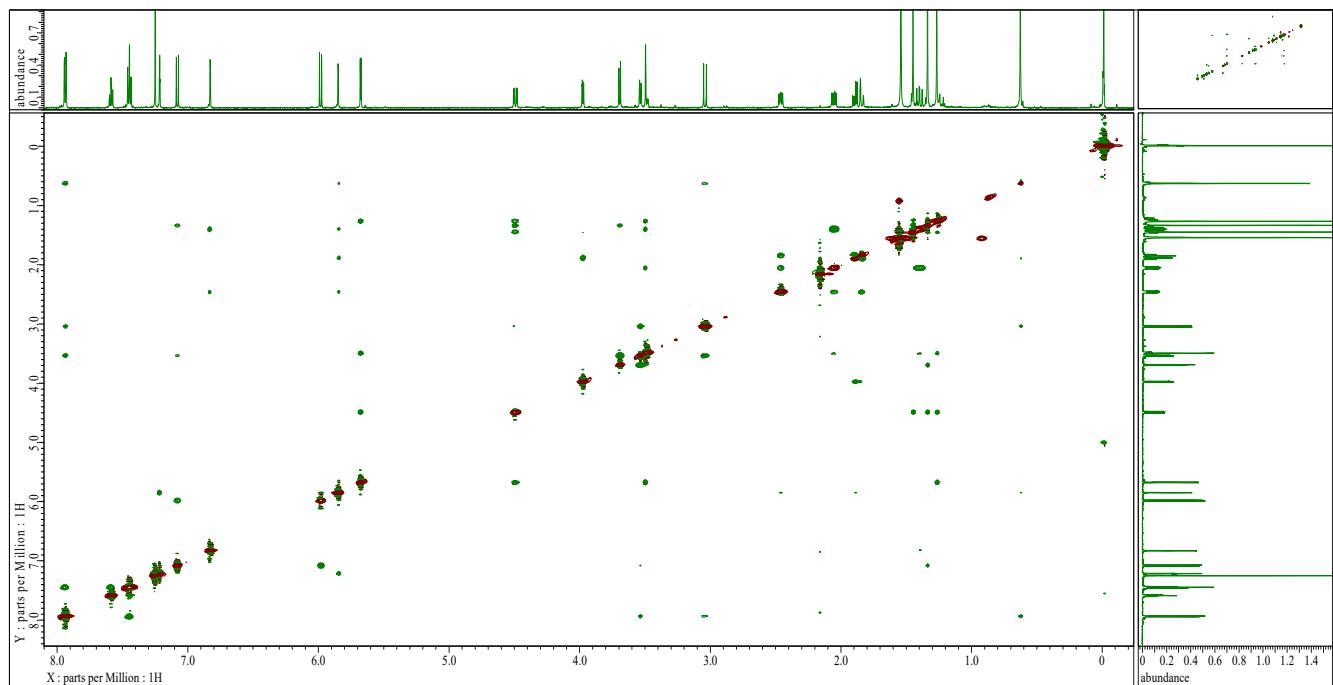


Figure S35. NOESY spectrum of compound **5** in CDCl_3 at 600MHz

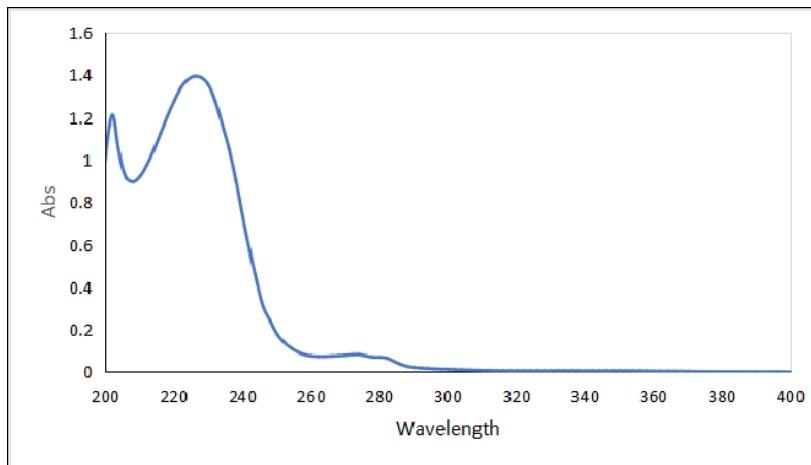


Figure S36. UV spectrum of compound **5** in MeOH

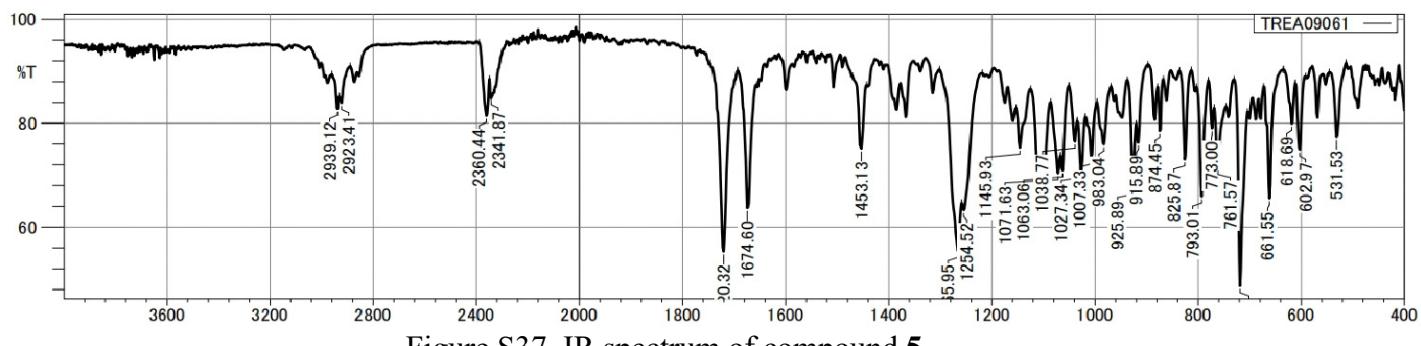


Figure S37. IR spectrum of compound **5**

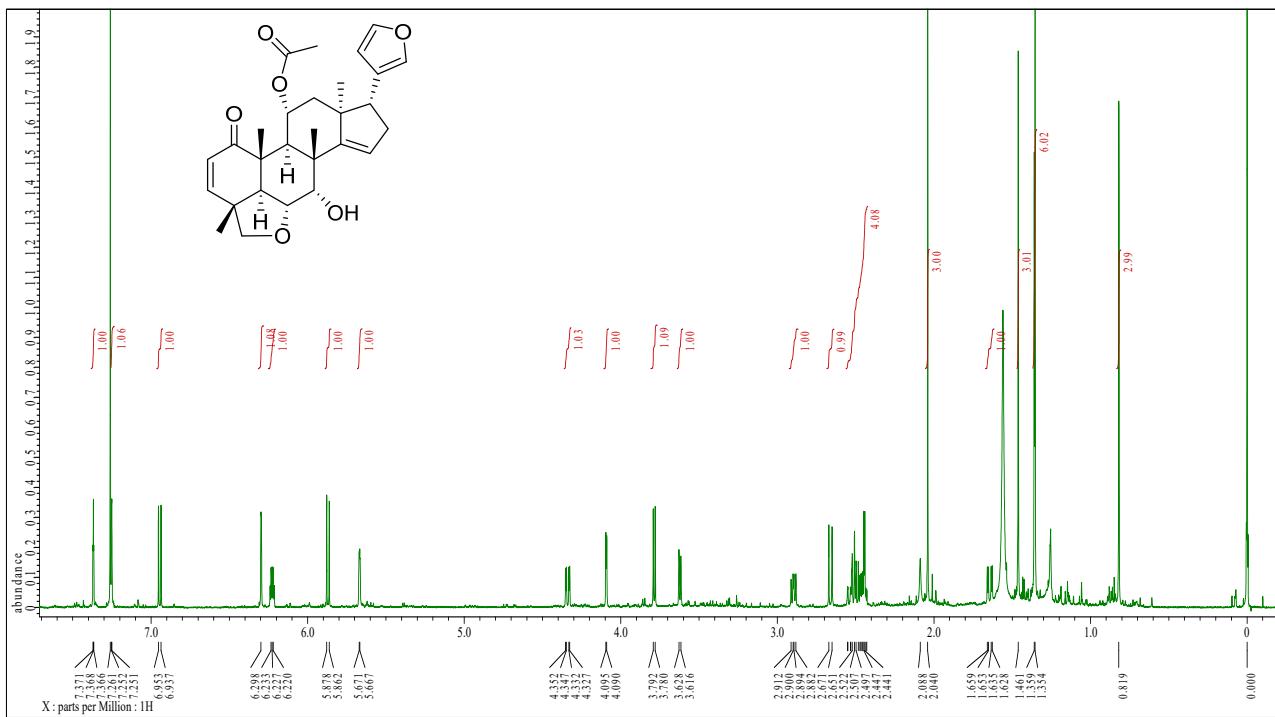


Figure S38. ^1H NMR spectrum of compound **6** in CDCl_3 at 600MHz

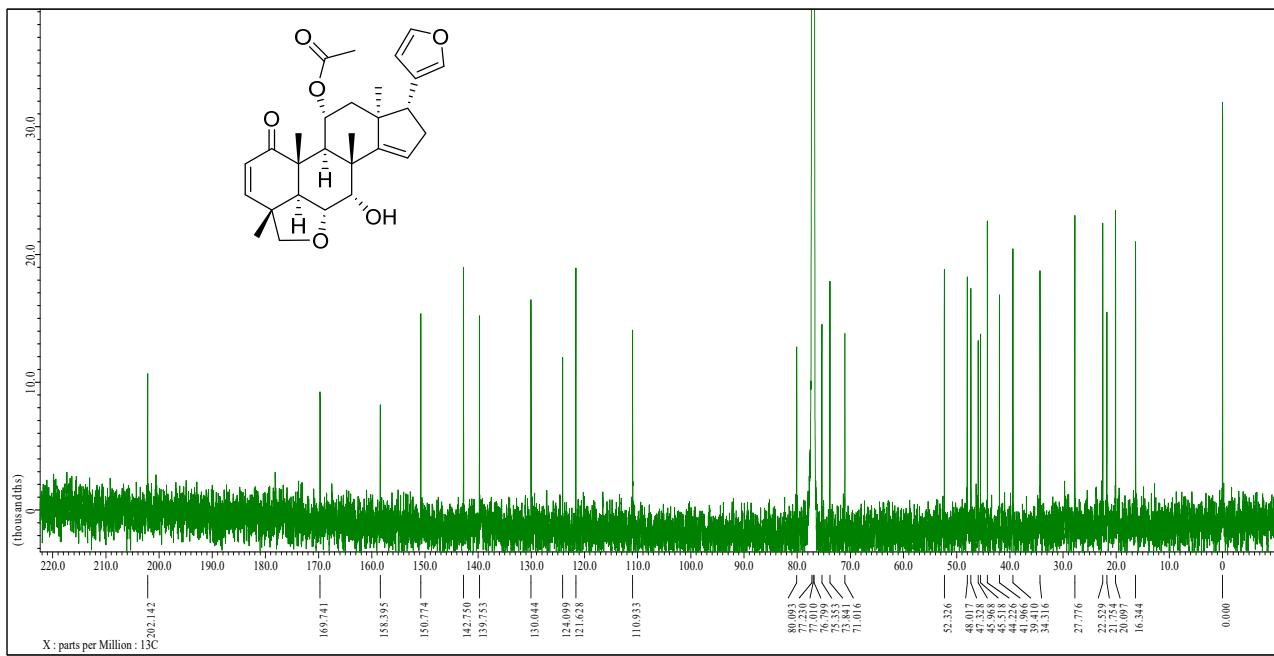


Figure S39. ^{13}C NMR spectrum of compound **6** in CDCl_3 at 150MHz

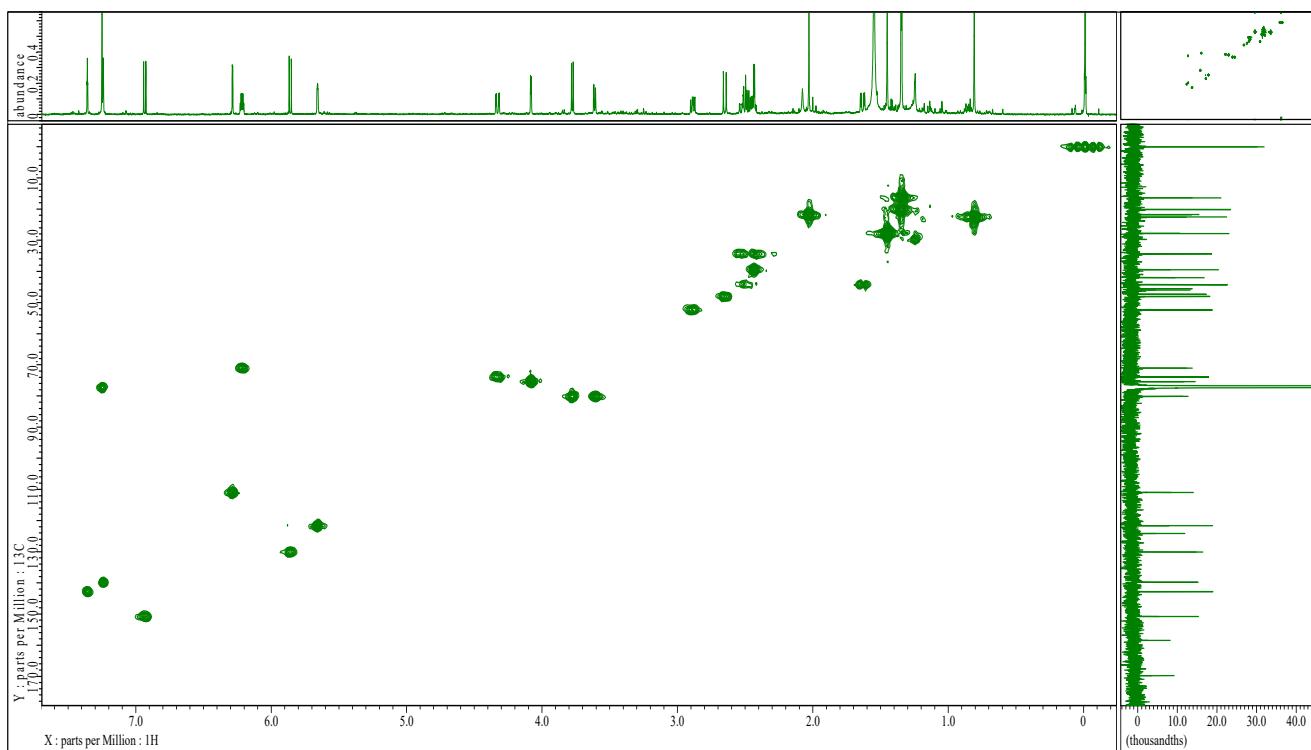


Figure S40. HMQC spectrum of compound **6** in CDCl_3 at 600MHz

