

Supplementary Information

Antimicrobial Activity of 3,4-*seco*-Diterpenes Isolated from *Croton blanchetianus* against *Streptococcus mutans* and *Streptococcus parasanguinis*

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Table S1. Fractions collected by selective desorption chromatography of NFCB1, NFCB4 and NFCB8

Fraction	Eluent	Mass / mg		
		Specimen 1	Specimen 4	Specimen 8
NFCBx-H1	hexane	106.2	104.0	72.6
NFCBx-H2	hexane	1.1	0.5	135.8
NFCBx-HD25-1	Hex-CH ₂ Cl ₂ 25%	487.5	485.2	11.8
NFCBx-HD25-2	Hex-CH ₂ Cl ₂ 25%	51.3	123.3	111.3
NFCBx-HD50-1	Hex-CH ₂ Cl ₂ 50%	287.3	329.3	329.9
NFCBx-HD50-2	Hex-CH ₂ Cl ₂ 50%	196.2	222.5	208.6
NFCBx-HD50-3	Hex-CH ₂ Cl ₂ 50%	—	—	318.0
NFCBx-D1	CH ₂ Cl ₂	578.2	398.3	416.1
NFCBx-D2	CH ₂ Cl ₂	276.7	354.0	339.7
NFCBx-D3	CH ₂ Cl ₂	—	—	216.3
NFCBx-DA25-1	CH ₂ Cl ₂ -AcOEt 25%	438.0	471.3	928.3
NFCBx-DA25-2	CH ₂ Cl ₂ -AcOEt 25%	49.1	54.2	1,049.6
NFCBx-DA25-3	CH ₂ Cl ₂ -AcOEt 25%	—	—	285.1
NFCBx-DA50-1	CH ₂ Cl ₂ -AcOEt 50%	—	—	283.8
NFCBx-DA50-2	CH ₂ Cl ₂ -AcOEt 50%	—	—	110.6
NFCBx-A1	AcOEt	65.8	64.0	55.1
NFCBx-A1	AcOEt	—	—	44.2
NFCBx-AM1	AcOEt-MeOH 50%	62.4	86.5	197.2
Total / g		2,599.8	2,693.1	5,114.0
Yield / %		98.24	97.19	97.94

In the notations of fractions, we have x = 1 for specimen 1, x = 4 for specimen 4, and x = 8 for specimen 8.

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Table S2. Fractions collected by selective desorption chromatography of AFCB1, AFCB4 and AFCB8

Fraction	Eluent	Mass / mg		
		Specimen 1	Specimen 4	Specimen 8
AFCBx-H1	hexane	1.4	2.6	2.9
AFCBx-HD50-1	Hex-CH ₂ Cl ₂ 50%	2,574.0	1,419.6	1,019.3
AFCBx-HD50-2	Hex-CH ₂ Cl ₂ 50%	961.9	1,271.2	777.1
AFCBx-D1	CH ₂ Cl ₂	838.1	1,382.9	597.5
AFCBx-D2	CH ₂ Cl ₂	308.2	605.1	139.5
AFCBx-D3	CH ₂ Cl ₂	148.6	259.3	27.5
AFCBx-DA25-1	CH ₂ Cl ₂ -AcOEt 25%	1,308.3	1,257.0	455.5
AFCBx-DA25-2	CH ₂ Cl ₂ -AcOEt 25%	38.2	40.8	41.6
AFCBx-A1	AcOEt	9.1	12.8	127.4
AFCBx-AM1	AcOEt-MeOH 50%	166.2	218.3	370.1
AFCBx-M1	MeOH	17.3	14.5	15.0
Total / mg		6,371.3	6,484.1	3,573.4
Yield / %		97.57	94.61	98.05

In the notations of fractions, we have x = 1 for specimen 1, for specimen 4, x = 4 and for specimen 8, x = 8.

Table S3. MIC and MBC values of the neutral fractions of CB1, CB4 and CB8 against *S. mutans* and *S. parasanguinis*

Neutral fraction	<i>S. mutans</i>		<i>S. parasanguinis</i>	
	MIC / (μg mL ⁻¹)	MBC / (μg mL ⁻¹)	MIC / (μg mL ⁻¹)	MBC / (μg mL ⁻¹)
NFCB1-H1	–	–	–	–
NFCB1-HD25/1	250	–	–	–
NCB1-HD50/1	62.5	–	500	–
NFCB1-HD50/2	500	–	–	–
NFCB1-D1	250	–	–	–
NFCB1-D2	62.5	–	62.5	125
NFCB1-DA25/1	–	–	–	–
NFCB1-A1	–	–	–	–
NFCB1-AM1	500	–	250	–
NFCB4-H1	–	–	–	–
NFCB4-HD25/1	–	–	500	–
NFCB4-HD50/1	62.5	–	500	–
NFCB4-HD50/2	125	–	250	–
NFCB4-D1	125	–	–	–
NFCB4-D2	250	–	500	–
NFCB4-DA25/1	–	–	–	–
NFCB4-A1	–	–	–	–
NFCB4-AM1	–	–	250	–

Table S3. MIC and MBC values of the neutral fractions of CB1, CB4 and CB8 against *S. mutans* and *S. parasanguinis* (cont.)

Neutral fraction	<i>S. mutans</i>		<i>S. parasanguinis</i>	
	MIC / ($\mu\text{g mL}^{-1}$)	MBC / ($\mu\text{g mL}^{-1}$)	MIC / ($\mu\text{g mL}^{-1}$)	MBC / ($\mu\text{g mL}^{-1}$)
NFCB8-H1	–	–	–	–
NFCB8-HD25/2	500	–	–	–
NFCB8-HD50/1	500	–	500	–
NFCB8-HD50/2	62.5	–	500	500
NFCB8-D1	125	–	500	500
NFCB8-D2	500	–	500	–
NFCB8-DA25/1	125	500	250	250
NFCB8-A1	500	–	–	–
NFCB8-AM	500	–	250	–

NFCBx: neutral fractions of the *C. blanchetianus*. H1: hexane; HD25/1: Hex/CH₂Cl₂ 25%; HD50/1 and HD50/2: Hex/CH₂Cl₂ 50%; D1 and D2: CH₂Cl₂; DA25/1: CH₂Cl₂/AcOEt 25%; A1: AcOEt; AM: AcOEt/MeOH; x = 1 for specimen 1, x = 4 for specimen 4 and x = 8 for specimen 8.

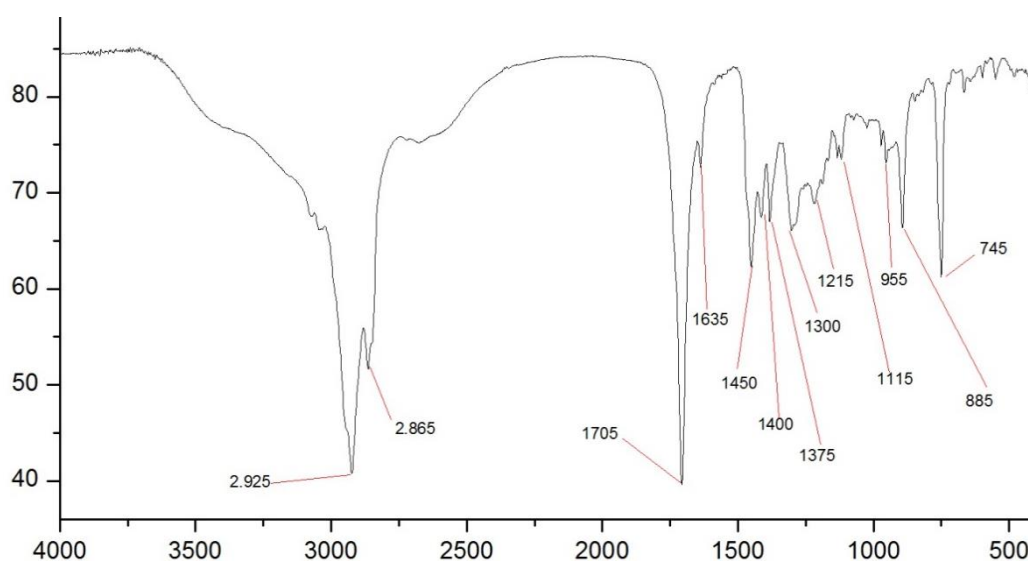


Figure S1. Infrared (UATR) spectrum of compound ICB1.



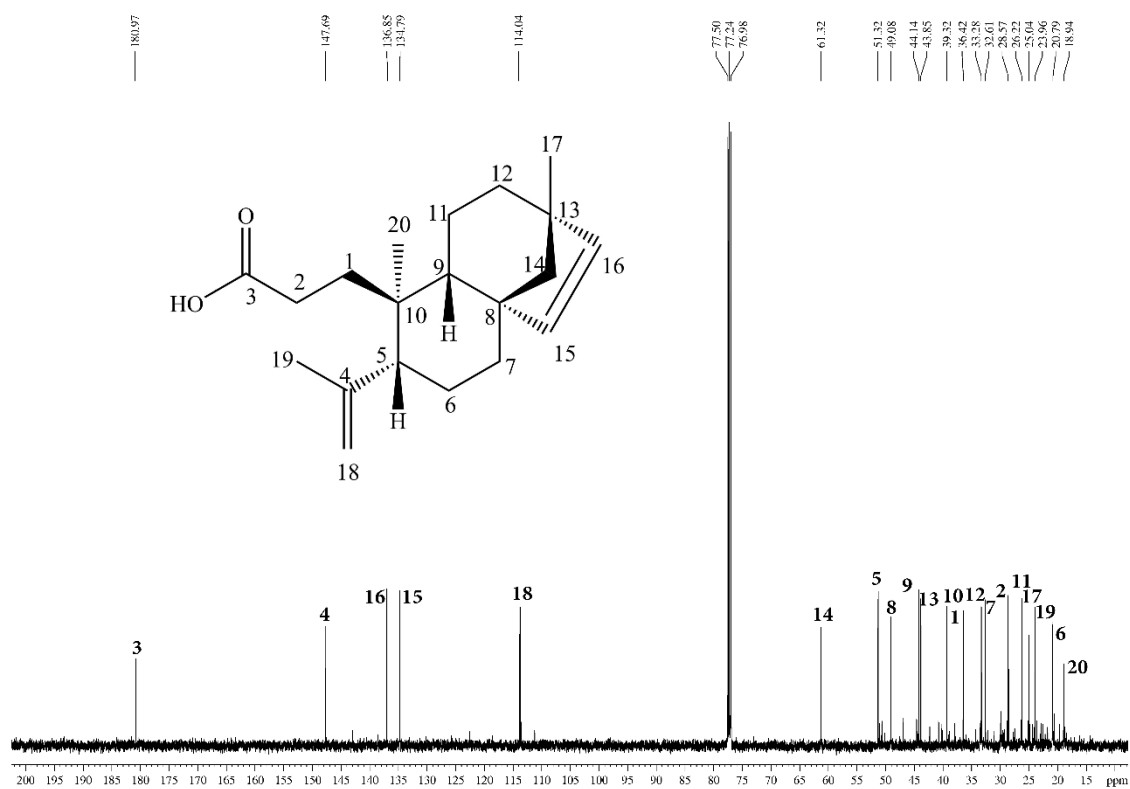


Figure S4. ^{13}C NMR spectrum (125 MHz, CDCl_3) of compound ICB1.

