10.1071/CH10036_AC

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Accessory Publication

Structural Variations to the 9-*N*-methyladeninium Diterpenoid Hybrid Commonly Isolated from *Agelas* Sponges*

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[D]Department of Pathology, University of California, San Francisco, CA 94158, United States.
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RECEIVED DATE (*AJC*_Supporting_Information_*Agelas*_8R3)

***** Marine Natural Products Chemistry Special Issue.

Contents

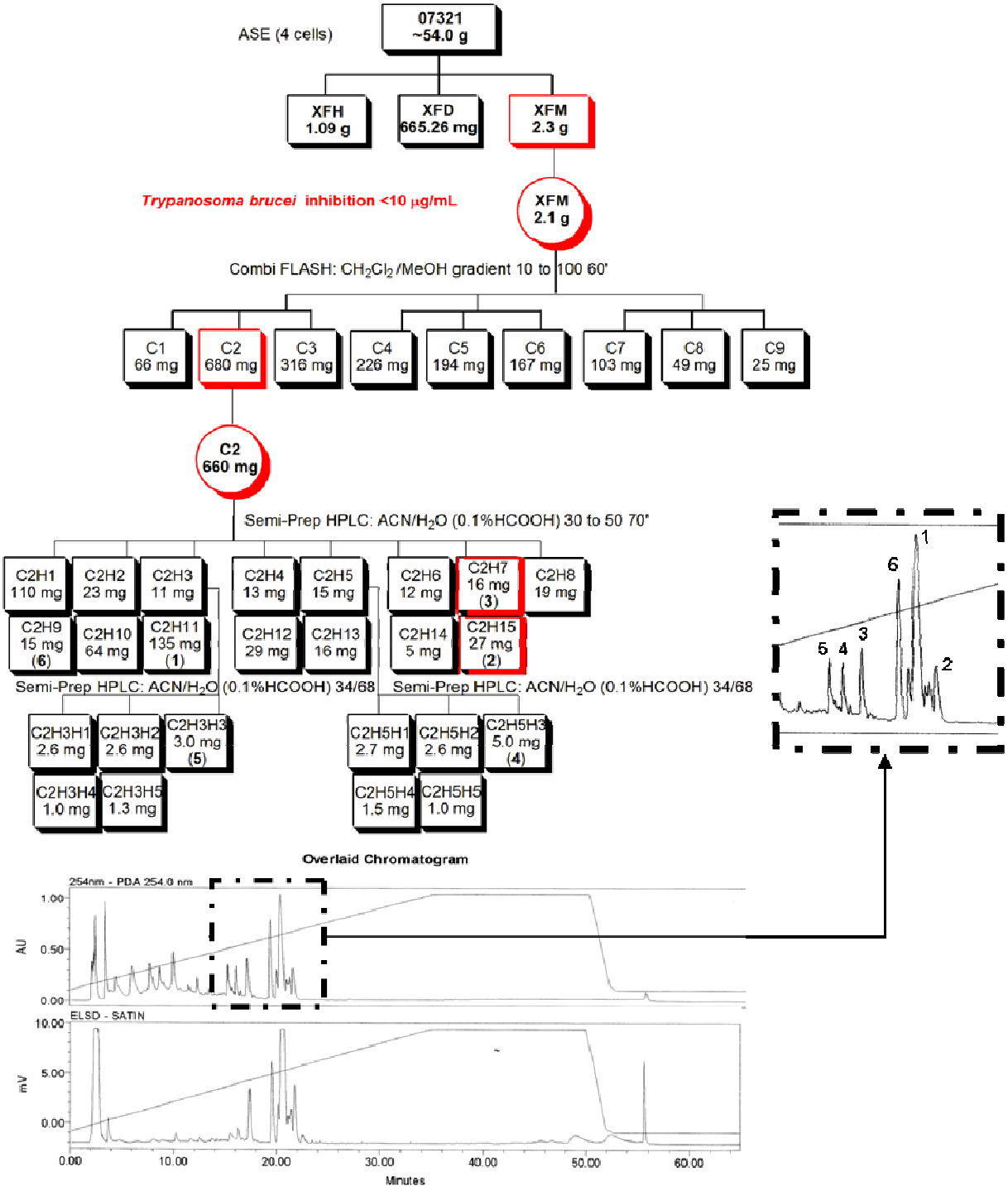


Fig. S1. ^{1}H and ^{13}C NMR data of sponge-derived 9-*N*-Methyladeninium clerodane diterpenoids

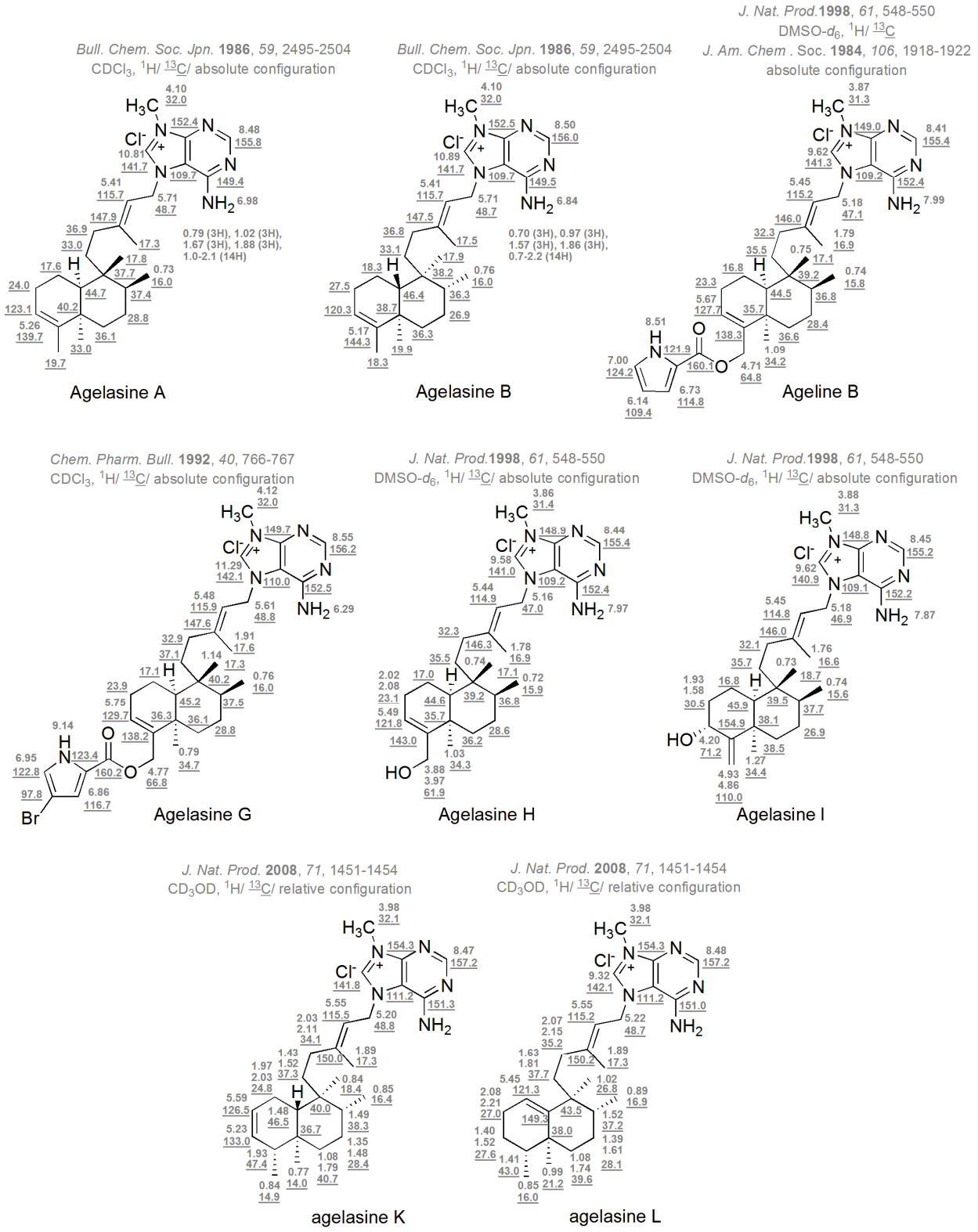
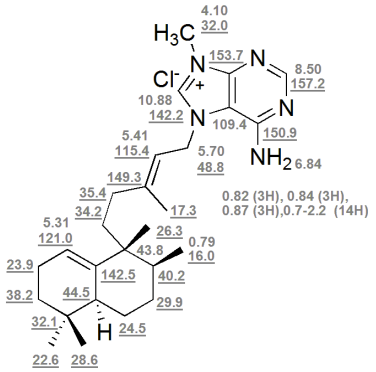


Fig. S2. ^{1}H and ^{13}C NMR data of sponge-derived 9-*N*-Methyladeninium labdane and thelepogane diterpenoids

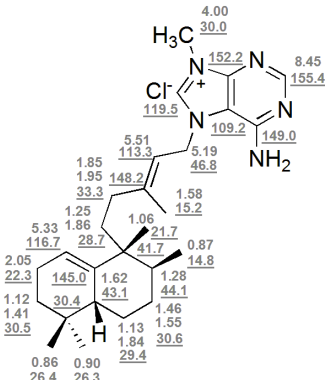
Labdane skeleton:

Bull. Chem. Soc. Jpn. **1986**, *59*, 2495-2504
$CDCl_3$, ^1H/ ^{13}C
Tetrahedron **2005**, *61*, 11672-11678
absolute configuration revised



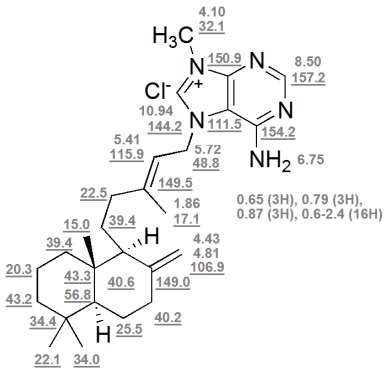
Agelasine C

J. Nat. Prod. **1997**, *60*, 411-413
CD_3OD, ^1H/ ^{13}C
Tetrahedron **2005**, *61*, 11672-11678
absolute configuration revised



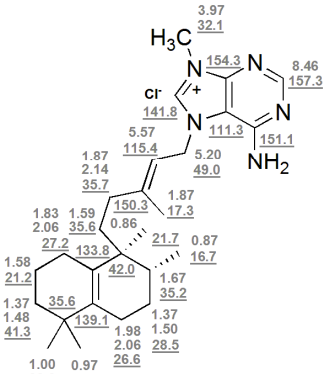
epi-Agelasine C

Bull. Chem. Soc. Jpn. **1986**, *59*, 2495-2504
$CDCl_3$, ^1H/ ^{13}C/ absolute configuration



Agelasine D

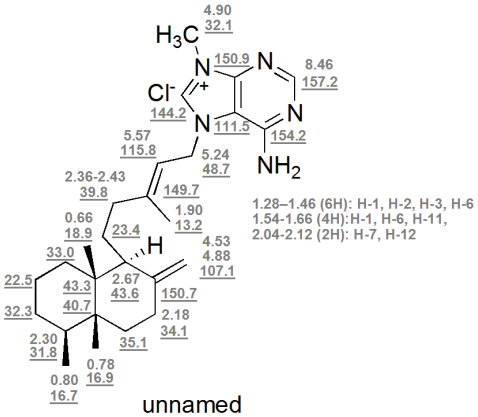
J. Nat. Prod. **2008**, *71*, 1451-1454
CD_3OD, ^1H/ ^{13}C/ absolute configuration



Agelasine J

Thelepogane skeleton:

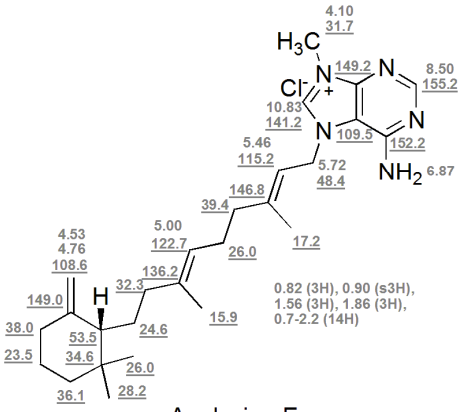
J. Nat. Prod. **1998**, *61*, 1310-1312
CD_3OD, ^1H/ ^{13}C/ relative configuration



unnamed

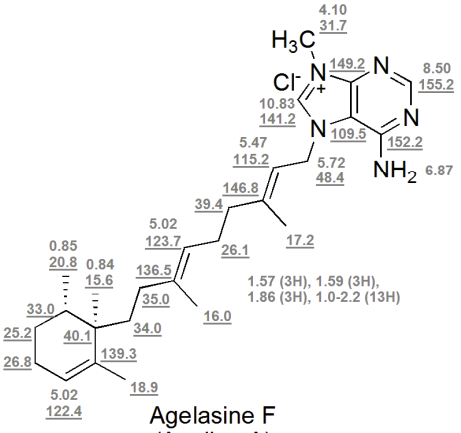
Fig. S3. ^1H and ^{13}C NMR data of sponge-derived 9-*N*-Methyladeninium monocyclic diterpenoids

Bull. Chem. Soc. Jpn. **1986**, *59*, 2495-2504
$CDCl_3$, ^1H/ ^{13}C
Tetrahedron lett. **1984**, *25*, 3719-3722
absolute configuration



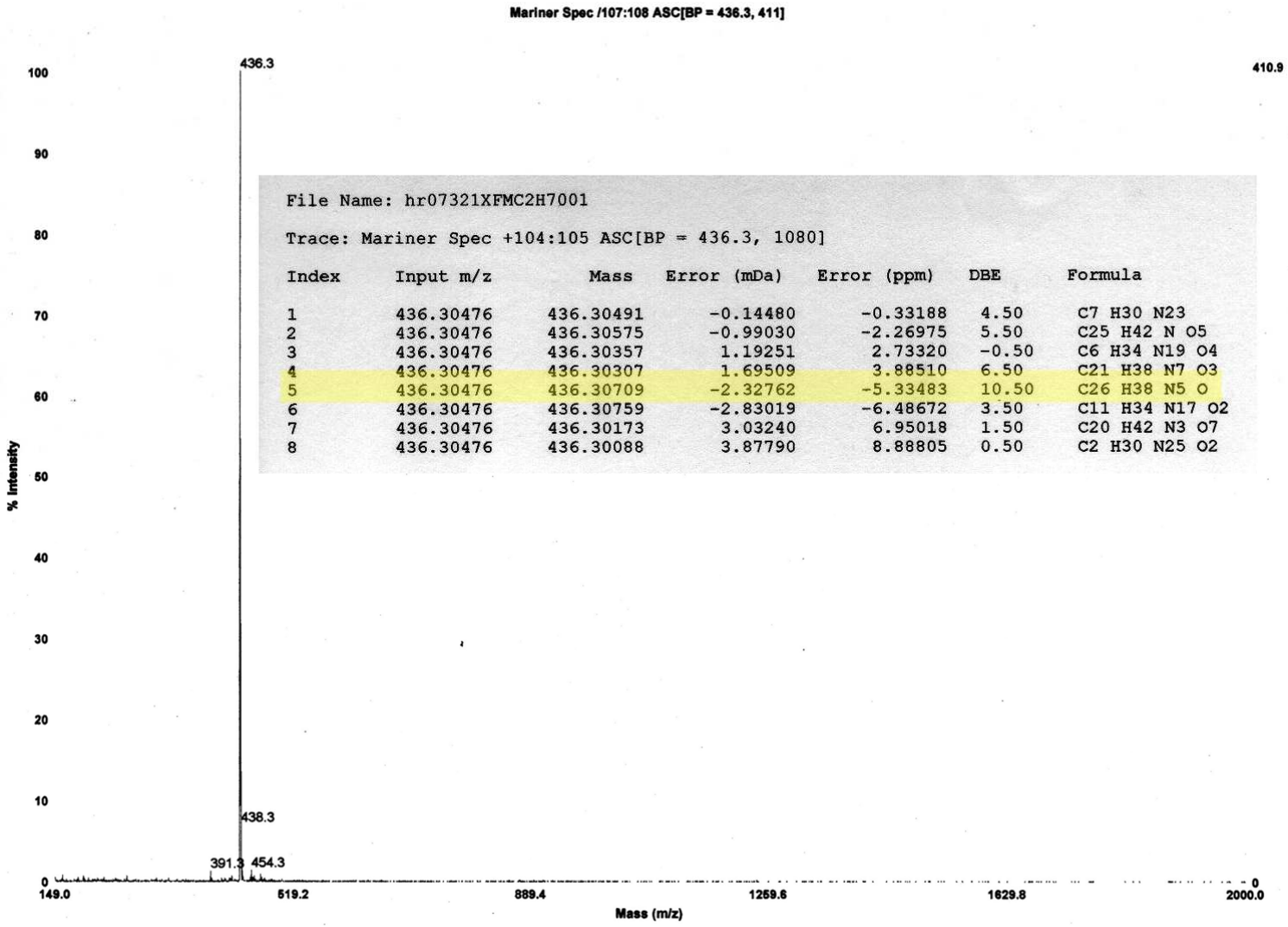
Agelasine E

Bull. Chem. Soc. Jpn. **1986**, *59*, 2495-2504
$CDCl_3$, ^1H/ ^{13}C
Tetrahedron lett. **1984**, *25*, 3719-3722
absolute configuration



Agelasine F
(Ageline A)

Fig. S4. Mass data HRESIMS in positive mode of compound **3**.



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3	436.30476	436.30357	1.19251	2.73320	-0.50	C6 H34 N19 O4
4	436.30476	436.30307	1.69509	3.88510	6.50	C21 H38 N7 O3
5	436.30476	436.30709	-2.32762	-5.33483	10.50	C26 H38 N5 O
6	436.30476	436.30759	-2.83019	-6.48672	3.50	C11 H34 N17 O2
7	436.30476	436.30173	3.03240	6.95018	1.50	C20 H42 N3 O7
8	436.30476	436.30088	3.87790	8.88805	0.50	C2 H30 N25 O2

Fig. S5. ^{1}H NMR spectrum of compound **3** (600 MHz, CD_3OD).

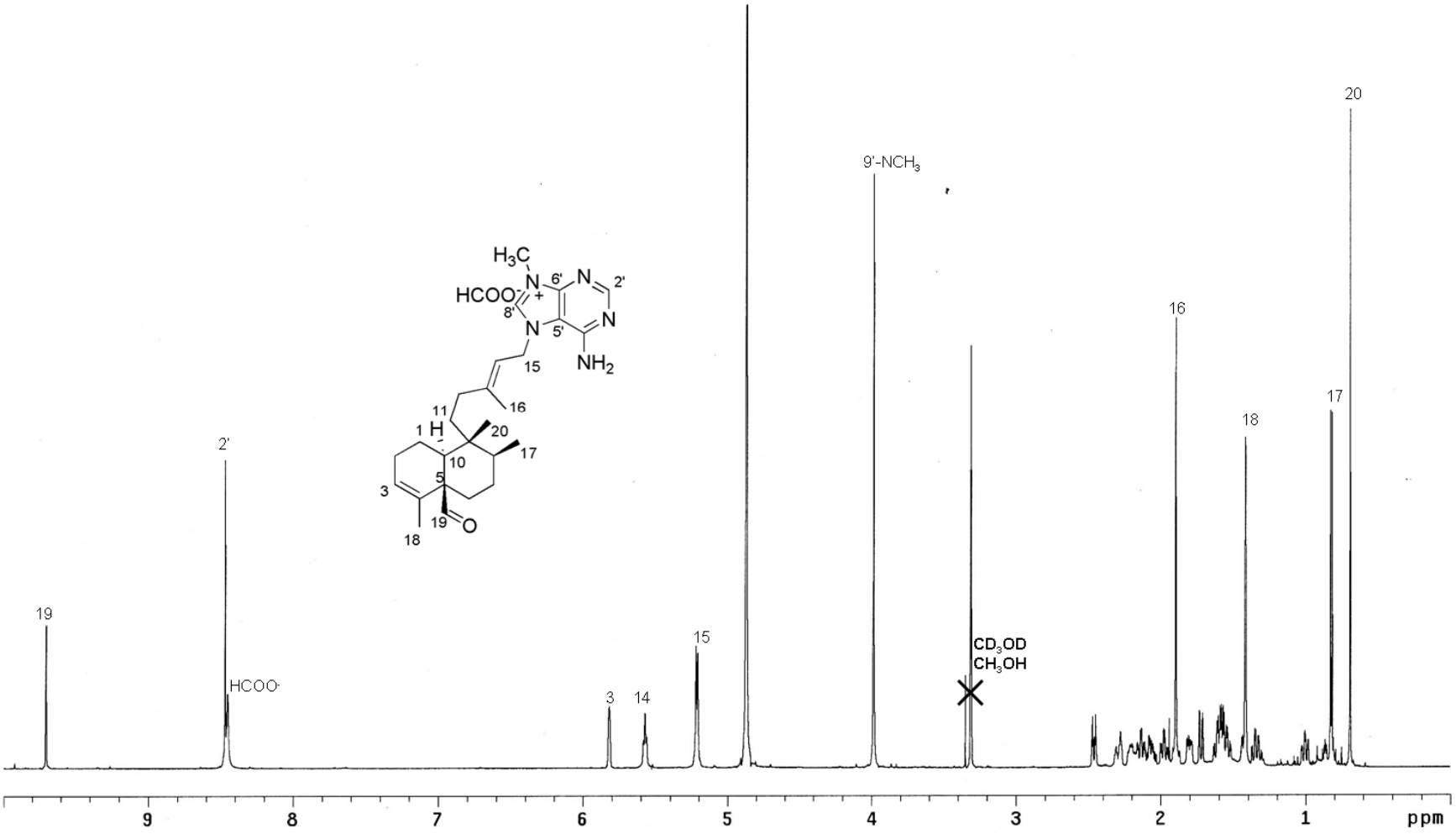

Fig. S6. ^{13}C NMR spectrum of compound **3** (125 MHz, CD_3OD).

