Supporting Information

Unusual Vilasinin-Class Limonoids from *Trichilia rubescens*

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[4] Molecular Targets Program, Center for Cancer Research, Division of Cancer Treatment and Diagnosis, National Cancer Institute, Frederick, MD 21702-1201, USA
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* Correspondence: kngoto@p.kanazawa-u.ac.jp
† Deceased 24 October 2021.

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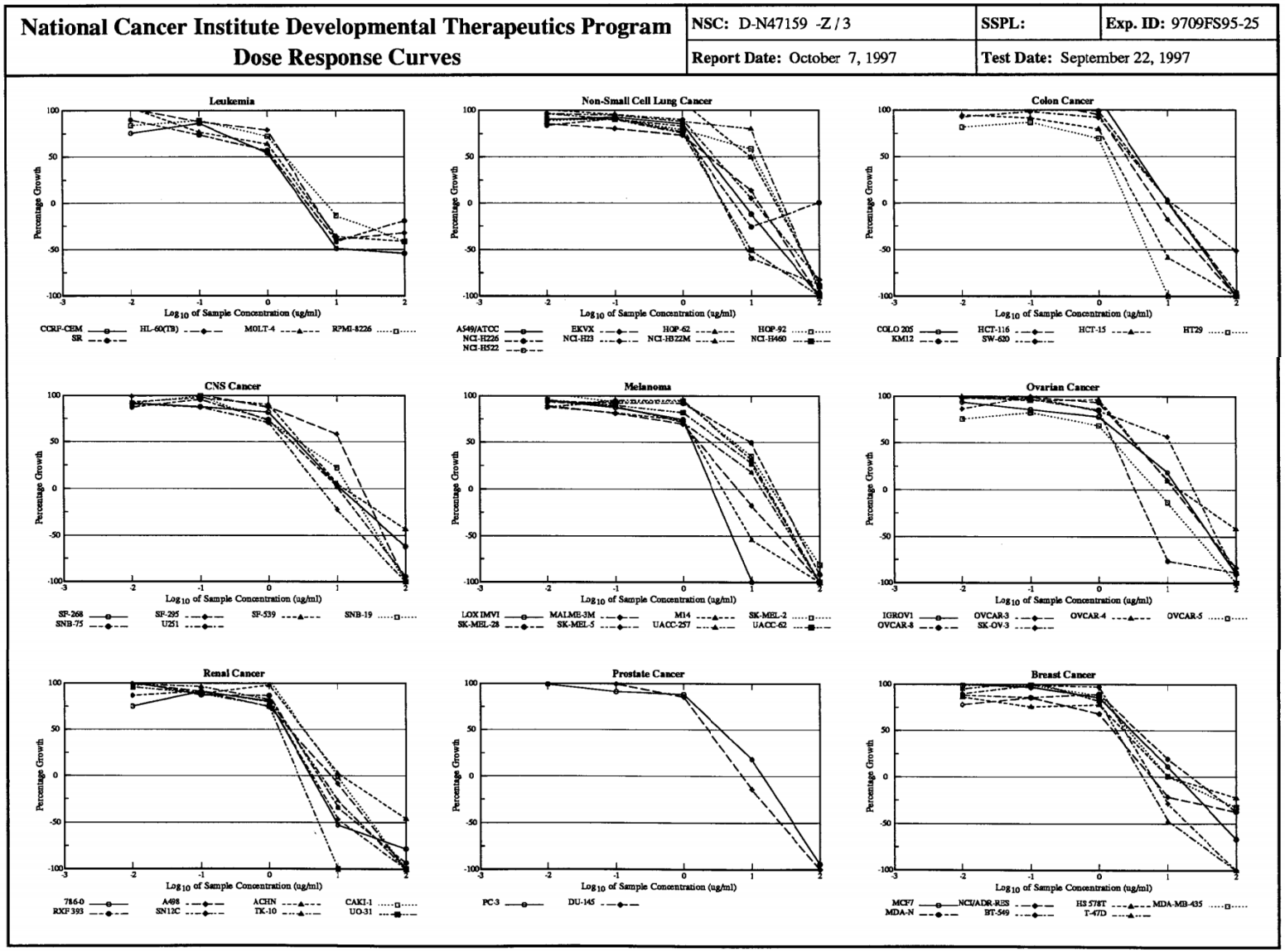


Figure S1. NCI-60 Human tumor cell line assay data for the crude organic extract of *T. rubescens* (N047159)