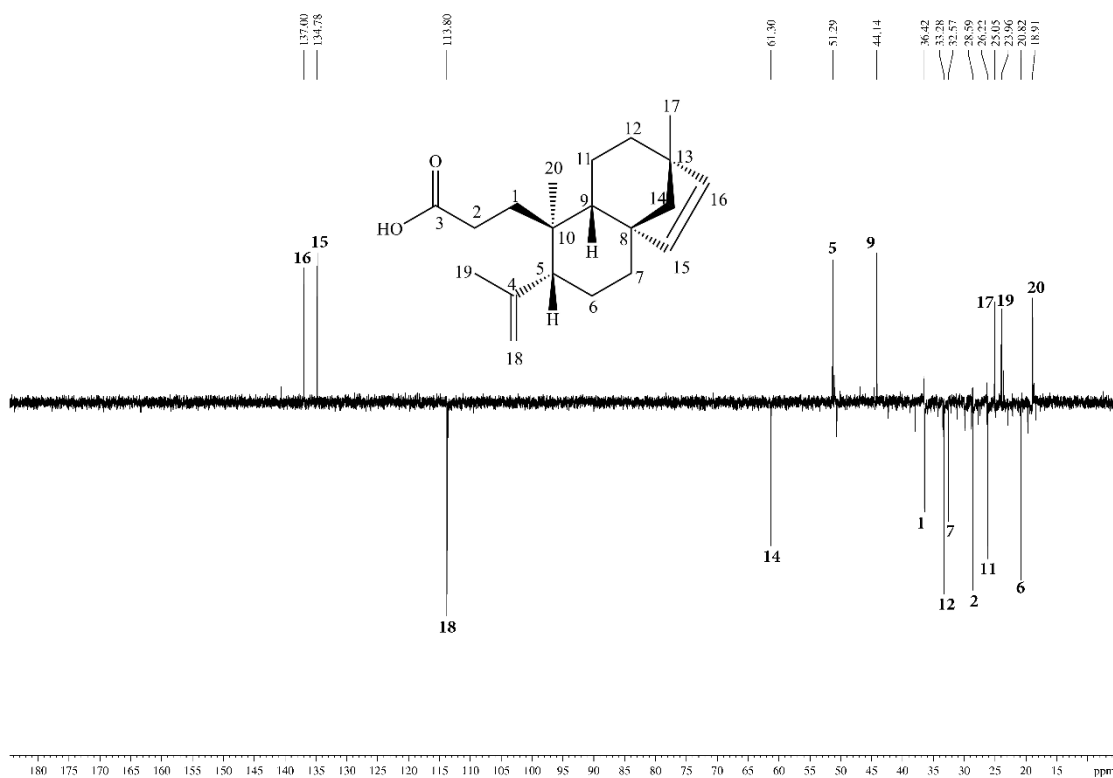


Figure S5. DEPT 135° NMR spectrum (125 MHz, CDCl_3) of compound ICB1.

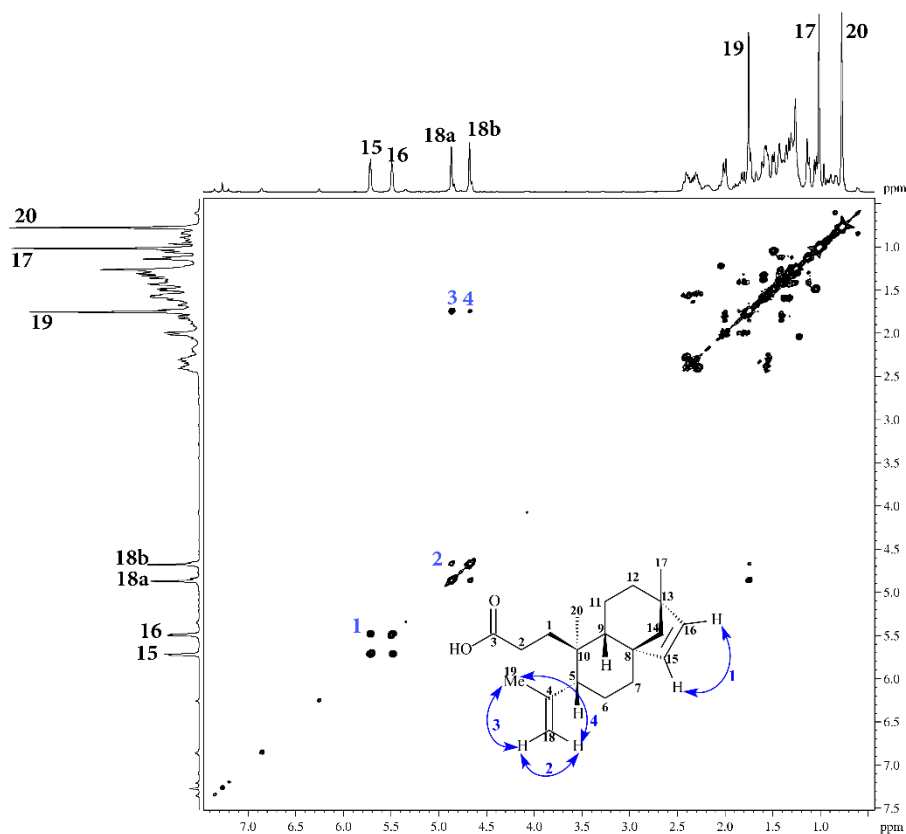


Figure S6. ^1H , ^1H COSY-NMR spectrum (500×500 MHz, CDCl_3) of compound ICB1.

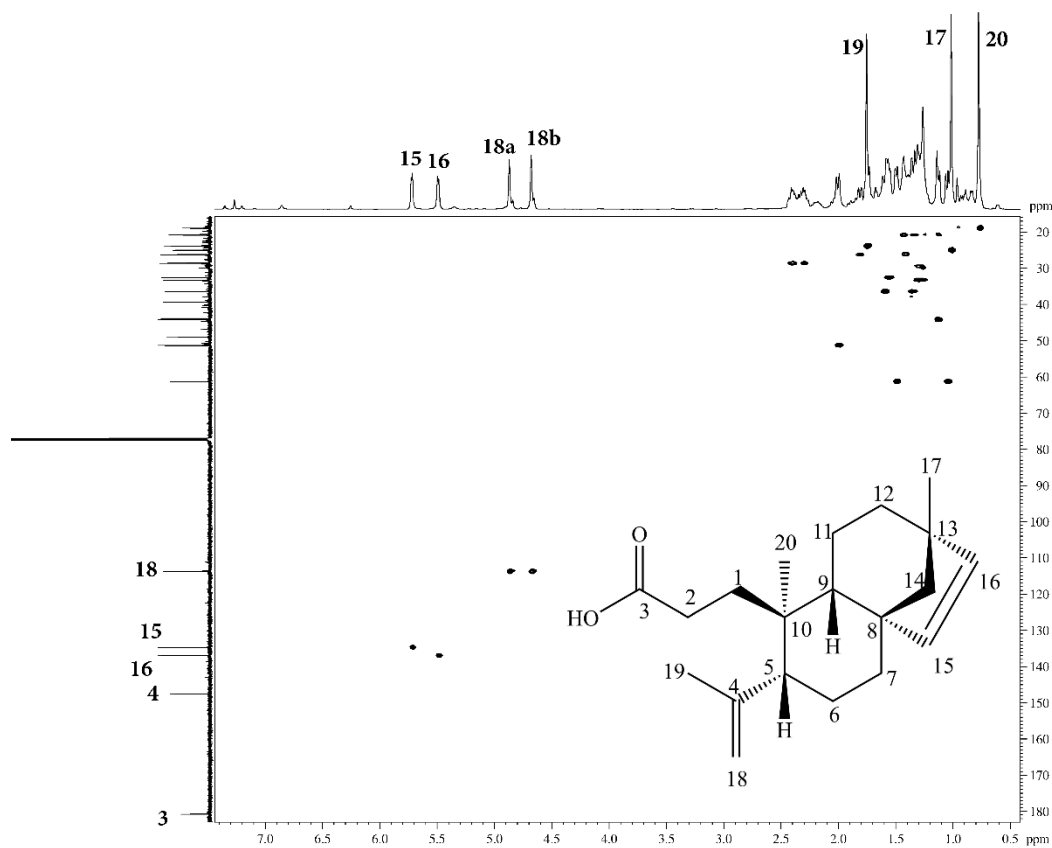


Figure S7. ^1H , ^{13}C HSQC-NMR spectrum (500×125 MHz, CDCl_3) of compound ICB1.

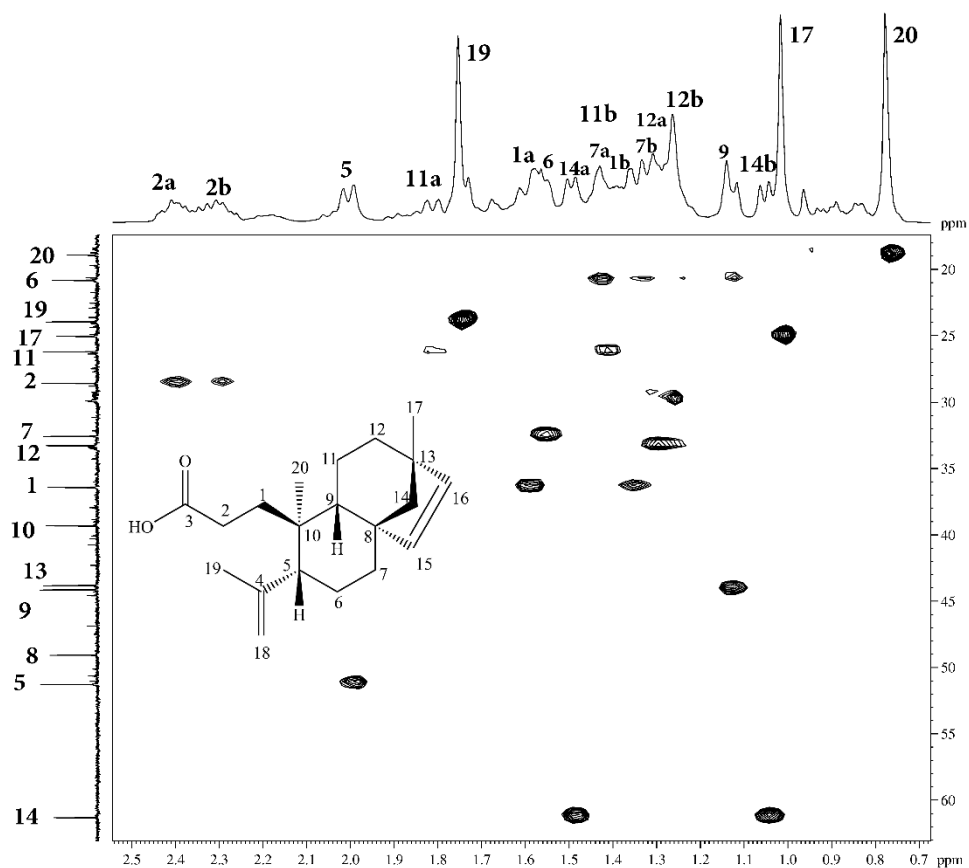


Figure S8. ^1H , ^{13}C HSQC-NMR spectrum (500 $\times$ 125 MHz, CDCl_3) of compound ICB1.

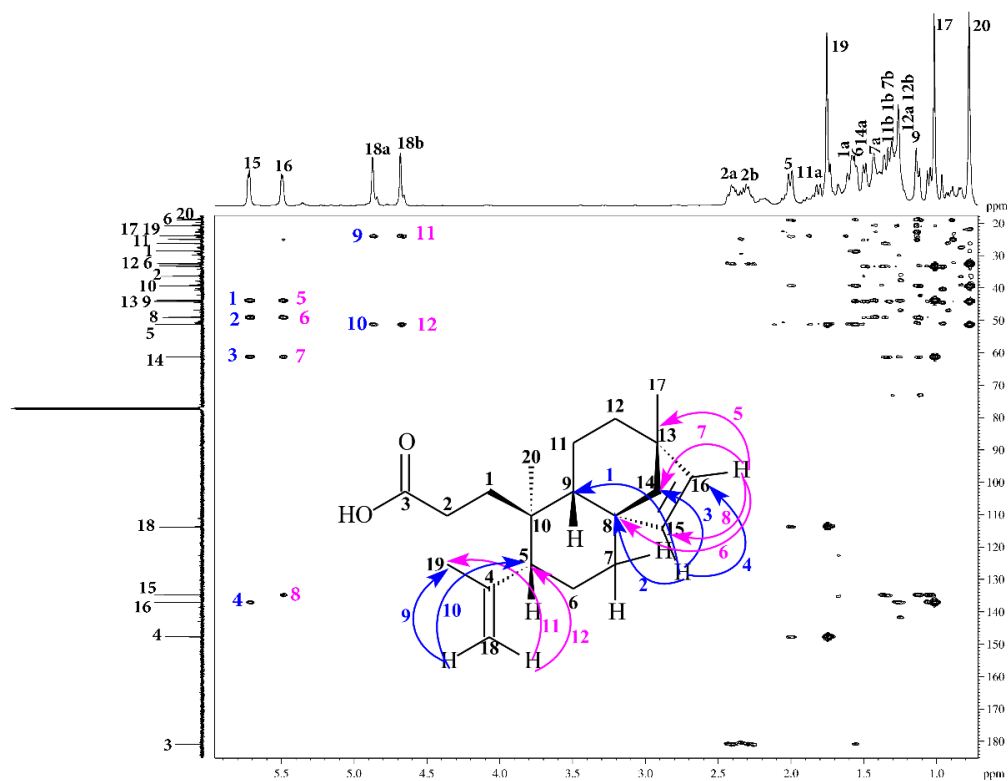


Figure S9. ^1H , ^{13}C HMBC-NMR spectrum (500 $\times$ 125 MHz, CDCl_3) of compound ICB1.