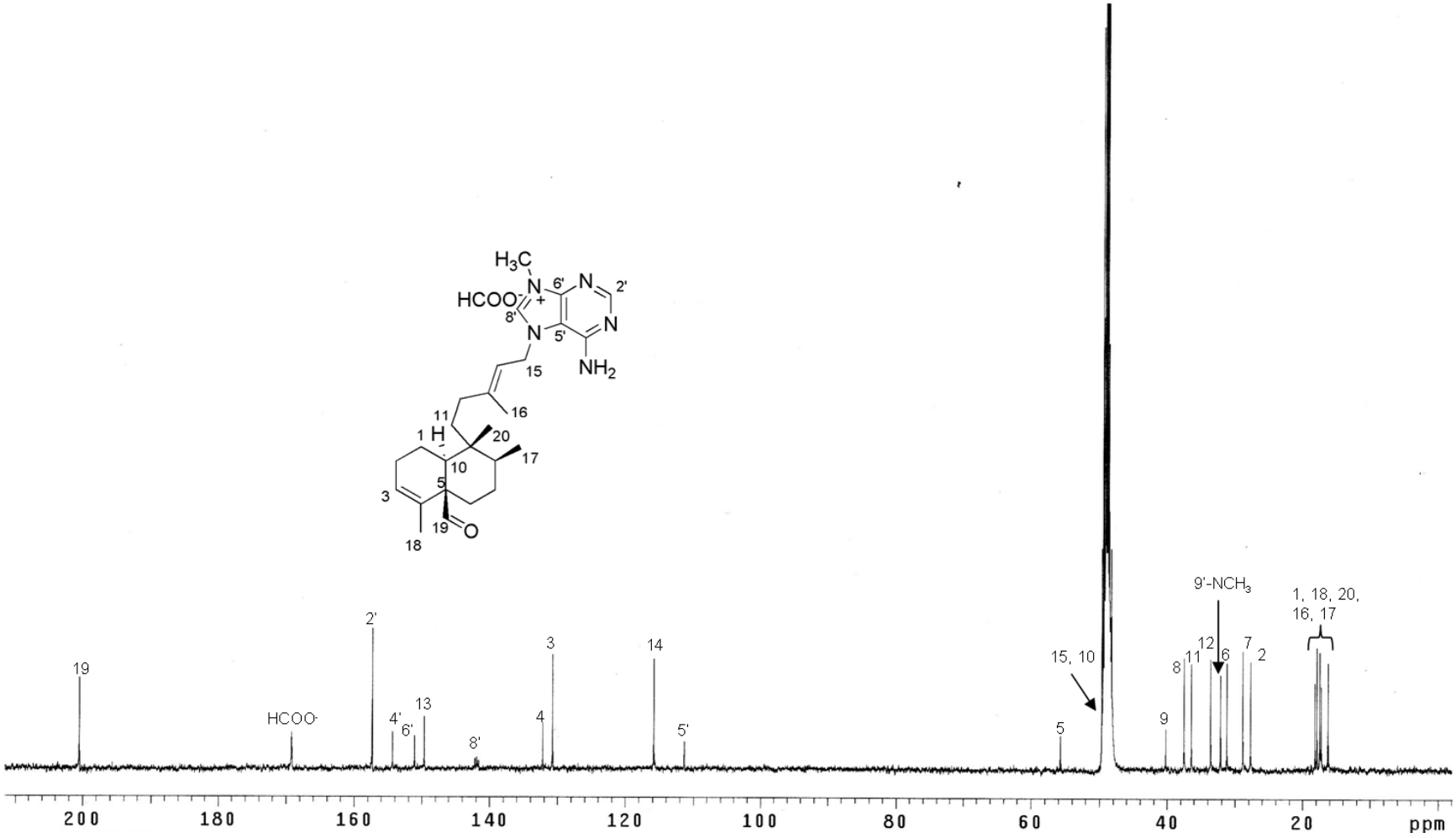

Fig. S7. DEPT spectrum of compound **3** (125 MHz, CD_3OD).

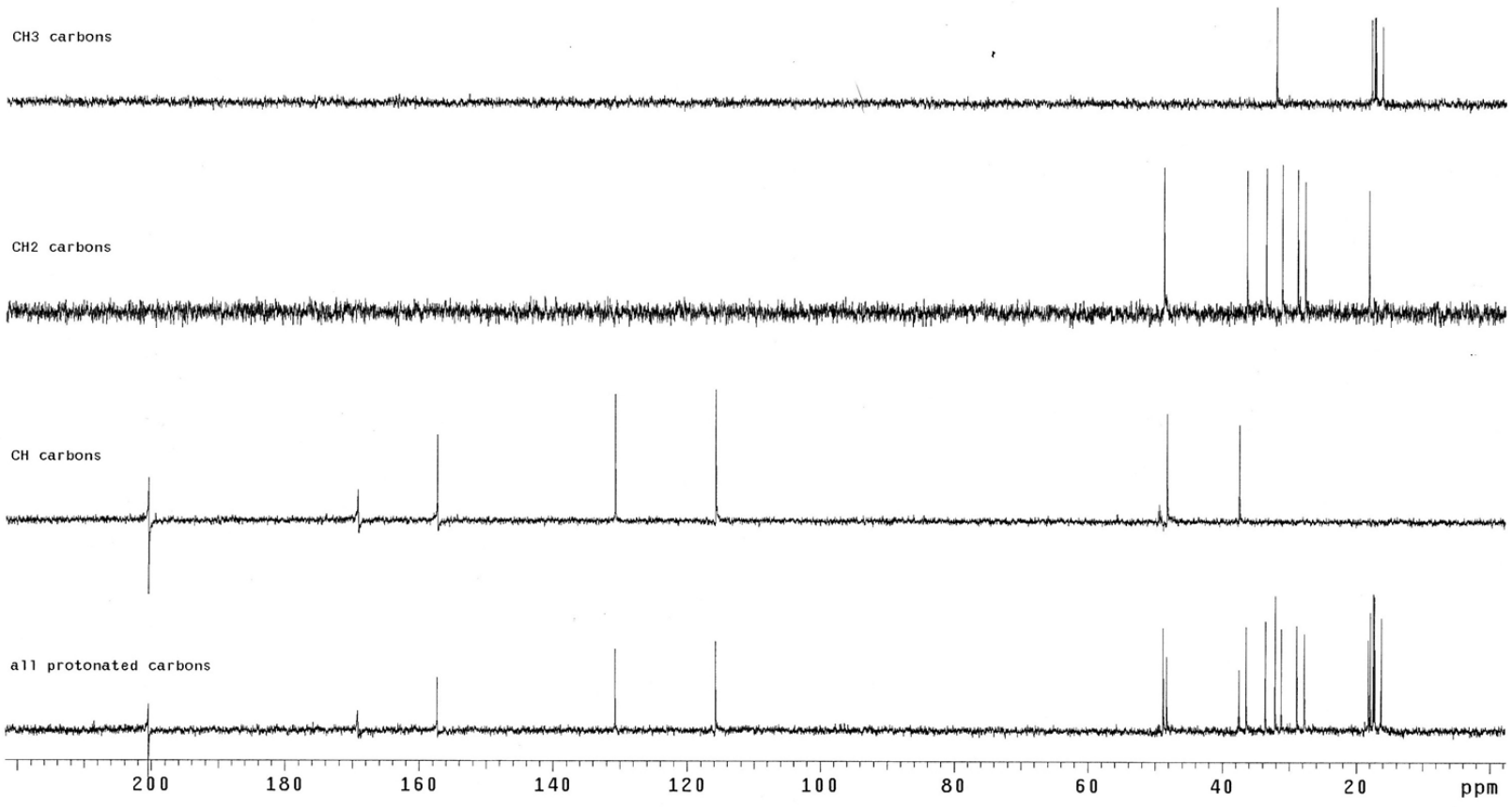

Fig. S8. *g*COSY spectrum of compound **3** (600 MHz, CD_3OD).

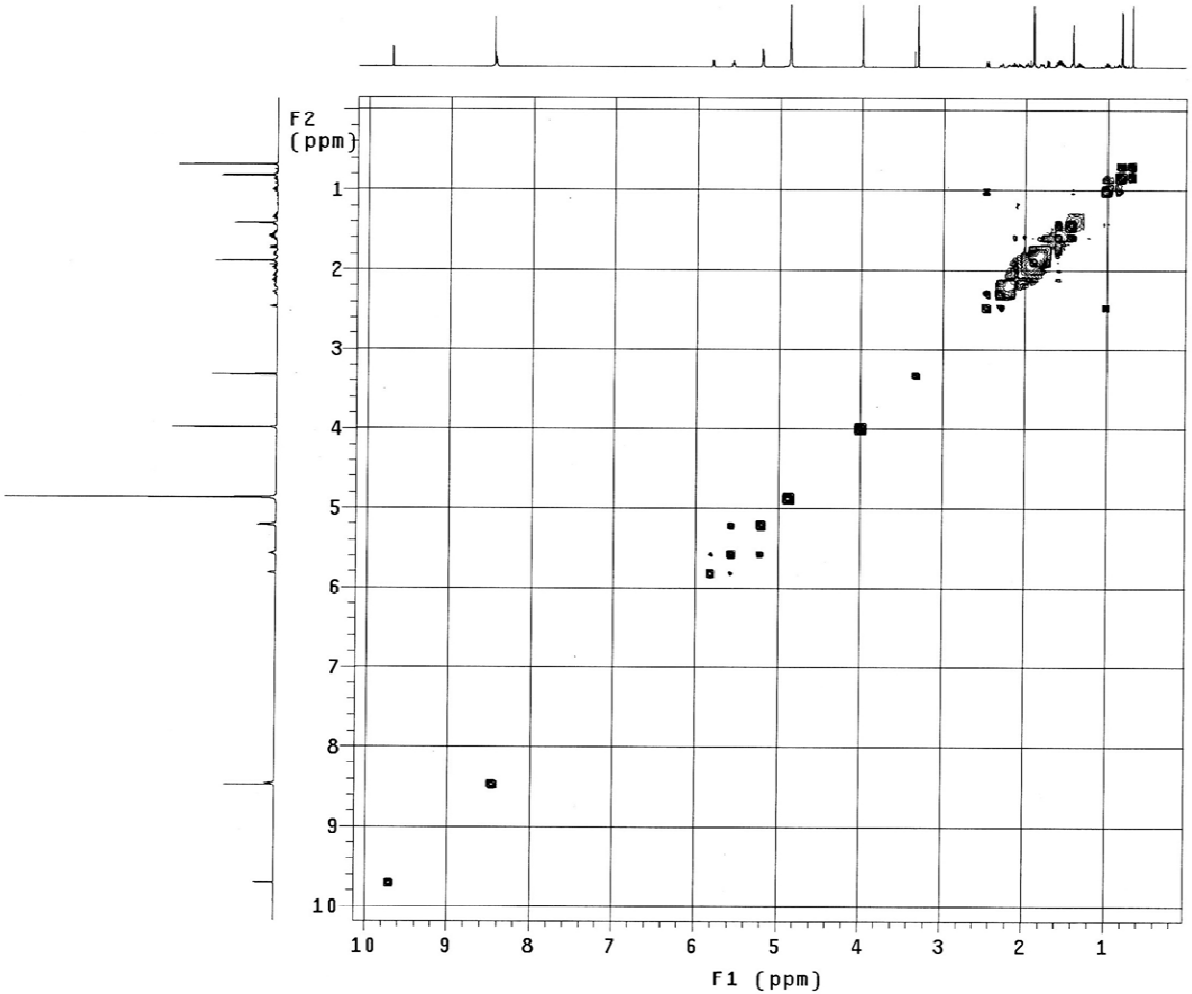

Fig. S9. HMQC spectrum of compound **3** (600 MHz, CD_3OD).

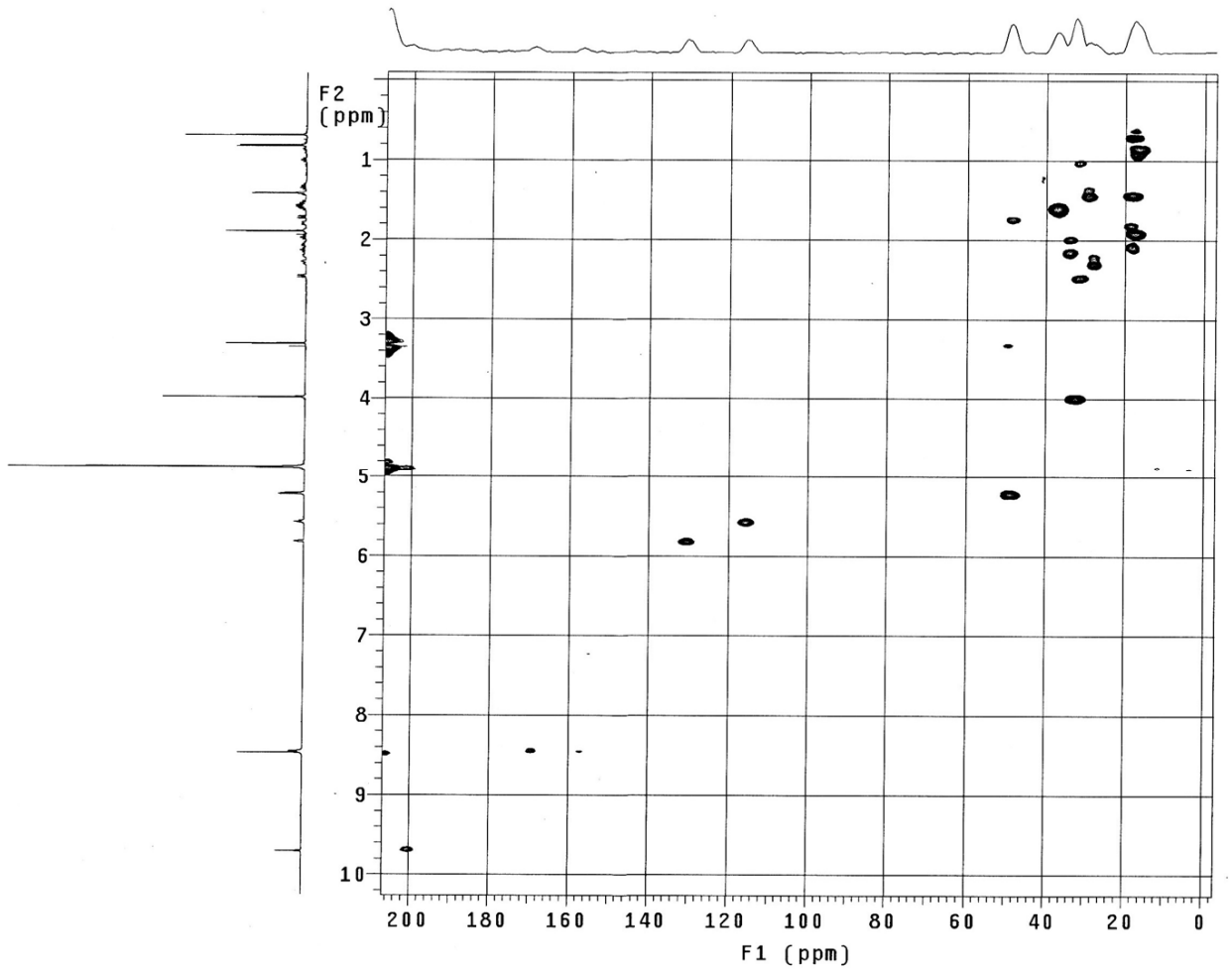

Fig. S10. *g*HMBC spectrum of compound **3** (600 MHz, CD_3OD).

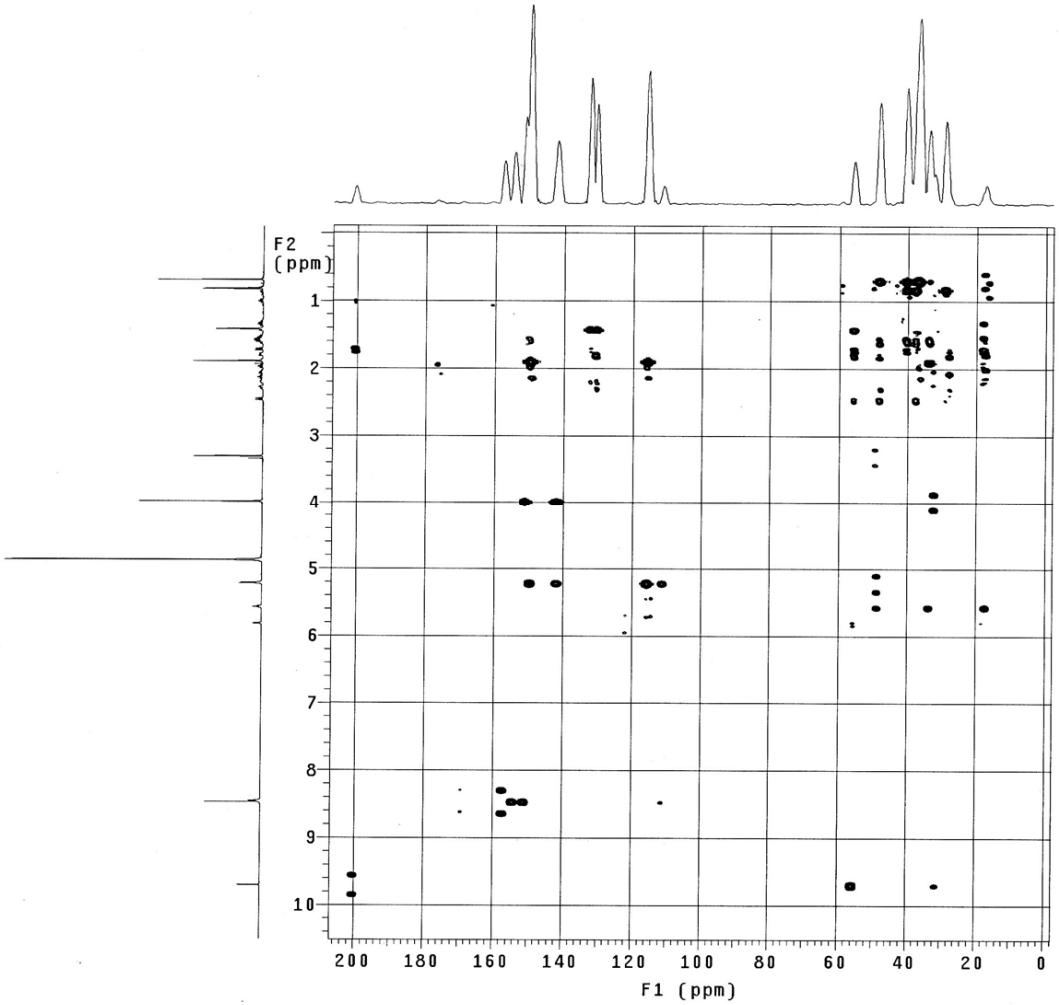

Fig. S11. 2D NOESY spectrum of compound **3** (600 MHz, CD_3OD).