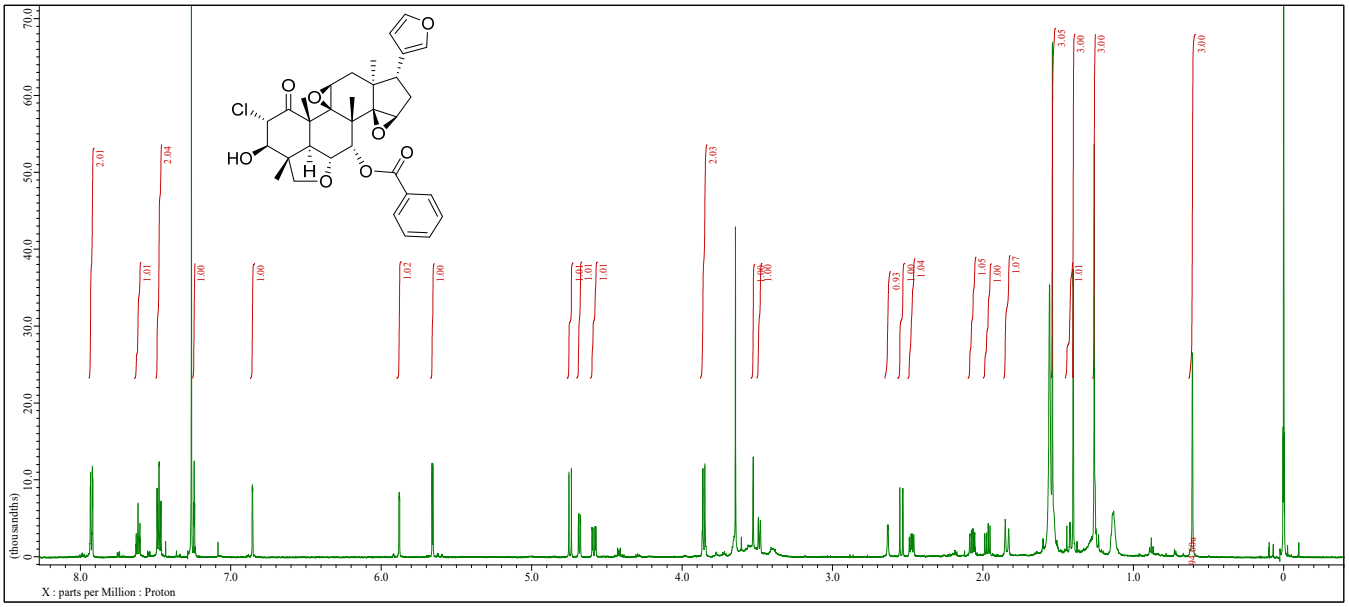


Figure S2. ^{1}H NMR spectrum of compound **1** in $CDCl_3$ at 600MHz

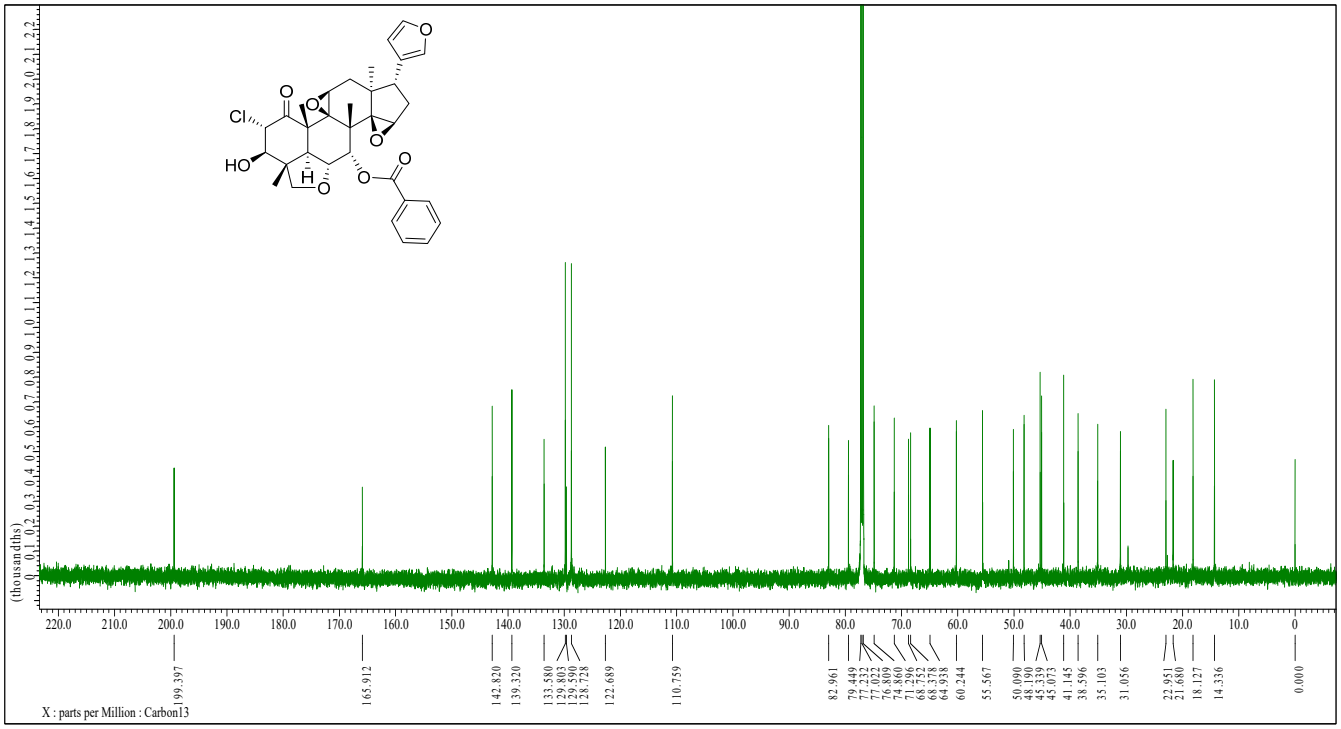


Figure S3. ^{13}C NMR spectrum of compound **1** in $CDCl_3$ at 150MHz

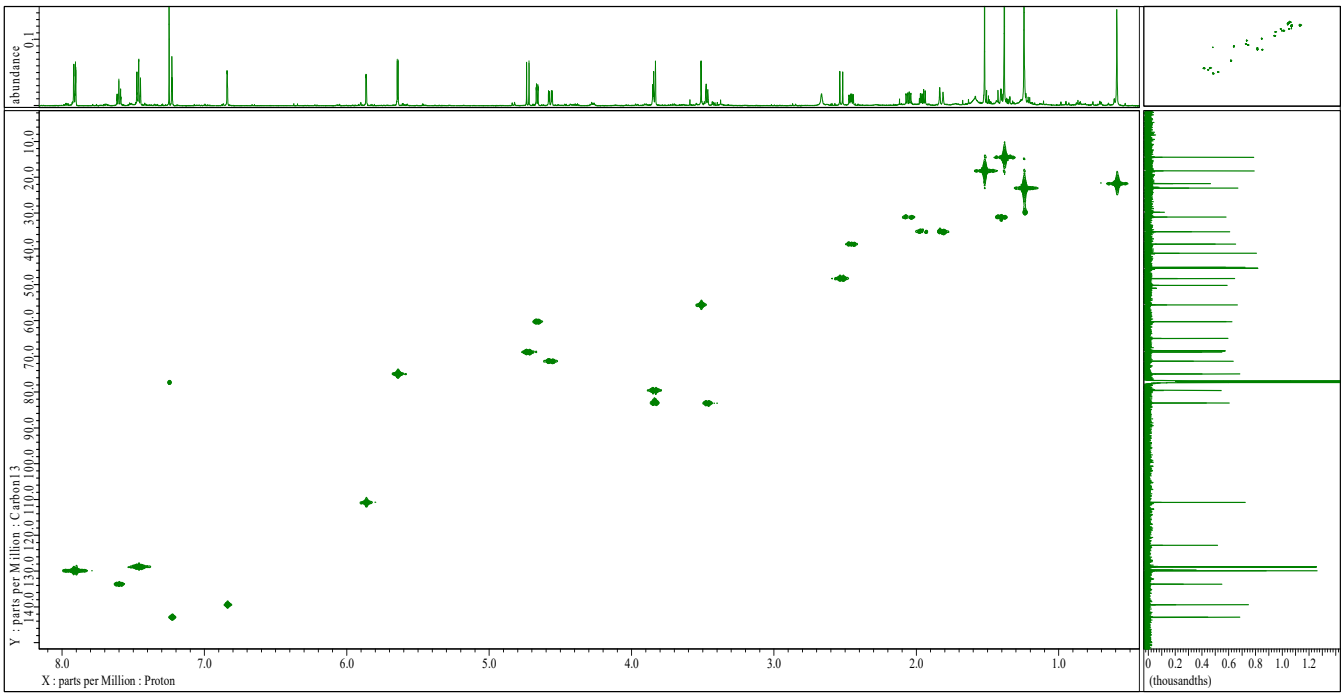


Figure S4. HMQC spectrum of compound **1** in CDCl3 at 600MHz

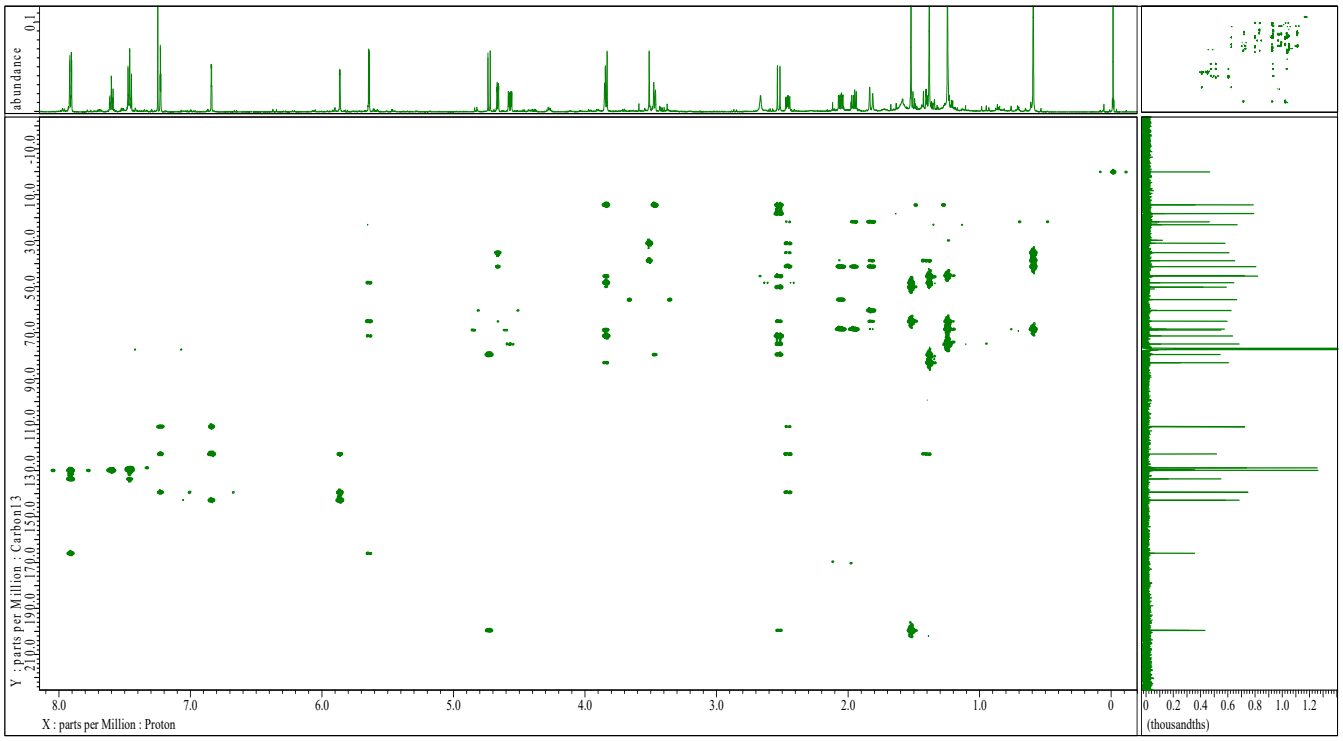


Figure S5. HMBC spectrum of compound **1** in $CDCl_3$ at 600MHz

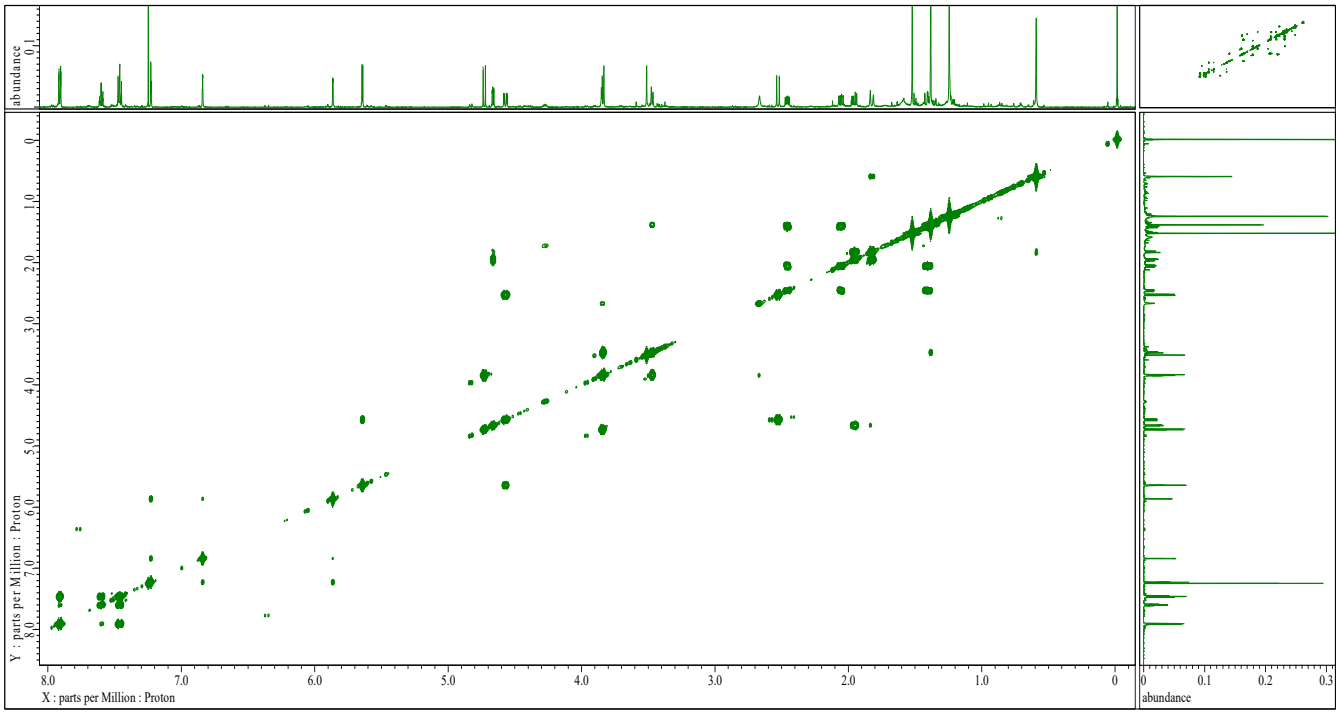


Figure S6. COSY spectrum of compound **1** in $CDCl_3$ at 600MHz

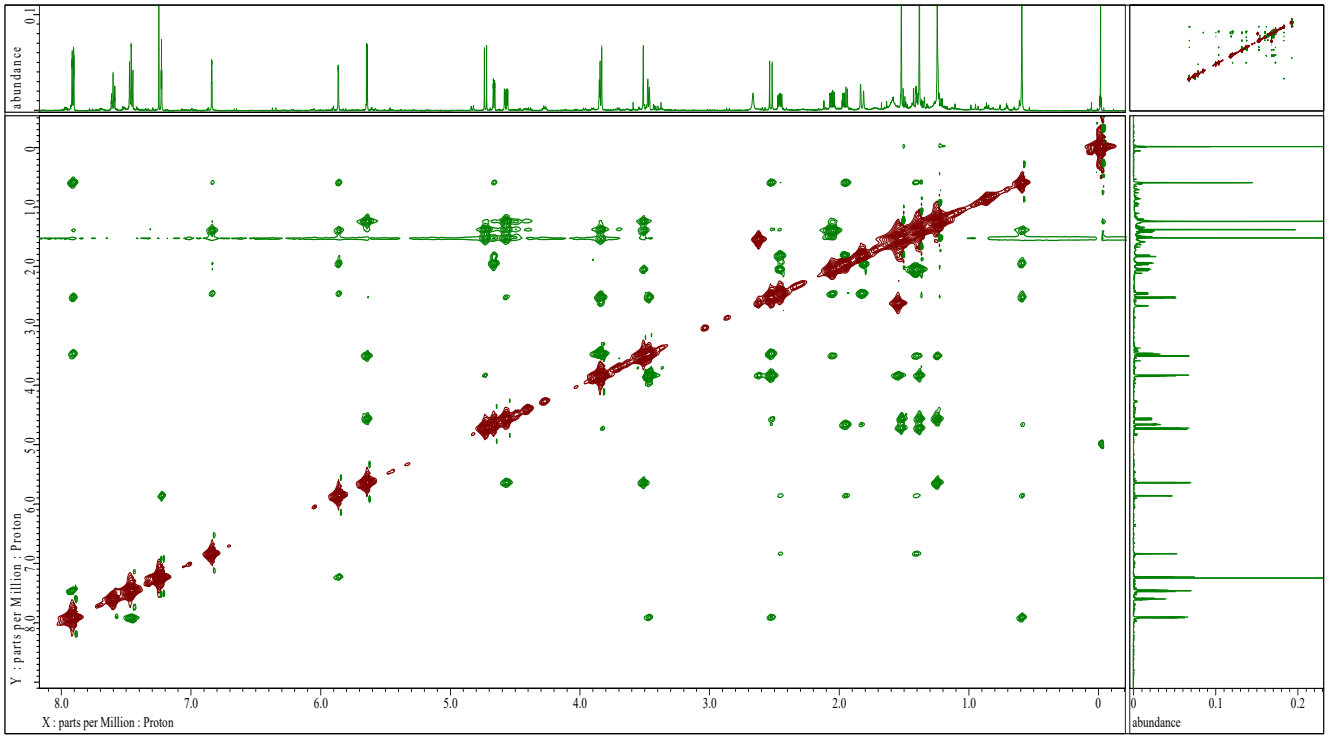


Figure S7. NOESY spectrum of compound **1** in $CDCl_3$ at 600MHz

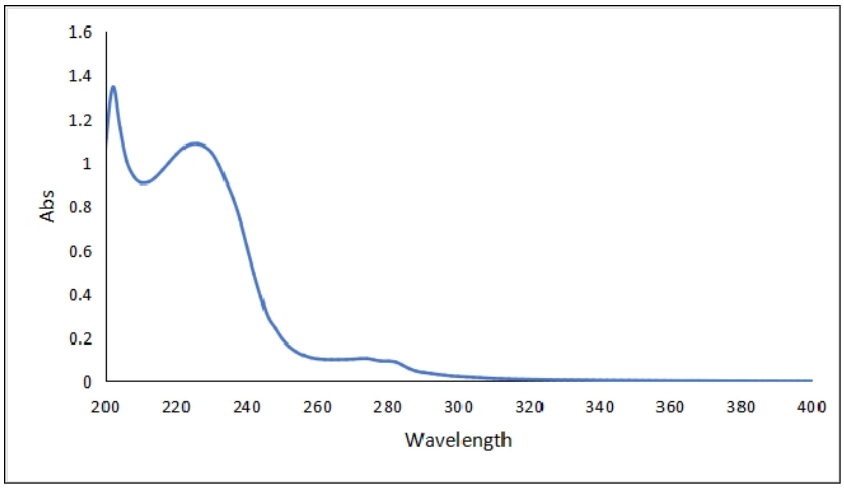


Figure S8. UV spectrum of compound **1** in MeOH

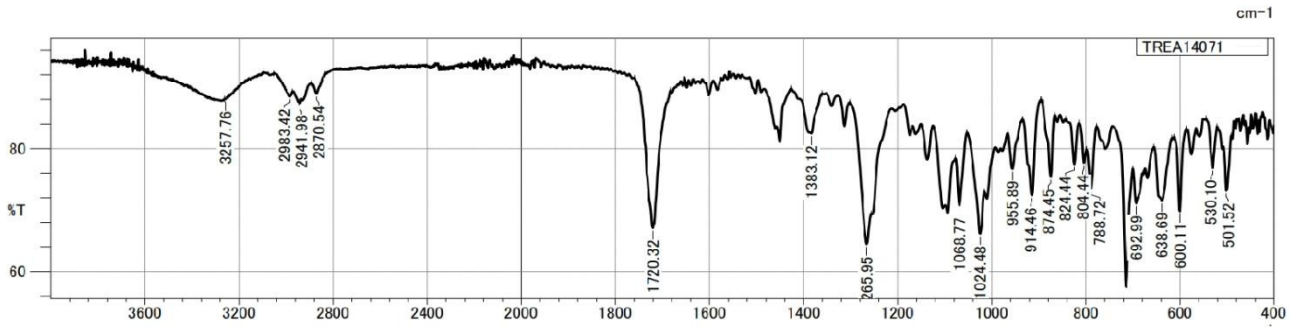


Figure S9. IR spectrum of compound **1**

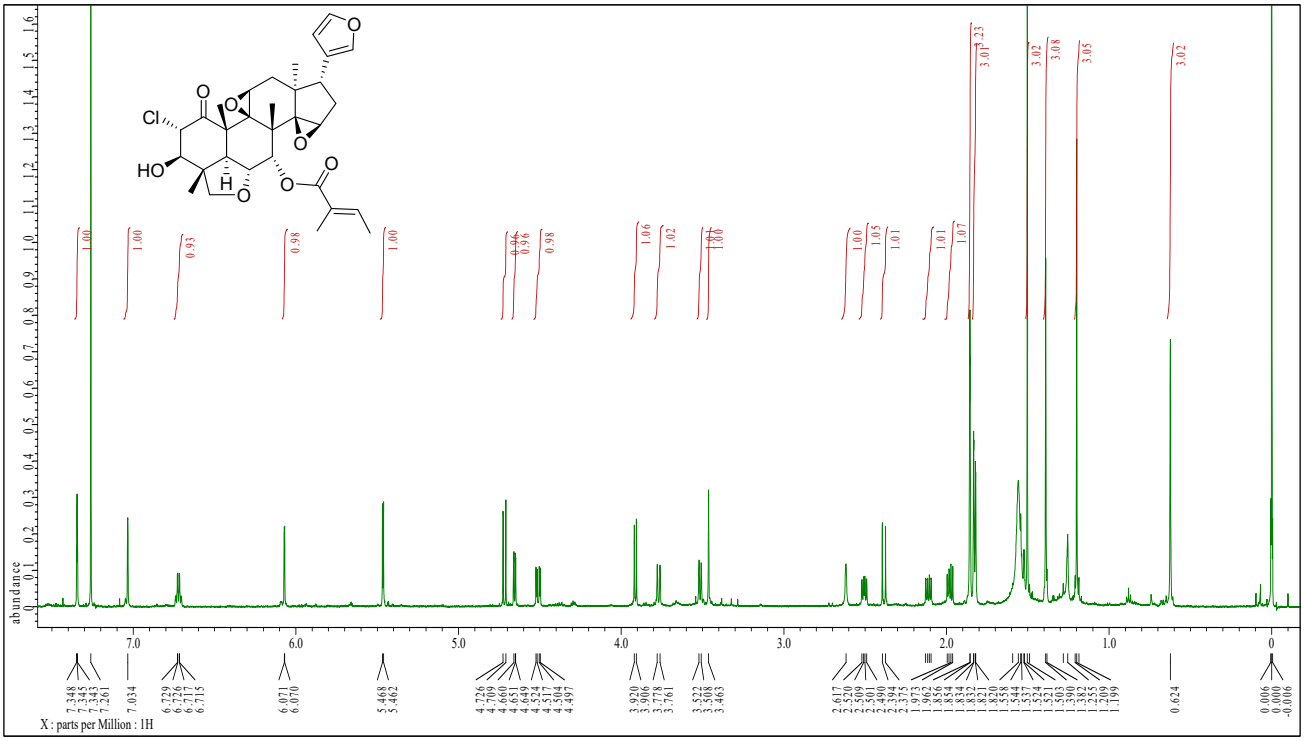


Figure S10. ^{1}H NMR spectrum of compound **2** in $CDCl_3$ at 600MHz

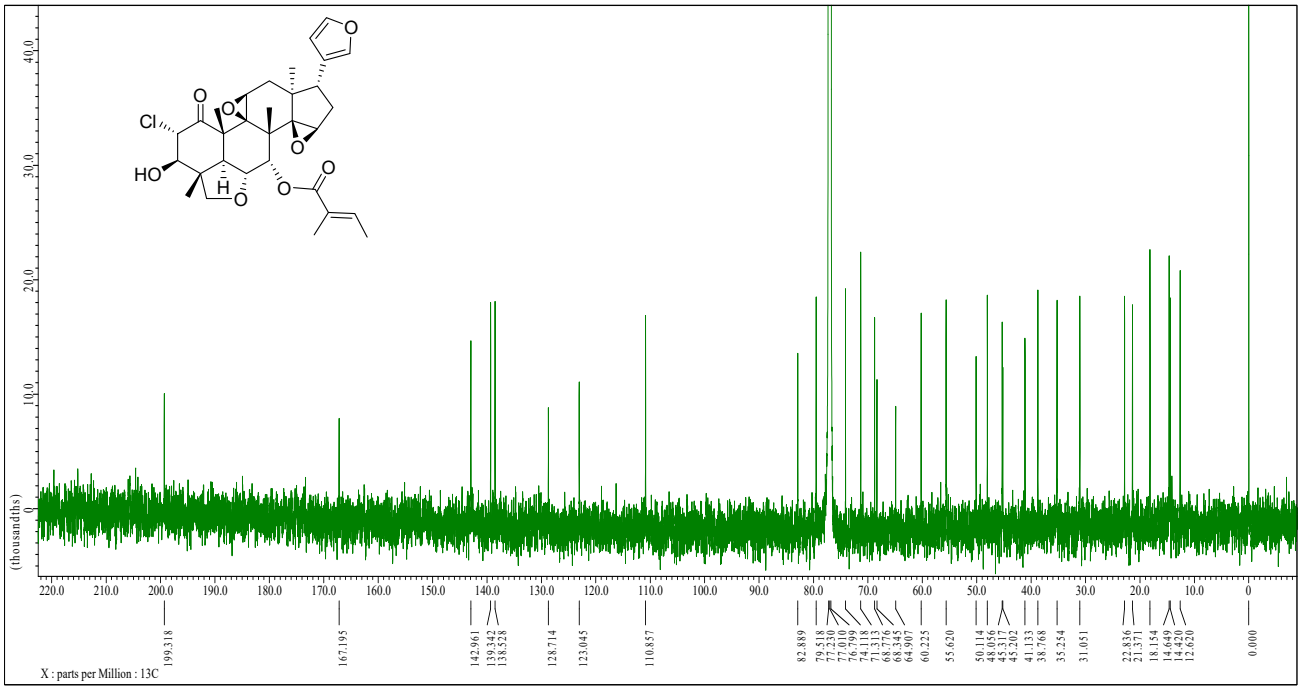


Figure S11.^{13}C NMR spectrum of compound **2** in $CDCl_3$ at 150MHz

