

Supplementary information

Physalins V-IX, 16,24-cyclo-13,14-seco withanolides from *Physalis angulata* and their antiproliferative and anti-inflammatory activities

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Li-Xia Chen^{2*}, Feng Qiu^{1,2*}

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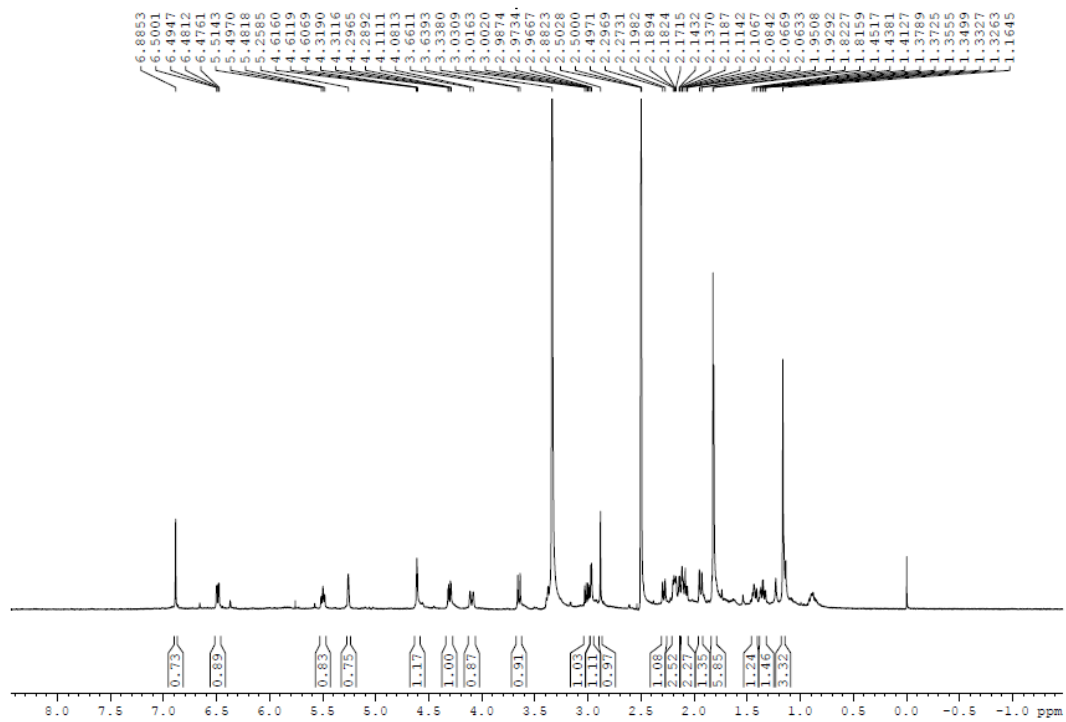
Supplementary Figure S50. HMBC spectrum of **6** (600 MHz, DMSO-*d*₆)

Supplementary Figure S51. Selected HMBC spectrum of **6** (600 MHz, DMSO-*d*₆)

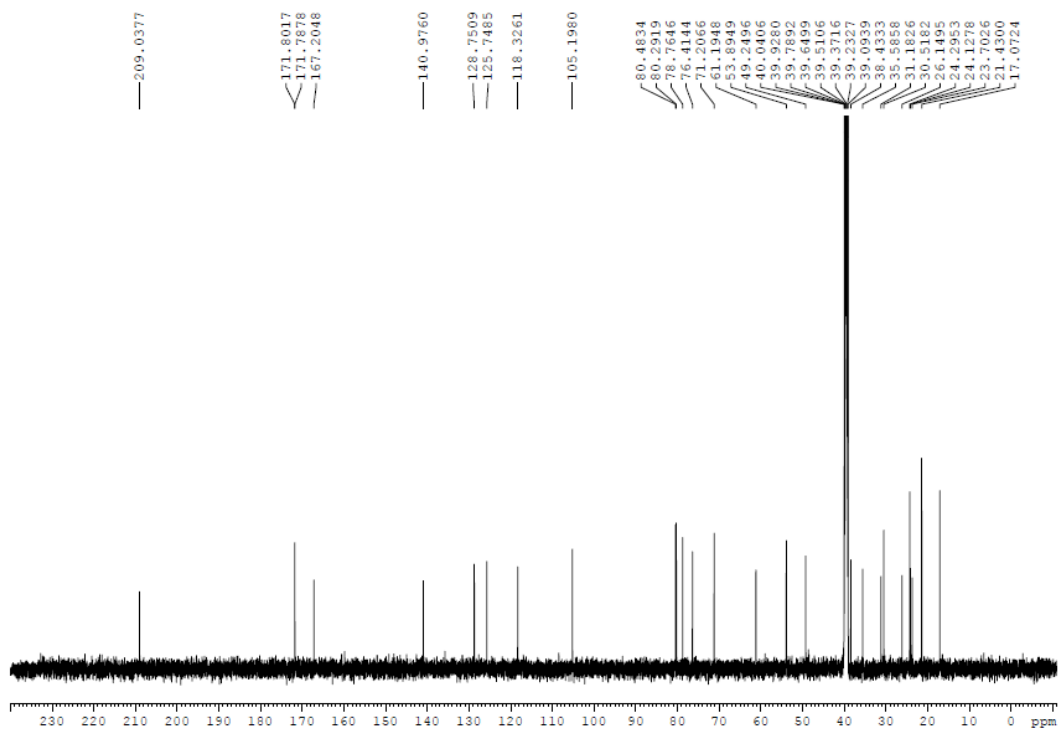
Supplementary Figure S52. NOESY spectrum of **6** (600 MHz, DMSO-*d*₆)

Supplementary Figure S53. Selected NOESY spectrum of **6** (600 MHz, DMSO-*d*₆)

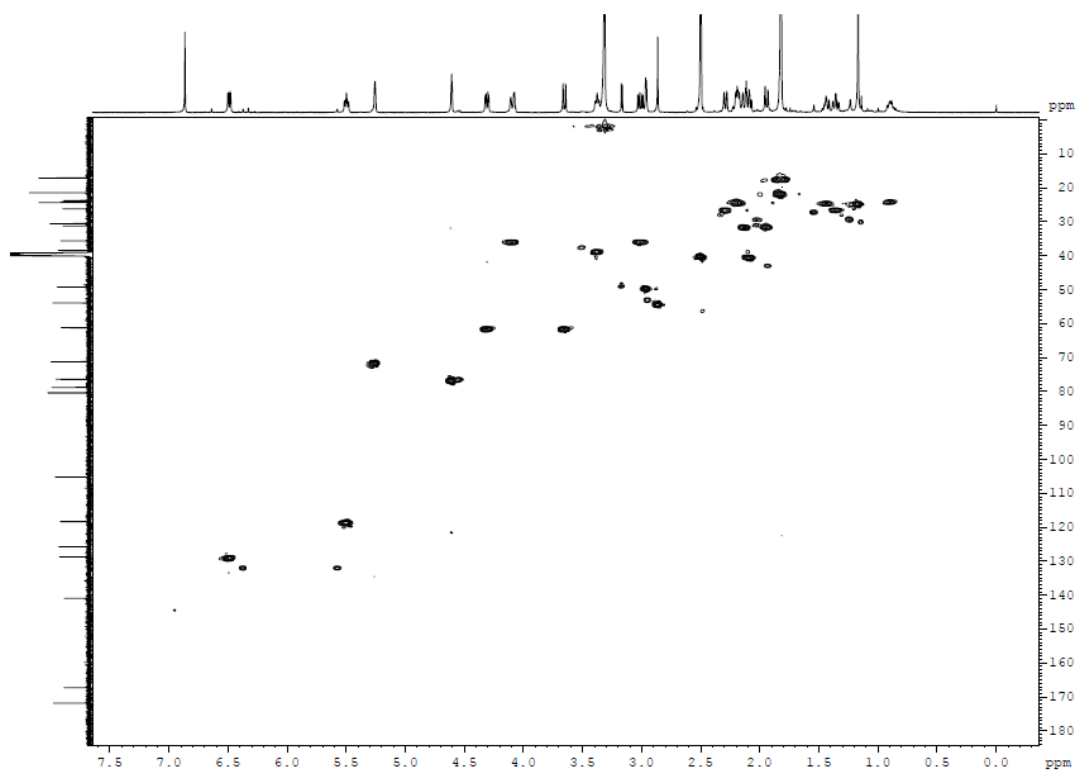
Supplementary Figure S54. HRESIMS spectrum of **6**



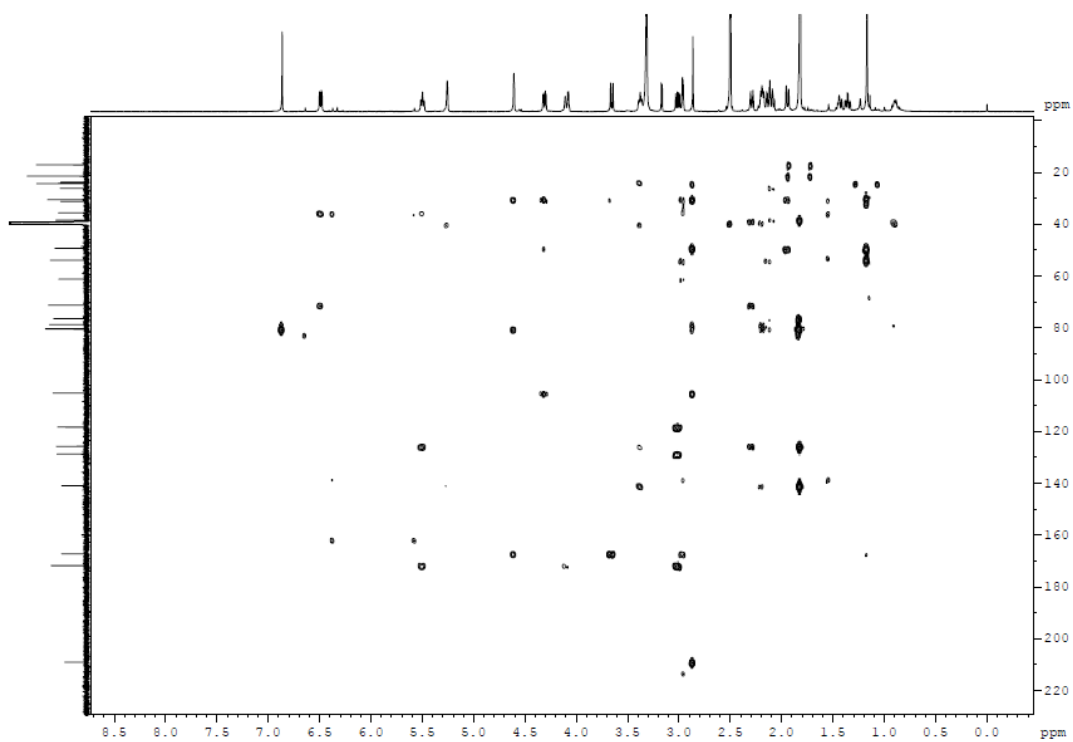
Supplementary Figure S1. ¹H NMR spectrum of **1** (600 MHz, DMSO-*d*₆)



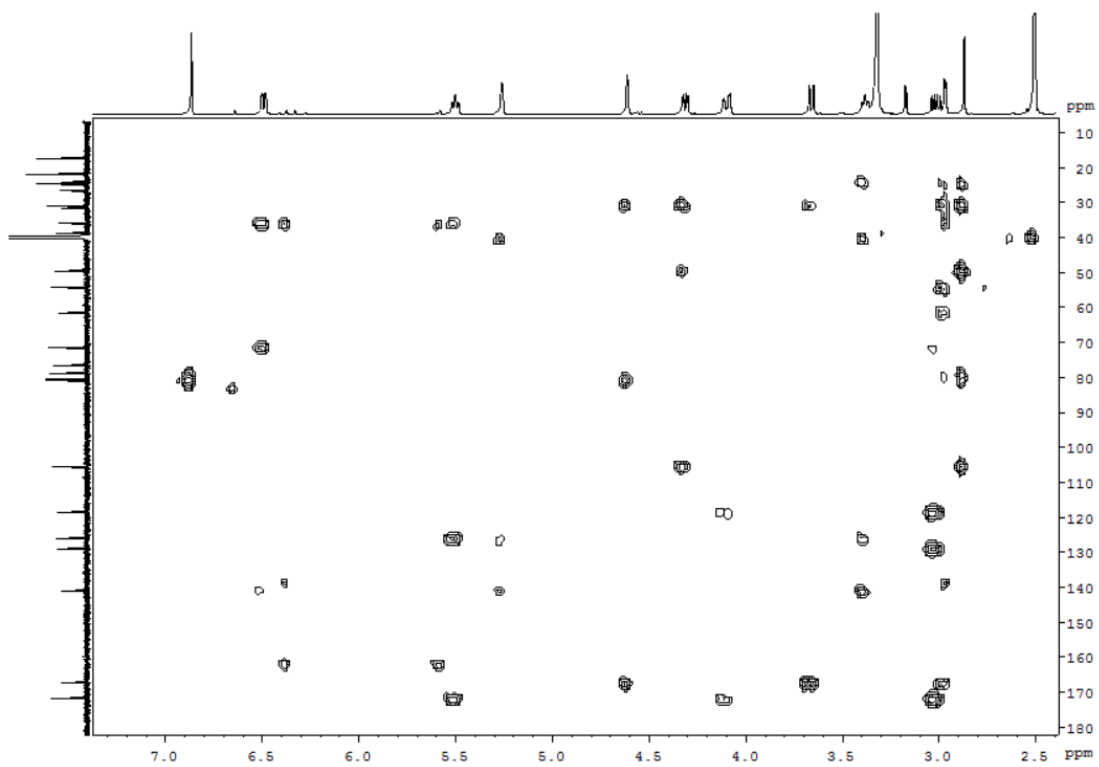
Supplementary Figure S2. ^{13}C NMR spectrum of **1** (150 MHz, $\text{DMSO-}d_6$)



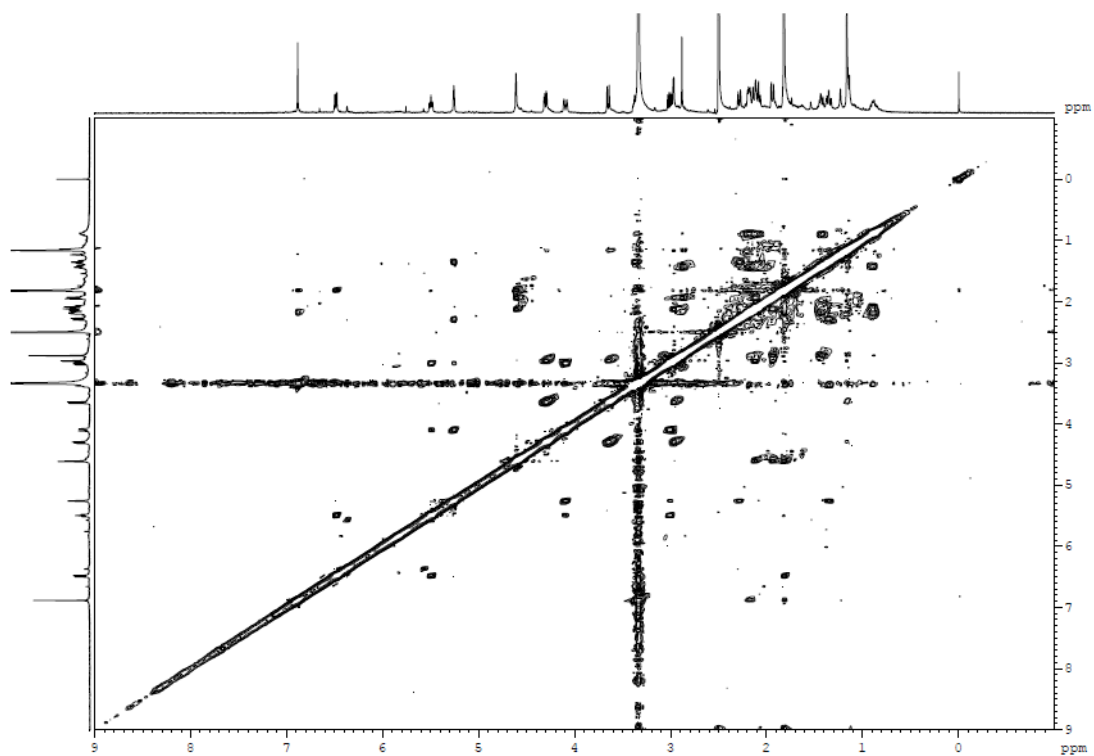
Supplementary Figure S3. HSQC spectrum of **1** (600 MHz, DMSO-*d*₆)



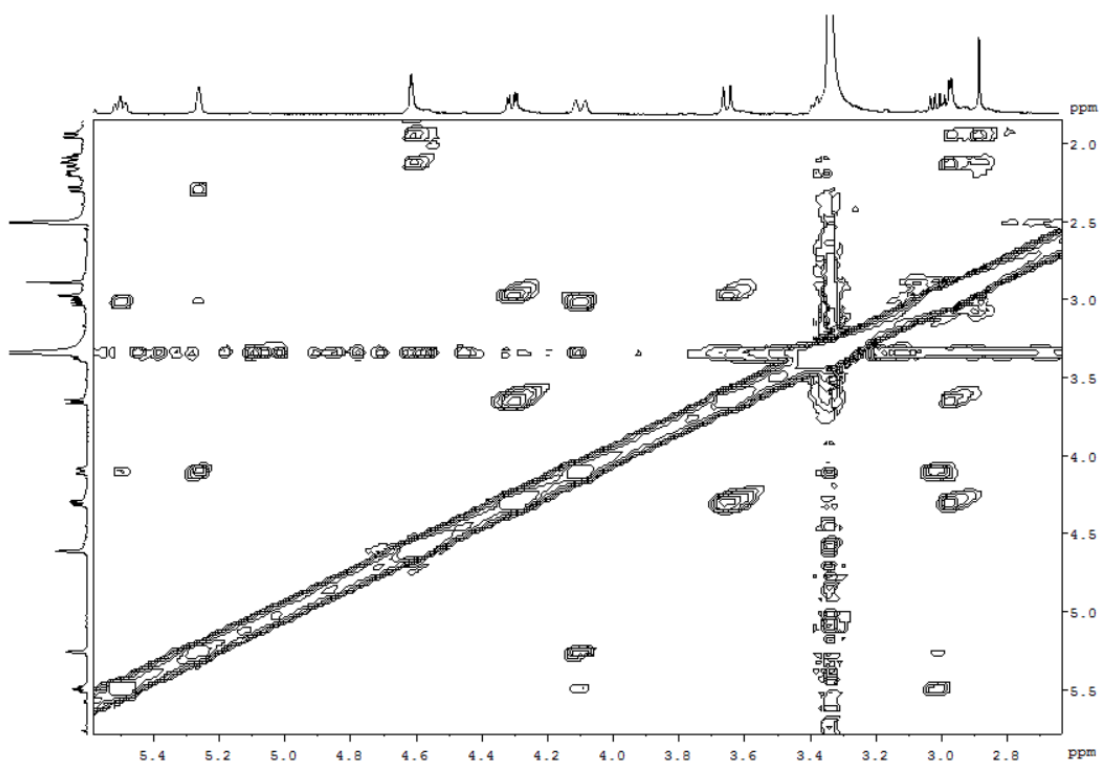
Supplementary Figure S4. HMBC spectrum of **1** (600 MHz, DMSO-*d*₆)



