

Supplementary Materials

Mesoscale Assembly of Bisteroidal Esters from Terephthalic Acid

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Bicholesterol ester (5).

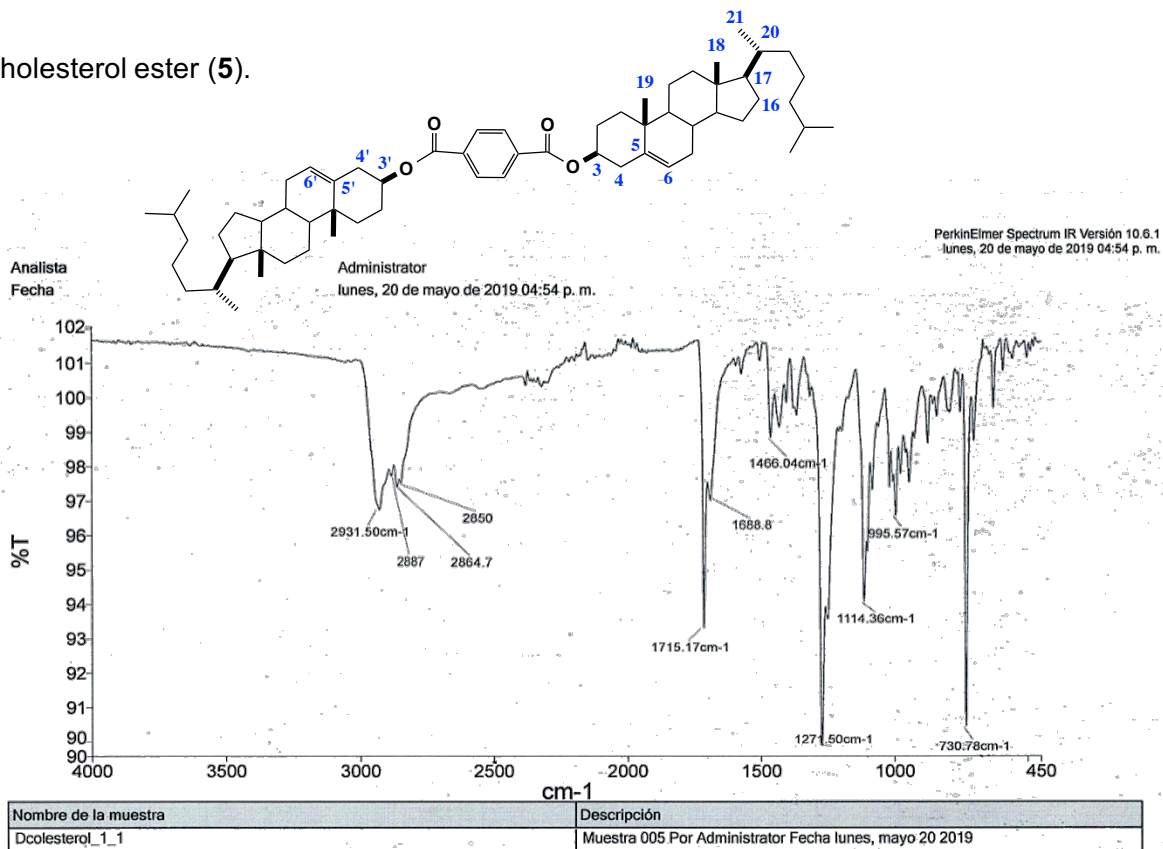


Figure S1. IR spectrum of Bicholesterol ester (5).

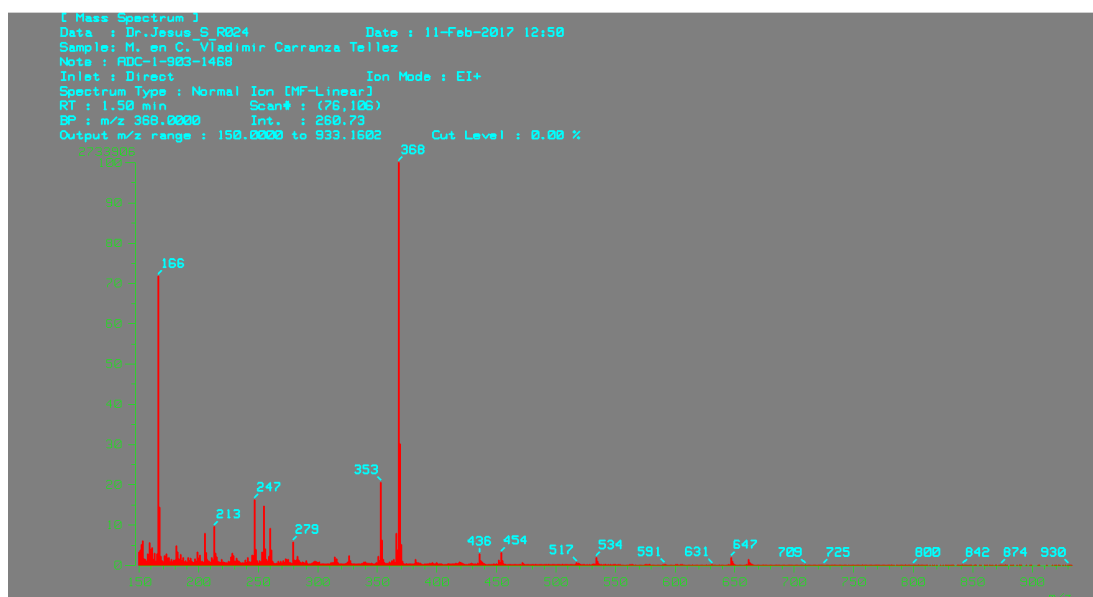


Figure S2. Mass spectrum of Bicholesterol ester (5).

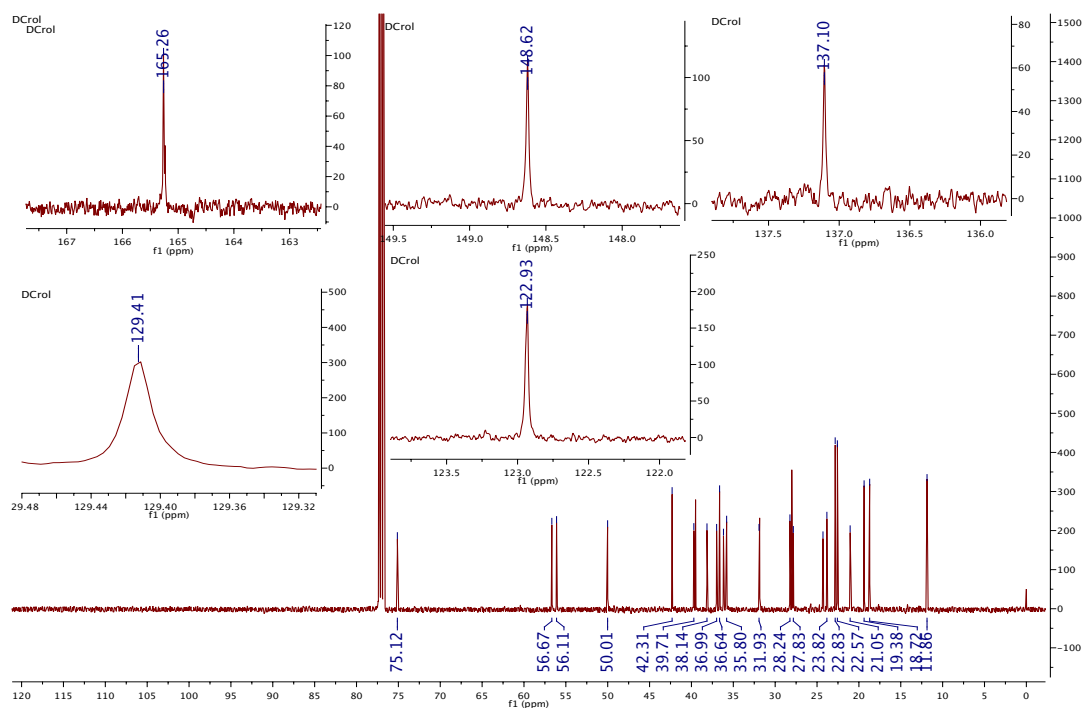


Figure S3. ^{13}C NMR spectrum at 125 MHz in CDCl_3 of Bicholesterol ester (5).

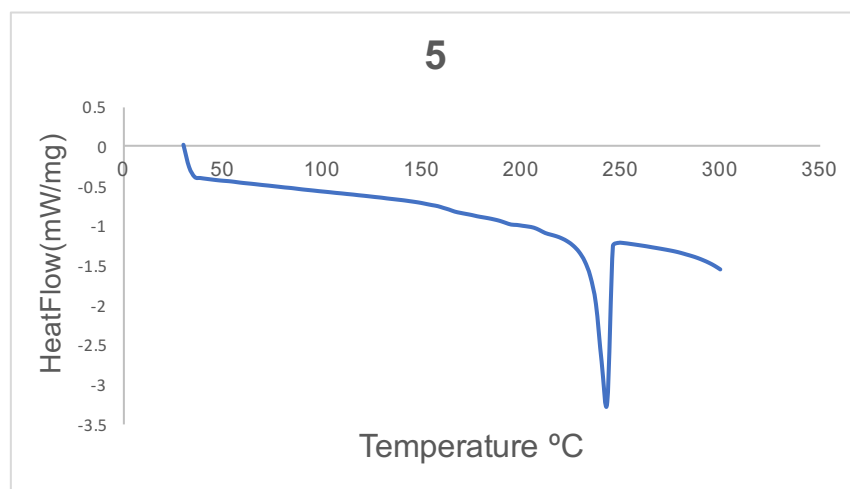


Figure S4. Differential scanning calorimetry (DSC) of Bicholesterol ester (5).

Bicholestanol ester (6)

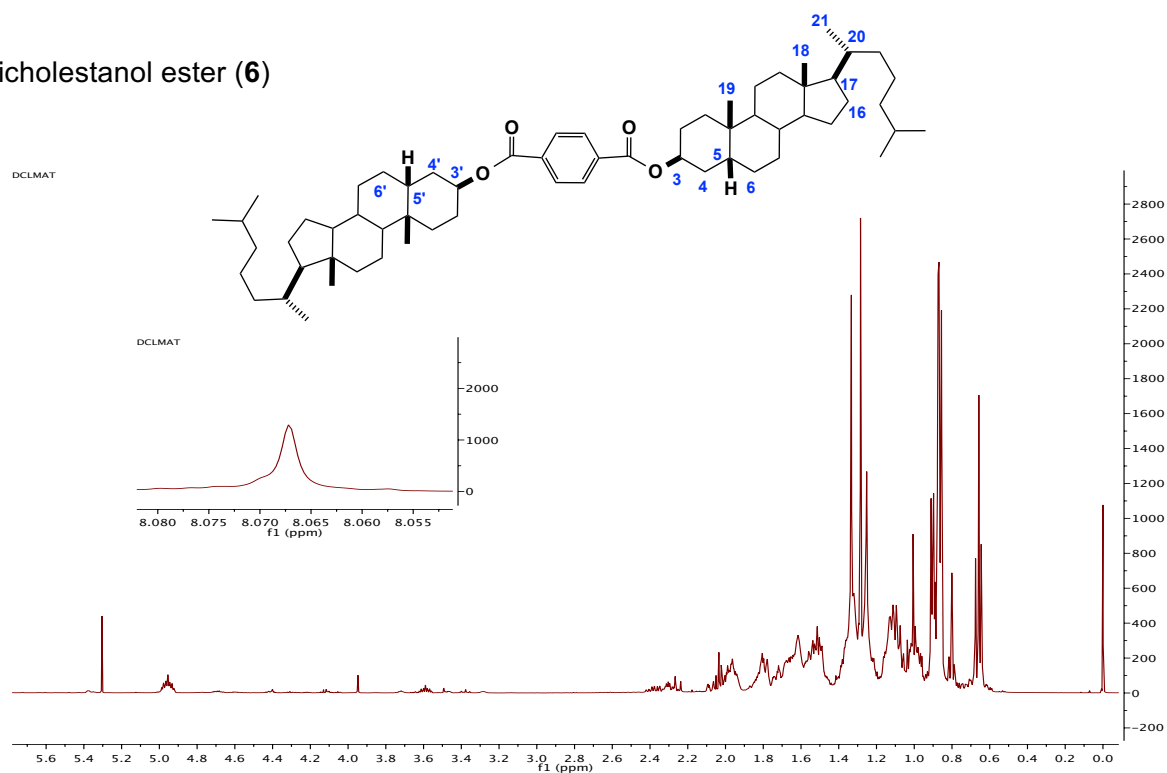


Figure S5. ^1H NMR spectrum at 500 MHz in CDCl_3 of Bicholestanol ester (6).

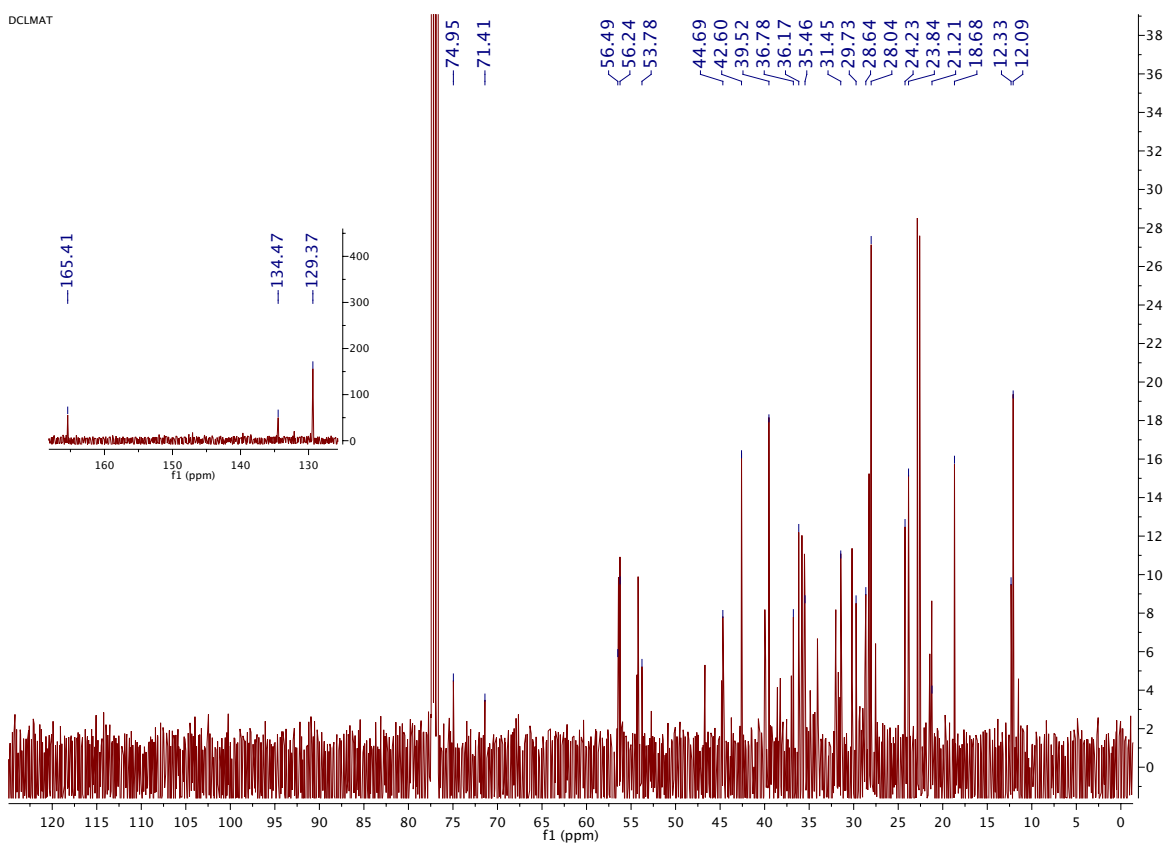


Figure S6. ^{13}C NMR spectrum at 125 MHz in CDCl_3 of the of Bicholestanol ester (6).