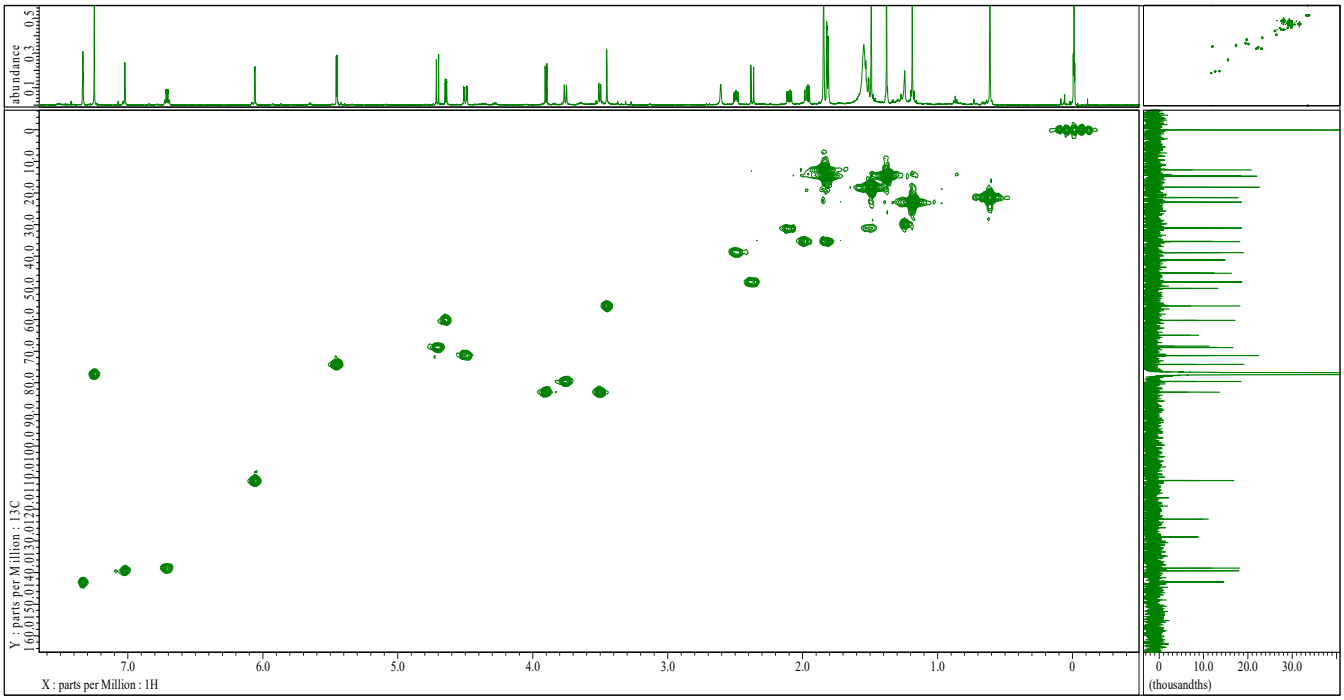


Figure S12. HMQC spectrum of compound **2** in $CDCl_3$ at 600MHz

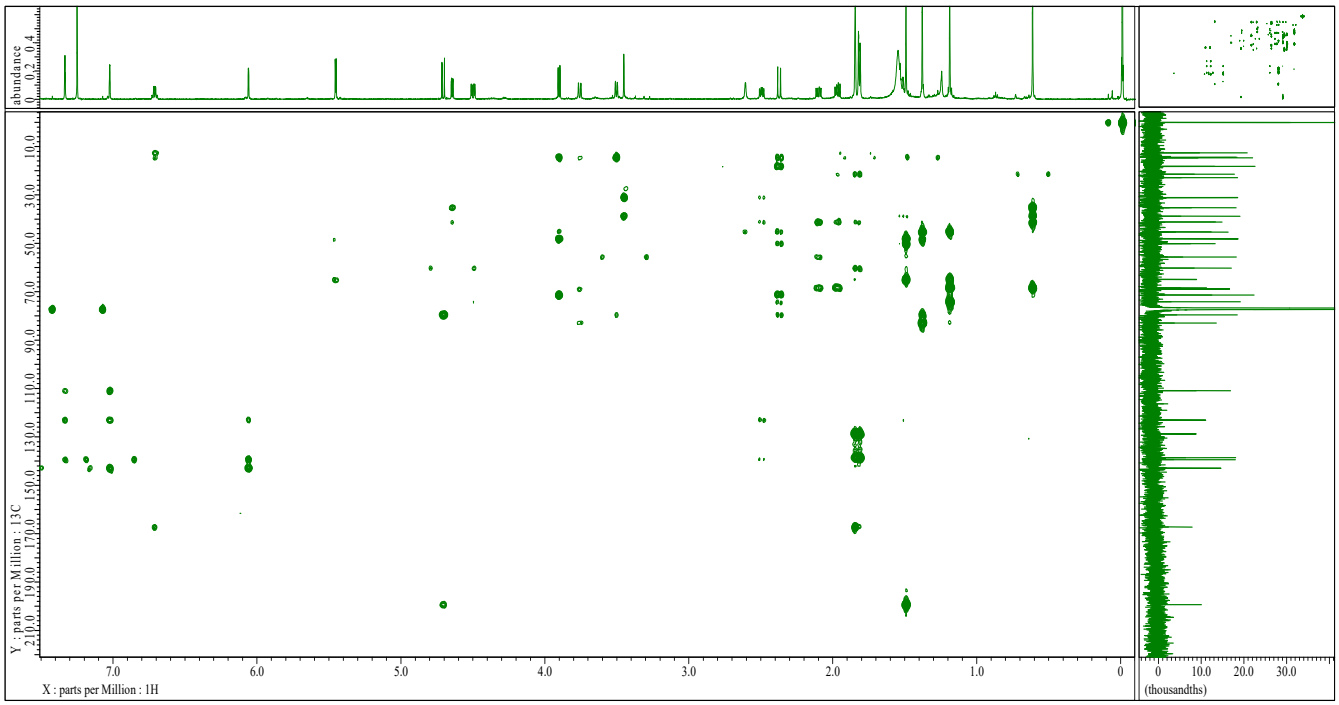


Figure S13. HMBC spectrum of compound **2** in $CDCl_3$ at 600MHz

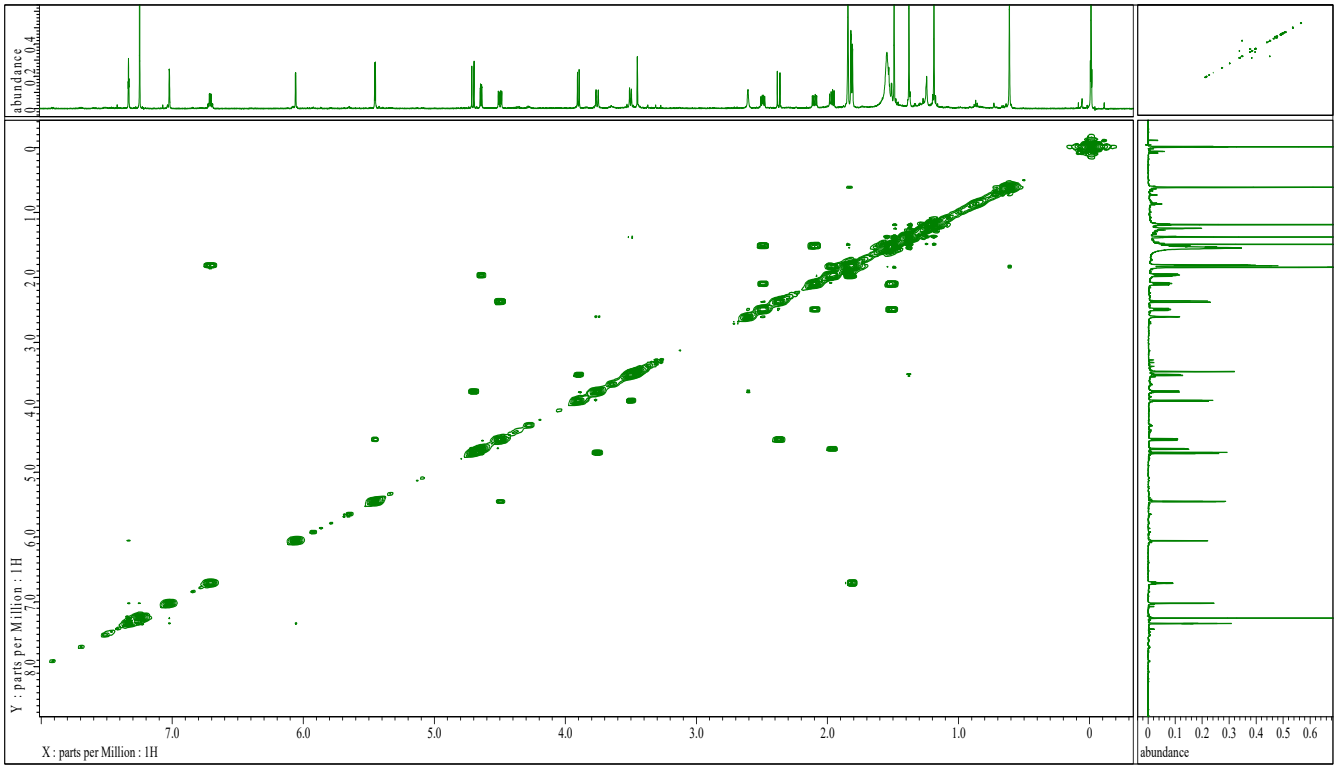


Figure S14. COSY spectrum of compound **2** in $CDCl_3$ at 600MHz

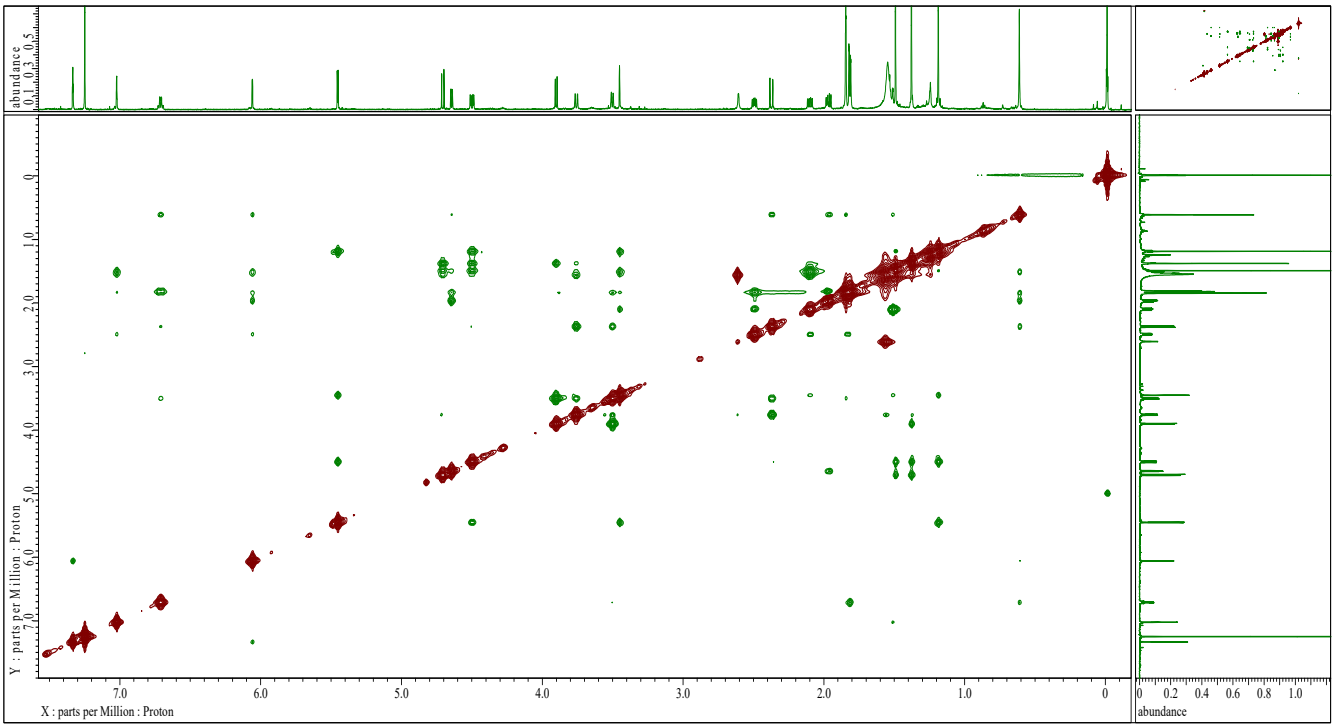


Figure S15. NOESY spectrum of compound **2** in $CDCl_3$ at 600MHz

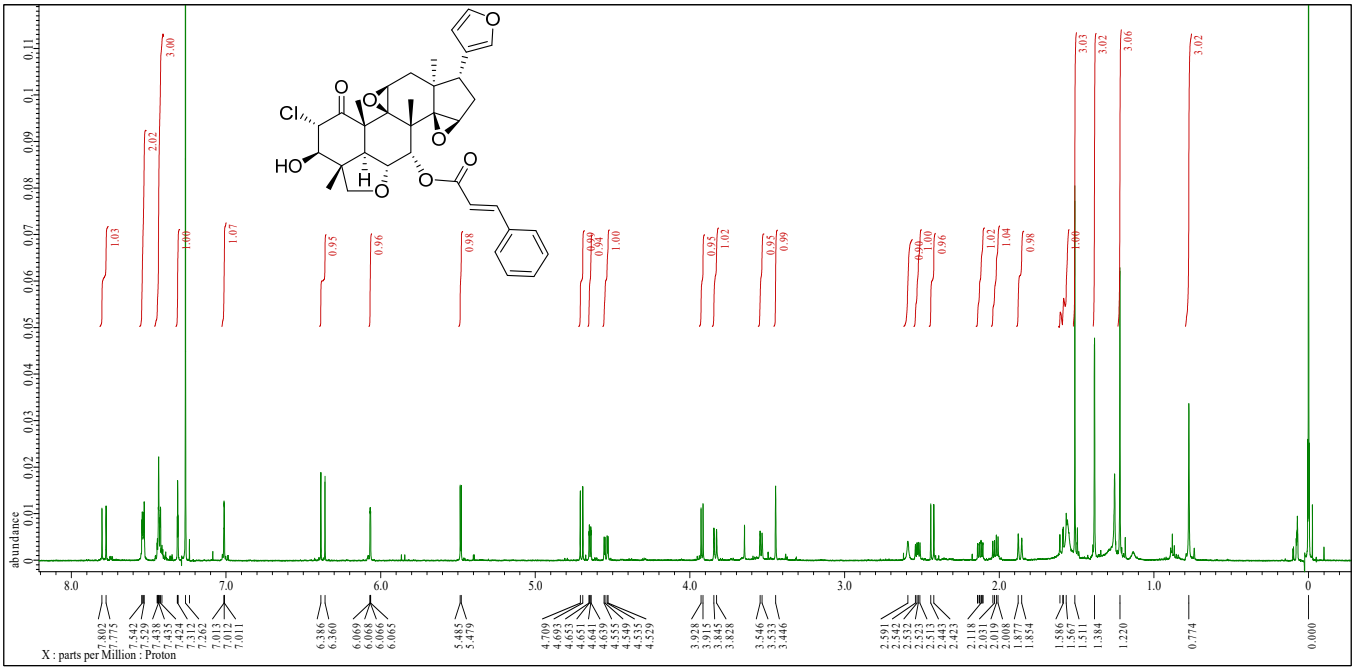


Figure S16. ^{1}H NMR spectrum of compound **3** in $CDCl_3$ at 600MHz

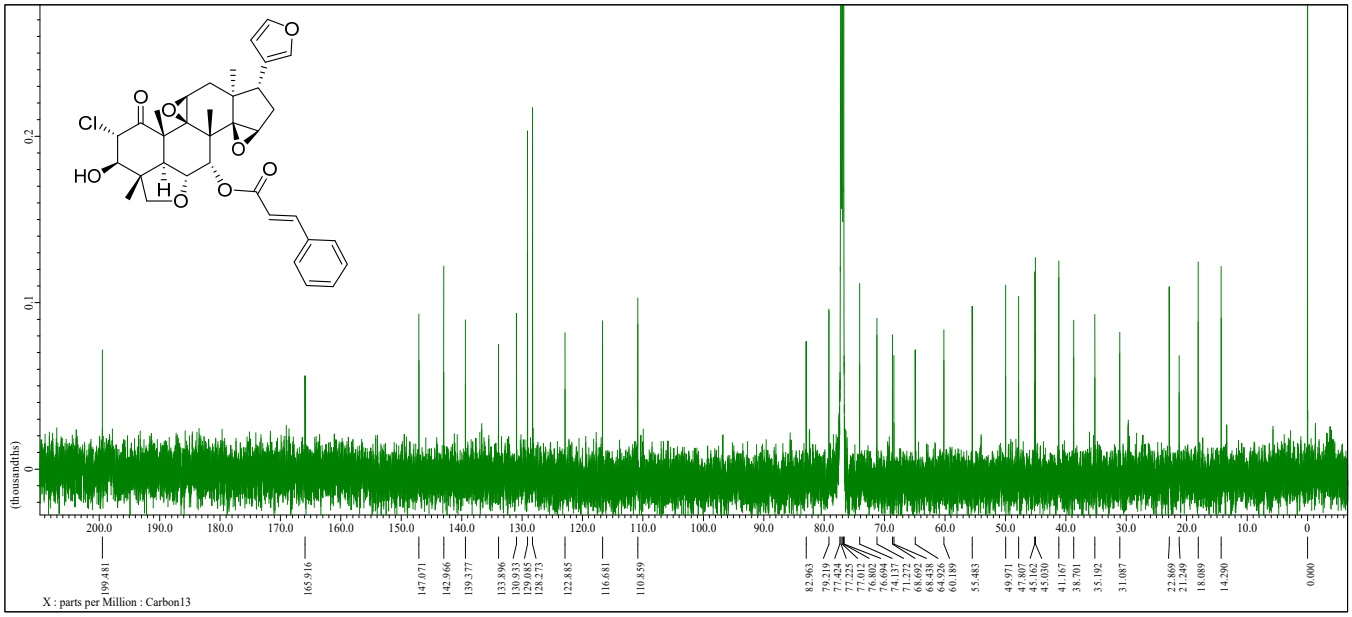


Figure S17.^{13}C NMR spectrum of compound **3** in $CDCl_3$ at 150MHz

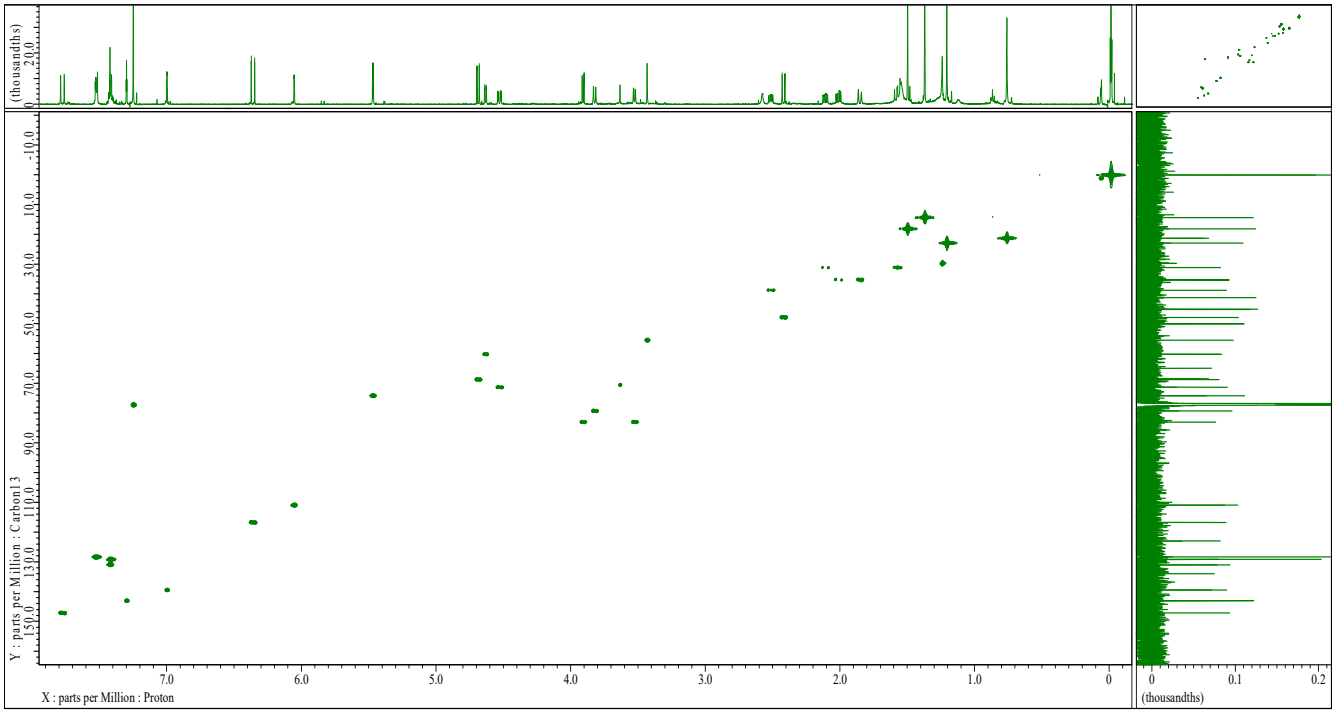


Figure S18. HMQC spectrum of compound **3** in $CDCl_3$ at 600MHz

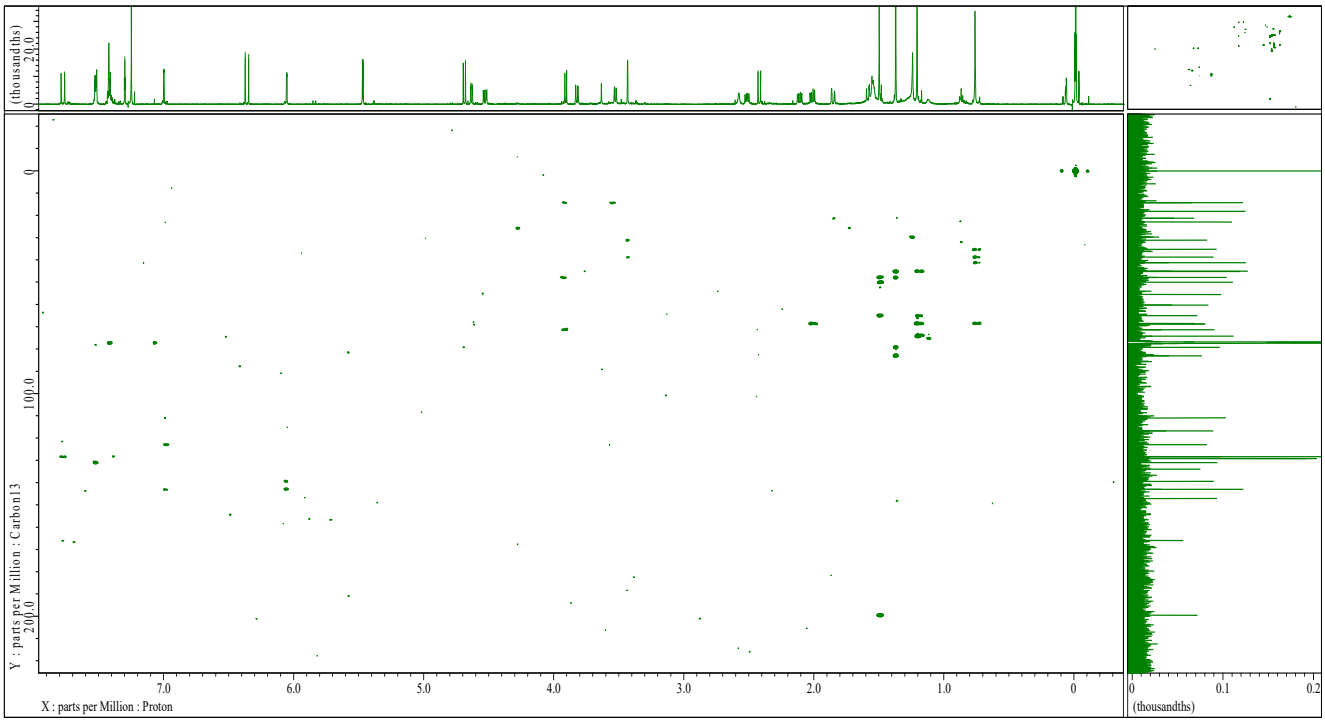


Figure S19. HMBC spectrum of compound **3** in $CDCl_3$ at 600MHz

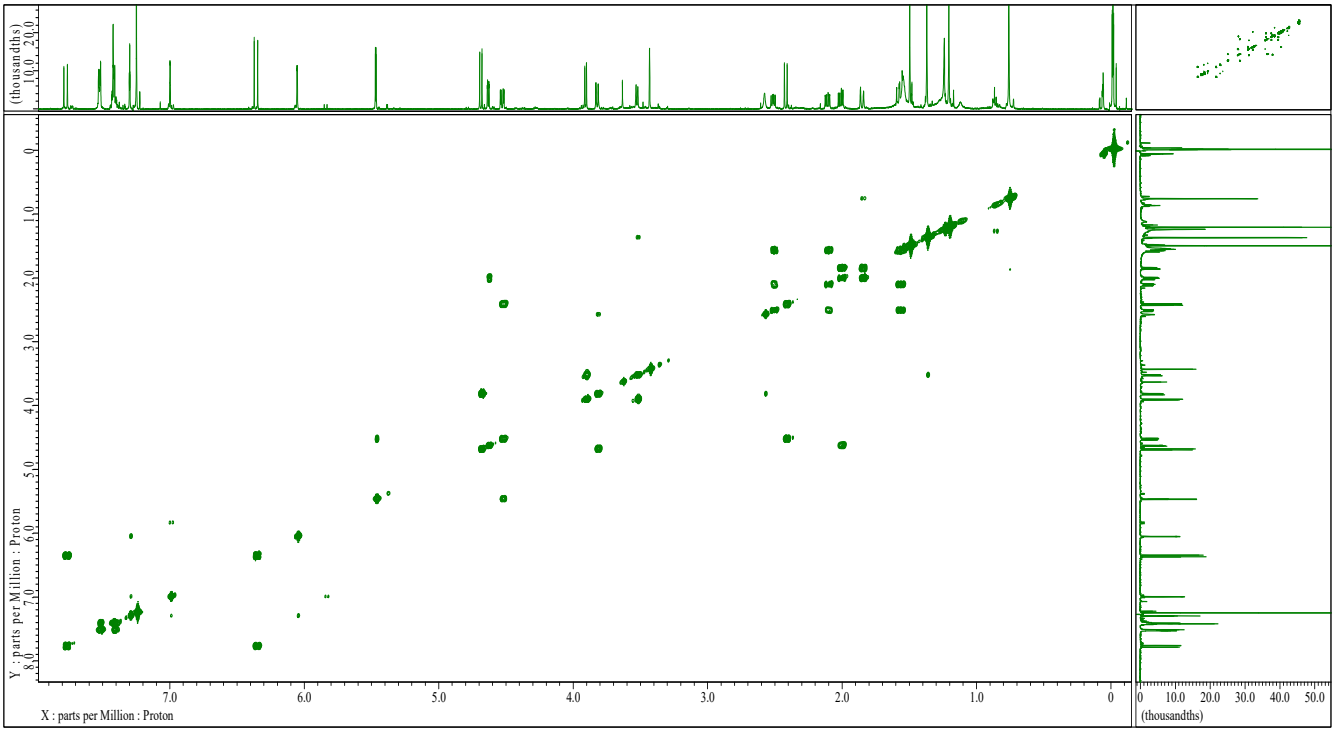


Figure S20. COSY spectrum of compound **3** in $CDCl_3$ at 600MHz

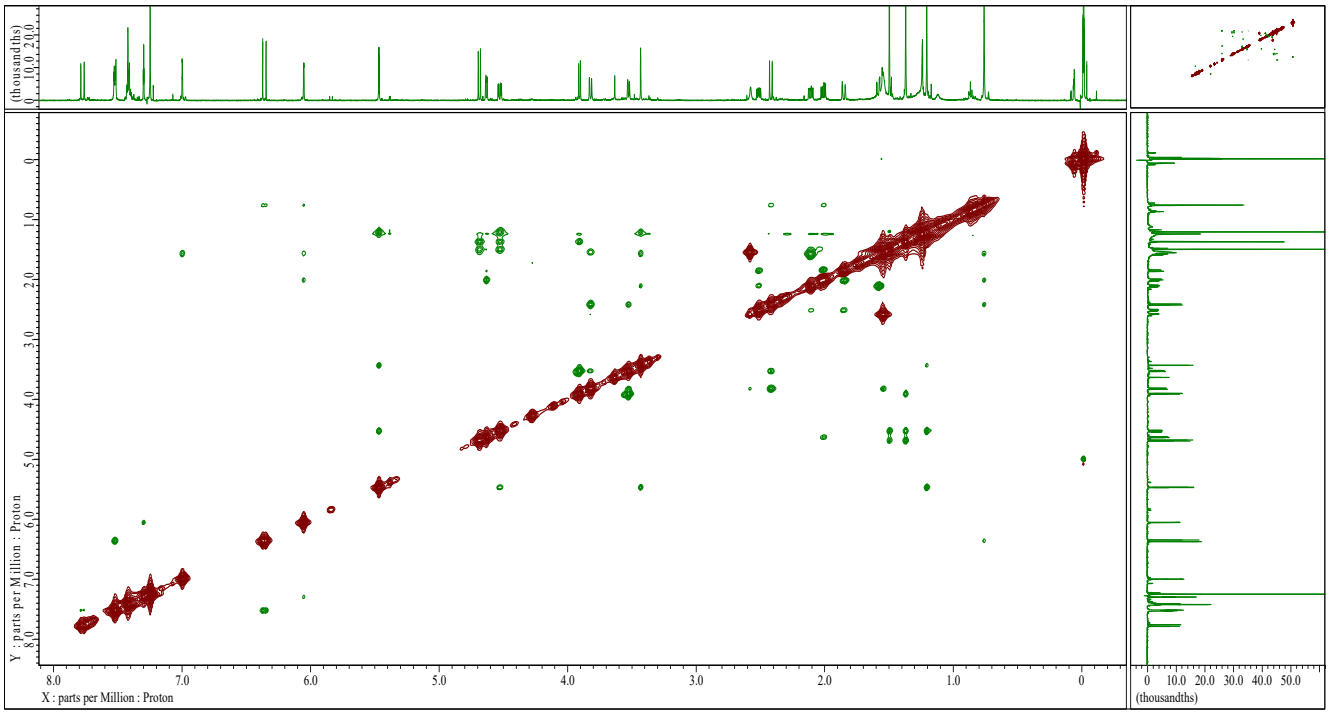


