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APPENDICES

ต้นฉบับ หน้าขาดหาย

ต้นฉบับ หน้าขาดหาย

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APPENDIX B

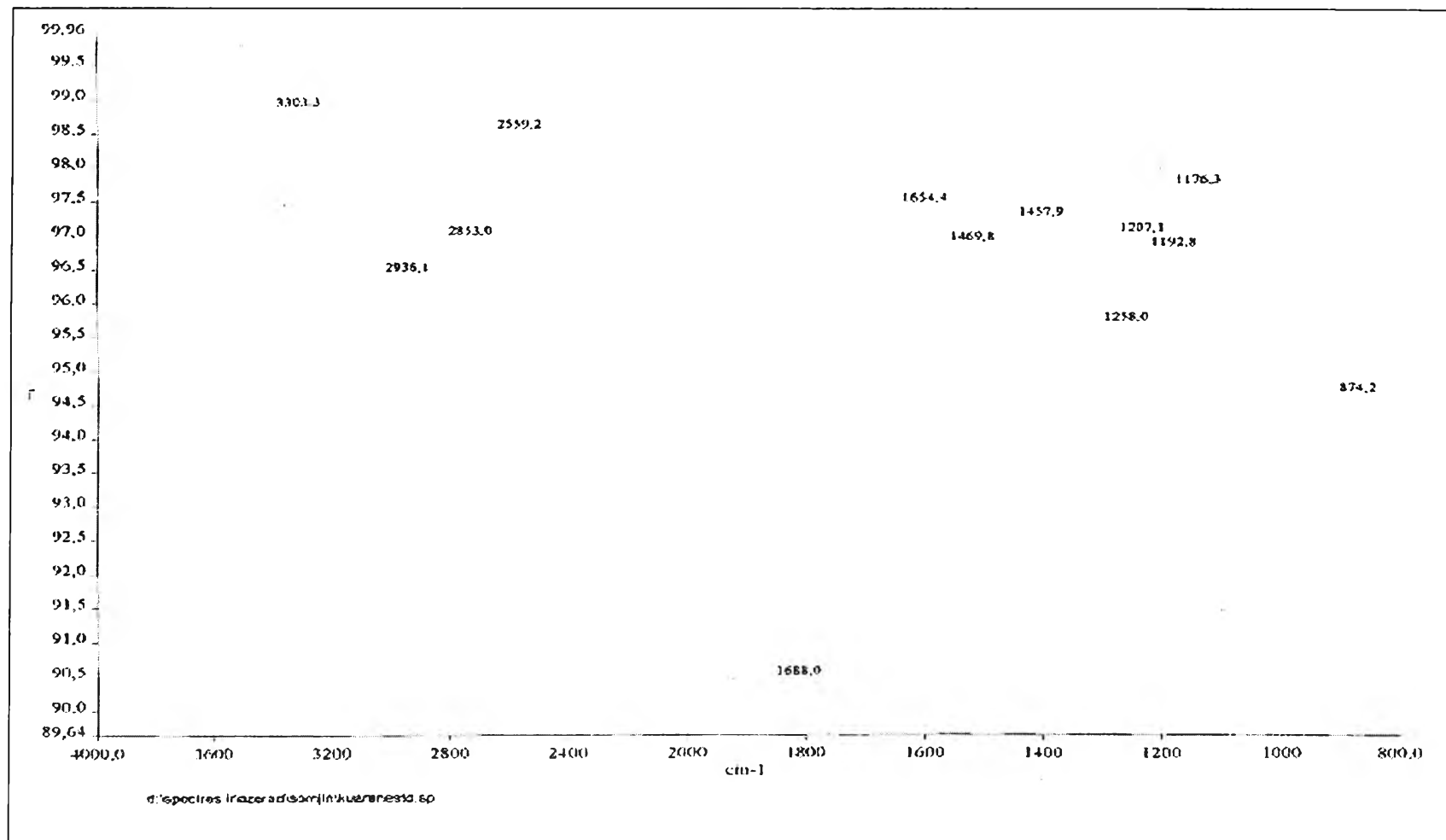


Figure B1. The IR spectrum of Compound 1.

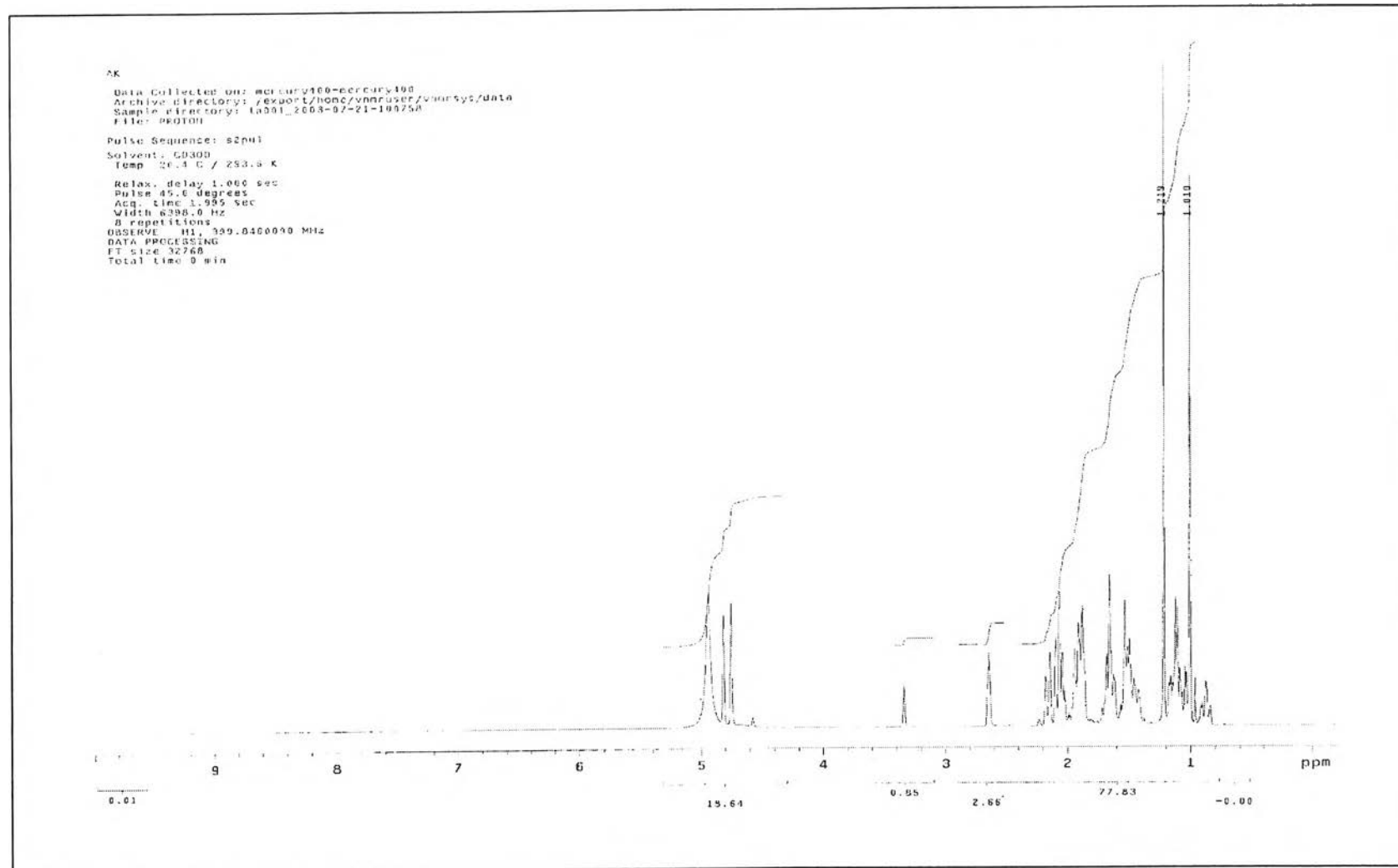


Figure B2. The ^1H -NMR spectrum of Compound 1.