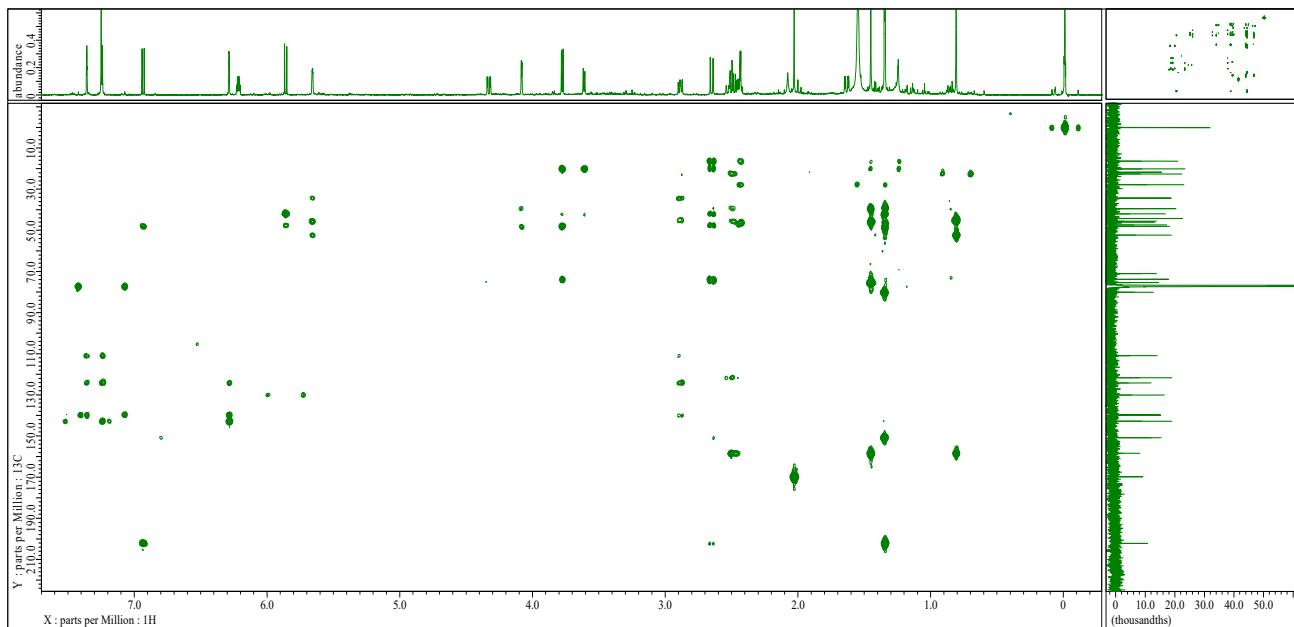


Figure S41. HMBC spectrum of compound **6** in CDCl_3 at 600MHz

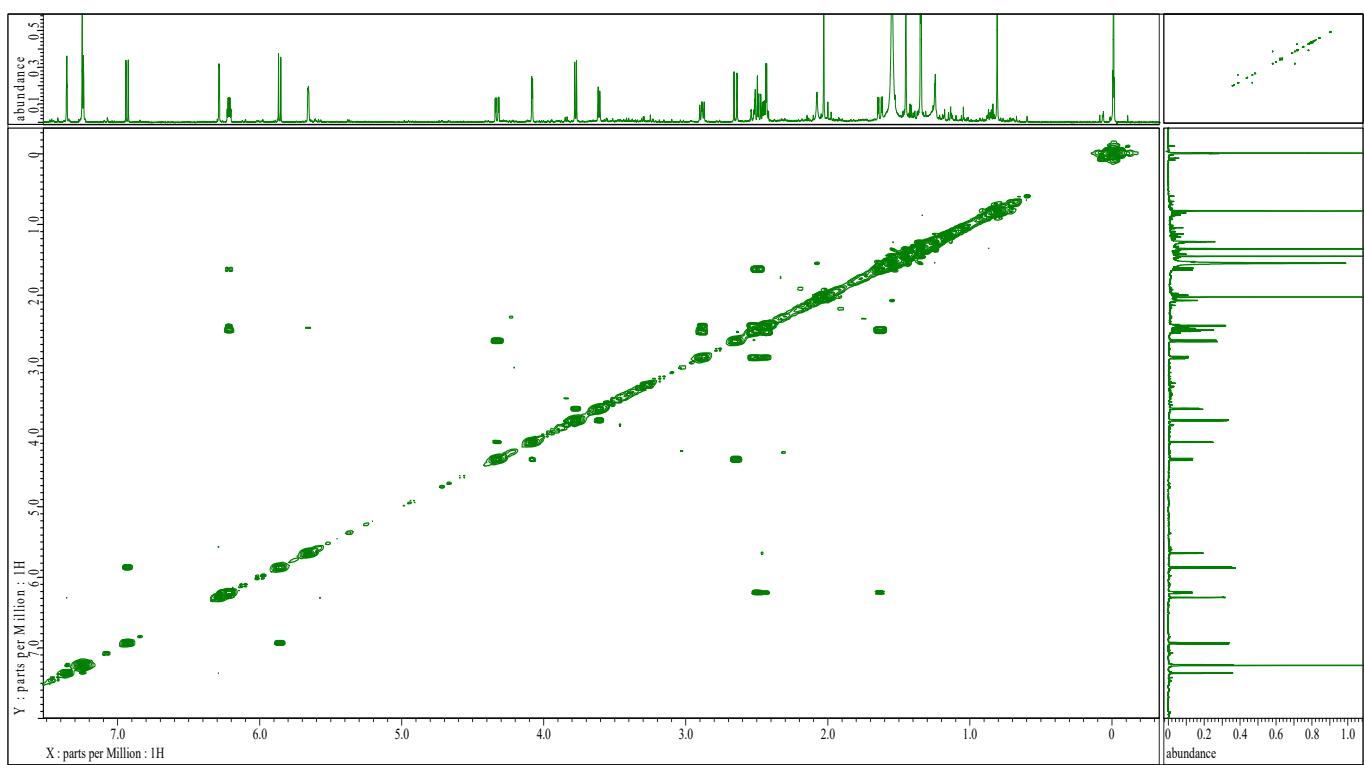


Figure S42. COSY spectrum of compound **6** in CDCl_3 at 600MHz

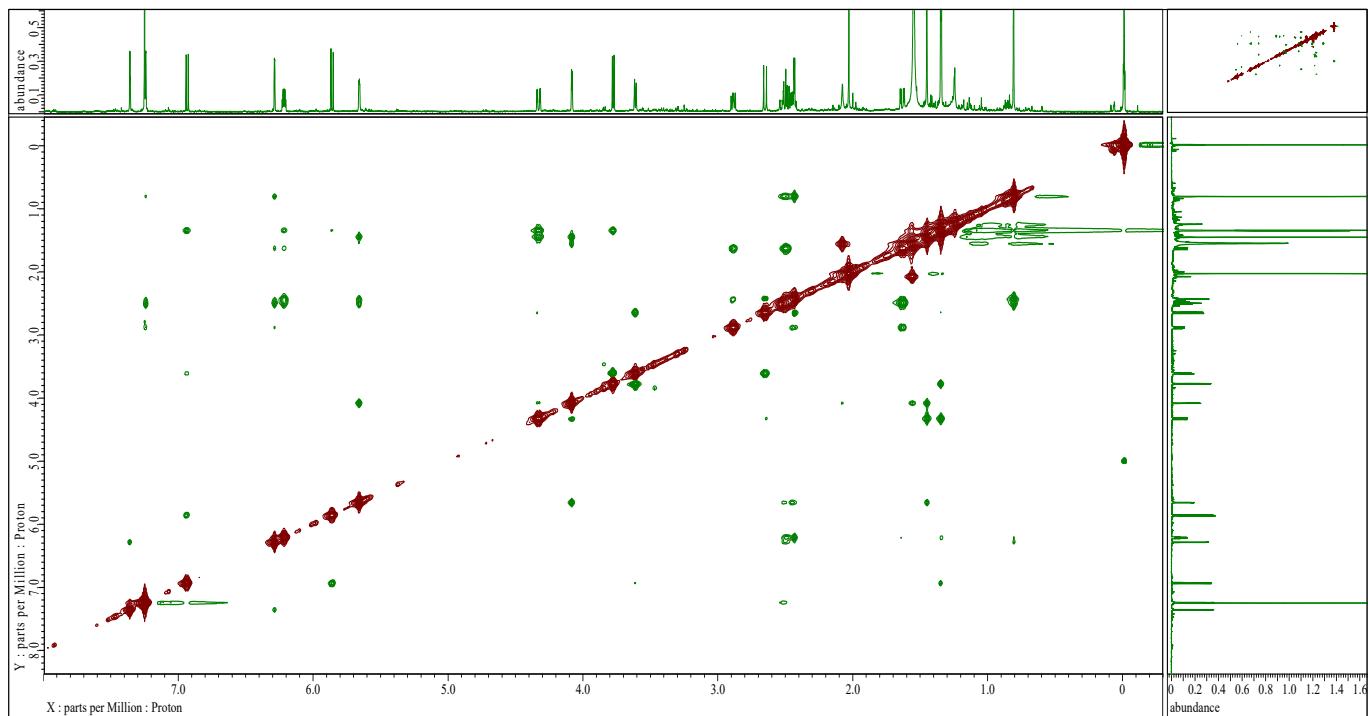


Figure S43. NOESY spectrum of compound **6** in CDCl_3 at 600MHz

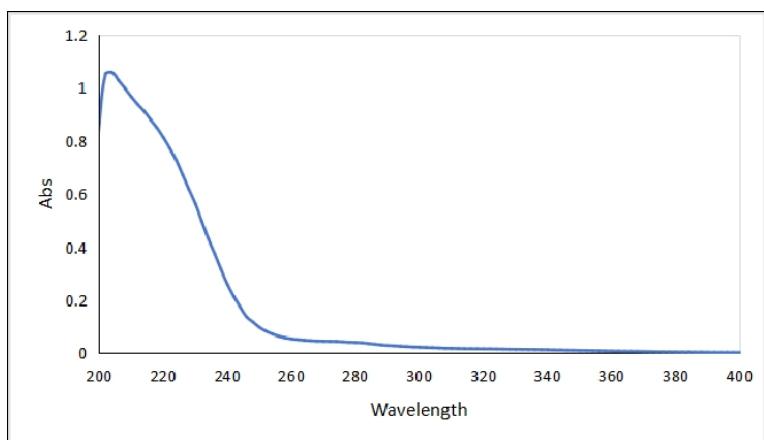


Figure S44. UV spectrum of compound **6** in MeOH

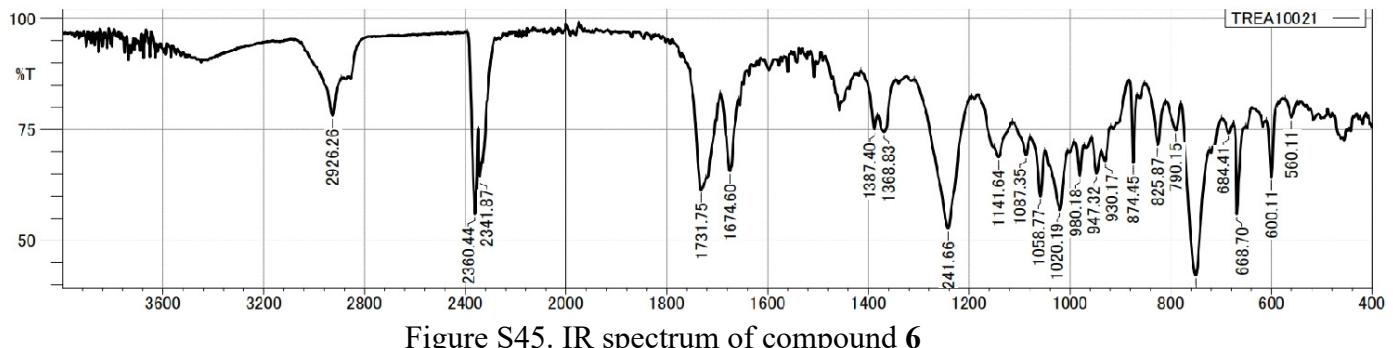


Figure S45. IR spectrum of compound **6**

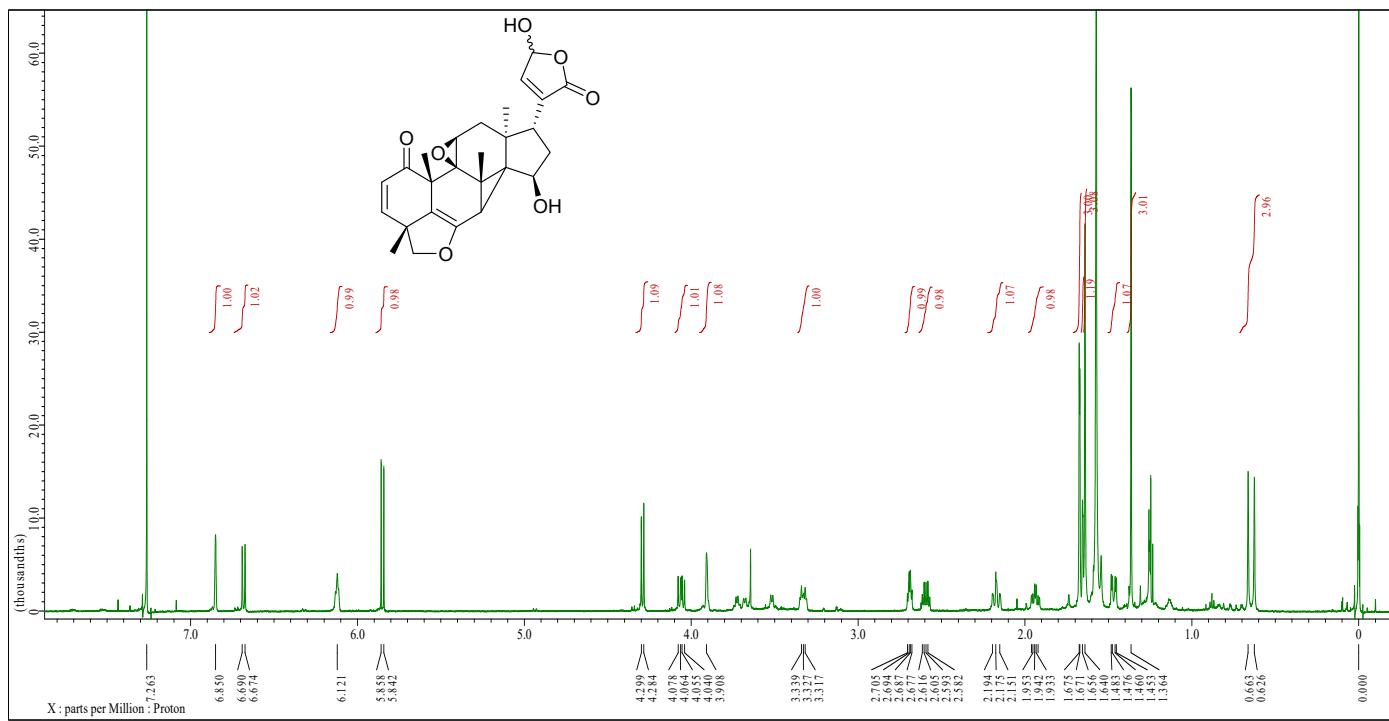