Figure S21. NOESY spectrum of compound **3** in $CDCl_3$ at 600MHz

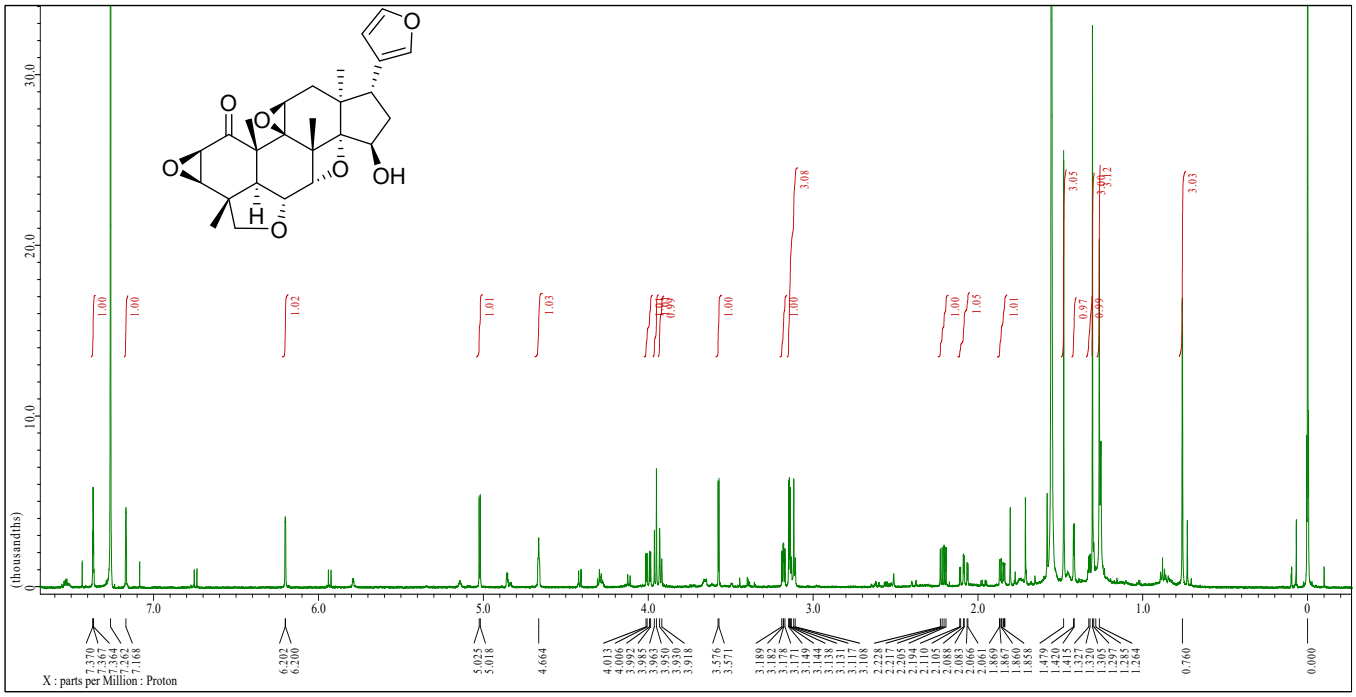


Figure S22. ^{1}H NMR spectrum of compound **4** in $CDCl_3$ at 600MHz

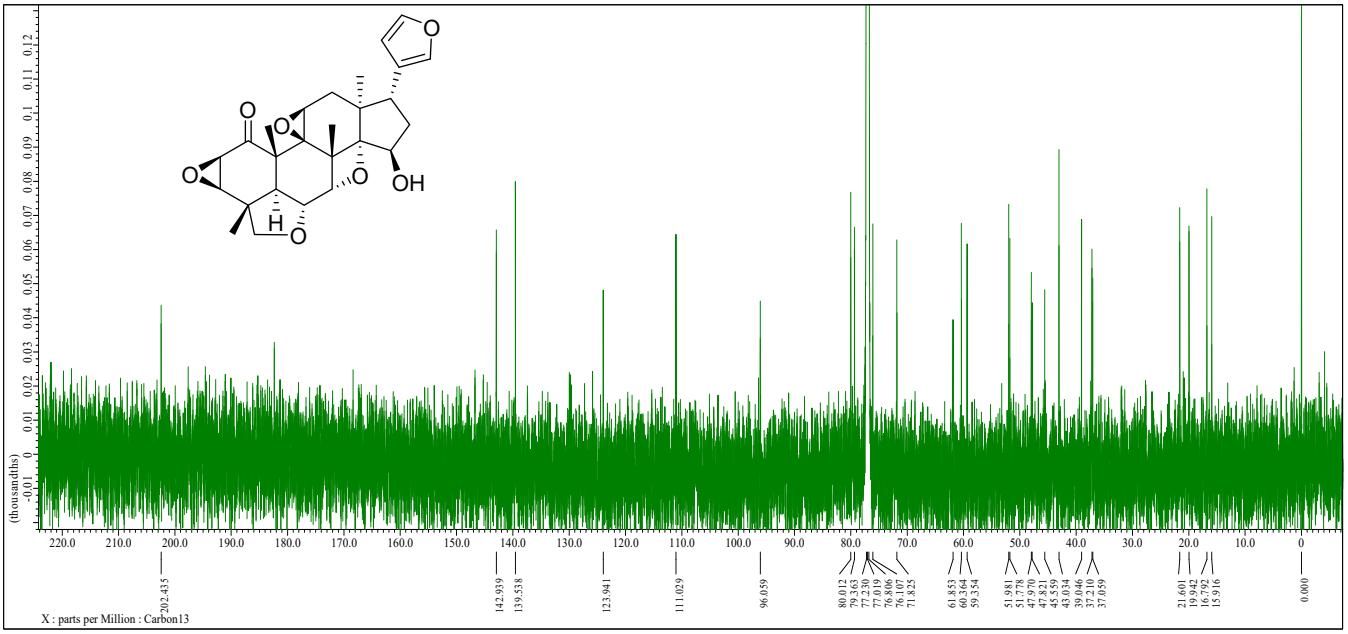


Figure S23.^{13}C NMR spectrum of compound **4** in $CDCl_3$ at 150MHz

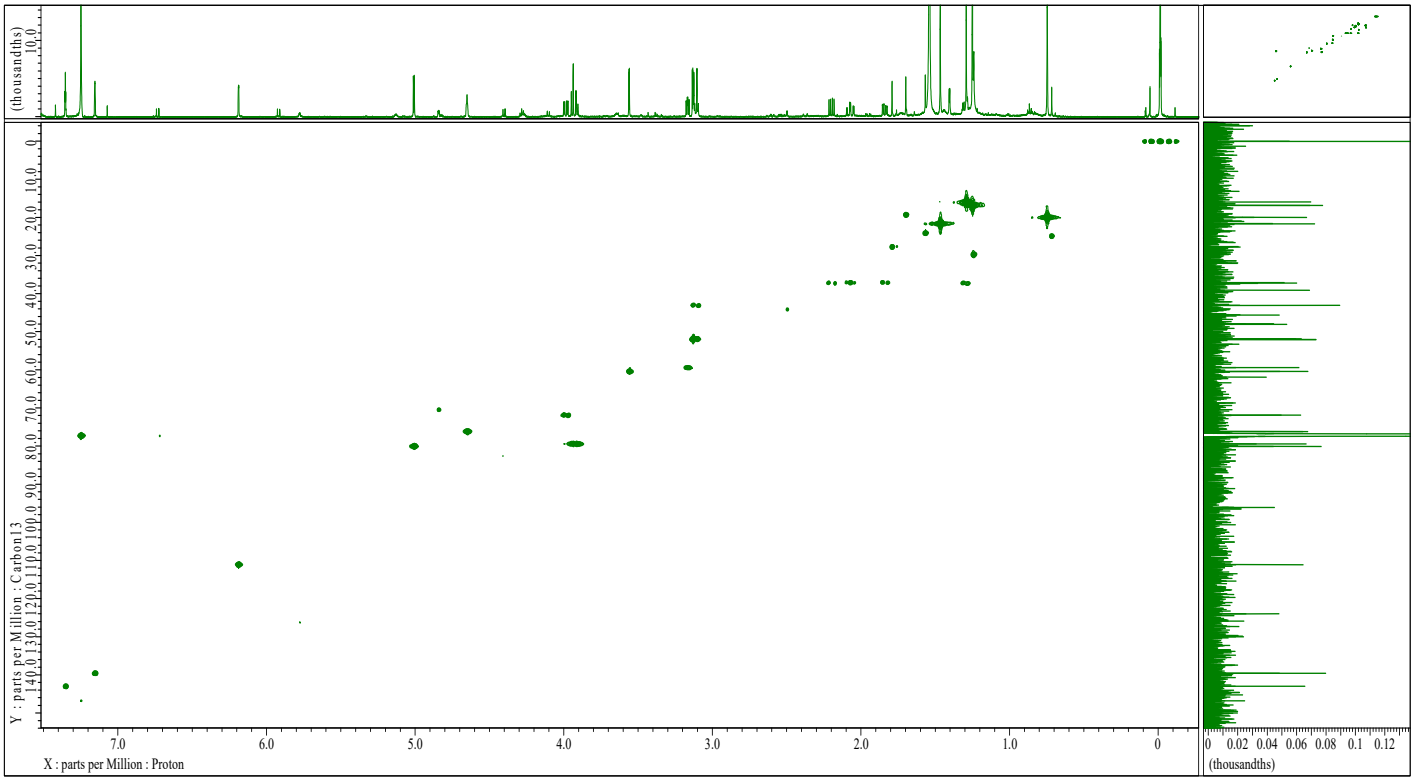


Figure S24. HMQC spectrum of compound **4** in $CDCl_3$ at 600MHz

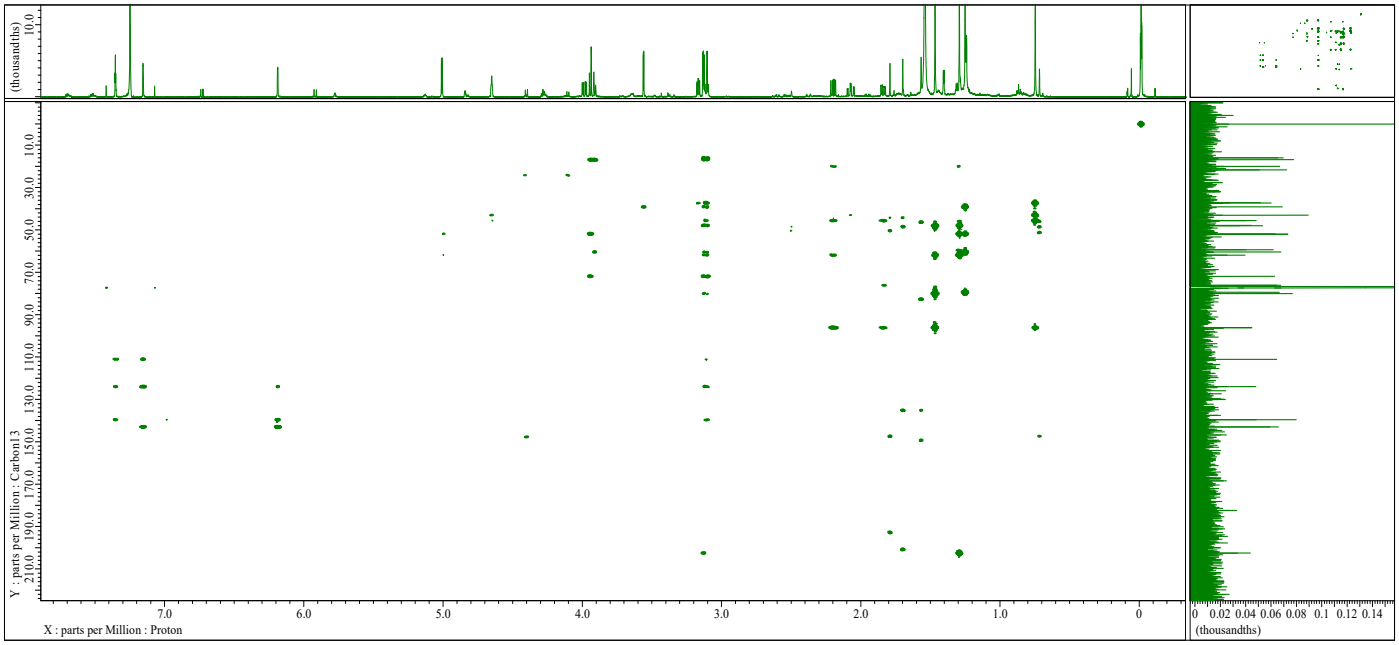


Figure S25. HMBC spectrum of compound **4** in $CDCl_3$ at 600MHz

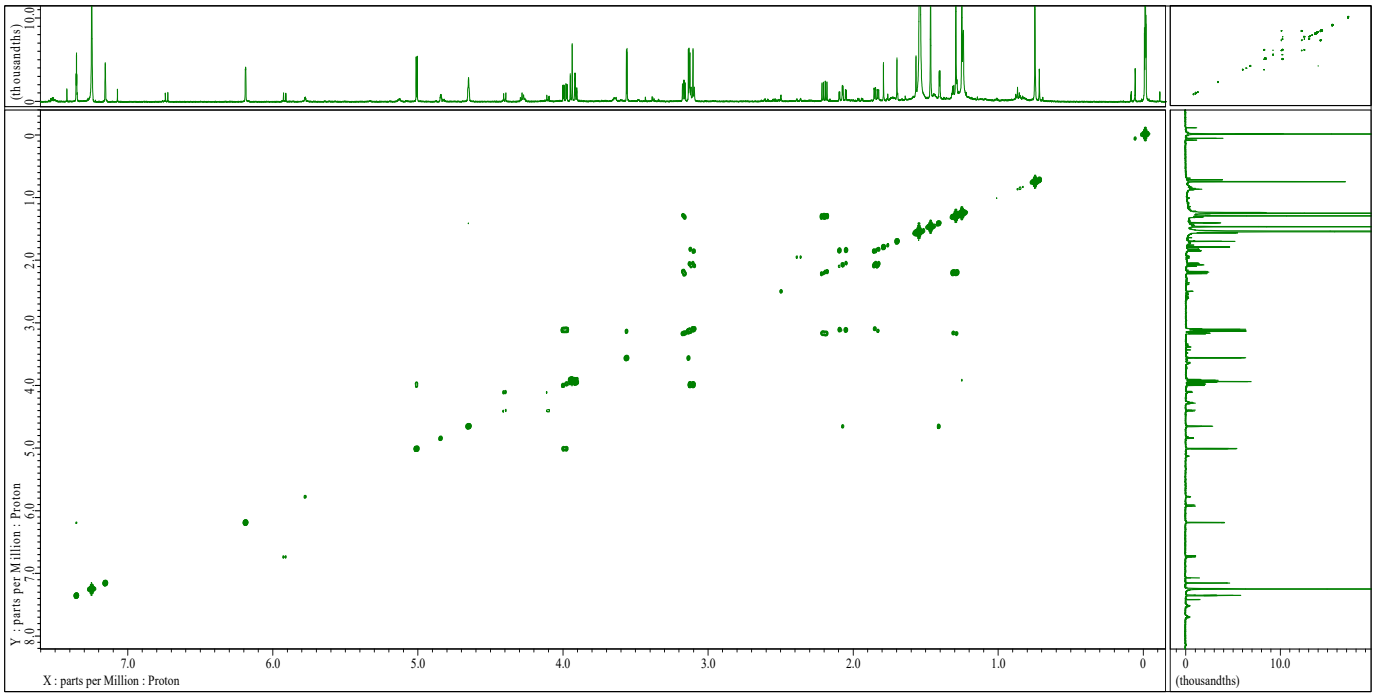


Figure S26. COSY spectrum of compound **4** in $CDCl_3$ at 600MHz

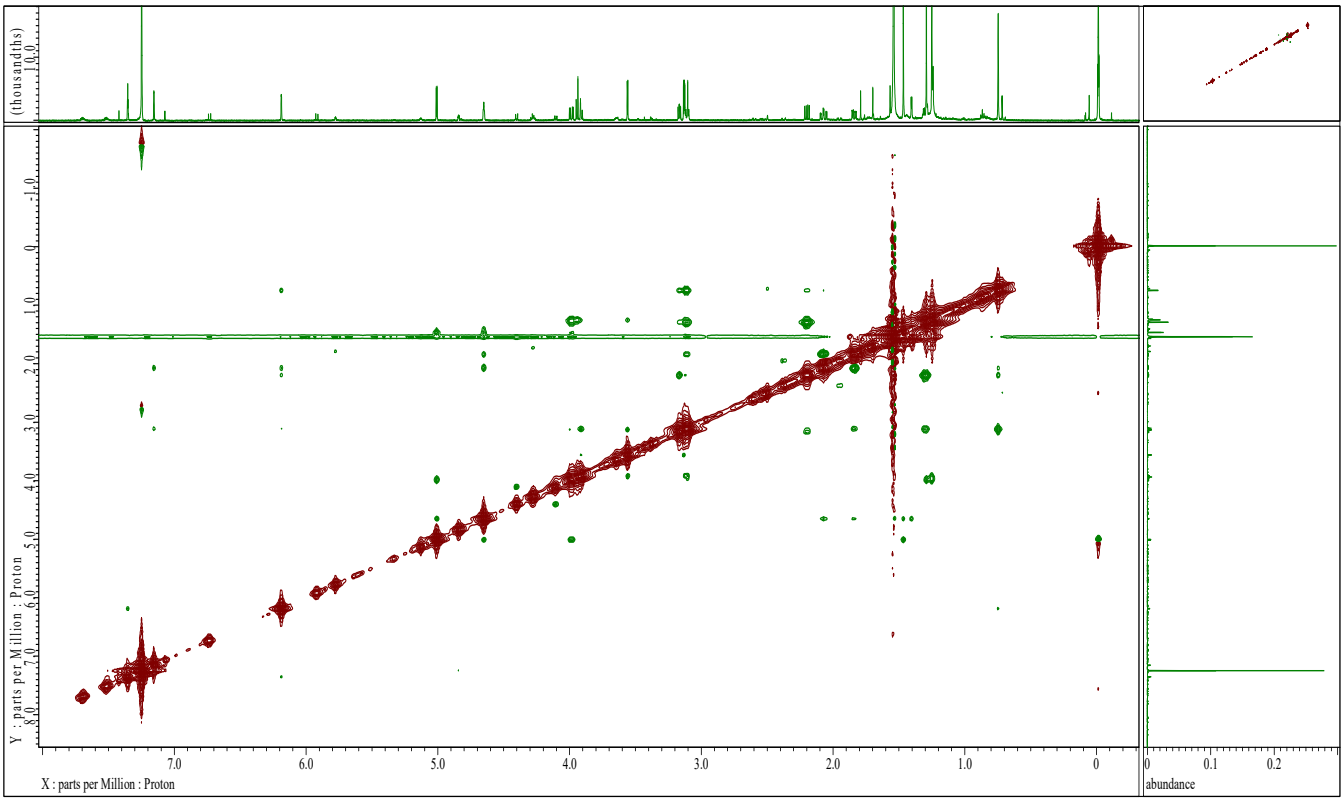


Figure S27. NOESY spectrum of compound **4** in $CDCl_3$ at 600MHz

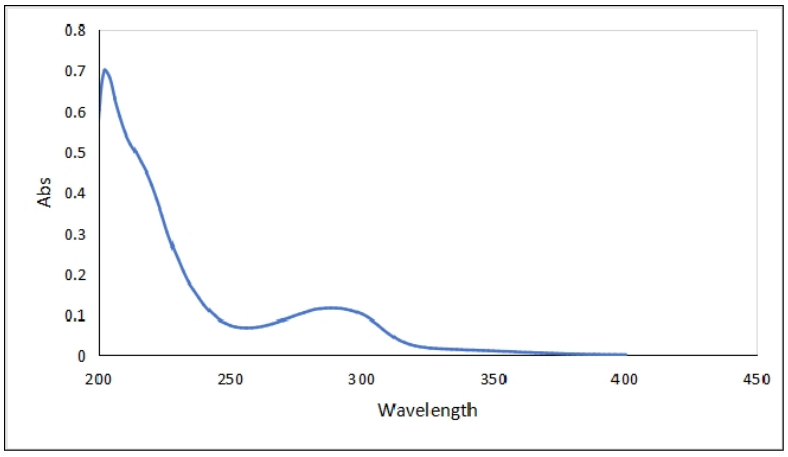


Figure S28. UV spectrum of compound **4** in MeOH

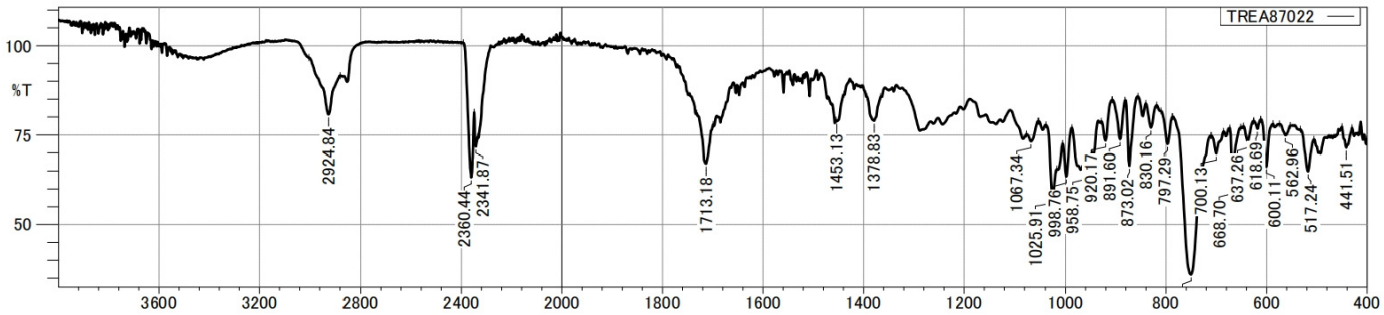


Figure S29. IR spectrum of compound **4**

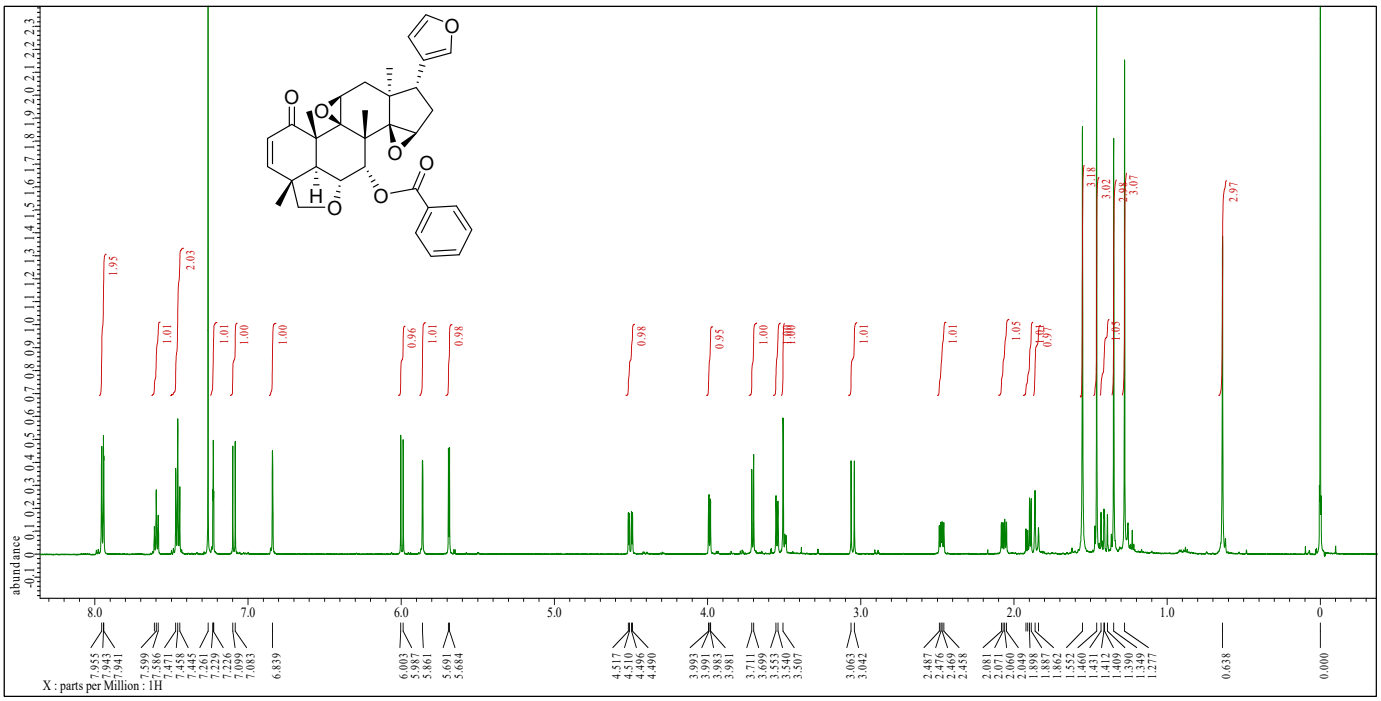


Figure S30. ^{1}H NMR spectrum of compound **5** in $CDCl_3$ at 600MHz

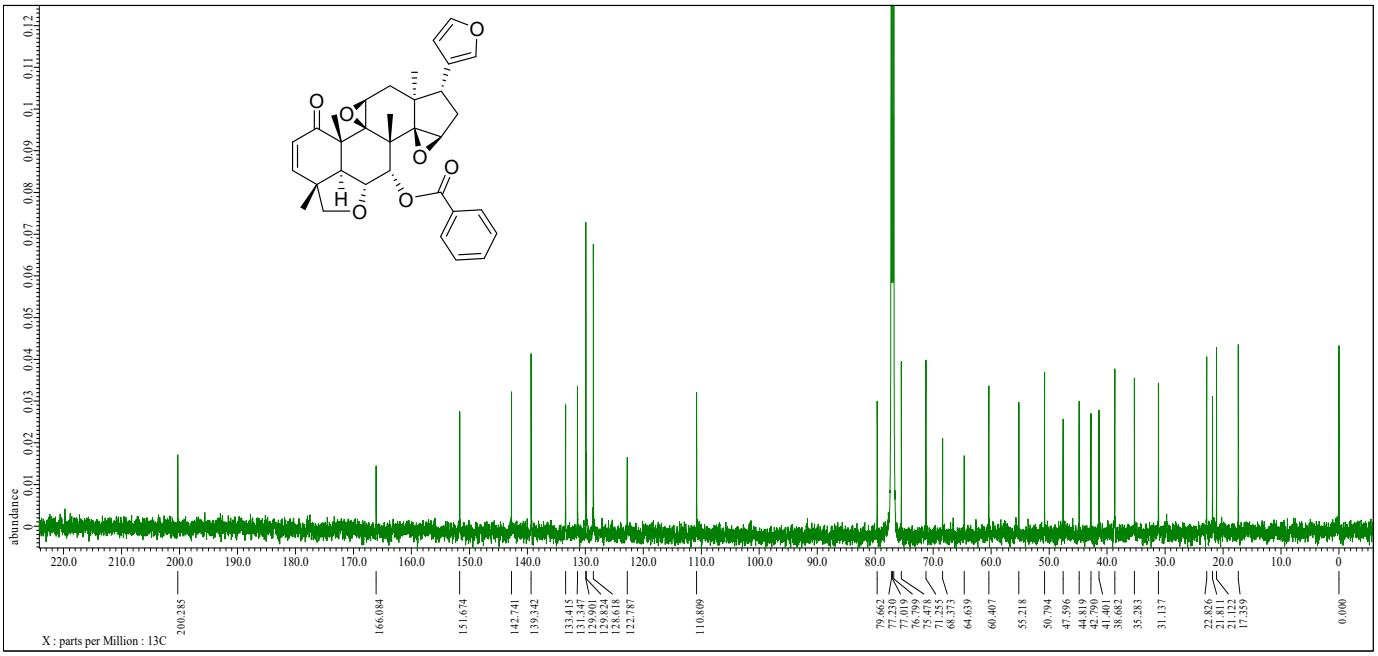