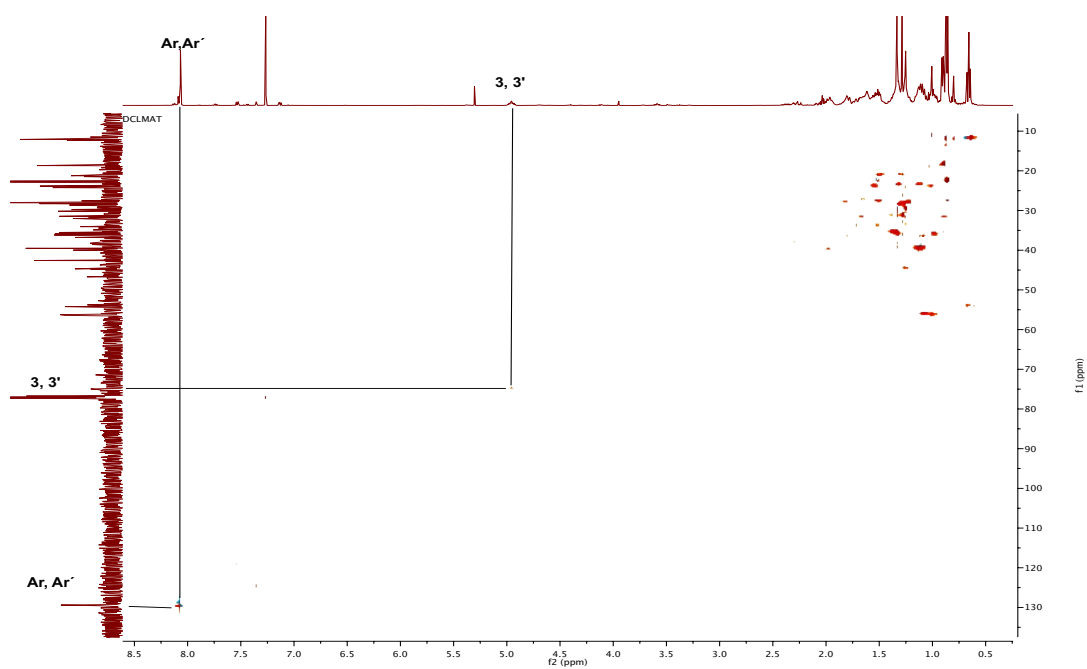


Figure S7. HSQC-NMR spectrum at 500 MHz of Bicholestanol ester (**6**).

Bidiosgenin terephthalate (**8a**).

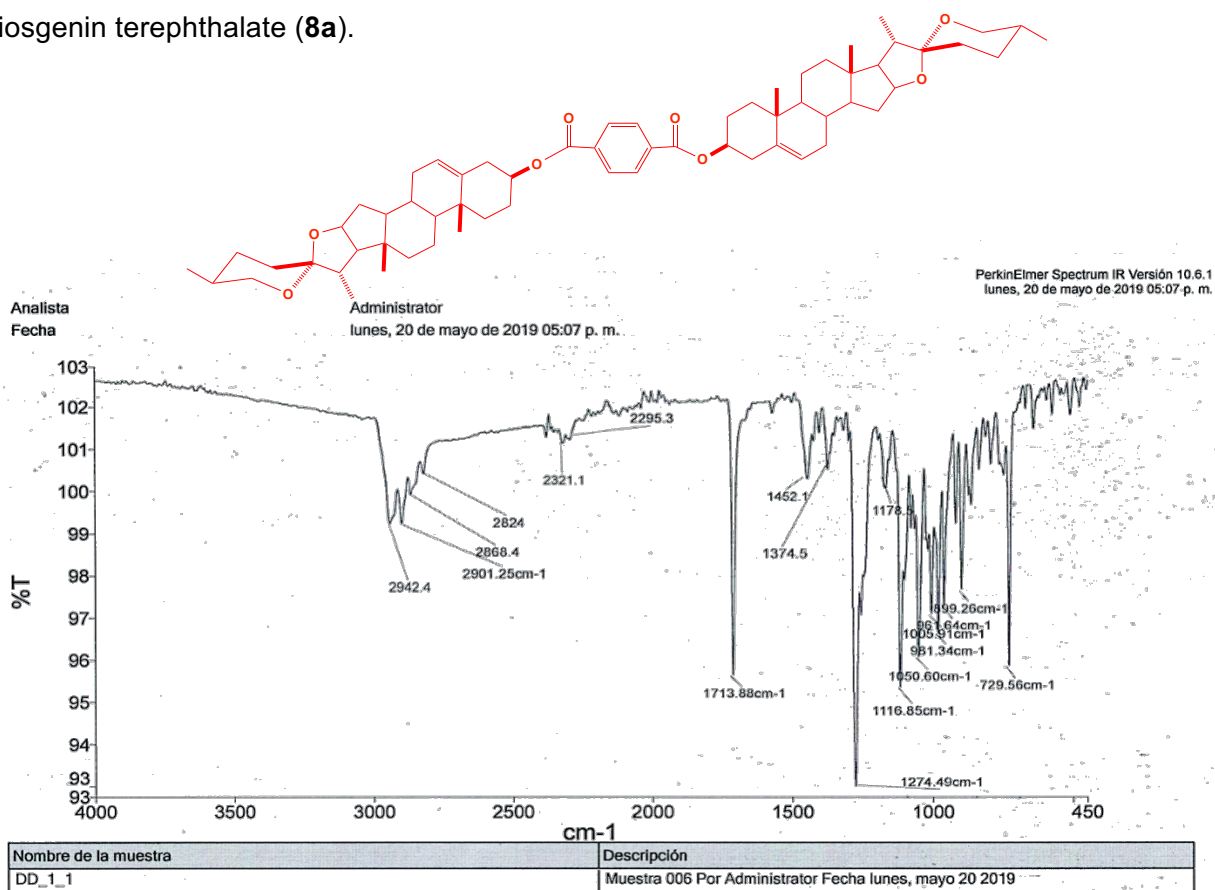


Figure S8. IR spectrum of Bidiosgenin ester (**8a**).

[Elemental Composition]
 Data : Dr-Jesus-Sandoval013
 Sample: STE-2215 LupDD
 Note : -
 Inlet : Direct
 RT : 0.36 min
 Elements : C 64/0, H 120/0, O 28/0
 Mass Tolerance : 1000ppm, 2mmu if m/z > 2
 Unsaturation (U.S.) : -0.5 - 30.0

Date : 14-Oct-2010 17:31

Page: 1

Ion Mode : FAB+
 Scan#: (1,6)

Observed m/z	Int%	Estimated m/z	Error[ppm]	U.S.	C	H	O
959.6414	85.7	959.6401	+1.4	19.5	62	87	8

Figure S9. HRMS data of Bidiosgenin ester (**8a**).

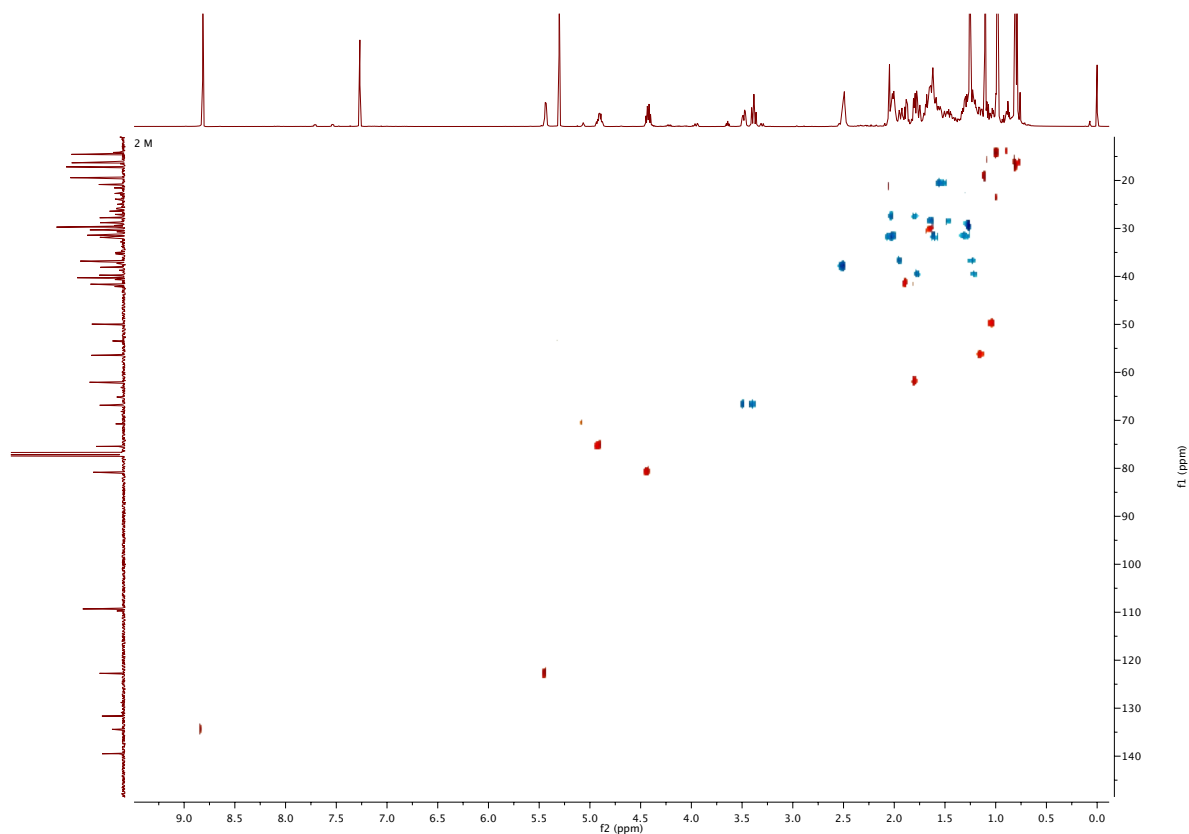


Figure S10. HSQC-NMR spectrum at 600 MHz in CDCl_3 of Bidiosgenin ester (**8a**).

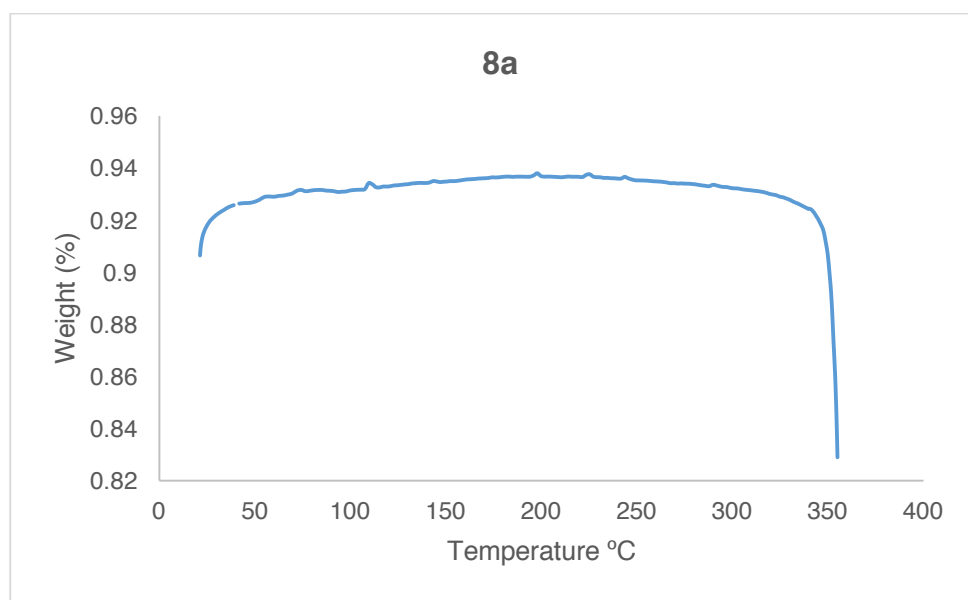


Figure S11. Thermogravimetric analysis of Bidiosgenin ester (**8a**).

Bihecogenin terephthalate (**8b**).

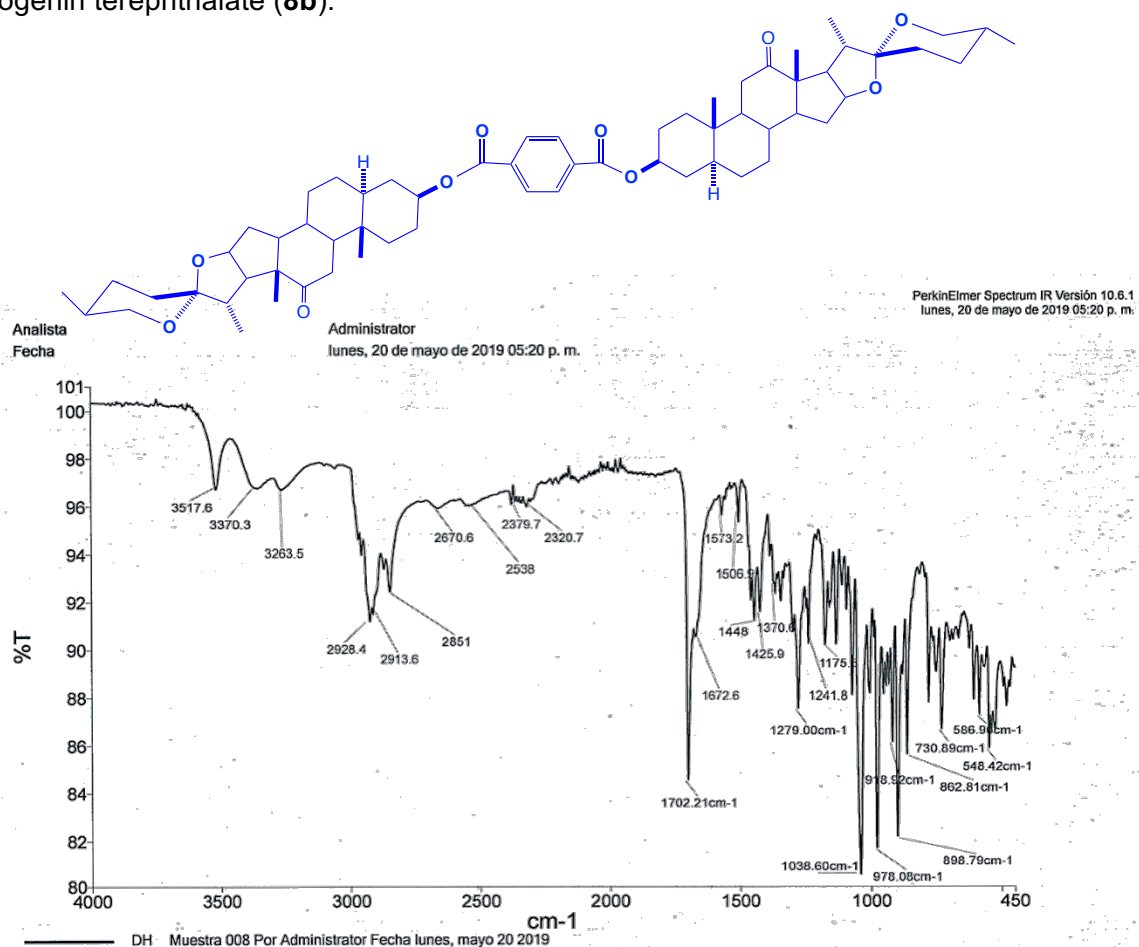


Figure S12. IR spectrum of Bihecogenin ester (**8b**).