Figure S46. ^1H NMR spectrum of compound 7 in CDCl_3 at 600MHz

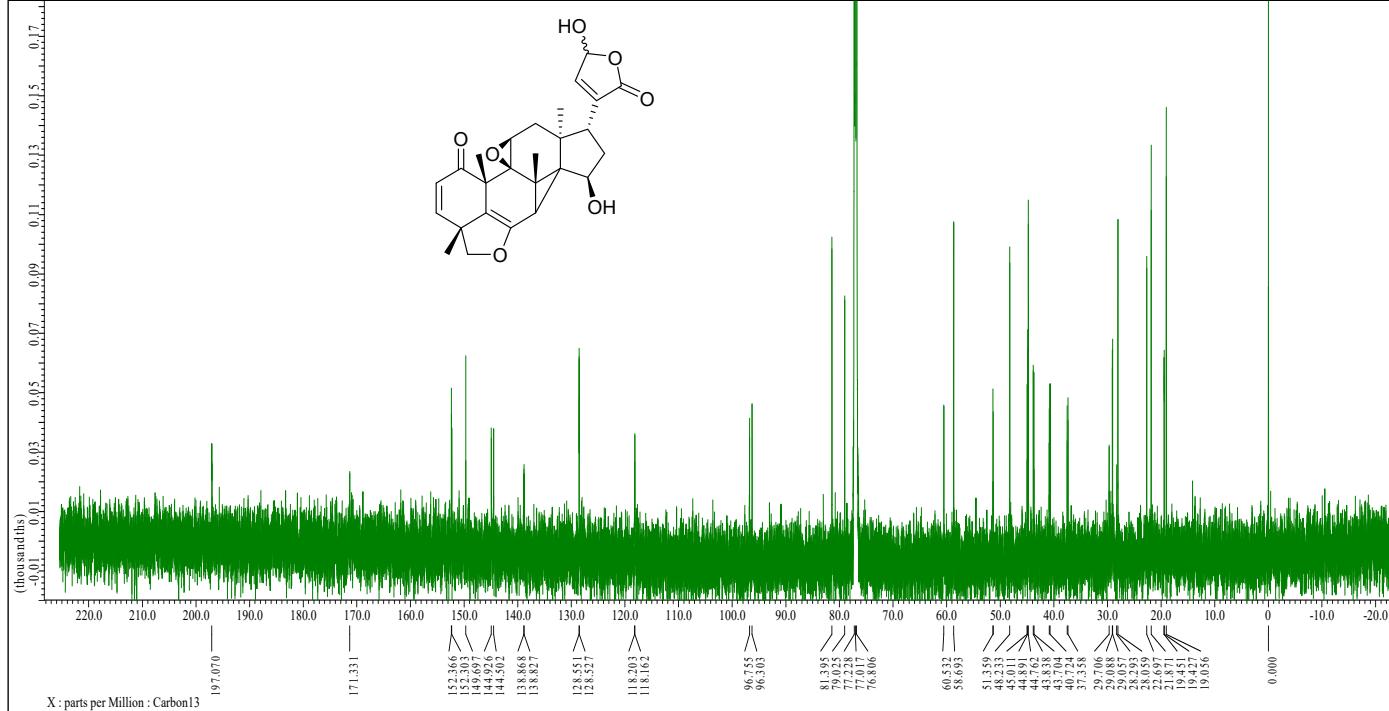


Figure S47. ^{13}C NMR spectrum of compound 7 in CDCl_3 at 150MHz

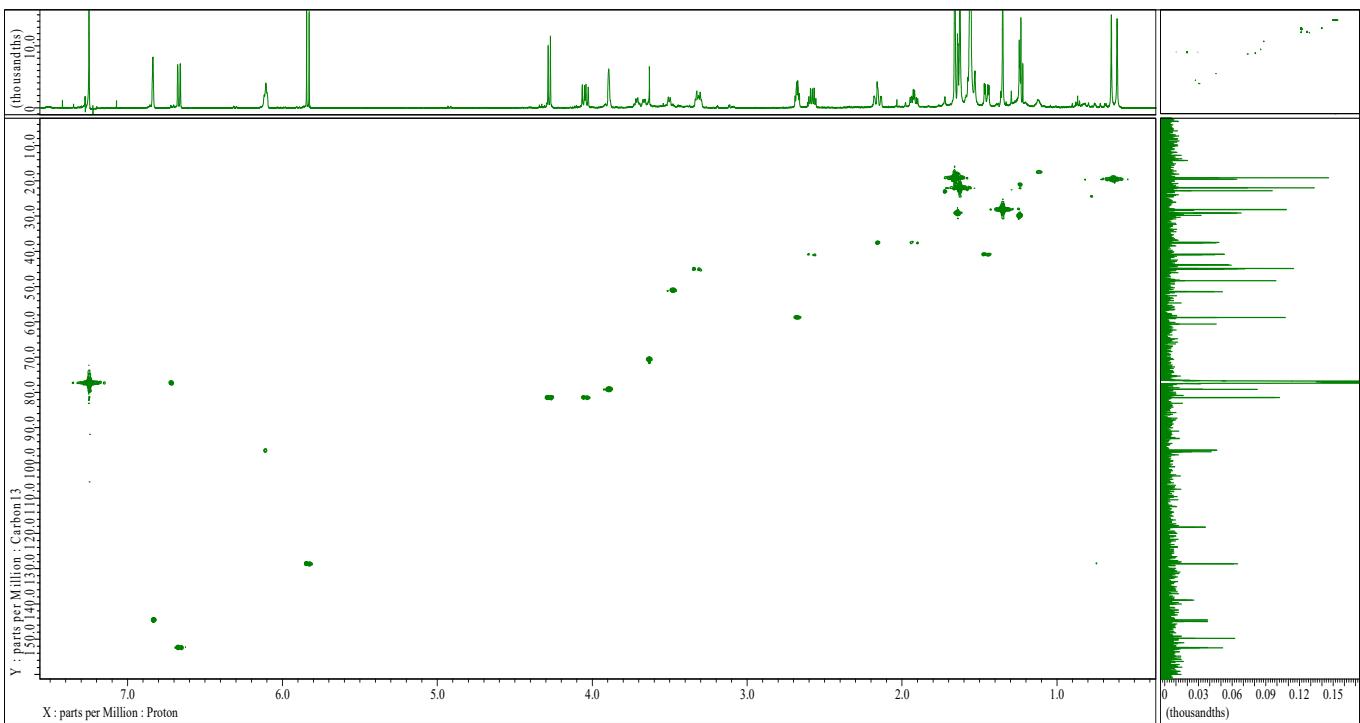


Figure S48. HMQC spectrum of compound 7 in CDCl_3 at 600MHz

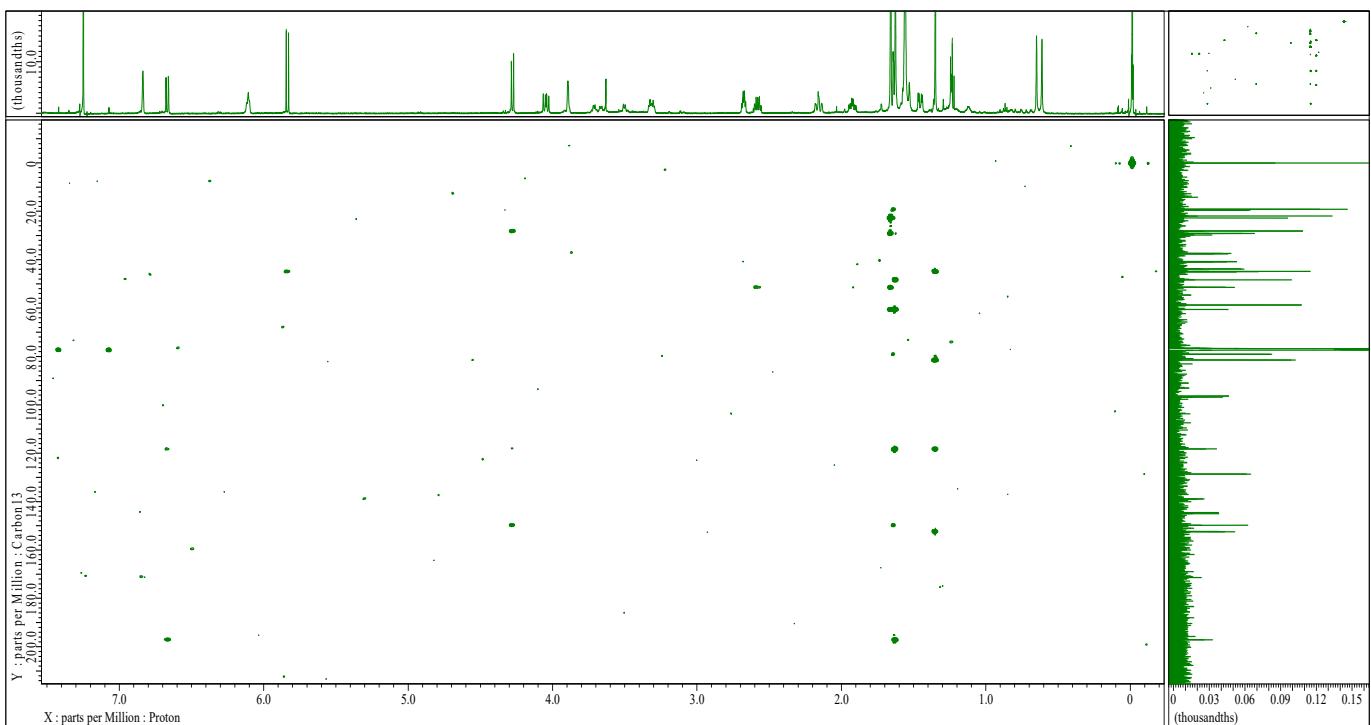


Figure S49. HMBC spectrum of compound 7 in CDCl_3 at 600MHz

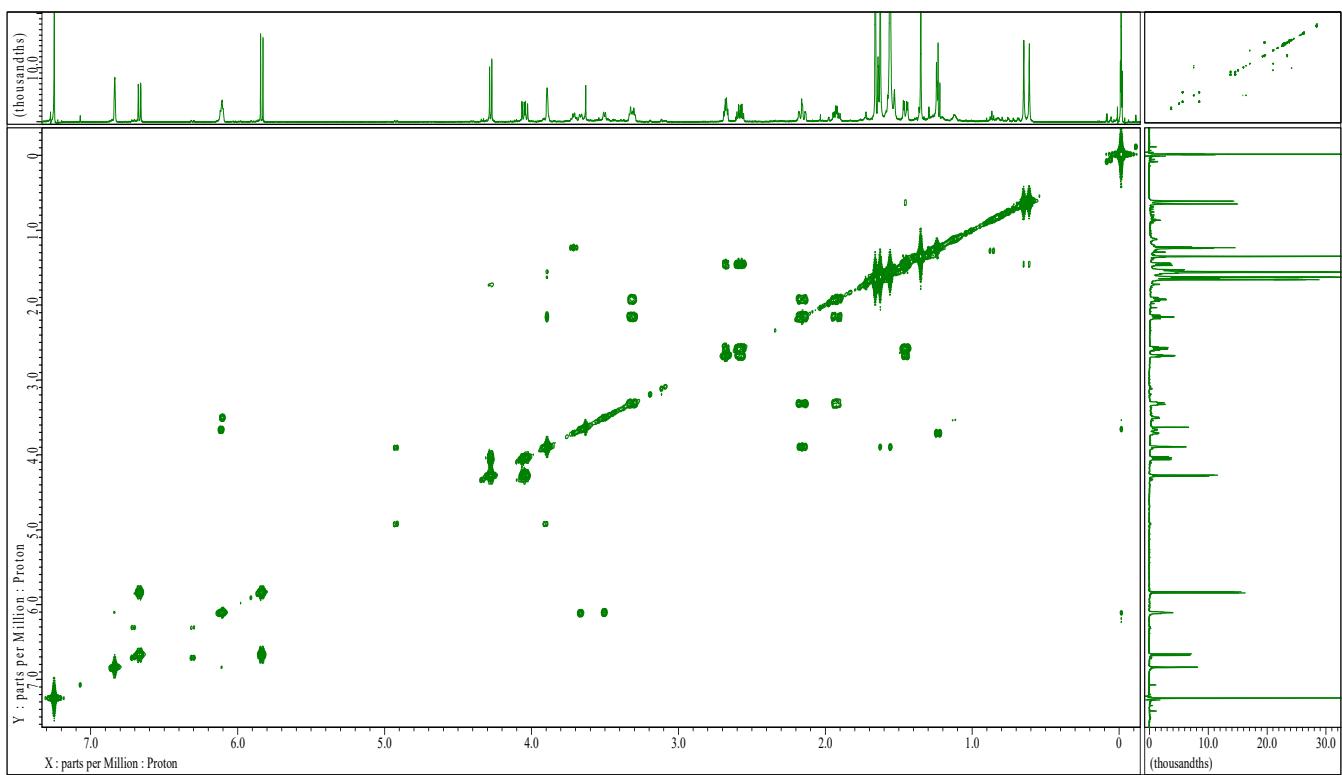


Figure S50. COSY spectrum of compound 7 in CDCl_3 at 600MHz

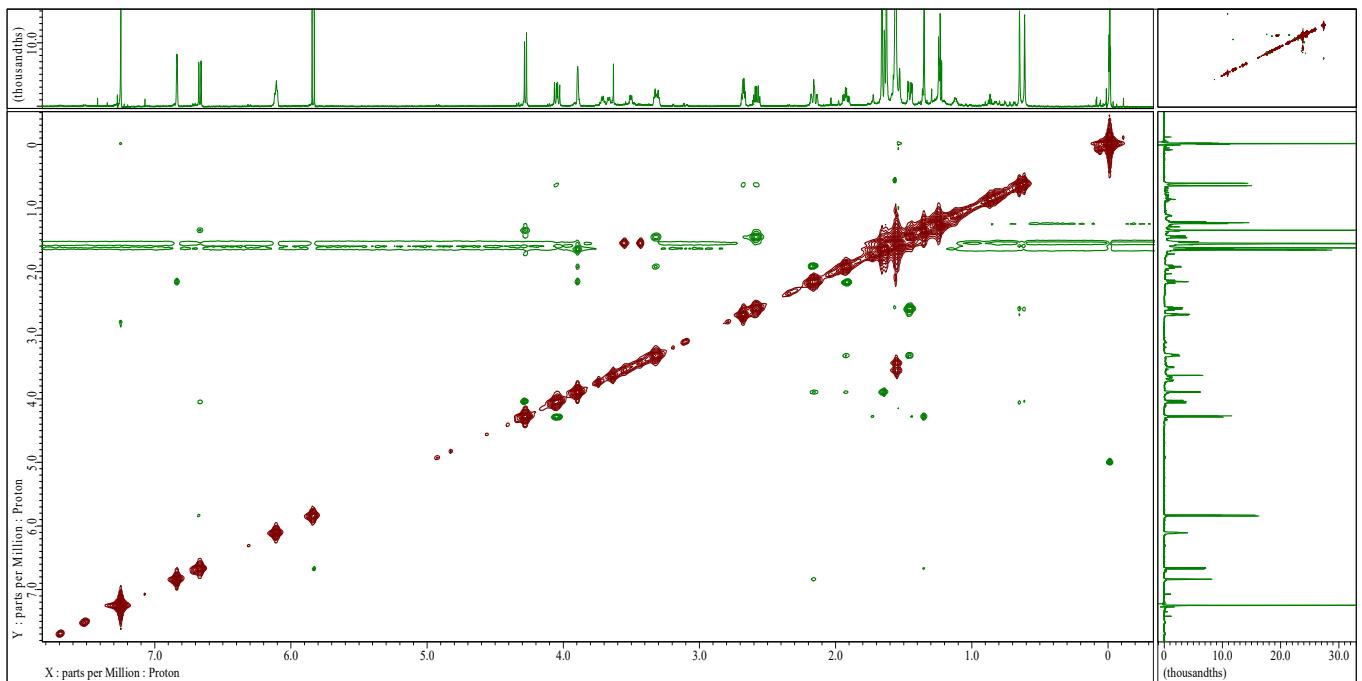


Figure S51. NOESY spectrum of compound 7 in CDCl_3 at 600MHz

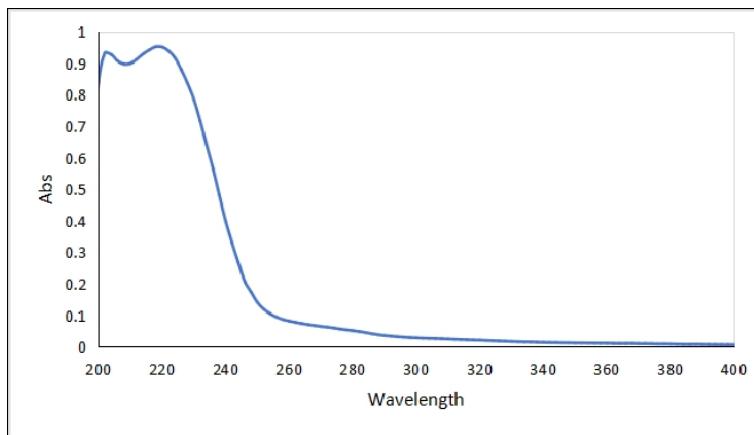