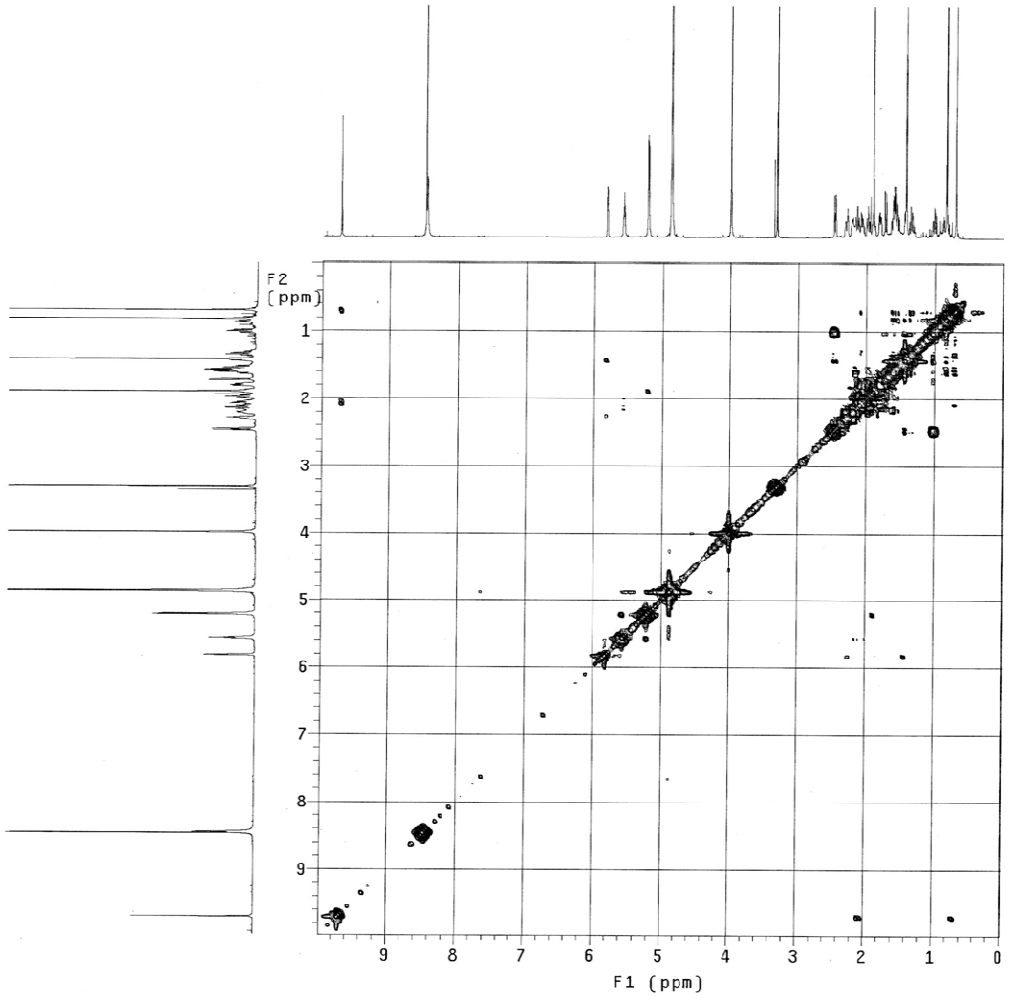

Fig. S12. IR spectrum of compound **3** (NaCl).

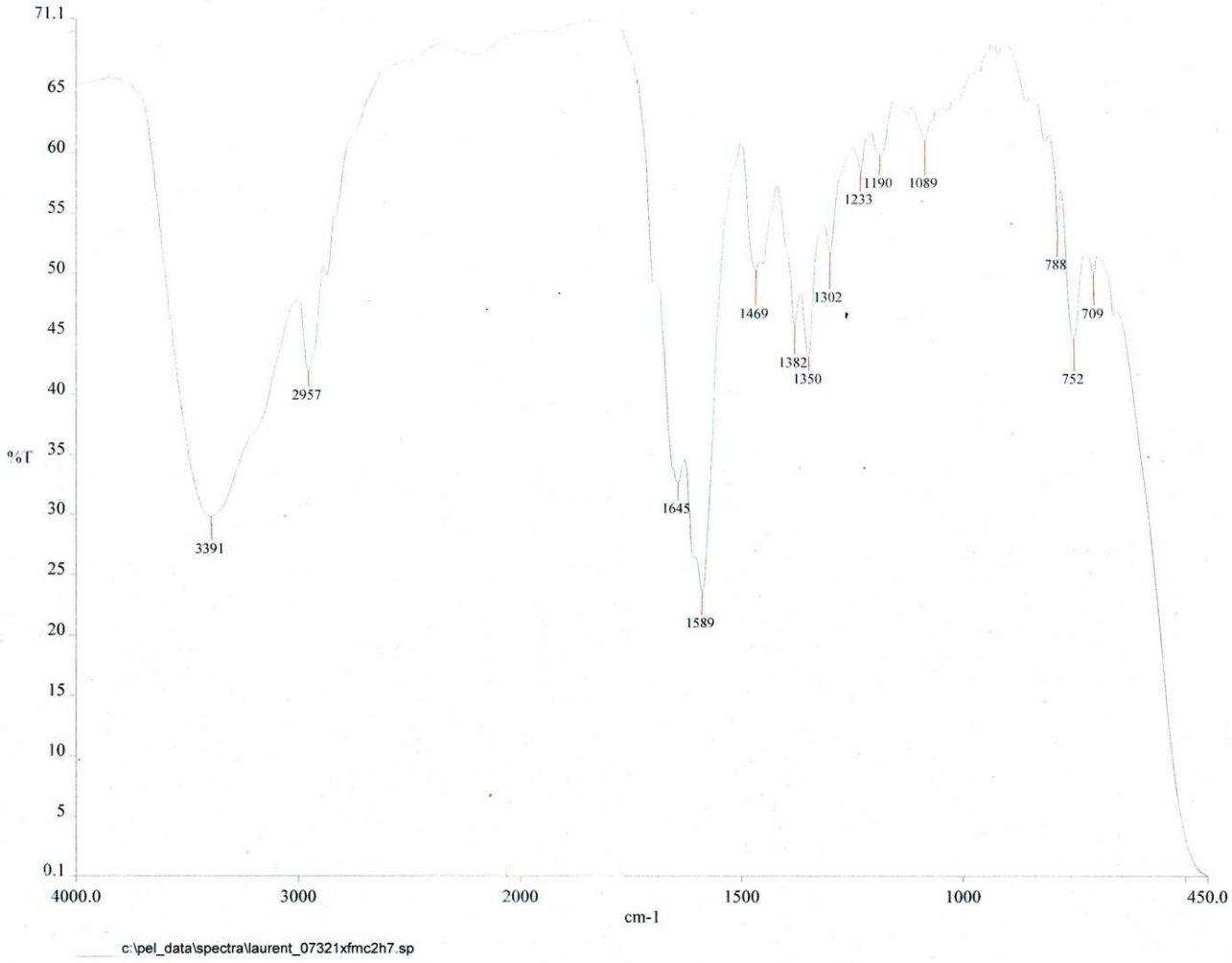

Fig. S13. UV spectrum of compound **3** (MeOH).

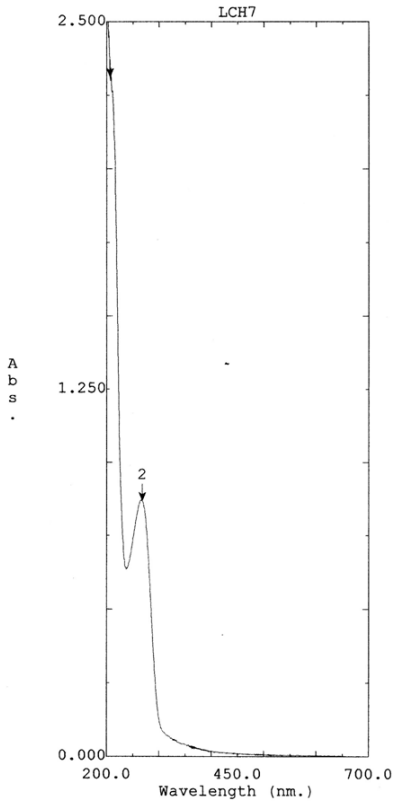

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Fig. S14. Mass data HRESIMS in positive mode of compound **4**.

Mass Spectrometry & Proteomics Facility

Mass Spectrum SmartFormula Report

Analysis Info

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Sample Name	07321xfmc2h5h3	Instrument / Ser#	micrOTOF 10314
Comment	in CH3CN		

Acquisition Parameter

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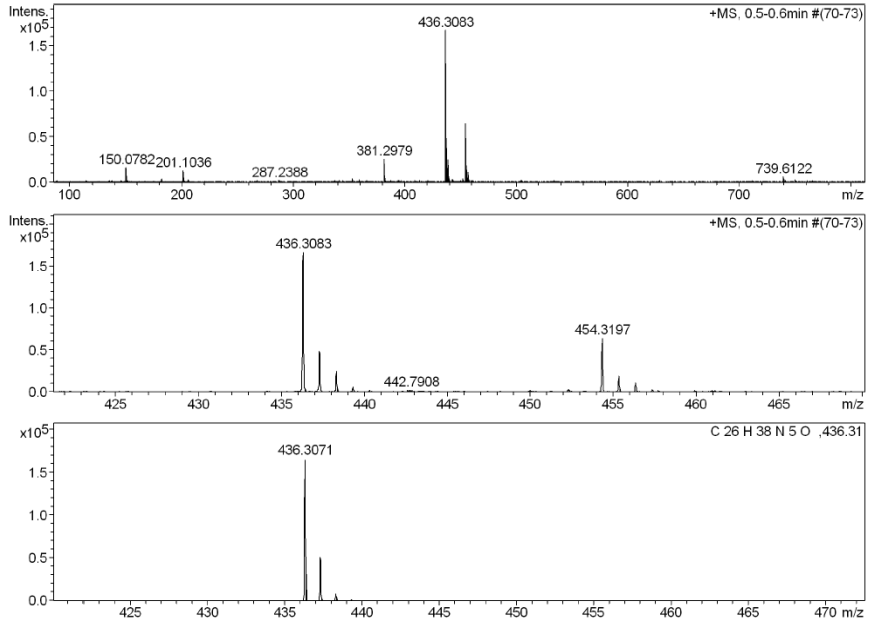


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf
436.3083	1	C 26 H 38 N 5 O	436.3071	-2.8	-4.7	10.5	ok	even

Fig. S15. ^{1}H NMR spectrum of compound **4** (600 MHz, CD_3OD).

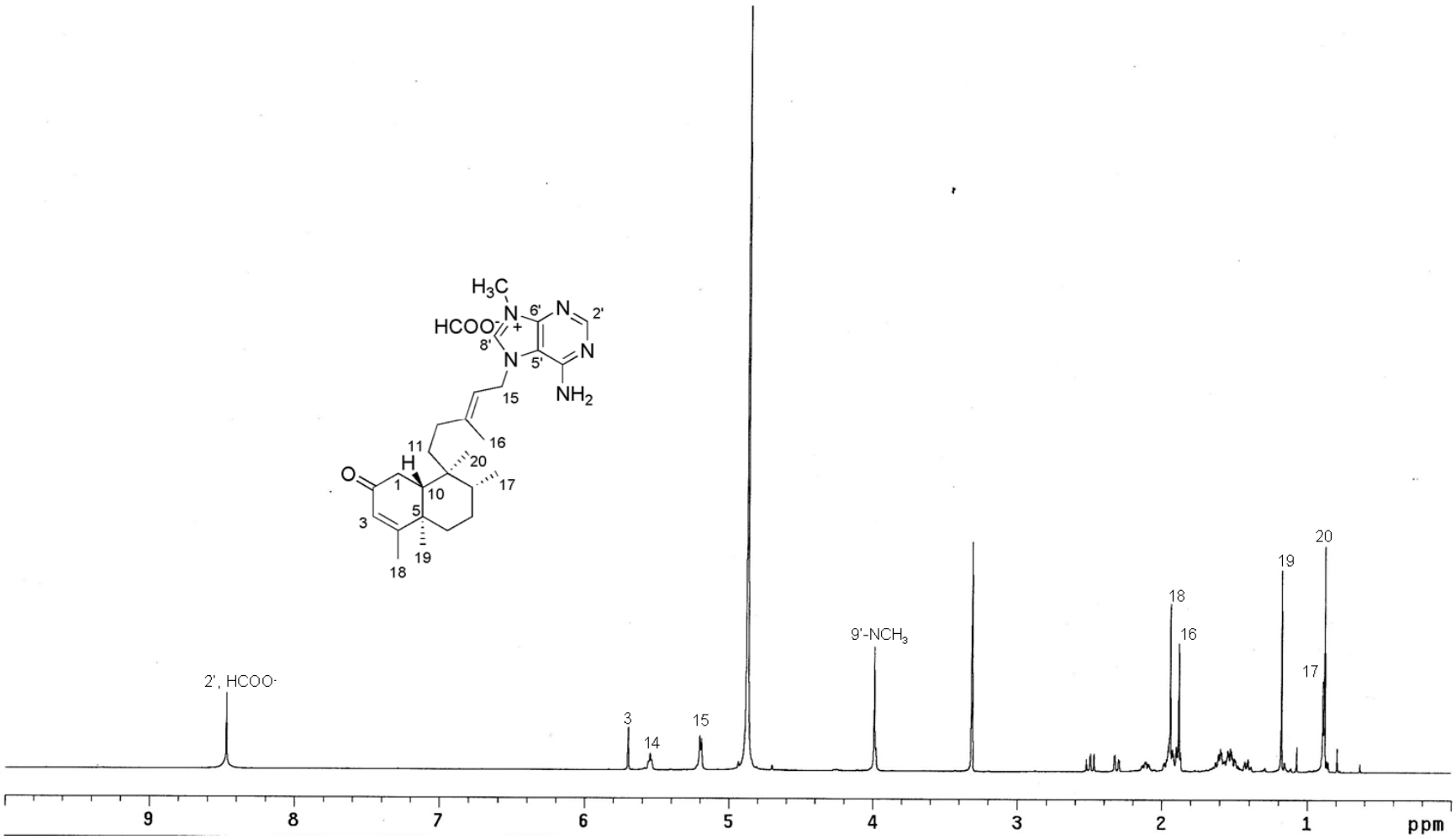


Fig. S16. ^{13}C NMR spectrum of compound **4** (125 MHz, CD_3OD).

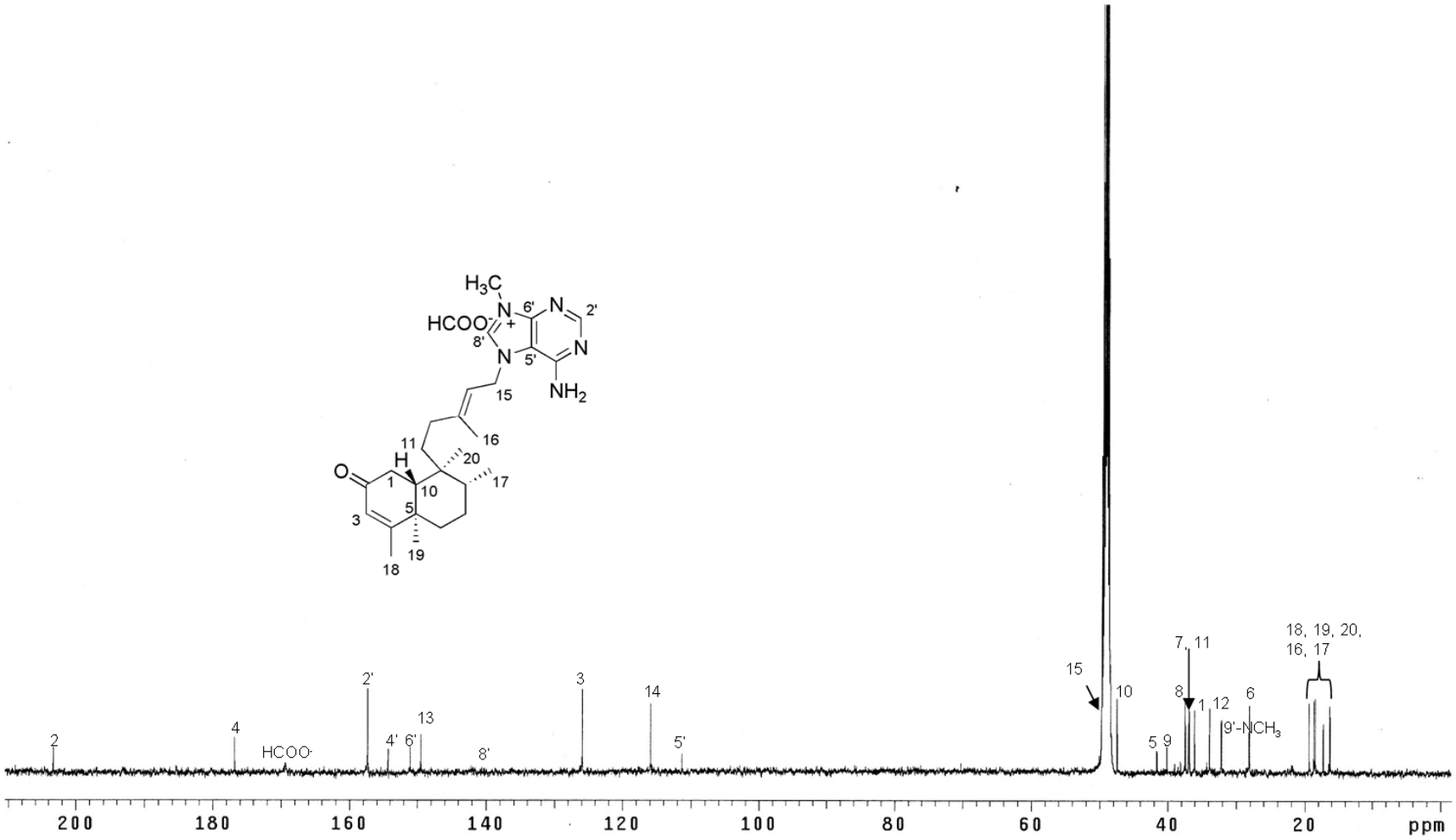


Fig. S17. DEPT spectrum of compound **4** (125 MHz, CD_3OD).

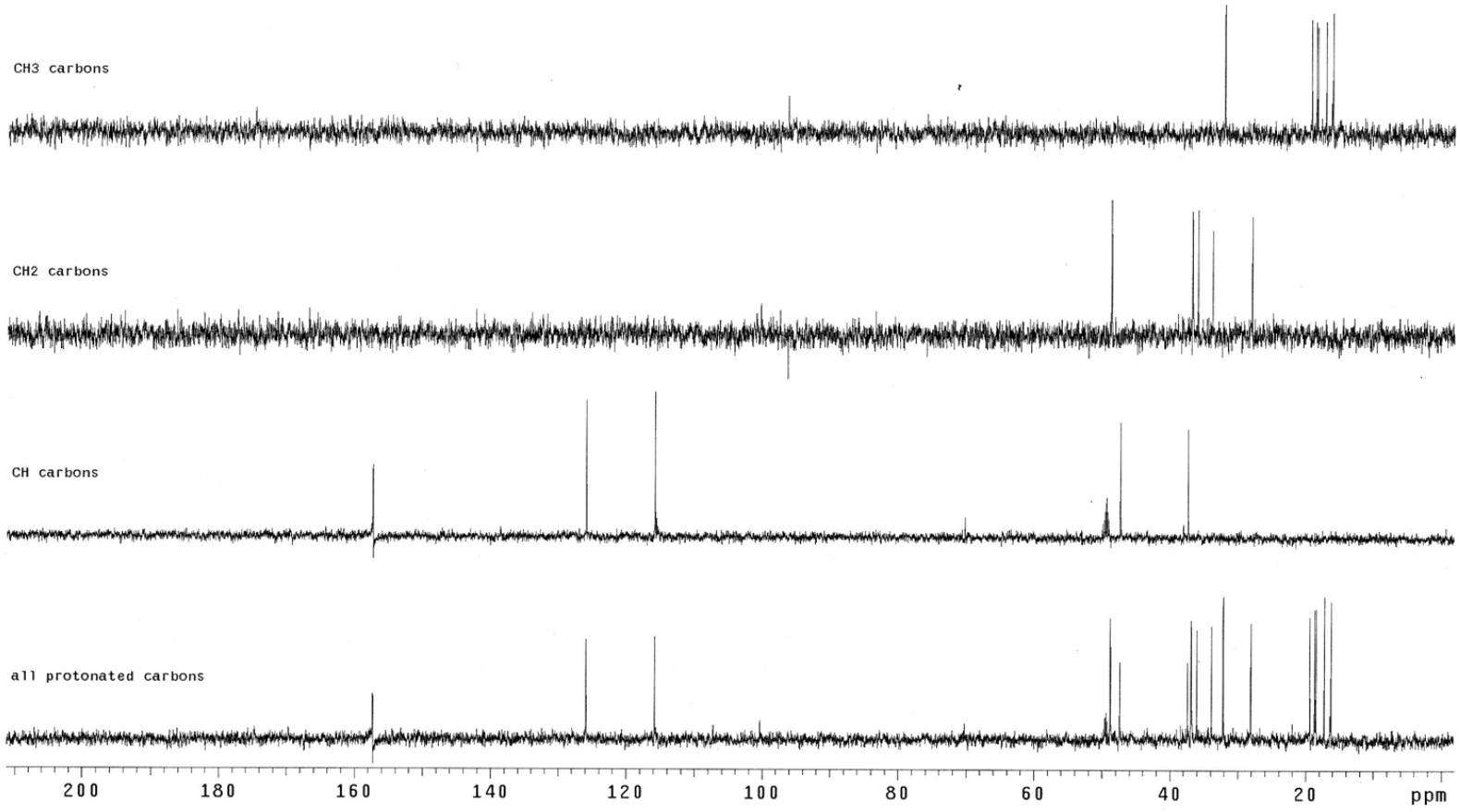


Fig. S18. *g*COSY spectrum of compound **4** (600 MHz, CD_3OD).