Supplementary Figure S5. Selected HMBC spectrum of **1** (600 MHz, DMSO-*d*₆)



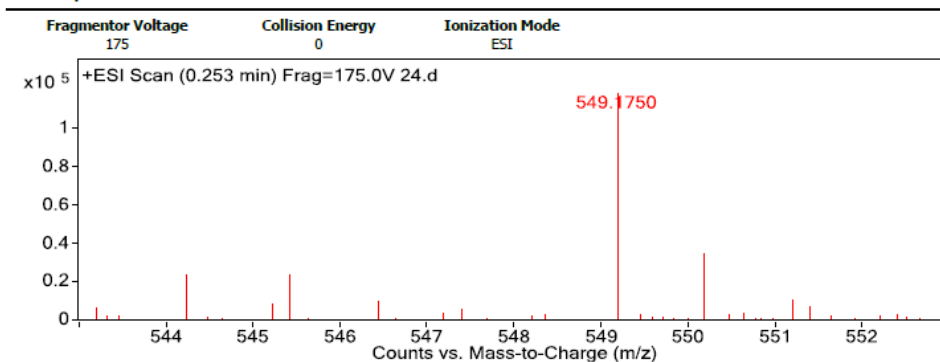
Supplementary Figure S6. NOESY spectrum of **1** (600 MHz, DMSO-*d*₆)



Supplementary Figure S7. Selected NOESY spectrum of **1** (600 MHz, DMSO-

User Chromatograms

User Spectra

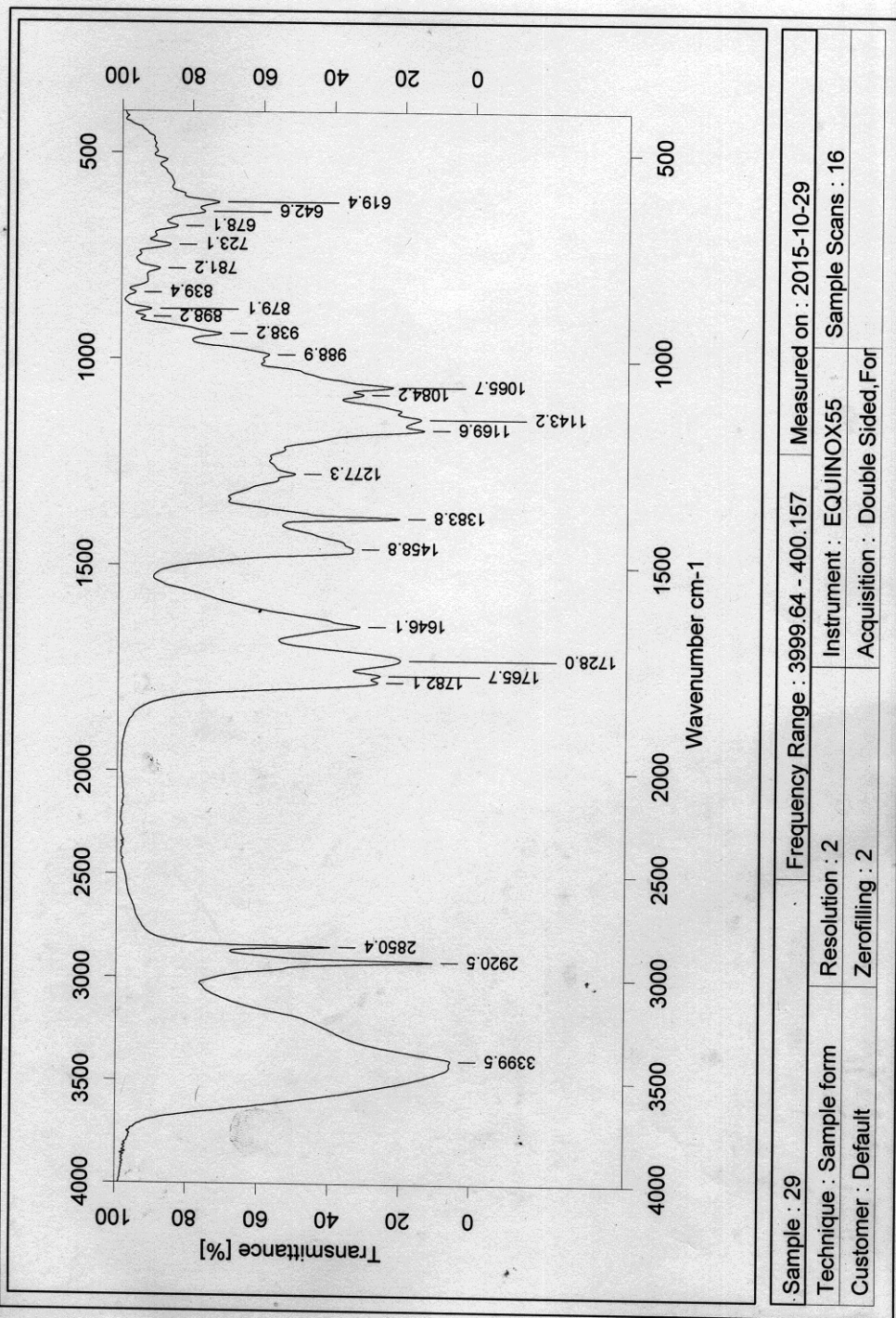


Formula Calculator Results

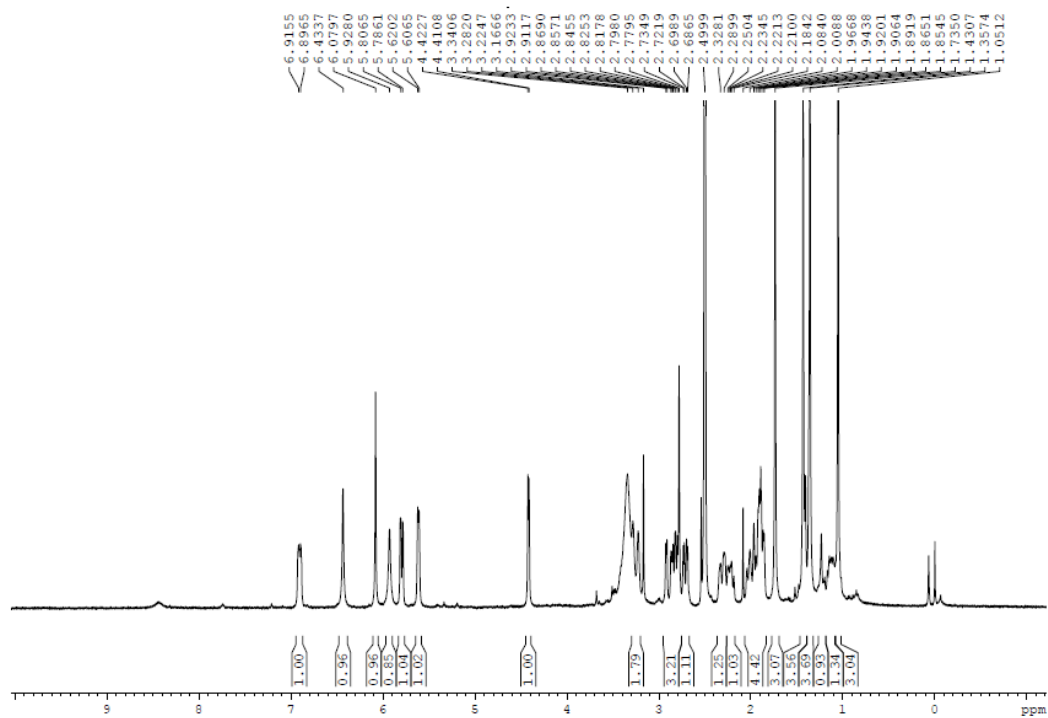
Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C ₂₈ H ₃₀ O ₁₀	TRUE	526.1857	526.1839	-3.52	C ₂₈ H ₃₀ NaO ₁₀	89.71

--- End Of Report ---

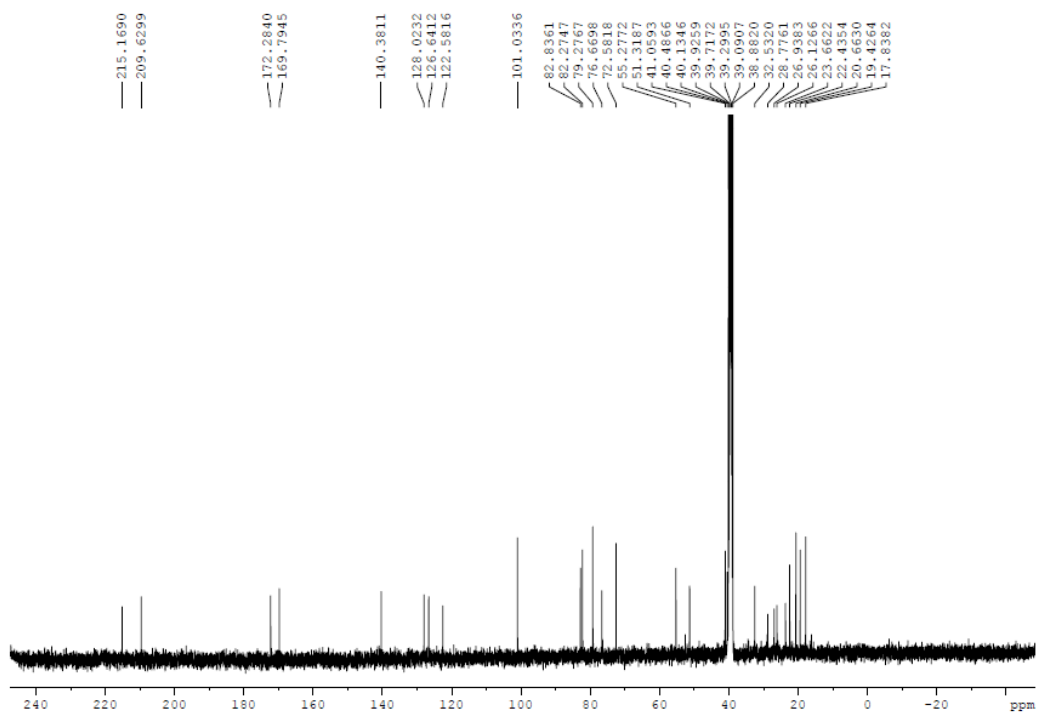
Supplementary Figure S8. HRESIMS spectrum of 1



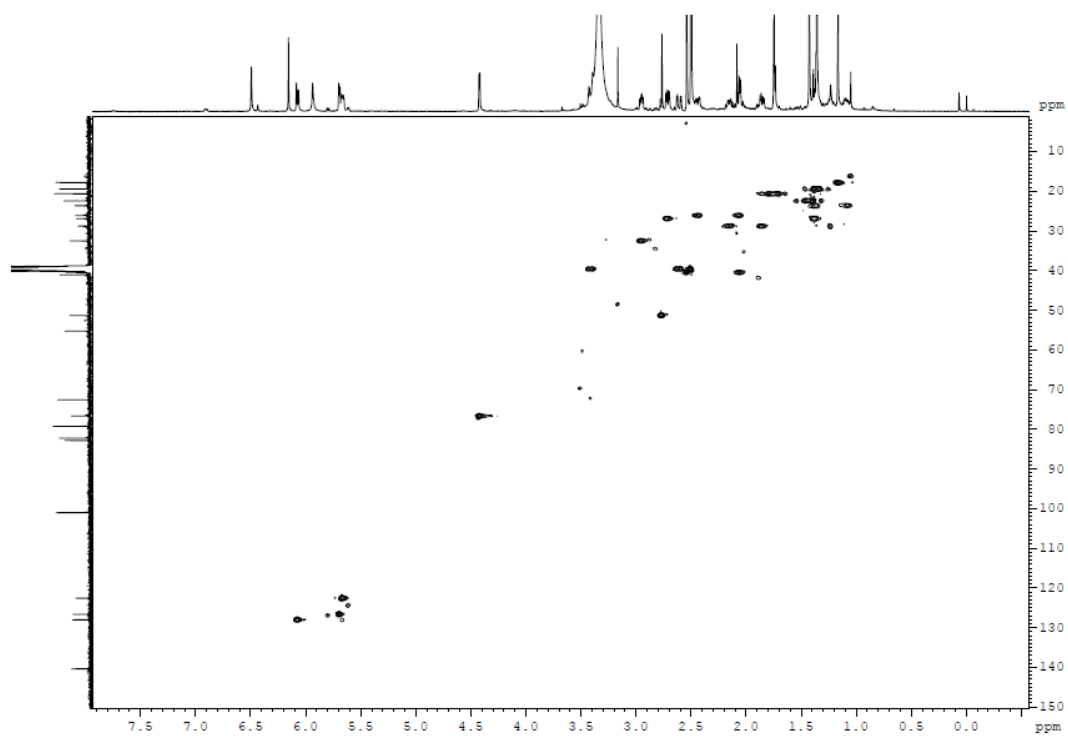
Supplementary Figure S9. IR spectrum of 1



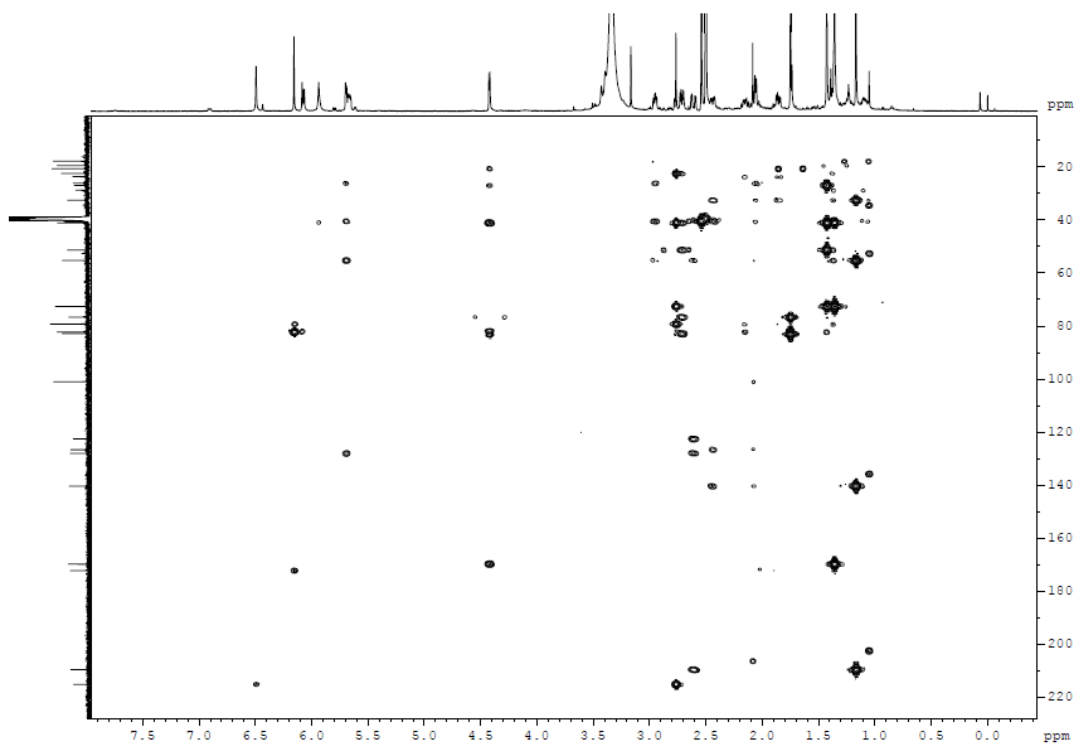
Supplementary Figure S10. ^1H NMR spectrum of **2** (400 MHz, $\text{DMSO-}d_6$)



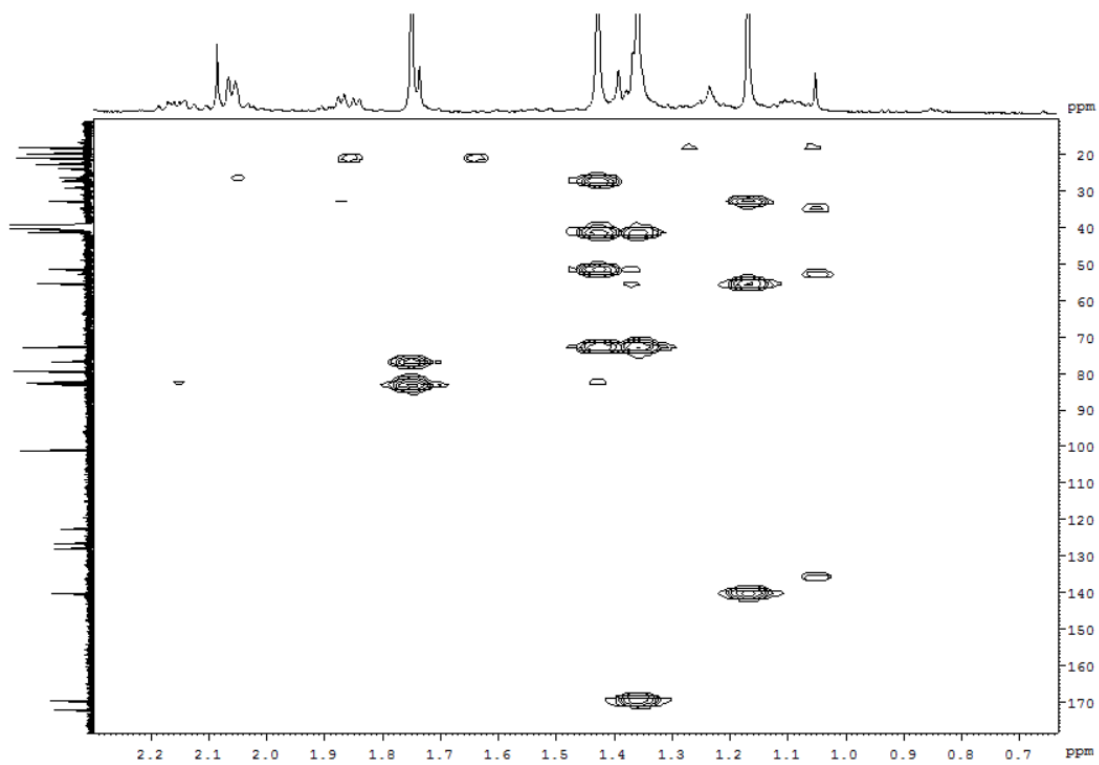
Supplementary Figure S11. ^{13}C NMR spectrum of **2** (100 MHz, $\text{DMSO-}d_6$)



