

Steroidal Glycosides from *Dregea sinensis* var. *corrugata* Based on
Chemical Screening with Liquid Chromatography-electrospray Ionization
Tandem Mass Spectrometry (LC-ESI-MSⁿ)

Yun-Bao Liu,¹ E-Nuo Su,² Jian-Bei Li,[†] Jin-Lan Zhang,[†] Shi-Shan Yu,^{*,†} Jing Qu,[†] Jing Liu,[†]
Yong Li[†]

*Key Laboratory of Bioactive Substances and Resources Utilization of Chinese Herbal Medicine,
Ministry of Education & Institute of Materia Medica, Peking Union Medical College and Chinese
Academy of Medical Sciences, No. 1 Xian Nong Tan Street, Beijing 100050, PR China, Shenyang
Pharmaceutical University, Shenyang 110016, PR China*

* To whom correspondence should be addressed. E-mail: yushishan@imm.ac.cn; Tel.: +86-10-63165324; +86-10-60212125; Fax: +86-10-63017757

¹ Institute of Materia Medica, Peking Union Medical College and Chinese Academy of Medical Sciences

² Shenyang Pharmaceutical University

Support Information

Figure S1. ESI-MSⁿ spectra of new compound 1

Figure S2. ¹H NMR (pyridine-d₅ 500 M Hz) spectra of new compound 1

Figure S3. ¹³C NMR (pyridine-d₅ 125 M Hz) spectra of new compound 1

Figure S4. Dept spectra of new compound 1

Figure S5. COSY spectra of new compound 1

Figure S6. HMQC spectra of new compound 1

Figure S7. HMBC spectra of new compound 1

Figure S8. ESI-MSⁿ spectra of new compound 4

Figure S9. ¹H NMR (pyridine-d₅ 500 M Hz) spectra of new compound 4