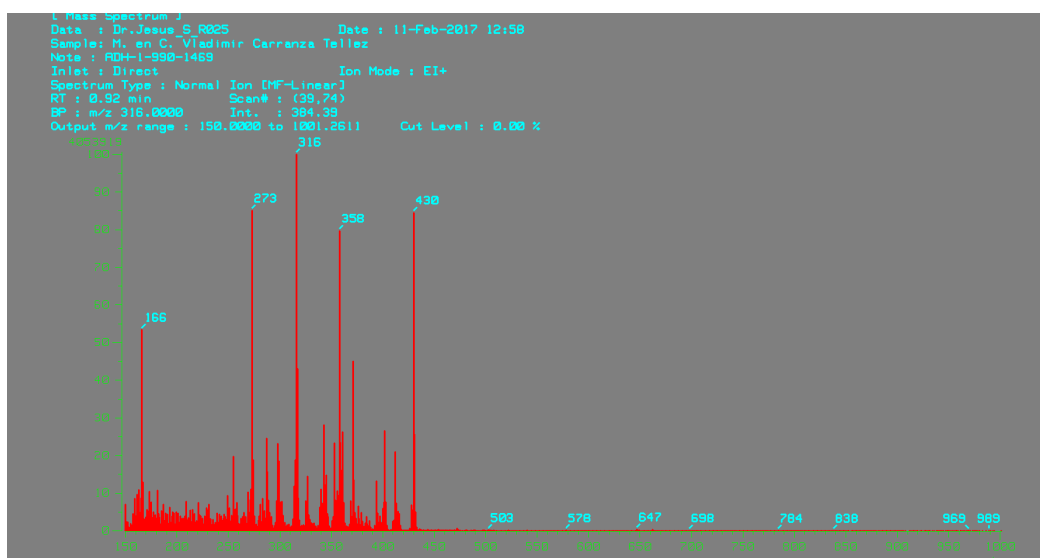


Figure S13. Mass spectrum of Bihecogenin ester (**8b**).

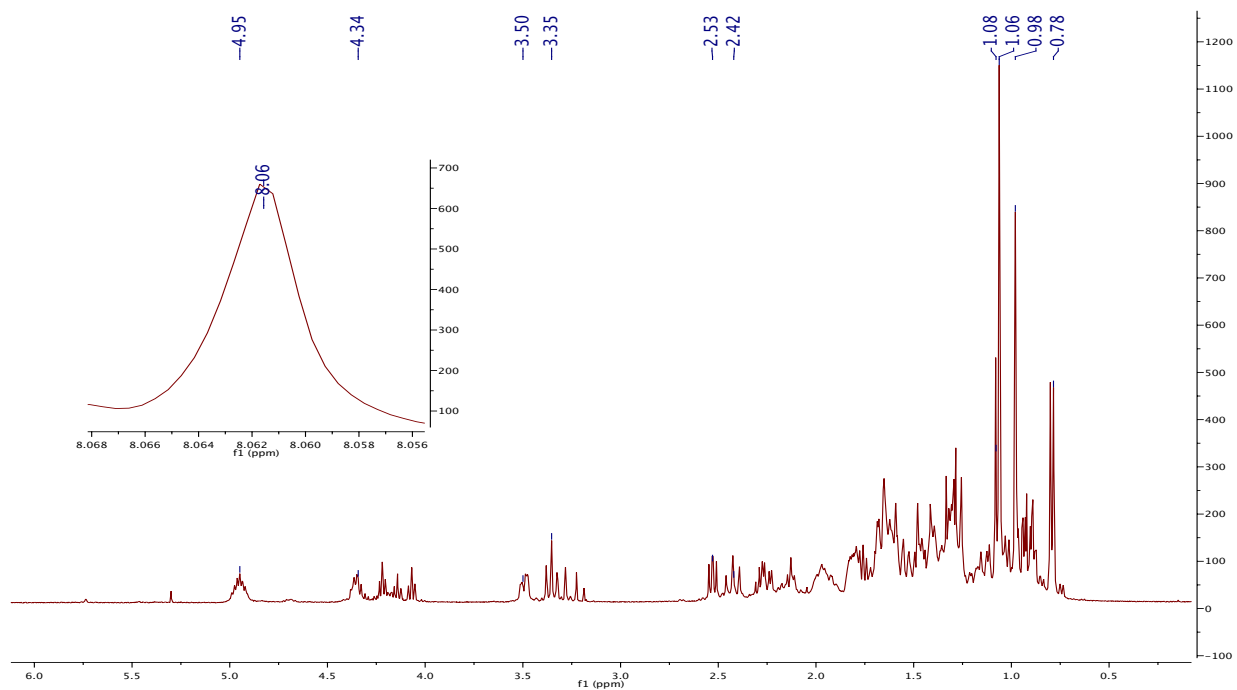


Figure S14. ¹H-NMR spectrum at 500 MHz in CDCl₃ of Bihecogenin ester (8b).

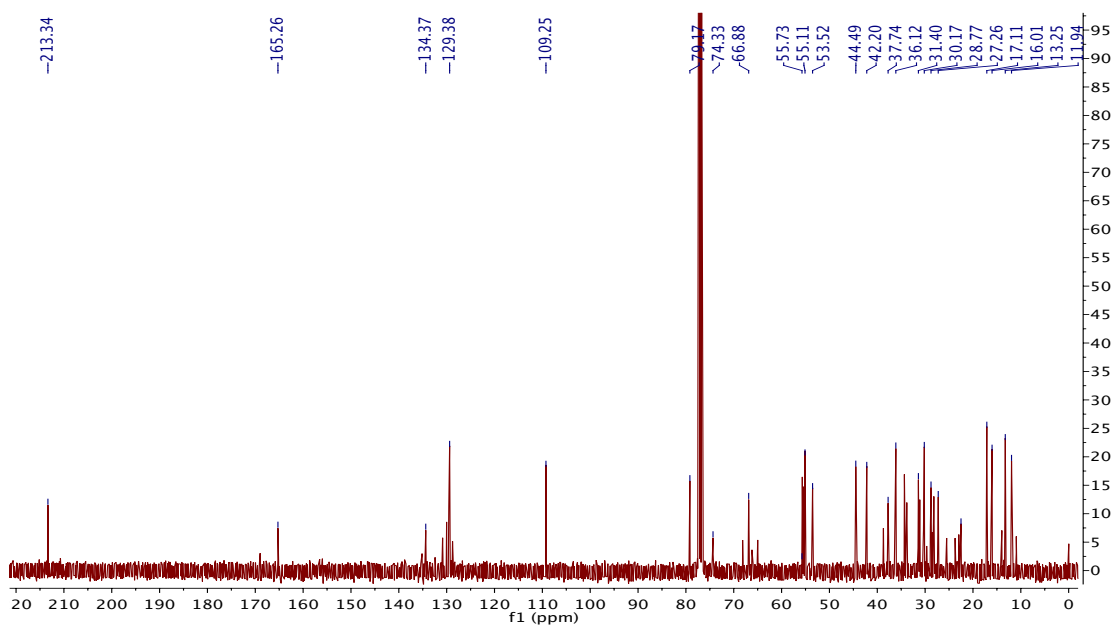


Figure S15. ¹³C-NMR spectrum at 125 MHz in CDCl₃ of Bihecogenin ester (8b).

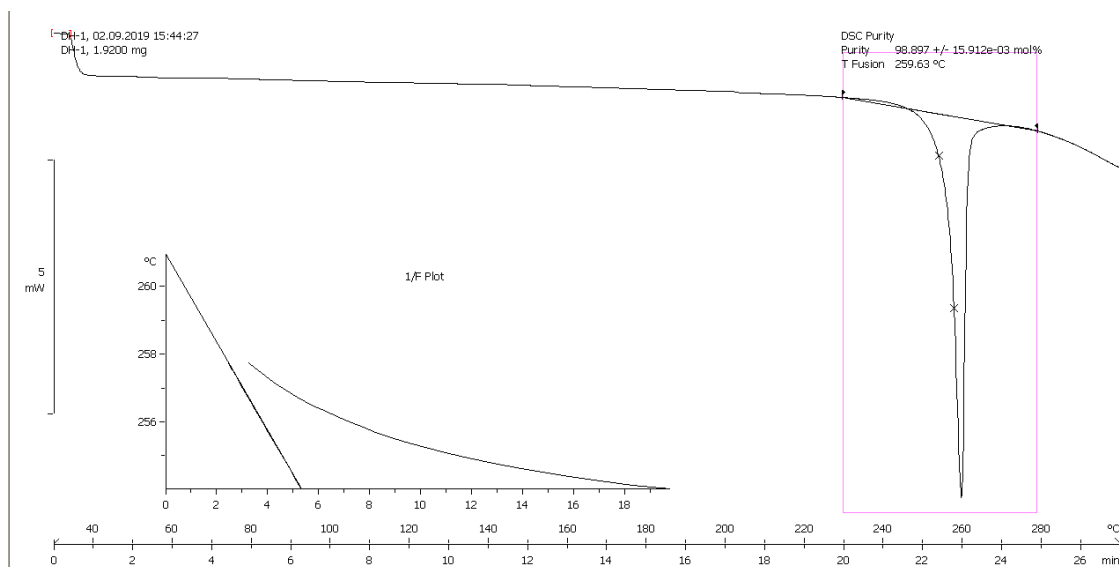


Figure S16. Differential Scanning Calorimetry analysis of Bihecogenin ester (**8b**).

Bisarsasapogenin terephthalate (**8c**)

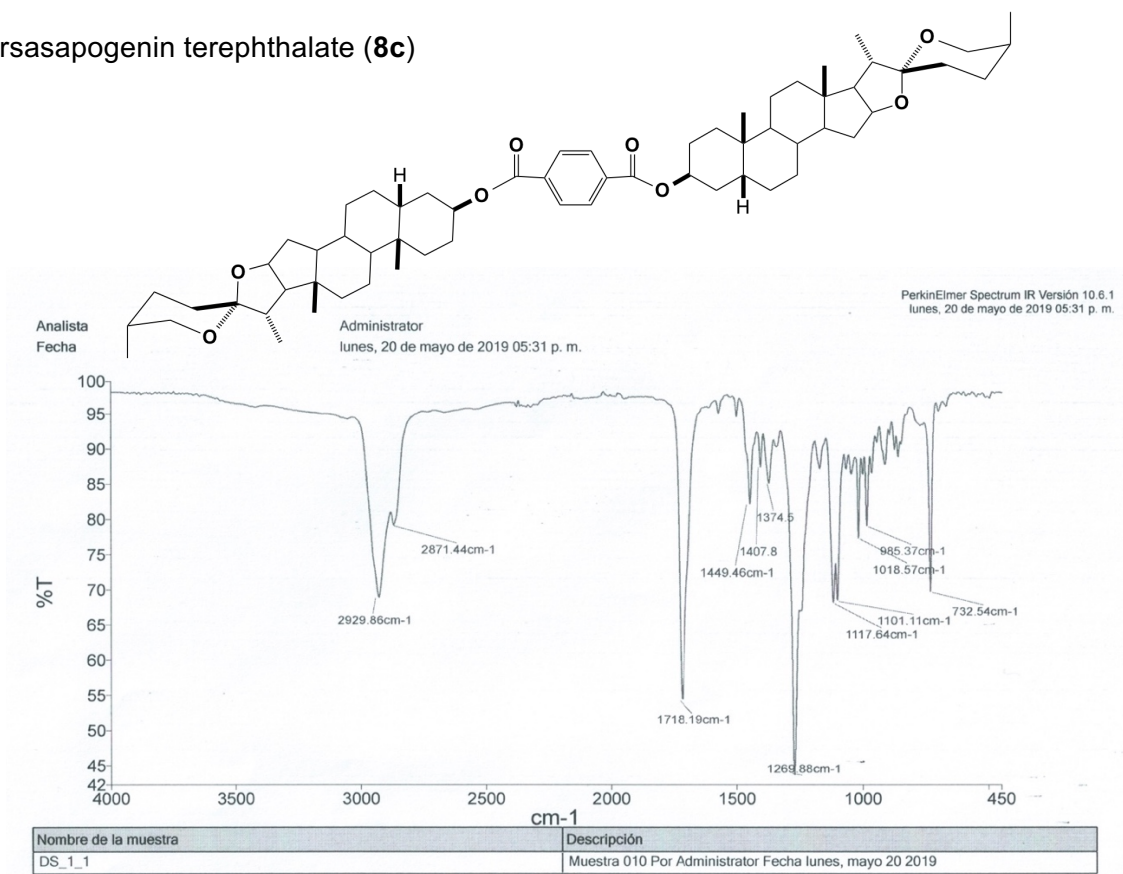


Figure S17. IR spectrum of Bisarsasapogenin ester (**8c**).

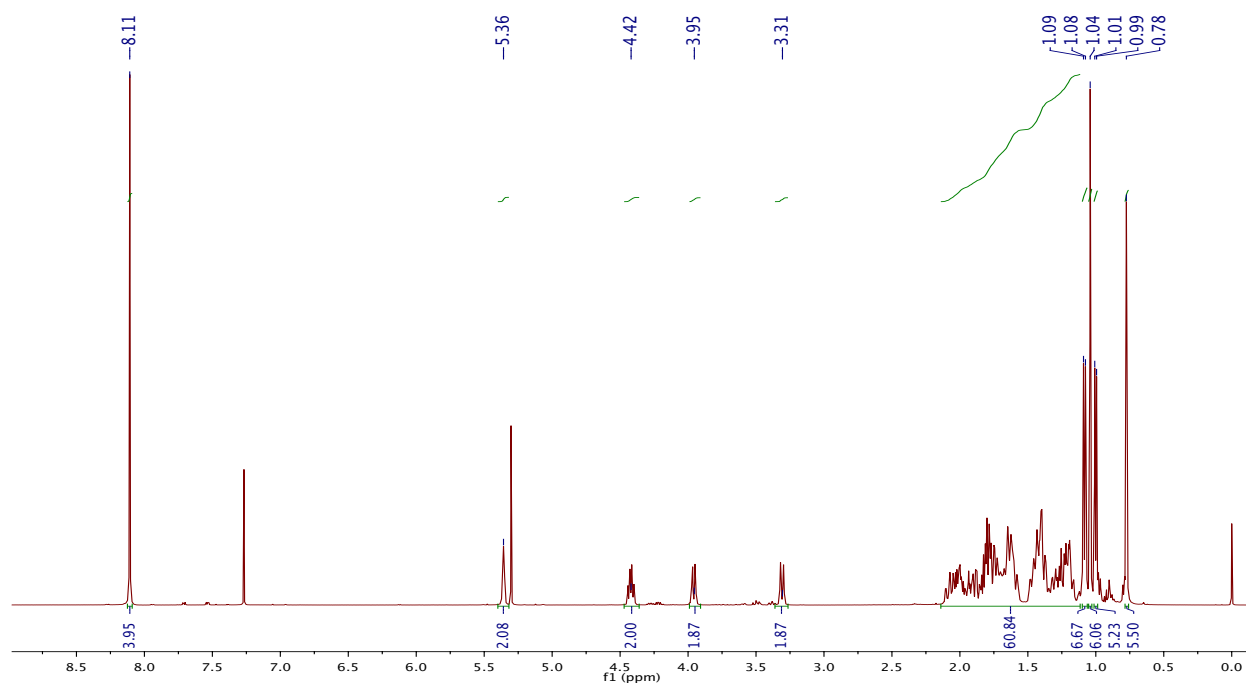


Figure S18. ¹H-NMR spectrum at 500 MHz in CDCl₃ of Bisarsasapogenin ester (**8c**).