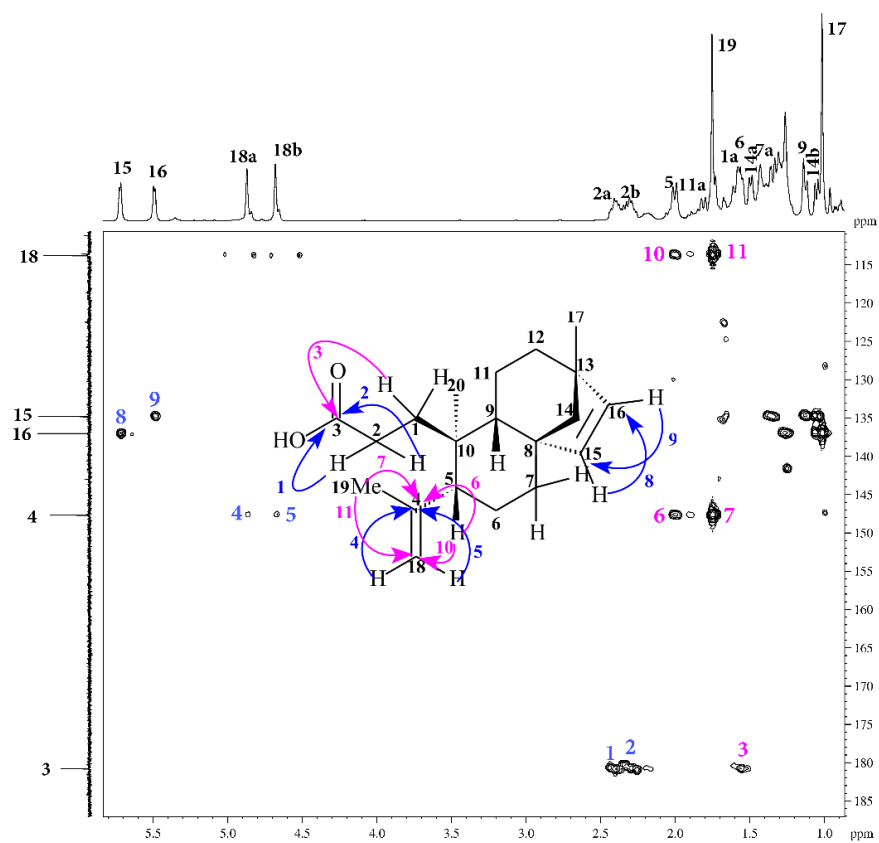


Figure S10. ^1H , ^{13}C HMBC-NMR spectrum (500 $\times$ 125 MHz, CDCl_3) of compound ICB1.

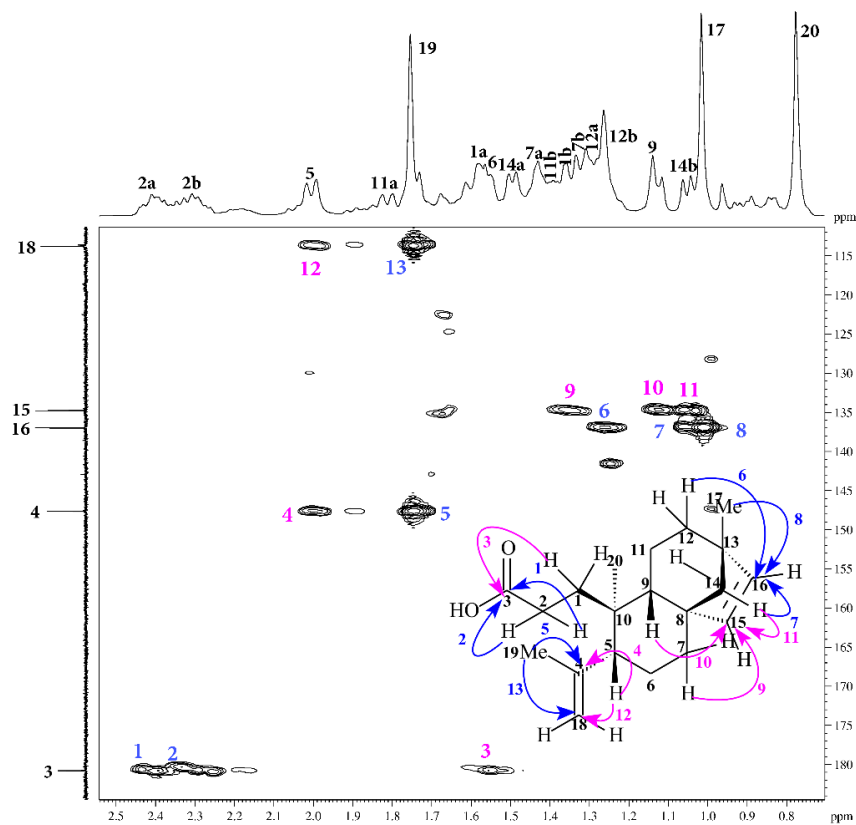


Figure S11. ^1H , ^{13}C HMBC-NMR spectrum (500 $\times$ 125 MHz, CDCl_3) of compound ICB1.

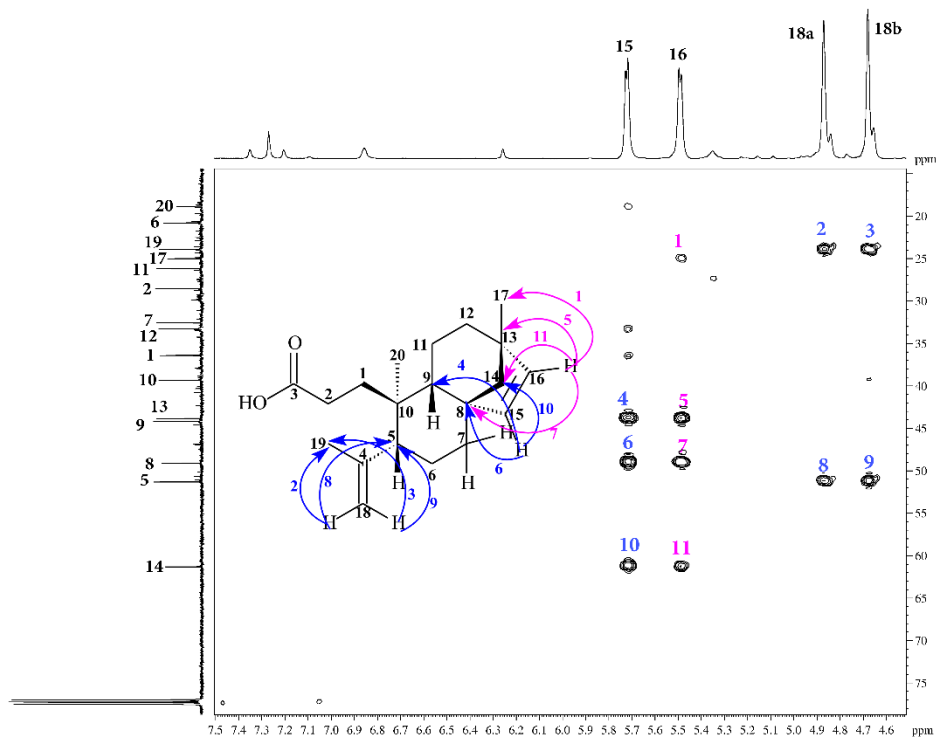


Figure S12. ^1H , ^{13}C HMBC-NMR spectrum (500 $\times$ 125 MHz, CDCl_3) of compound ICB1.

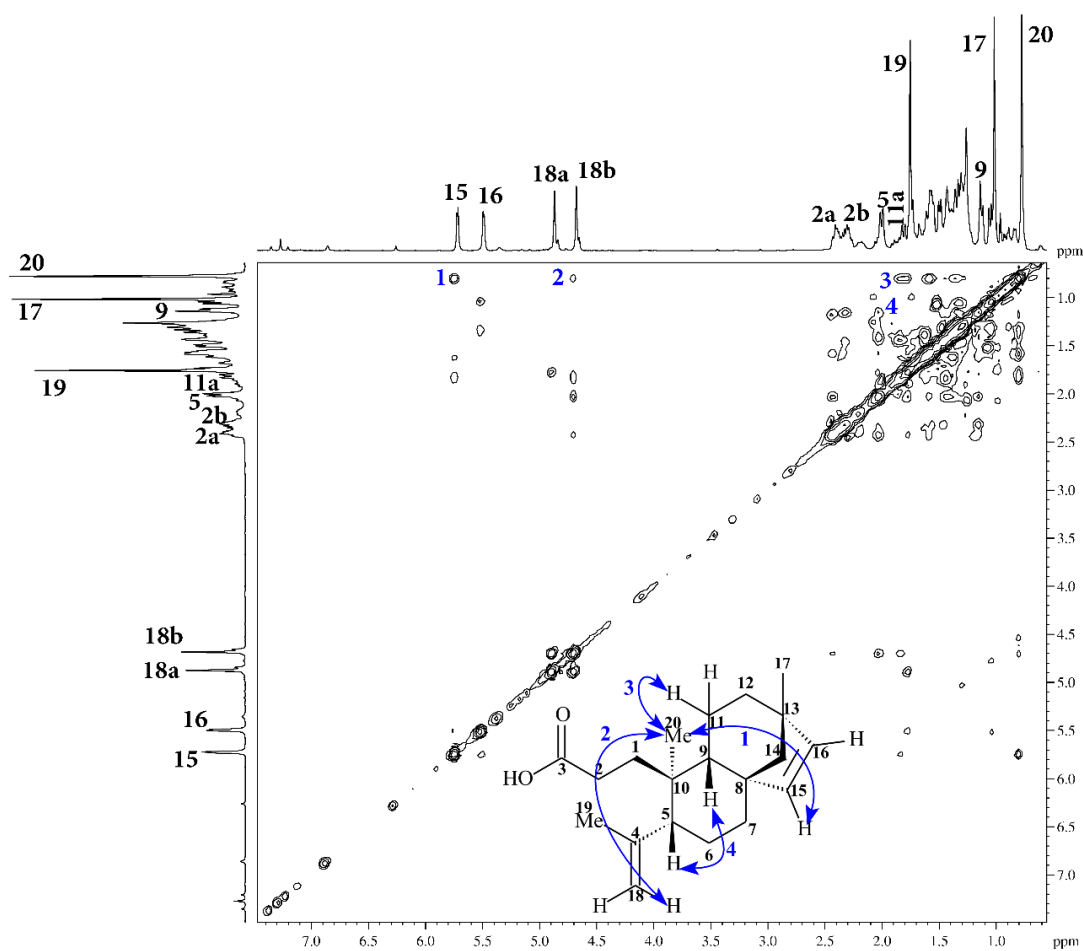


Figure S13. ^1H , ^1H NOESY-NMR spectrum (500 $\times$ 500 MHz, CDCl_3) of compound ICB1.