Figure S10. ¹³C NMR (pyridine-d₅ 125 M Hz) spectra of new compound 4

Figure S11. Dept spectra of new compound 4

Figure S12. COSY spectra of new compound 4

Figure S13. HMQC spectra of new compound 4

Figure S14. HMBC spectra of new compound 4

Figure S15. ESI-MSⁿ spectra of new compound 7

Figure S16. ¹H NMR (pyridine-d₅ 500 M Hz) spectra of new compound 7

Figure S17. ¹³C NMR (pyridine-d₅ 125 M Hz) spectra of new compound 7

Figure S18. Dept spectra of new compound 7

Figure S19. COSY spectra of new compound 7

Figure S20. HMQC spectra of new compound 7

Figure S21. HMBC spectra of new compound 7

Figure S1. ESI-MSⁿ spectra of new compound 1

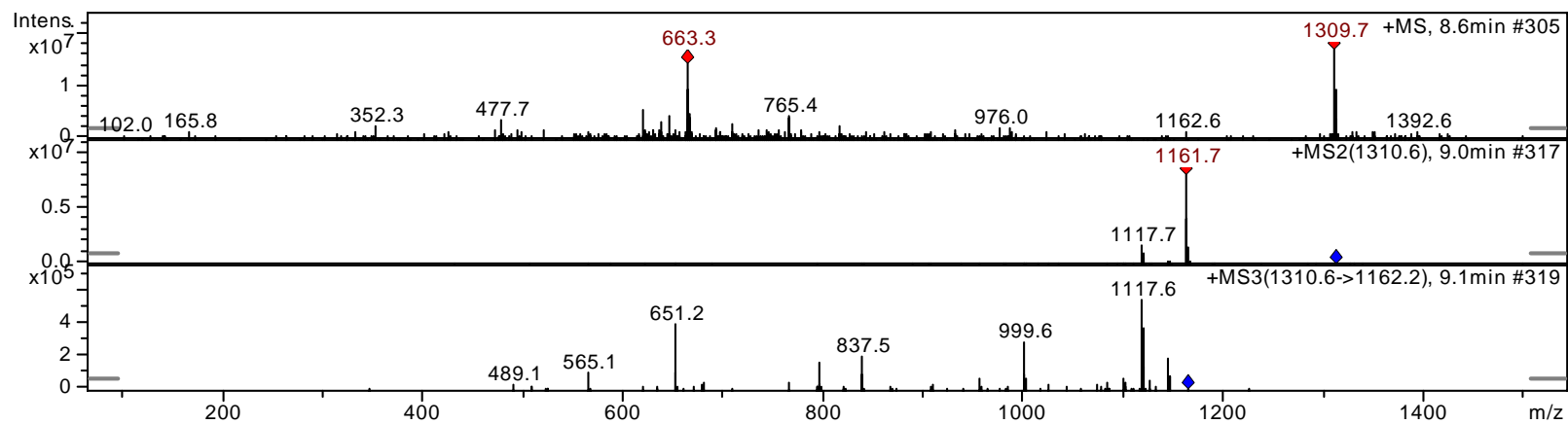


Figure S2. ^1H NMR (pyridine- d_5 500 M Hz) spectra of new compound 1

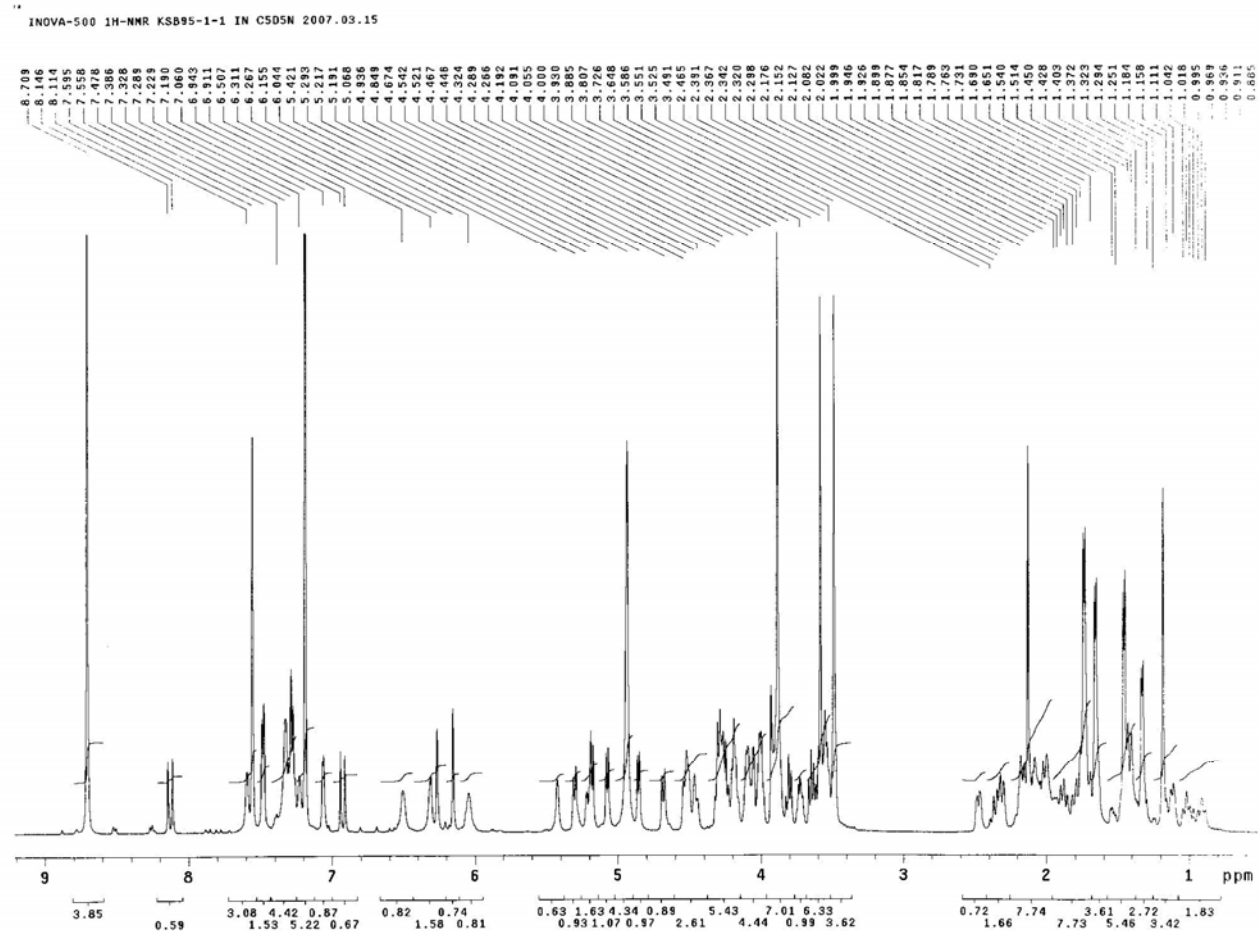


Figure S3. ^{13}C NMR (pyridine- d_5 500 M Hz) spectra of new compound 1

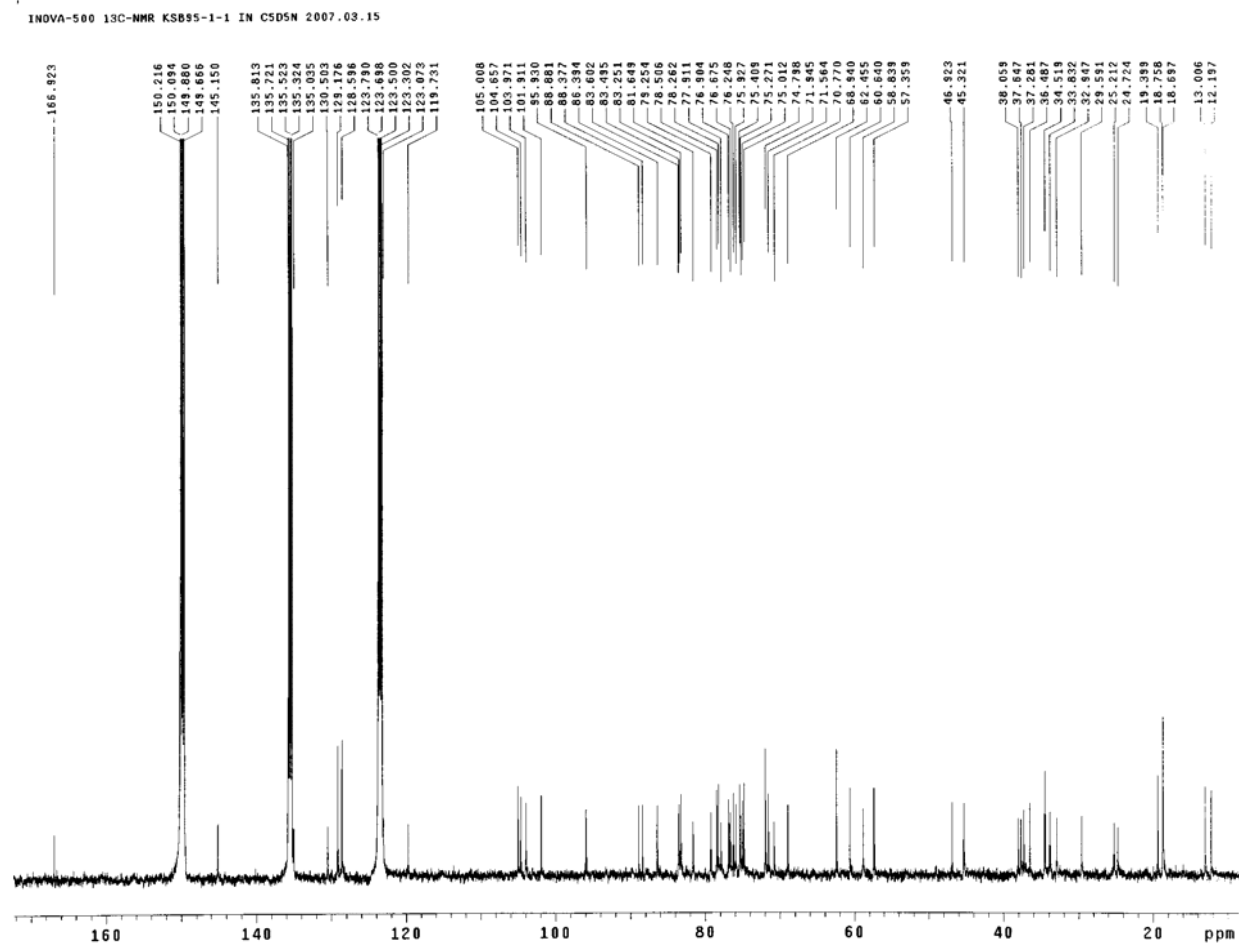


Figure S4. Dept spectra of new compound 1

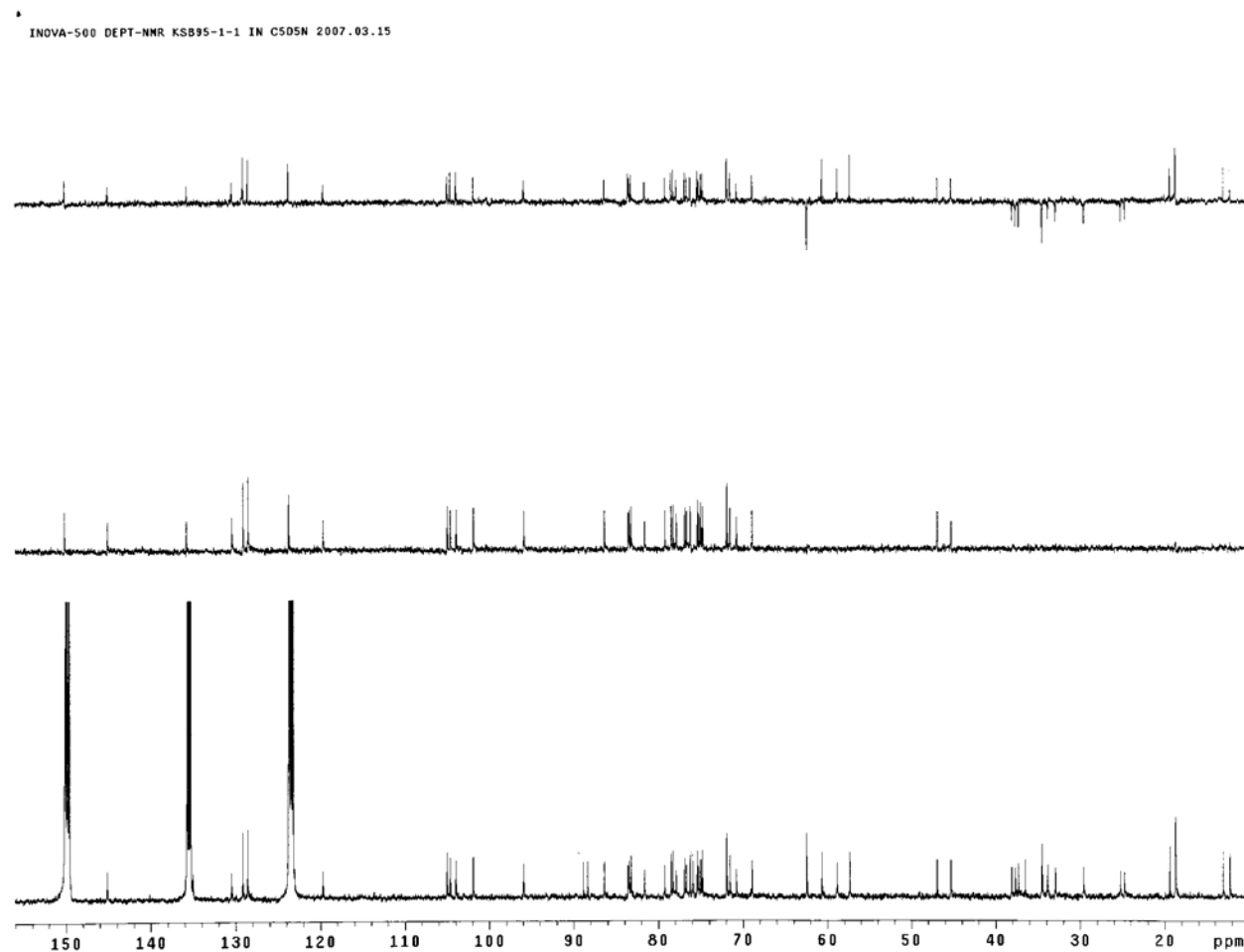


Figure S5. COSY spectra of new compound 1

INOVA-501 gCOSY KSB95-1-1 IN C505N 07.05.08

Solvent: Pyridine
Temp. 25.0 C / 288.1 K
INOVA-500 "INM-501"

Relax. delay 1.000 sec
Acq. time 0.218 sec
Width 4700.9 Hz
2D Width 4700.9 Hz
2 repetitions
256 increments
OBSERVE H1, 499.7702145 MHz
DATA PROCESSING
Sine bell 0.109 sec
F1 DATA PROCESSING
Sine bell 0.026 sec
FT size 2048 x 2048
Total time 11 min, 3 sec