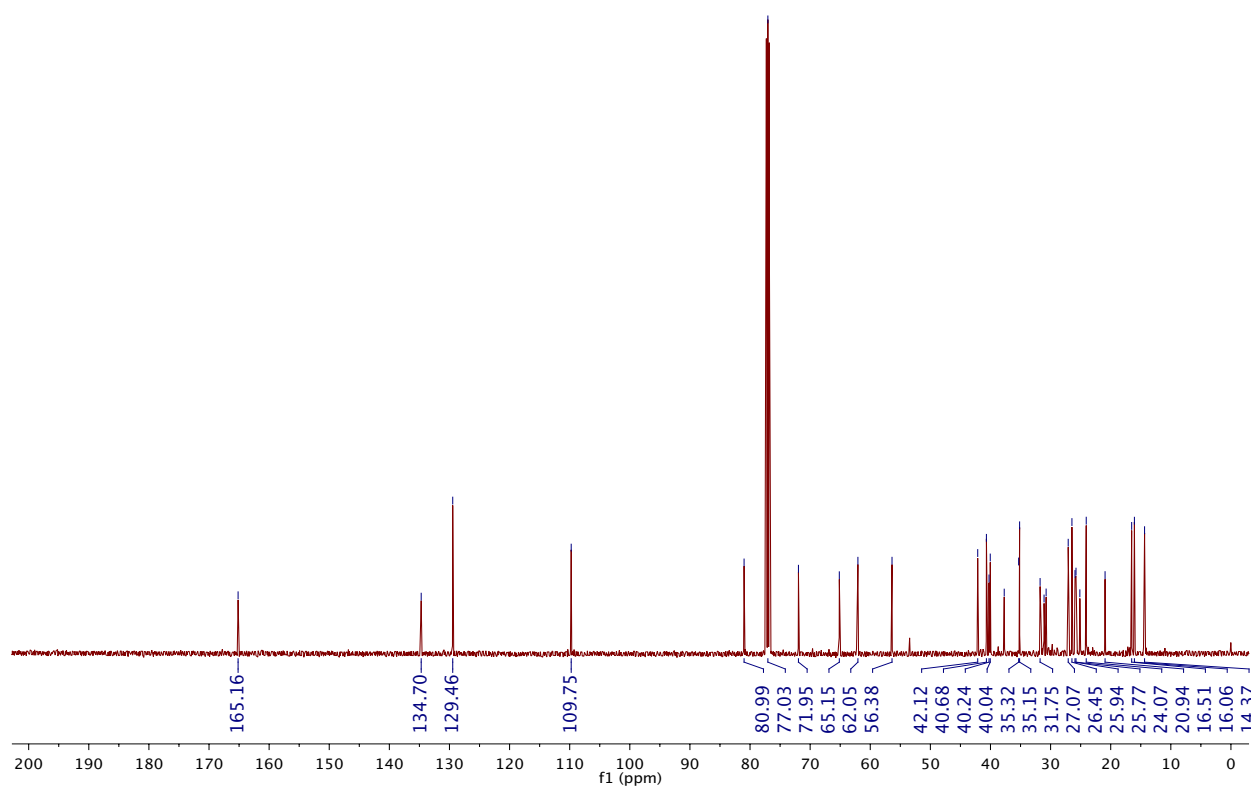


Figure S19. ¹³C-NMR spectrum at 125 MHz in CDCl₃ of Bisarsasapogenin ester (**8c**).

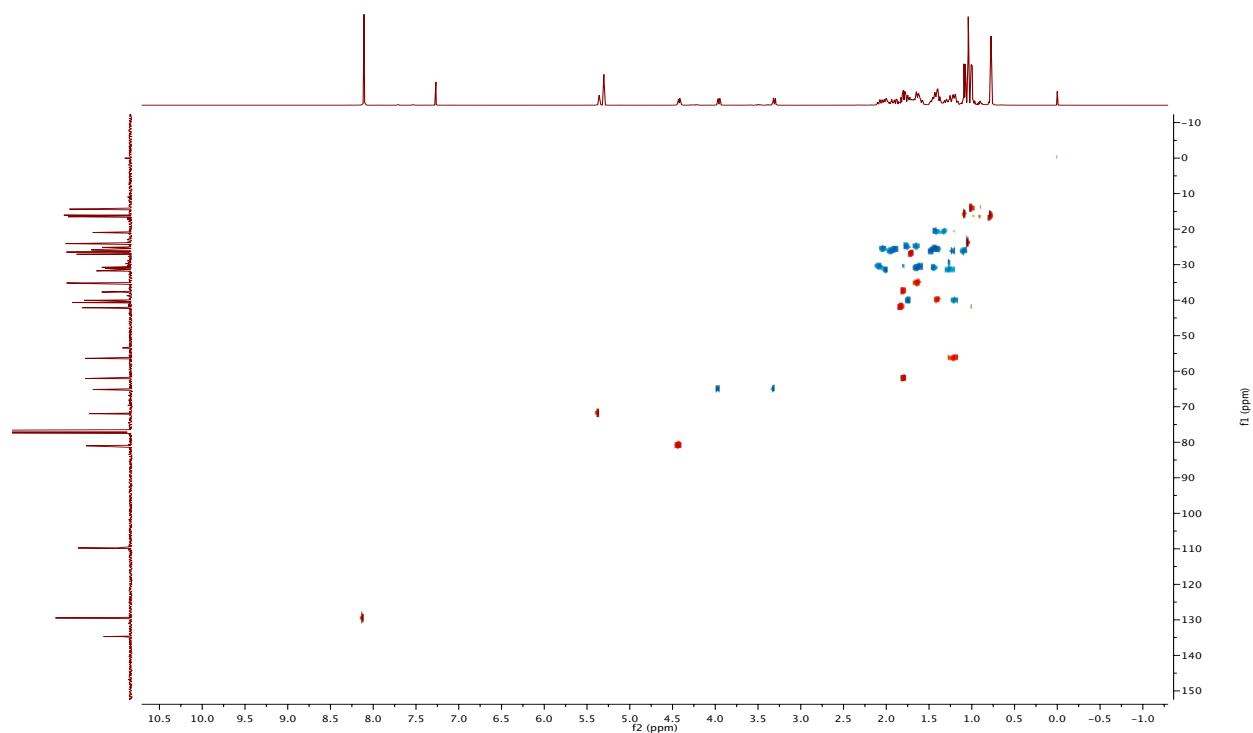


Figure S20. HSQC-NMR spectrum at 500 MHz in CDCl₃ of Bisarsasapogenin ester (**8c**).

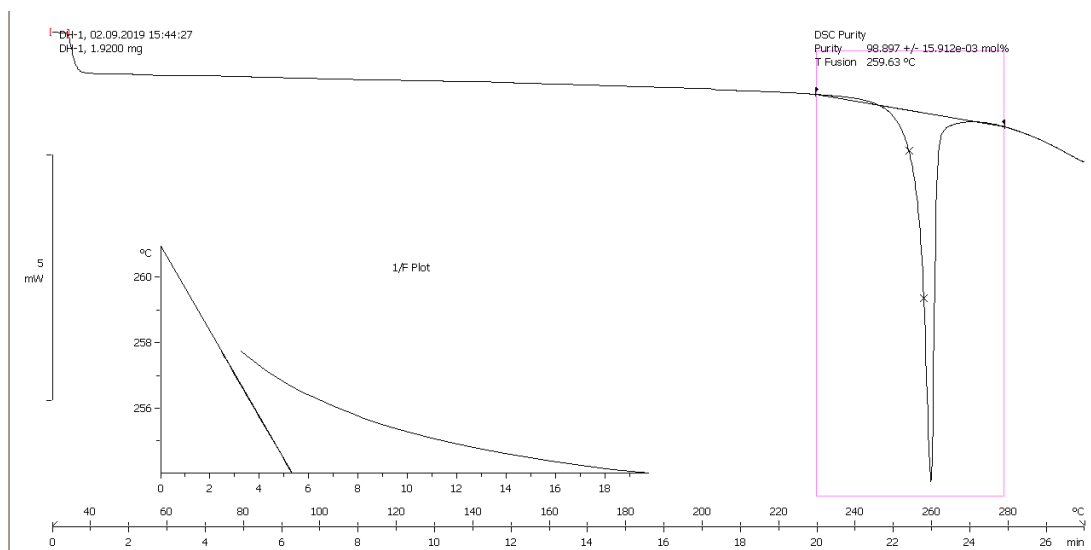


Figure S21. Differential Scanning Calorimetry analysis of Bisarsasapogenin ester (**8c**).

Bi-23-acetyldiosgenin terephthalate (**10a**).

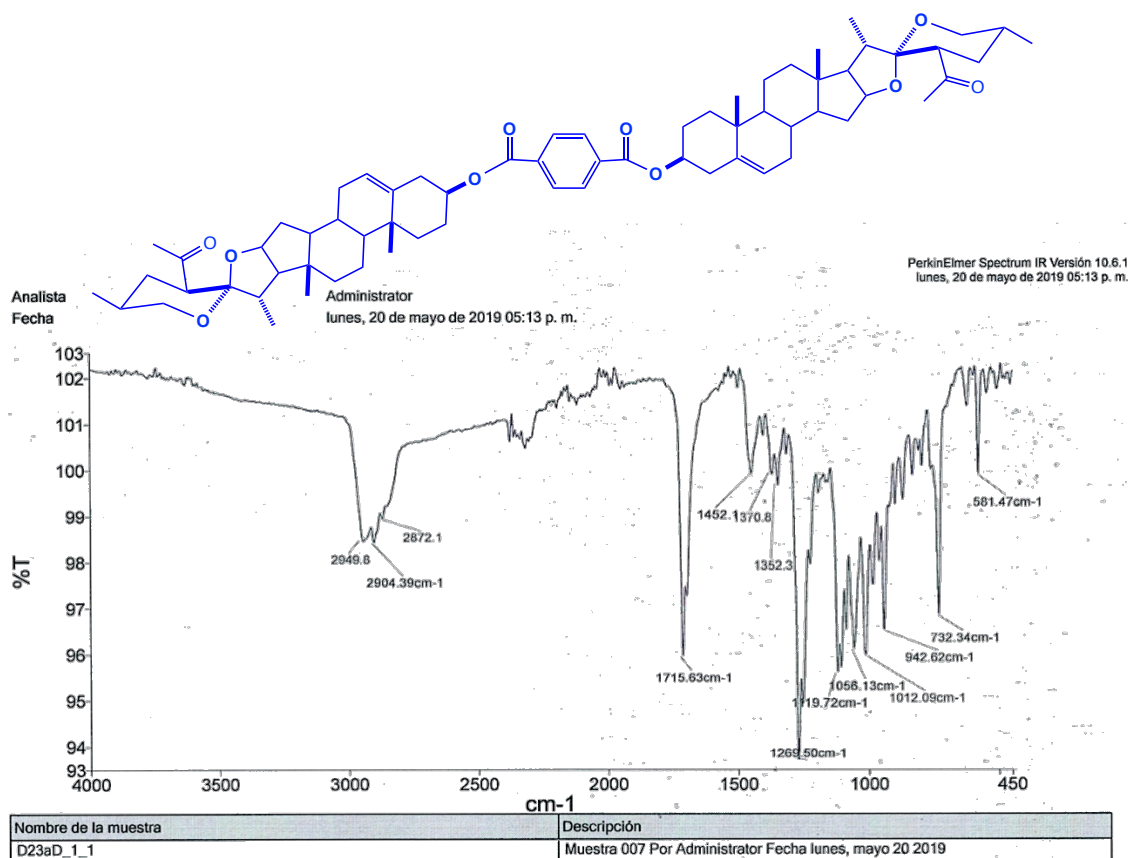


Figure S22. IR spectrum of Bi-23-acetyldiosgenin ester (**10a**).

[Elemental Composition]

Data : Dr-Jesus-Sandoval039

Date : 08-Oct-2010 13:20

Sample: 65-STE-2216 Lup-23aD

Note : Luis-Velasco

Inlet : Direct

Ion Mode : FAB+

RT : 9.15 min

Scan#: (65,76)

Elements : C 70/1, H 95/1, O 12/1

Mass Tolerance : 1000ppm, 2mmu if m/z > 2

Unsaturation (U.S.) : 0.0 - 50.0

Observed m/z	Int%				
1043.6609	41.2				
Estimated m/z	Error[ppm]	U.S.	C	H	O
1043.6612	-0.3	21.5	66	91	10

Figure S23. Mass spectrum data of Bi-23-acetyldiosgenin ester (**10a**).

tmpstudy_data_PROTON_01
MAF-D23aD

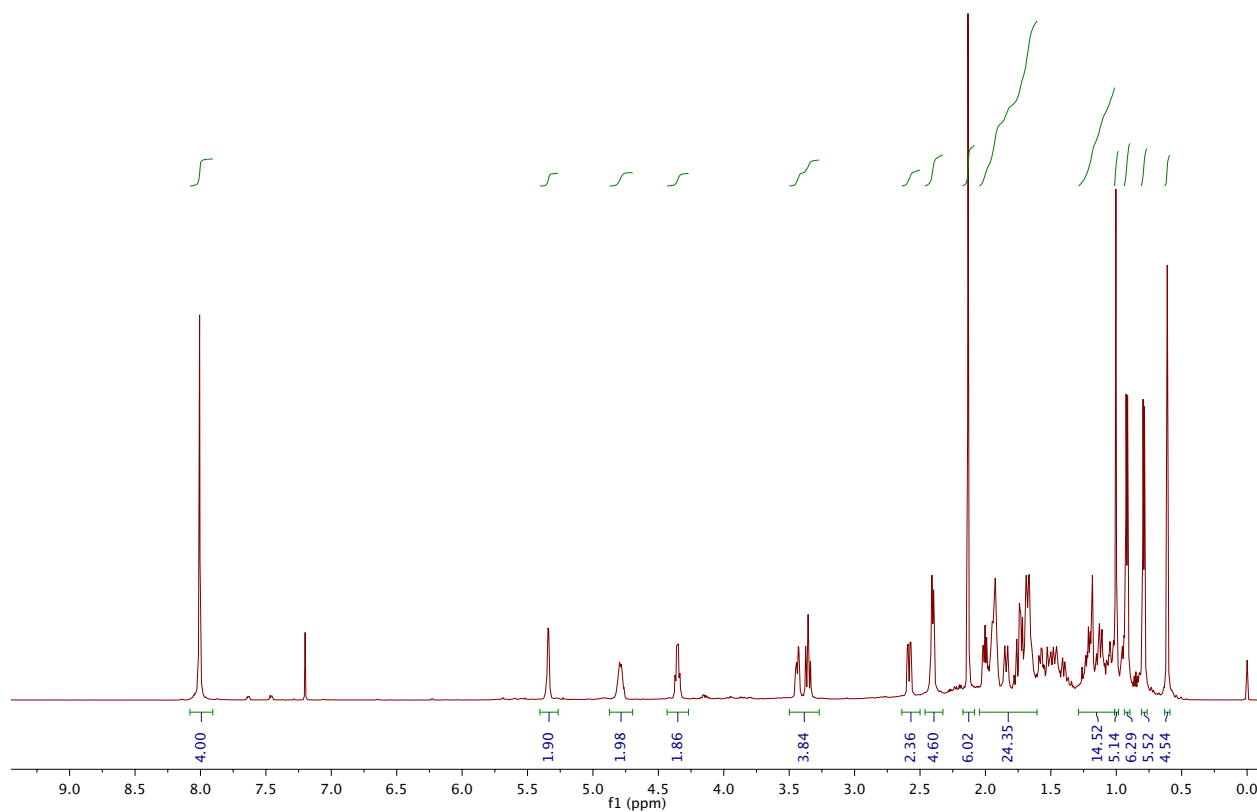


Figure S24. ^1H -NMR spectrum at 600 MHz in CDCl_3 of Bi-23-acetyldiosgenin ester (**10a**).