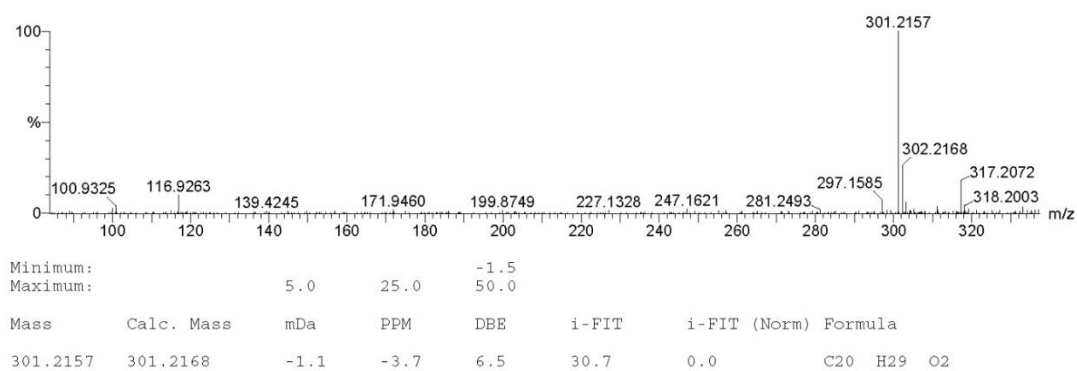


Figure S14. High resolution mass spectrum (negative ion mode) of compound ICB1.

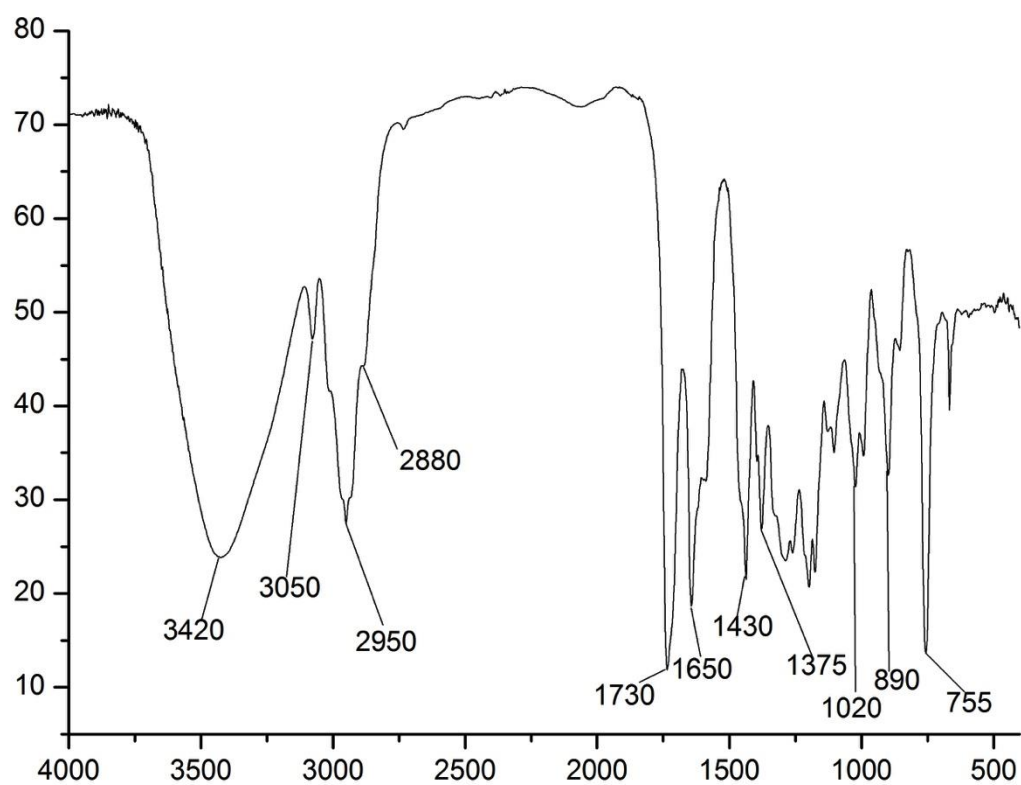


Figure S15. Infrared (UATR) spectrum of compound ICB4.

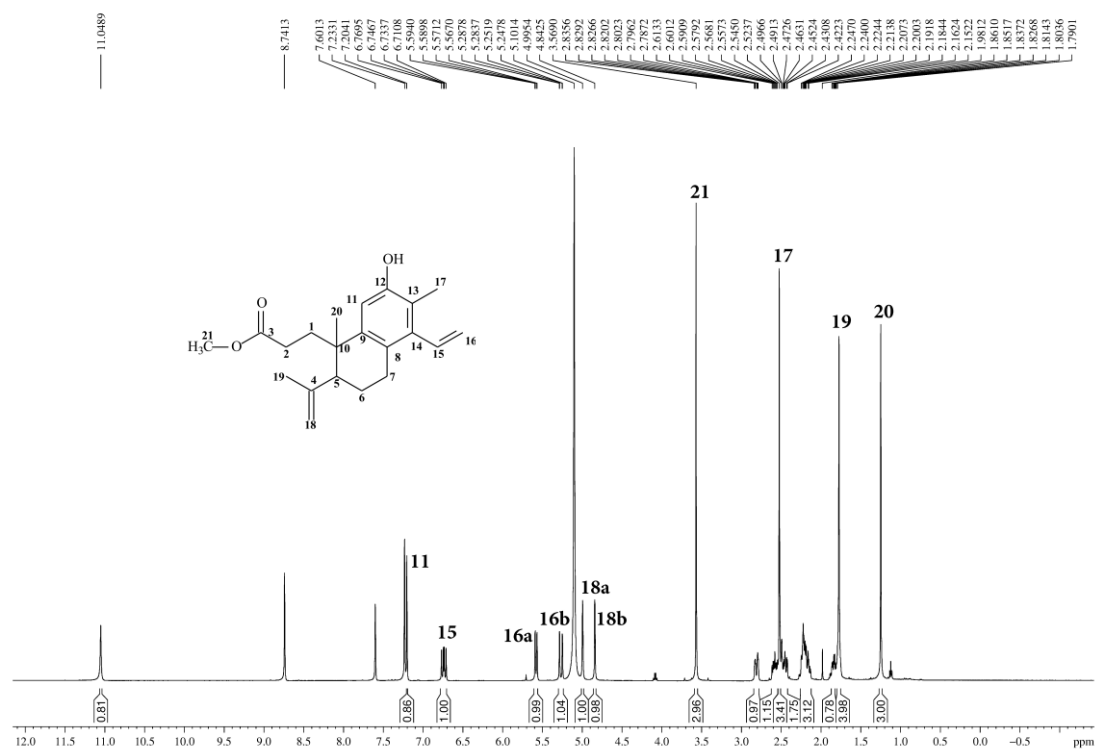


Figure S16. ¹H NMR spectrum (500 MHz, C₅D₅N) of compound ICB4.

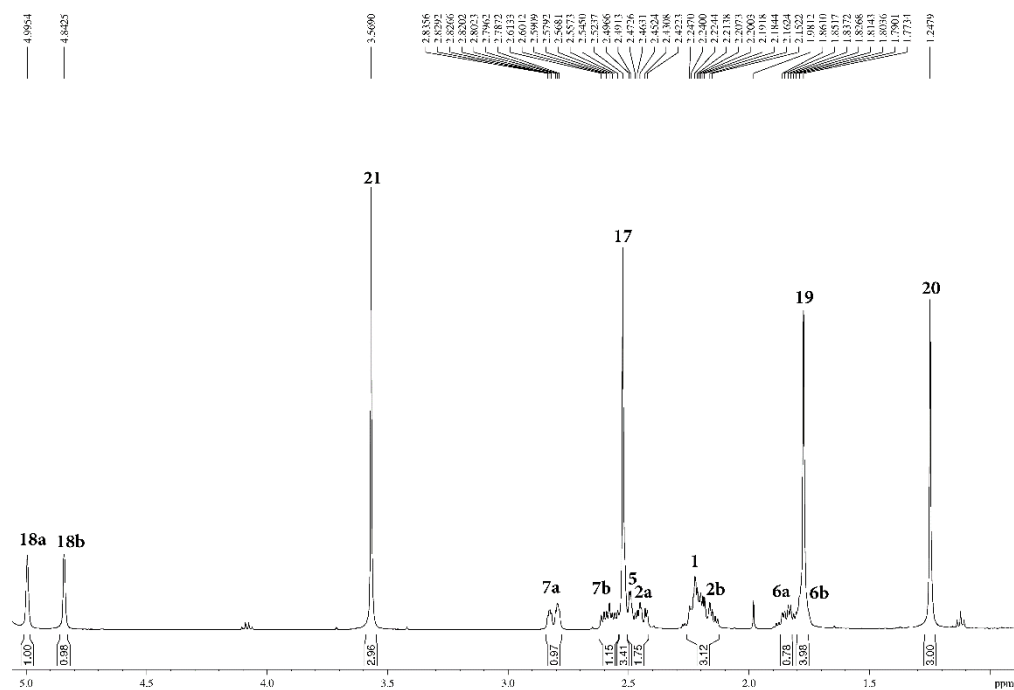


Figure S17. ¹H NMR spectrum (500 MHz, C₅D₅N) of compound ICB4.

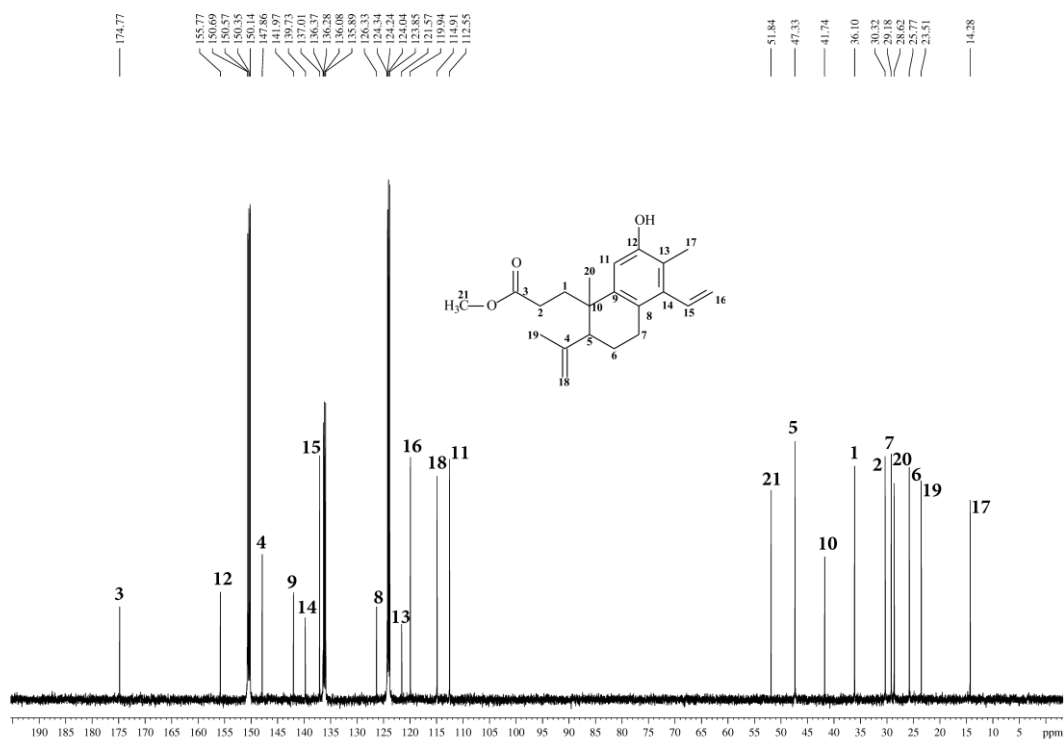


Figure S18. ¹³C NMR spectrum (125 MHz, C₅D₅N) of compound ICB4.