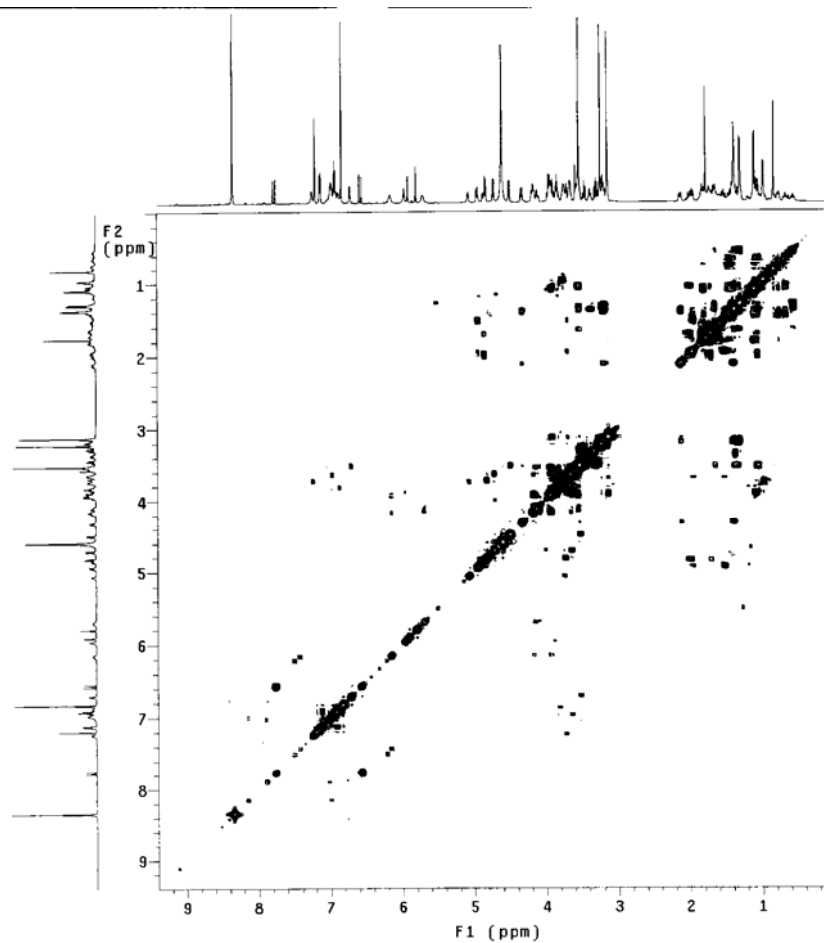


Figure S6. HMQC spectra of new compound 1

INOVA-501 gHSQC KS895-1-1 IN C5D5N 07.05.08

Solvent: Pyridine
Temp: 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "IMH-501"

Relax. delay 1.000 sec
Acq. time 0.219 sec
Width 4672.1 Hz
2D Width 21511.2 Hz
16 repetitions
256 increments
OBSERVE H1, 499.7702145 MHz
DECOUPLE C13, 125.6785340 MHz
Power 48 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Sine bell 0.046 sec
F1 DATA PROCESSING
Sine bell 0.006 sec
FT size 2048 x 4096
Total time 1 hr, 28 min, 53 sec

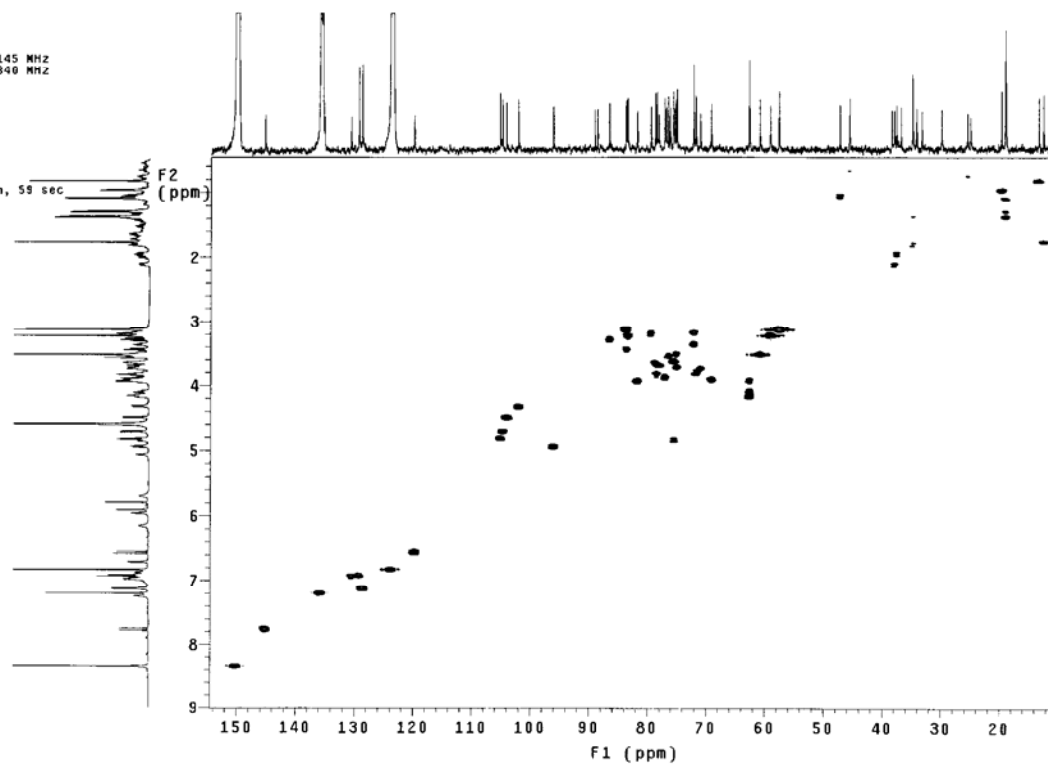


Figure S7. HMBC spectra of new compound 1

INOVA-501 gHMBC KSB95-1-1 IN C505N 07.05.08

Solvent: Pyridine
Temp. 25.0 C / 298.1 K
User: i-14-87
INOVA-500 "JNM-501"

Relax. delay 1.000 sec
Acq. time 0.221 sec
Width 4643.1 Hz
2D Width 21511.2 Hz
32 repetitions
320 increments
OBSERVE H1, 499.7702145 MHz
DATA PROCESSING
Sine bell 0.046 sec
F1 DATA PROCESSING
Sine bell 0.004 sec
FT size 2048 x 4096
Total time 3 hr, 44 min, 13 sec