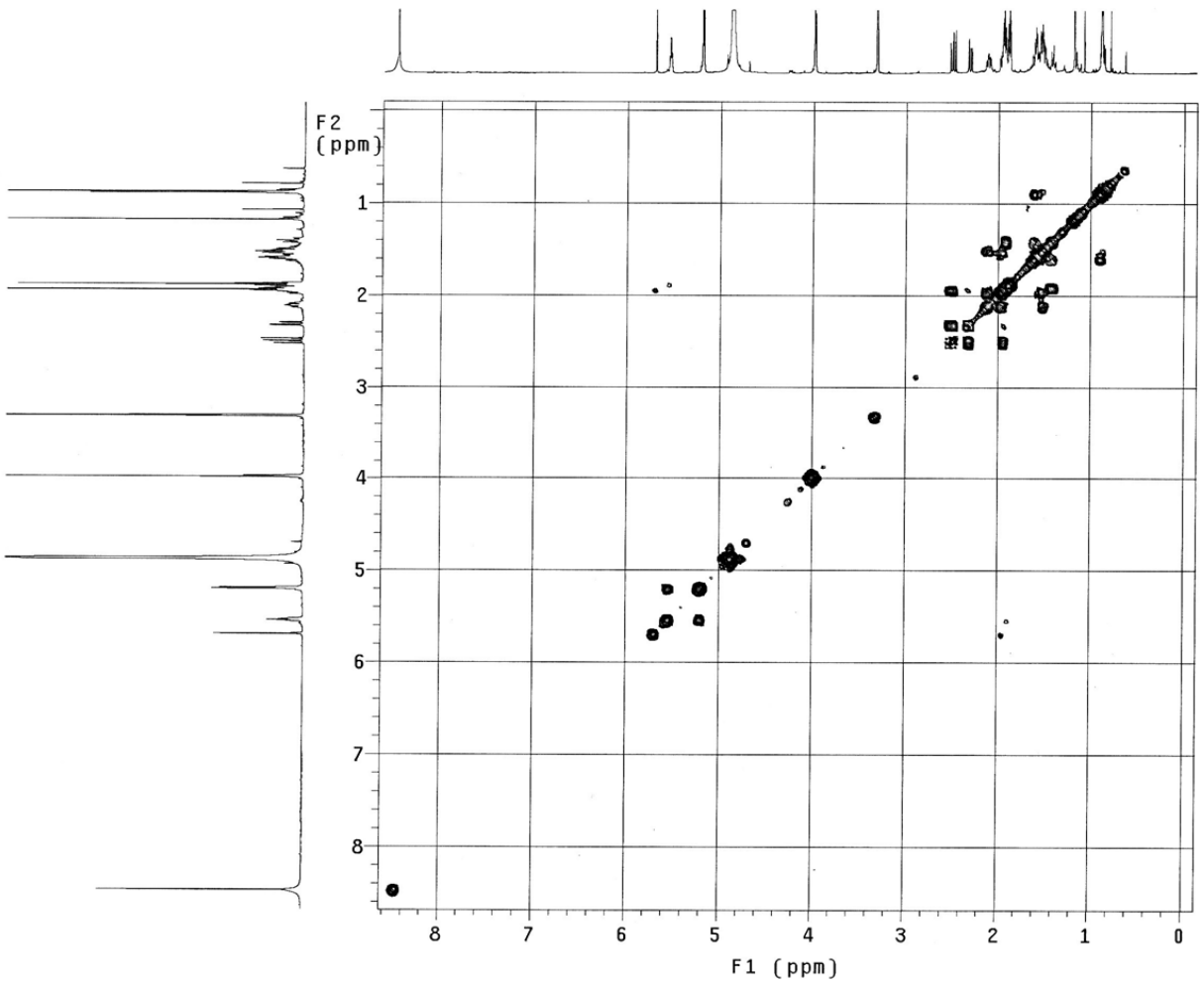


Fig. S19. HMQC spectrum of compound **4** (600 MHz, CD_3OD).

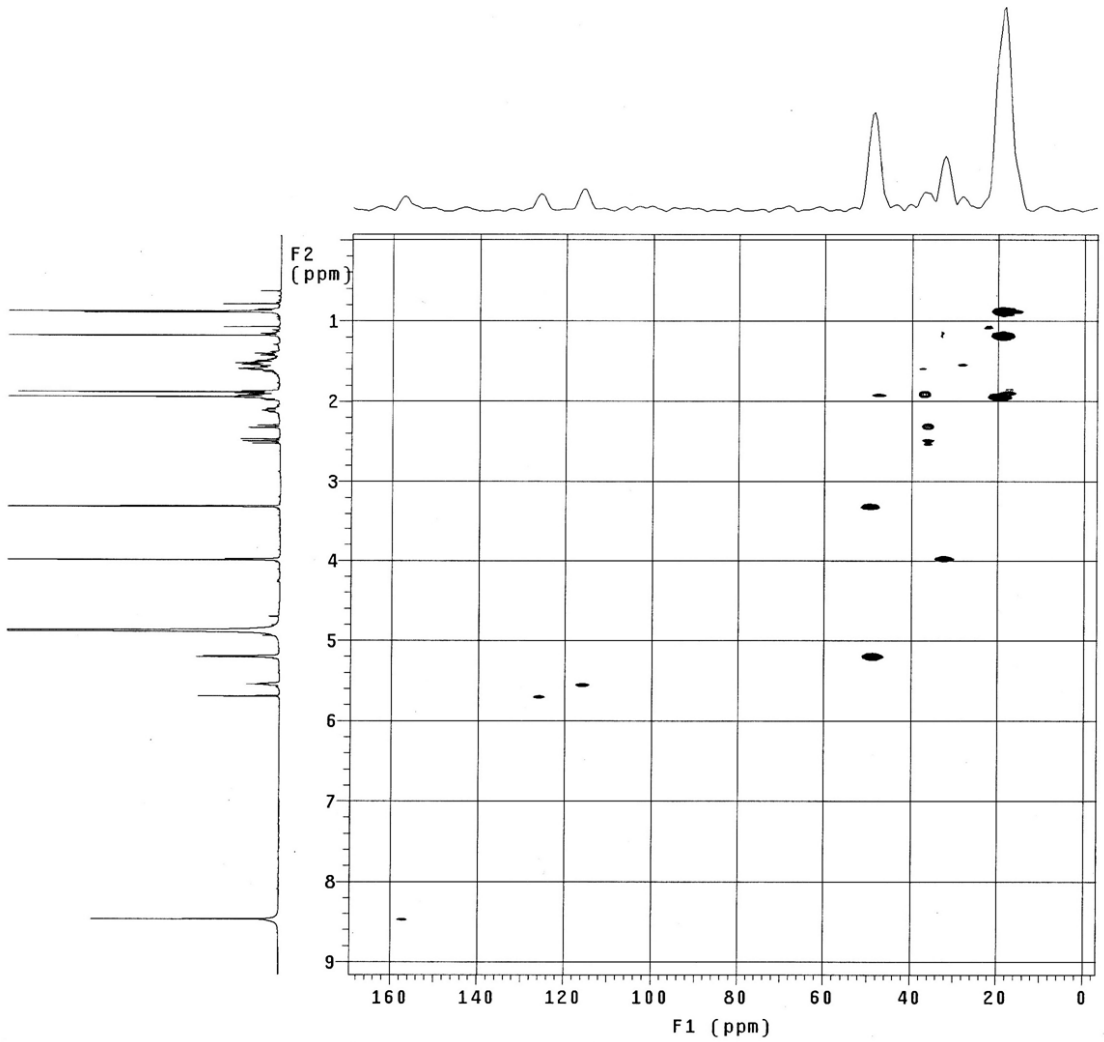


Fig. S20. *g*HMBC spectrum of compound **4** (600 MHz, CD_3OD).

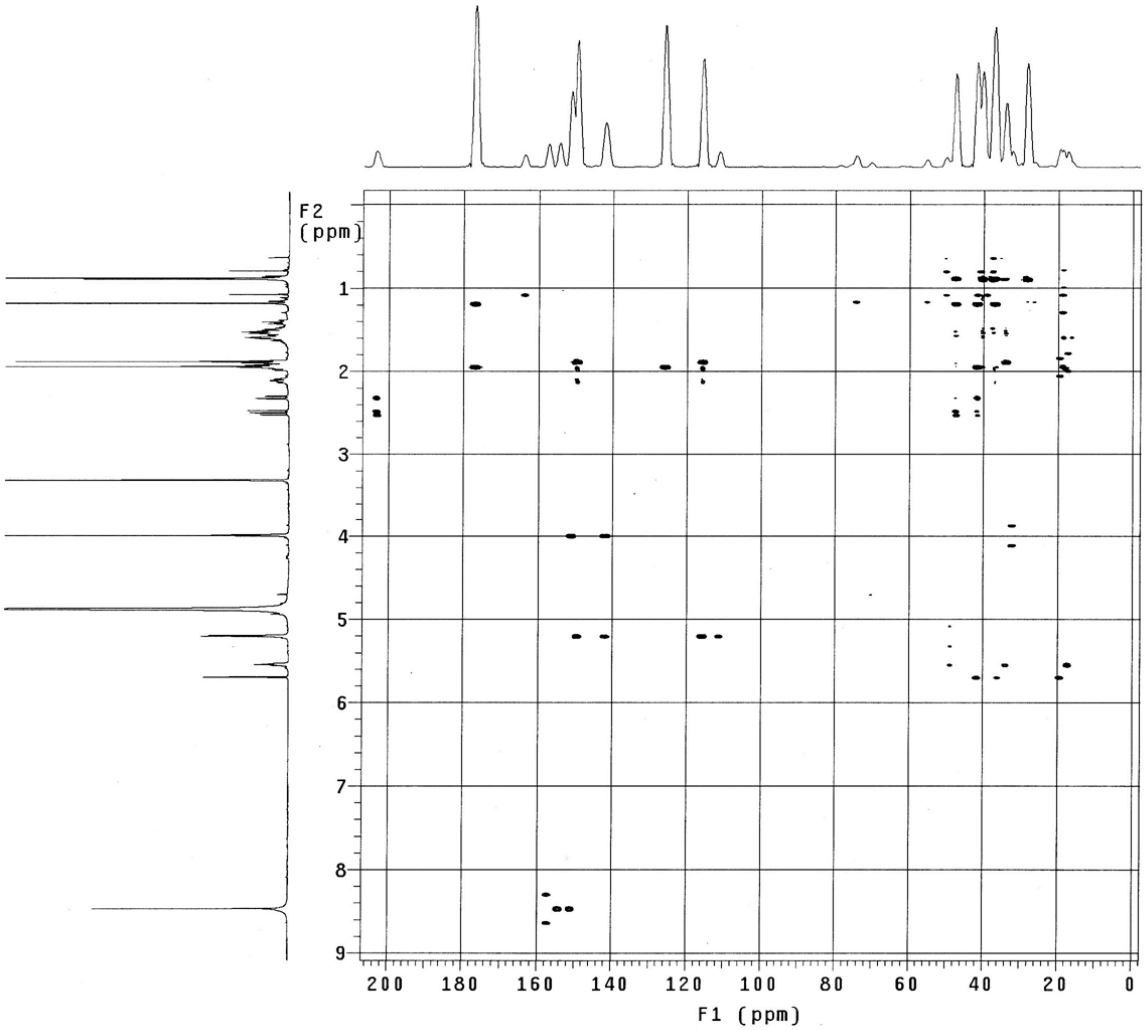


Fig. S21. NOESY spectrum of compound **4** (600 MHz, CD_3OD).

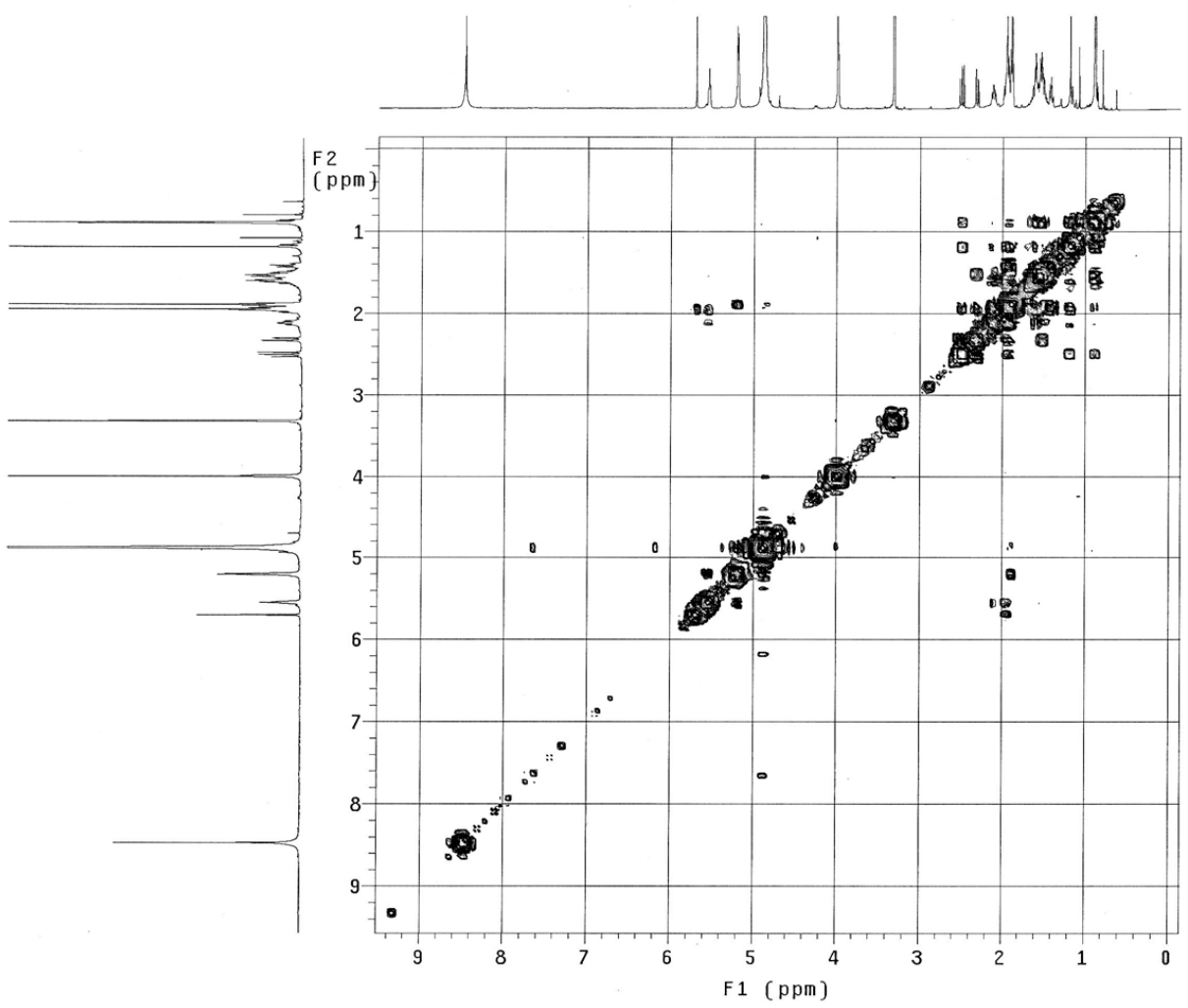


Fig. S22. IR spectrum of compound **4** (NaCl).

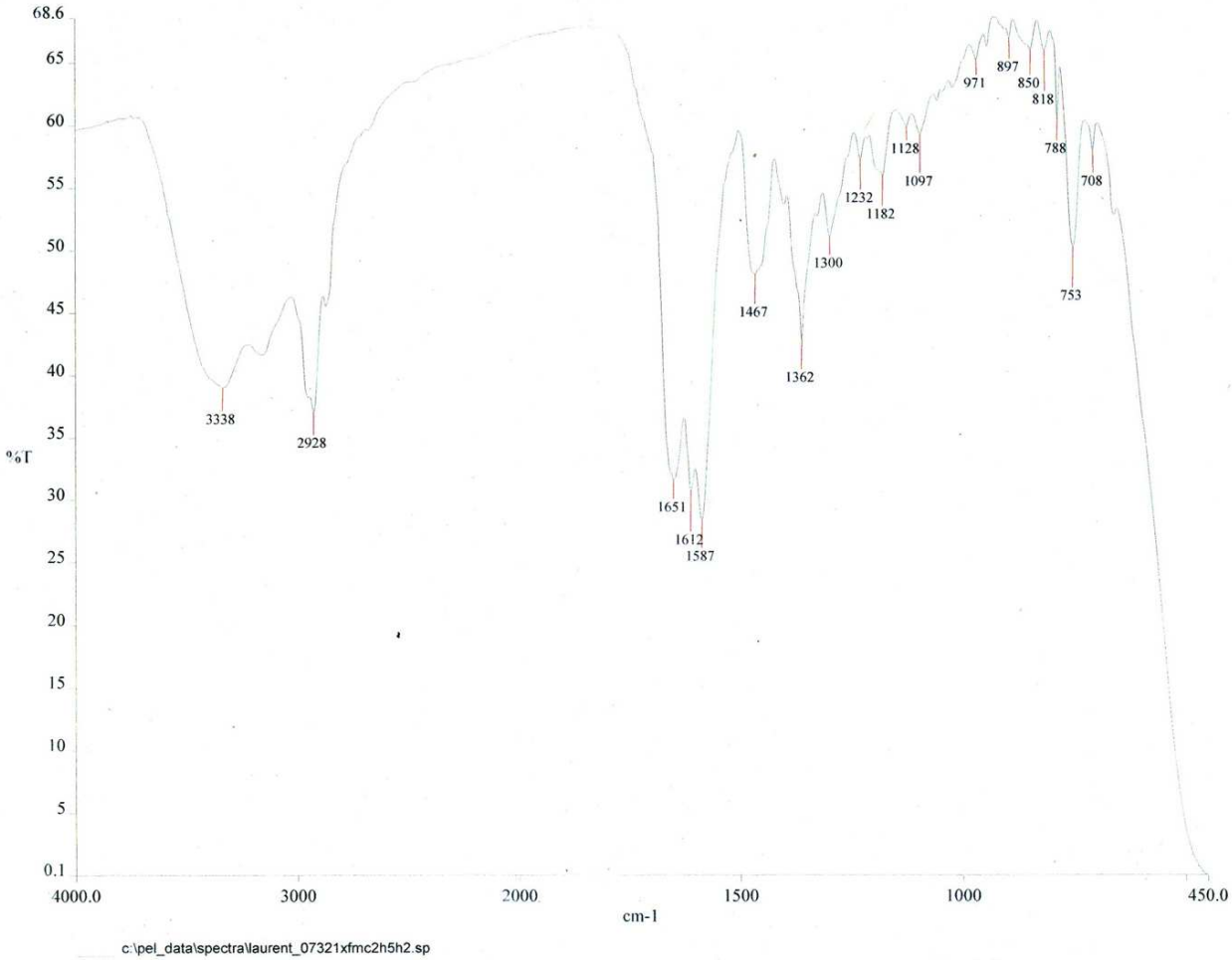
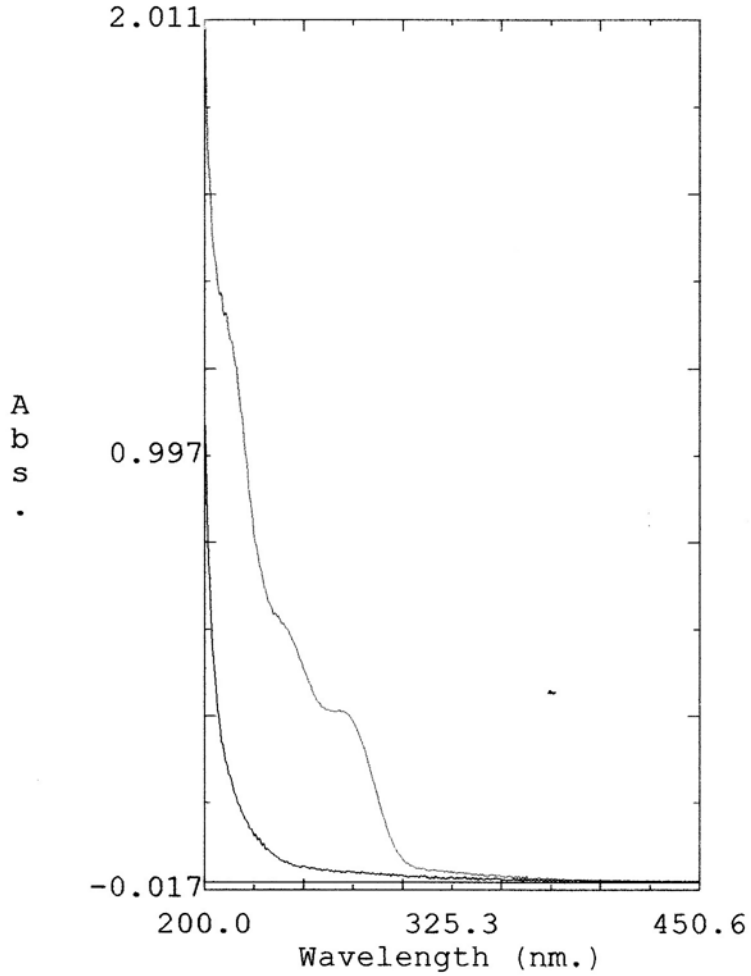


Fig. S23. UV spectrum of compound **4** (MeOH).