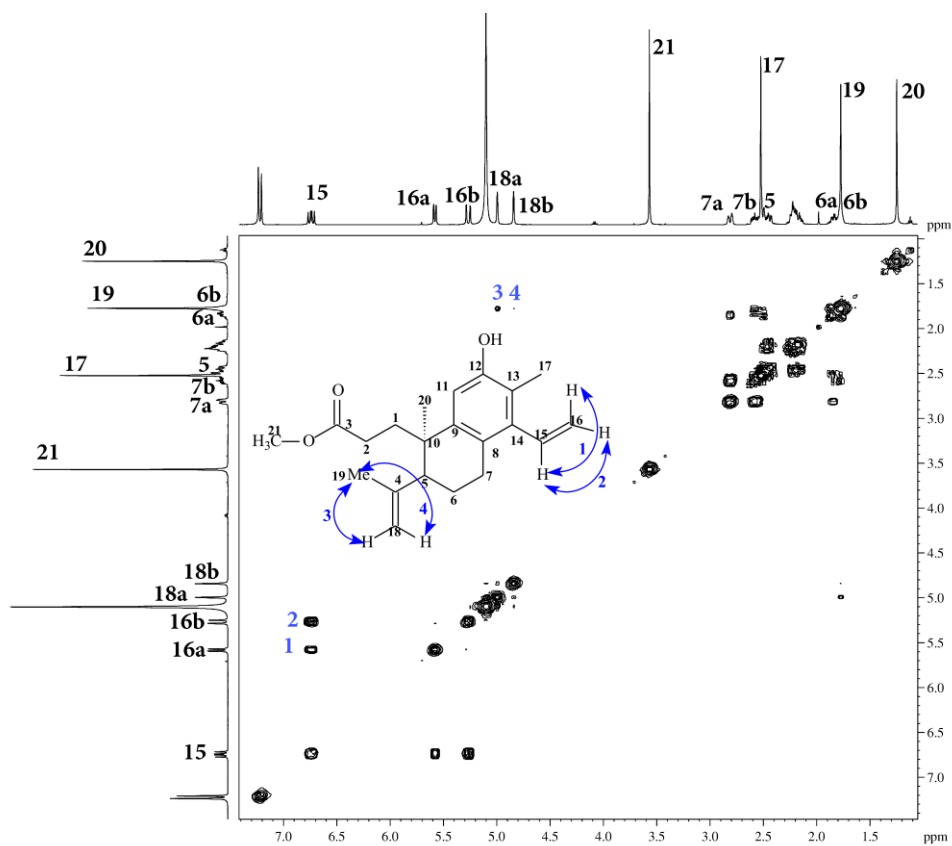


Figure S19. ¹H, ¹H COSY-NMR spectrum (500 × 500 MHz, C₅D₅N) of compound ICB4.

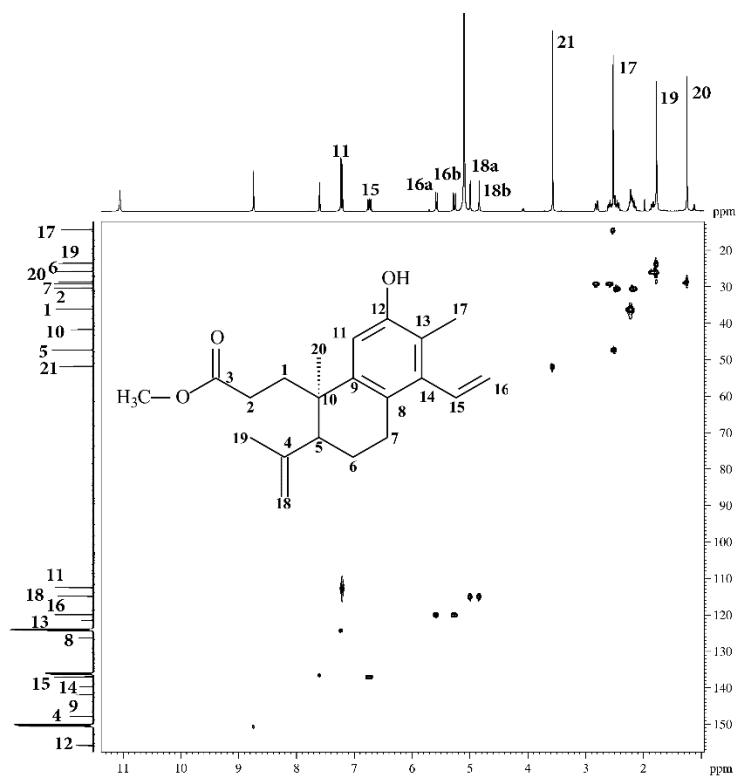


Figure S20. ^1H , ^{13}C HSQC-NMR spectrum (500 $\times$ 125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB4.

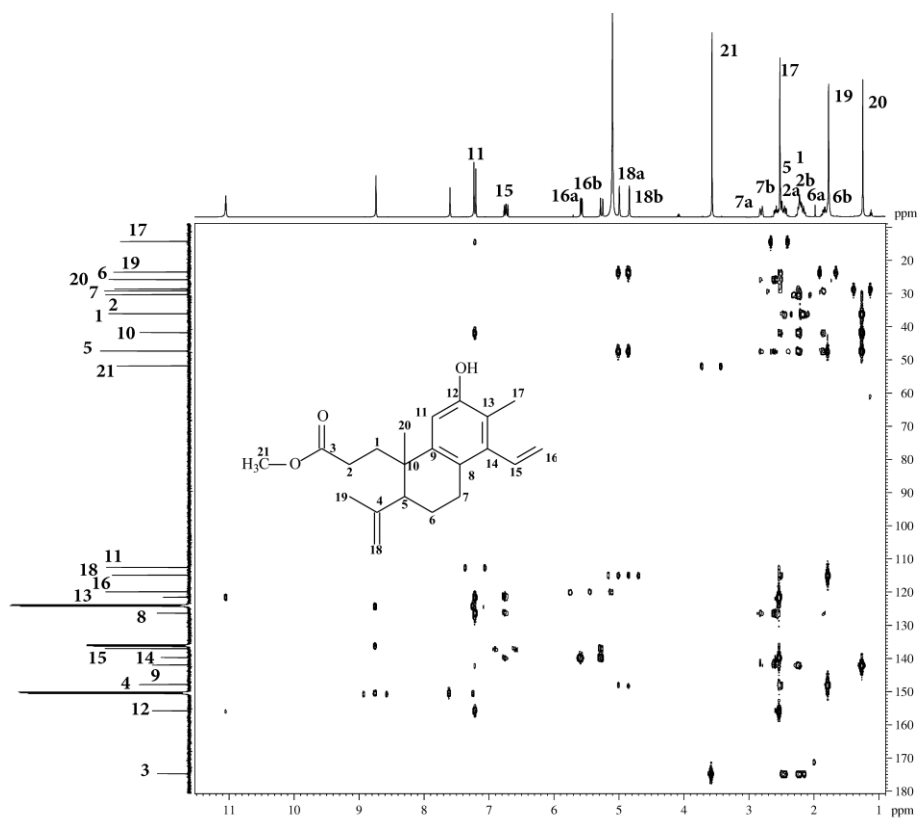


Figure S21. ^1H , ^{13}C HMBC-NMR spectrum (500 $\times$ 125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB4.

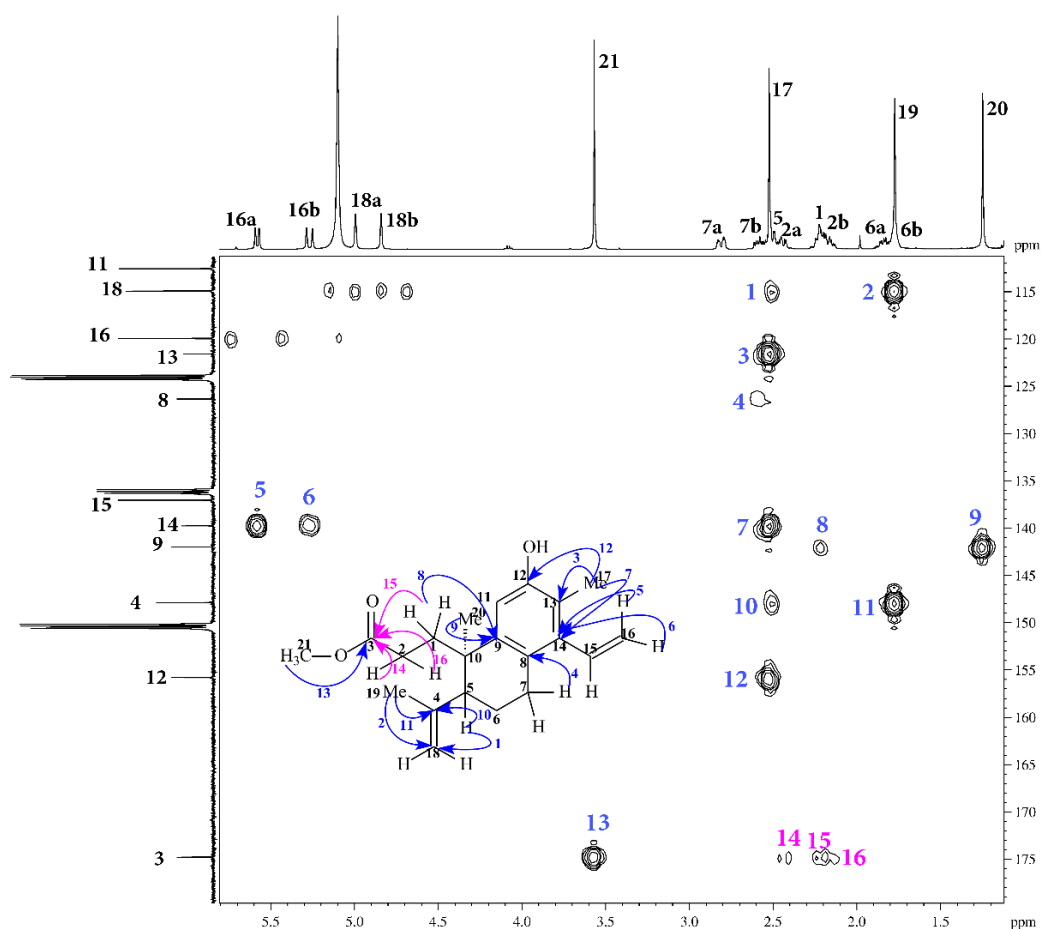


Figure S22. ^1H , ^{13}C HMBC-NMR spectrum (500×125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB4.

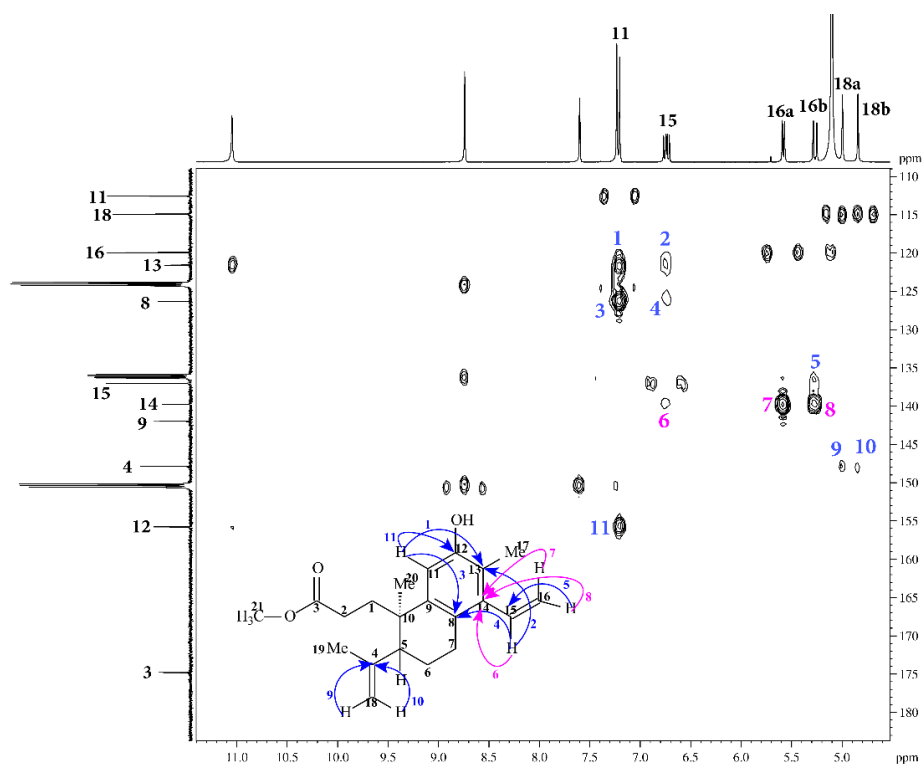


Figure S23. ^1H , ^{13}C HMBC-NMR spectrum (500×125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB4.