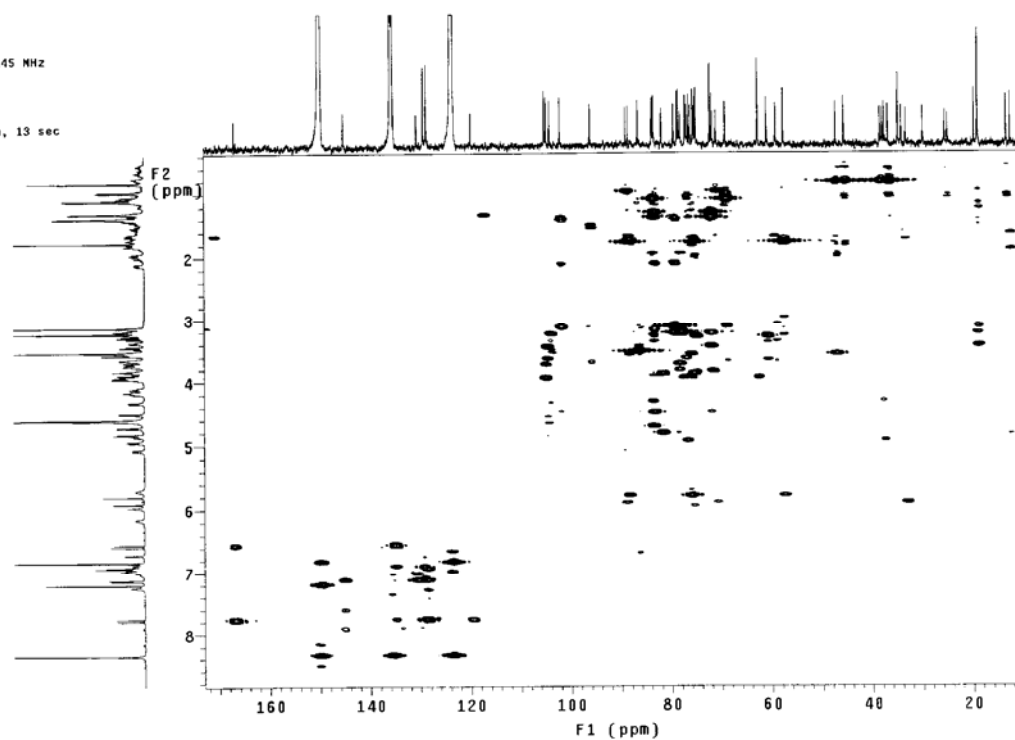


Figure S8. ESI-MSⁿ spectra of new compound 4

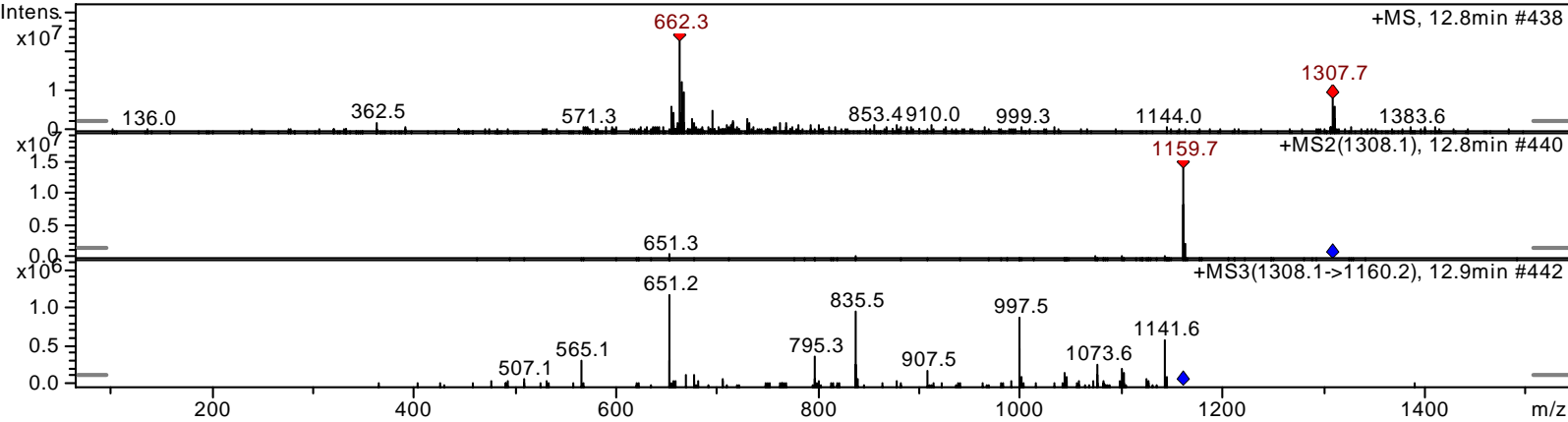


Figure S9. ^1H NMR (pyridine- d_5 500 M Hz) spectra of new compound 2

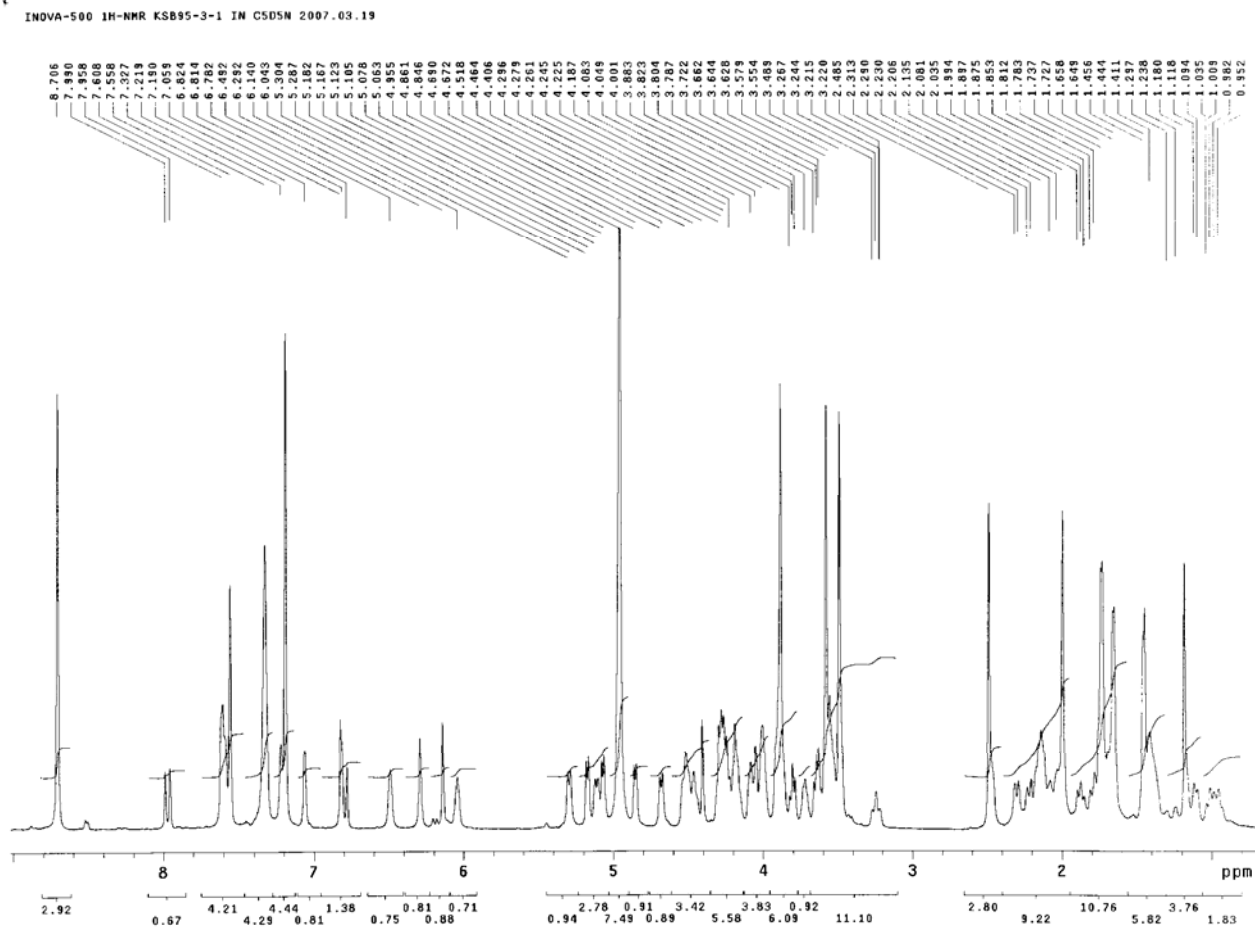


Figure S10. ^{13}C NMR (pyridine- d_5 125 M Hz) spectra of compound 4

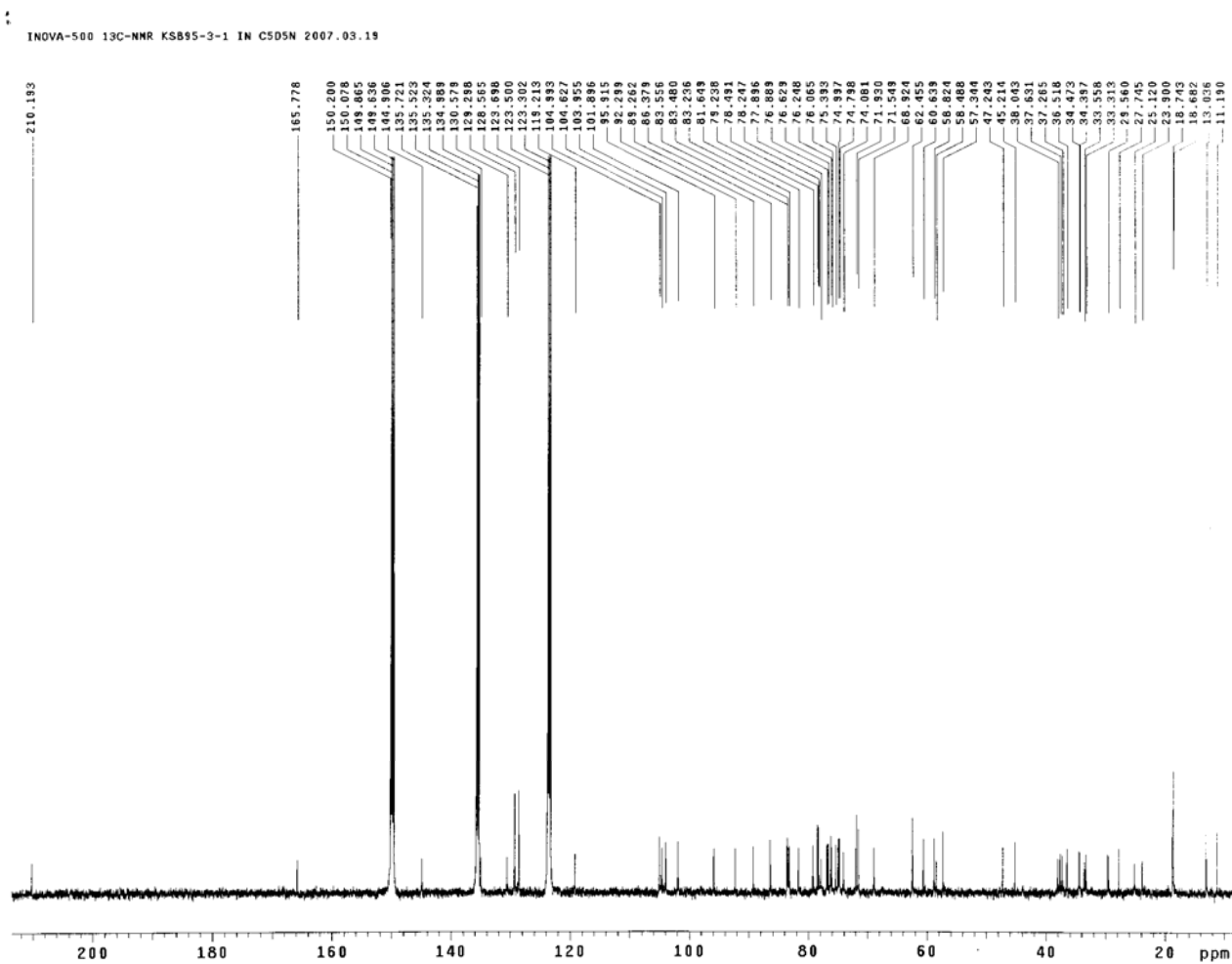


Figure S11. Dept spectra of new compound 4

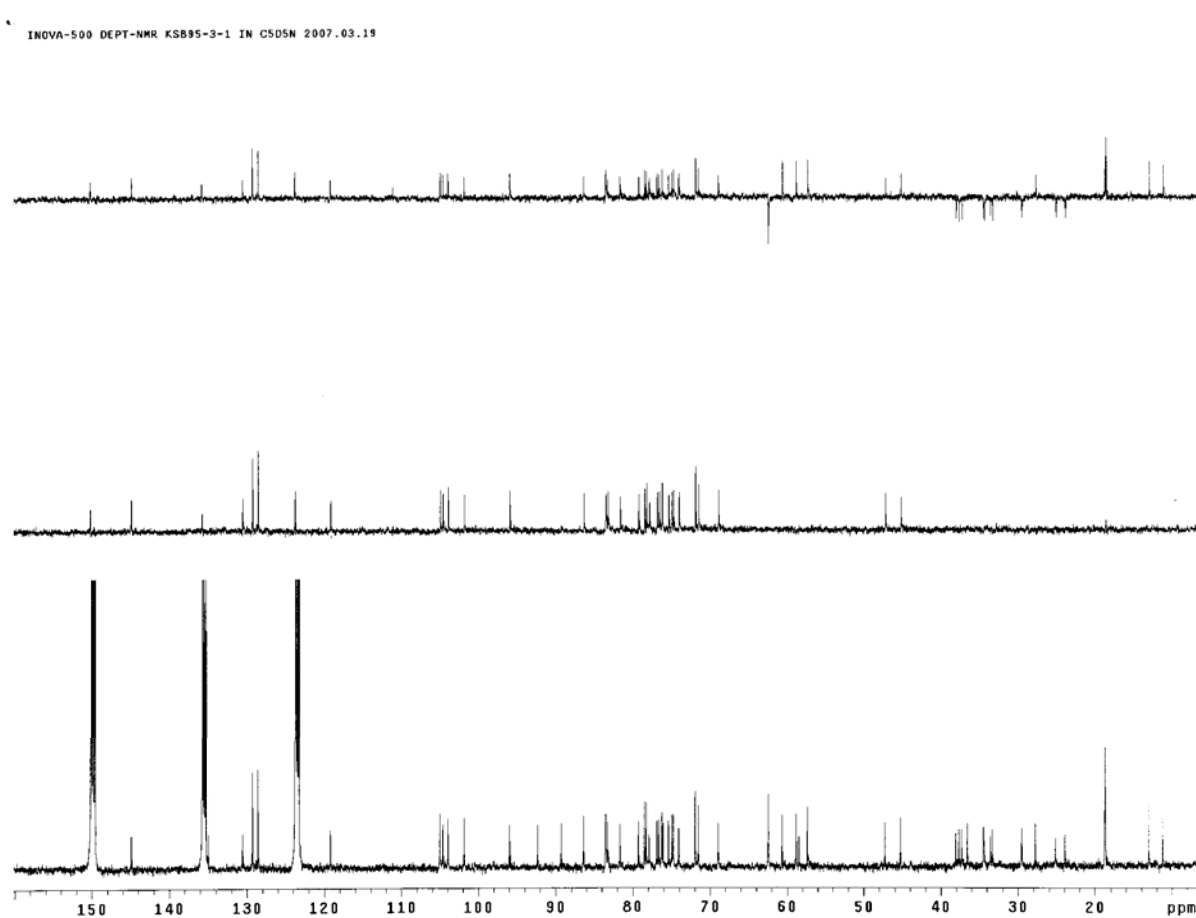


Figure S12. COSY spectra of new compound 4

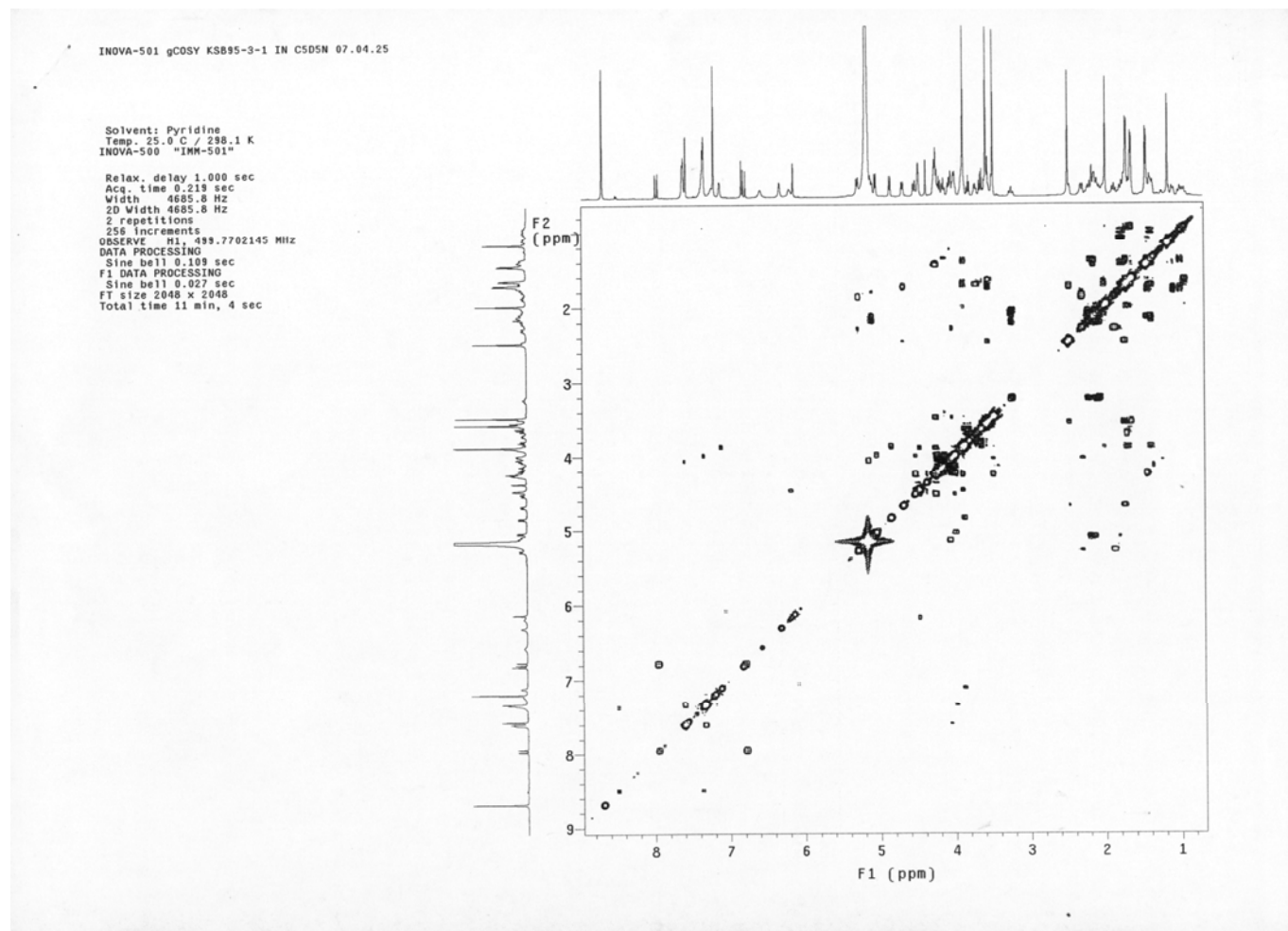