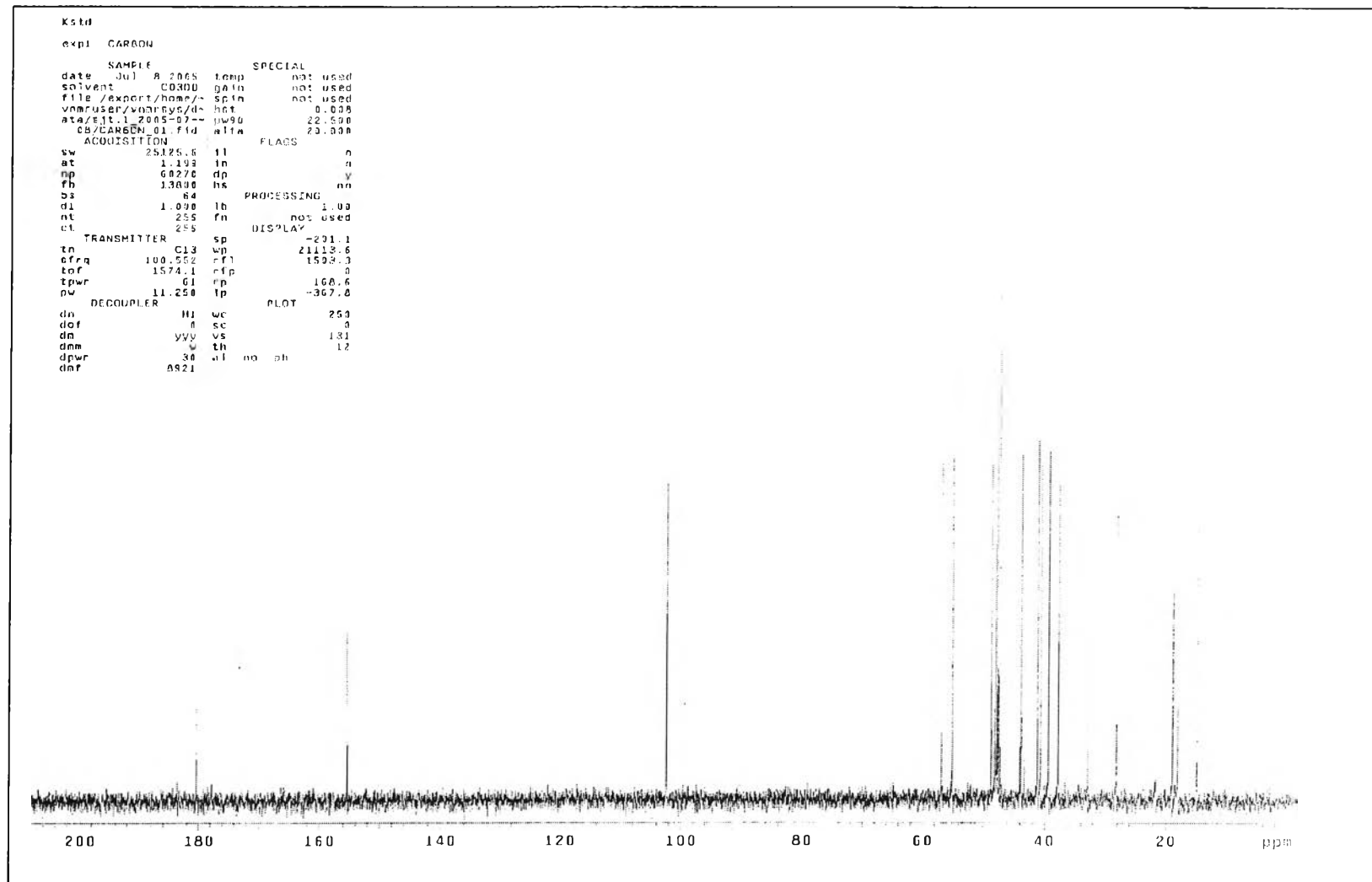


Figure B3. The ^{13}C -NMR spectrum of Compound 1.

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Vial Number : 1

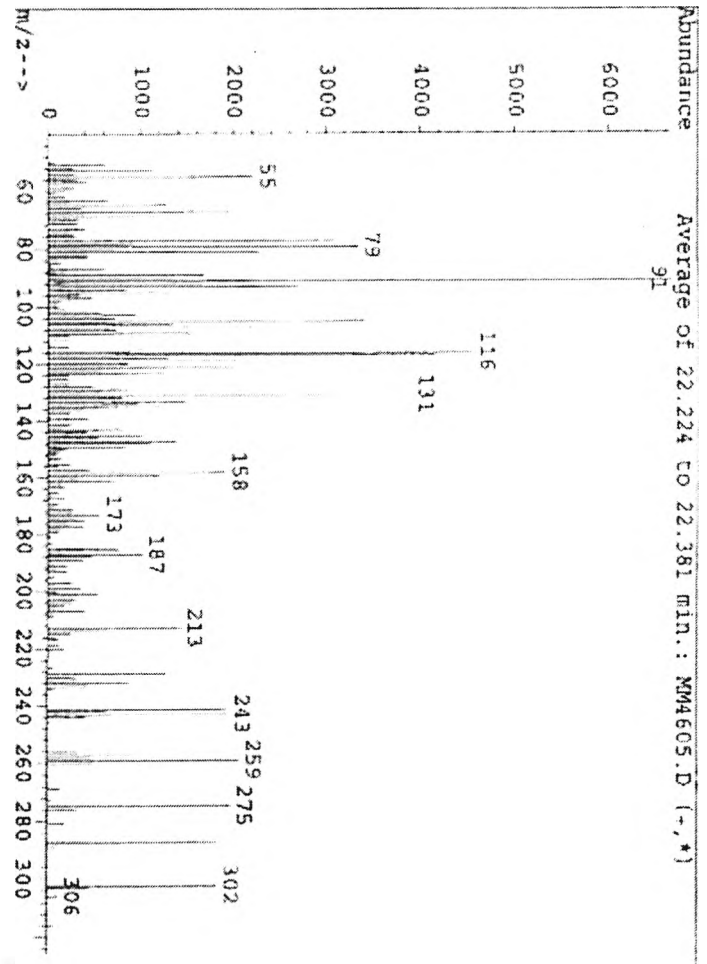
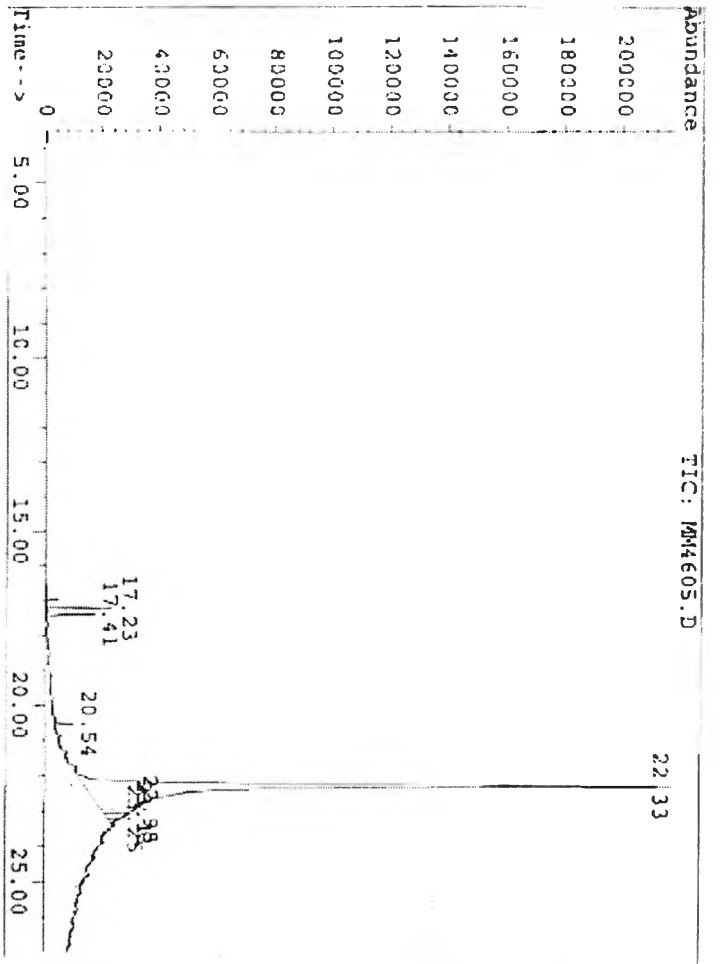


Figure B4. The GC- MS spectrum of Compound 1.