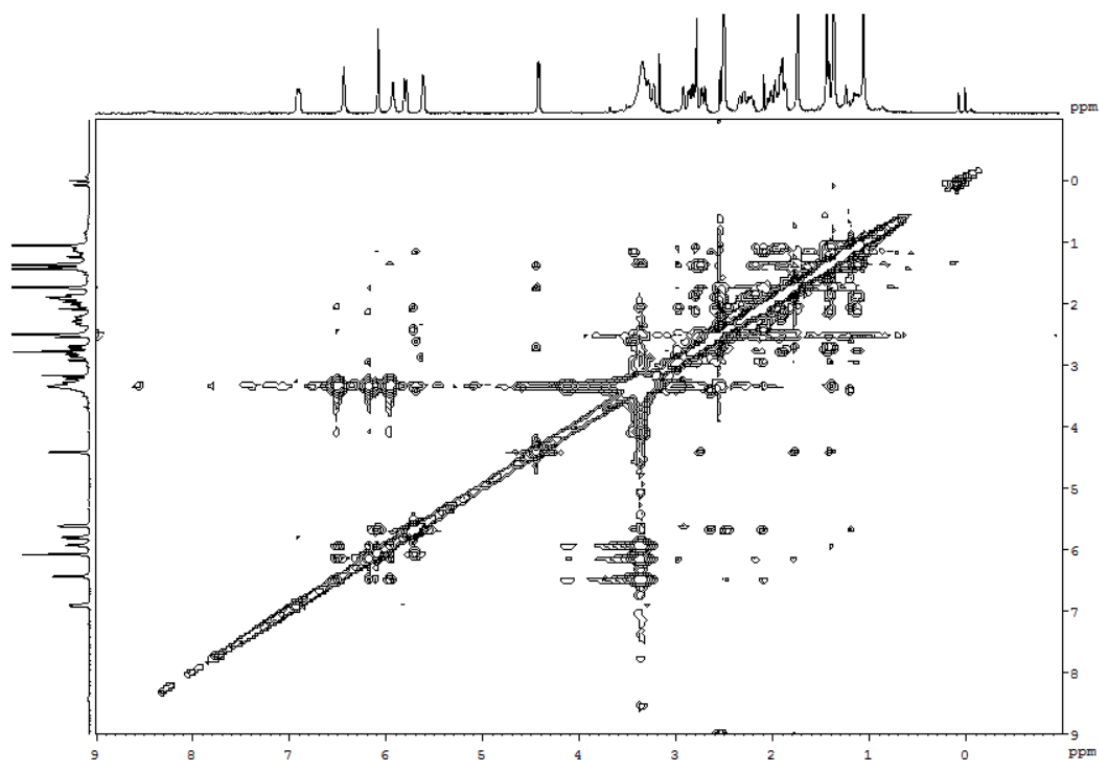
Supplementary Figure S12. HSQC spectrum of **2** (600 MHz, DMSO-*d*₆)



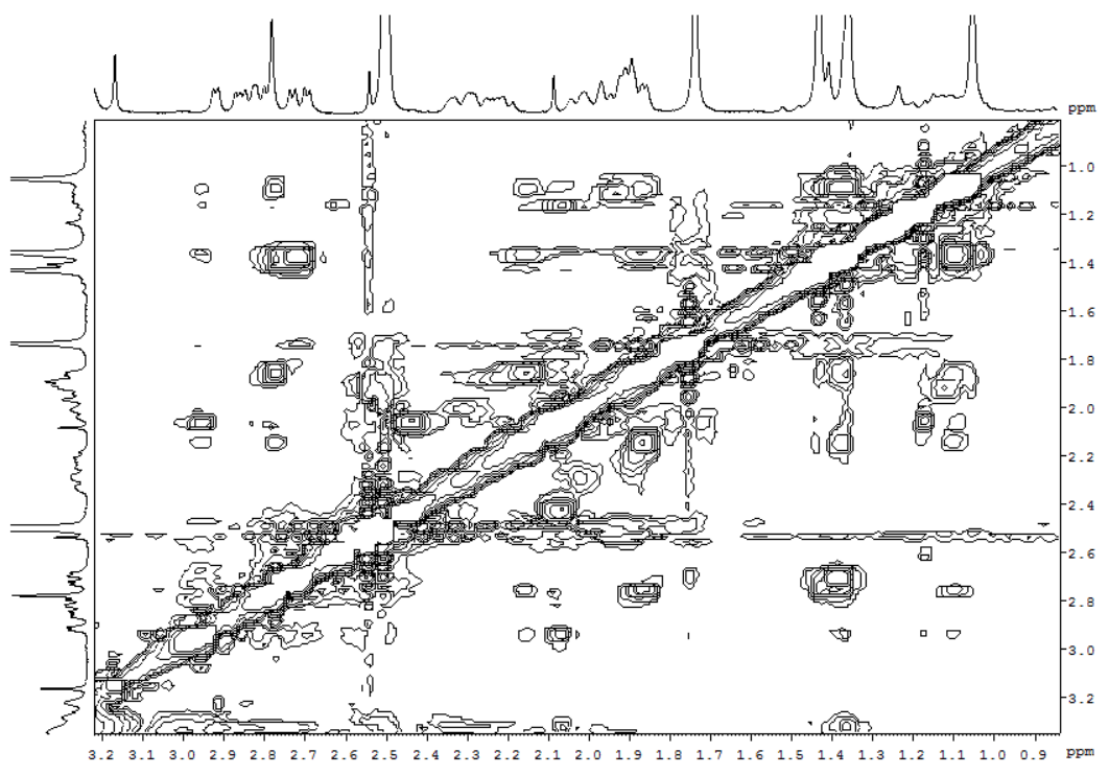
Supplementary Figure S13. HMBC spectrum of **2** (600 MHz, DMSO-*d*₆)



Supplementary Figure S14. Selected HMBC spectrum of **2** (600 MHz, DMSO-*d*₆)



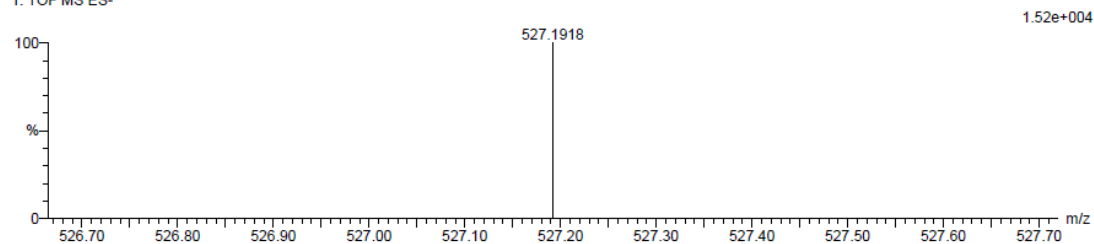
Supplementary Figure S15. NOESY spectrum of **2** (600 MHz, DMSO-*d*₆)



Supplementary Figure S16. Selected NOESY spectrum of **2** (600 MHz, DMSO-*d*₆)

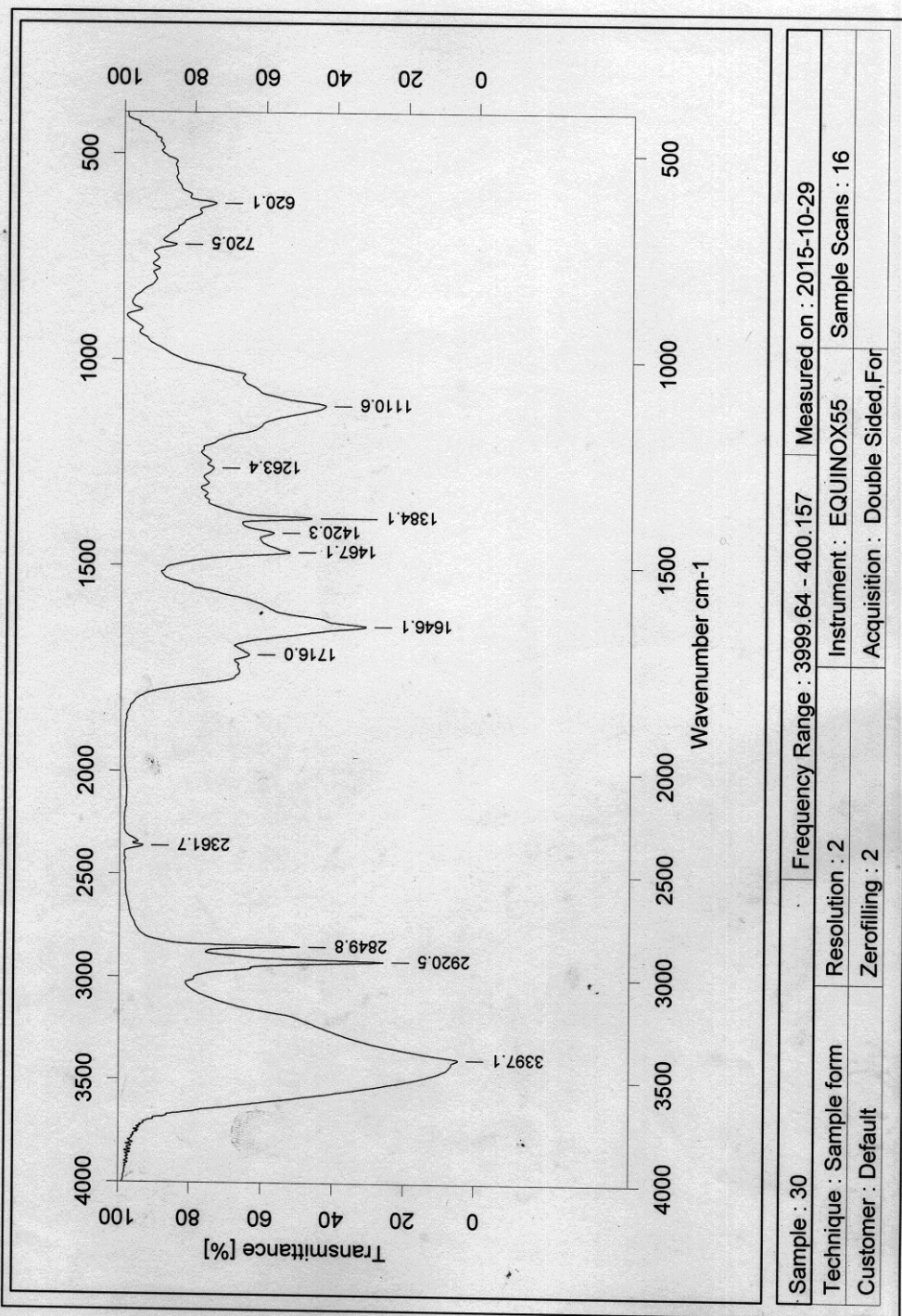
Tolerance = 2.1 PPM / DBE: min = 3.0, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
 250 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
 Elements Used:
 C: 0-100 H: 0-100 O: 0-200 Na: 0-1
 SCPs12 neg 49 (0.205)
 1: TOF MS ES-

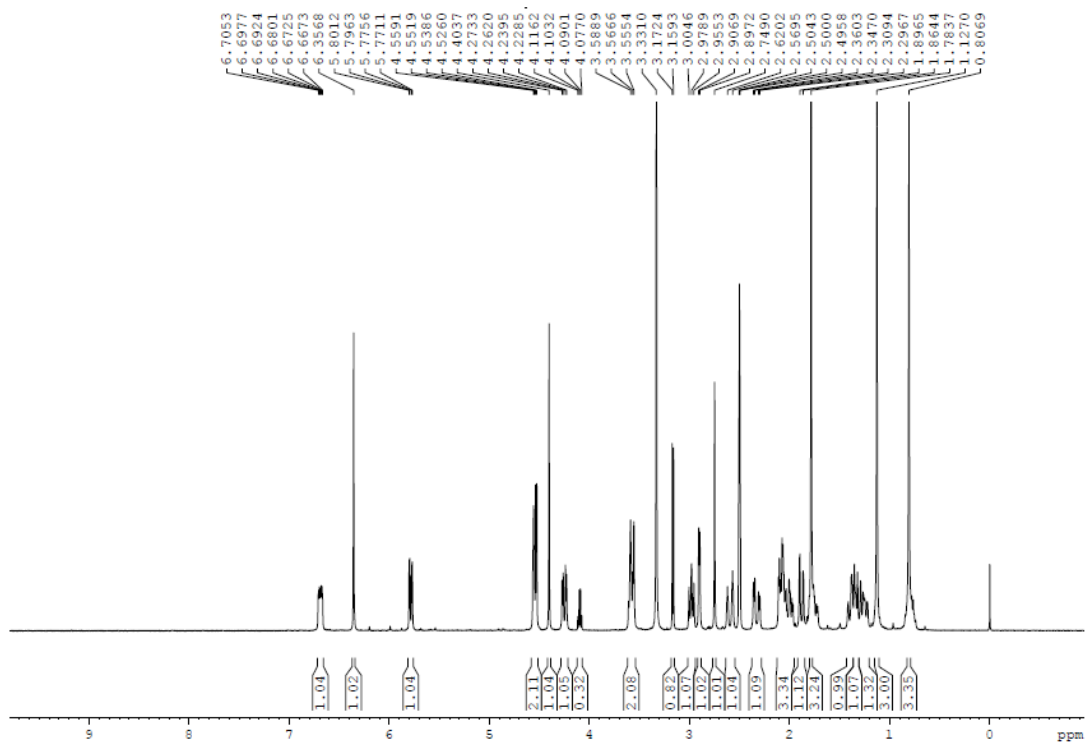


Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
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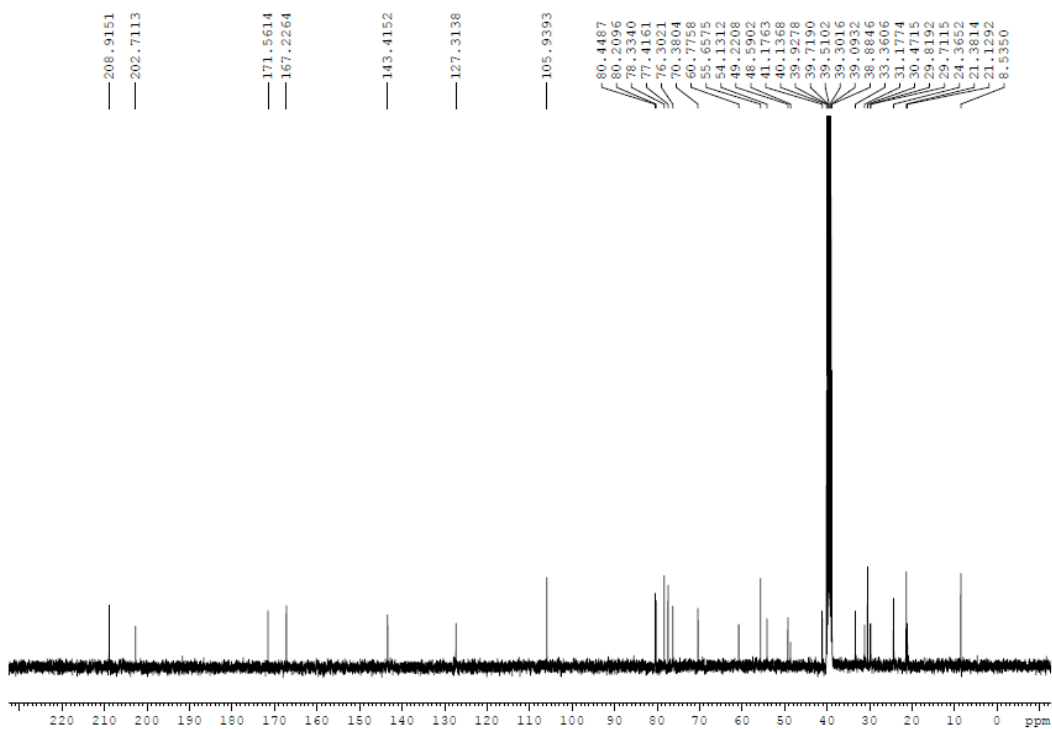
Supplementary Figure S17. HRESIMS spectrum of **2**