Figure S13. HMQC spectra of new compound 4

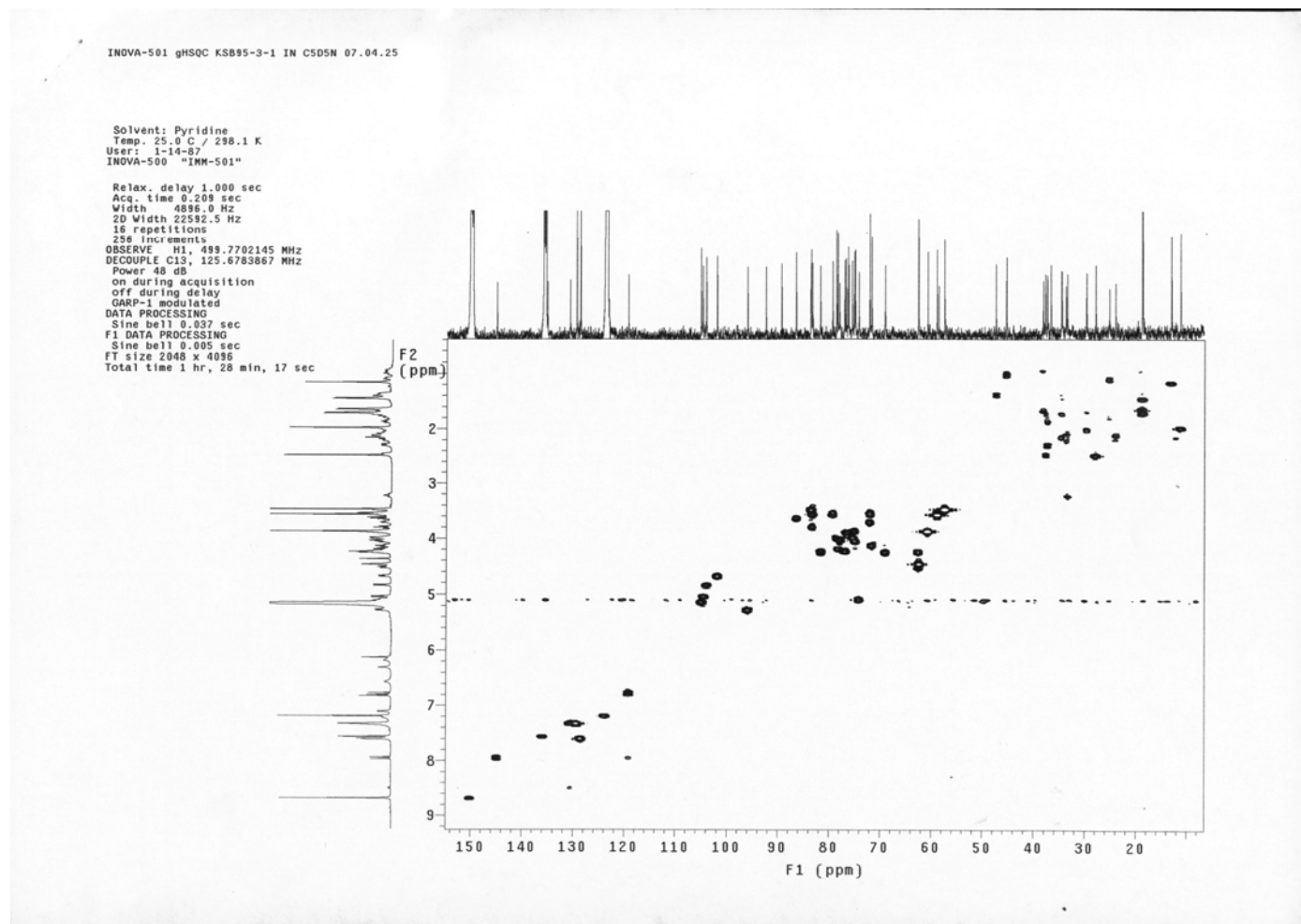


Figure S14. HMBC spectra of new compound 4

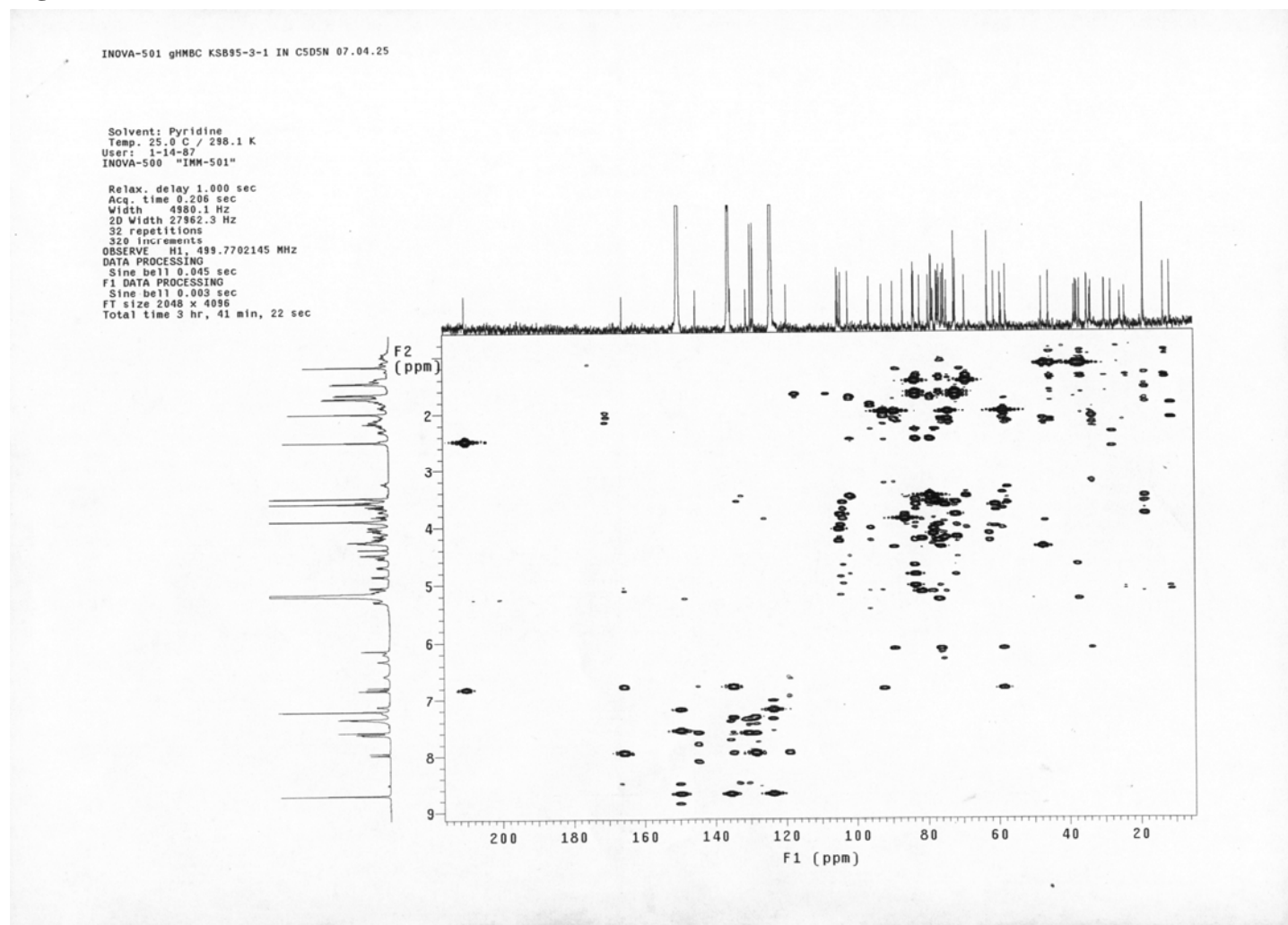


Figure S15 ESI-MSⁿ spectra of new compound 7

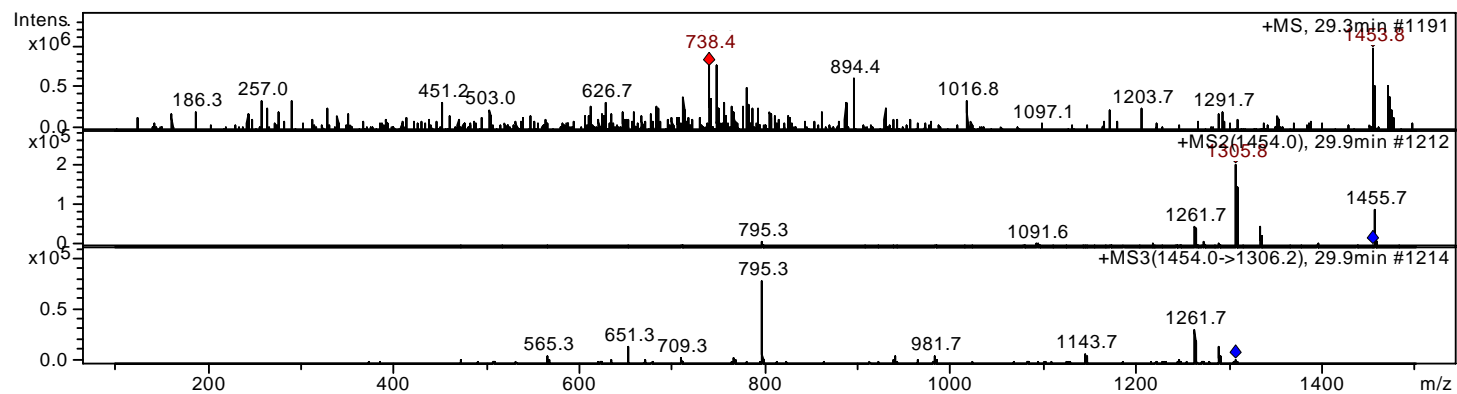


Figure S16. ^1H NMR (pyridine- d_5 500 M Hz) spectra of new compound 7

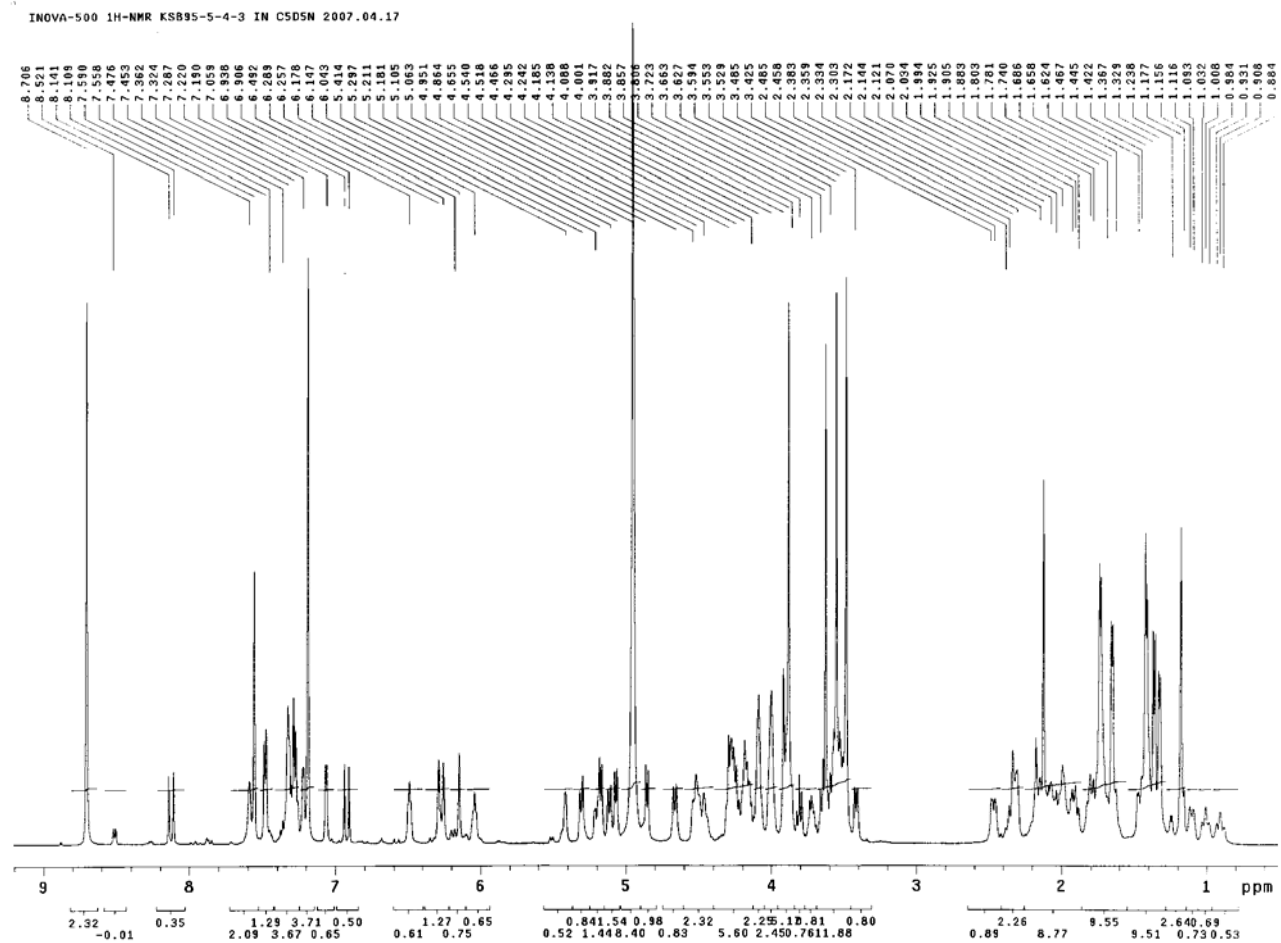


Figure S17. ^{13}C NMR (pyridine- d_5 125 M Hz) spectra of new compound 7

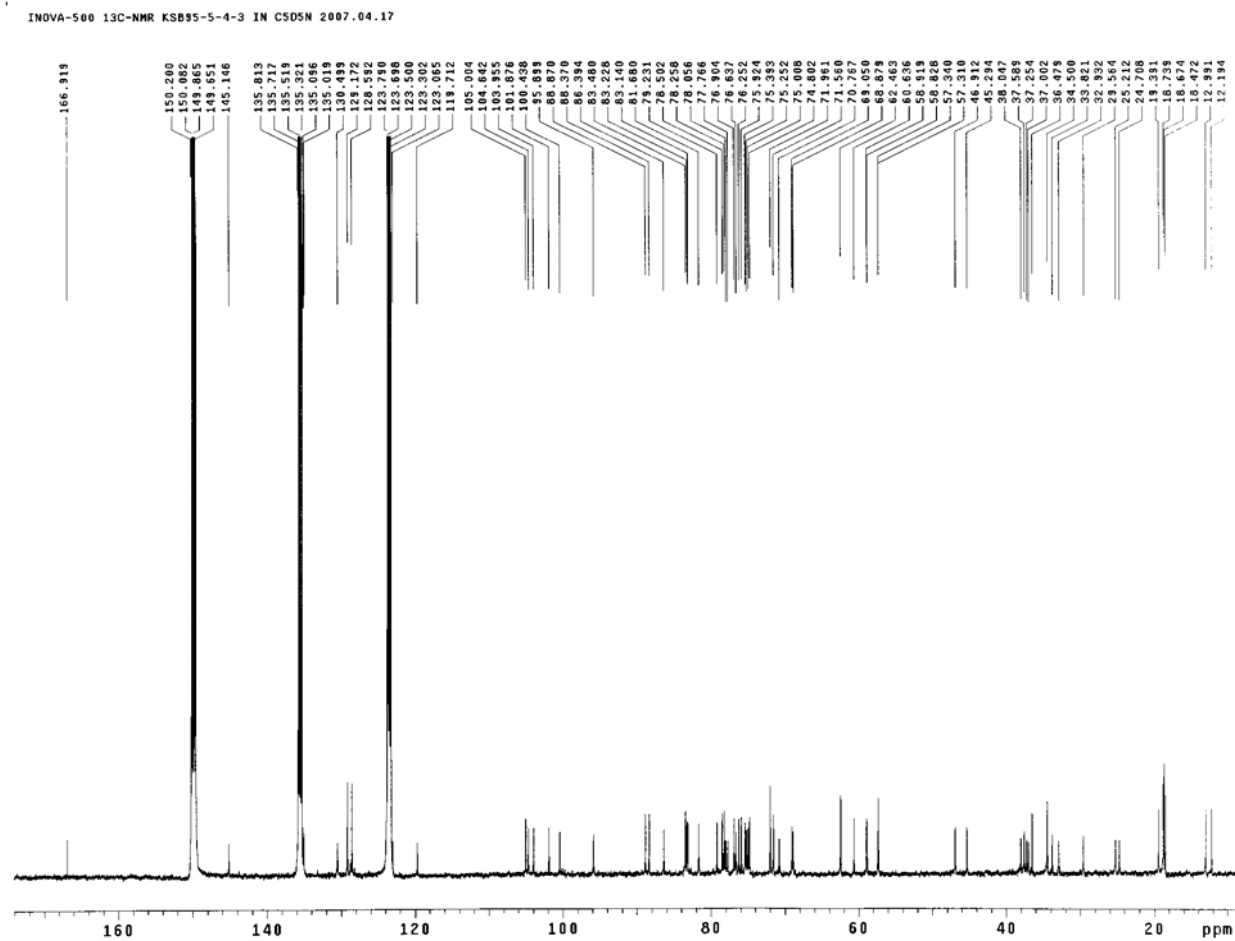