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Created: 09:31 09/22/09

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270.00	0.3991
698.50	-0.0002

Fig. S24. UV spectrum of compound **4** (MeOH).

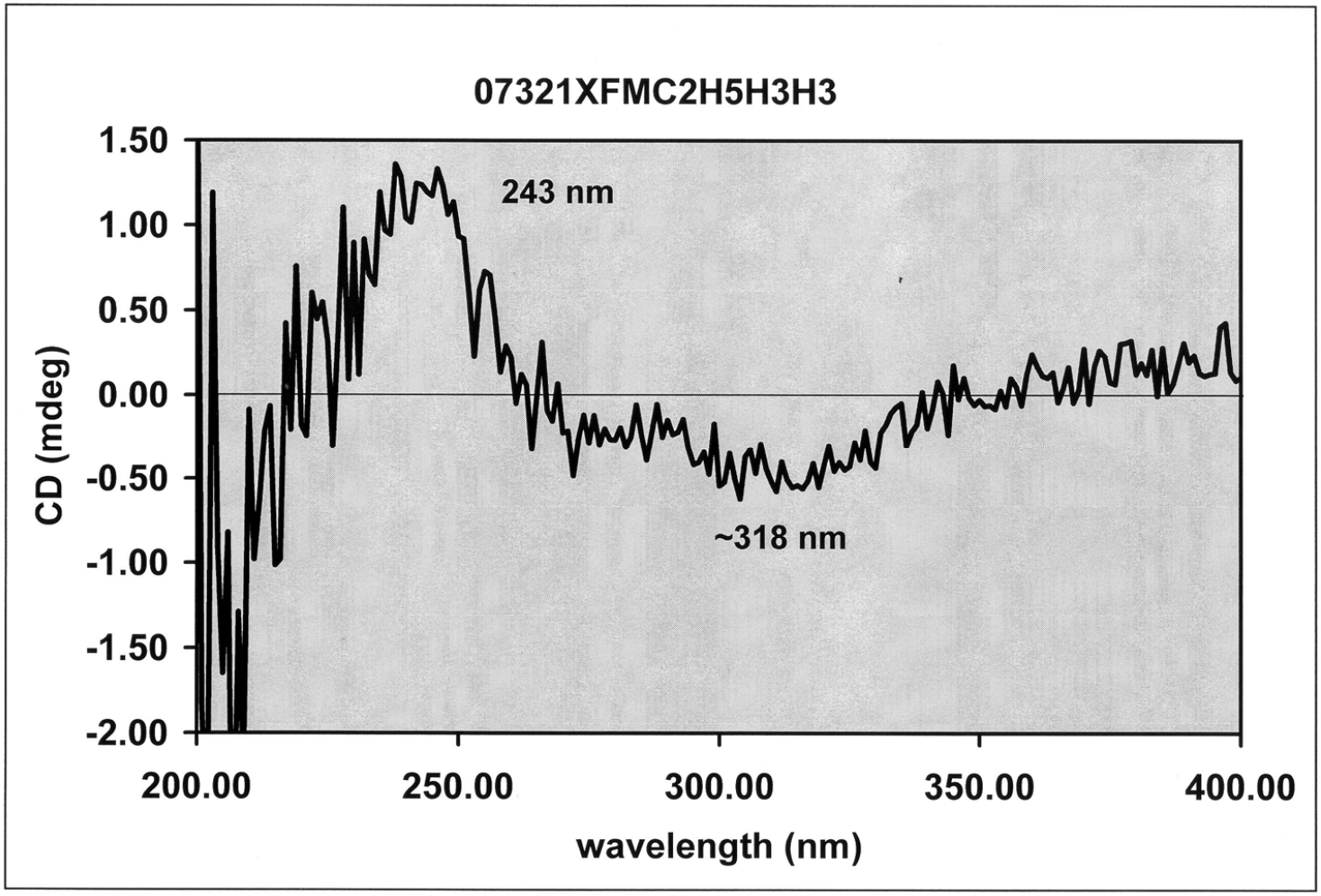

Fig. S25. Mass data HRESIMS in positive mode of compound **5**.

Mass Spectrometry & Proteomics Facility

Mass Spectrum SmartFormula Report

Analysis Info

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Sample Name	07321xfmc2h3h3	Instrument / Ser#	micrOTOF 10314
Comment	in CH3CN		

Acquisition Parameter

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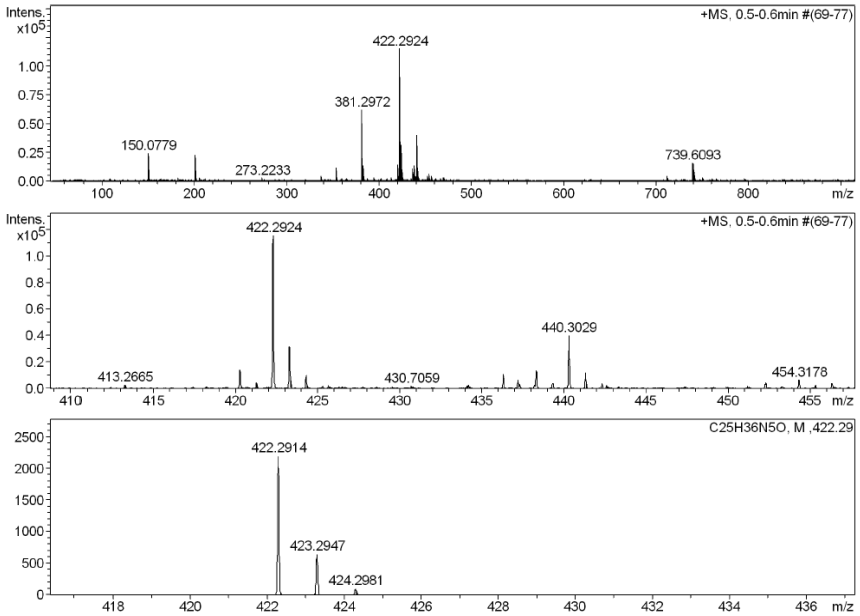
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	n/a	n/a	Set Divert Valve	Source



Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf
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Fig. S26. ^{1}H NMR spectrum of compound **5** (600 MHz, CD_3OD).

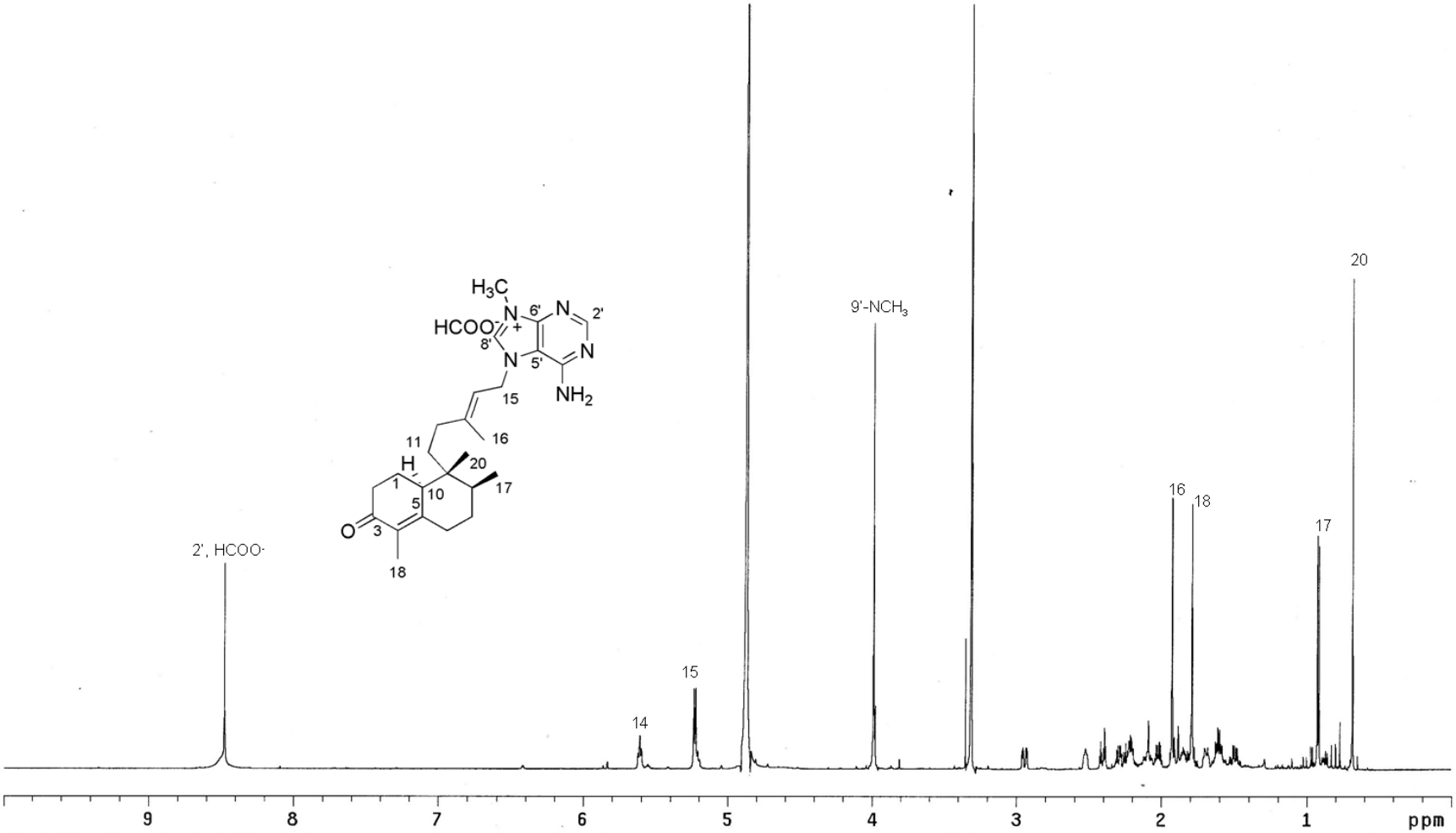


Fig. S27. ^{1}H NMR spectrum of compound **5** (600 MHz, CD_3OD).

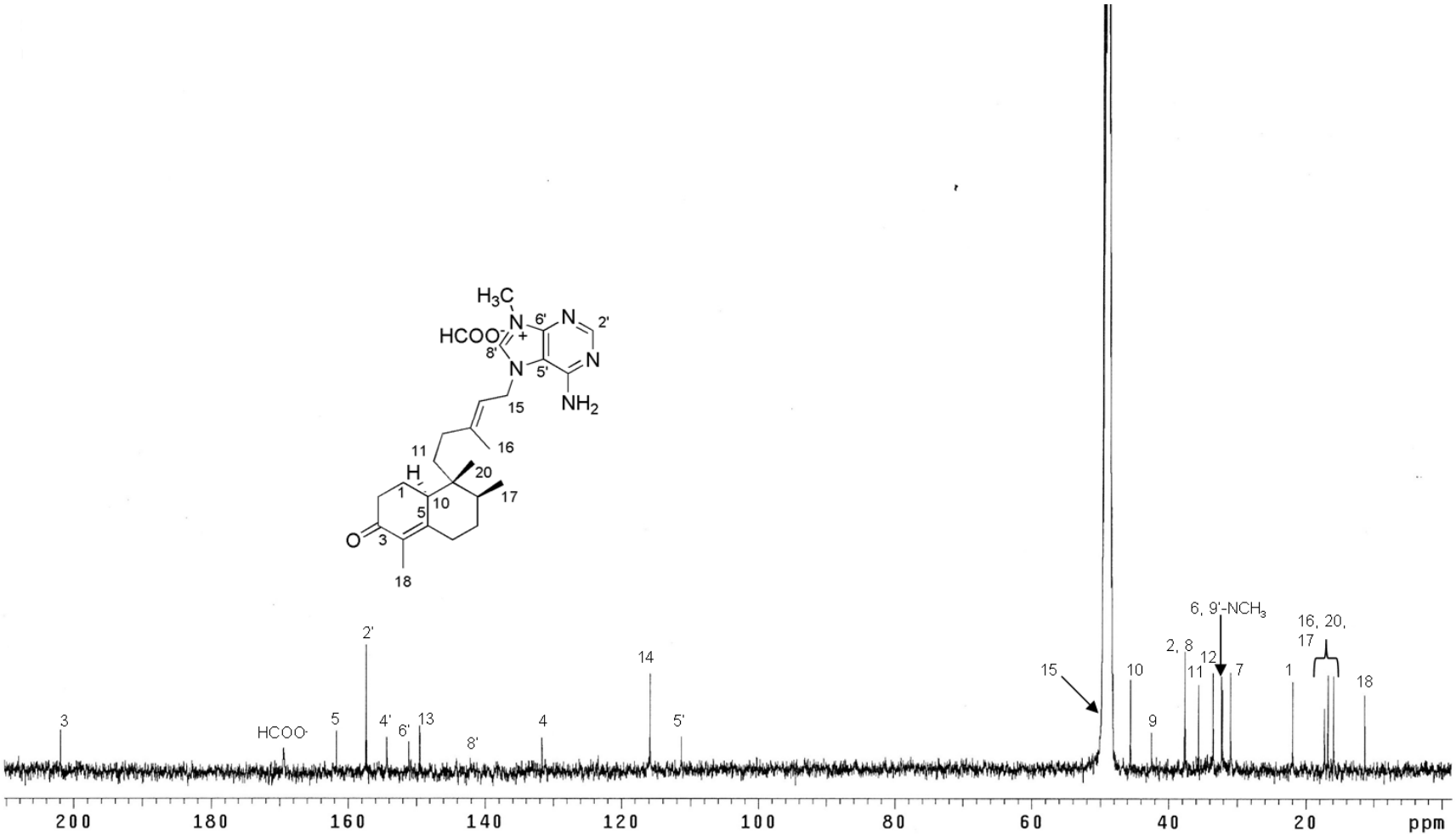


Fig. S28. DEPT spectrum of compound **5** (125 MHz, CD_3OD).

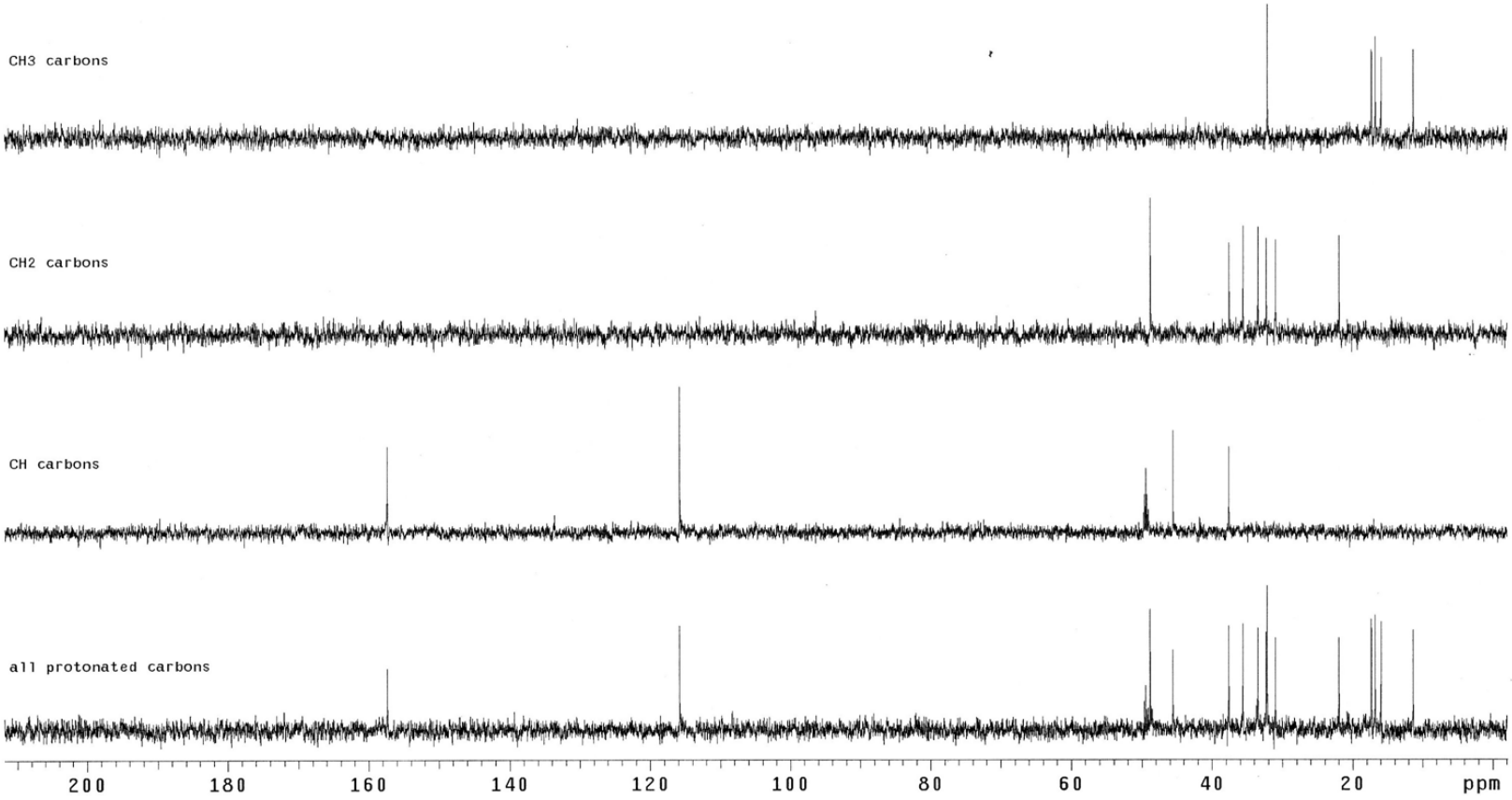


Fig. S29. *g*COSY spectrum of compound **5** (600 MHz, CD_3OD).