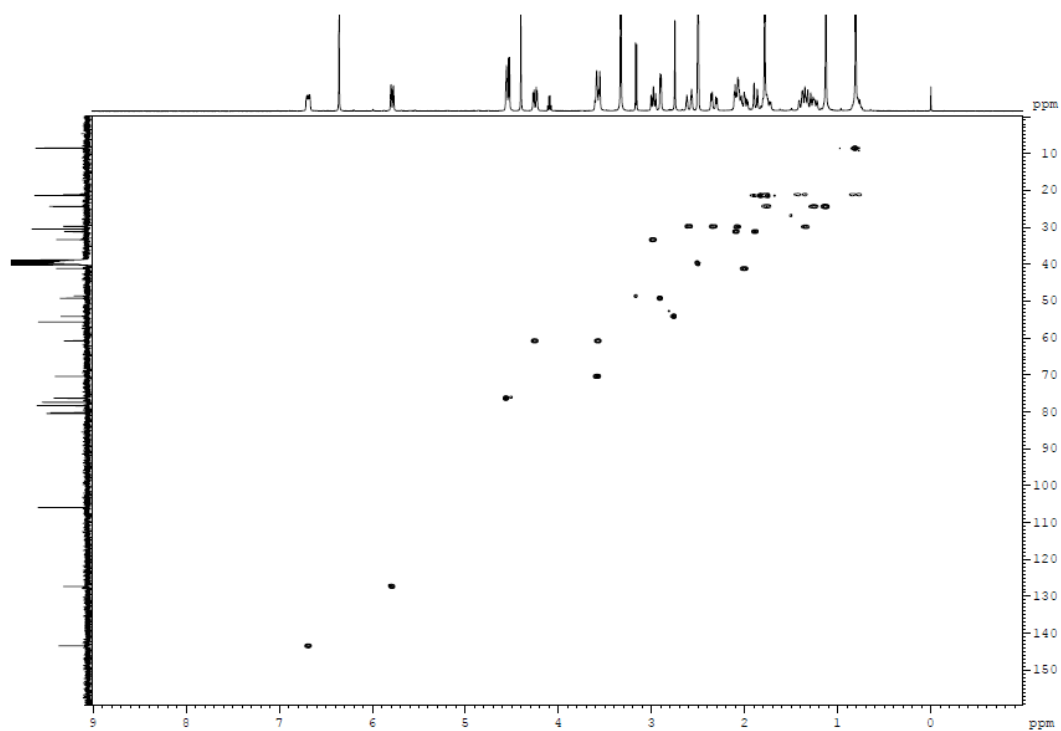
Supplementary Figure S18. IR spectrum of 2



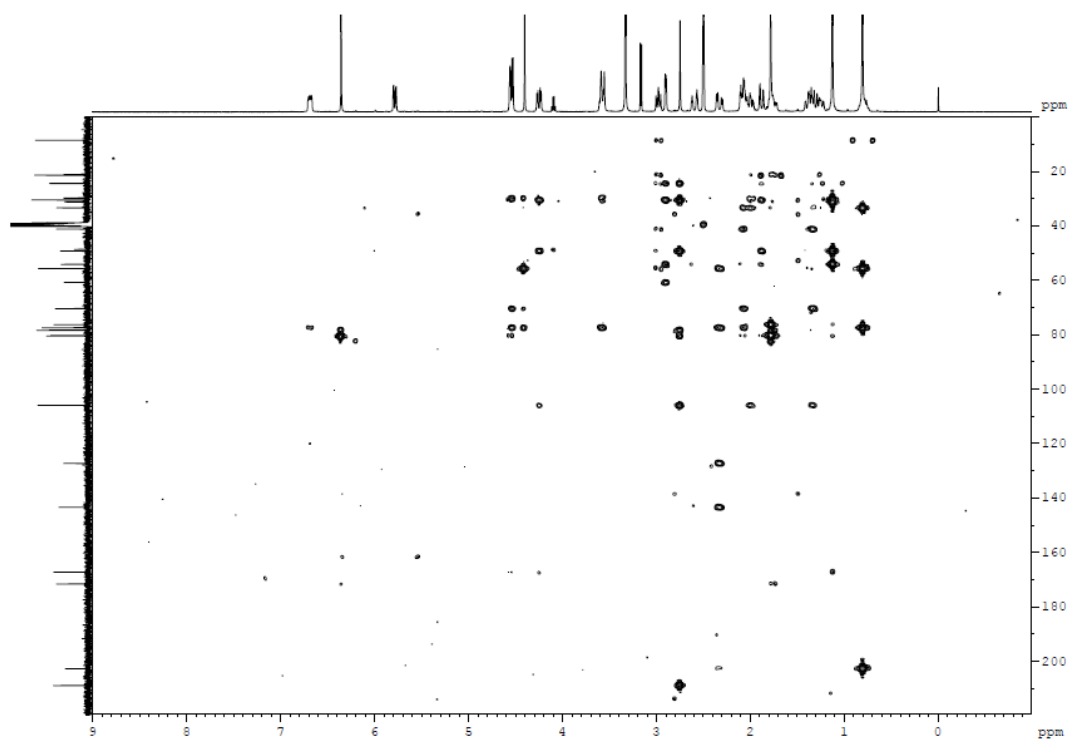
Supplementary Figure S19. ^1H NMR spectrum of **3** (400 MHz, $\text{DMSO-}d_6$)



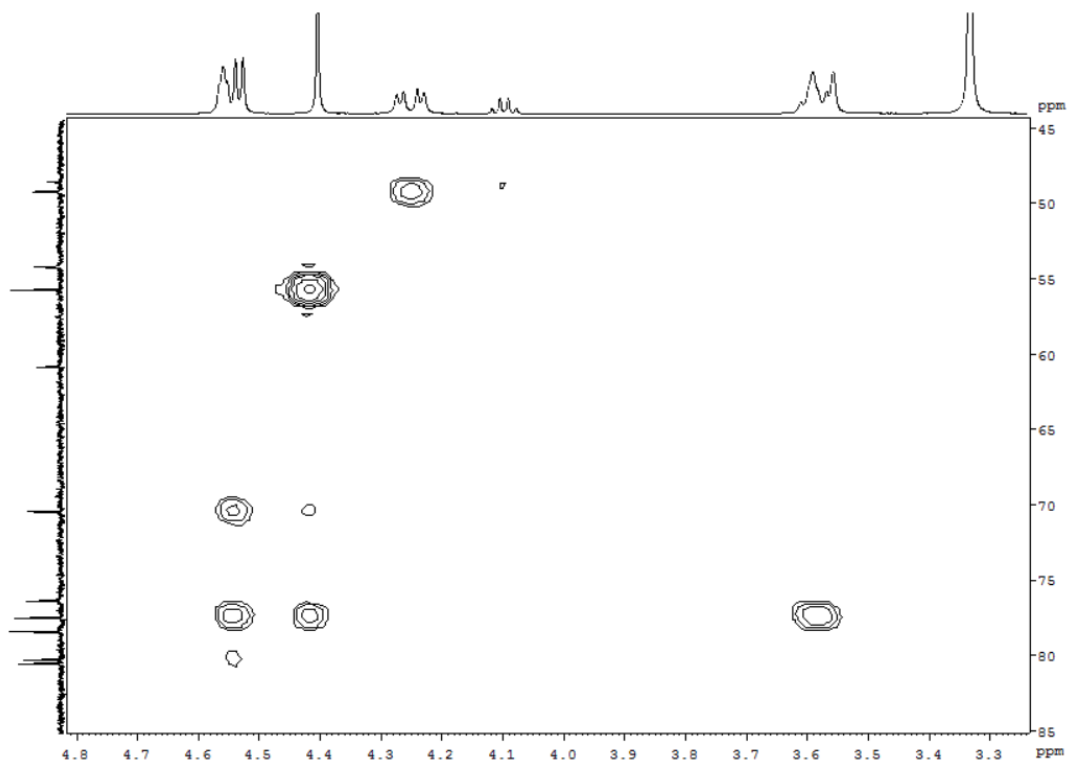
Supplementary Figure S20. ^{13}C NMR spectrum of **3** (100 MHz, $\text{DMSO-}d_6$)



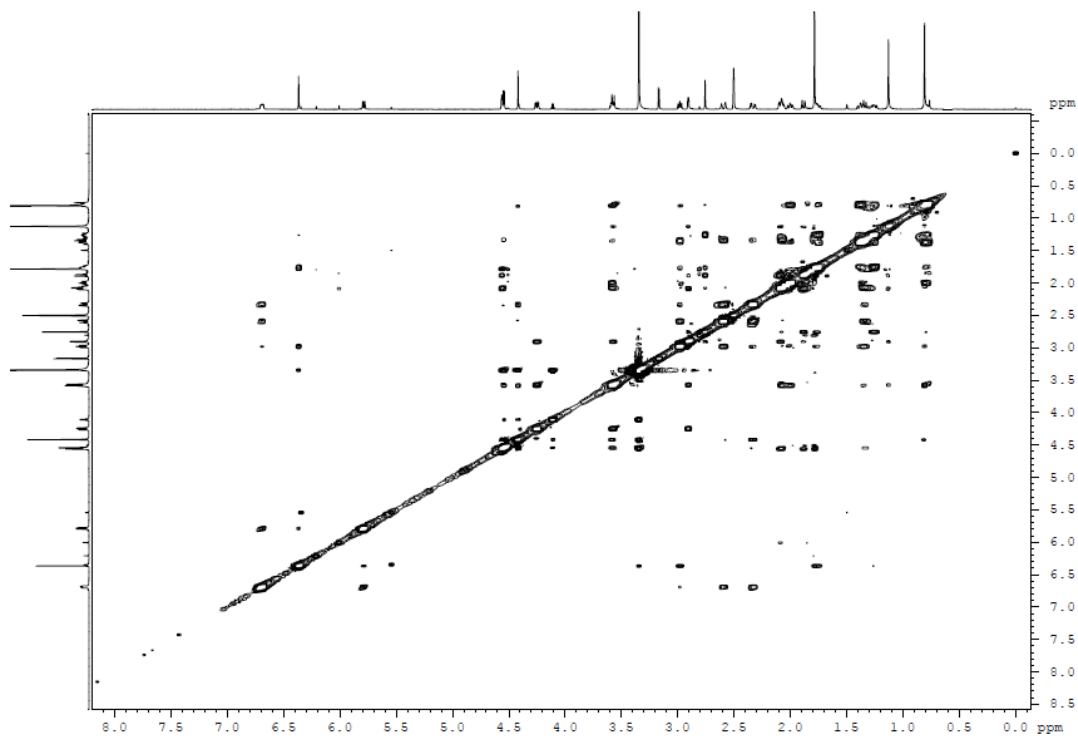
Supplementary Figure S21. HSQC spectrum of **3** (600 MHz, DMSO-*d*₆)



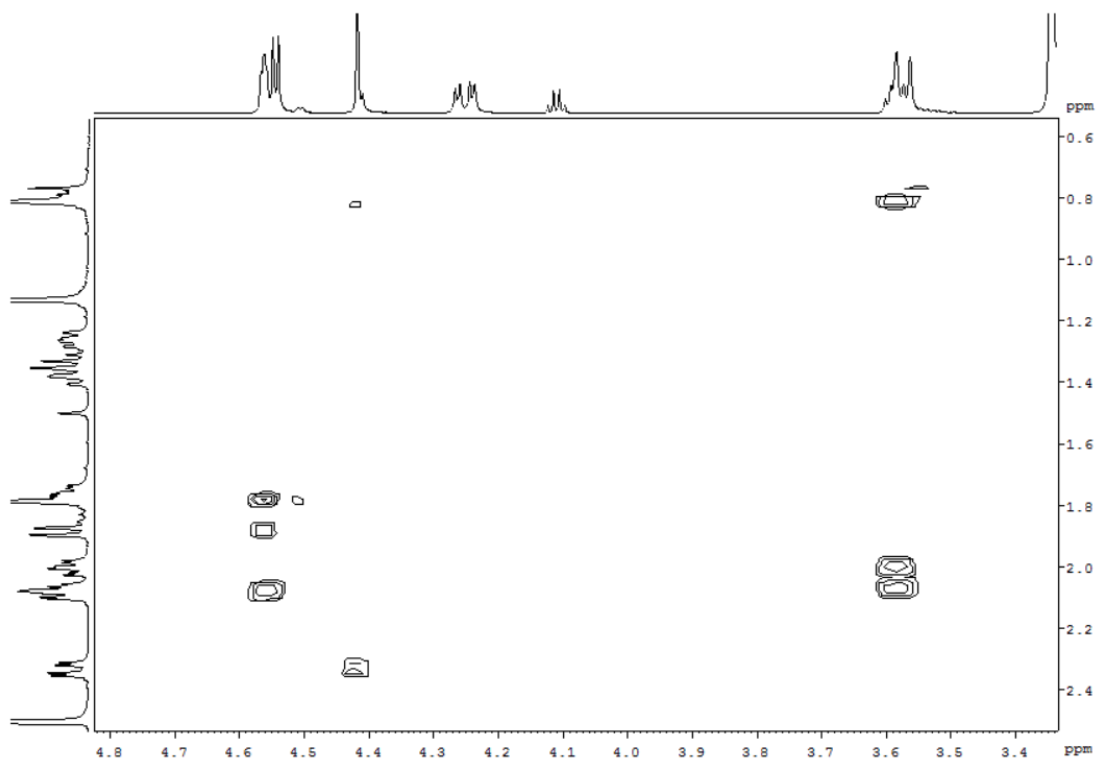
Supplementary Figure S22. HMBC spectrum of **3** (600 MHz, DMSO-*d*₆)



Supplementary Figure S23. Selected HMBC spectrum of **3** (600 MHz, DMSO-*d*₆)



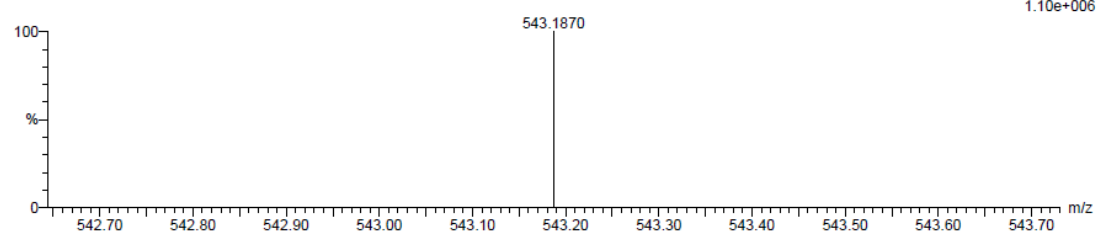
Supplementary Figure S24. NOESY spectrum of **3** (600 MHz, DMSO-*d*₆)



Supplementary Figure S25. Selected NOESY spectrum of **3** (600 MHz, DMSO-*d*₆)

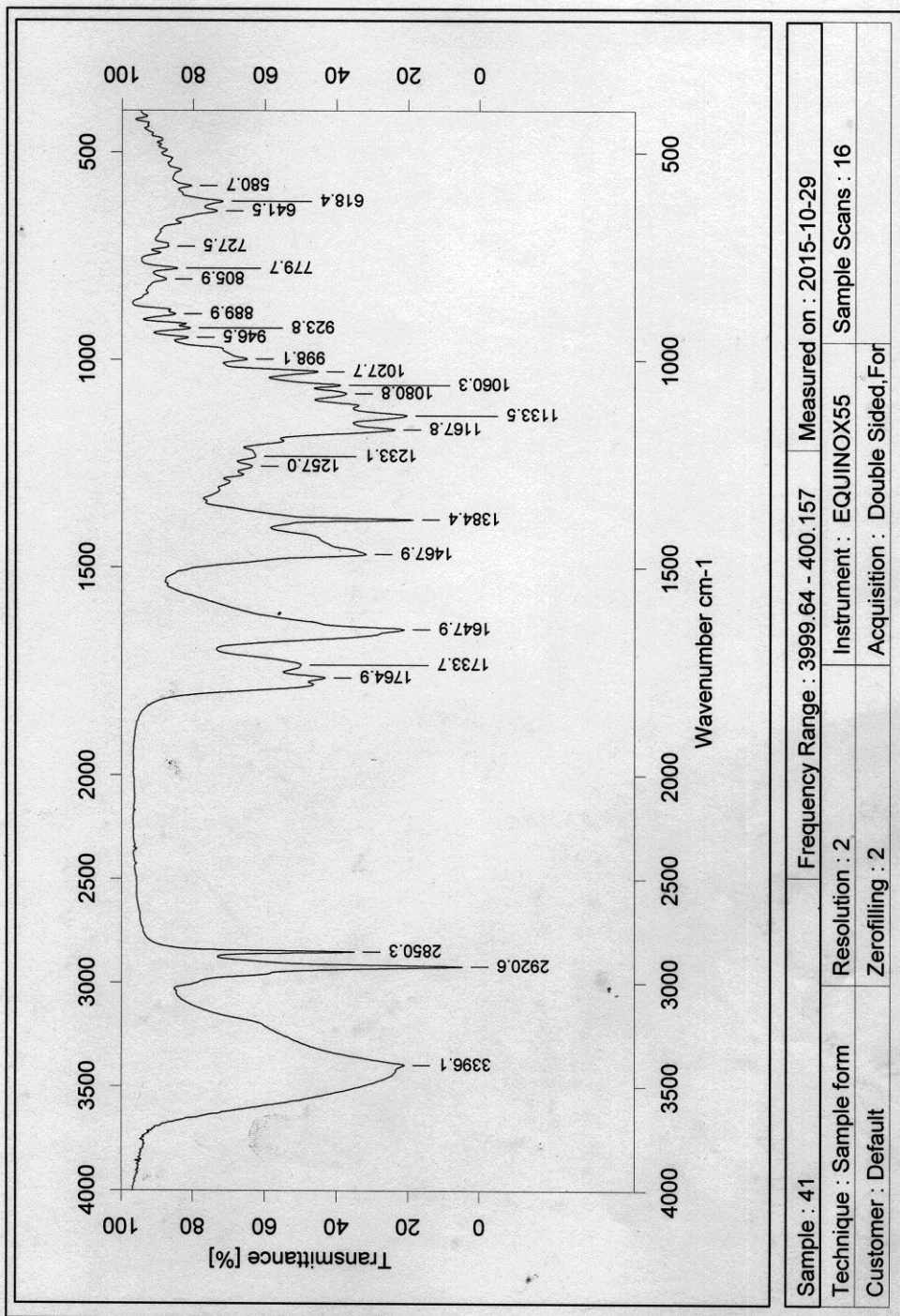
Tolerance = 2.0 PPM / DBE: min = 3.0, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
 139 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
 Elements Used:
 C: 0-100 H: 0-100 O: 0-200
 SCPs23 neg 41 (0.170) Cm (41:55)
 1: TOF MS ES-