Figure S18. Dept spectra of new compound 7

INOVA-500 DEPT-NMR KSB95-5-4-3 IN C5D5N 2007.04.17

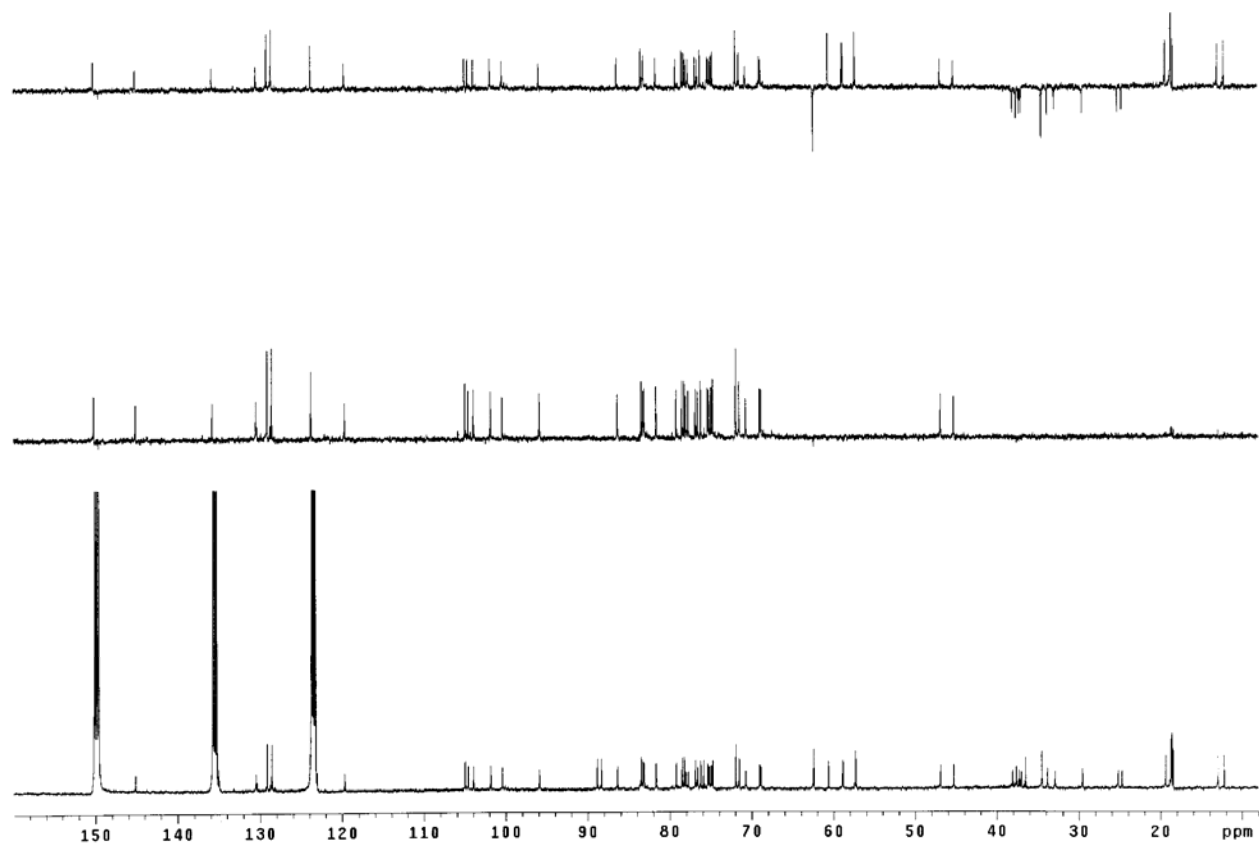


Figure S19. COSY spectra of new compound 7

INOVA-501 gCOSY KSB95-5-4-3 IN C5DSN 07.05.10

Solvent: Pyridine
Temp: 25.0 C / 298.1 K
INOVA-500 "1H-501"

Relax. delay 1.000 sec
Acq. time 0.236 sec
Width 4344.5 Hz
2D Width 4344.5 Hz
2 repetitions
256 increments
OBSERVE H1, 499.7702145 MHz
DATA PROCESSING
Sine bell 0.118 sec
F1 DATA PROCESSING
Sine bell 0.029 sec
FT size 2048 x 2048
Total time 11 min, 14 sec