Figure S52. UV spectrum of compound 7 in MeOH

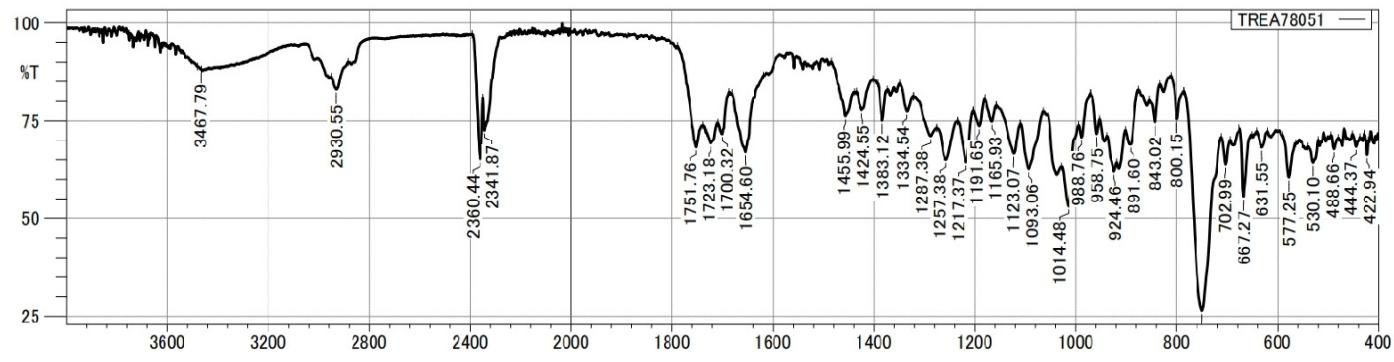


Figure S53. IR spectrum of compound 7

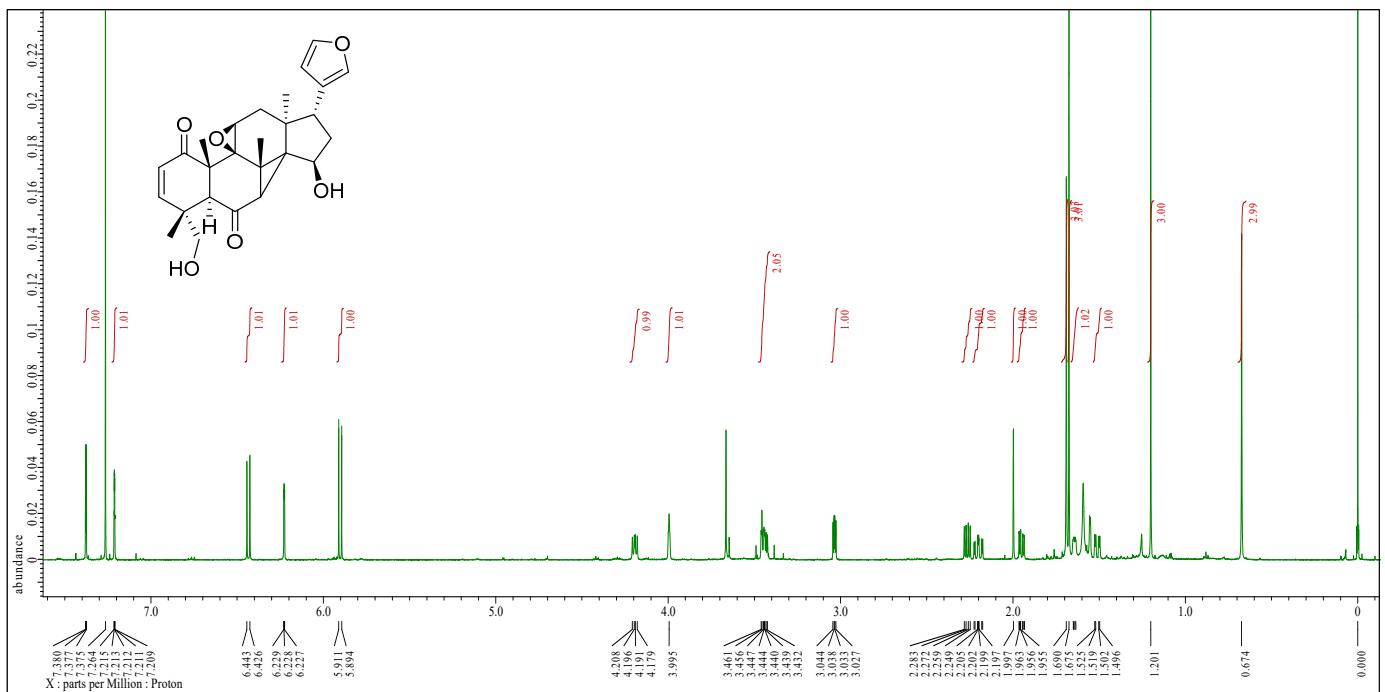


Figure S54. ^1H NMR spectrum of compound 8 in CDCl_3 at 600MHz

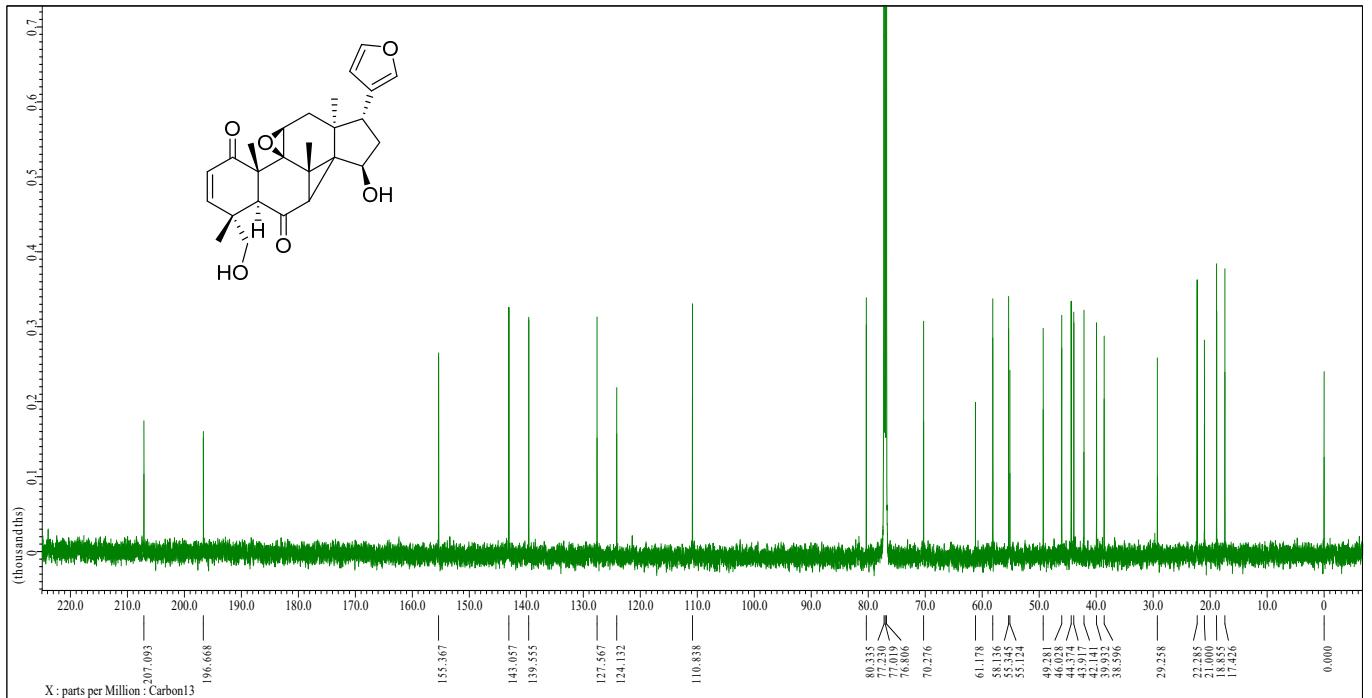


Figure S55. ^{13}C NMR spectrum of compound 8 in CDCl_3 at 150MHz

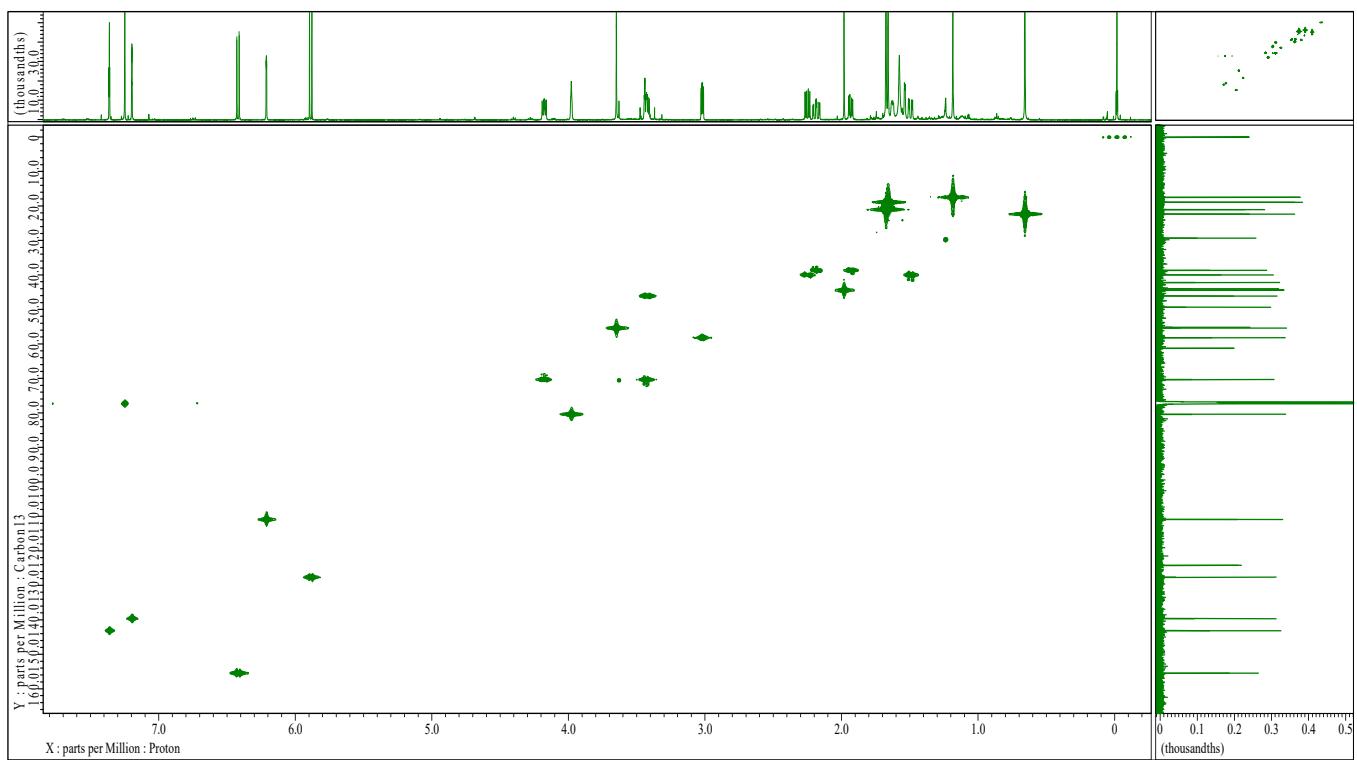


Figure S56. HMQC spectrum of compound **8** in CDCl_3 at 600MHz

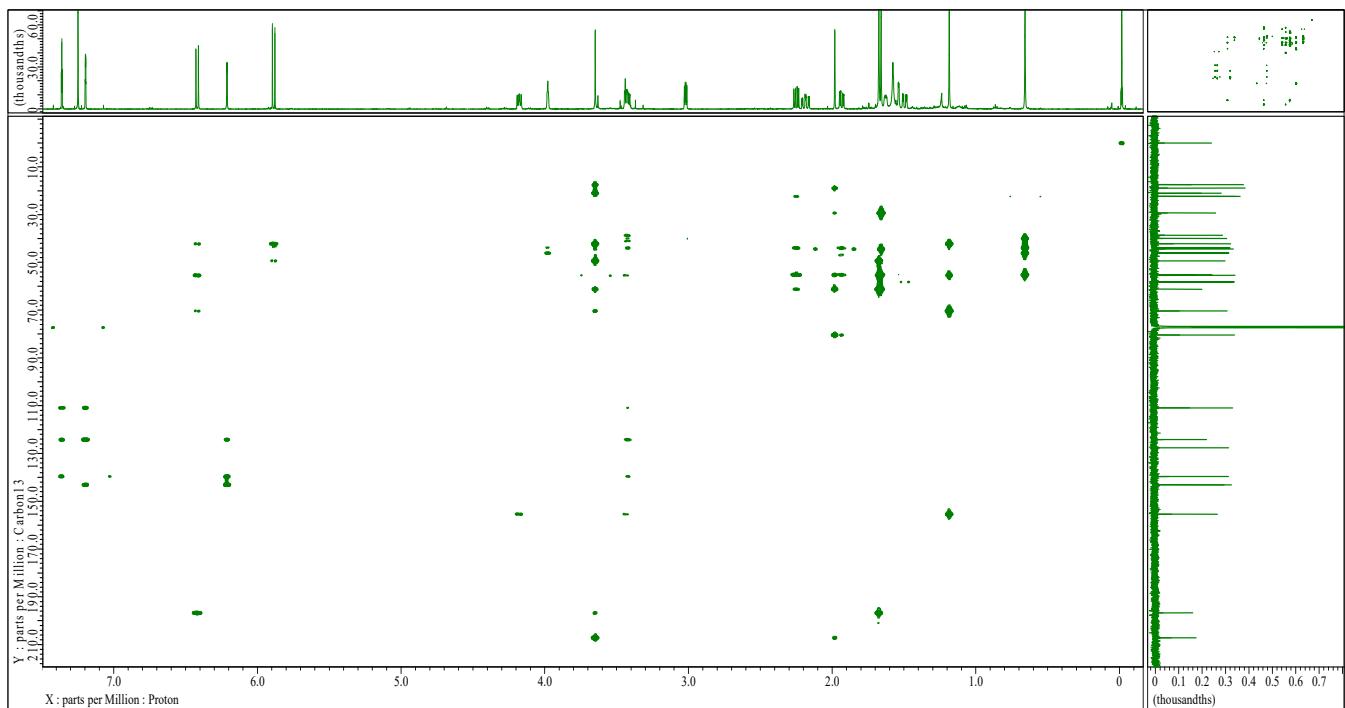


Figure S57. HMBC spectrum of compound **8** in CDCl_3 at 600MHz