Figure S31.^{13}C NMR spectrum of compound **5** in $CDCl_3$ at 150MHz

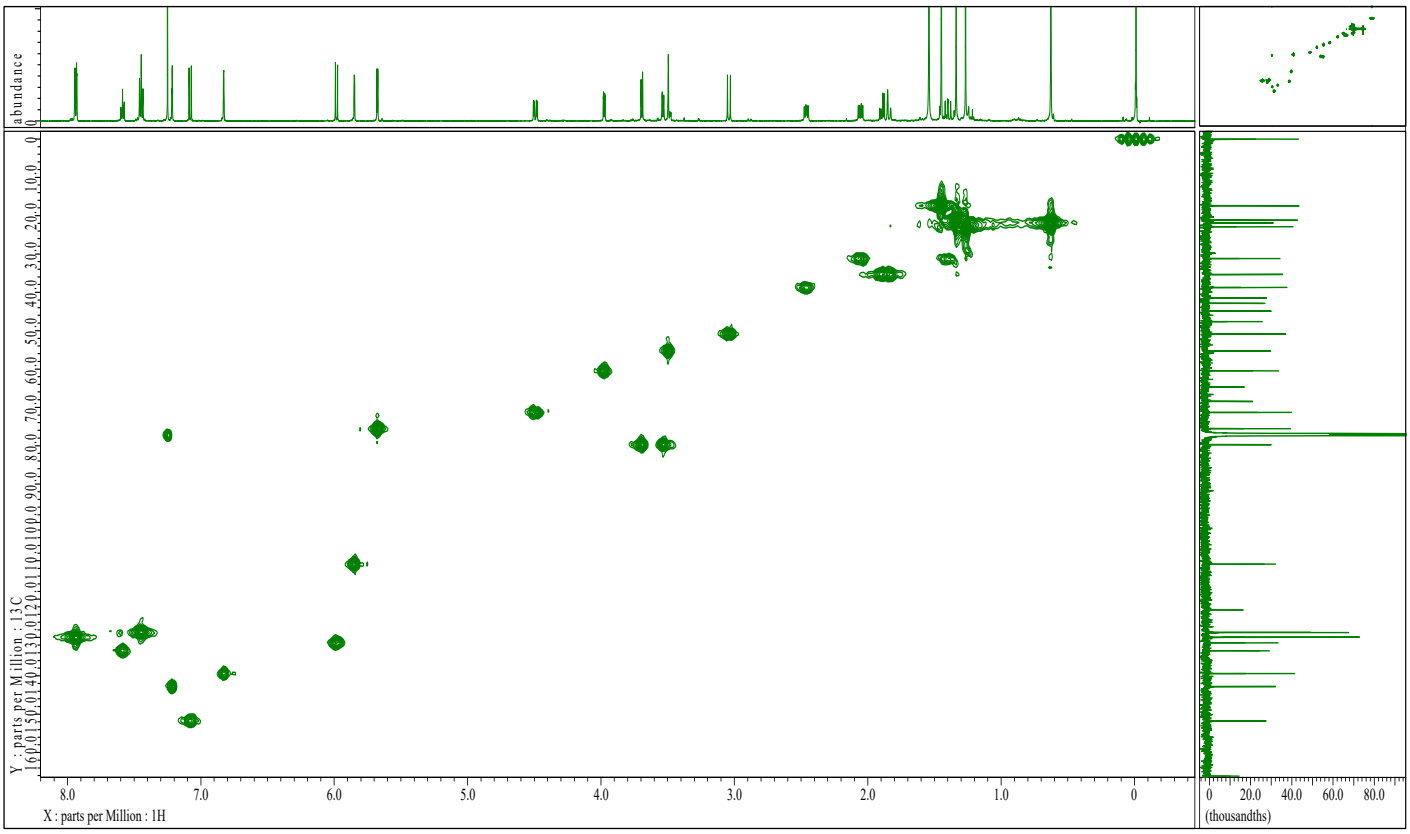


Figure S32. HMQC spectrum of compound **5** in $CDCl_3$ at 600MHz

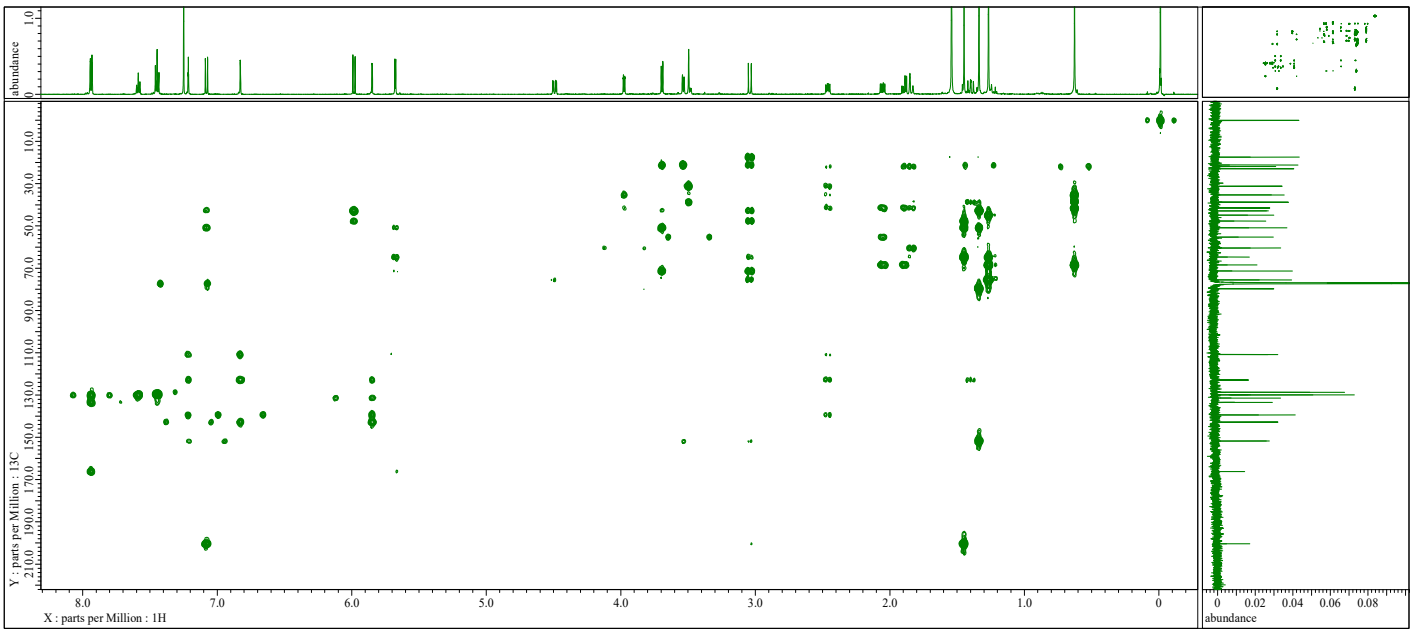


Figure S33. HMBC spectrum of compound **5** in $CDCl_3$ at 600MHz

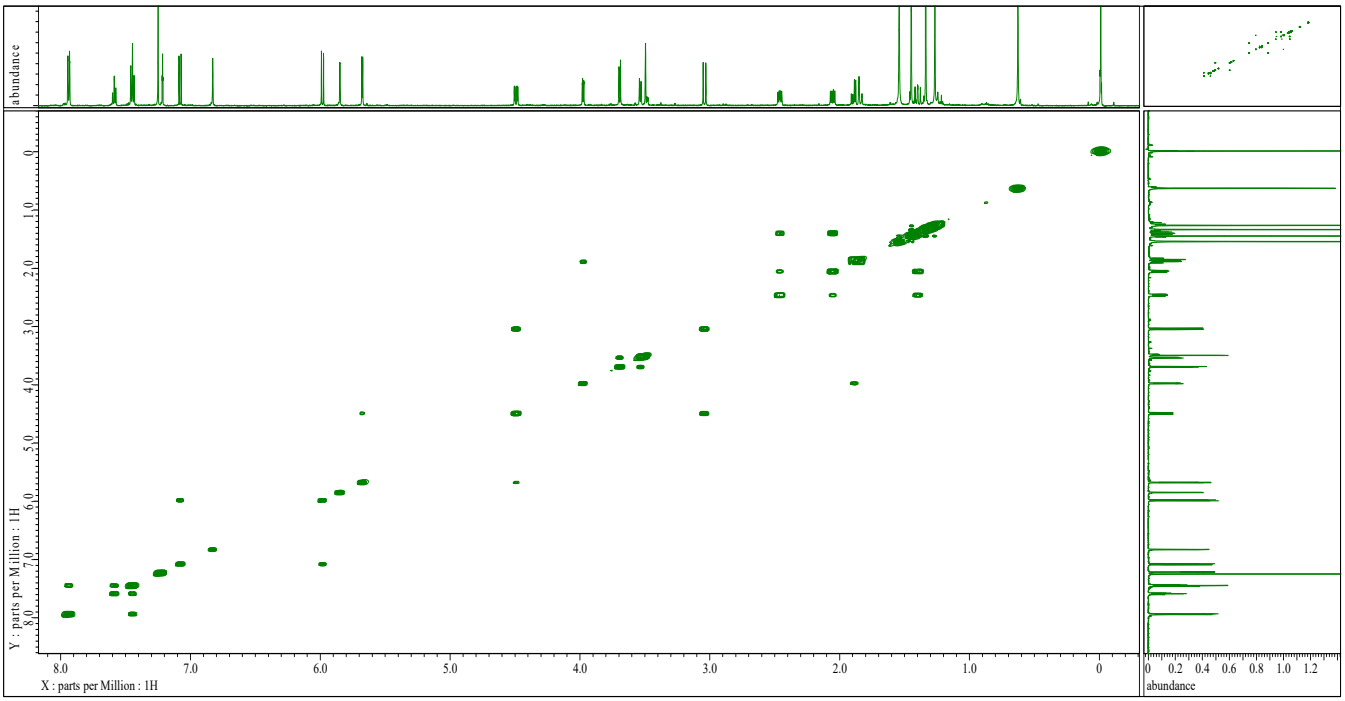


Figure S34. COSY spectrum of compound **5** in $CDCl_3$ at 600MHz

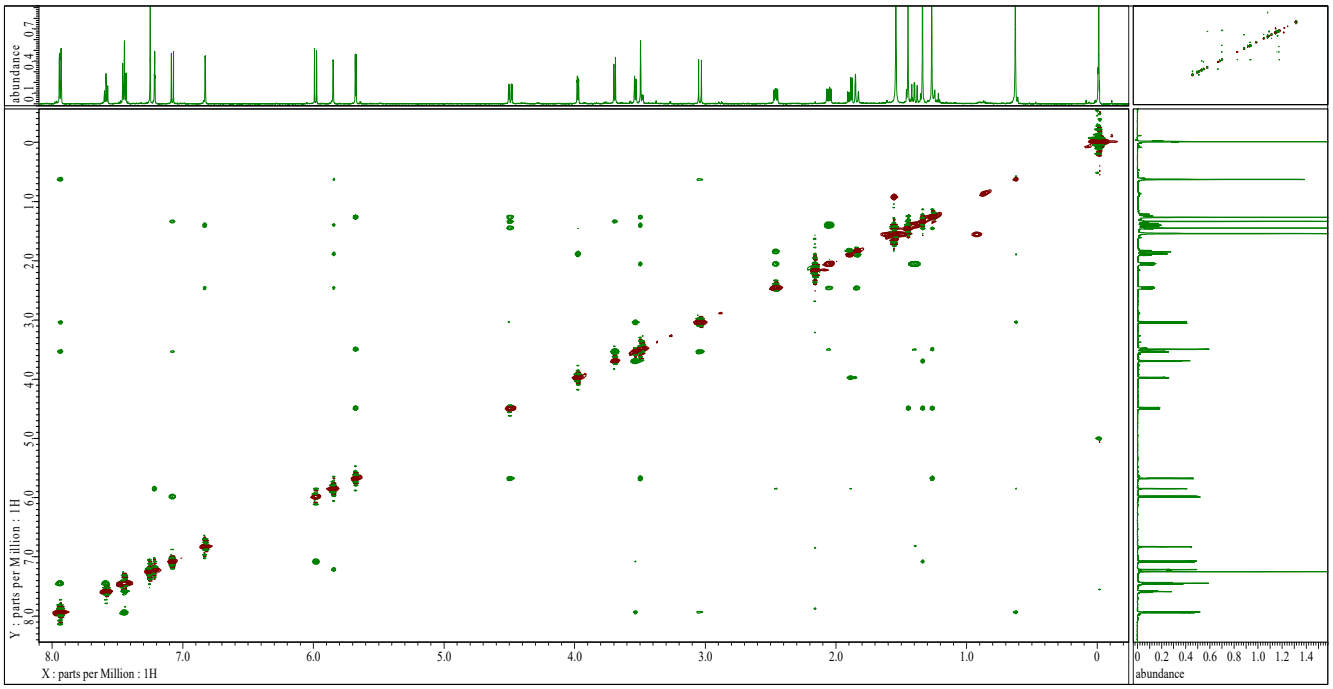


Figure S35. NOESY spectrum of compound **5** in $CDCl_3$ at 600MHz

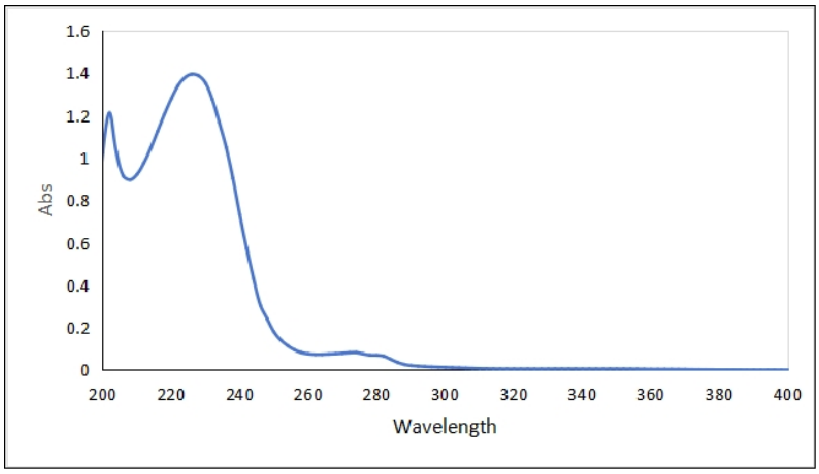


Figure S36. UV spectrum of compound **5** in MeOH

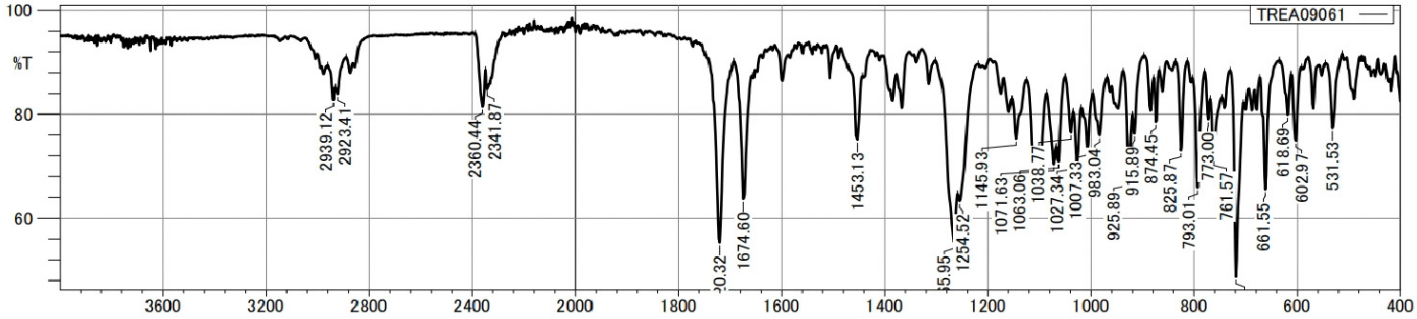


Figure S37. IR spectrum of compound **5**

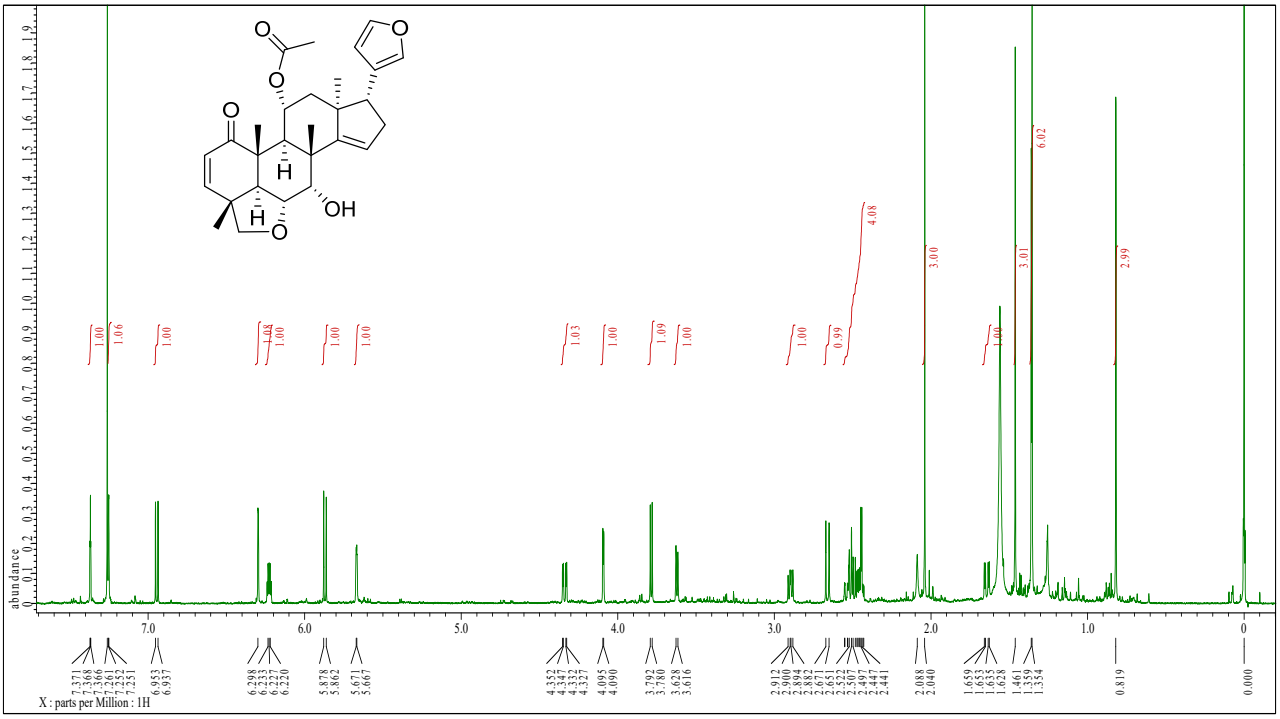


Figure S38. ^{1}H NMR spectrum of compound **6** in $CDCl_3$ at 600MHz

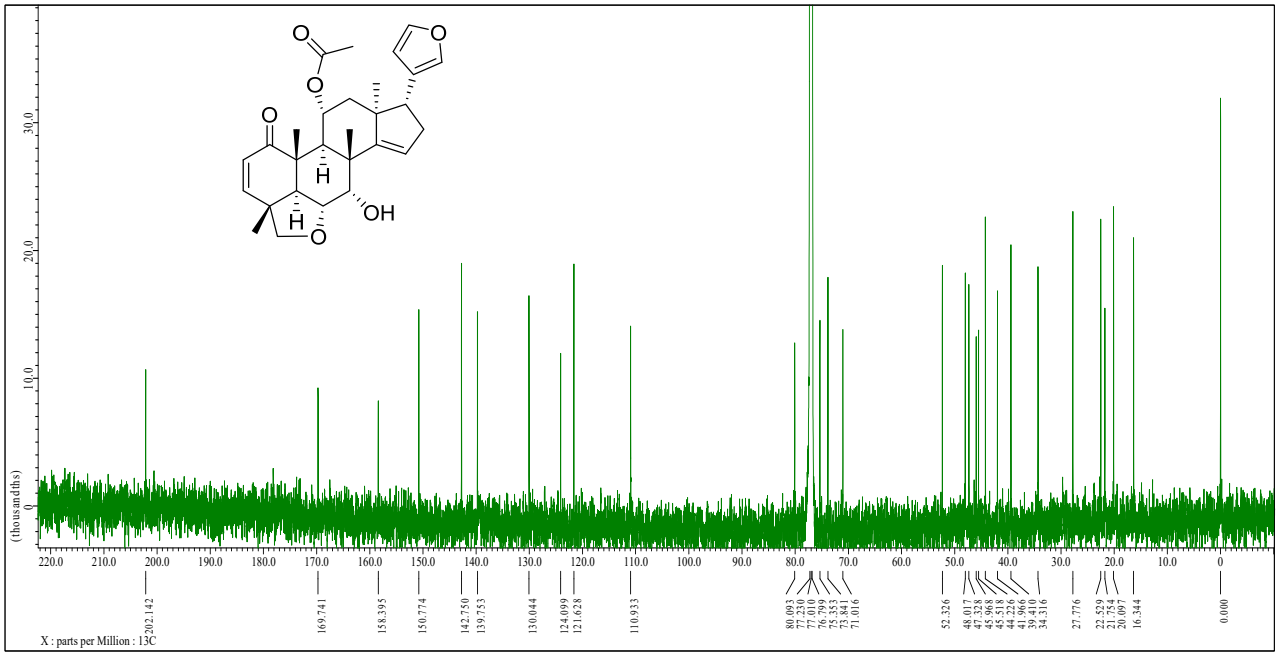


Figure S39.^{13}C NMR spectrum of compound **6** in $CDCl_3$ at 150MHz

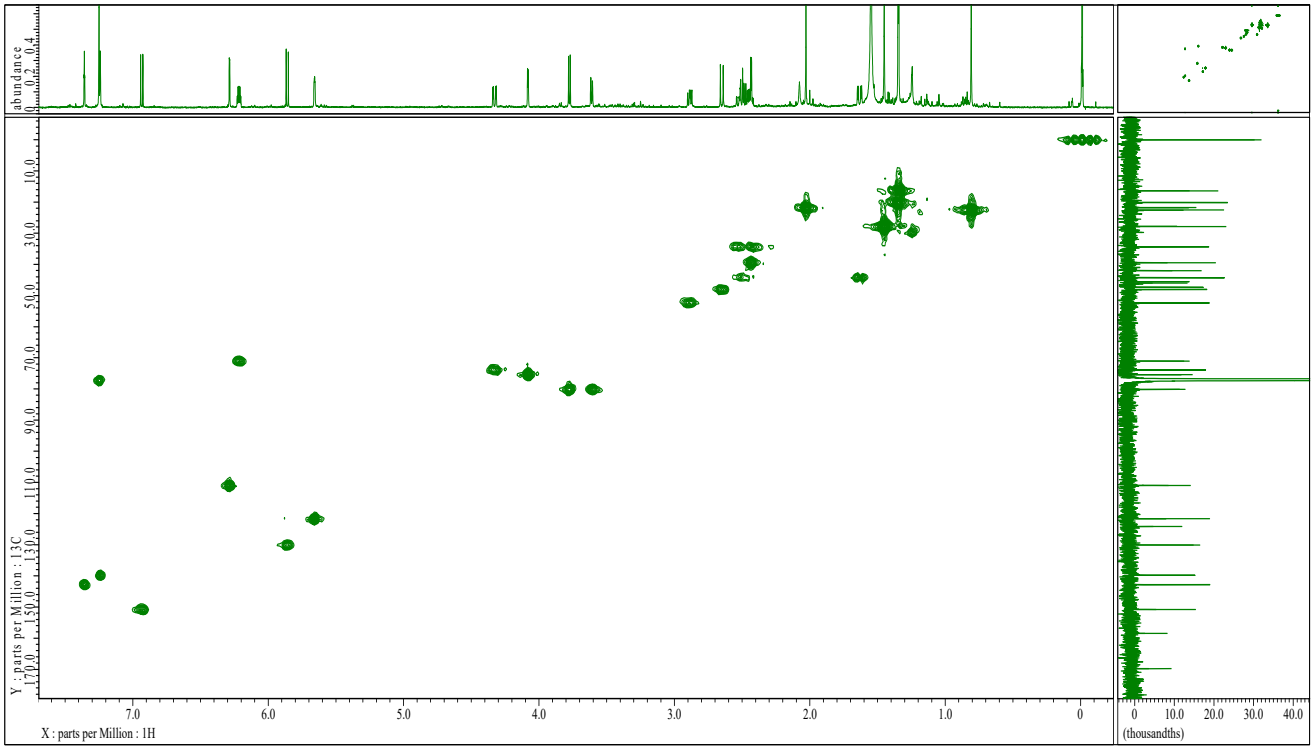


Figure S40. HMQC spectrum of compound **6** in $CDCl_3$ at 600MHz