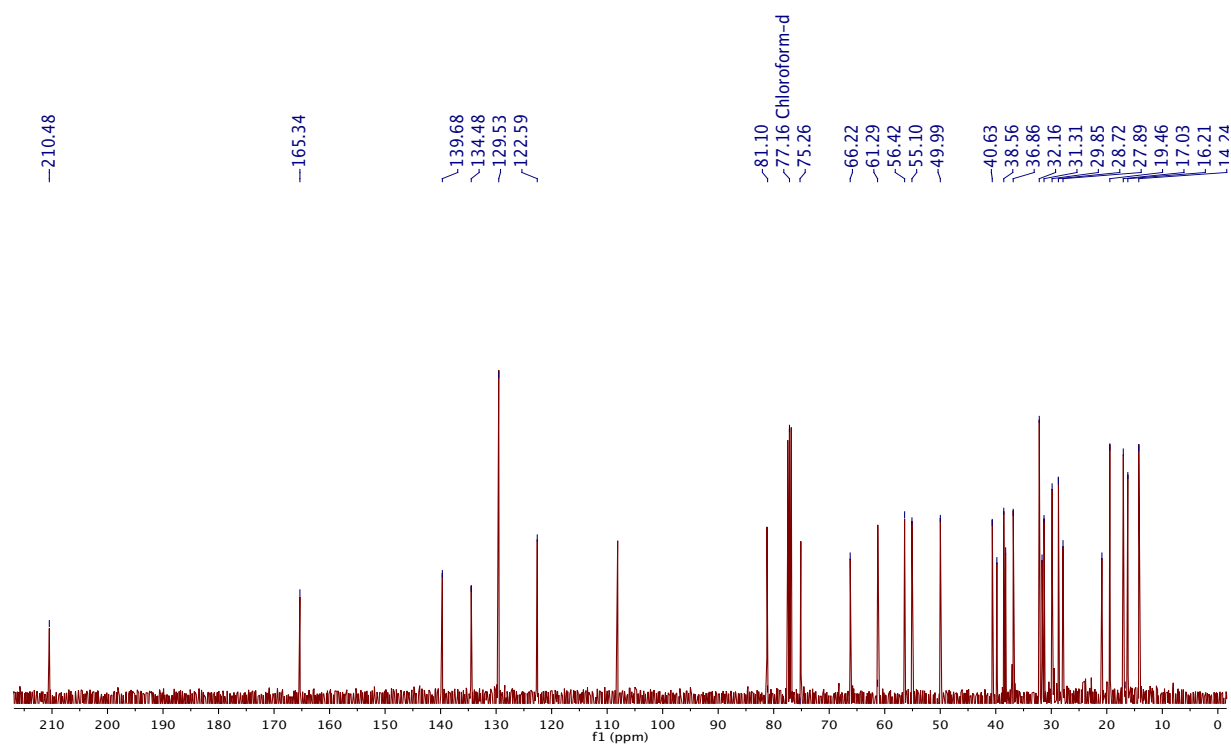


Figure S25. ^{13}C -NMR spectrum at 150 MHz in CDCl_3 of Bi-23-acetyldiosgenin ester (**10a**).

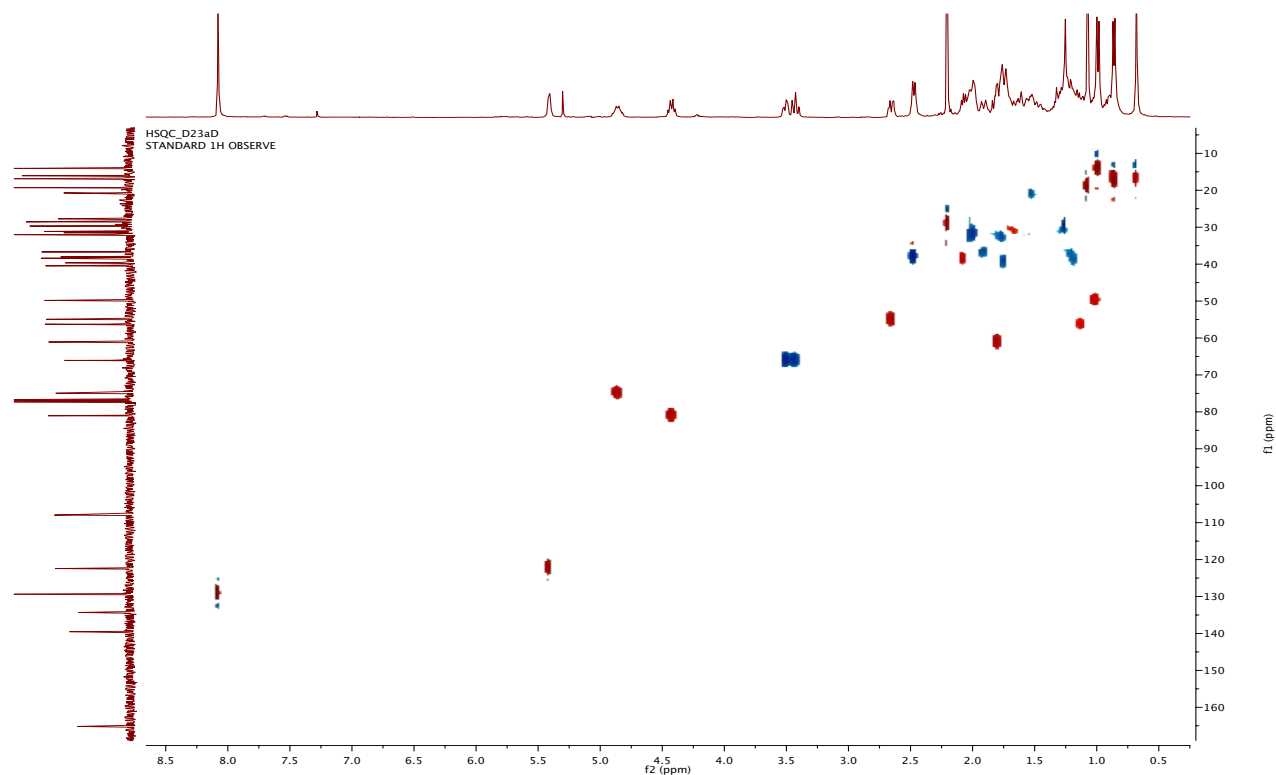


Figure S26. HSQC-NMR spectrum at 500 MHz in CDCl_3 of Bi-23-acetyldiosgenin ester (**10a**).

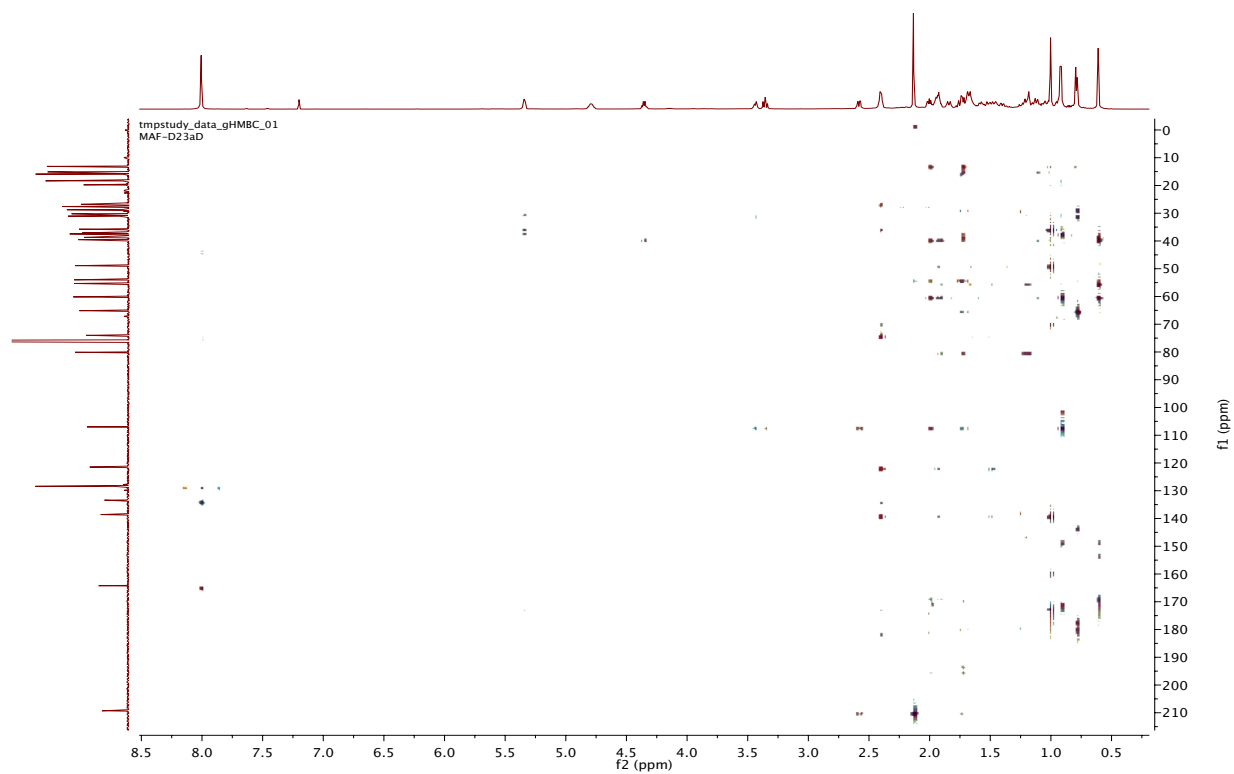


Figure S27. HMBC-NMR experiment at 600 MHz in CDCl_3 of Bi-23-acetyldiosgenin ester (**10a**).

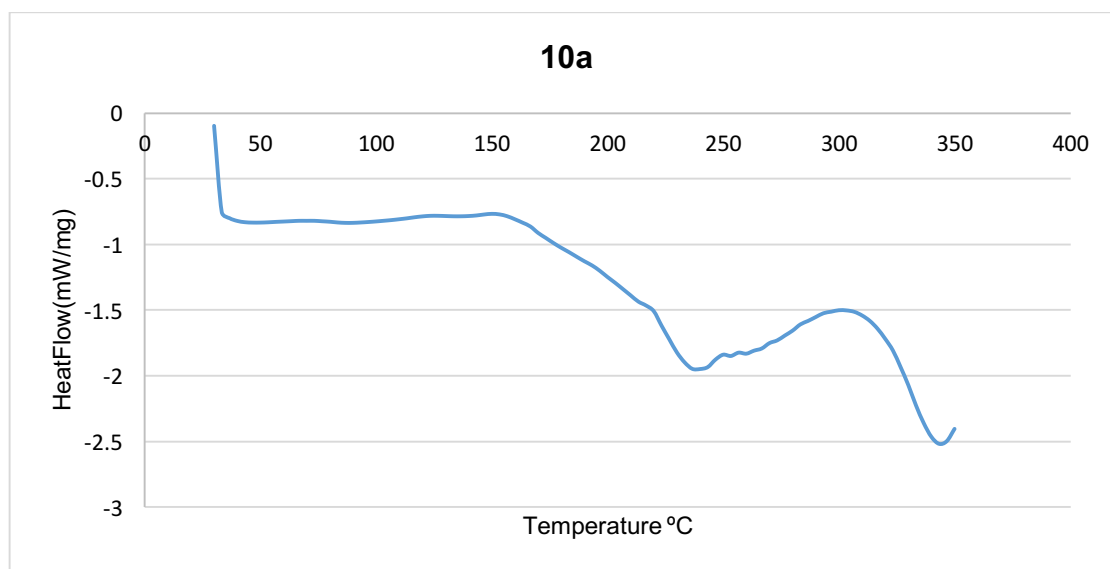


Figure S28. Differential Scanning Calorimetry analysis of Bi-23-acetyldiosgenin ester (**10a**).

Bi-23-acetylhecogenin terephthalate (**10b**).

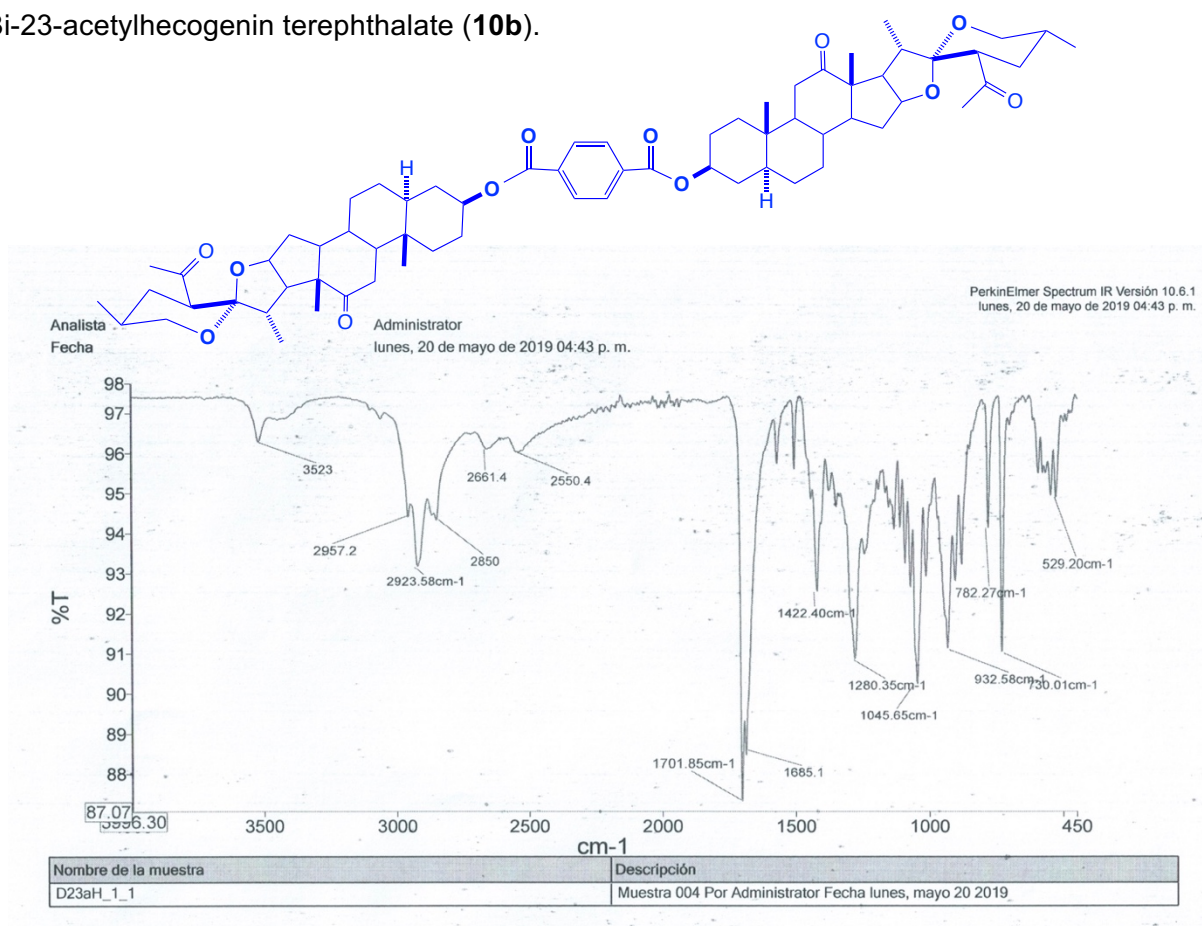


Figure S29. IR spectrum of Bi-23-acetylhecogenin ester (**10b**).