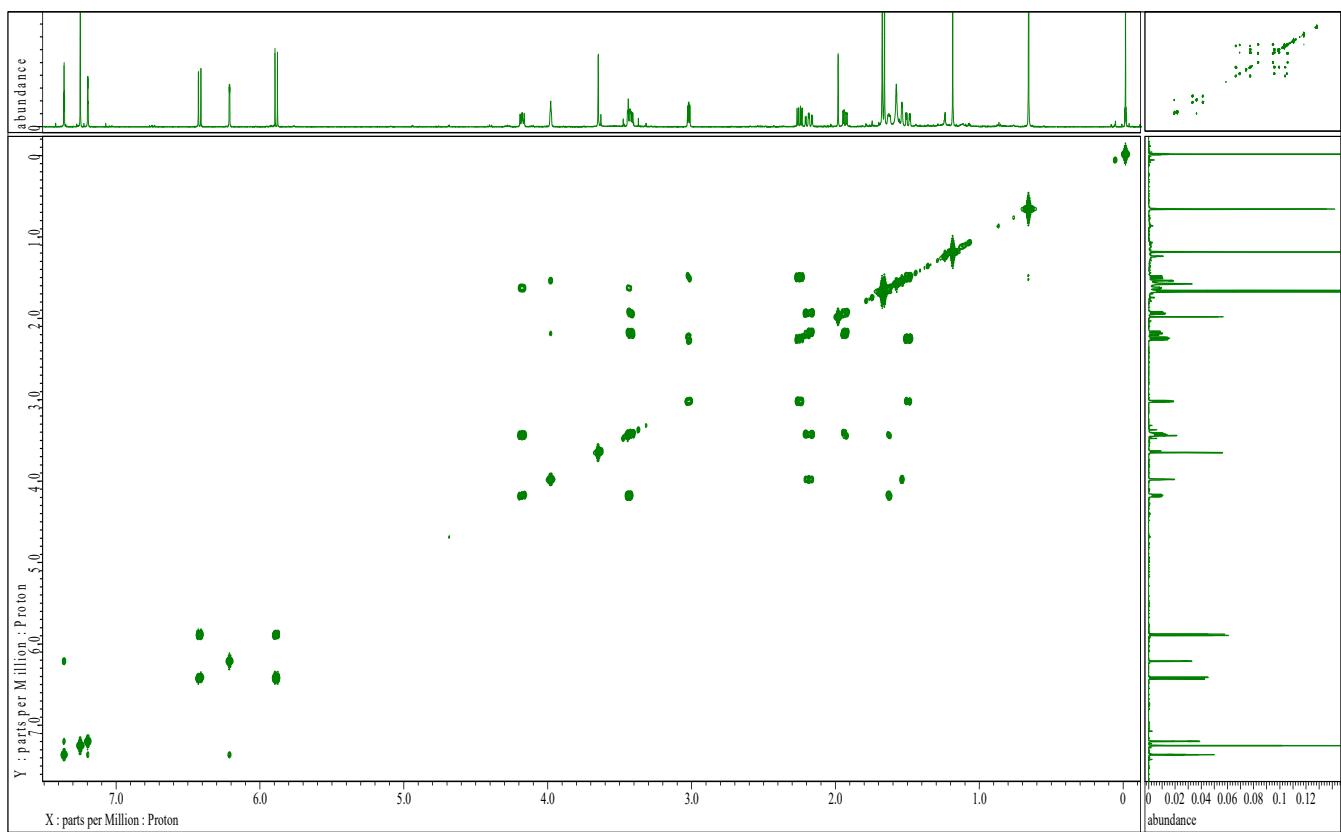


Figure S58. COSY spectrum of compound **8** in CDCl_3 at 600MHz

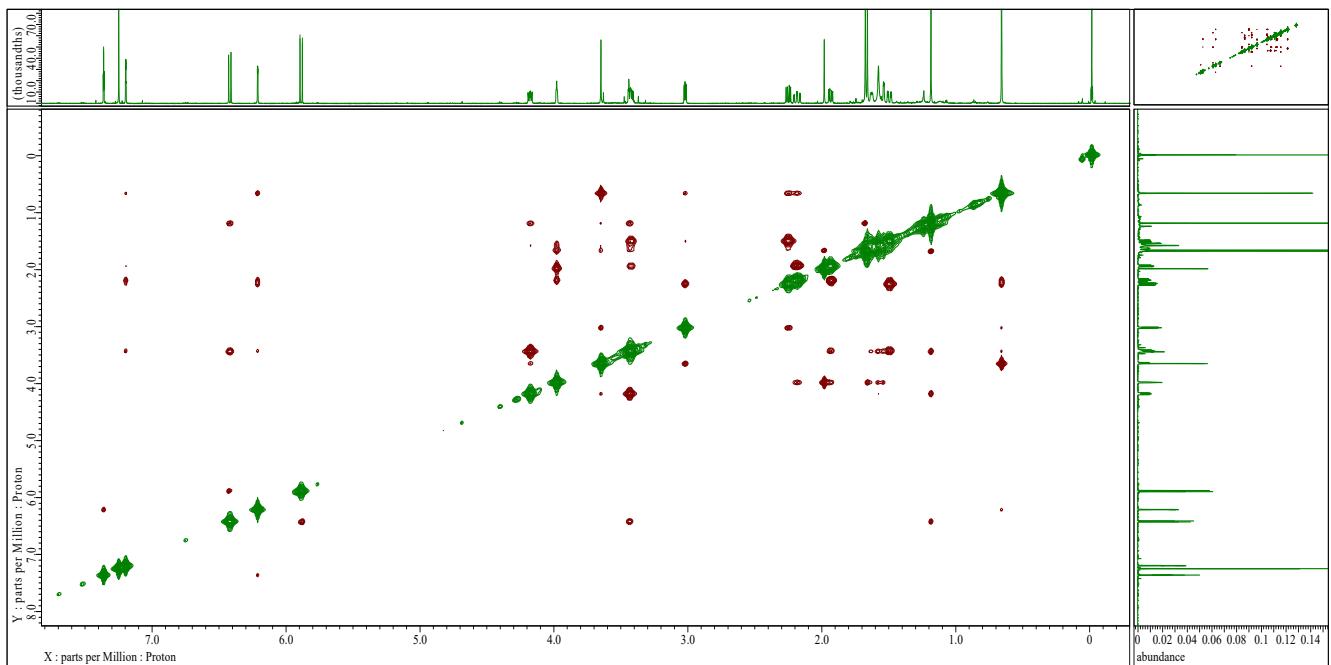


Figure S59. NOESY spectrum of compound **8** in CDCl_3 at 600MHz

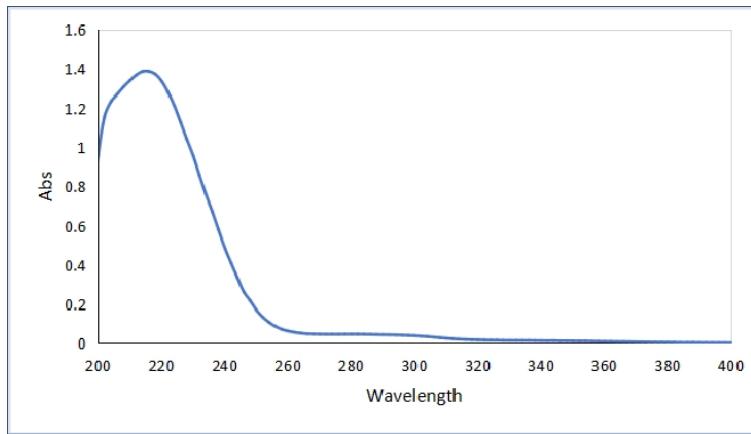


Figure S60. UV spectrum of compound **8** in MeOH

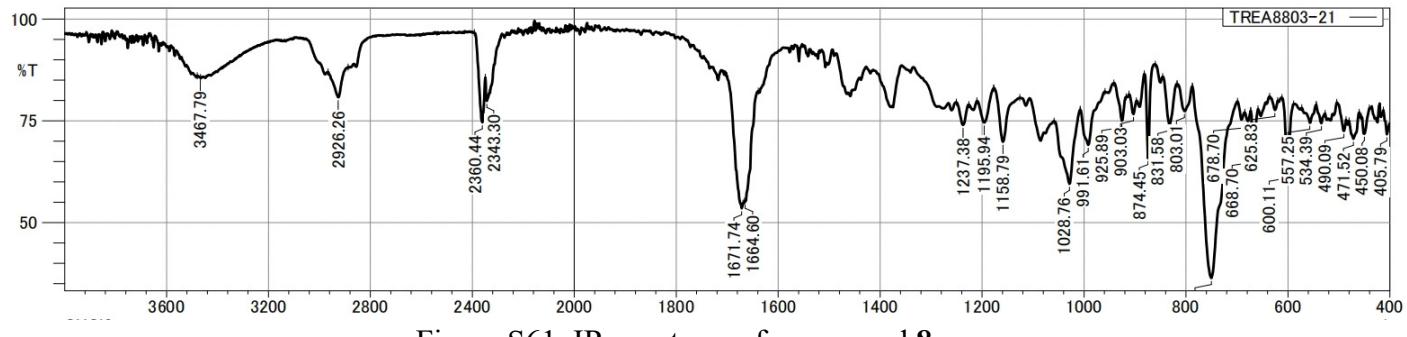


Figure S61. IR spectrum of compound **8**

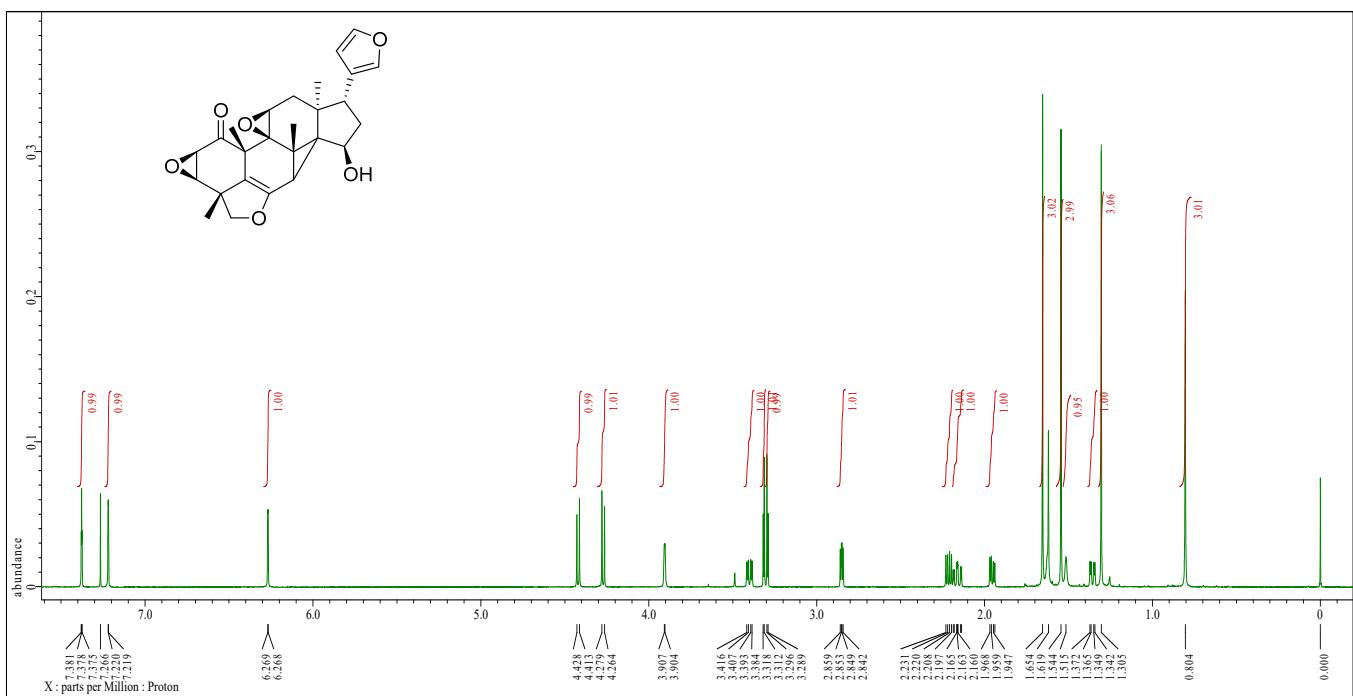


Figure S62. ^1H NMR spectrum of compound 17 in CDCl_3 at 600 MHz

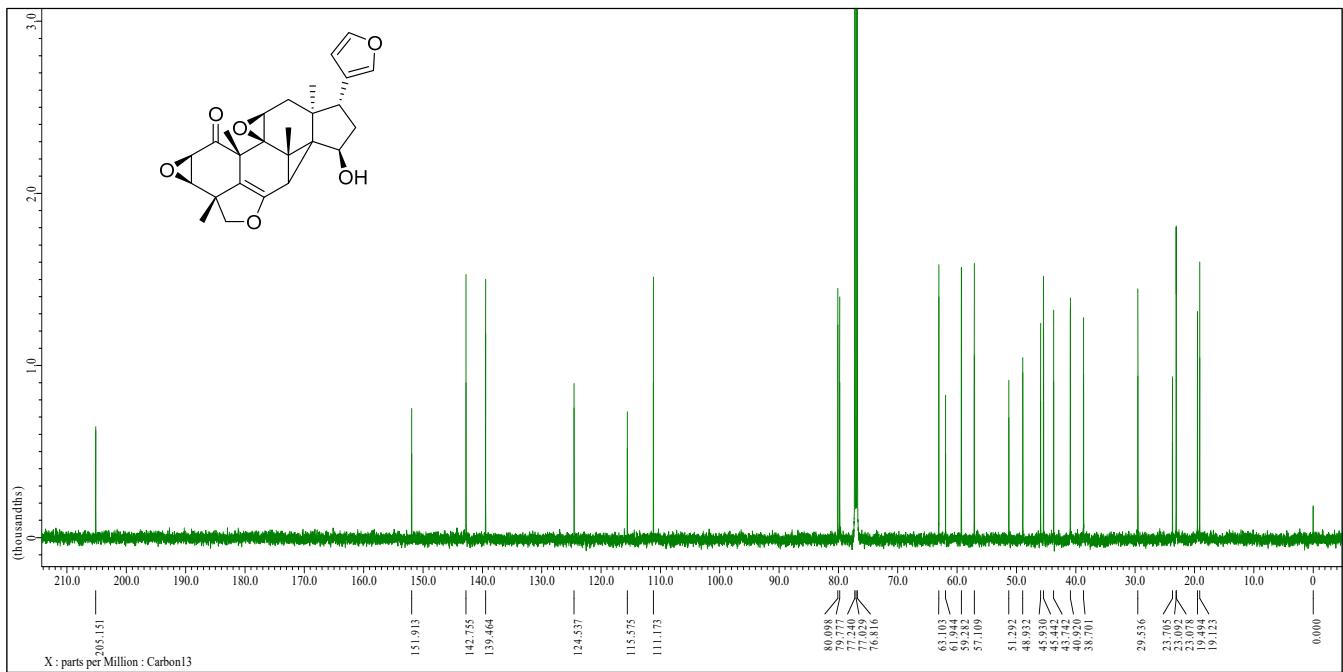
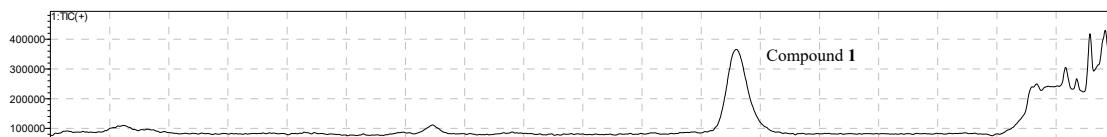