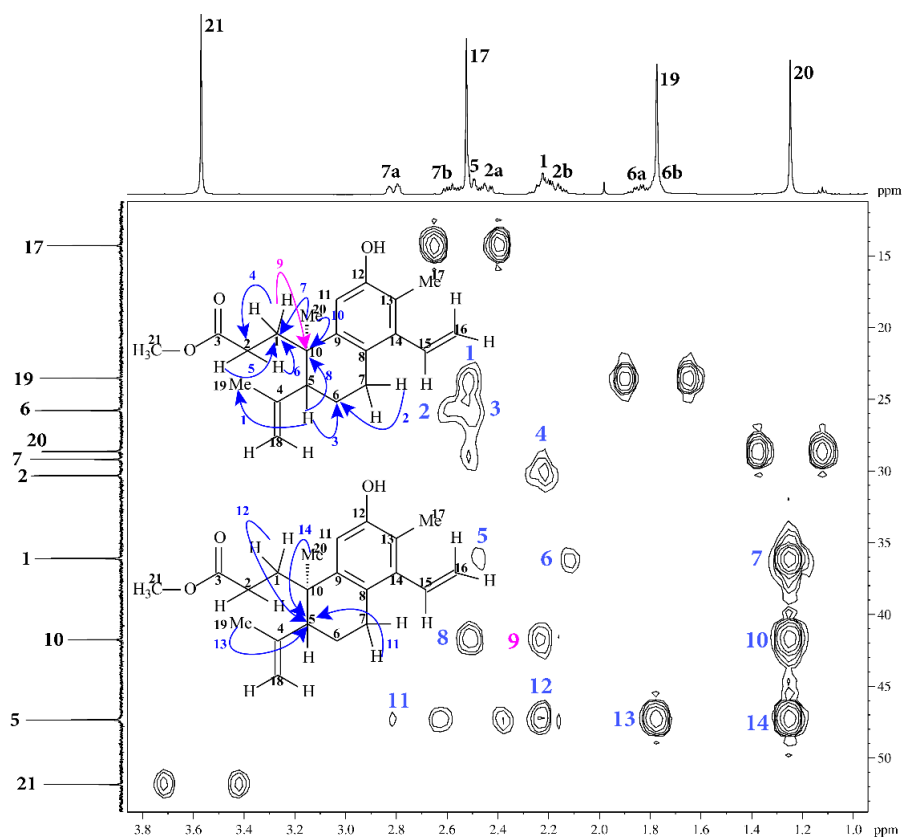


Figure S24. ^1H , ^{13}C HMBC-NMR spectrum (500×125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB4.

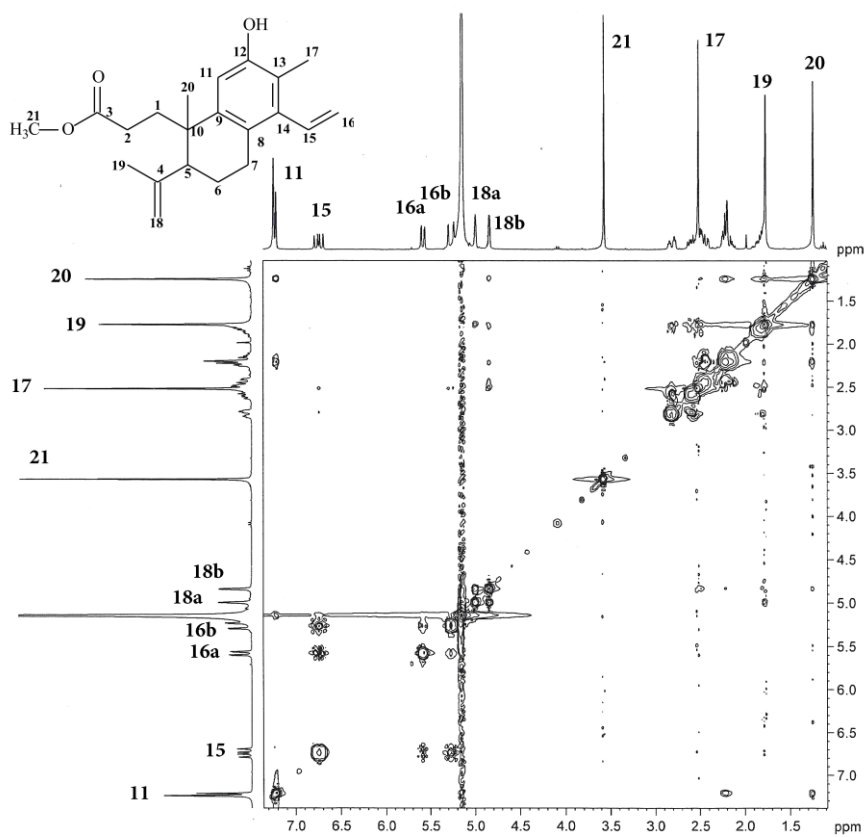


Figure S25. ^1H , ^1H NOESY-NMR spectrum (500×500 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB4.

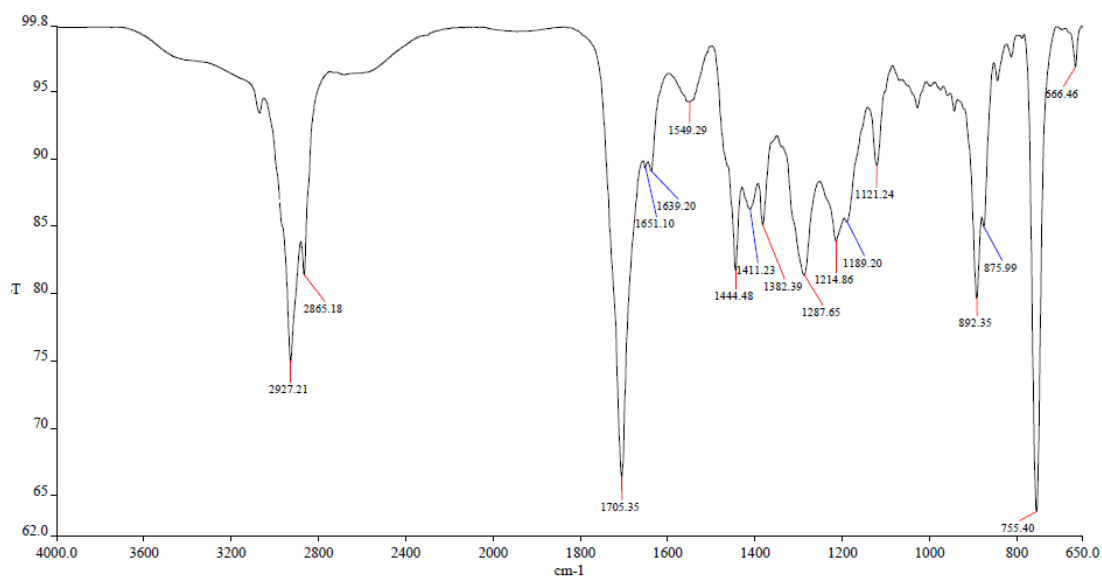


Figure S26. Infrared (UATR) spectrum of compound ICB8.

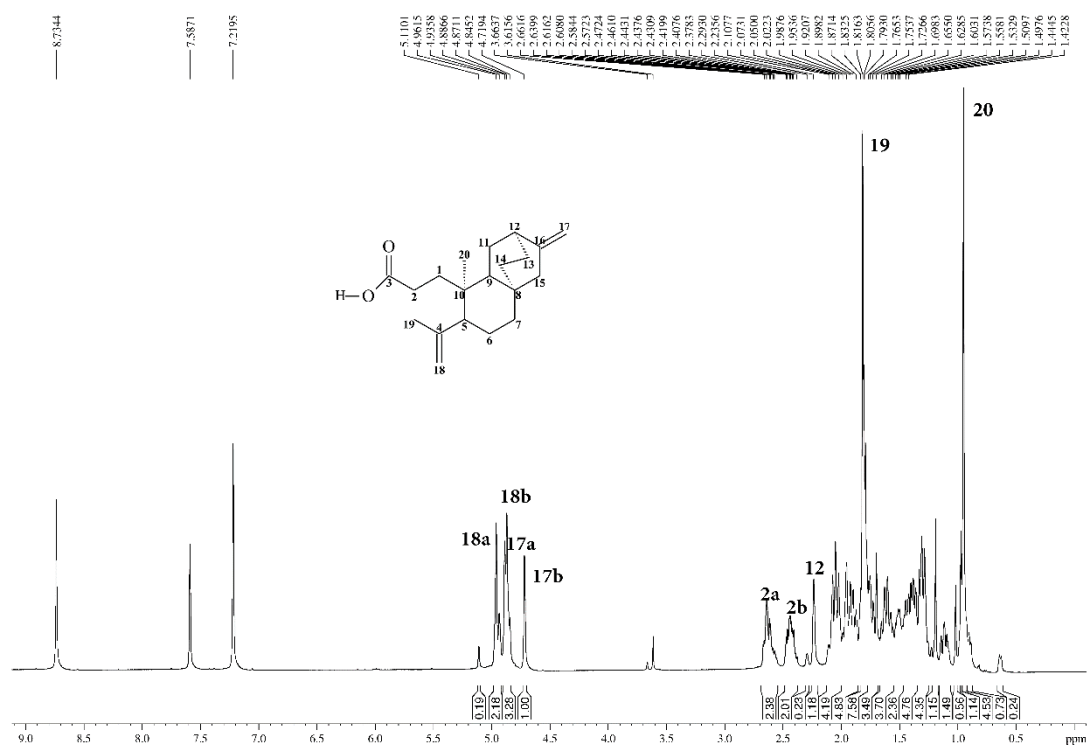


Figure S27. ^1H NMR spectrum (500 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB8.

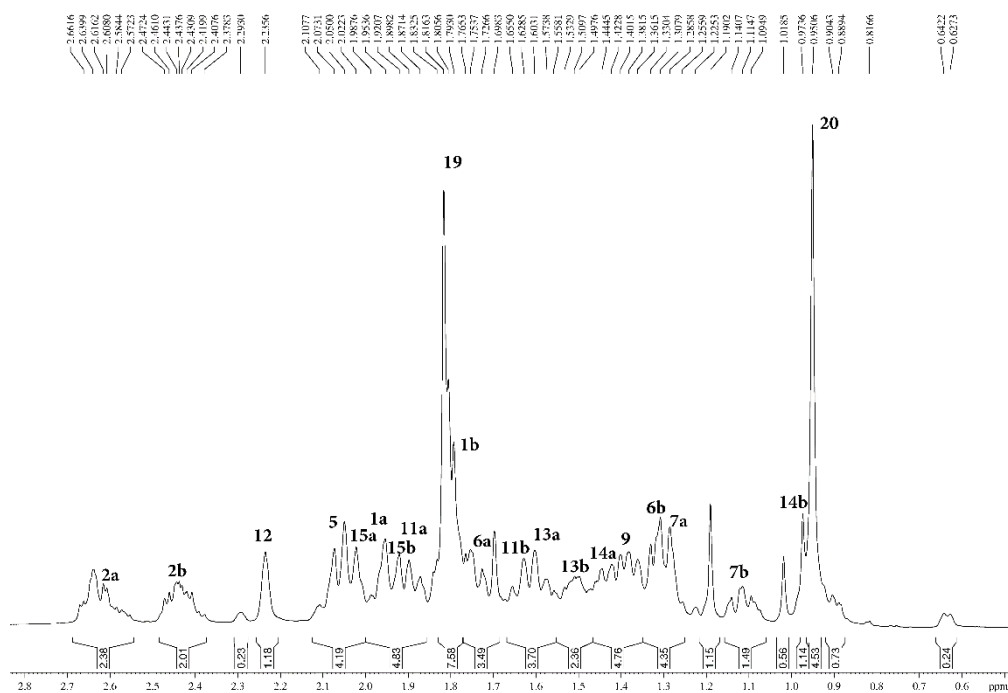


Figure S28. ¹H NMR spectrum (500 MHz, C₅D₅N) of compound ICB8.