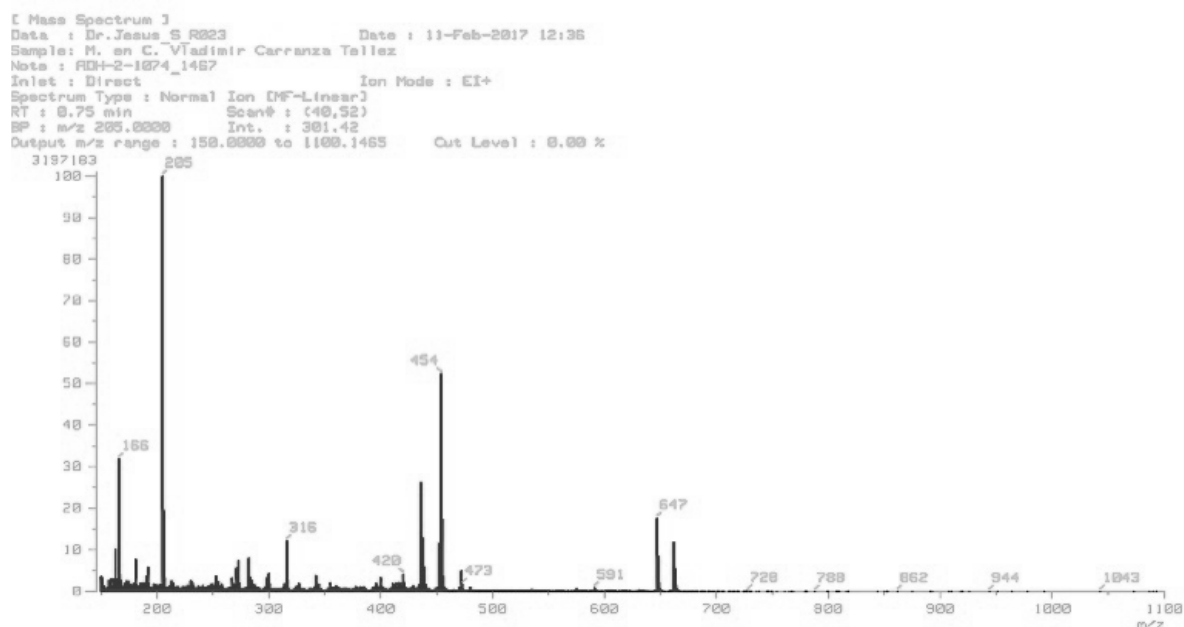


Figure S30. Mass spectrum of Bi-23-acetylhecogenin ester (**10b**).

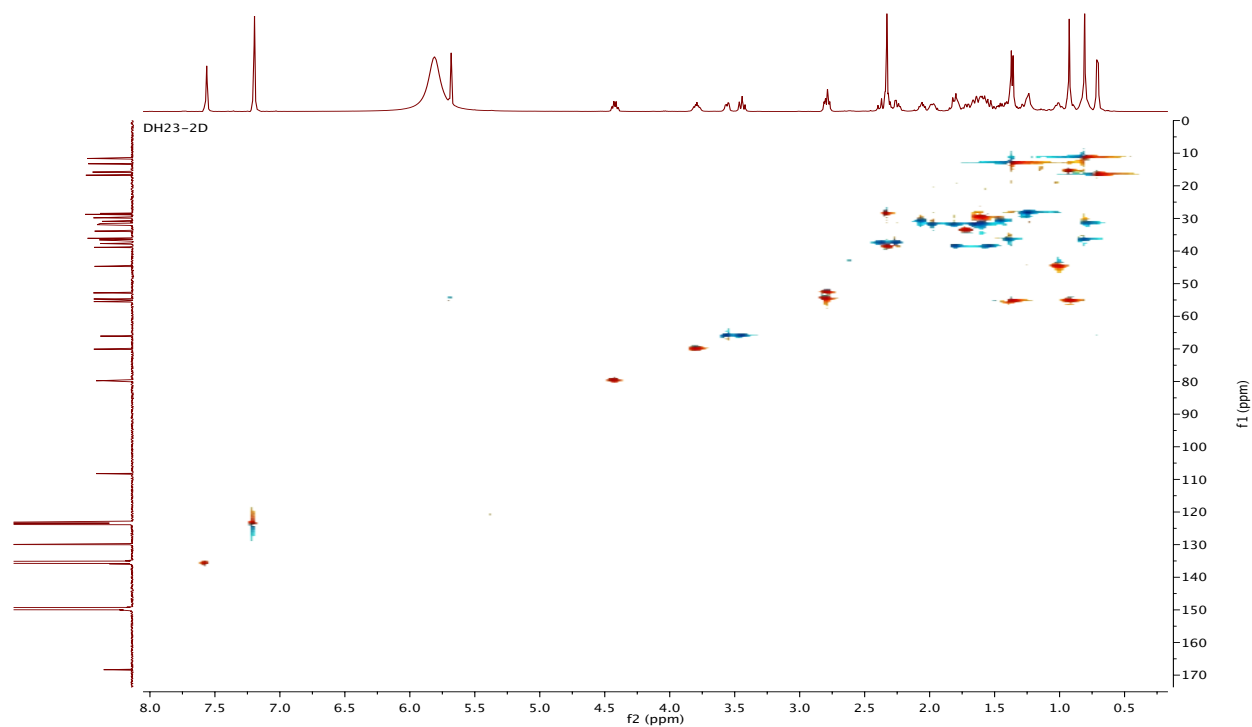


Figure S31. HSQC-NMR spectrum at 500 MHz in CDCl_3 of Bi-23-acetylhecogenin ester (**10b**).

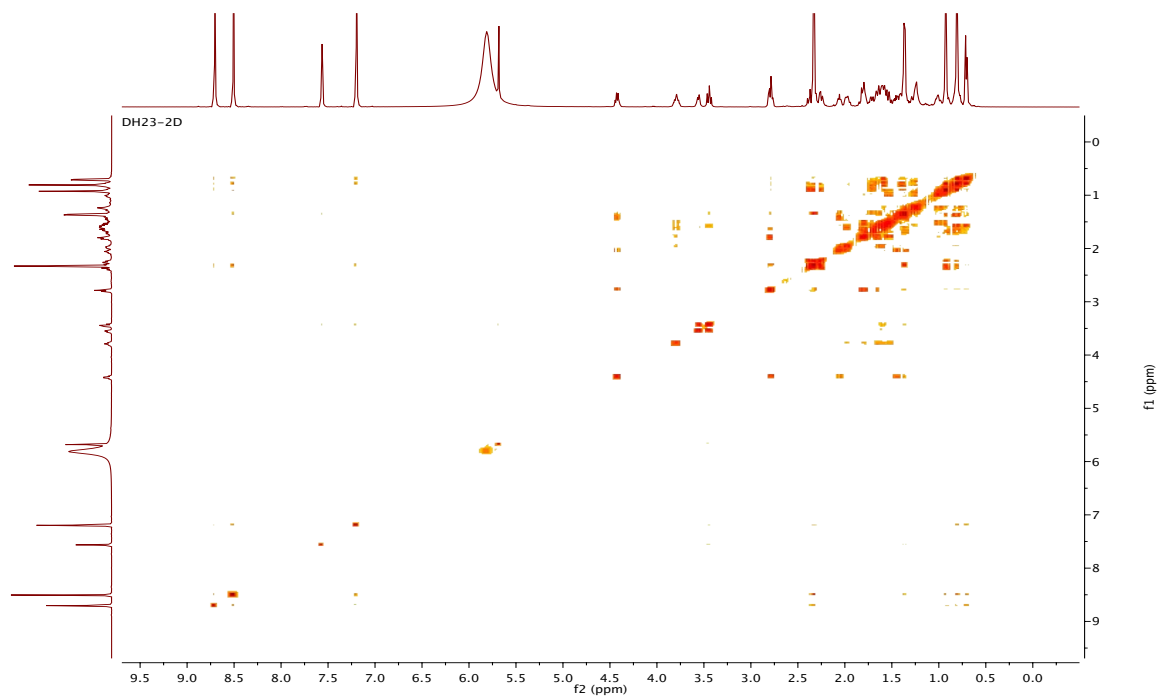


Figure S32. COSY-NMR spectrum at 500 MHz in CDCl_3 of Bi-23-acetylhecogenin ester (**10b**).

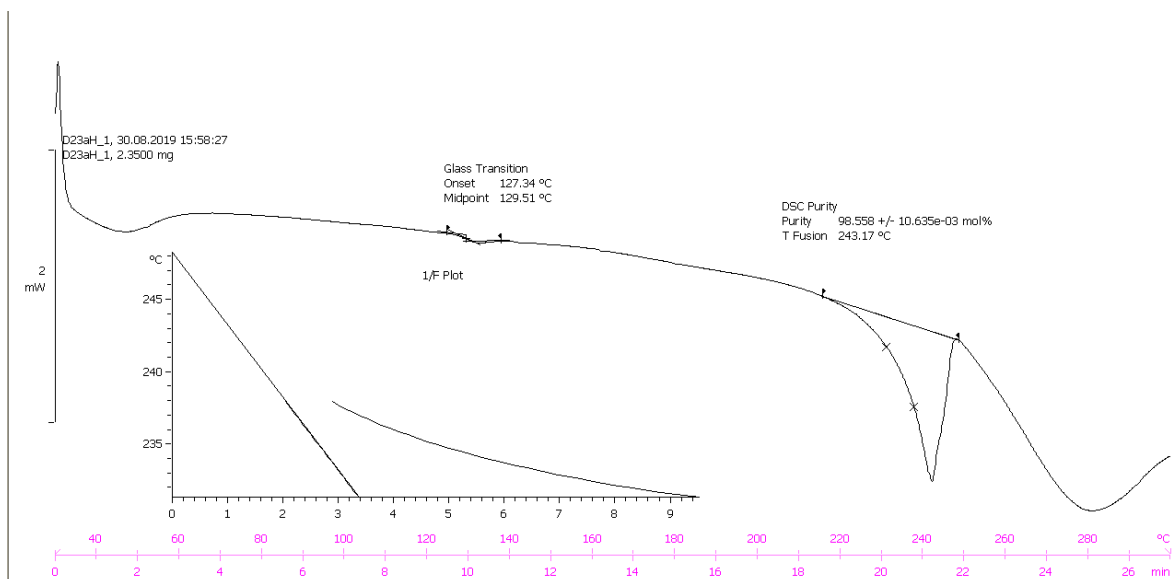


Figure S33. Differential Scanning Calorimetry analysis of Bi-23-acetylhecogenin ester (**10b**).

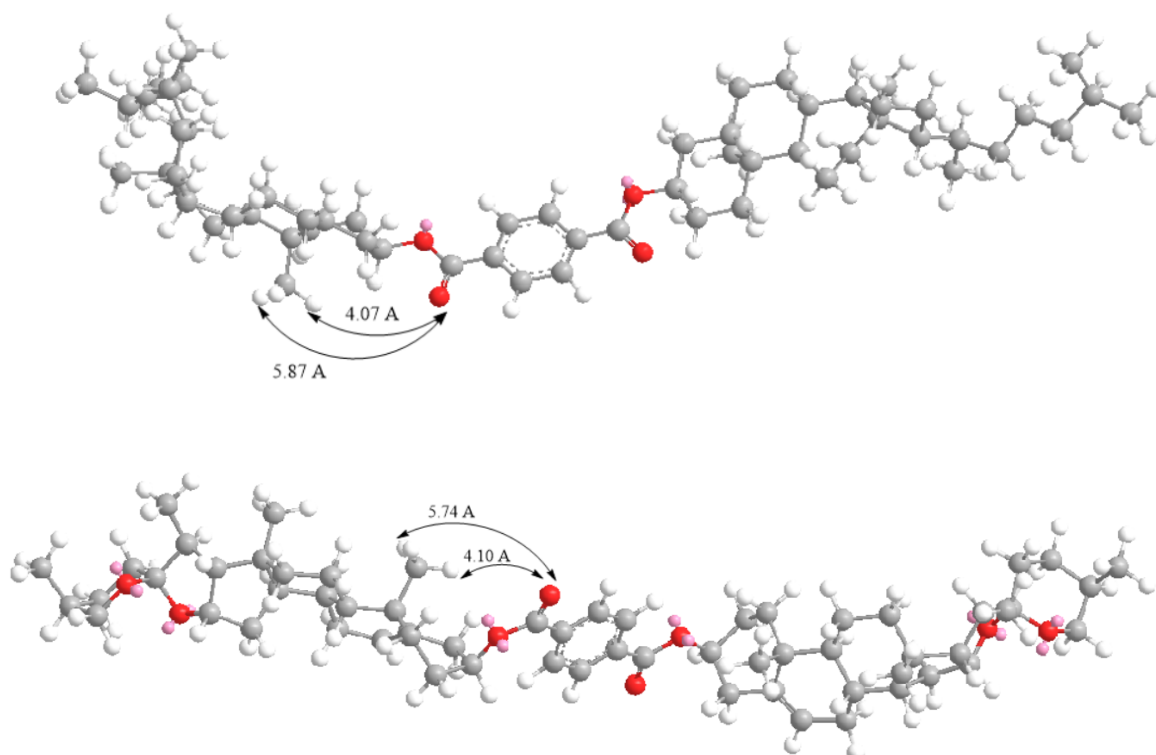


Figure S34. Molecular structure with MM2 energy minimization method for series: 5 α (**6**, top) and 5 β (**8c**, down).

Scanning Electron Microscopy (SEM) Images.