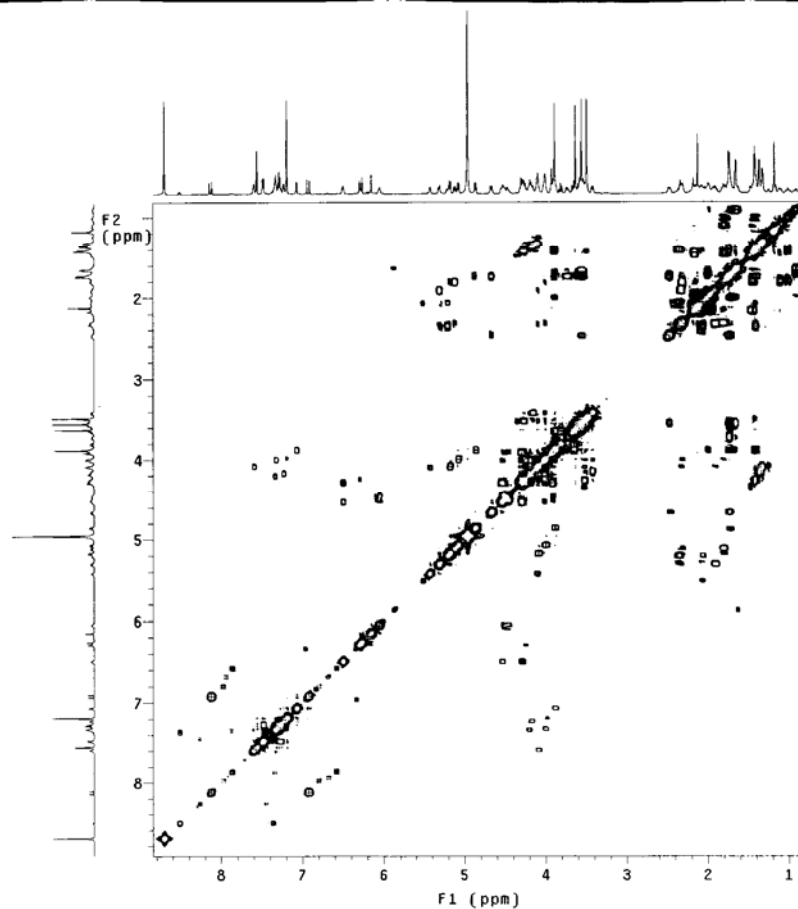


Figure S20. HMQC spectra of new compound 7

INOVA-501 gHSQC KSB95-5-4-3 IN C5D5N 07.05.10

Solvent: Pyridine
Temp. 25.0 °C / 298.1 K
User: 1-14-87
INOVA-500 "IMM-501"

Relax. delay 1.000 sec
Acq. time 0.232 sec
Width 4012.1 Hz
2D Width 21248.3 Hz
16 repetitions
256 increments
OBSERVE H1, 499.7702145 MHz
DECOUPLE C13, 125.6784017 MHz
Power 48 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Sine bell 0.050 sec
F1 DATA PROCESSING
Sine bell 0.006 sec
F1 size 2048 x 4096