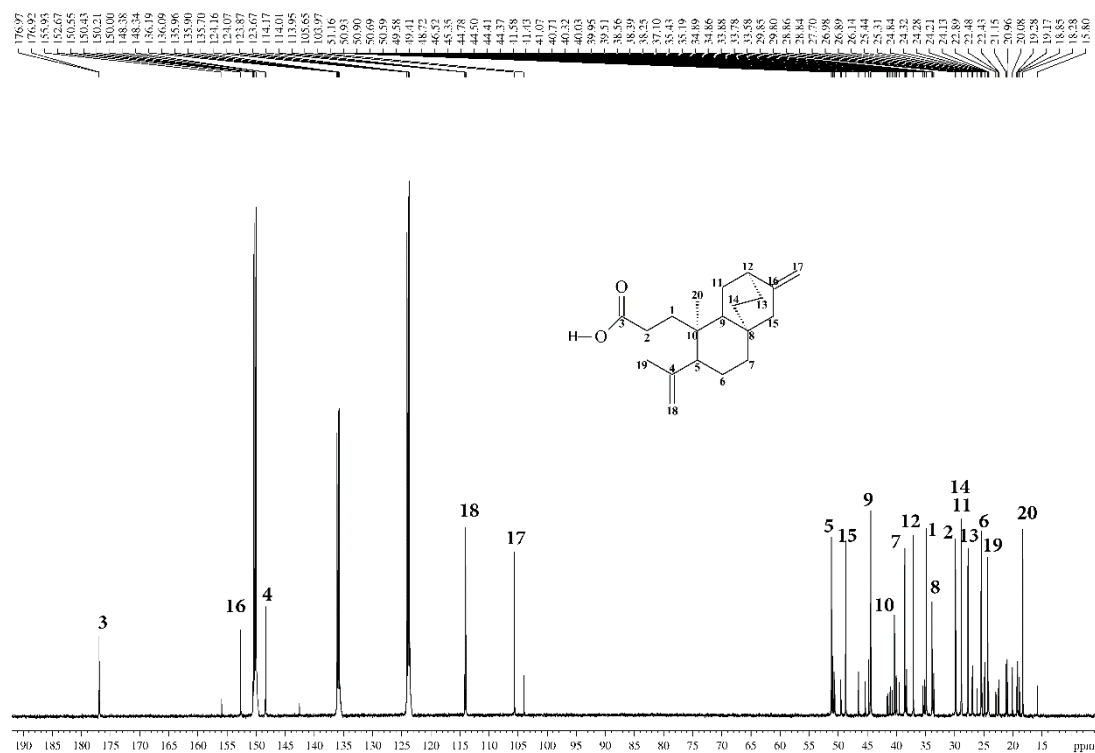


Figure S29. ¹³C NMR spectrum (125 MHz, C₅D₅N) of compound ICB8.

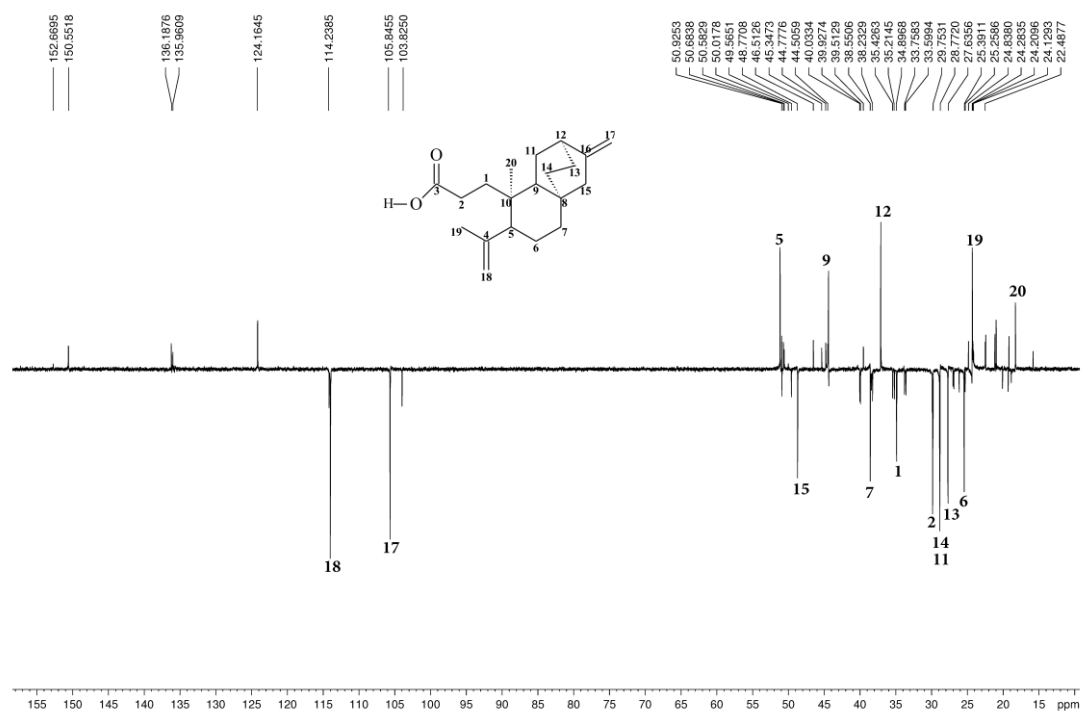


Figure S30. DEPT 135° NMR spectrum (125 MHz, C₅D₅N) of compound ICB8.

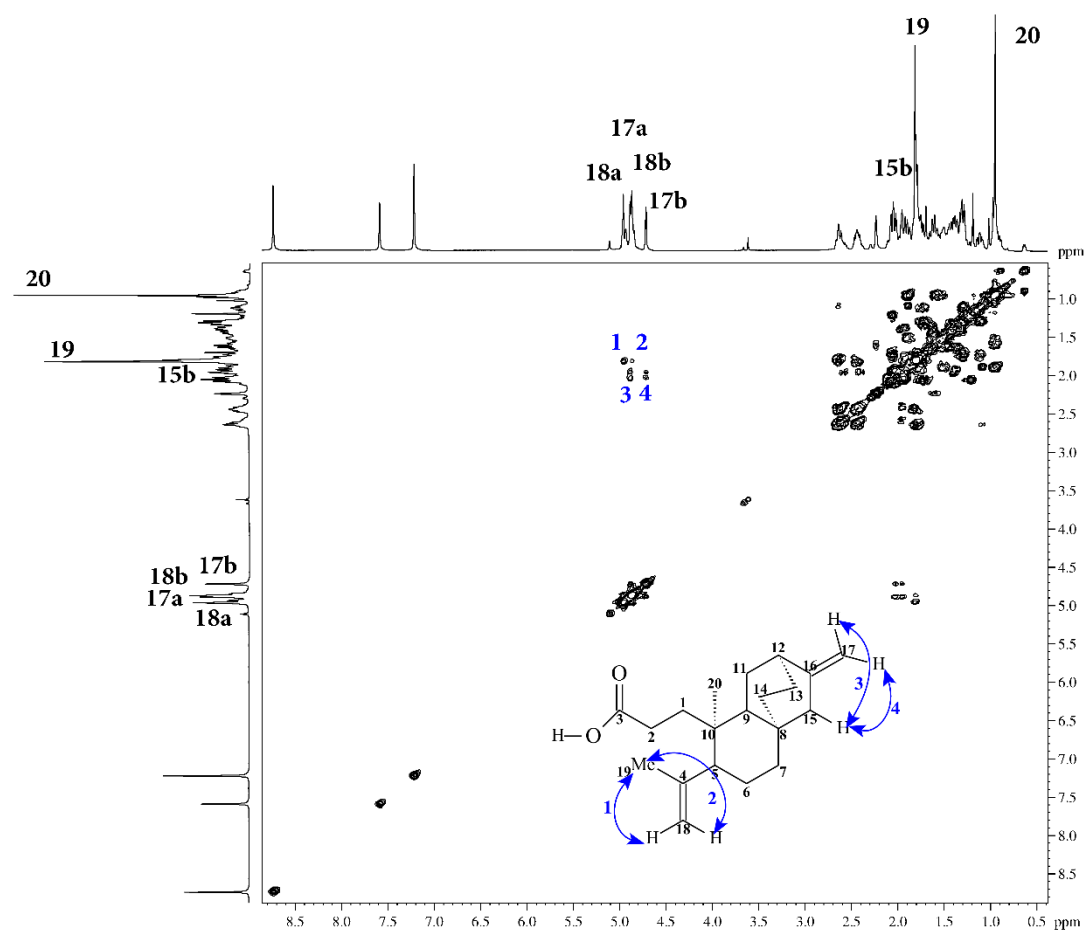


Figure S31. ¹H, ¹H COSY-NMR spectrum (500 × 500 MHz, C₅D₅N) of compound ICB8.

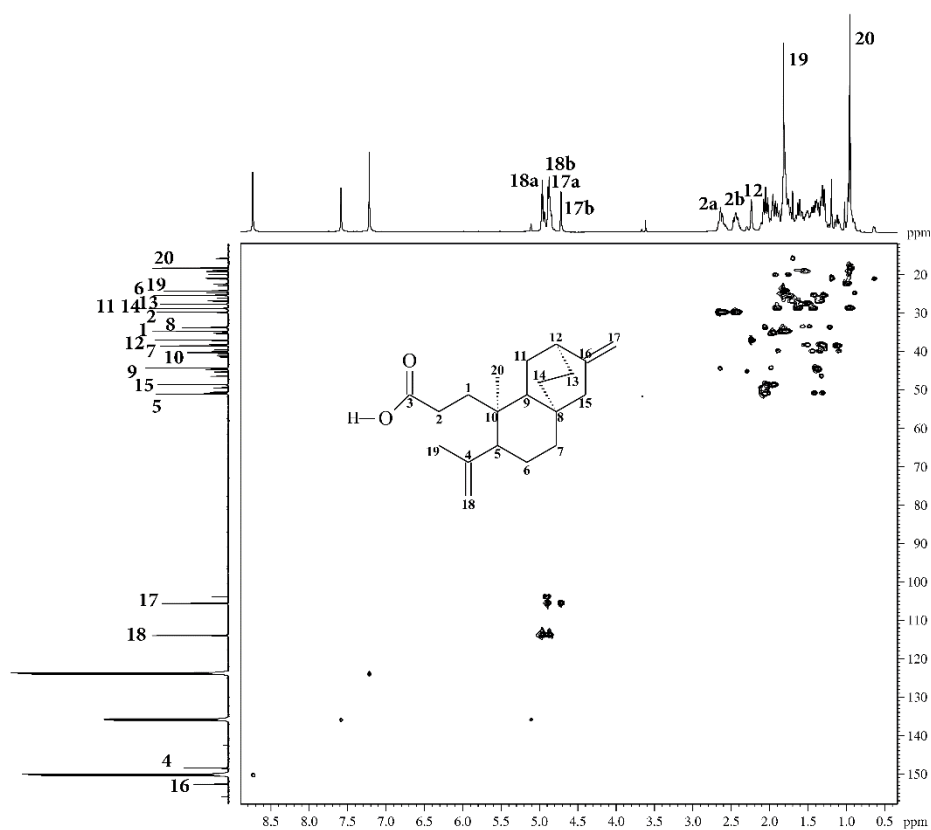


Figure S32. ^1H , ^{13}C HSQC-NMR spectrum (500 $\times$ 125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB8.