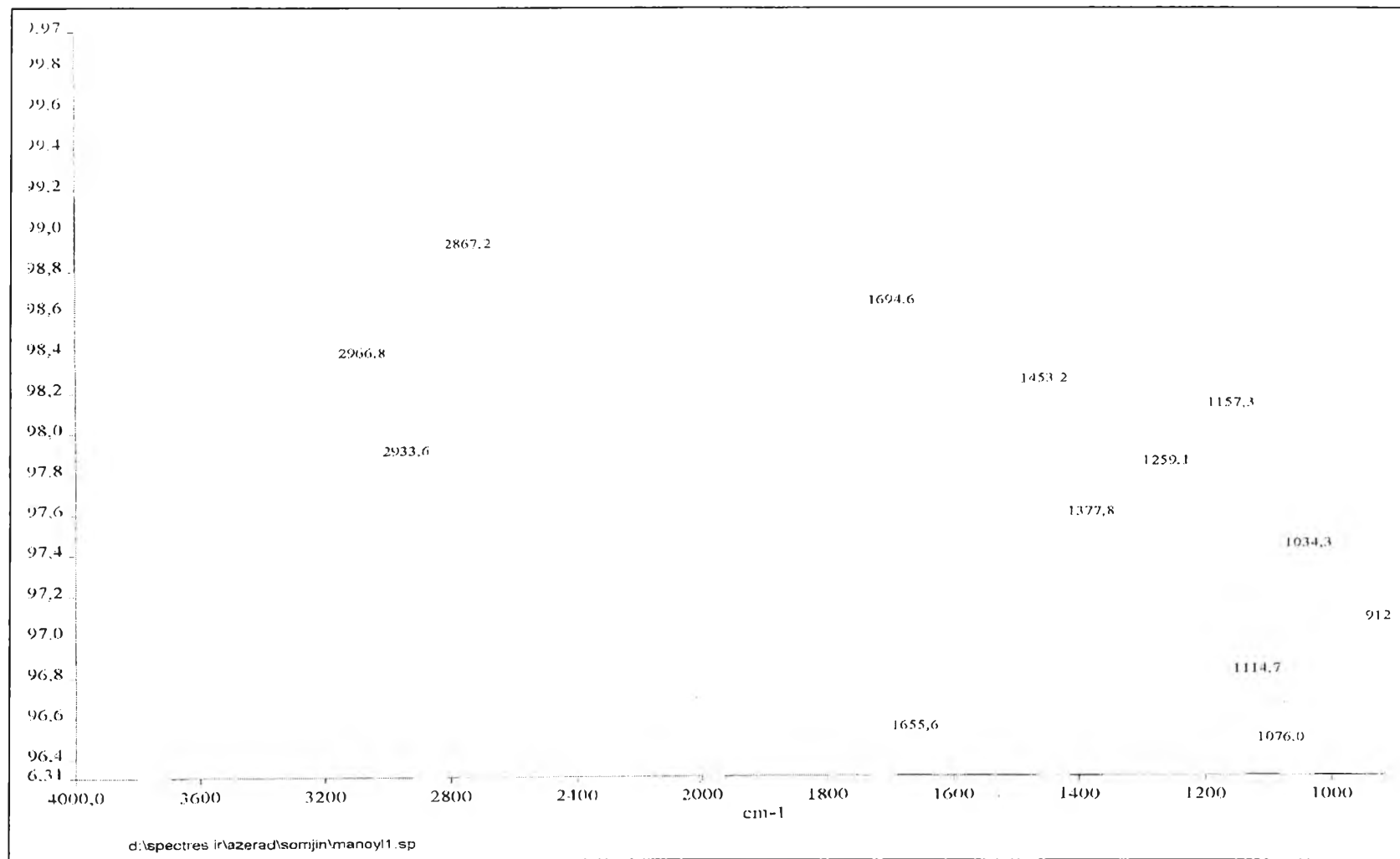


Figure B5. The IR spectrum of Compound 2.

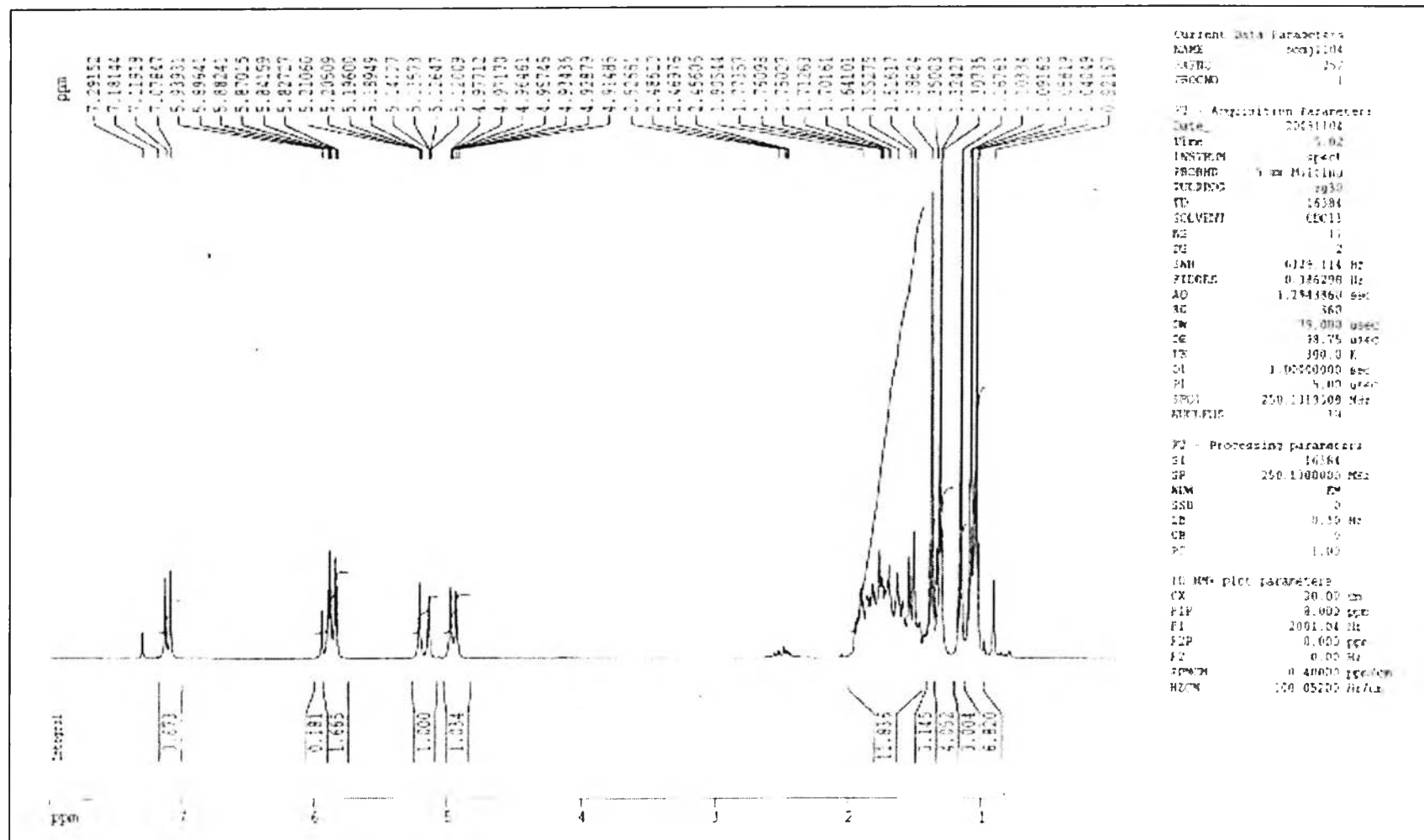
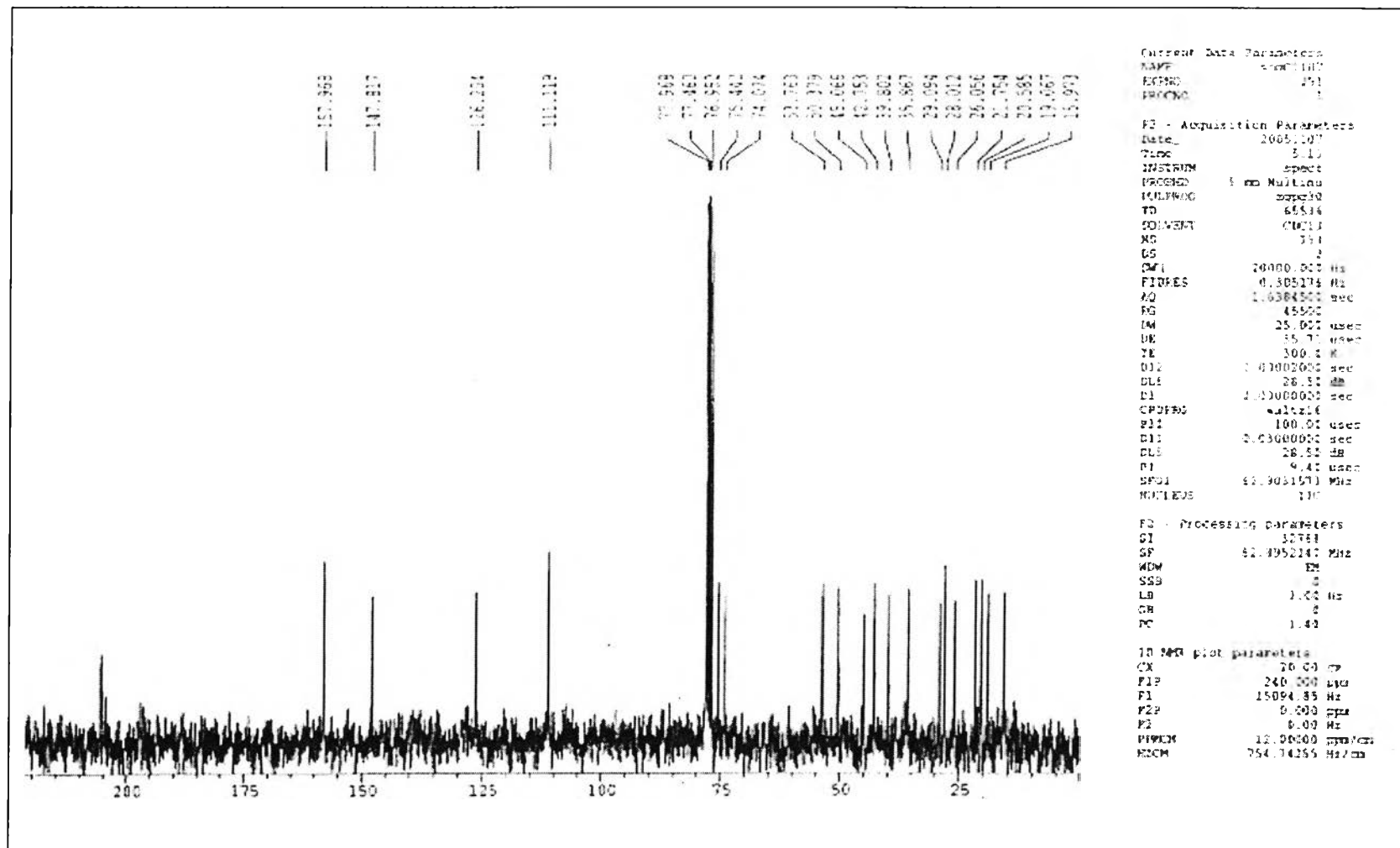