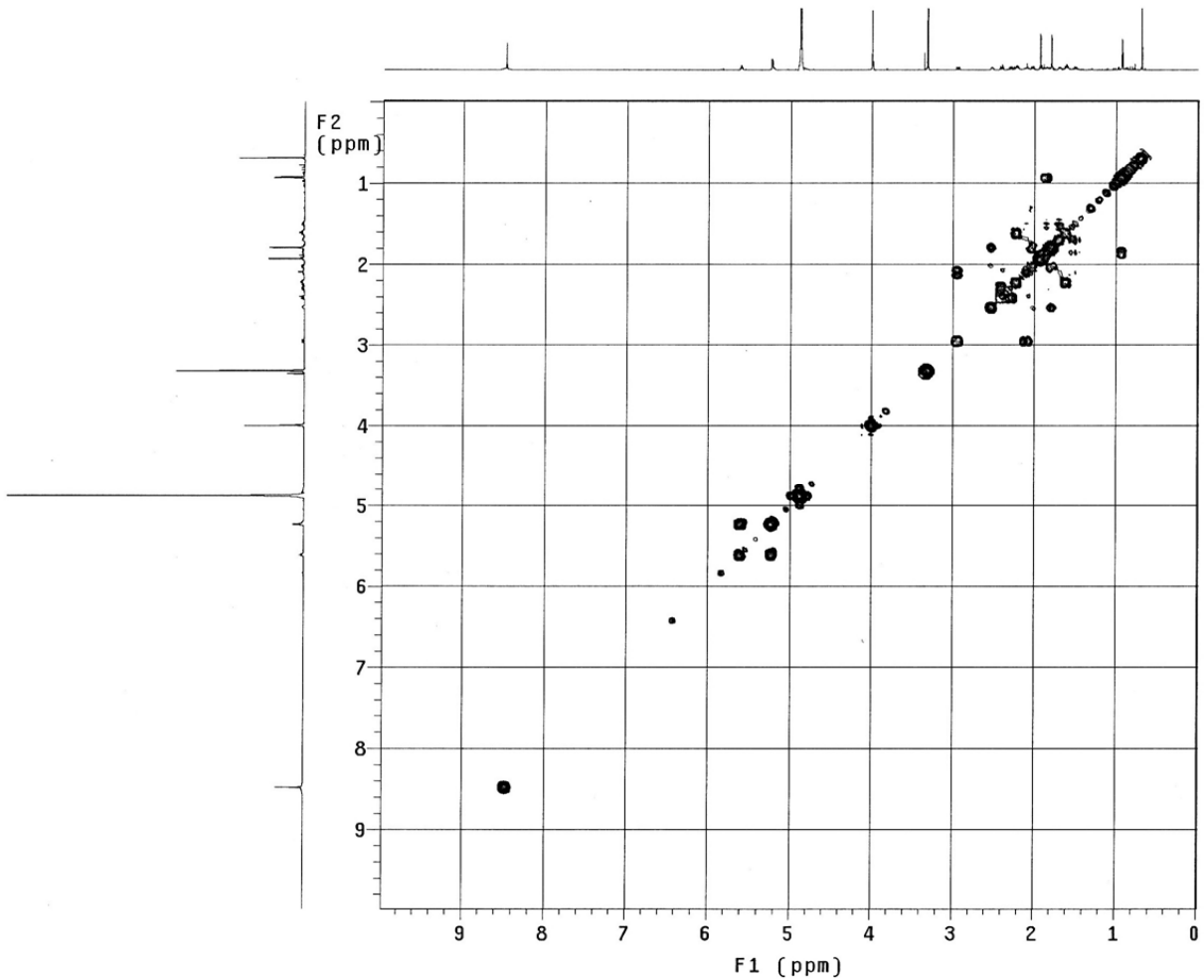


Fig. S30. HMQC spectrum of compound **5** (600 MHz, CD_3OD).

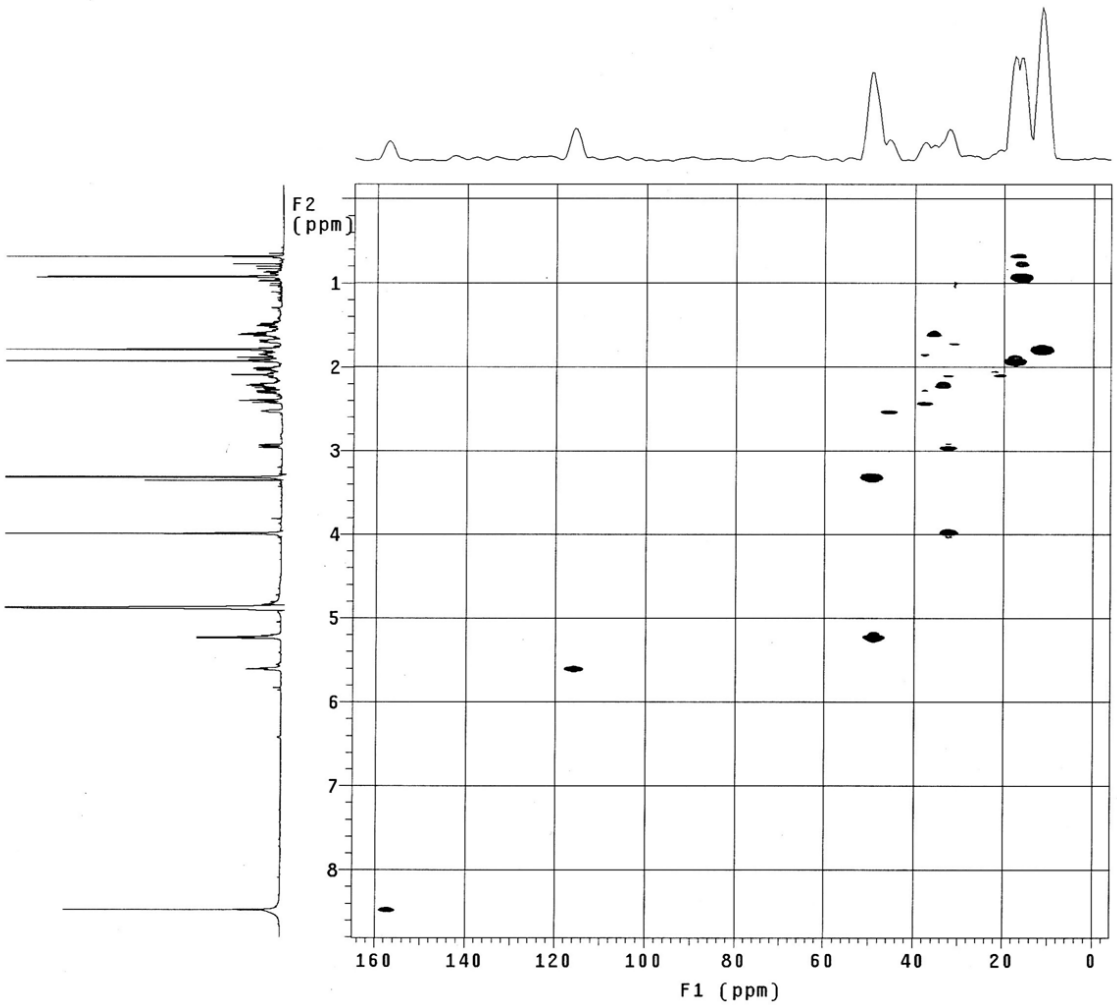


Fig. S31. *g*HMBC spectrum of compound **5** (600 MHz, CD_3OD).

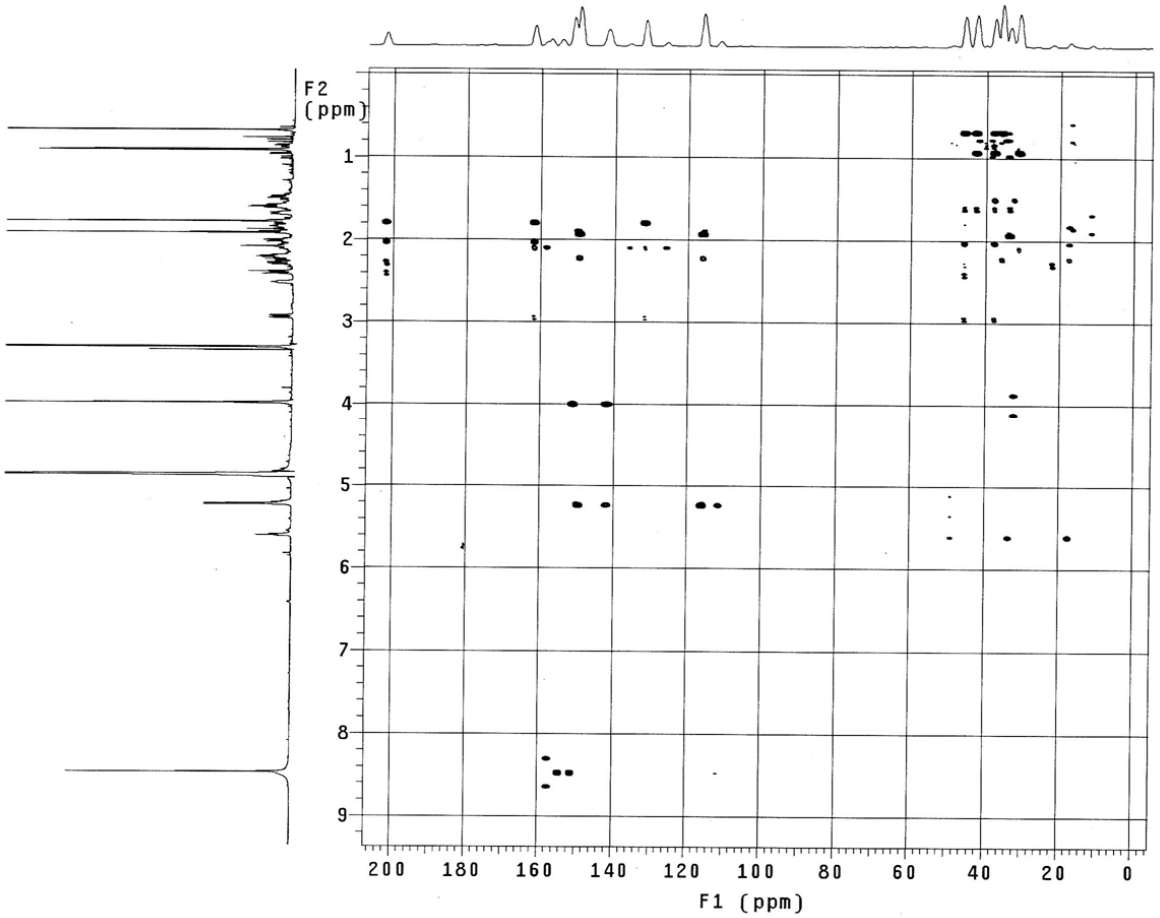


Fig. S32. NOESY spectrum of compound **5** (600 MHz, CD_3OD).

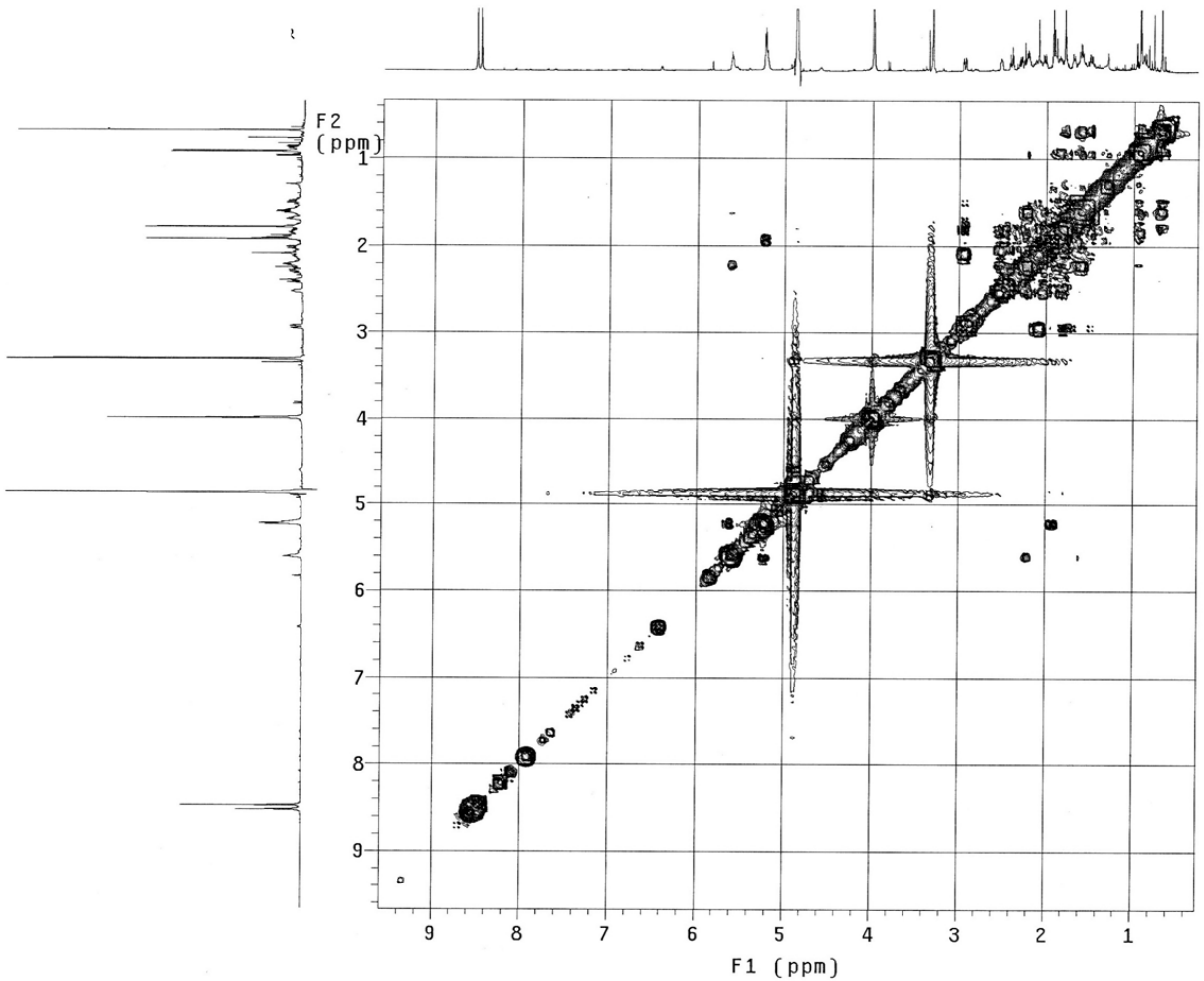


Fig. S33. IR spectrum of compound **5** (NaCl).

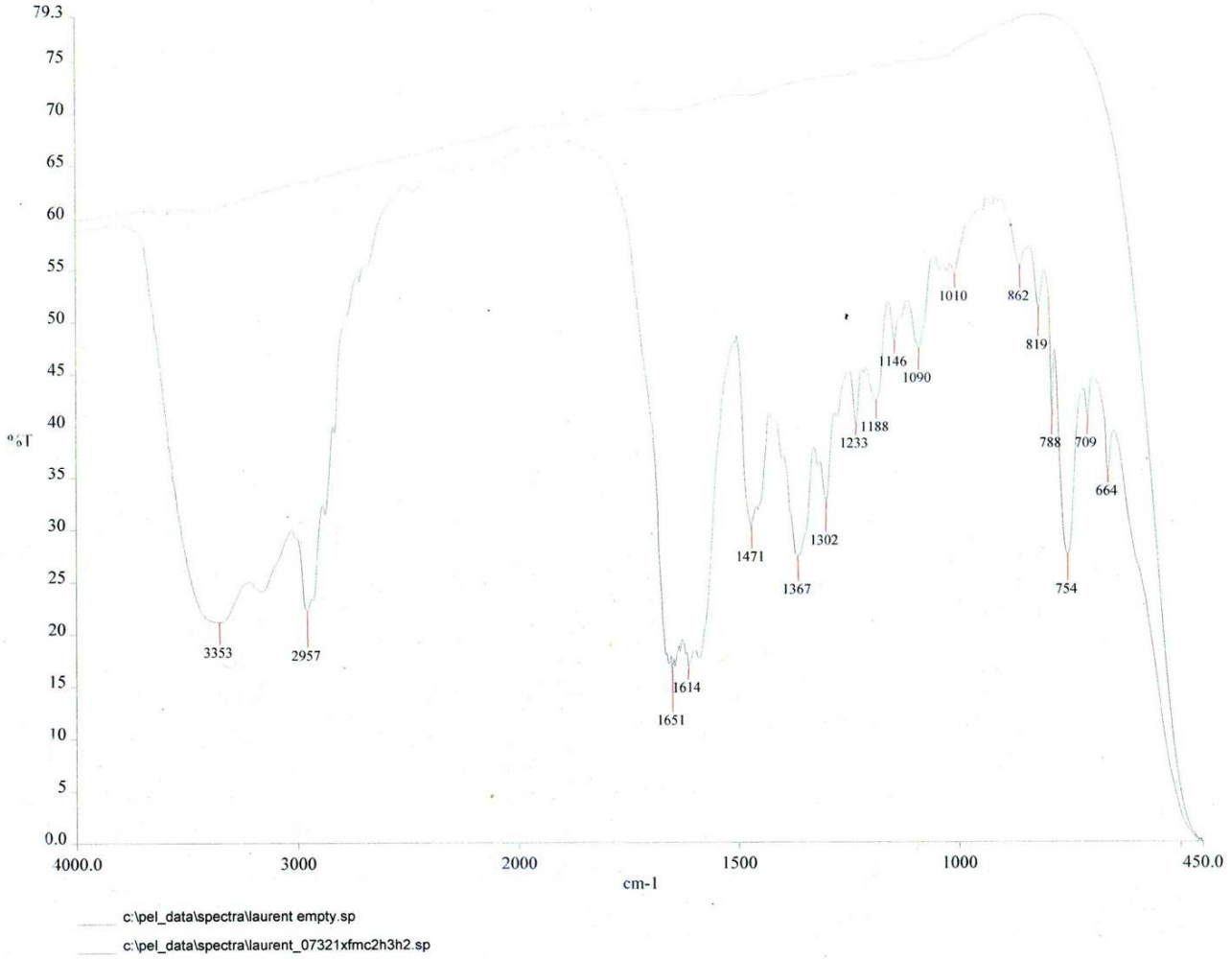
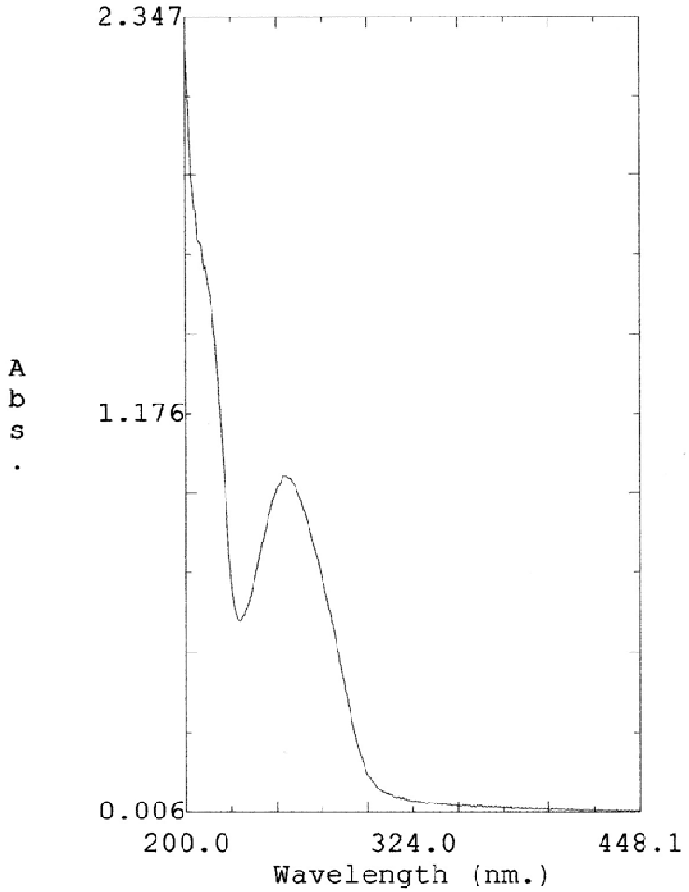


Fig. S34. UV spectrum of compound **5** (MeOH).