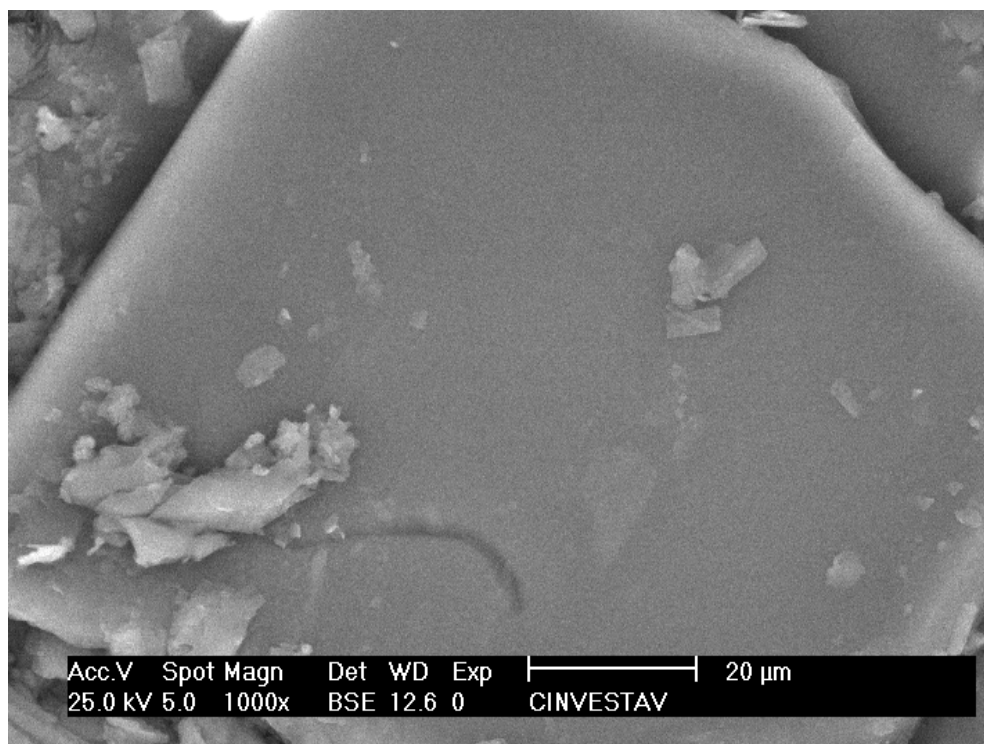


Figure S35 Bicholesterol ester (**5**) in hexane/ EtOAc showing membrane-shaped structures.

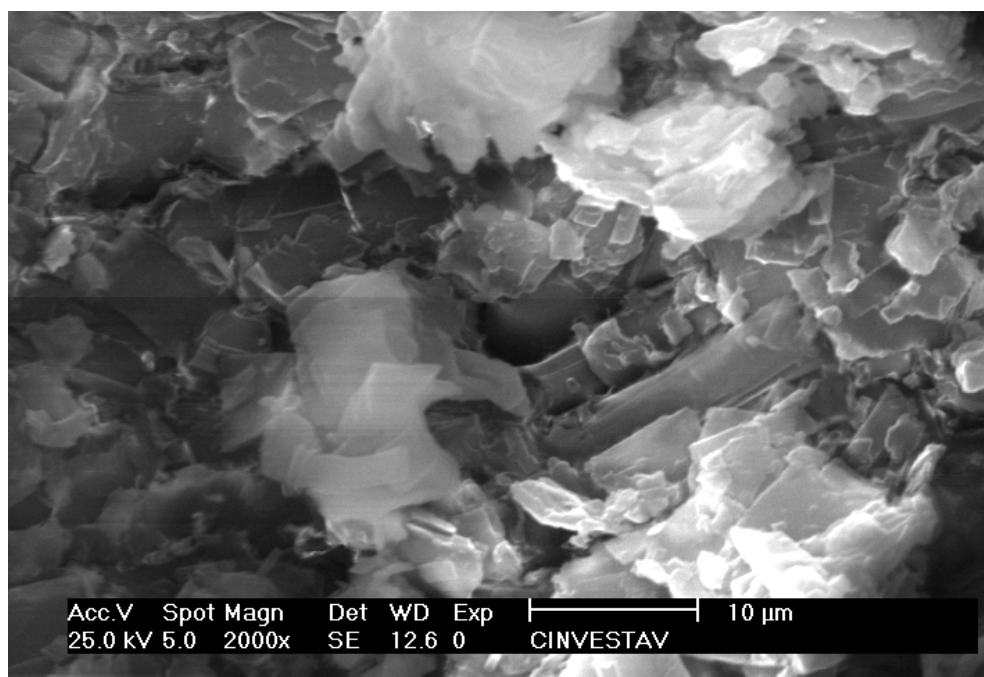


Figure S36 Bicholesterol ester (**5**) in hexane/ EtOAc showing membrane-shaped structures.

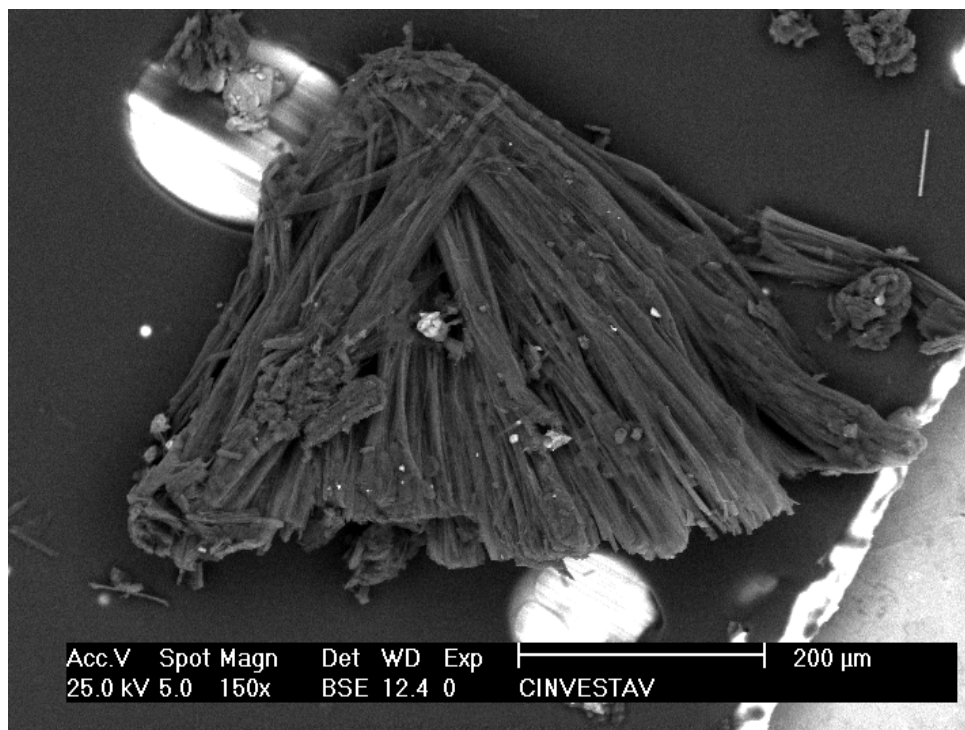


Figure S37. Bicholesterol ester (**5**) EtOAc showing strand-shaped structures.

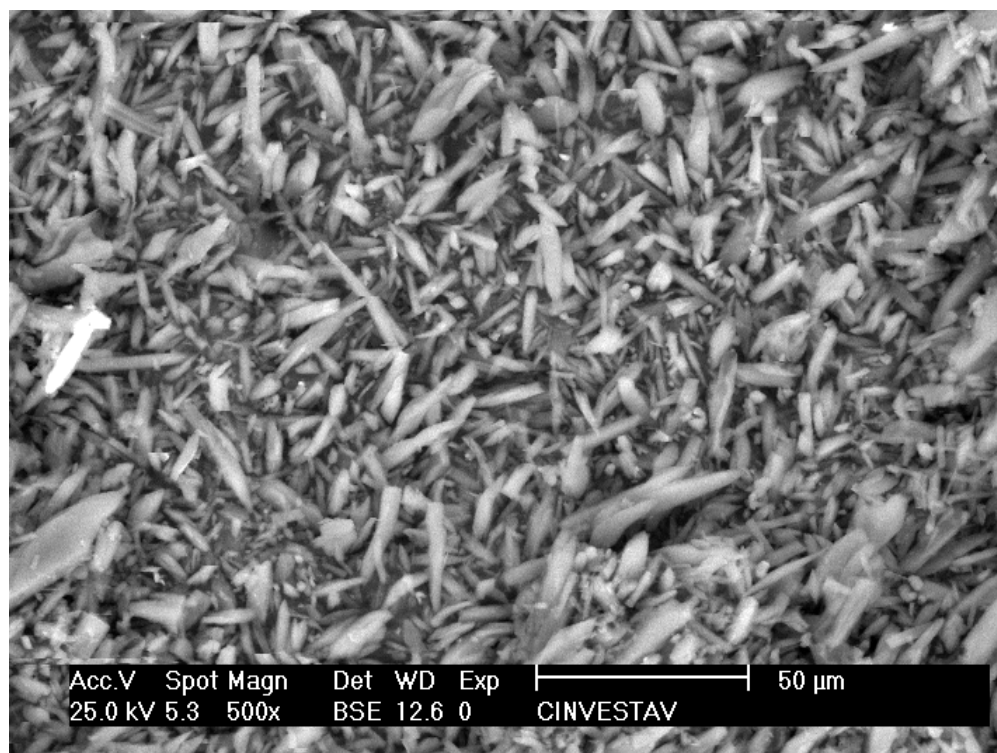


Figure S38. Bidiosgenin ester (**8a**) in EtOAc showing strand-shaped structures.

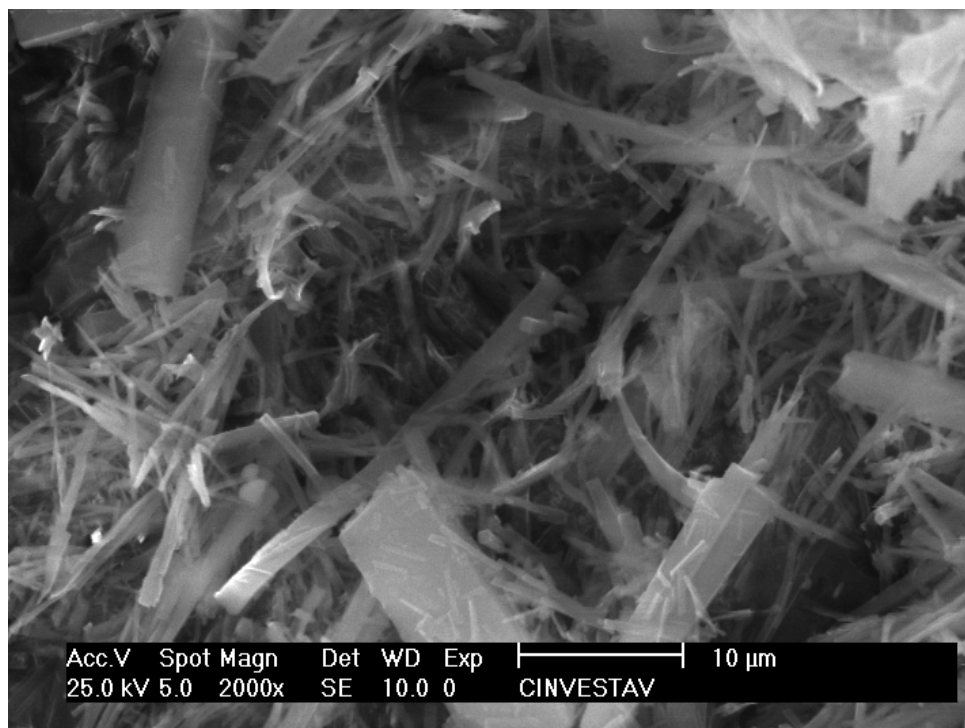


Figure S39. Bidiosgenin ester (**8a**) in $\text{CHCl}_3/\text{MeOH}$.



Figure S40. Bidiosgenin ester (**8a**) in $\text{CHCl}_3/\text{MeOH}$

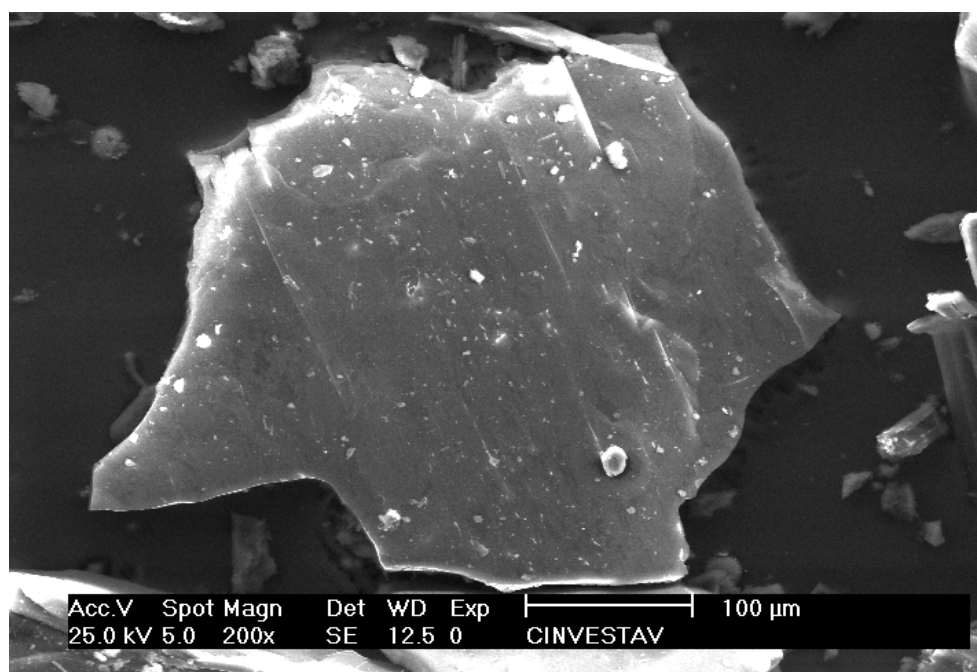


Figure S41. Bisarsasapogenin ester (**8c**) in hexane/EtOAc showing membrane-shaped structures.