Figure B6. The ¹H-NMR spectrum of Compound 2



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Instrument : 5972 - In
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Vial Number: 1

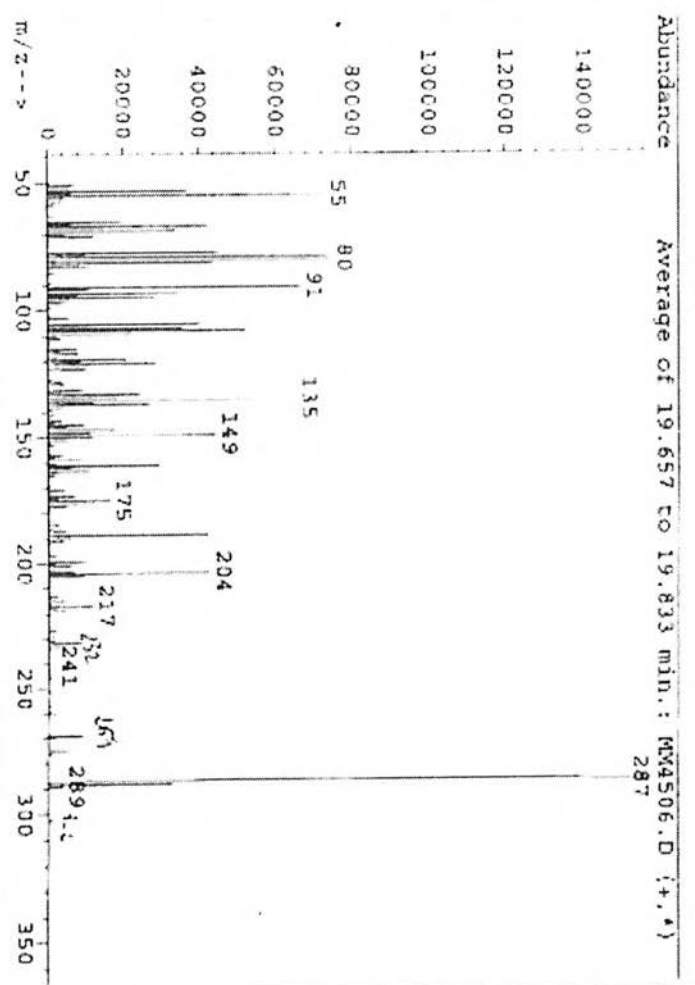
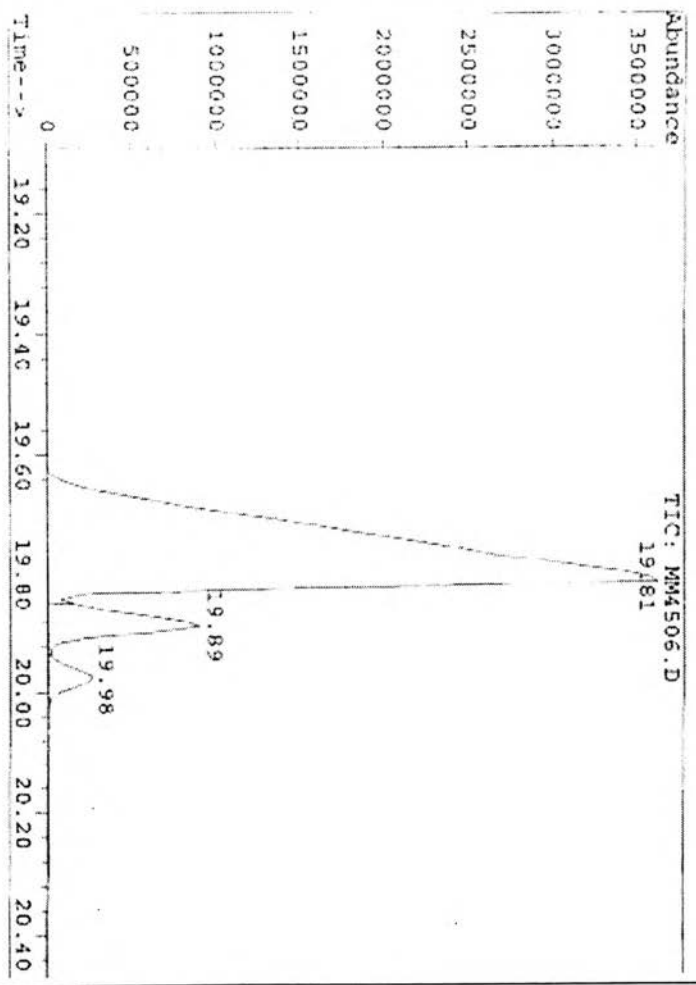


Figure B8. The GC- MS spectrum of Compound 2.