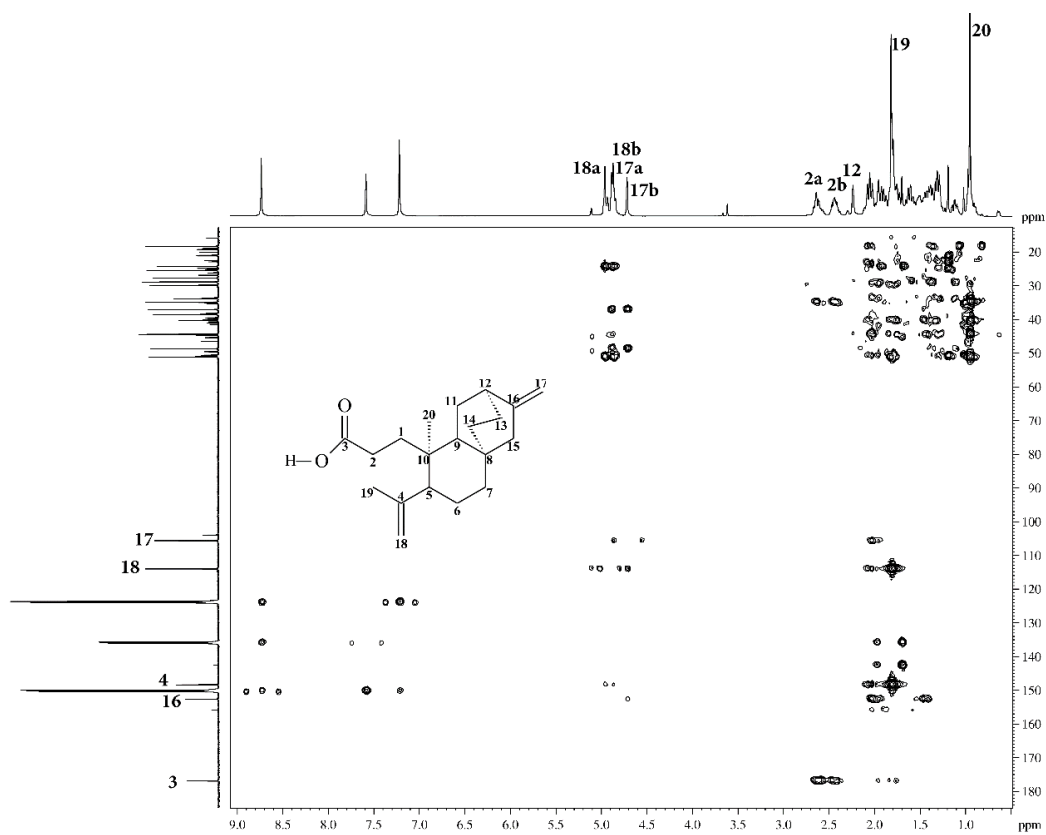


Figure S33. ^1H , ^{13}C HMBC-NMR spectrum (500 $\times$ 125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB8.

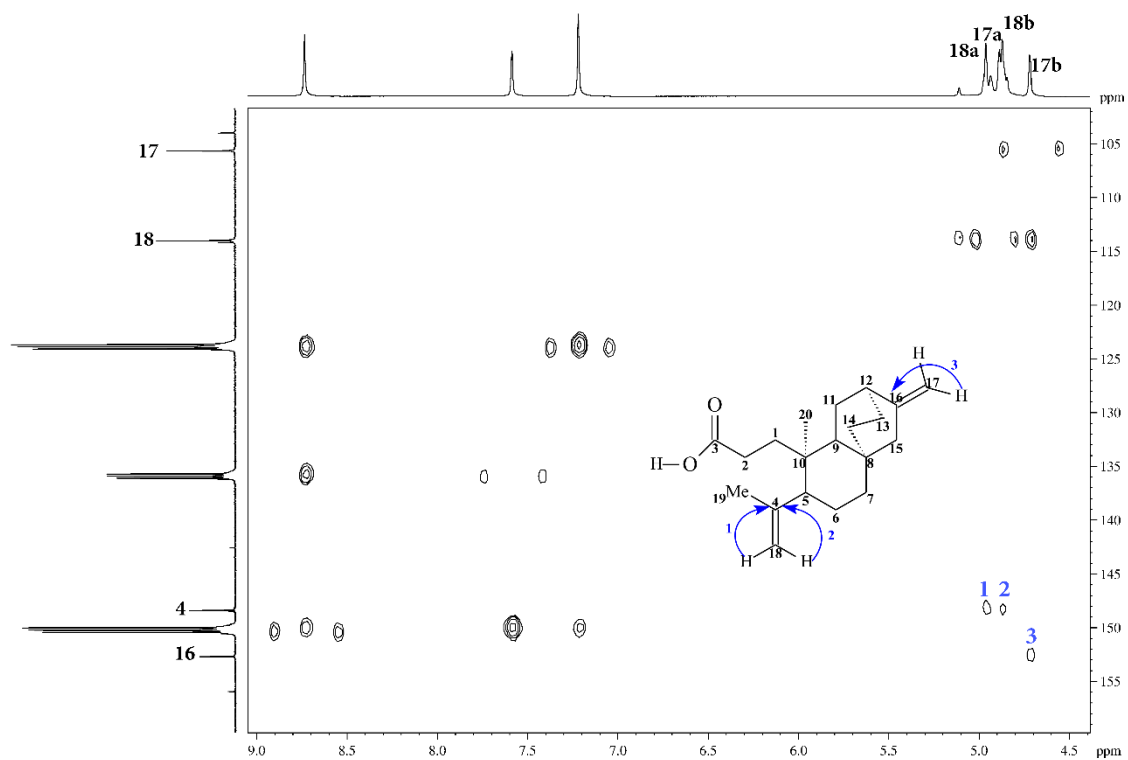


Figure S34. ^1H , ^{13}C HMBC-NMR spectrum (500×125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB8.

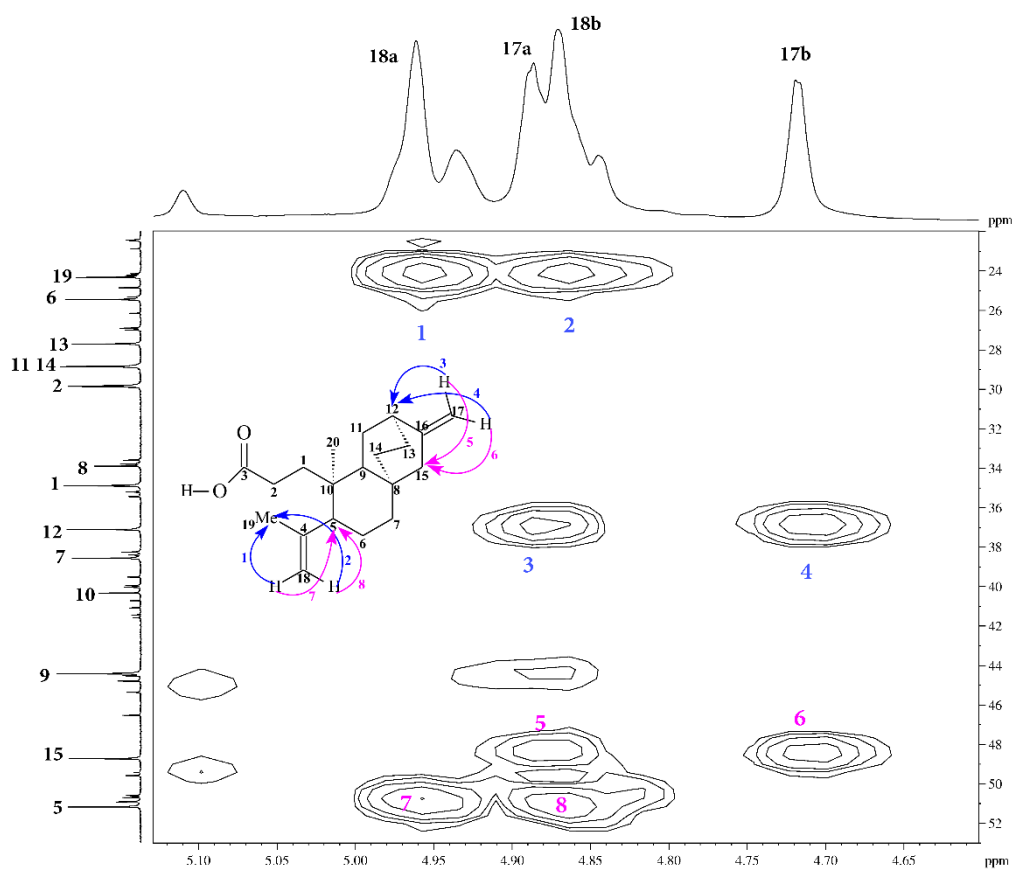


Figure S35. ^1H , ^{13}C HMBC-NMR spectrum (500×125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB8.

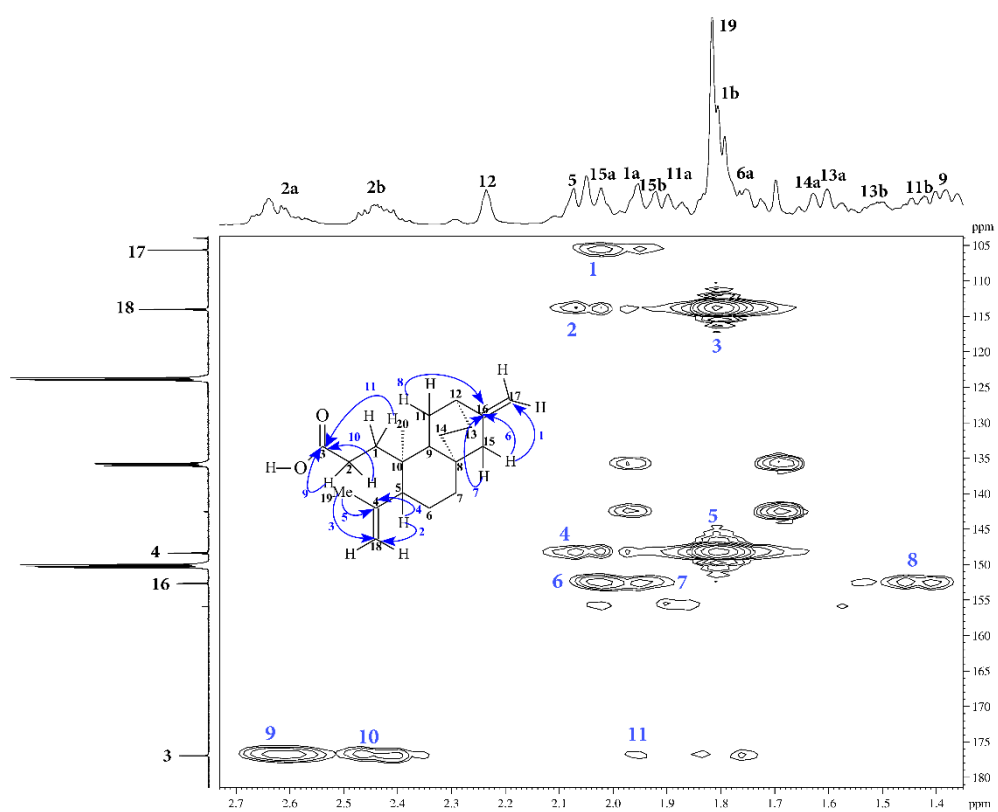


Figure S36. ^1H , ^{13}C HMBC-NMR spectrum (500 $\times$ 125 MHz, $\text{C}_5\text{D}_5\text{N}$) of compound ICB8.