Total time 1 hr, 29 min, 53 sec

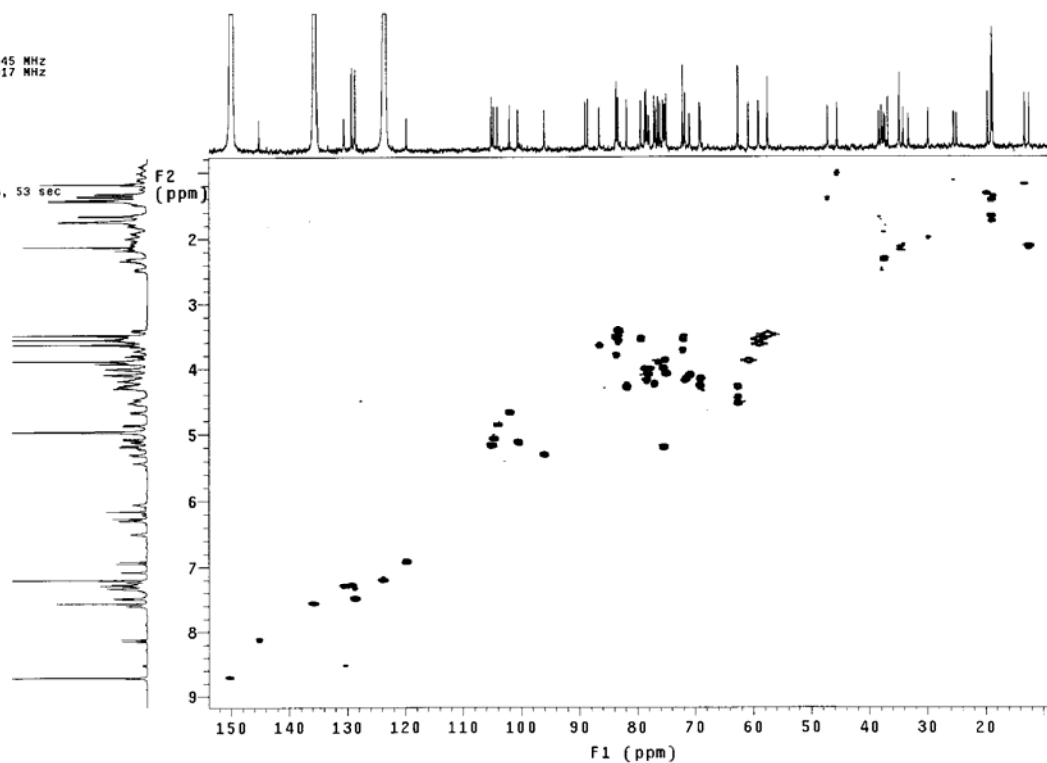


Figure S21. HMBC spectra of new compound 7

INOVA-501 gHMBC KSB95-5-4-3 IN C505N 07.05.11

Solvent: Pyridine
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "1HN-501"

Relax. delay 1.000 sec
Acq. time 0.219 sec
Width 4682.2 Hz
2D Width 22061.1 Hz
32 repetitions
320 increments
OBSERVE R1, 499.7702145 MHz
DATA PROCESSING
Sine bell 0.050 sec
F1 DATA PROCESSING
Sine bell 0.004 sec
FT size 2048 x 4096
Total time 3 hr, 43 min, 52 sec