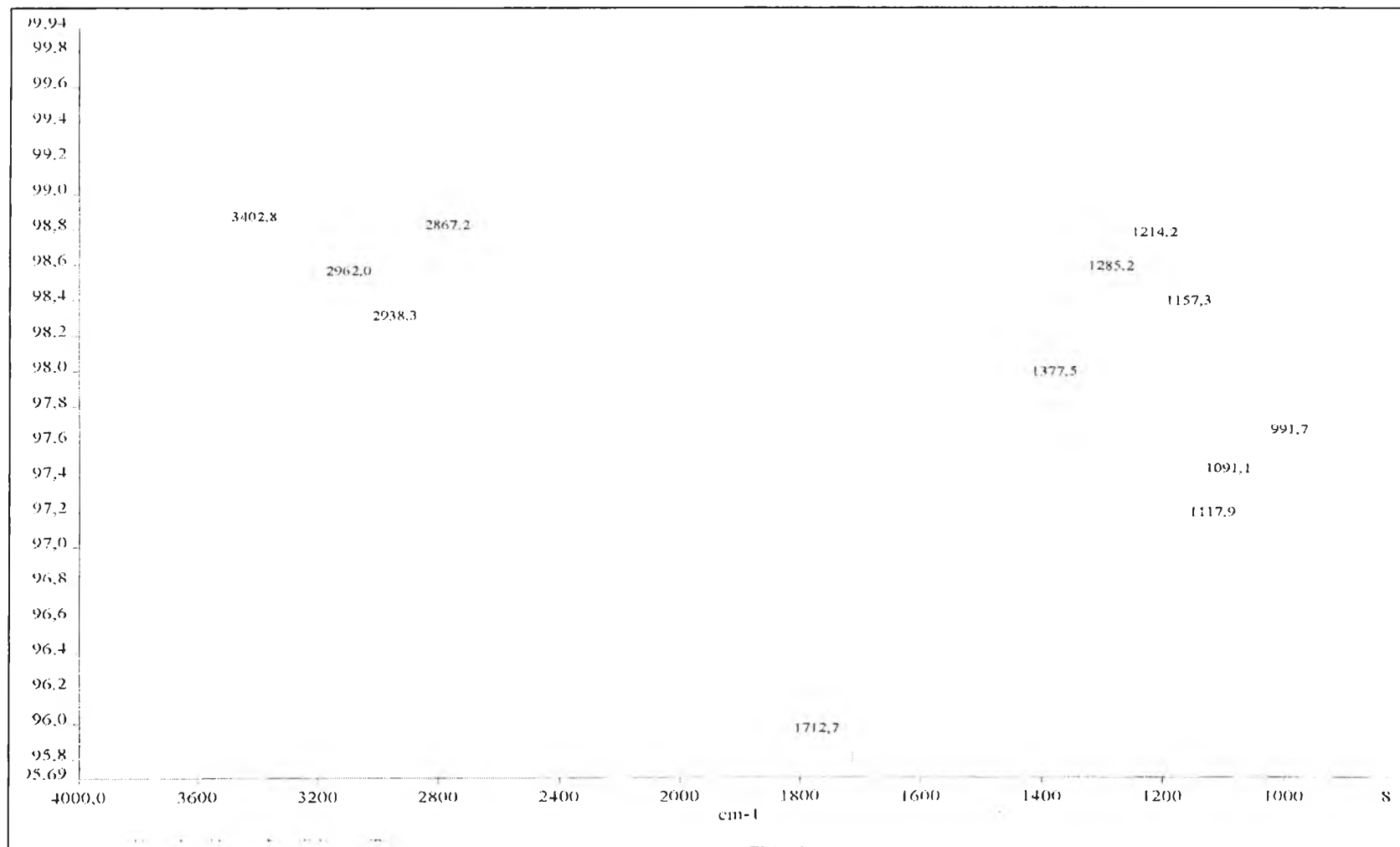


Figure B9. The IR spectrum of Compound 3.

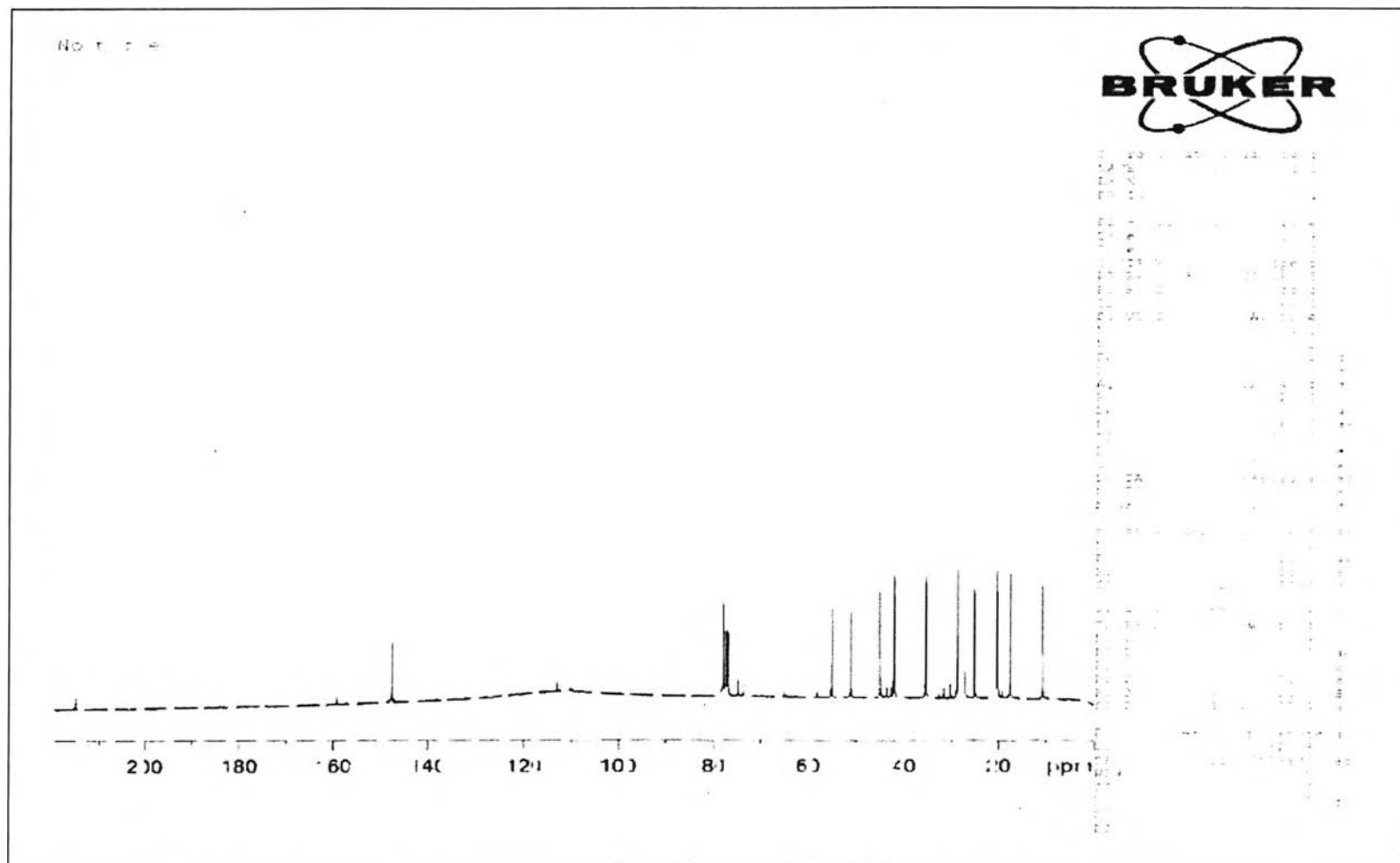


Figure B11. The ^{13}C -NMR spectrum of Compound 3.