Figure S63. ^{13}C NMR spectrum of compound 17 in CDCl_3 at 150 MHz

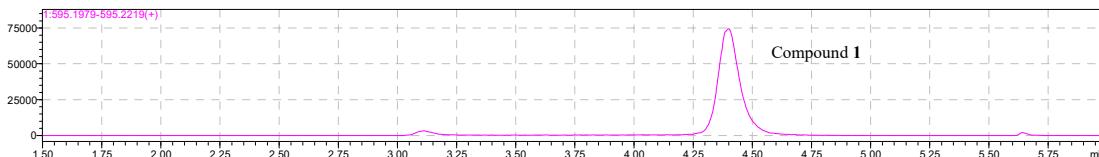
LC/MS analysis of compound 1 and fraction 7.2

An authentic sample of compound **1** was diluted with acetonitrile/water (3:1) containing 0.1% formic acid to 10 μ M. A concentration of the fraction 7.2 was also prepared to 10 μ M with the same solvent, assuming an average molecular weight of compounds containing in the fraction as 500. The sample was subjected to LCMS-9030 (Shimadzu, Kyoto, Japan) connected with Candenza CX-C18 (2 x 50 mm, 3 μ m, Imtakt, Kyoto, Japan). The mobile phases were solvent A: 0.1% formic acid in water, and solvent B: 0.1 % formic acid in acetonitrile. The gradient elution was performed as follows: 0–0.5 min, A90% /B5% ; 0.5–1.0 min, A90% /B10% B to A65% /B35% ; 1.0–5.0 min; A55% /B45%. The column temperature was maintained at 50°C, and the flow rate was 0.4 mL/min. The autosampler temperature was maintained at 4°C, and the injection volume was 3 μ L. The sample was ionized by atmospheric pressure chemical ionization (APCI) and the data were obtained in the positive ion mode with a centroid format at m/z 100–1000 on a quadrupole time-of-flight (Q-TOF) mass spectrometer.