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698.50	0.0003

Fig. S35. CD spectrum of compound **5** (MeOH).

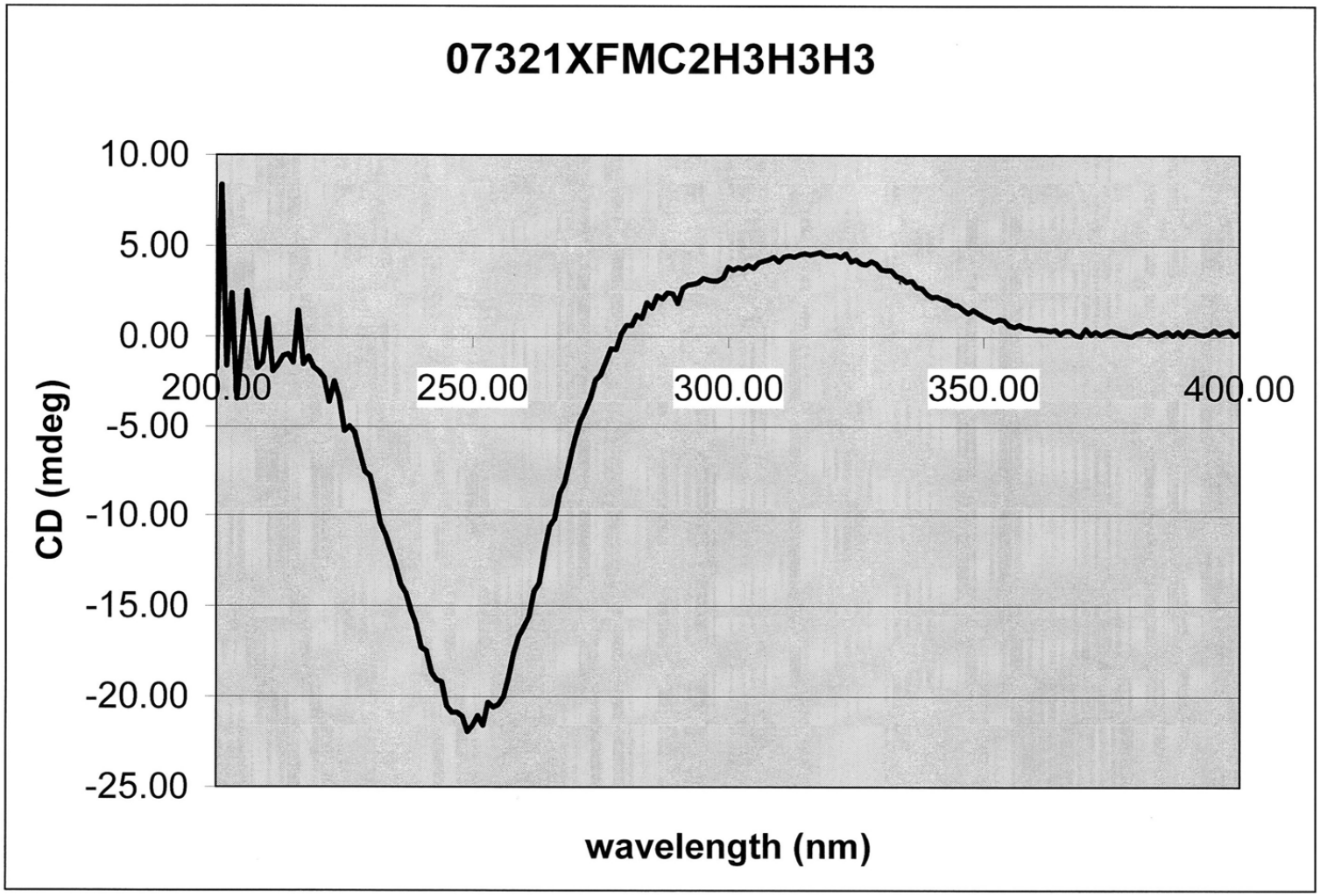
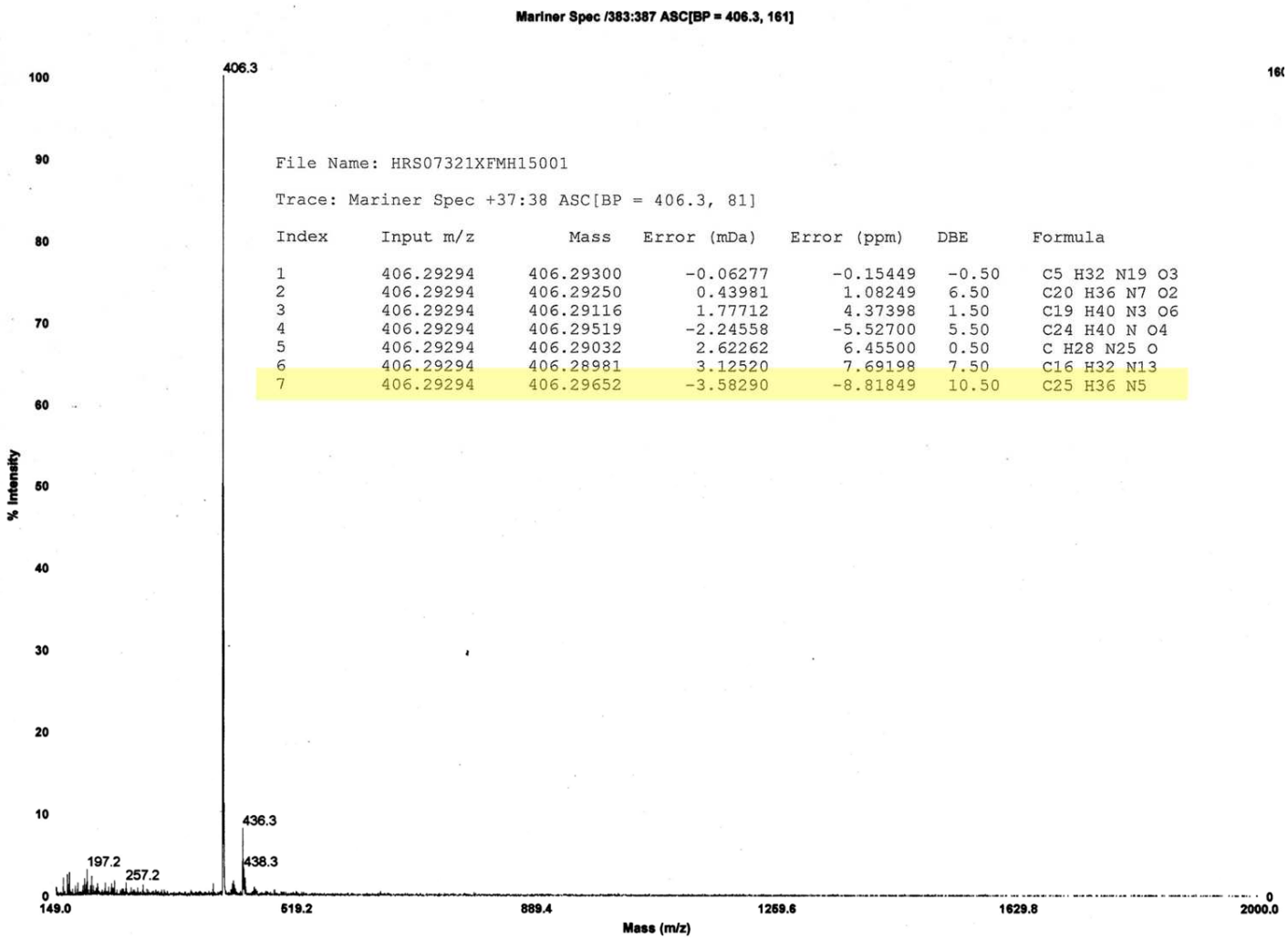


Fig. S36. Mass data HRESIMS in positive mode of compound **6**.



File Name: HRS07321XFMH15001

Trace: Mariner Spec +37:38 ASC[BP = 406.3, 81]

Index	Input m/z	Mass	Error (mDa)	Error (ppm)	DBE	Formula
1	406.29294	406.29300	-0.06277	-0.15449	-0.50	C5 H32 N19 O3
2	406.29294	406.29250	0.43981	1.08249	6.50	C20 H36 N7 O2
3	406.29294	406.29116	1.77712	4.37398	1.50	C19 H40 N3 O6
4	406.29294	406.29519	-2.24558	-5.52700	5.50	C24 H40 N O4
5	406.29294	406.29032	2.62262	6.45500	0.50	C H28 N25 O
6	406.29294	406.28981	3.12520	7.69198	7.50	C16 H32 N13
7	406.29294	406.29652	-3.58290	-8.81849	10.50	C25 H36 N5

Fig. S37. ^{1}H NMR spectrum of compound **6** (600 MHz, CD_3OD).

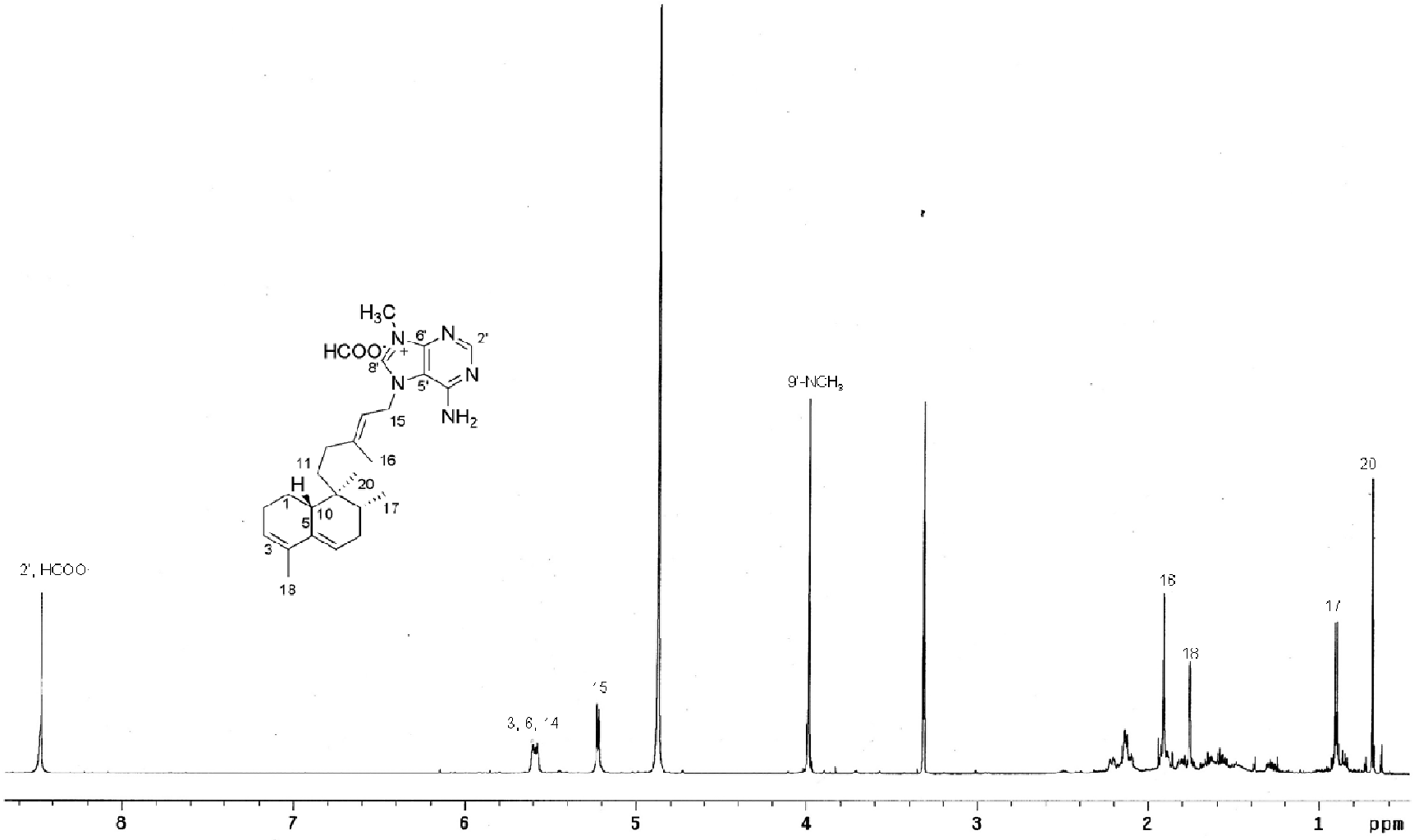


Fig. S38. ^{13}C NMR spectrum of compound **6** (125 MHz, CD_3OD).

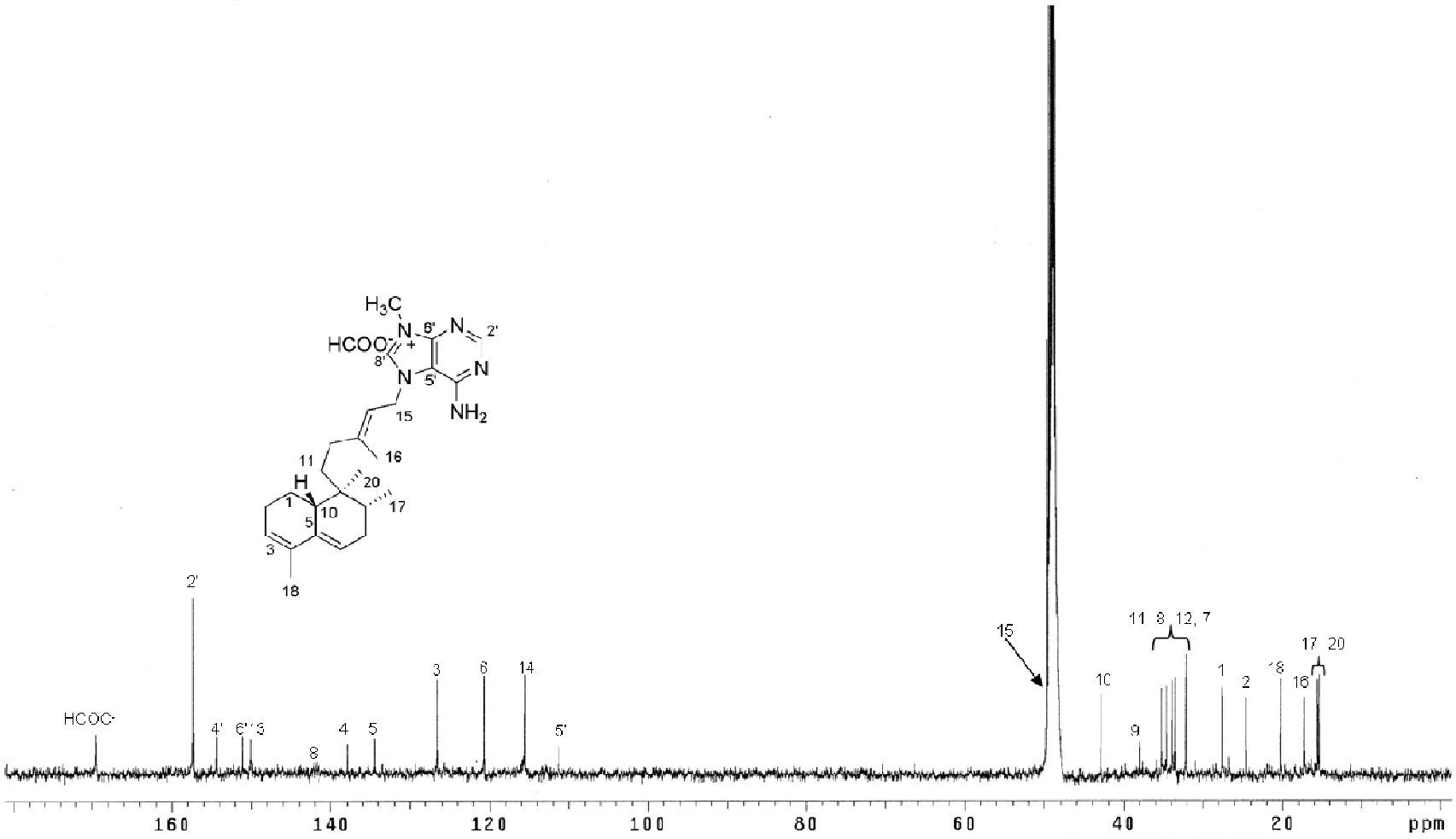


Fig. S39. DEPT spectrum of compound **6** (125 MHz, CD_3OD).

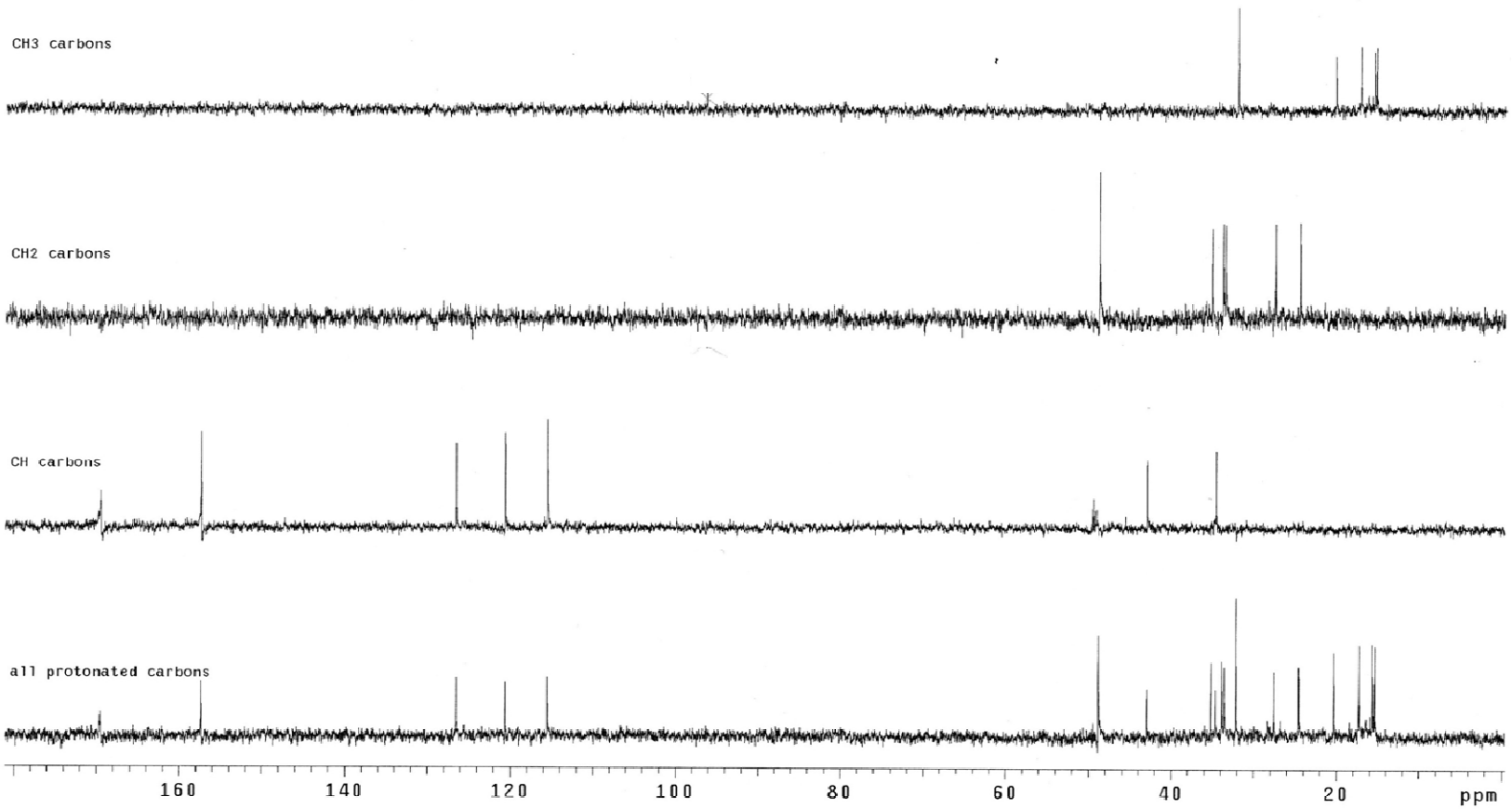


Fig. S40. *g*COSY spectrum of compound **6** (600 MHz, CD_3OD).