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Vial Number: 1

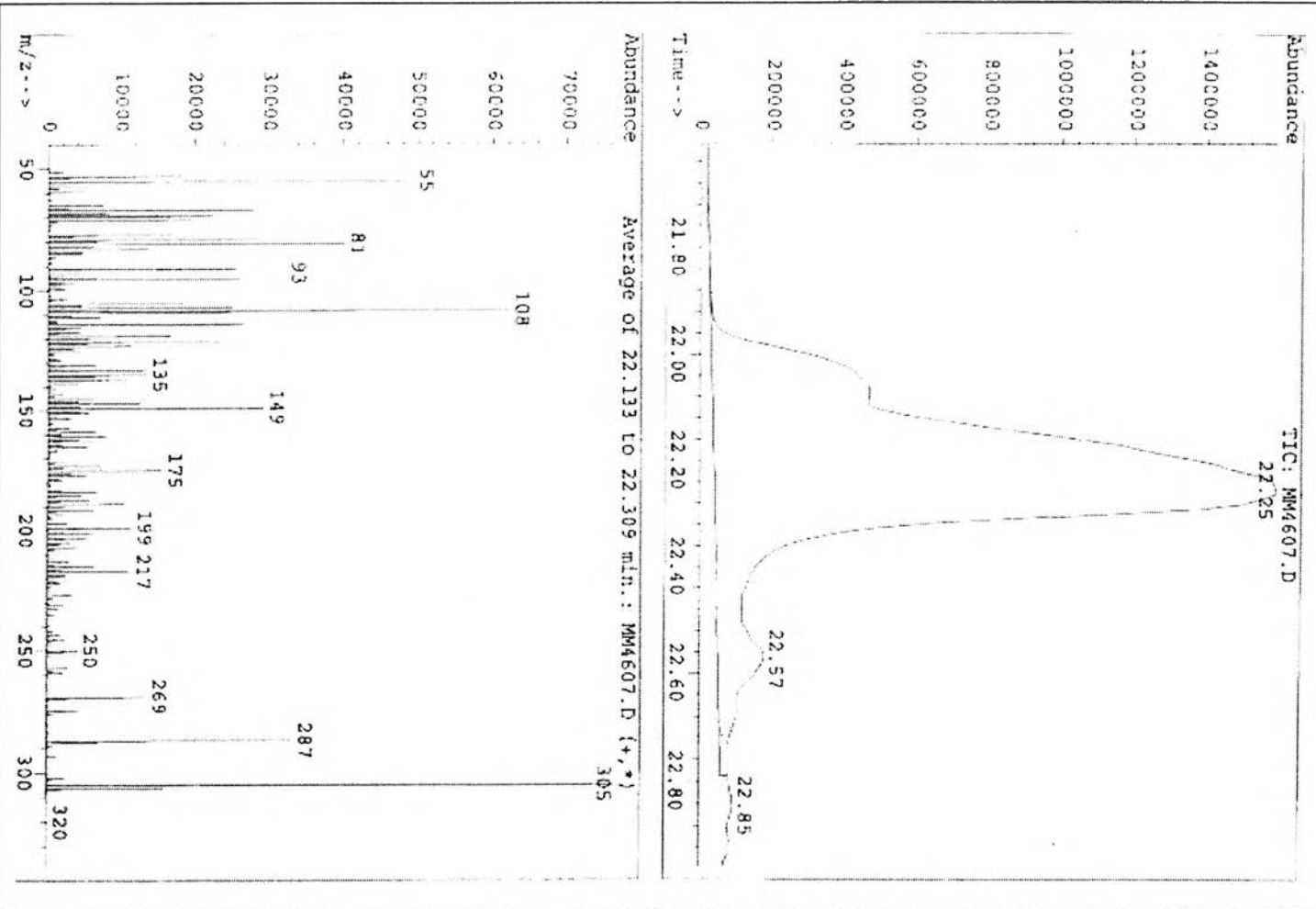


Figure B12. The GC- MS spectrum of Compound 3.

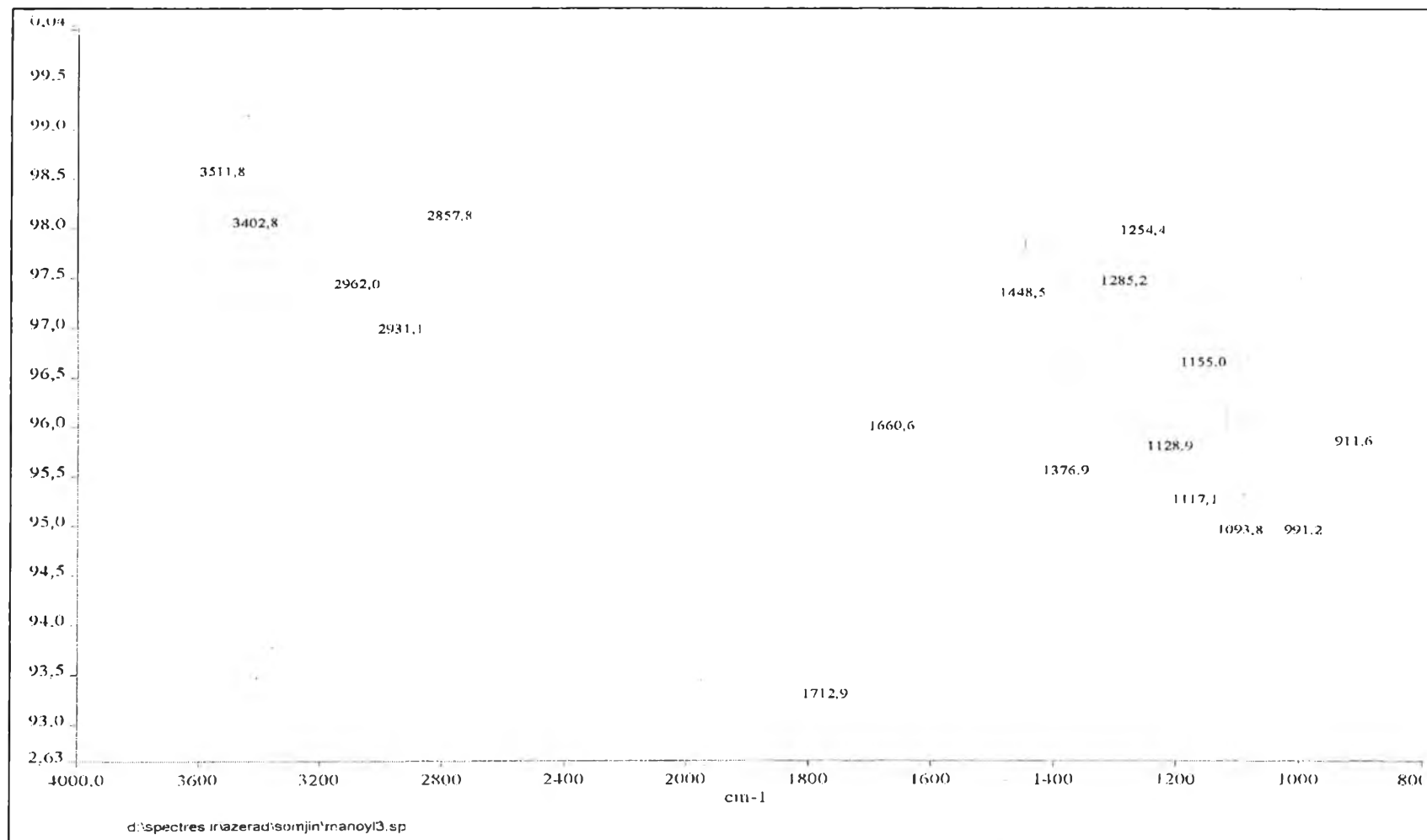


Figure B13. The IR spectrum of Compound 4.