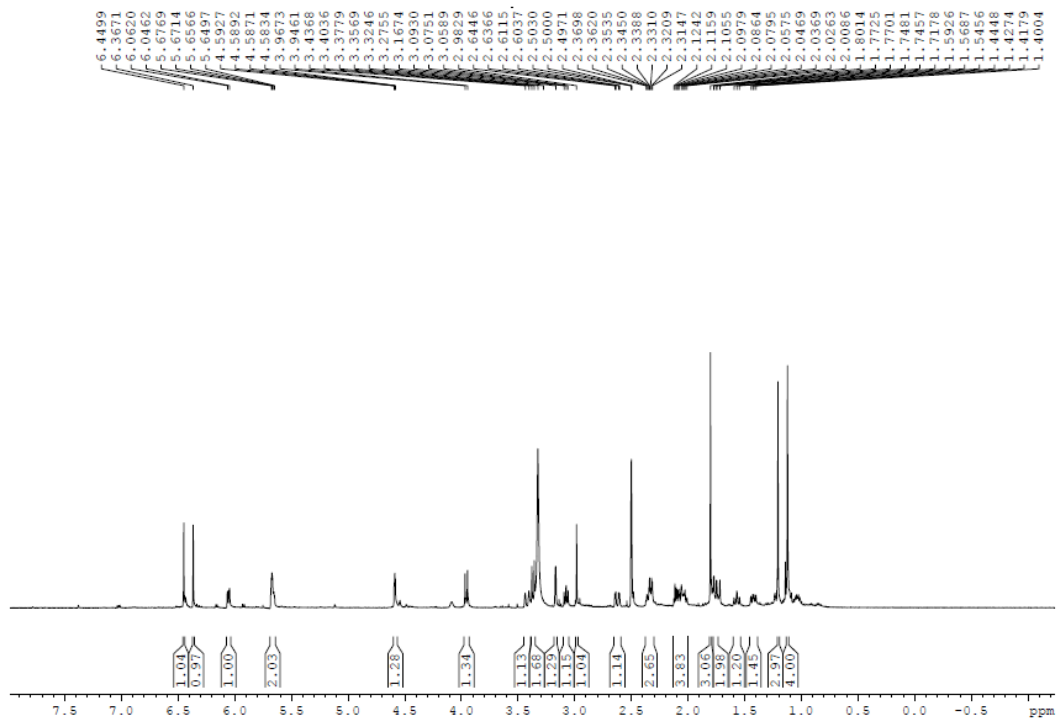


Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
543.1870	543.1866	0.4	0.7	13.5	42.4	n/a	n/a	C ₂₈ H ₃₁ O ₁₁

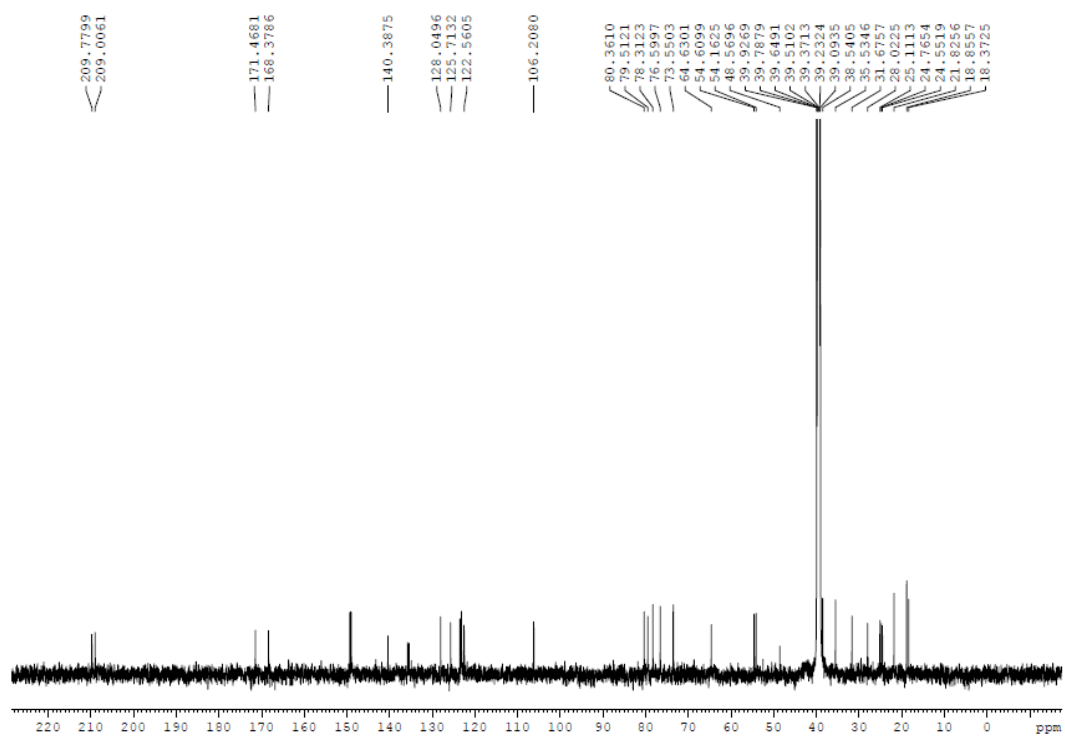
Supplementary Figure S26. HRESIMS spectrum of **3**



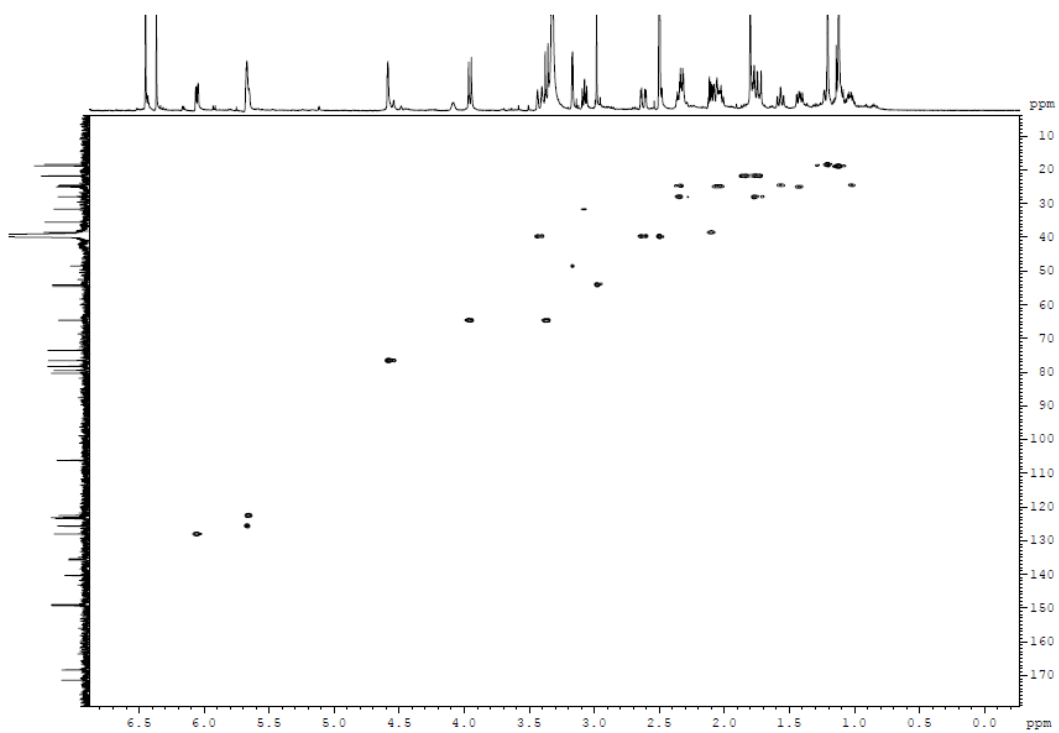
Supplementary Figure S27. IR spectrum of 3



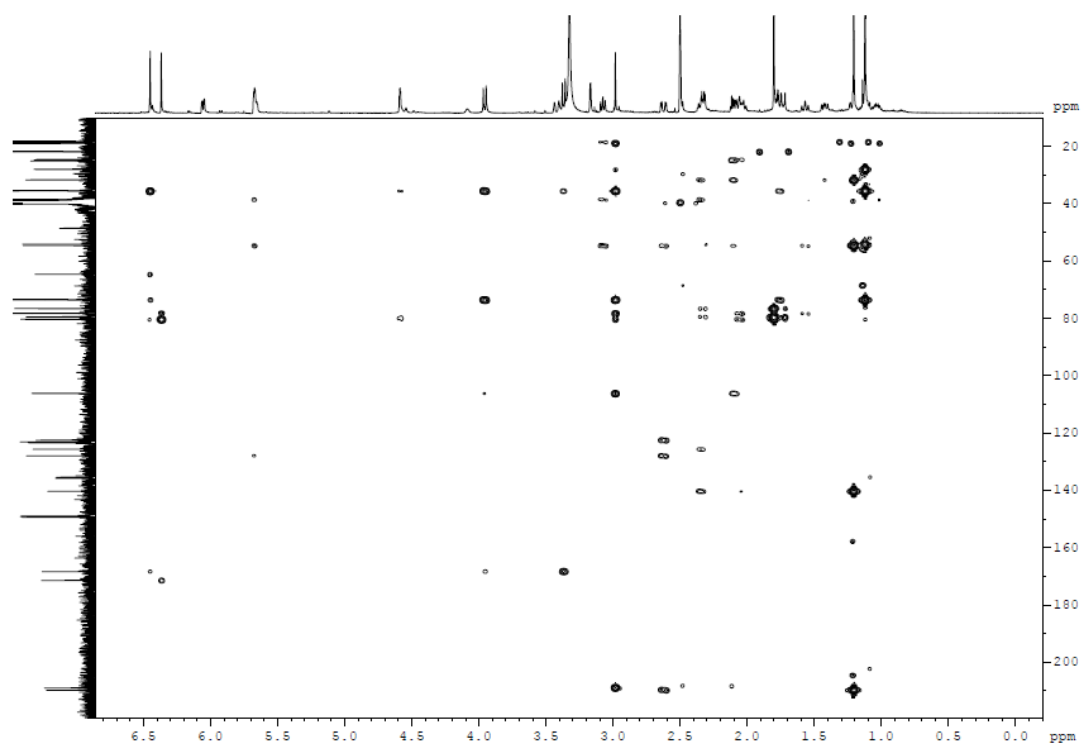
Supplementary Figure S28. ^1H NMR spectrum of **4** (600 MHz, $\text{DMSO-}d_6$)



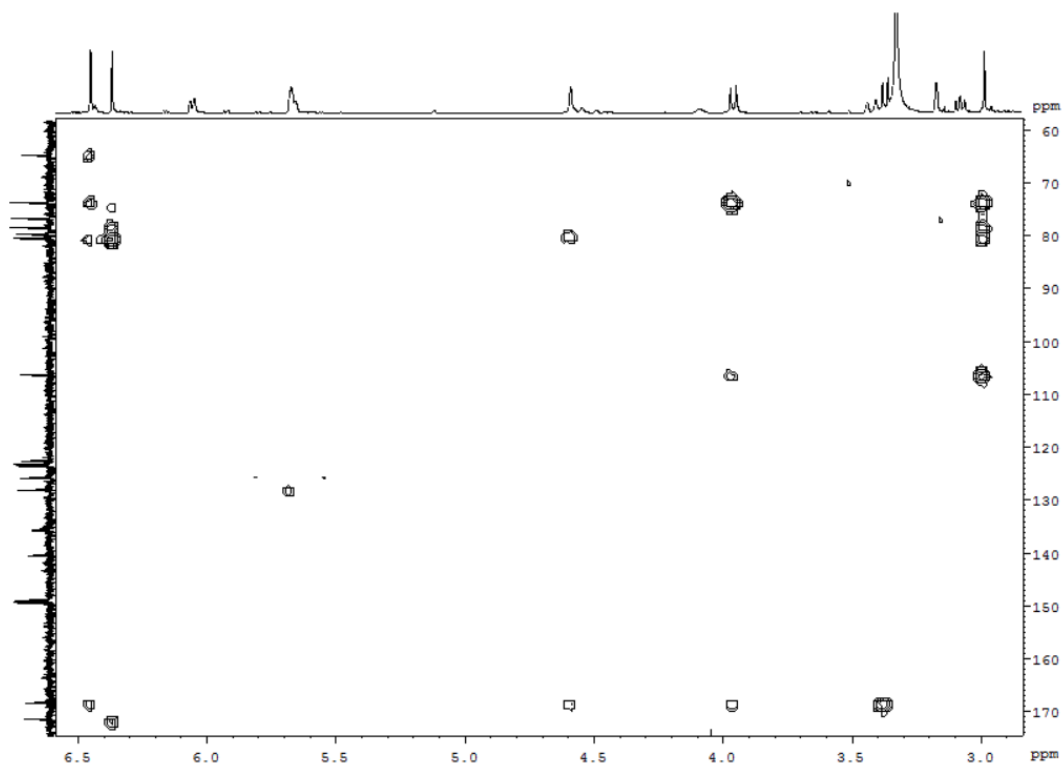
Supplementary Figure S29. ^{13}C NMR spectrum of **4** (150 MHz, $\text{DMSO-}d_6$)



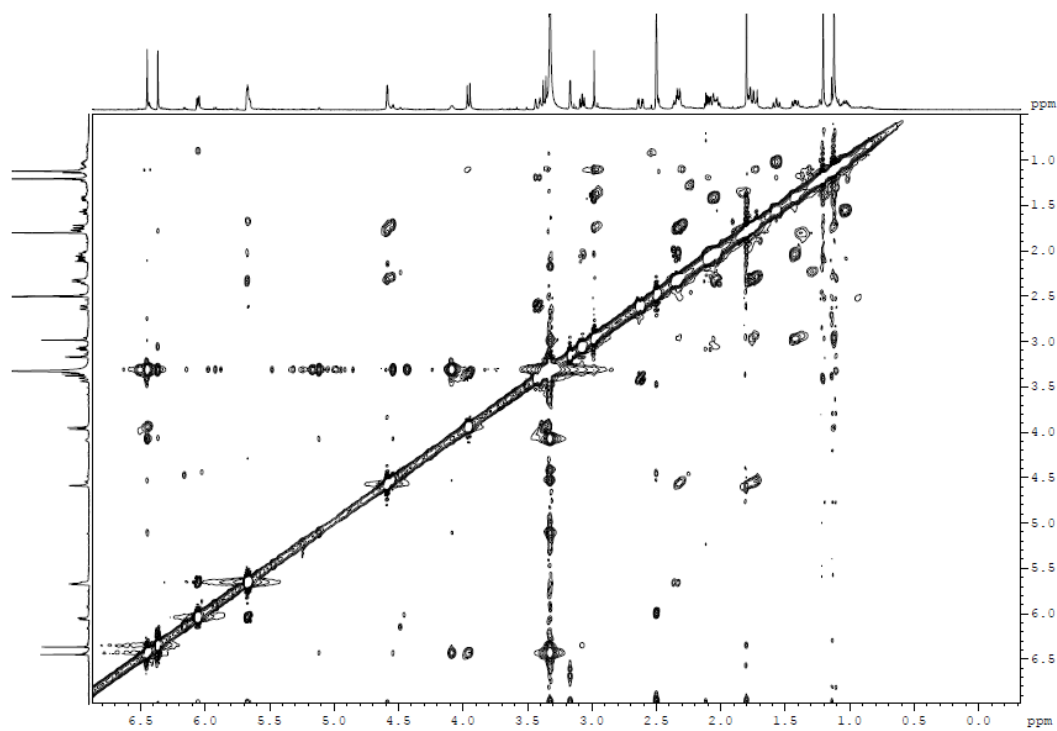
Supplementary Figure S30. HSQC spectrum of **4** (600 MHz, DMSO-*d*₆)



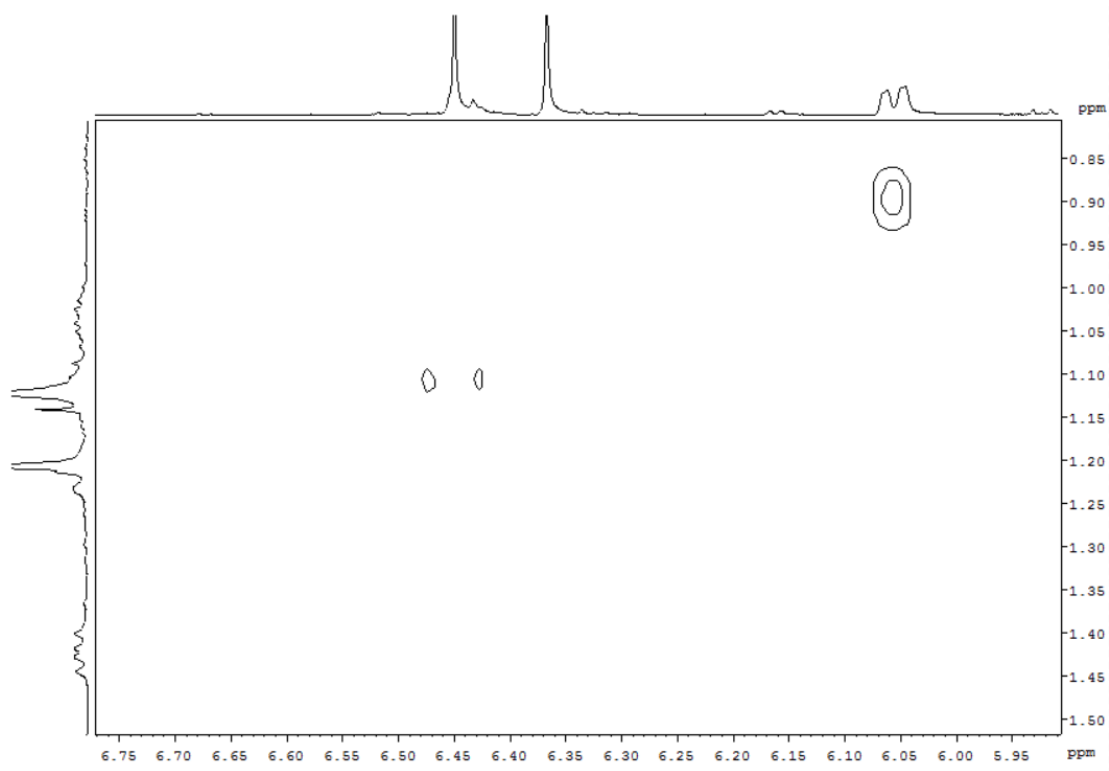
Supplementary Figure S31. HMBC spectrum of **4** (600 MHz, DMSO-*d*₆)