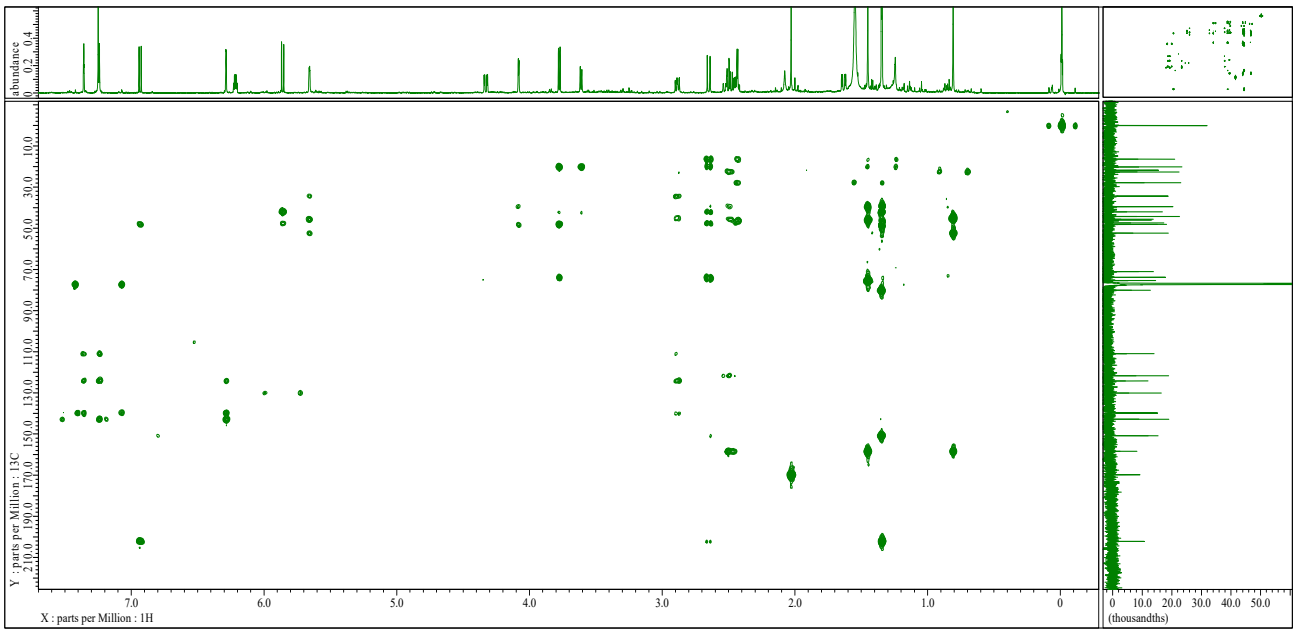


Figure S41. HMBC spectrum of compound **6** in $CDCl_3$ at 600MHz

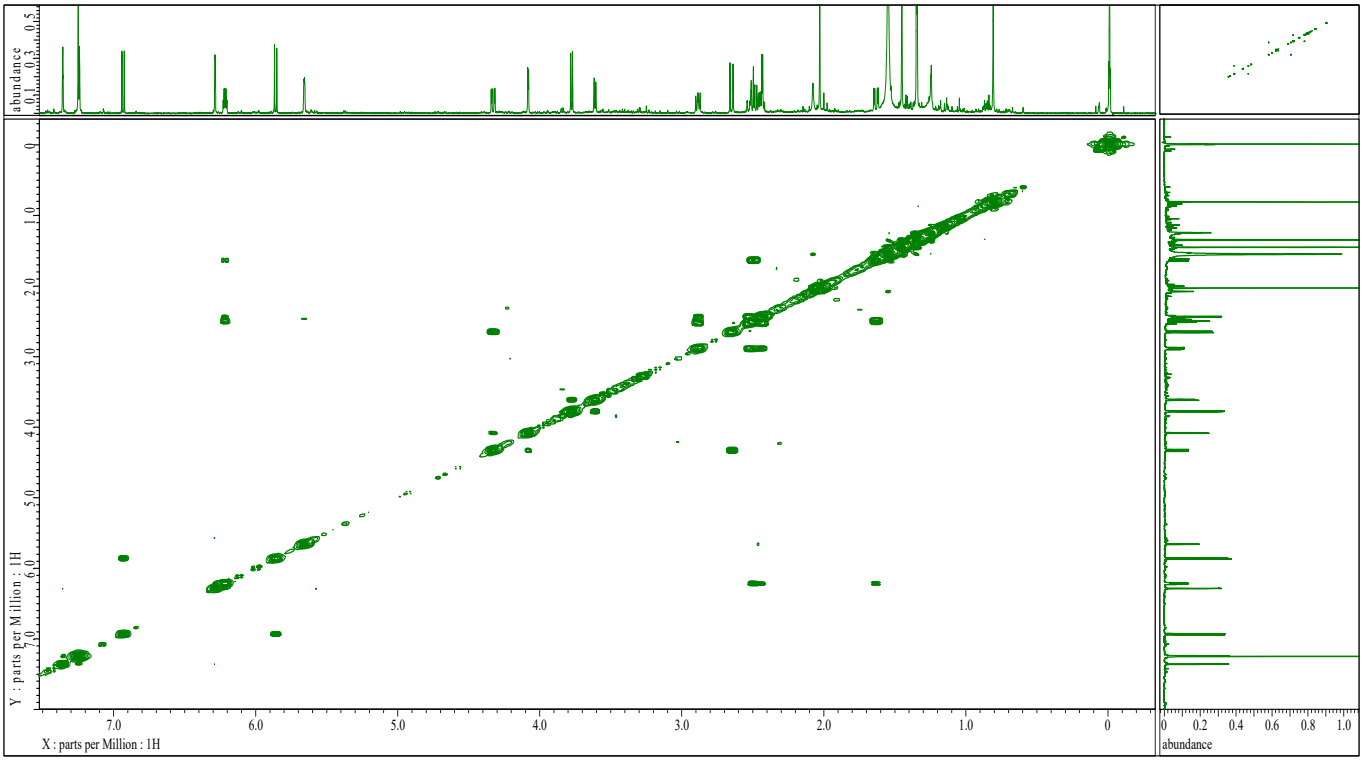


Figure S42. COSY spectrum of compound **6** in $CDCl_3$ at 600MHz

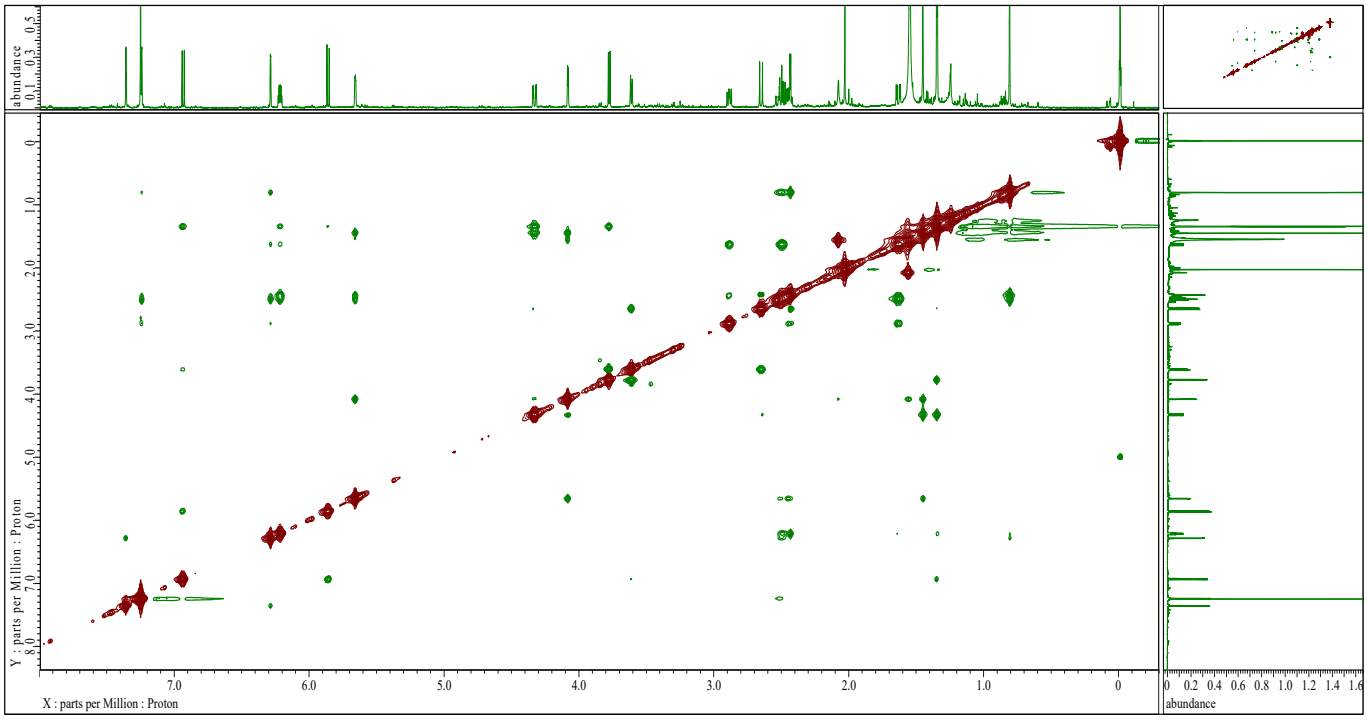


Figure S43. NOESY spectrum of compound **6** in $CDCl_3$ at 600MHz

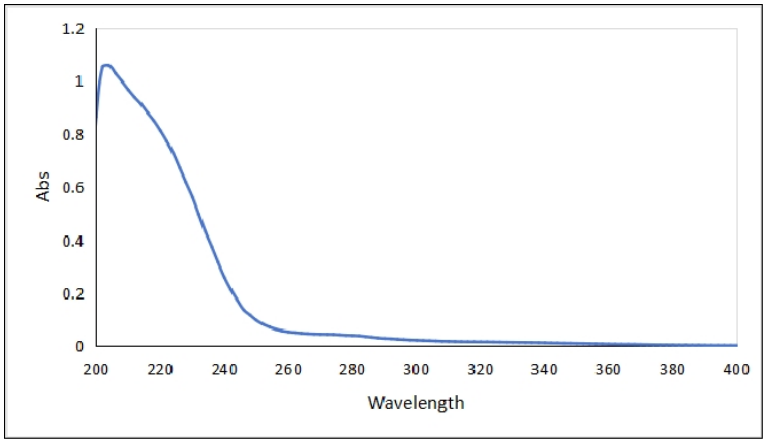


Figure S44. UV spectrum of compound **6** in MeOH

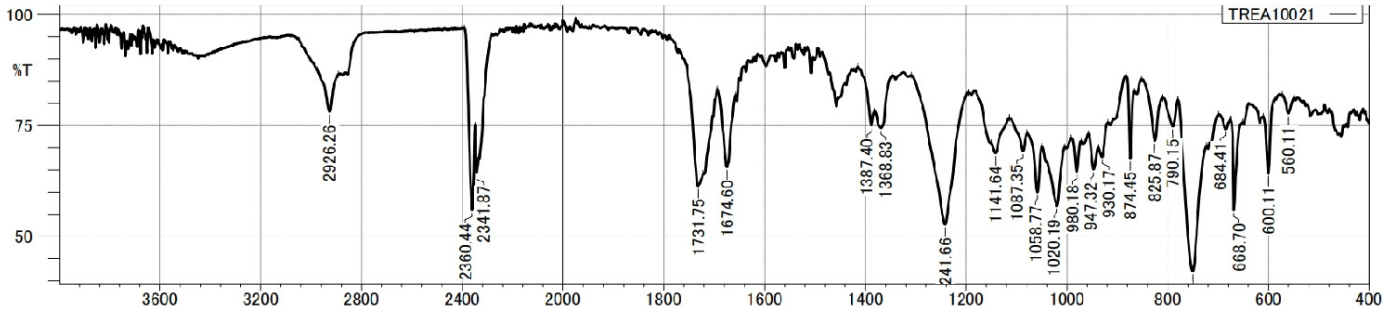


Figure S45. IR spectrum of compound **6**

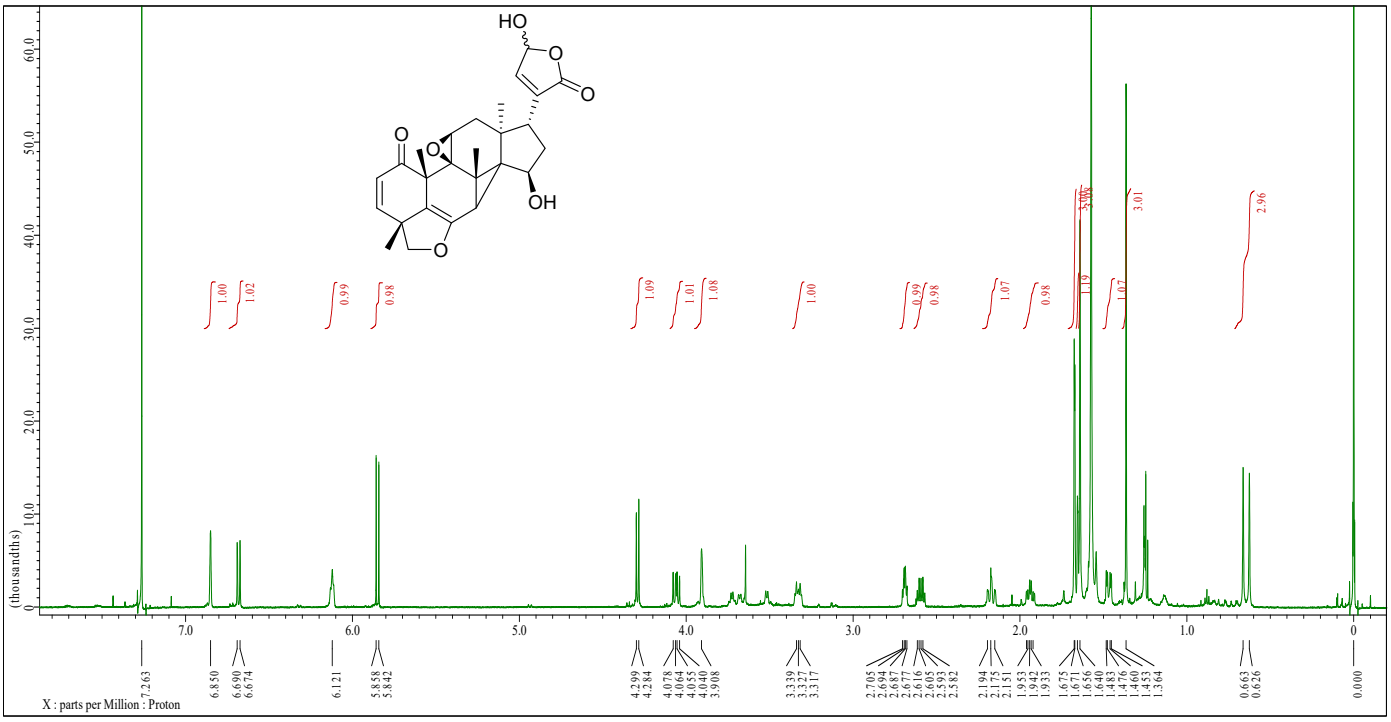


Figure S46. ^{1}H NMR spectrum of compound **7** in $CDCl_3$ at 600MHz

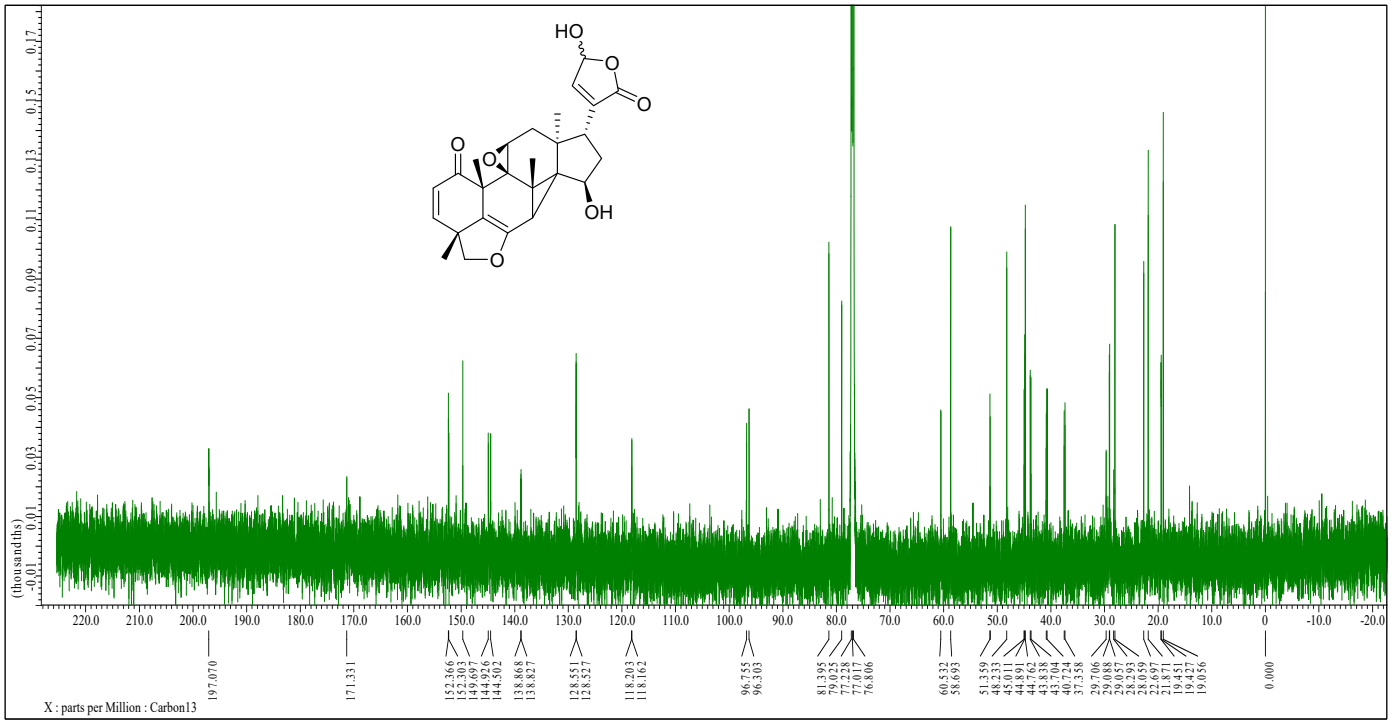


Figure S47.^{13}C NMR spectrum of compound **7** in $CDCl_3$ at 150MHz

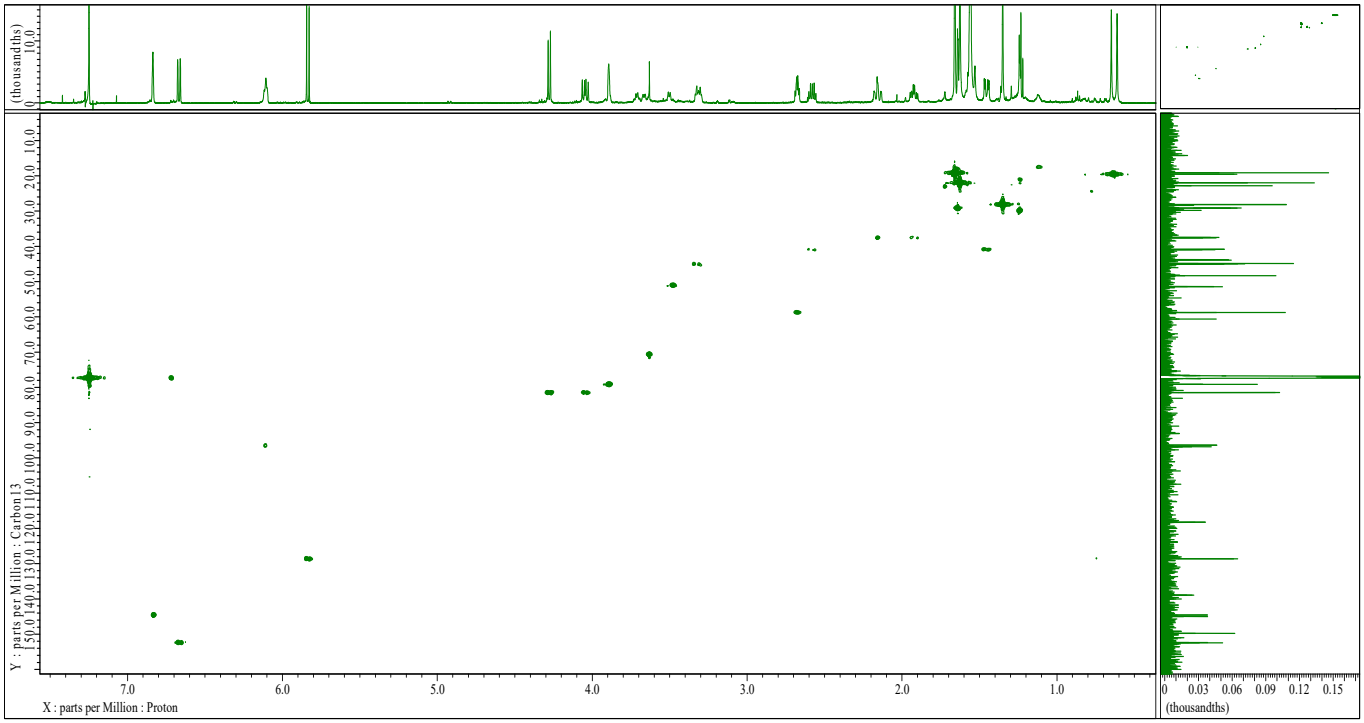


Figure S48. HMQC spectrum of compound **7** in $CDCl_3$ at 600MHz

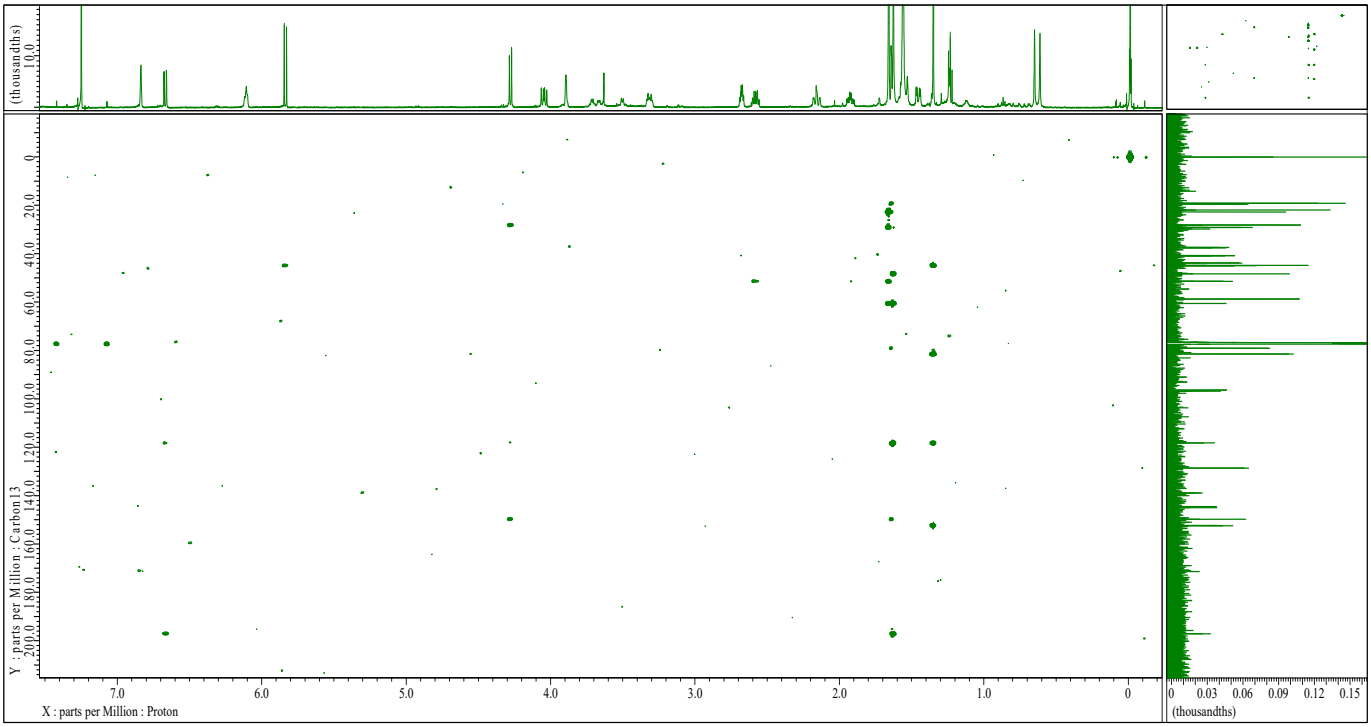


Figure S49. HMBC spectrum of compound **7** in $CDCl_3$ at 600MHz

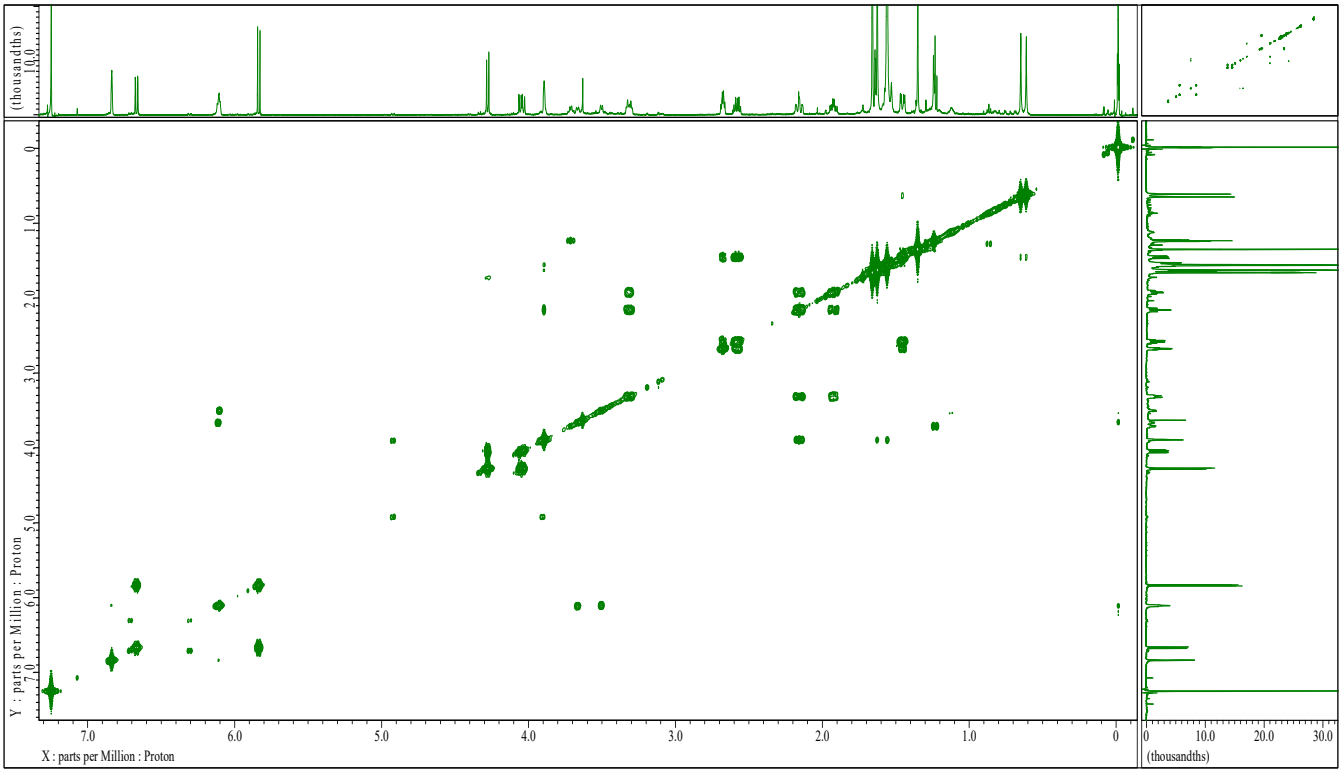


Figure S50. COSY spectrum of compound **7** in $CDCl_3$ at 600MHz