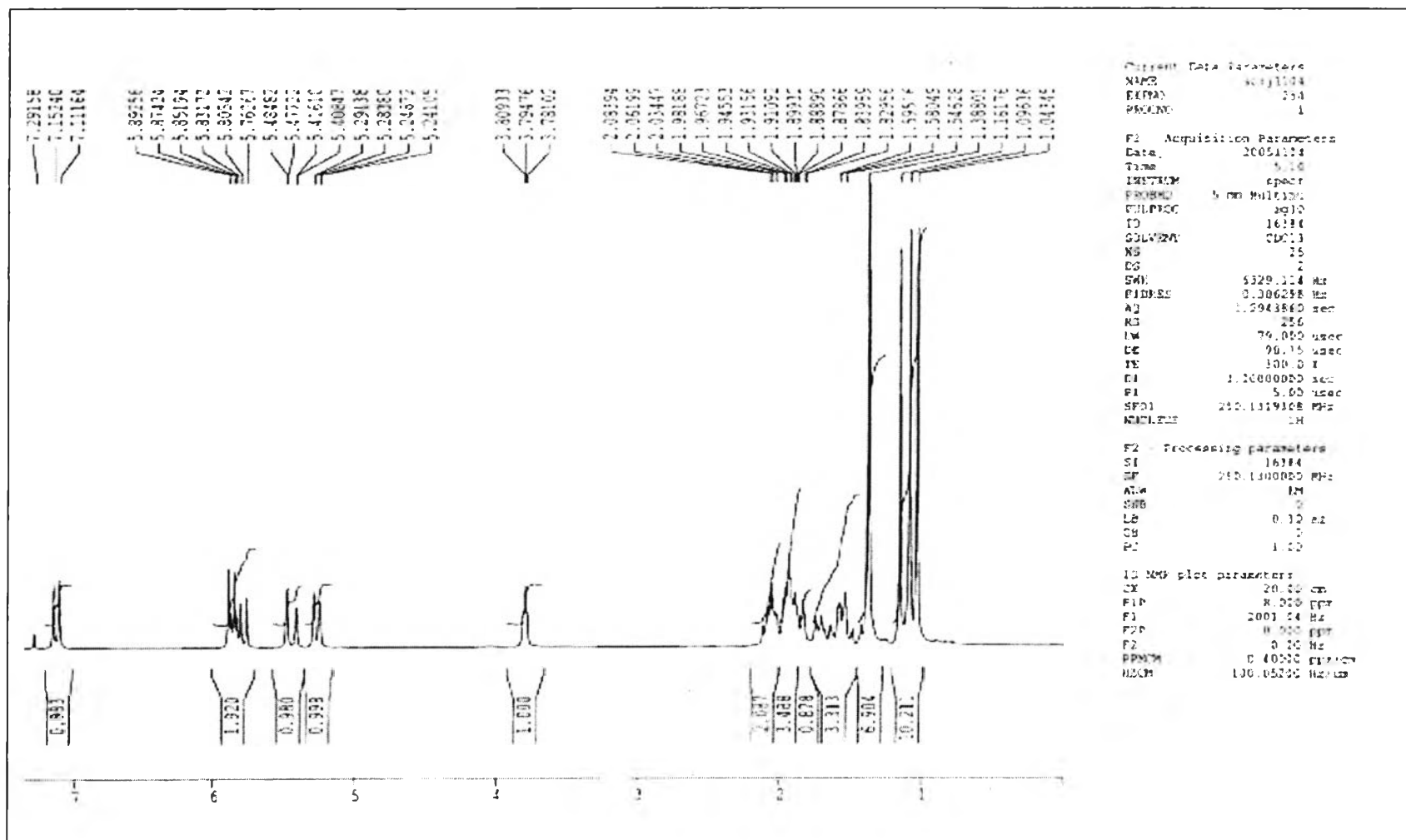


Figure B14. The ¹H-NMR spectrum of Compound 4.

No title



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PROCNO    1

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SI        16394
SOLVENT   Acetone
NS        18432
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SWH       20001.000 MHz
FIDRES    0.449580 Hz
AQ        0.2294000 sec
RG        32768
DQ        19.500 usec
DE        6.00 usec
TE        300.2 K
D1        1.00000000 sec
d11       0.10000000 sec
DELTA    0.99999999 sec
MORPH1    0.00000000 sec
MORPH2    0.01000000 sec

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P1        12.00 usec
PC1       50.00 dB
SFO1      125.7611797 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2       120.00 dB
PL12      23.00 dB
PL13      23.00 dB
SFO2      500.137797 MHz

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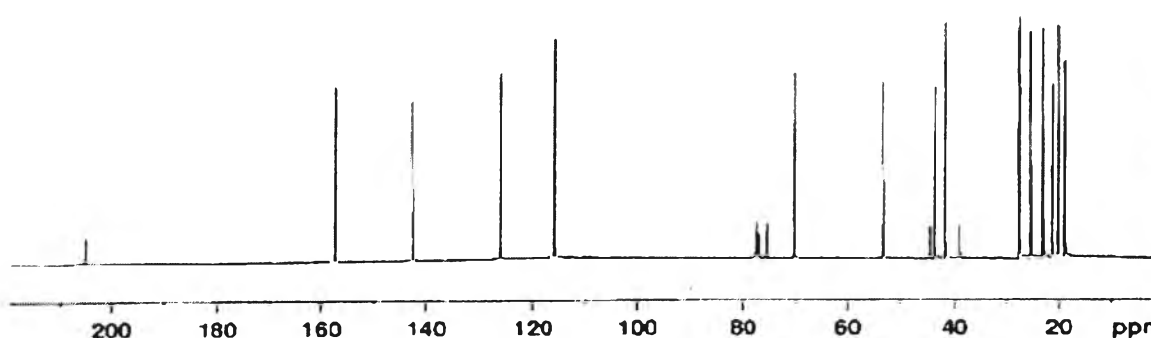


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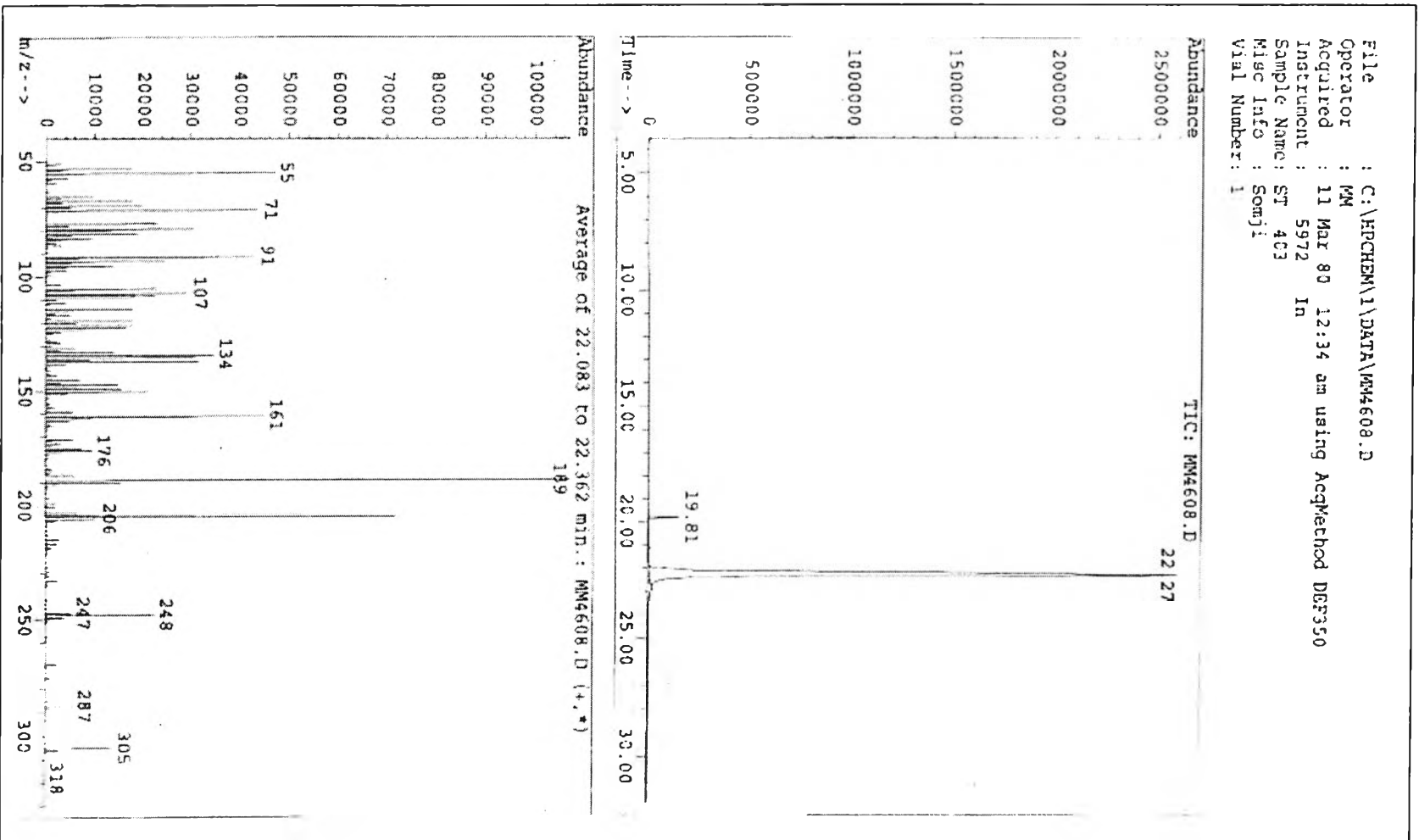


Figure B16. The GC- MS spectrum of Compound 4.