Supplementary Figure S32. Selected HMBC spectrum of **4** (600 MHz, DMSO-*d*₆)



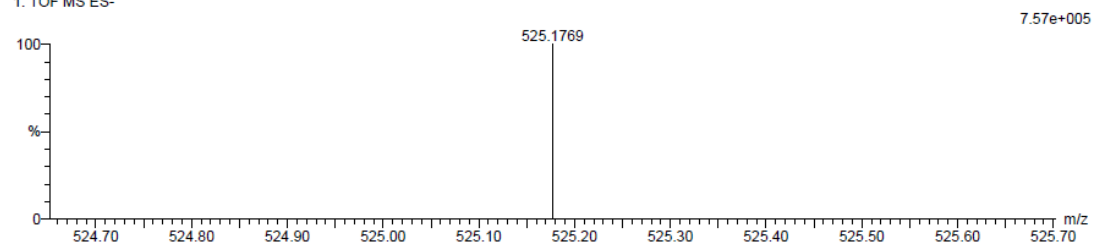
Supplementary Figure S33. NOESY spectrum of **4** (600 MHz, DMSO-*d*₆)



Supplementary Figure S34. Selected NOESY spectrum of **4** (600 MHz, DMSO-*d*₆)

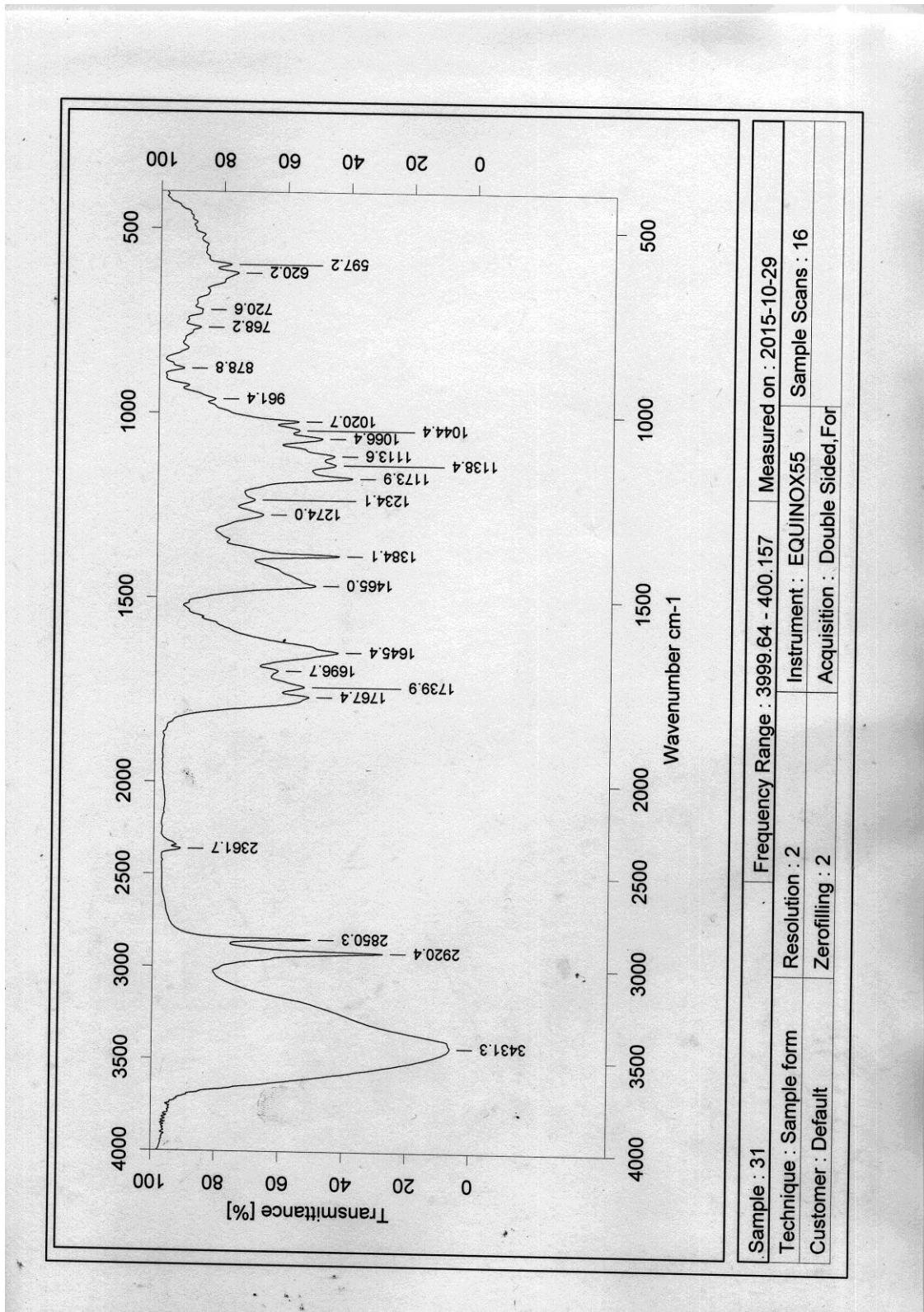
Tolerance = 2.1 PPM / DBE: min = 3.0, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
 238 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
 Elements Used:
 C: 0-100 H: 0-100 O: 0-200 Na: 0-1
 SCPs13 neg 24 (0.109)
 1: TOF MS ES-

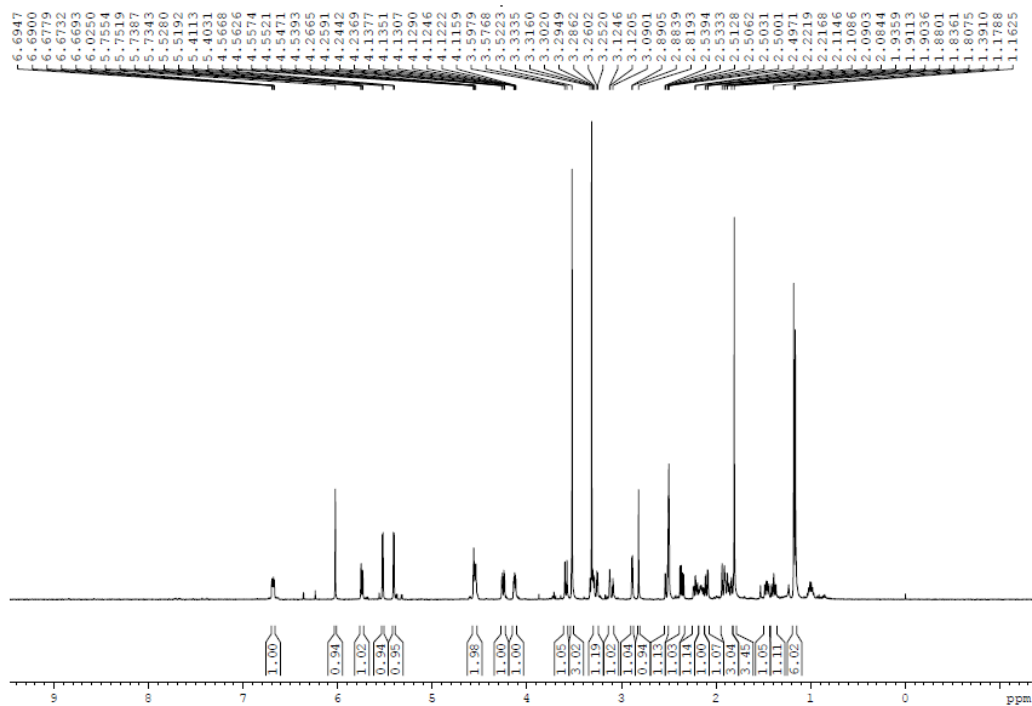


Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
525.1769	525.1761	0.8	1.5	14.5	41.9	n/a	n/a	C ₂₈ H ₂₉ O ₁₀

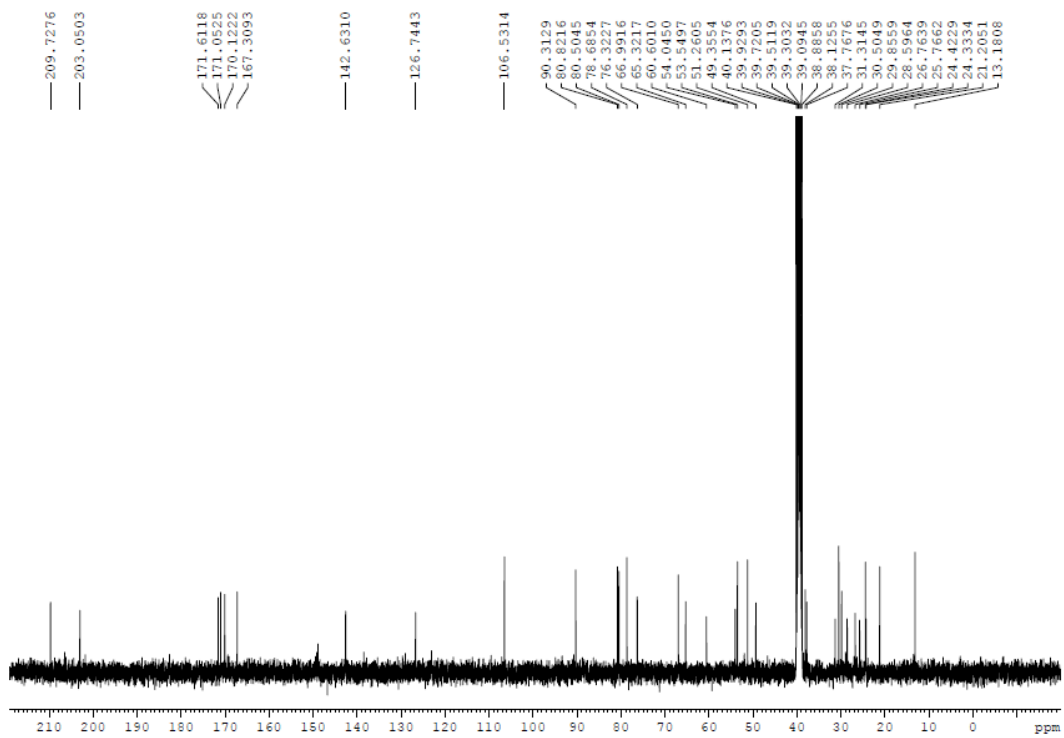
Supplementary Figure S35. HRESIMS spectrum of **4**



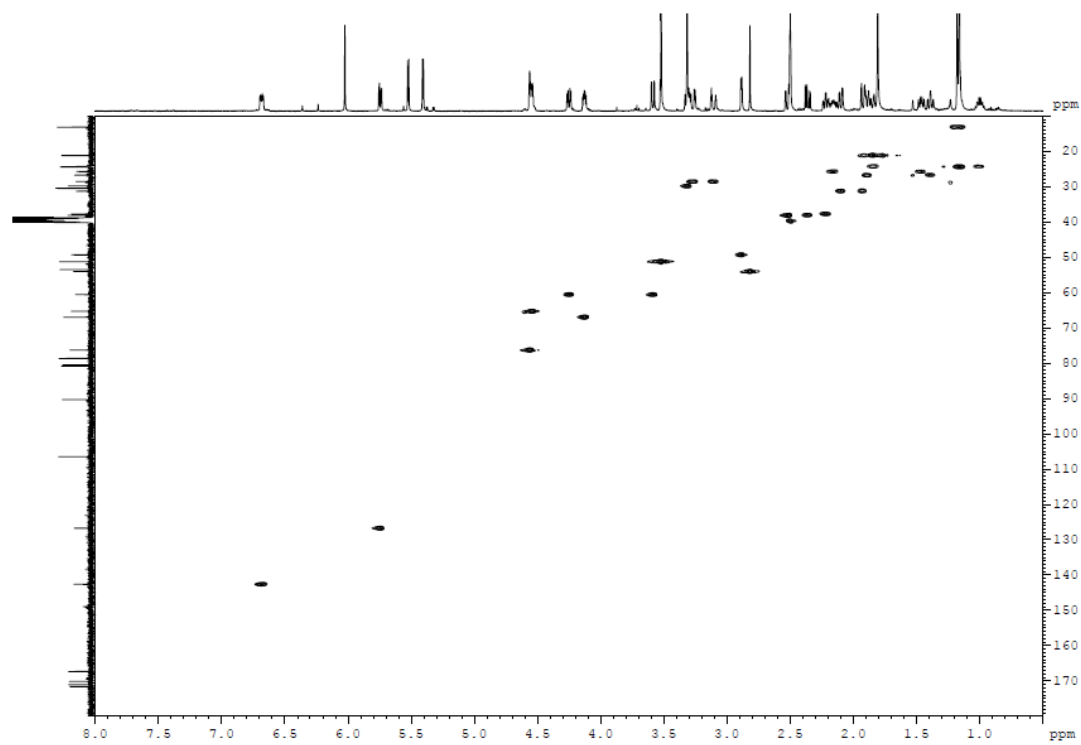
Supplementary Figure S36. IR spectrum of 4



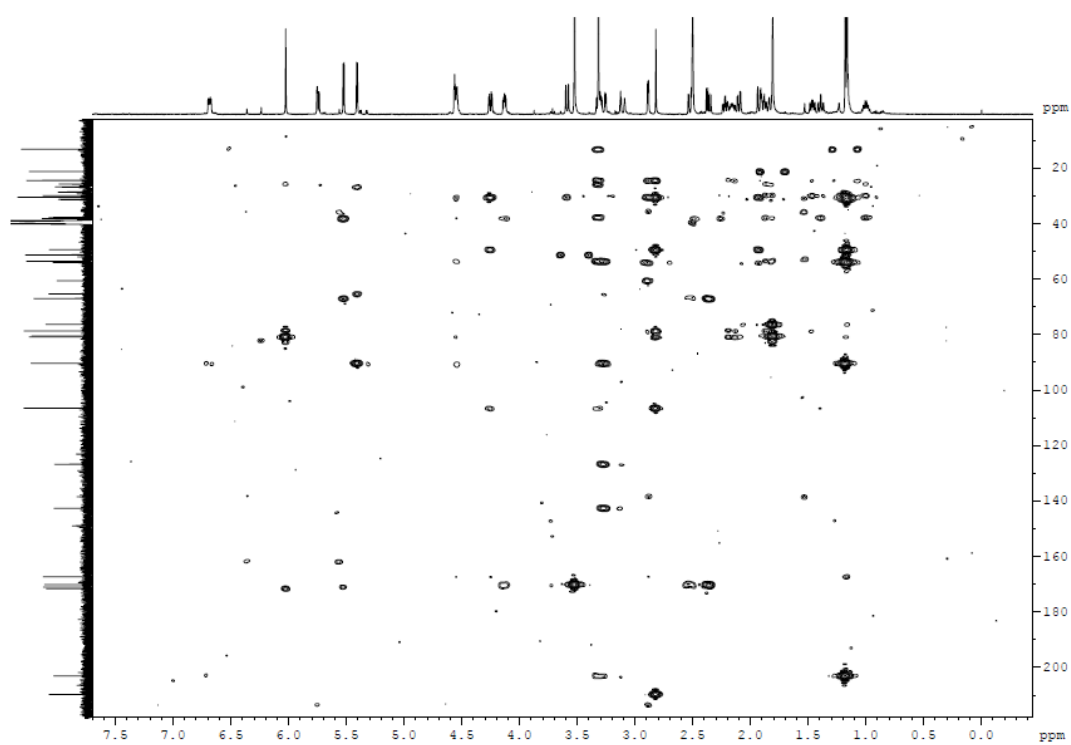
Supplementary Figure S37. ^1H NMR spectrum of **5** (400 MHz, $\text{DMSO-}d_6$)



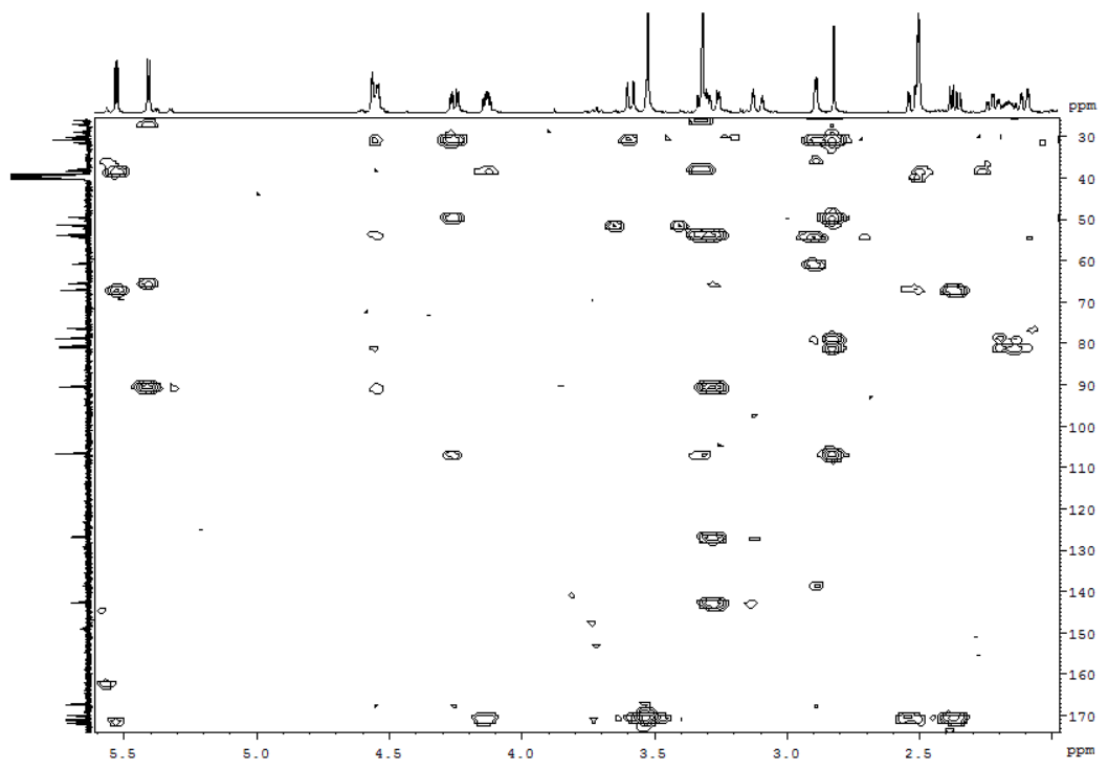
Supplementary Figure S38. ^{13}C NMR spectrum of **5** (100 MHz, $\text{DMSO-}d_6$)