Total ion chromatogram (TIC)



IC of 595.1979–595.2219



Mass spectrum at 4.4 min.

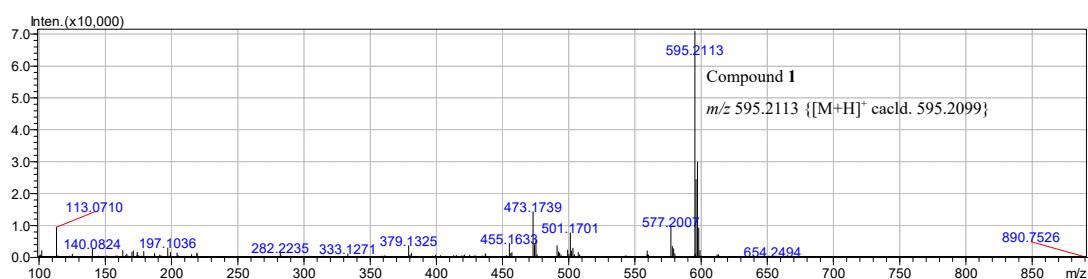
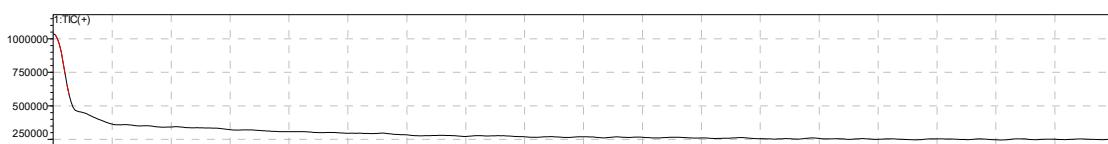
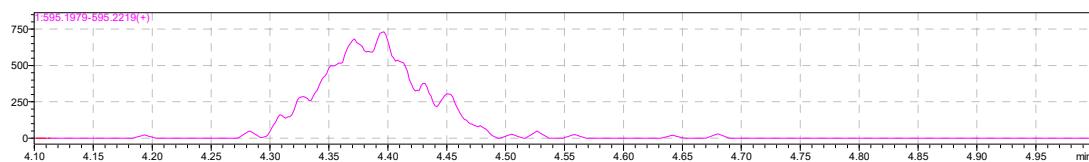


Figure S64. LC/MS analysis of compound **1**

TIC



IC of 595.1979–595.2219



Mass spectrum at 4.4 min.

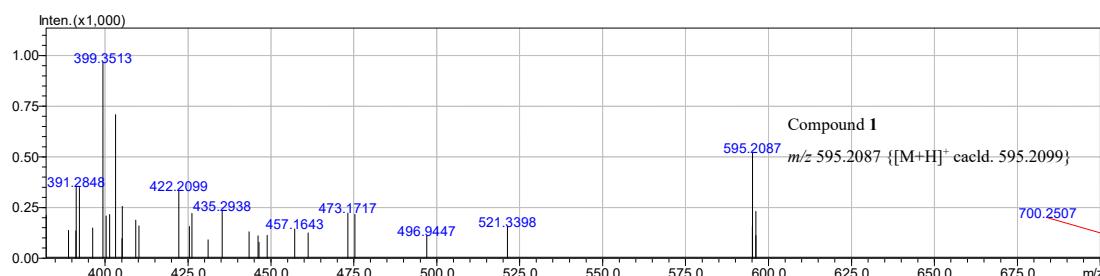
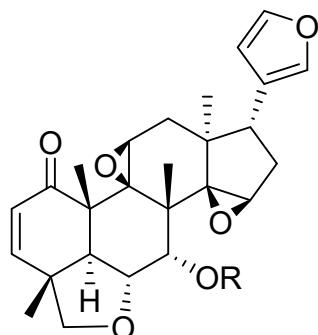
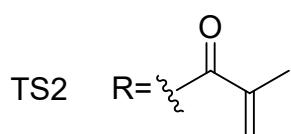


Figure S65. LC/MS analysis of fraction 7.2



TS1 R= H



TS2 R= $\text{CH}_2\text{C}(=\text{O})\text{CH}_2$

Figure S66. Structures of TS1 and TS2