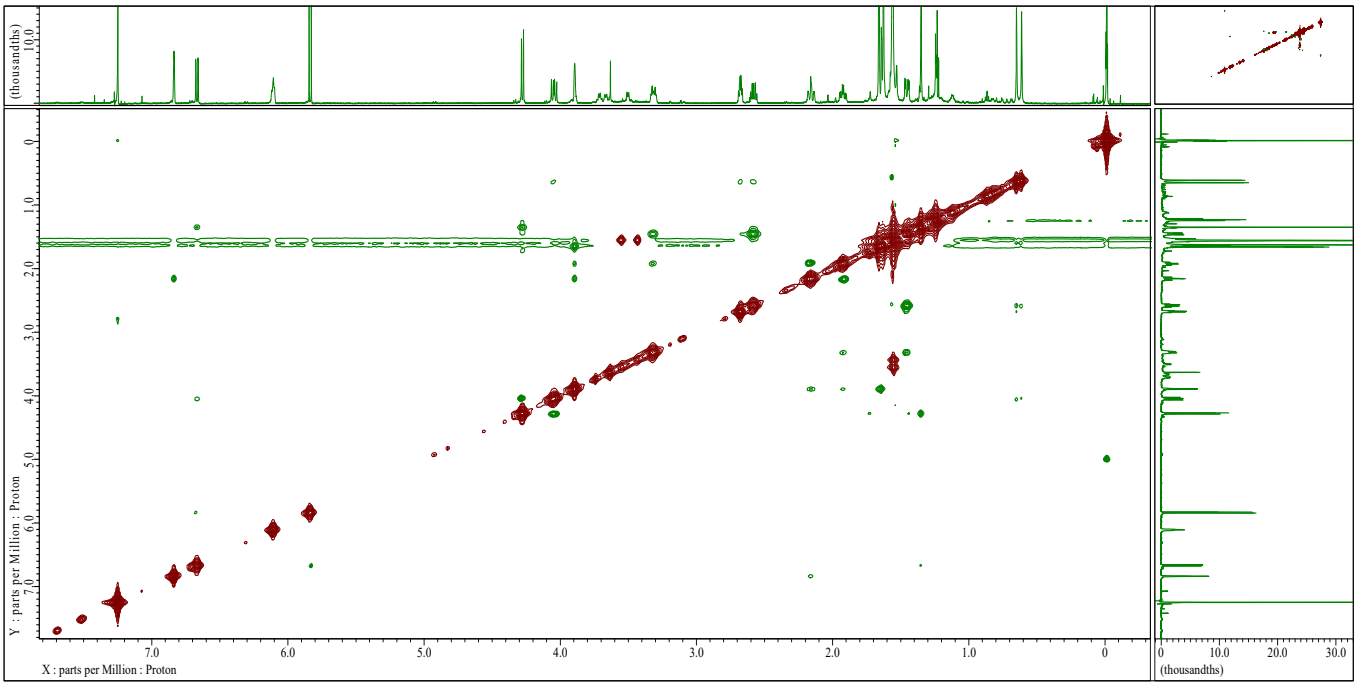


Figure S51. NOESY spectrum of compound **7** in $CDCl_3$ at 600MHz

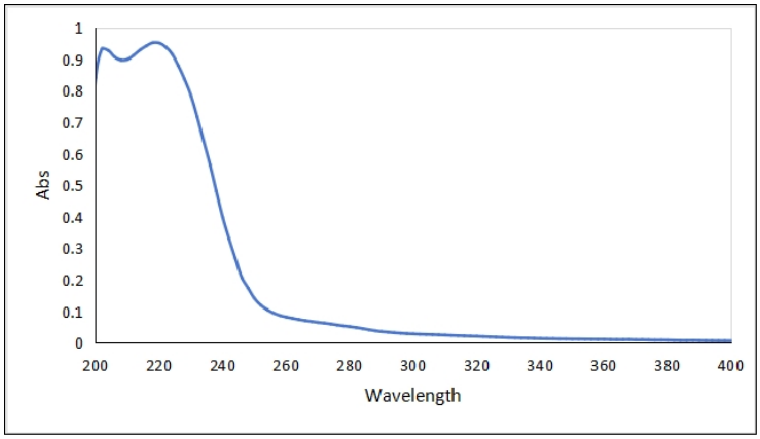


Figure S52. UV spectrum of compound **7** in MeOH

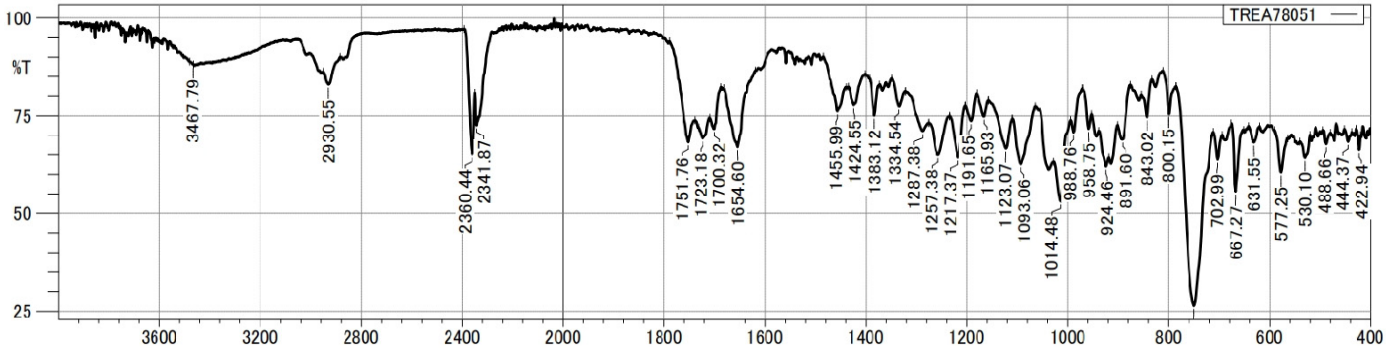


Figure S53. IR spectrum of compound **7**

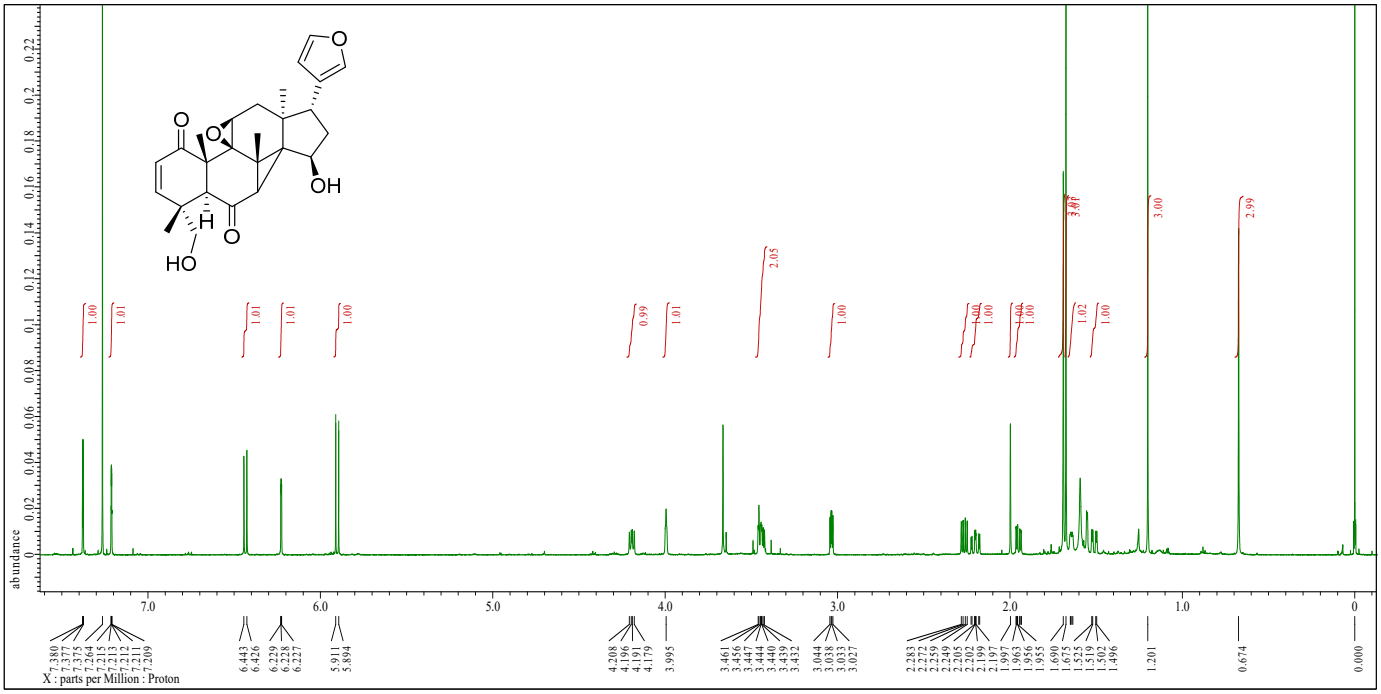


Figure S54. ^{1}H NMR spectrum of compound **8** in $CDCl_3$ at 600MHz

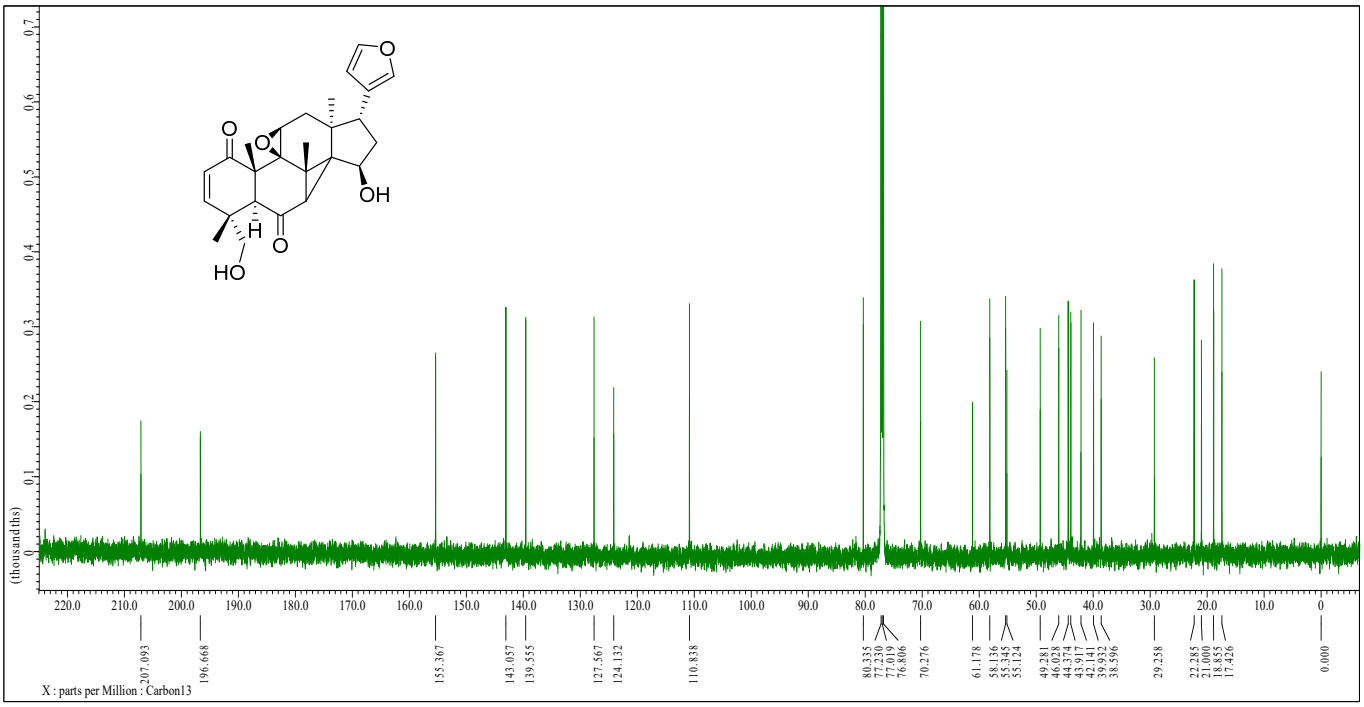


Figure S55.^{13}C NMR spectrum of compound **8** in $CDCl_3$ at 150MHz

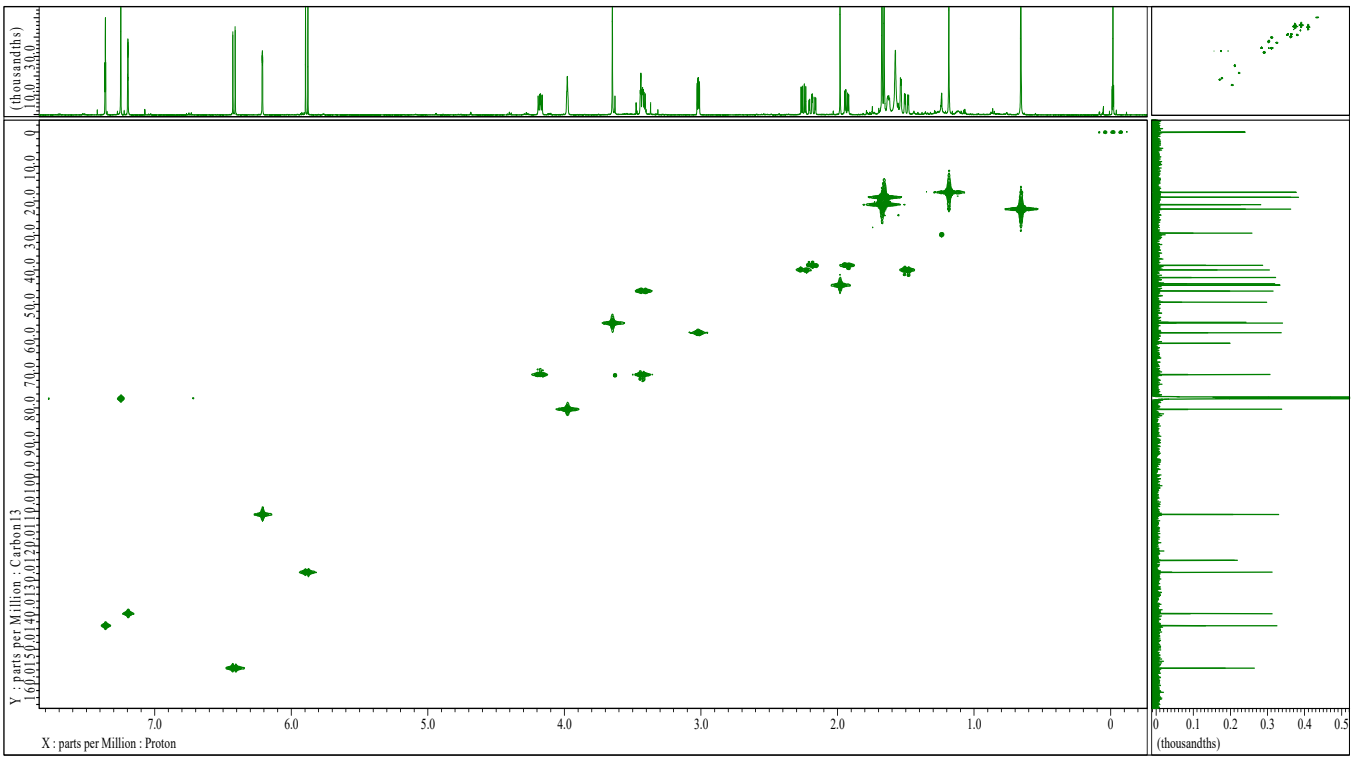


Figure S56. HMQC spectrum of compound **8** in $CDCl_3$ at 600MHz

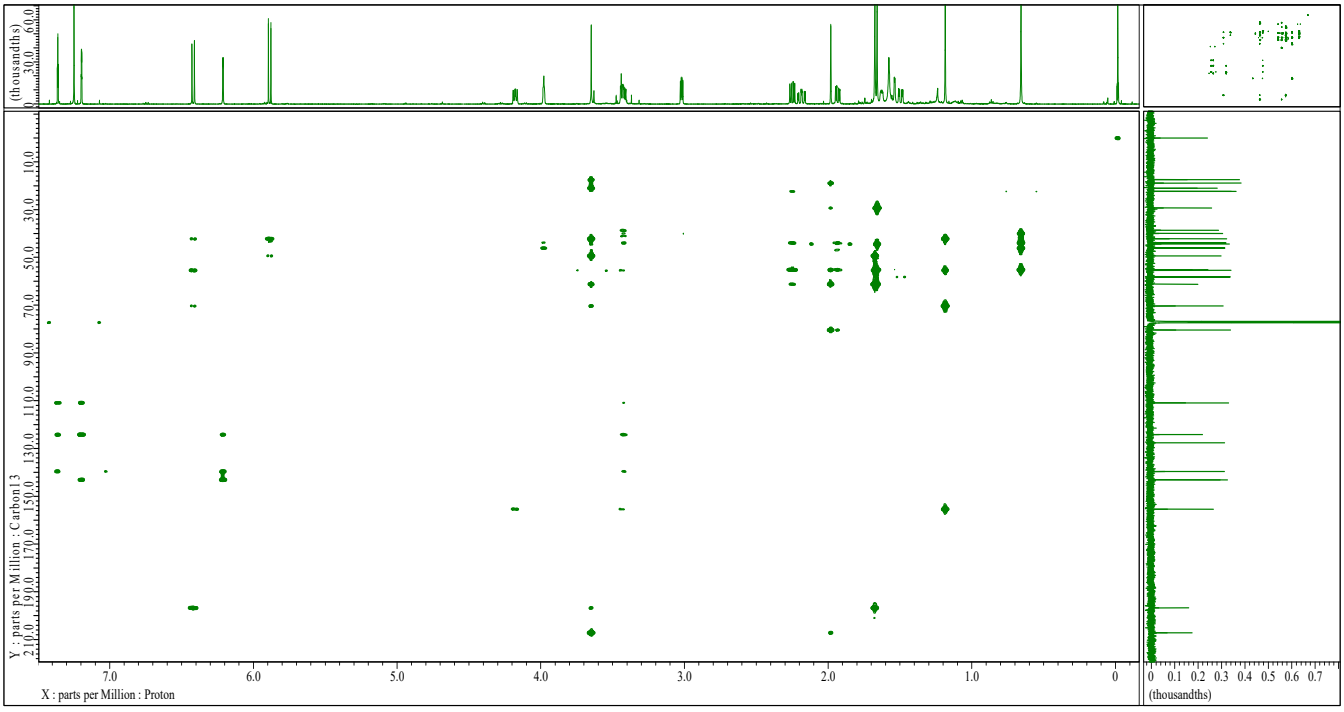


Figure S57. HMBC spectrum of compound **8** in $CDCl_3$ at 600MHz

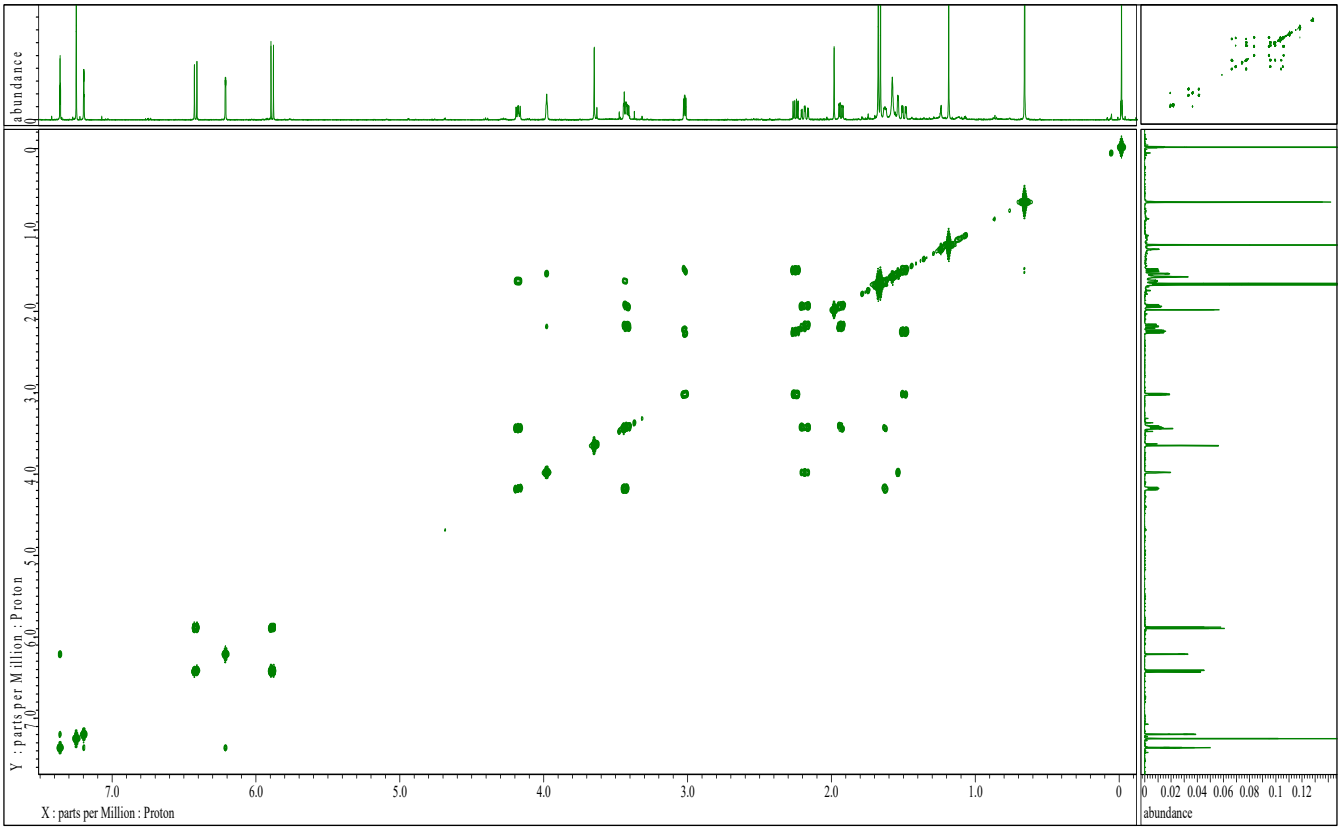


Figure S58. COSY spectrum of compound **8** in $CDCl_3$ at 600MHz

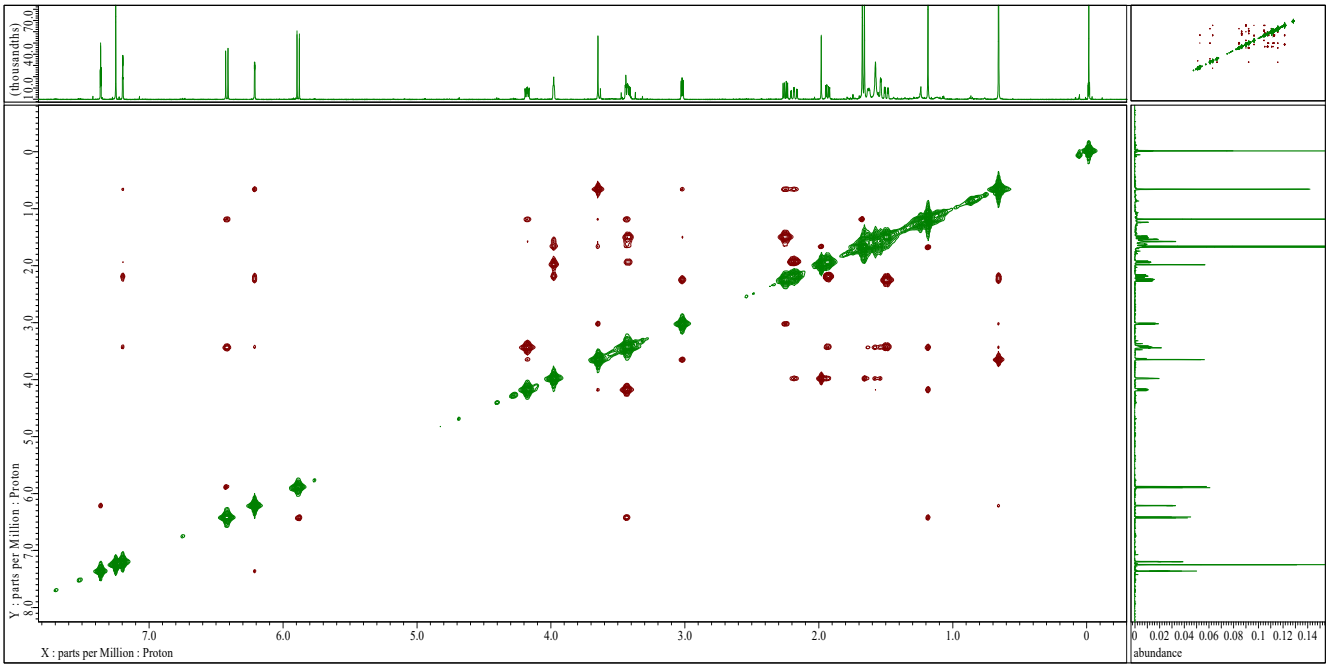


Figure S59. NOESY spectrum of compound **8** in $CDCl_3$ at 600MHz

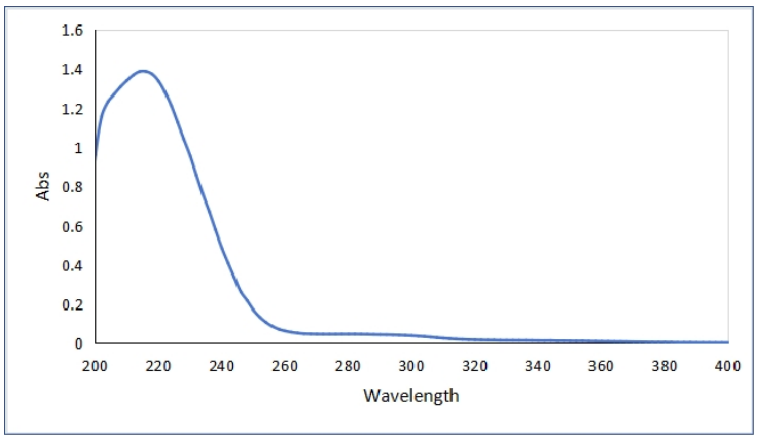


Figure S60. UV spectrum of compound **8** in MeOH