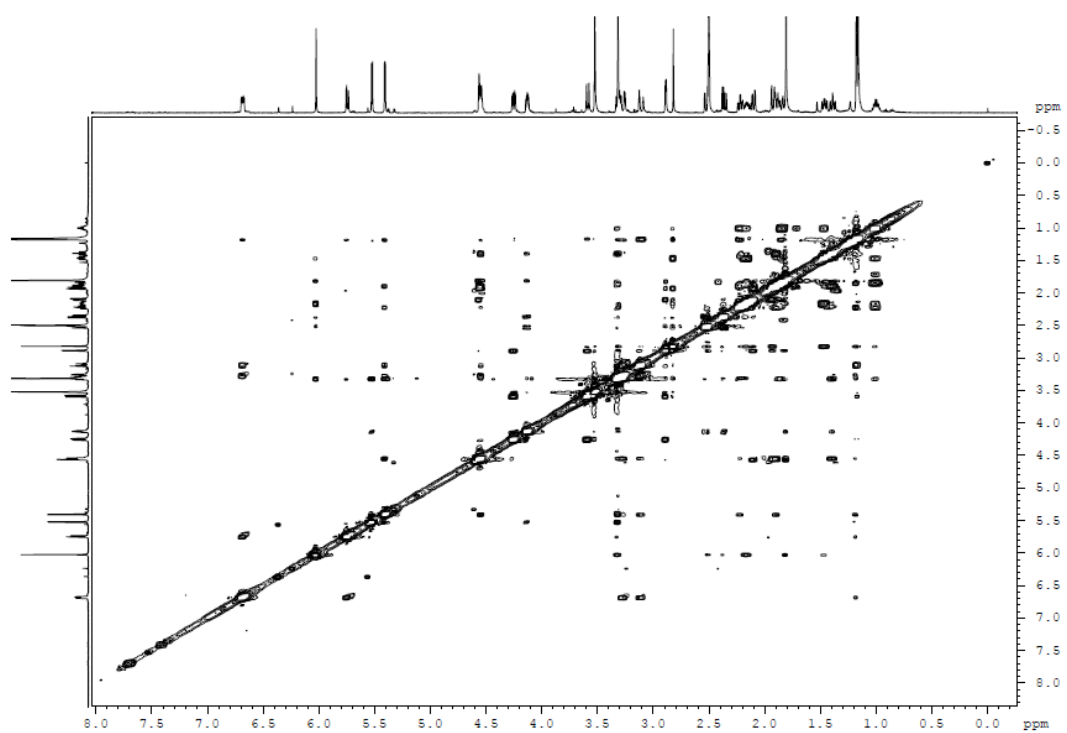
Supplementary Figure S39. HSQC spectrum of **5** (600 MHz, DMSO-*d*₆)



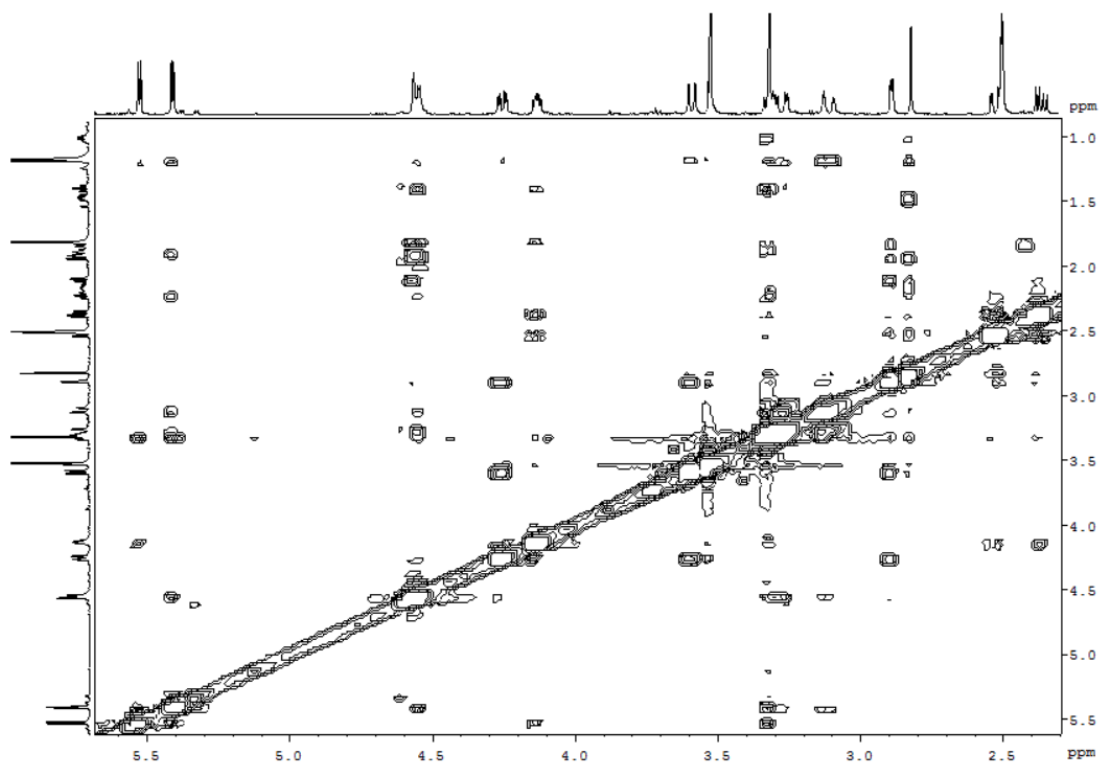
Supplementary Figure S40. HMBC spectrum of **5** (600 MHz, DMSO-*d*₆)



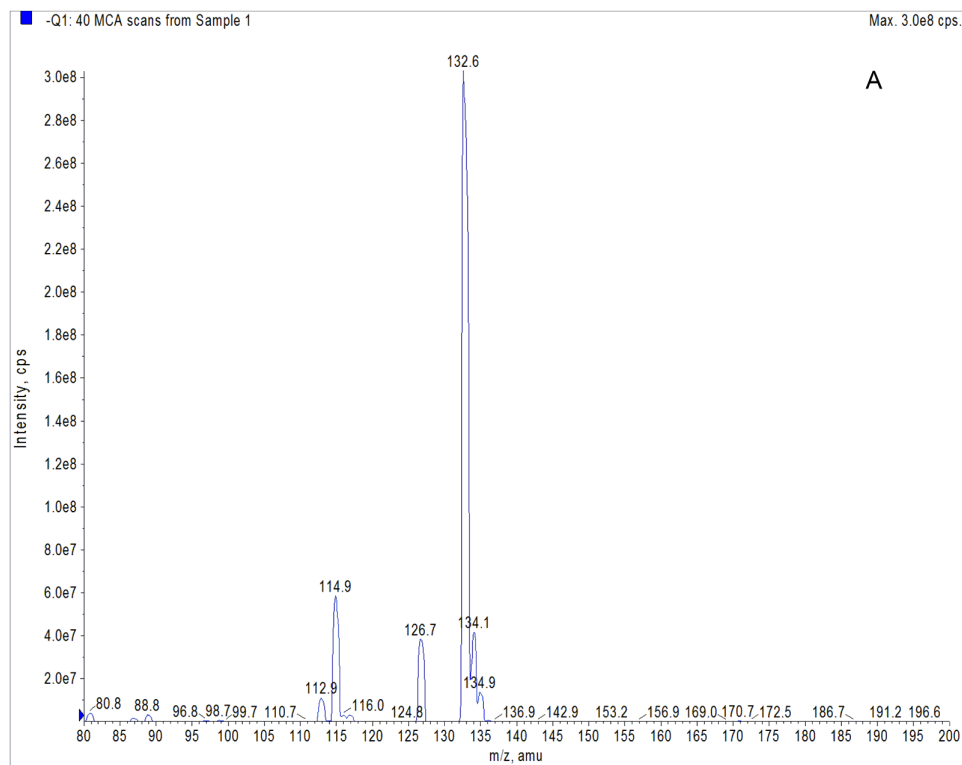
Supplementary Figure S41. Selected HMBC spectrum of **5** (600 MHz, DMSO-*d*₆)

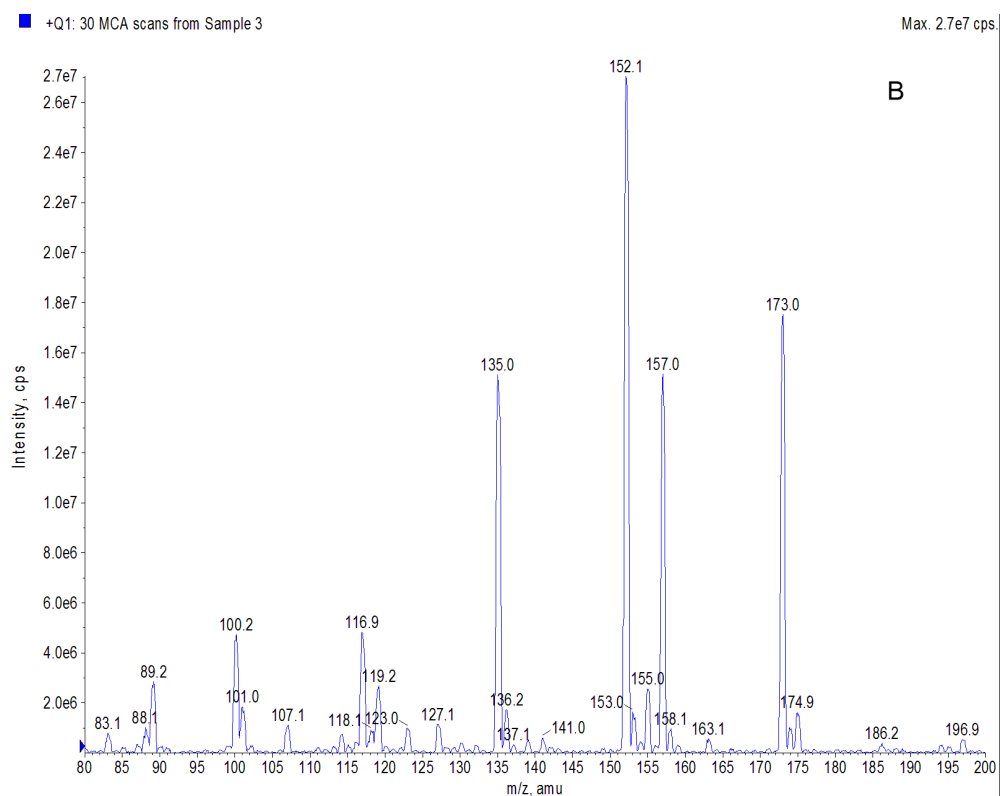


Supplementary Figure S42. NOESY spectrum of **5** (600 MHz, DMSO-*d*₆)



Supplementary Figure S43. Selected NOESY spectrum of **5** (600 MHz, DMSO-*d*₆)

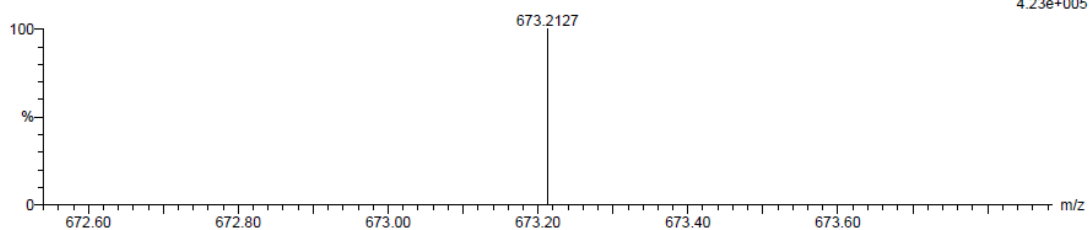




Supplementary Figure S44. MS spectrum (A, negative mode; B, positive mode) of the hydrolyzed product for **5**

Tolerance = 2.1 PPM / DBE: min = 3.0, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

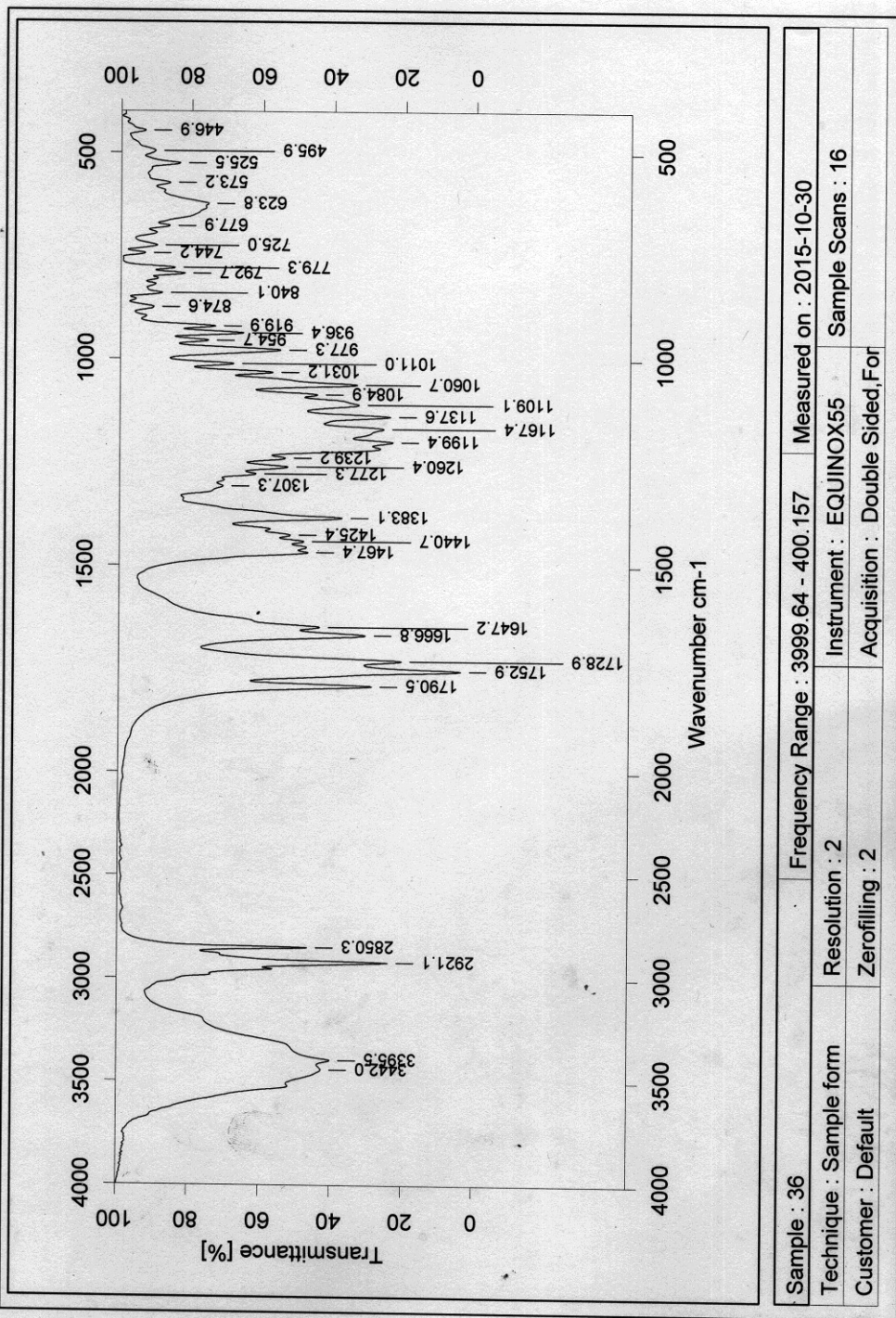
Monoisotopic Mass, Even Electron Ions
380 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-100 H: 0-100 O: 0-200 Na: 0-1
SCPs18 neg 26 (0.116)
1: TOF MS ES-