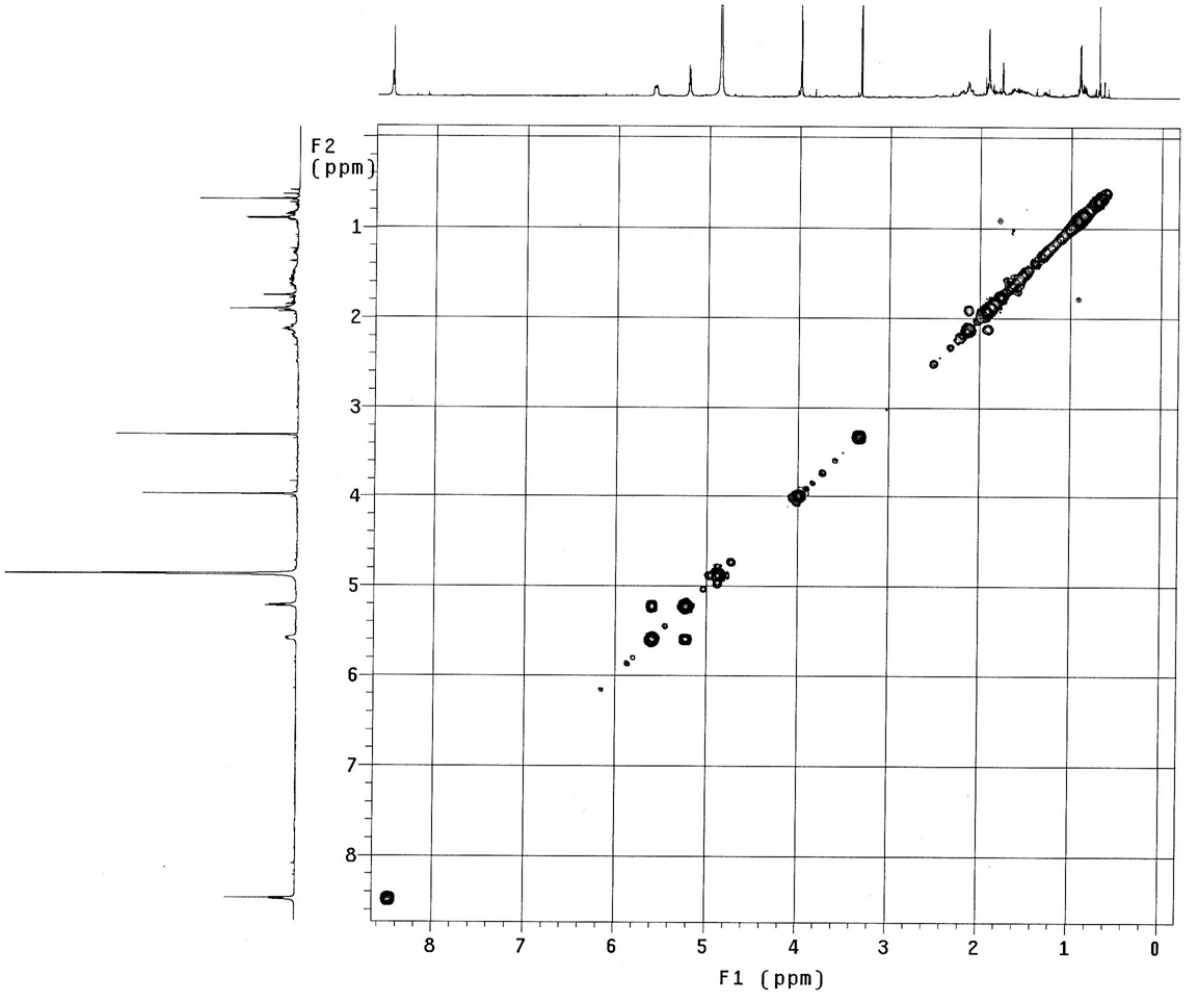


Fig. S41. HMQC spectrum of compound **6** (600 MHz, CD_3OD).

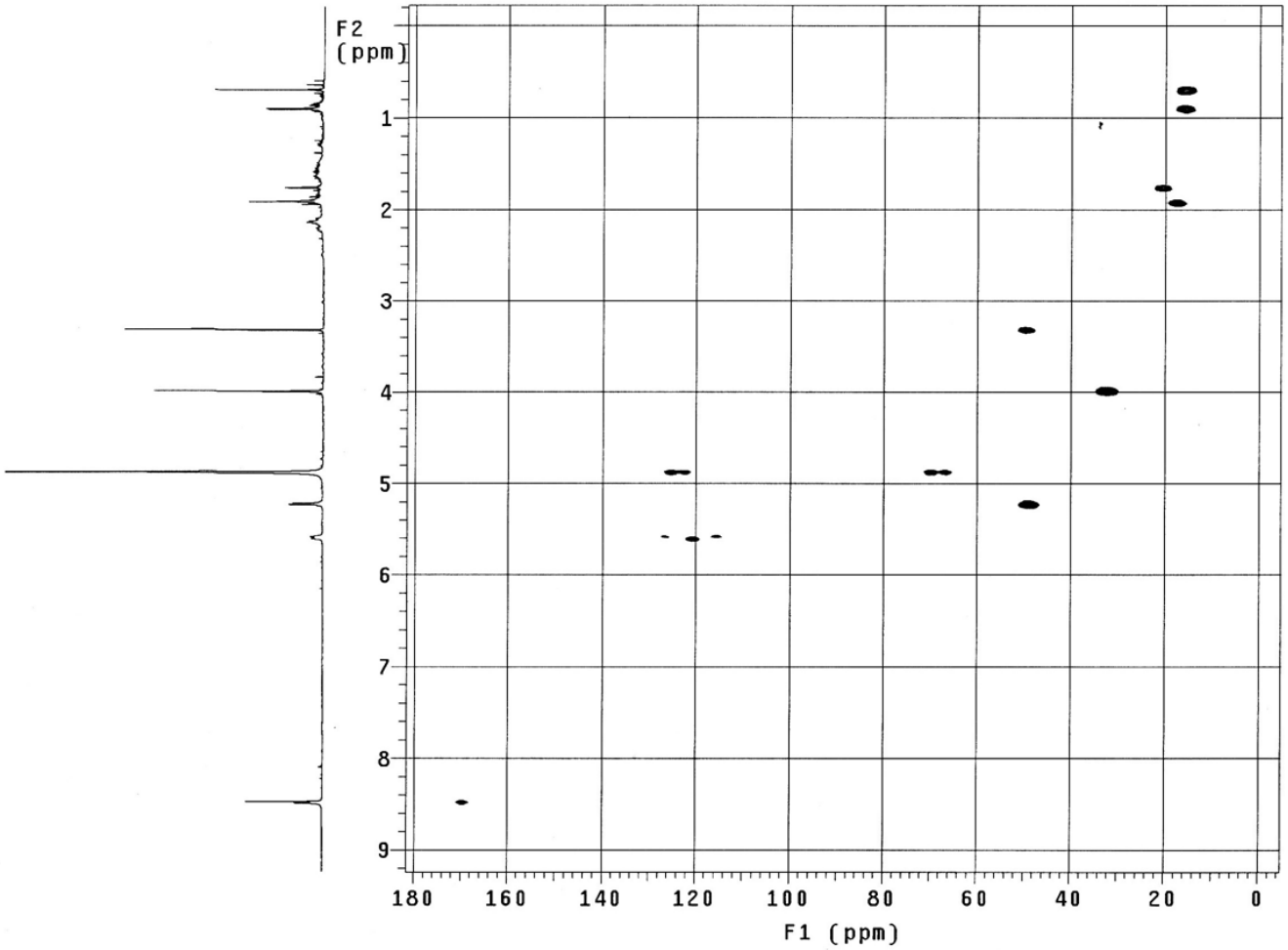


Fig. S42. *g*HMBC spectrum of compound **6** (600 MHz, CD_3OD).

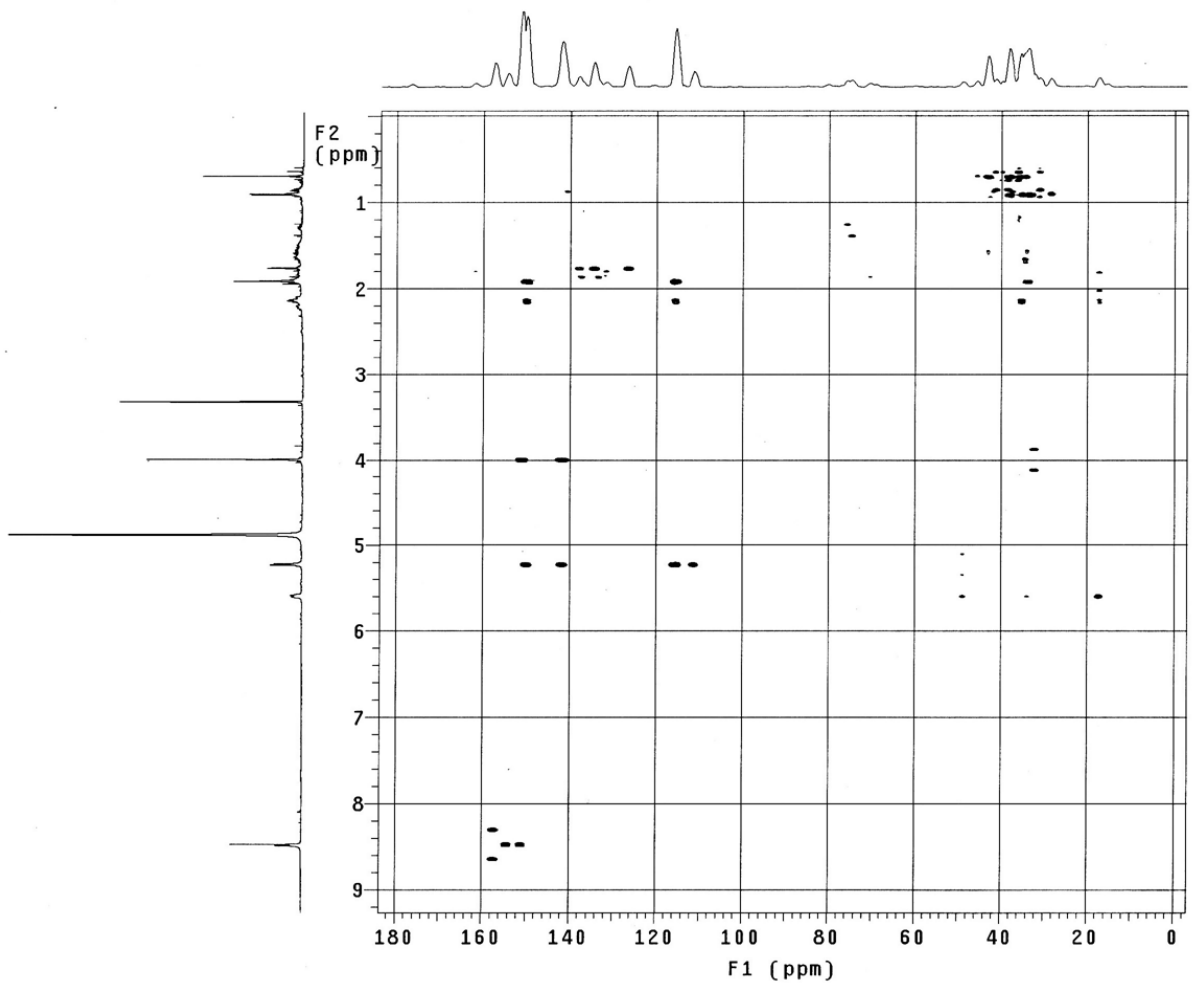


Fig. S43. NOESY spectrum of compound **6** (600 MHz, CD_3OD).

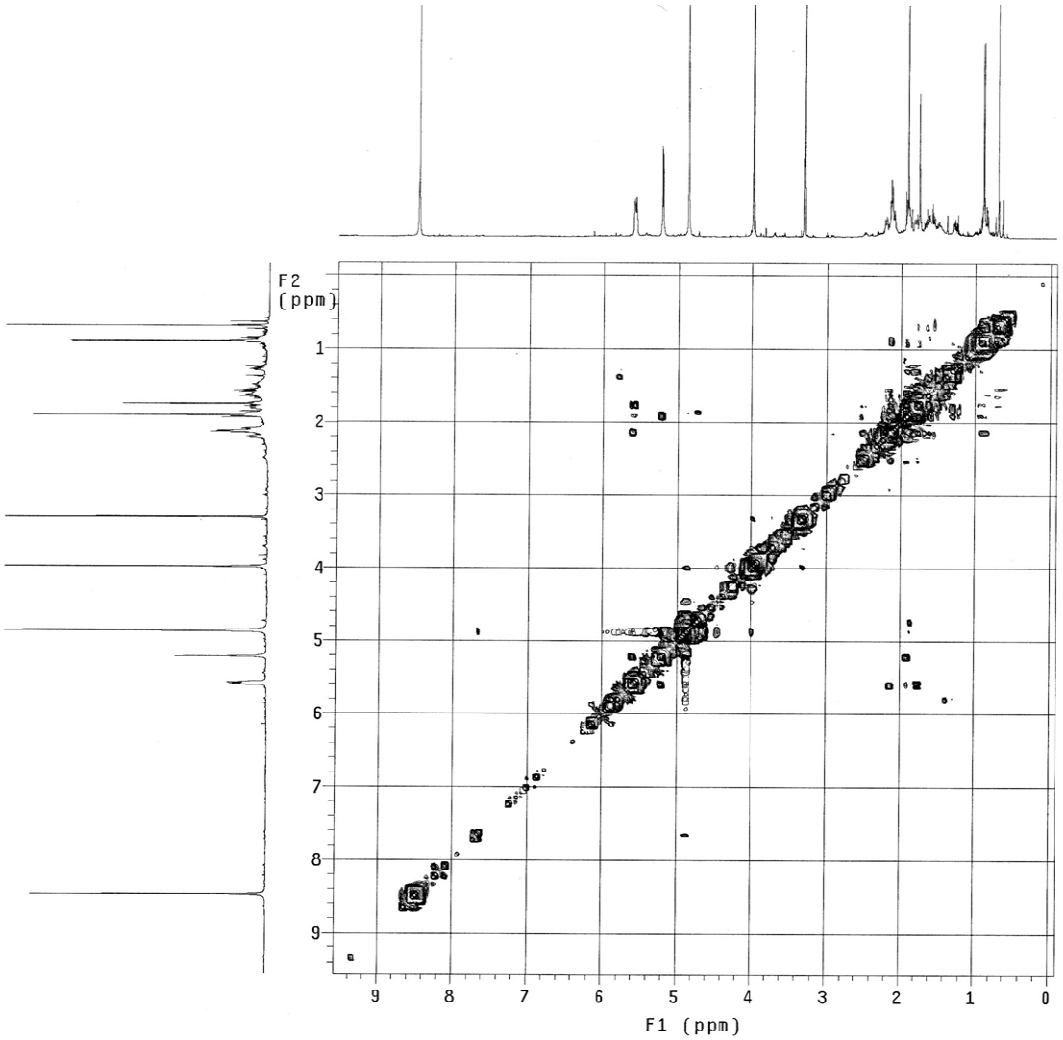


Fig. S44. IR spectrum of compound **6** (NaCl).

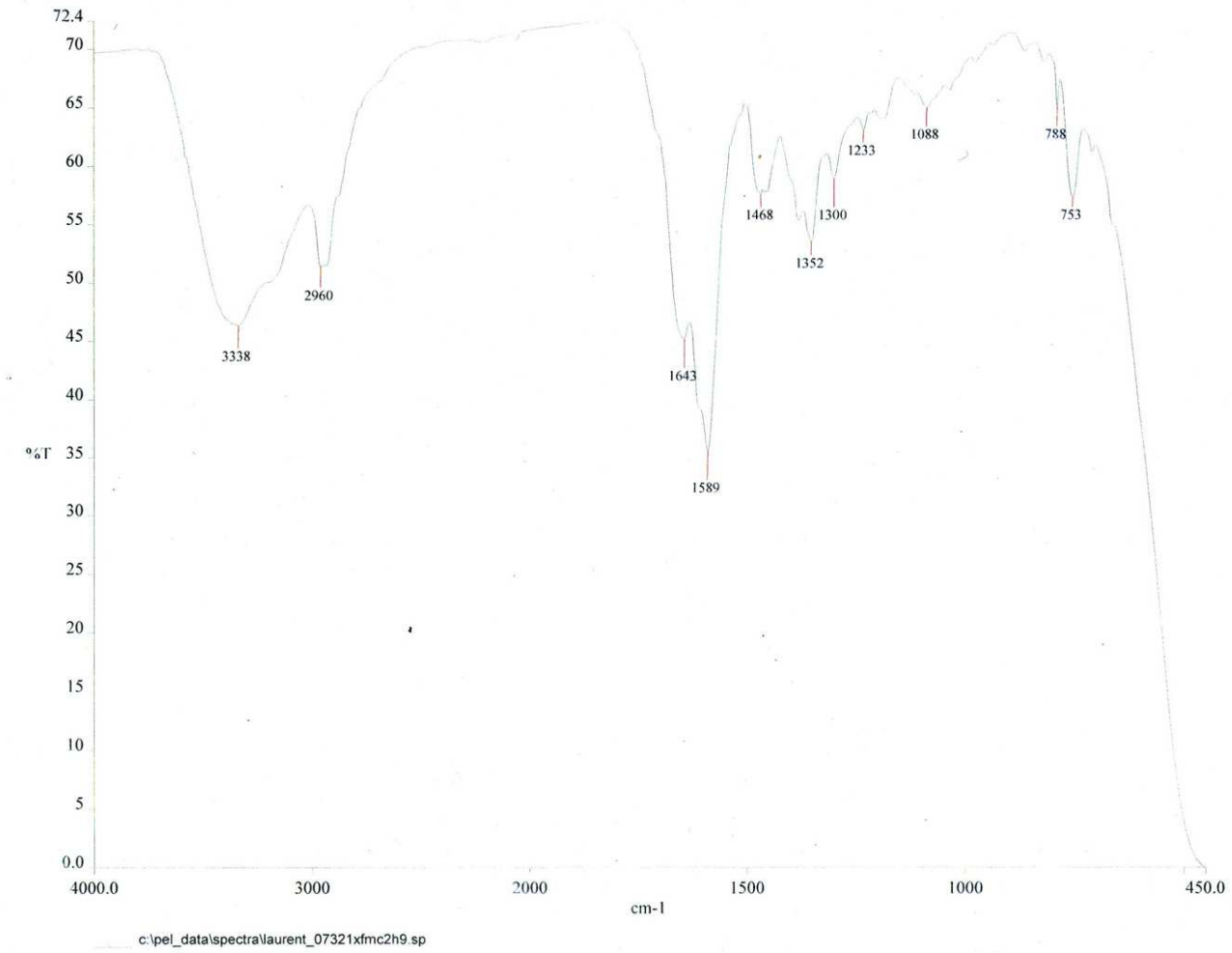
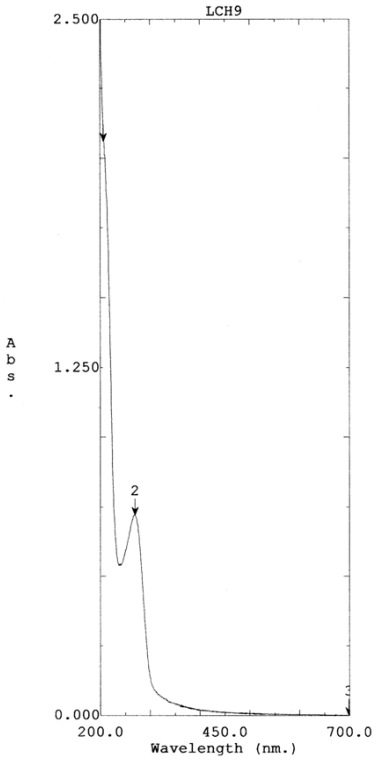


Fig. S45. UV spectrum of compound **6** (MeOH).



Point Pick

No.	Wavelength (nm.)	Abs.
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2	269.50	0.7215
3	698.50	0.0003

File Name: LCH9
07321XFMC2H9_093009

Created: 10:50 09/30/09
Data: Original

Measuring Mode: Abs.
Scan Speed: Fast
Slit Width: 1.0
Sampling Interval: 0.5