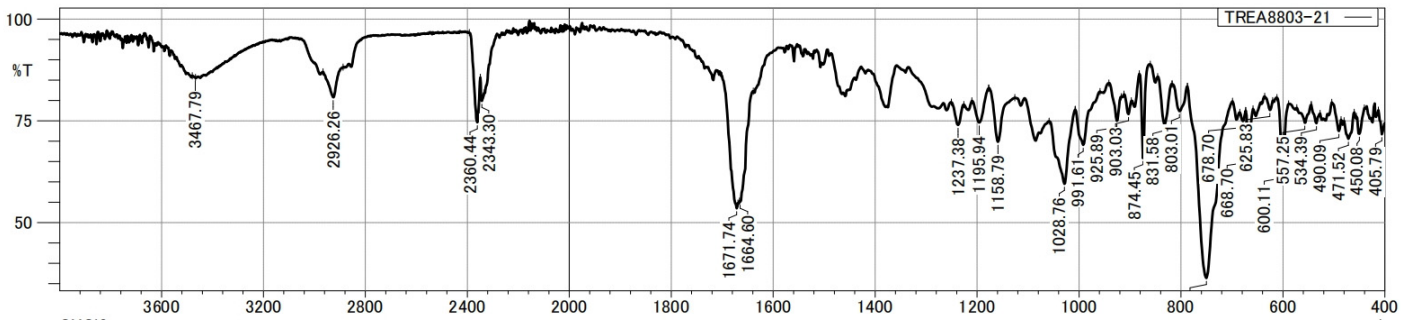


Figure S61. IR spectrum of compound **8**

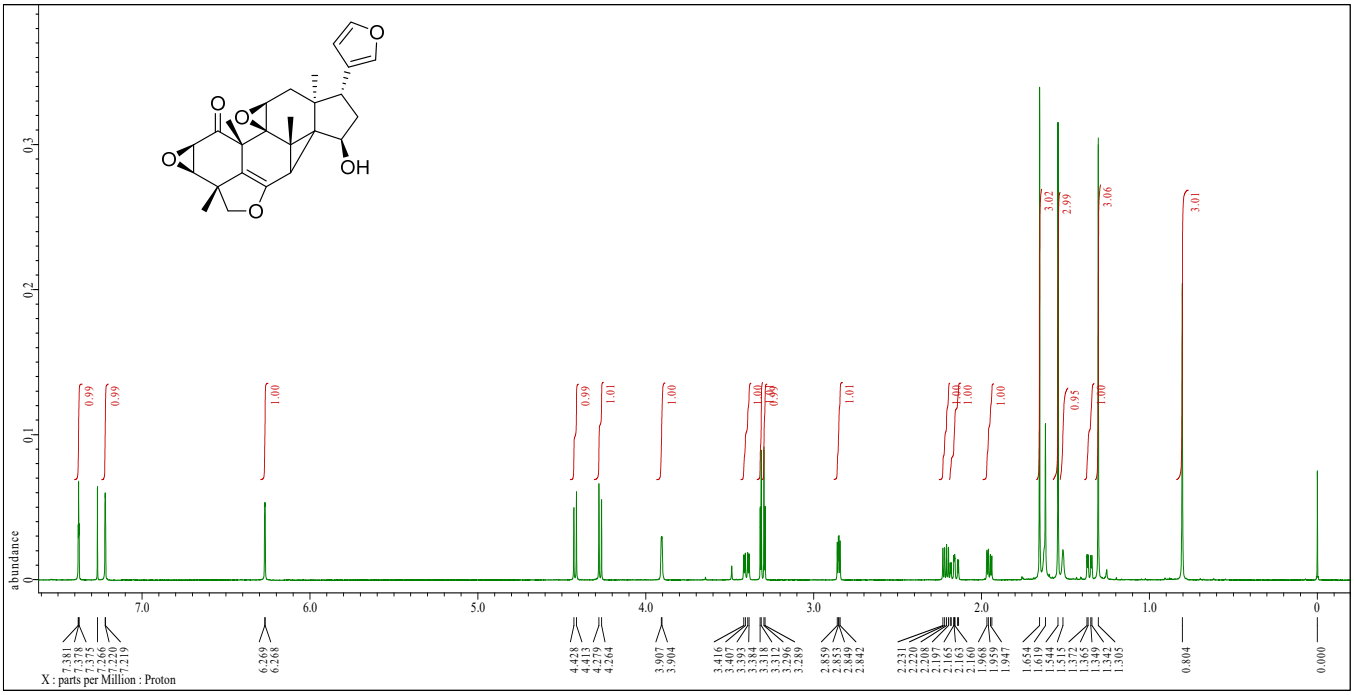


Figure S62. ^{1}H NMR spectrum of compound **17** in $CDCl_3$ at 600 MHz

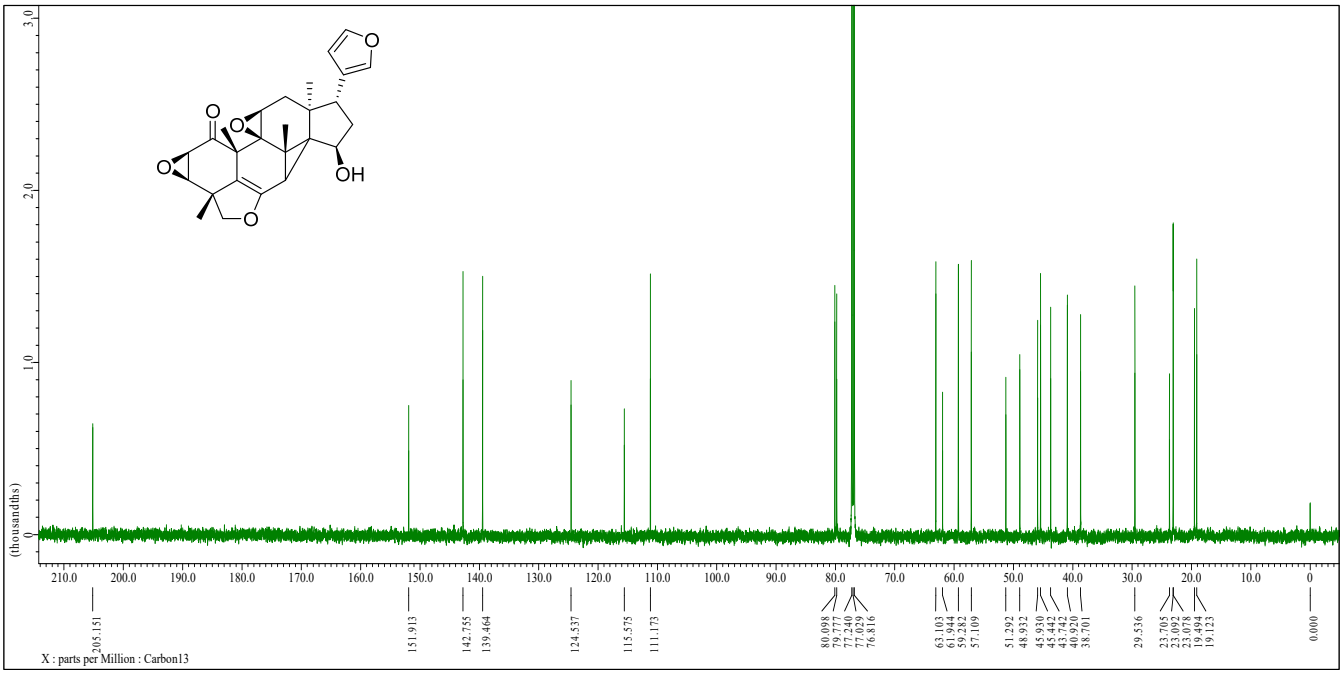


Figure S63.^{13}C NMR spectrum of compound **17** in $CDCl_3$ at 150 MHz

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 µM. A concentration of the fraction 7.2 was also prepared to 10 µM 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 µm, 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 µL. 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.

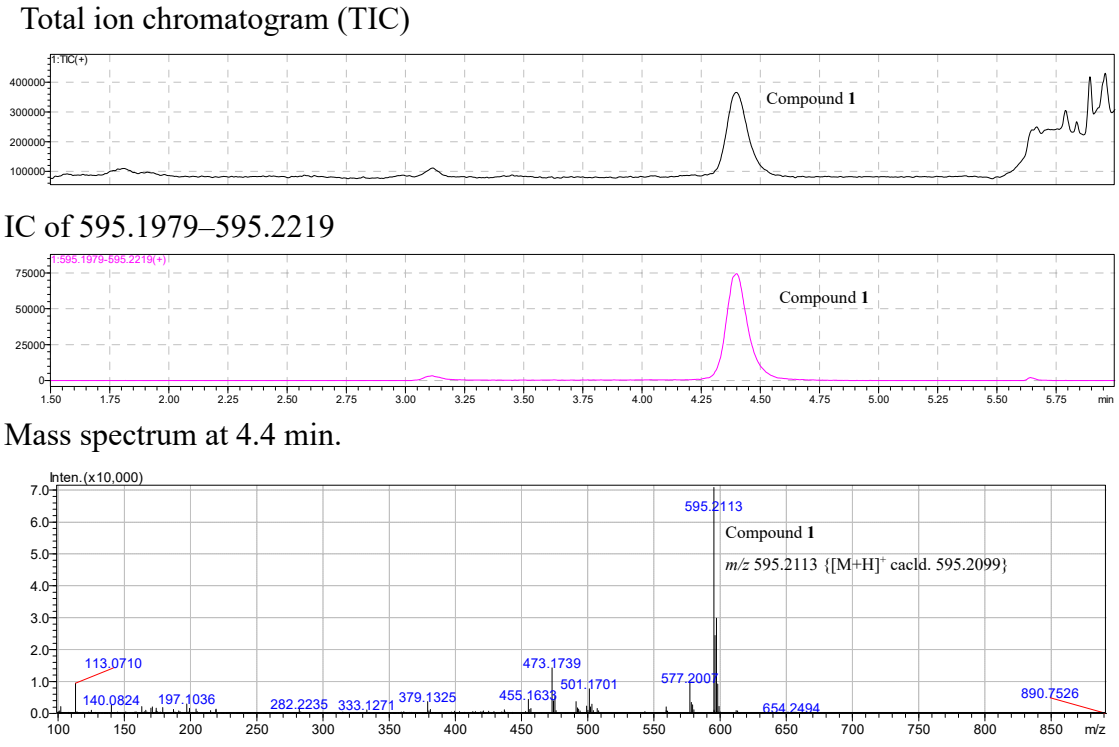
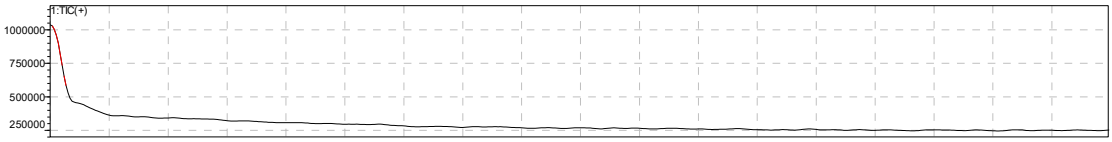
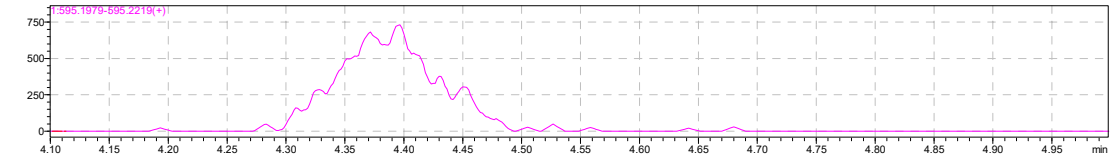


Figure S64. LC/MS analysis of compound **1**

TIC



IC of 595.1979–595.2219



Mass spectrum at 4.4 min.

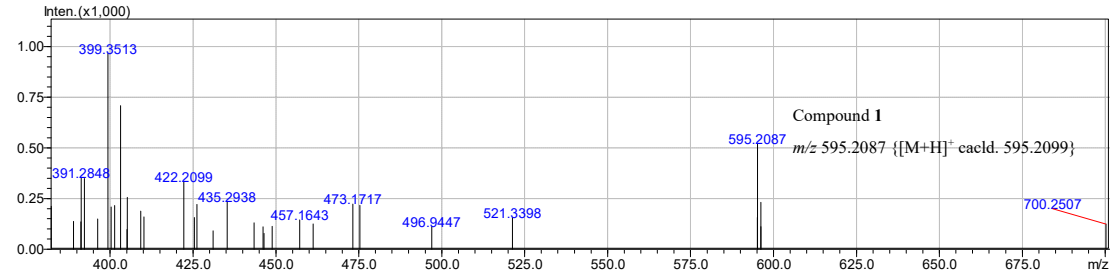
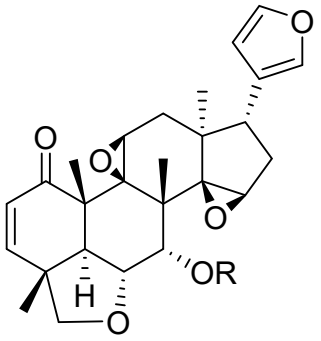


Figure S65. LC/MS analysis of fraction 7.2



TS1 R= H

TS2 R=

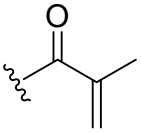


Figure S66. Structures of TS1 and TS2