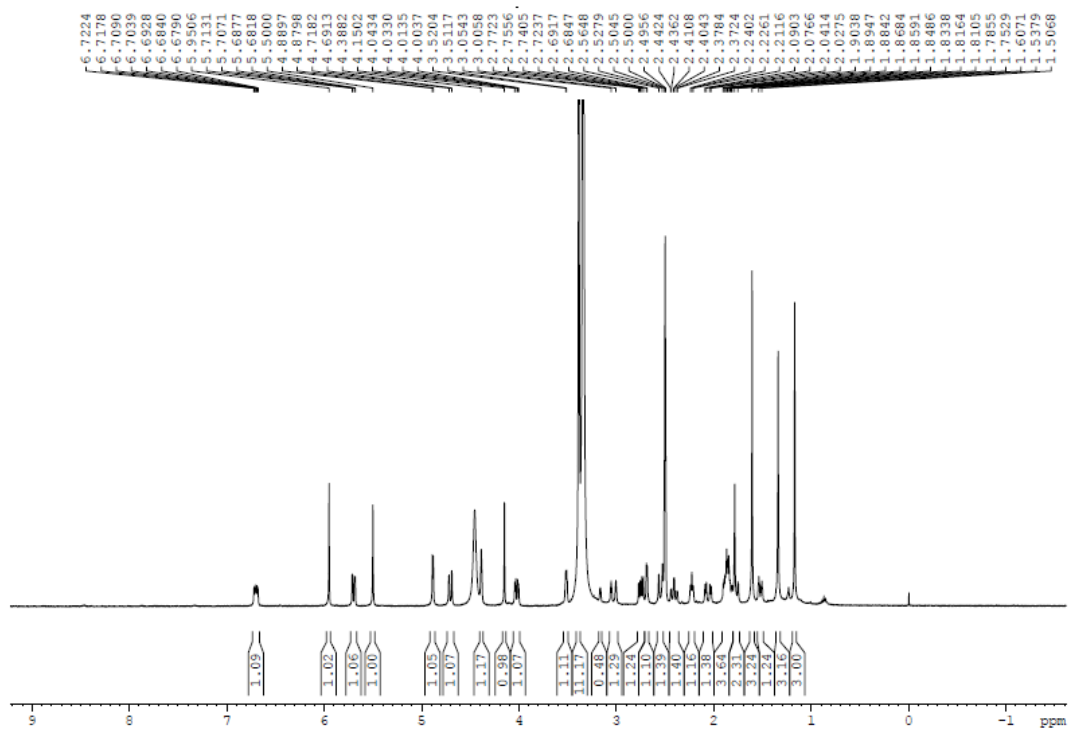
Minimum: 3.0
Maximum: 4.0 2.1 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
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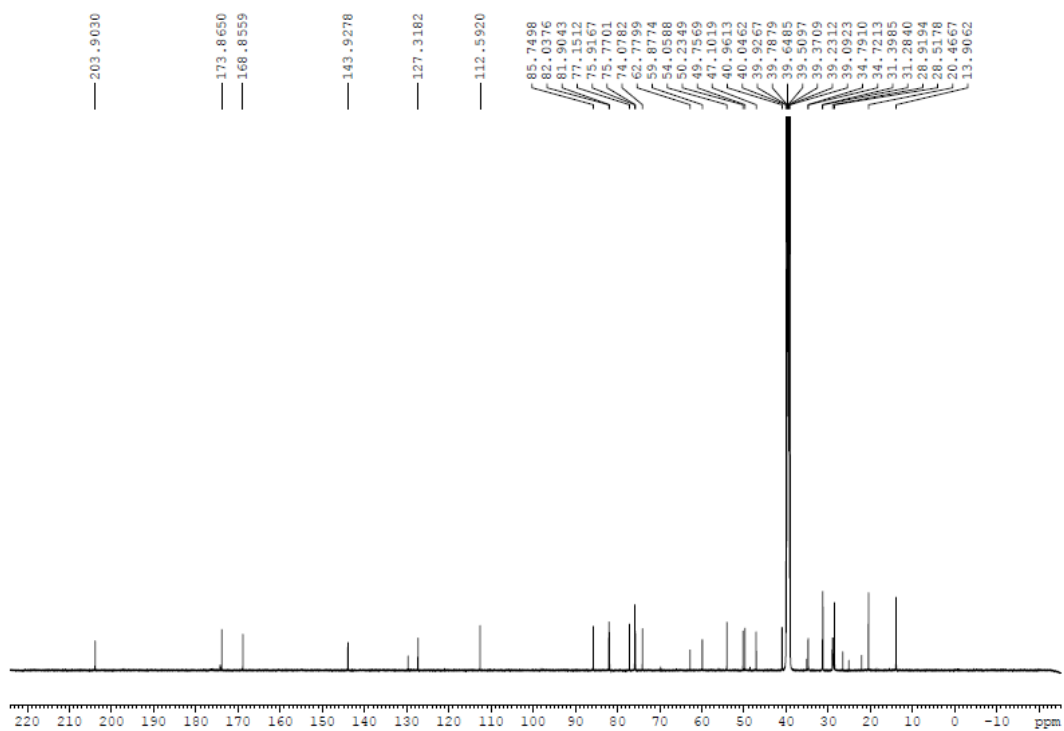
Supplementary Figure S45. HRESIMS spectrum of **5**



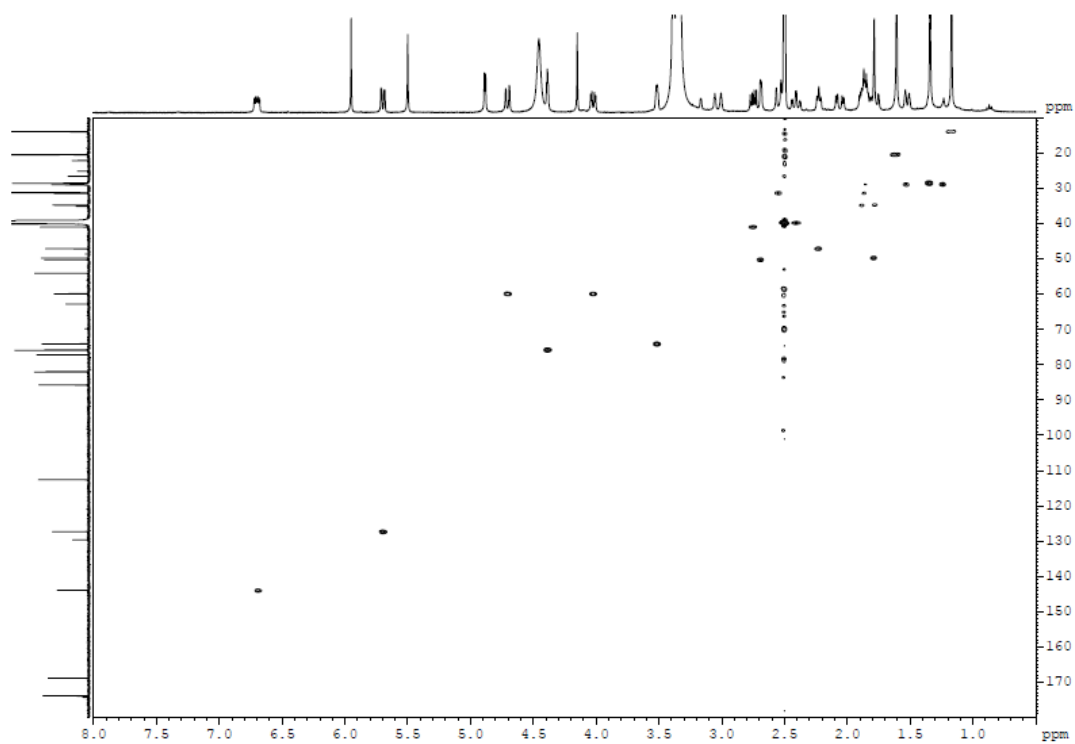
Supplementary Figure S46. IR spectrum of 5



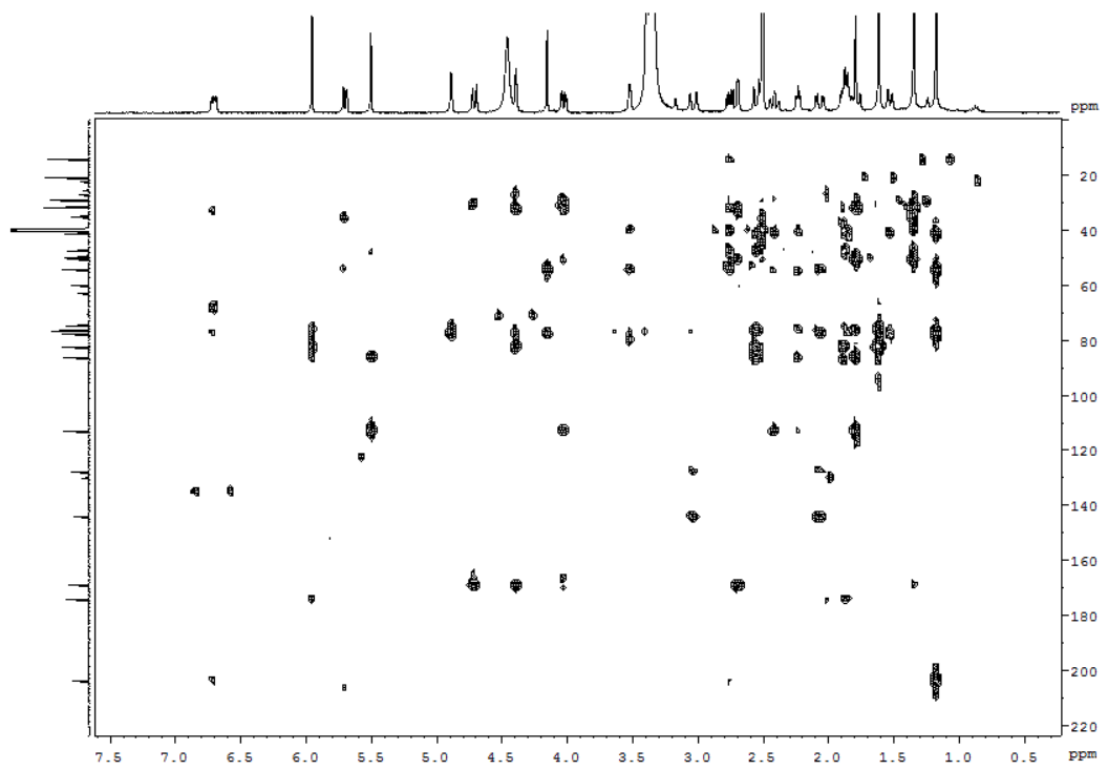
Supplementary Figure S47. ^1H NMR spectrum of **6** (600 MHz, $\text{DMSO-}d_6$)



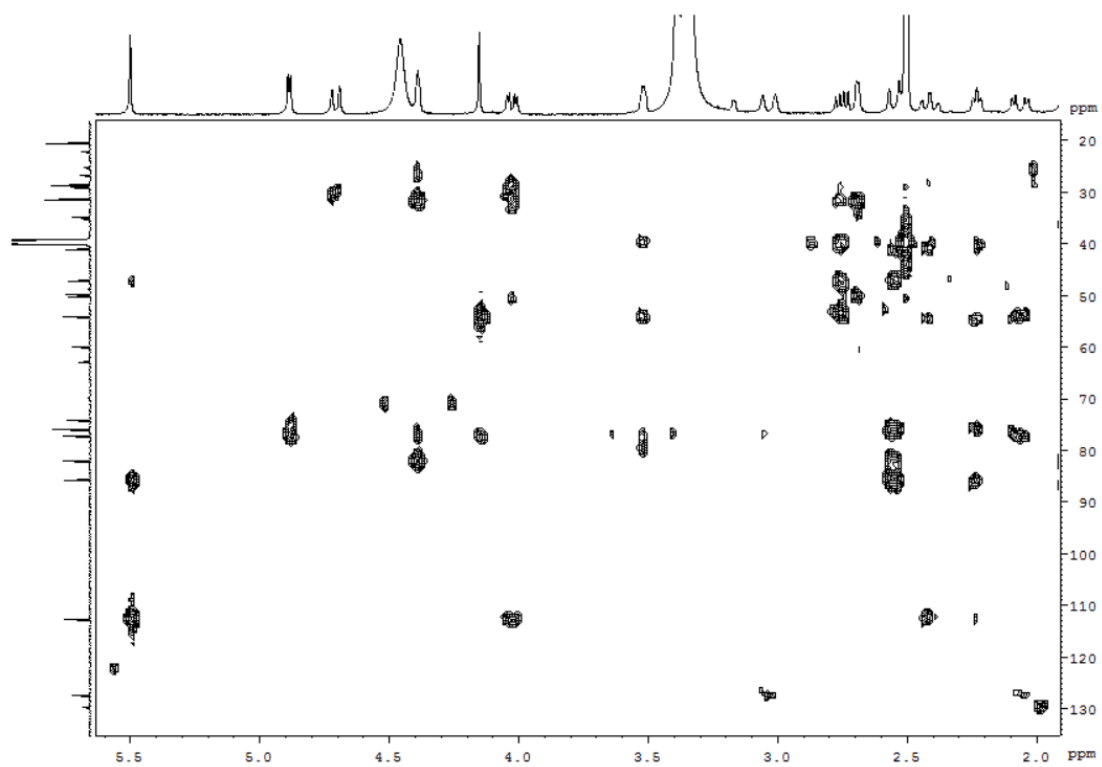
Supplementary Figure S48. ^{13}C NMR spectrum of **6** (150 MHz, $\text{DMSO}-d_6$)



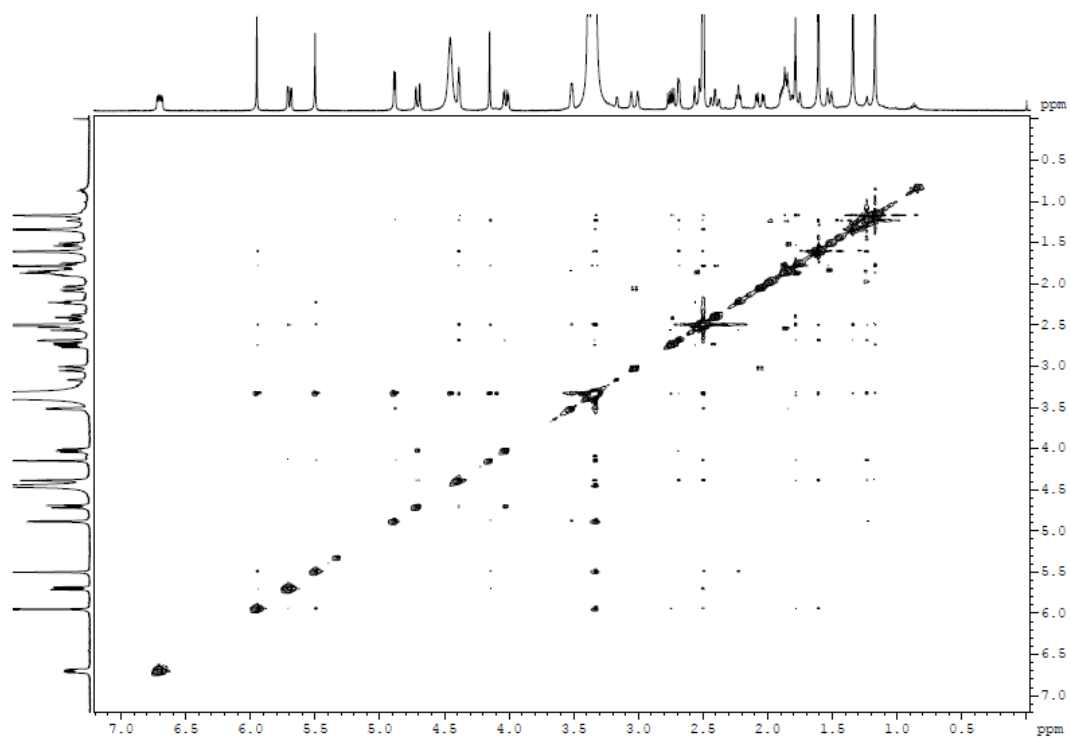
Supplementary Figure S49. HSQC spectrum of **6** (600 MHz, $\text{DMSO}-d_6$)



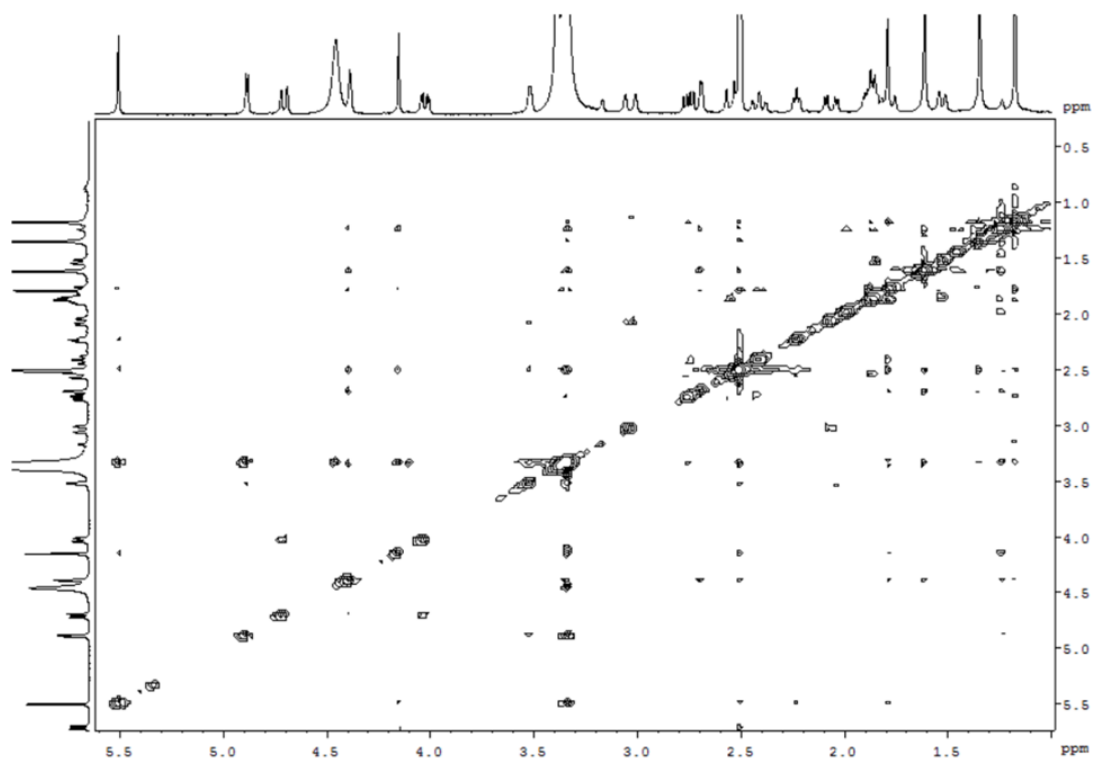
Supplementary Figure S50. HMBC spectrum of **6** (600 MHz, DMSO-*d*₆)



Supplementary Figure S51. Selected HMBC spectrum of **6** (600 MHz, DMSO-*d*₆)



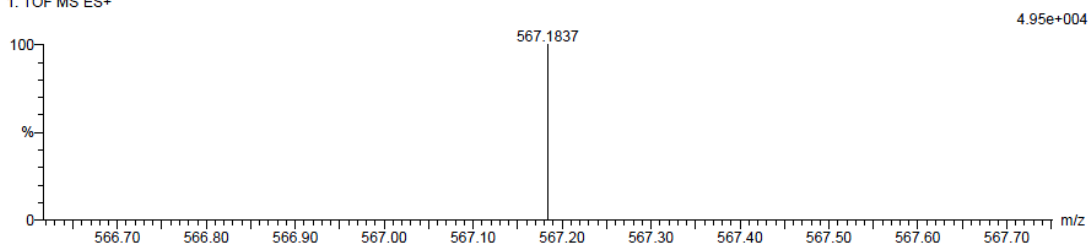
Supplementary Figure S52. NOESY spectrum of **6** (600 MHz, DMSO-*d*₆)



Supplementary Figure S53. Selected NOESY spectrum of **6** (600 MHz, DMSO-*d*₆)

Tolerance = 5.0 PPM / DBE: min = 3.0, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
234 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-38 H: 0-100 O: 0-200 Na: 0-1
SY0923pos16 24 (0.109)
1: TOF MS ES+



Minimum:				3.0					
Maximum:		4.0	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula	
567.1837	567.1842	-0.5	-0.9	12.5	34.4	n/a	n/a	C28 H32 O11 Na	

Supplementary Figure S54. HRESIMS spectrum of **6**