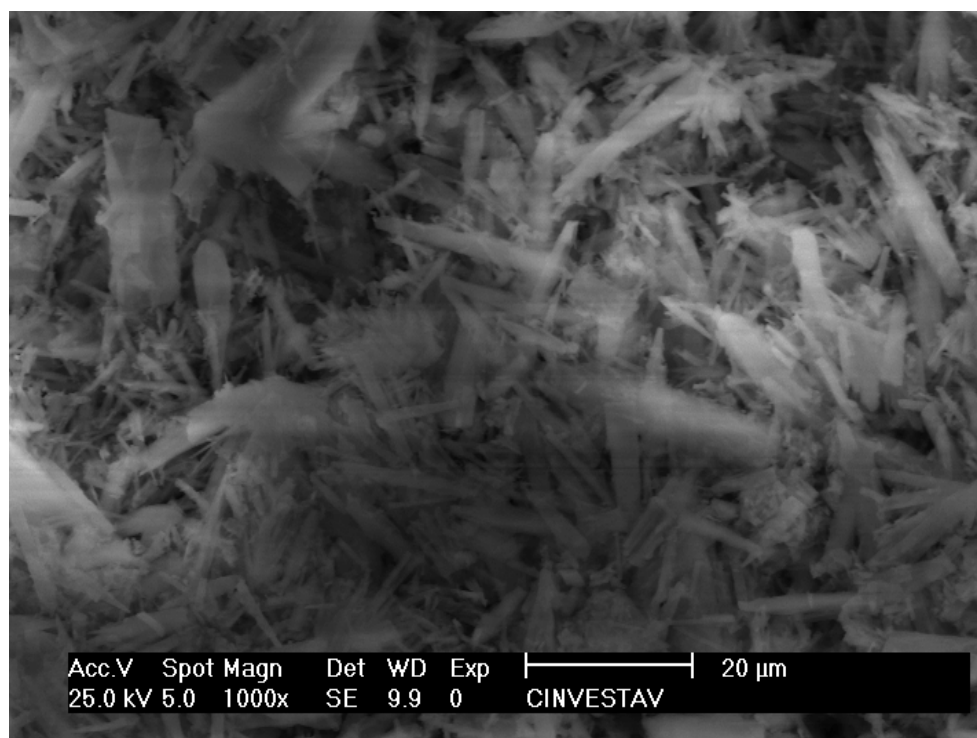


Figure S42. Bi-23-acetyldiosgenin ester (**10a**) in EtOAc showing strand-shaped structures.

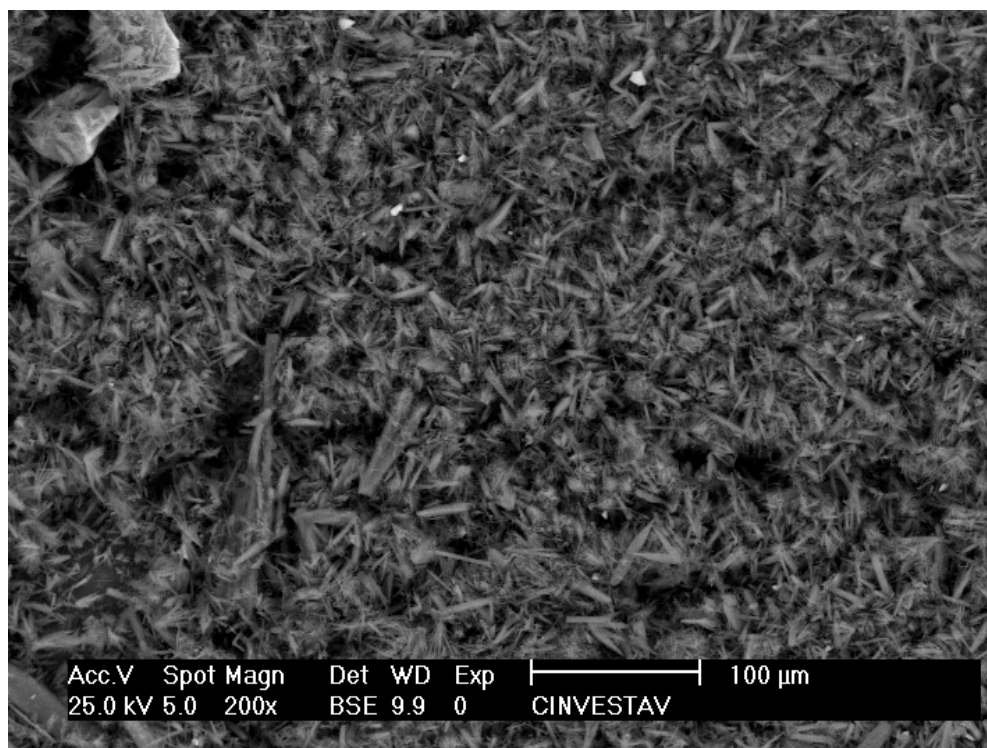


Figure S43. Bi-23-acetyldiosgenin ester (**10a**) in EtOAc showing strand-shaped structures.

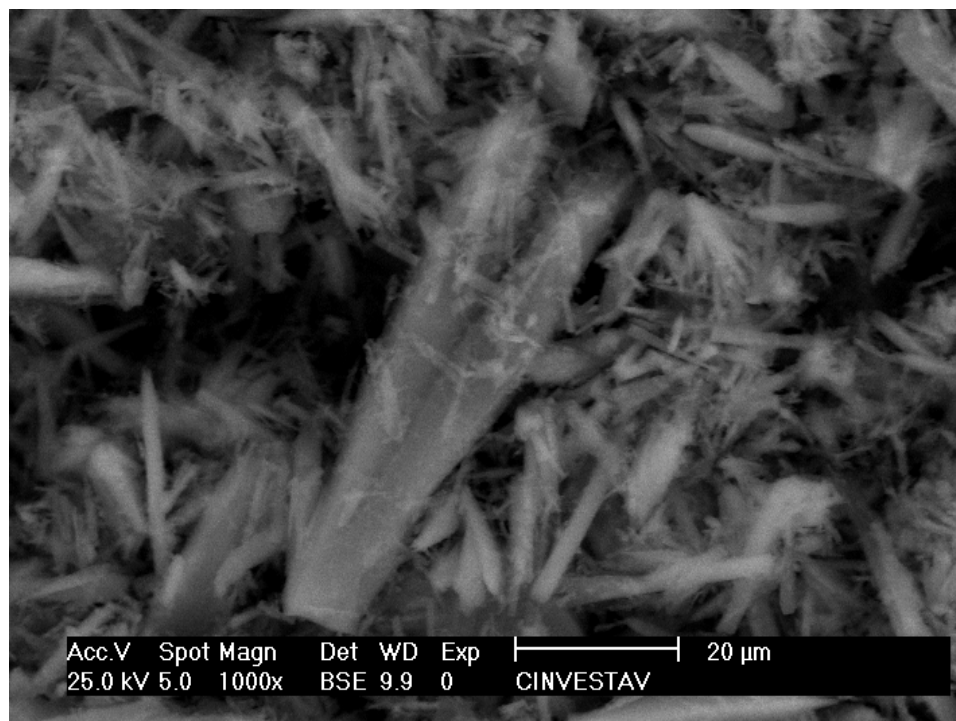


Figure S44. Bi-23-acetyldiosgenin ester (**10a**) in EtOAc showing strand-shaped structures.

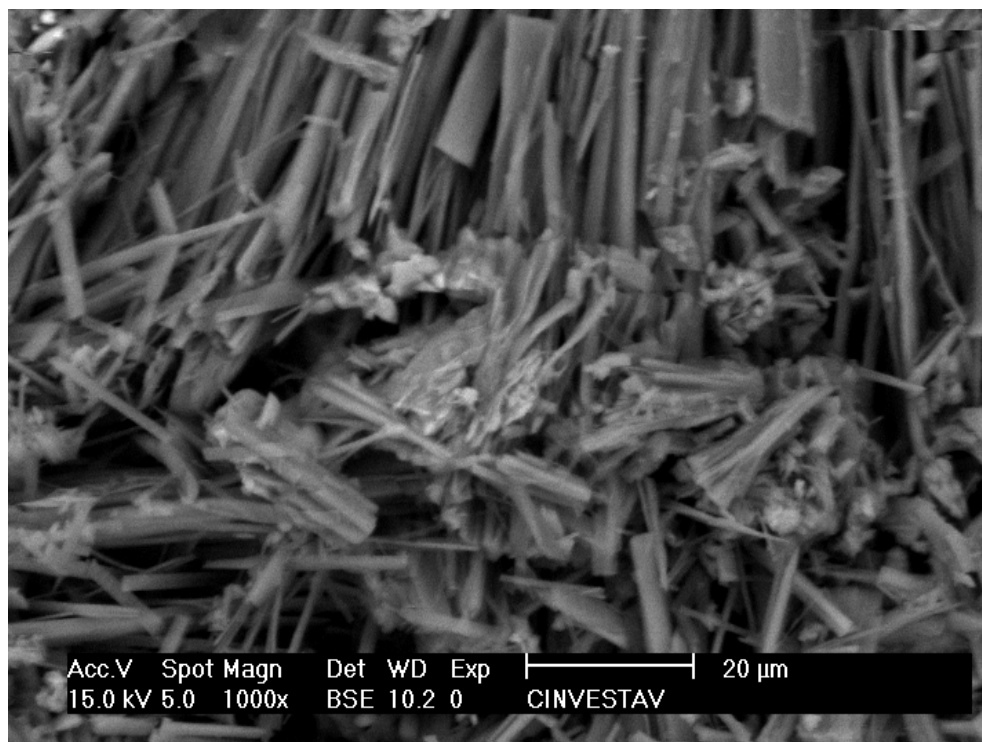


Figure S45. Bi-23-acetylhecogenin ester (**10b**) in EtOAc showing strand-shaped structures.

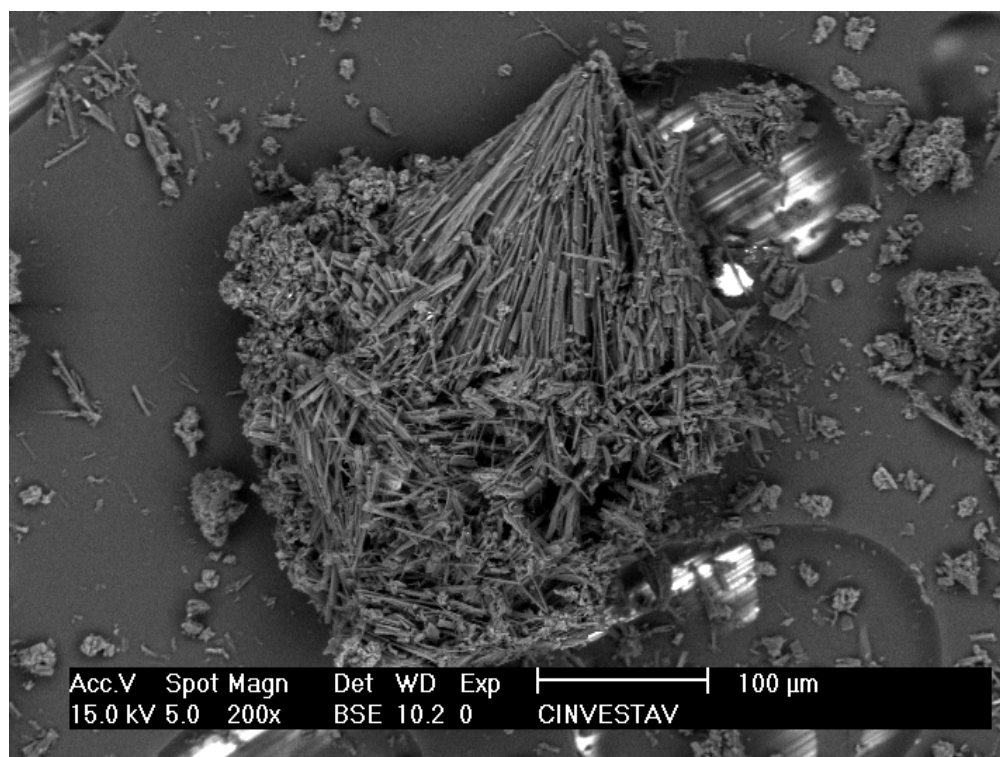


Figure S46. Bi-23-acetylhecogenin ester (**10b**) in EtOAc showing strand-shaped structures.

Powder X-ray diffraction (PXRD) analysis.

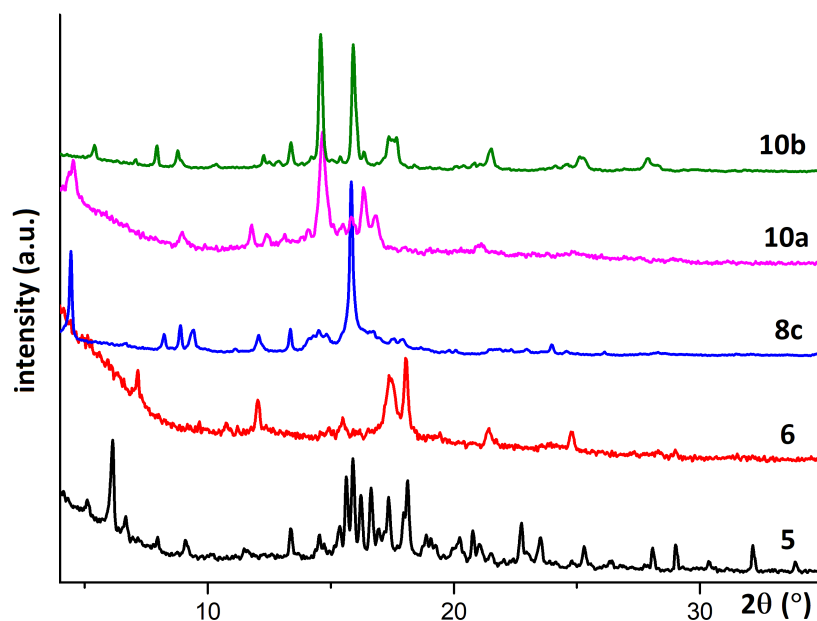


Figure S47. Powder X-ray diffraction patterns for raw materials of steroidal dimers **5**, **6**, **8c**, **10a** and **10b**. Patterns were collected with the Cu- $K\alpha$ radiation, and are uncorrected for amorphous contribution.

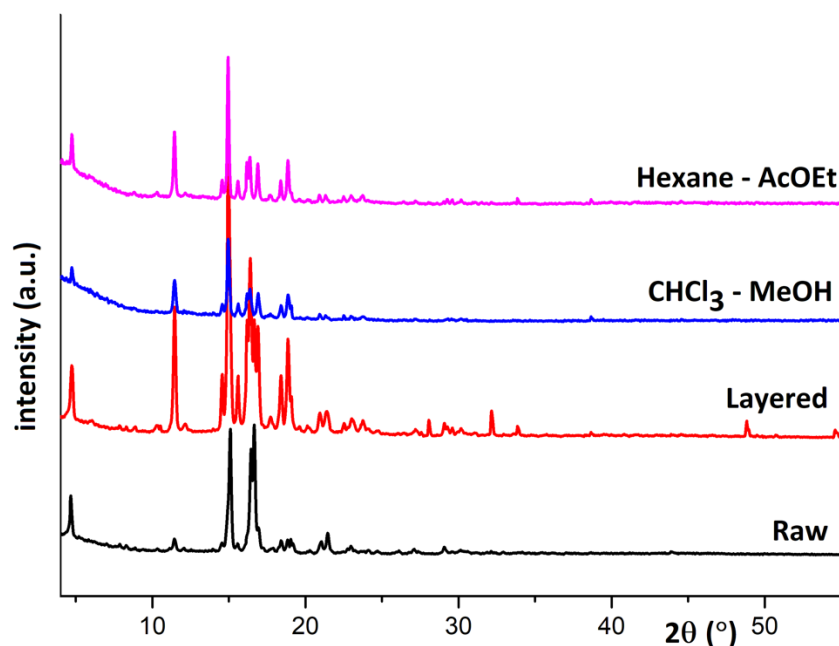


Figure S48. Comparison of the X-ray diffraction patterns of bidiosgenin ester (**8a**) under different conditions: raw material, layered material, dimer in contact with chloroform-methanol, and with hexane-ethyl acetate. Patterns were collected with the Cu- $K\alpha$ radiation, and are uncorrected for amorphous contribution. Note the intensity variation for the peak at $2\theta = 11.5^\circ$, as a consequence of the self-organization of the steroidal dimer.