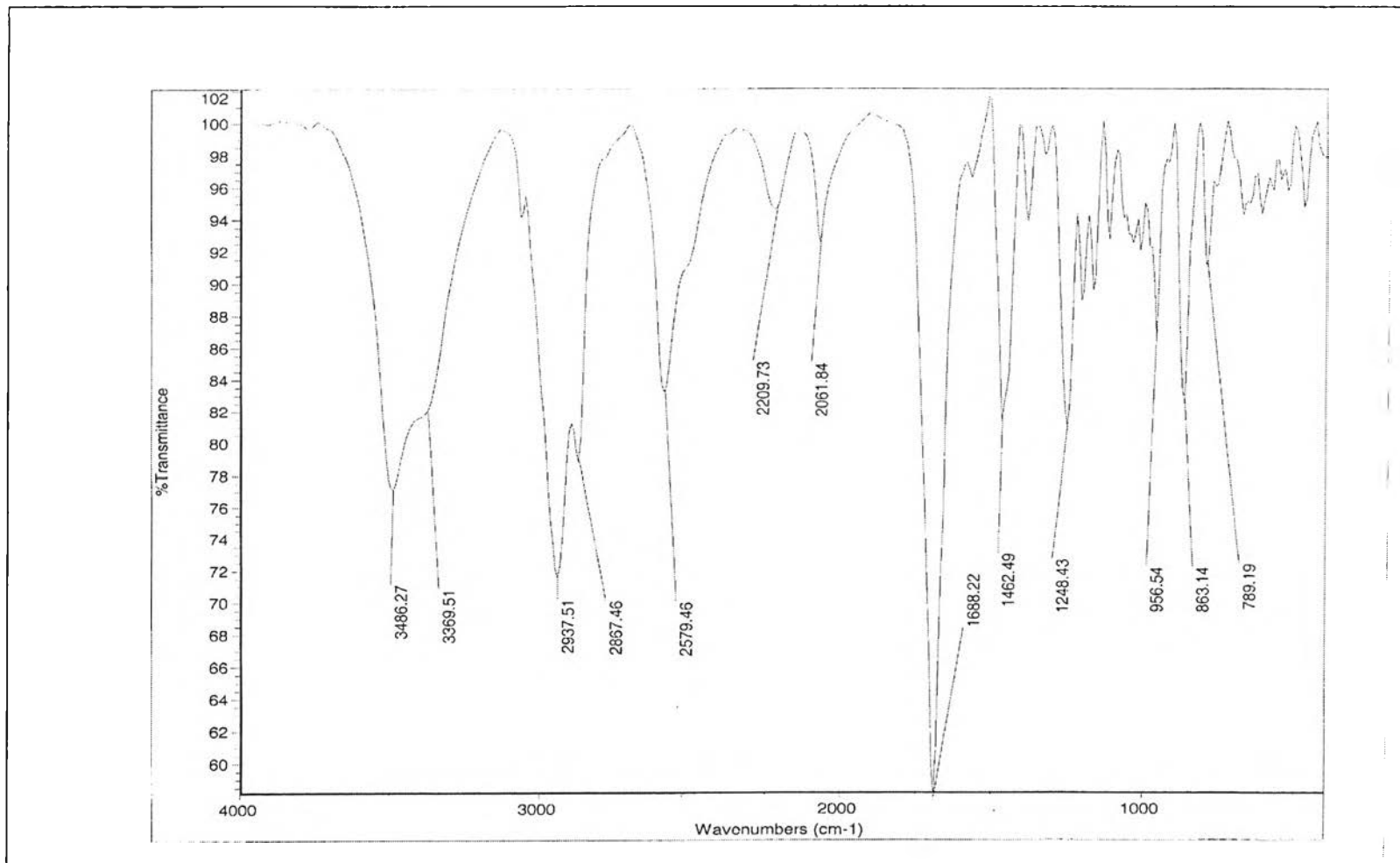


Figure B17. The IR spectrum of Metabolite 1a.

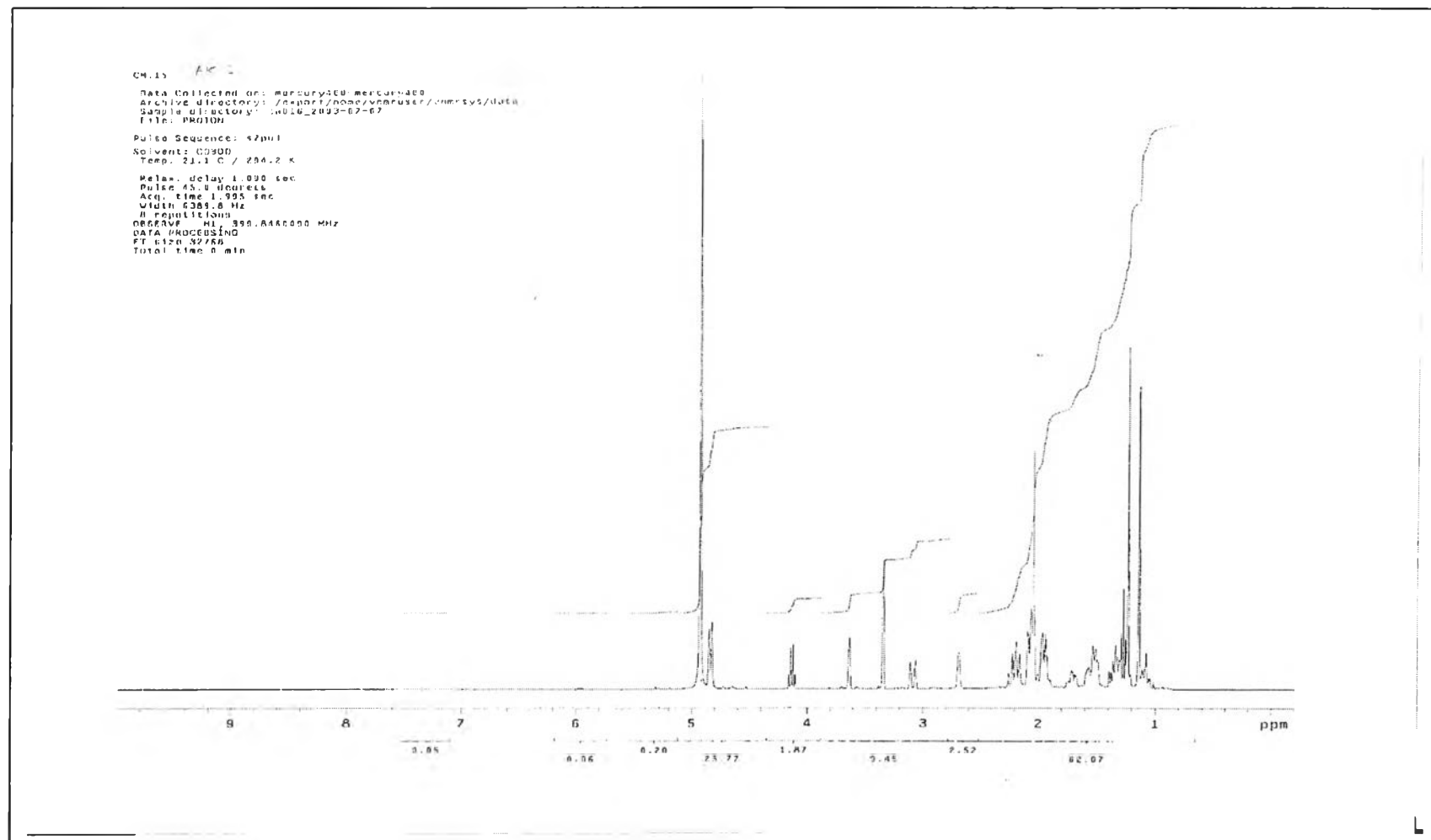


Figure B18. The ^1H -NMR spectrum of Metabolite 1a.

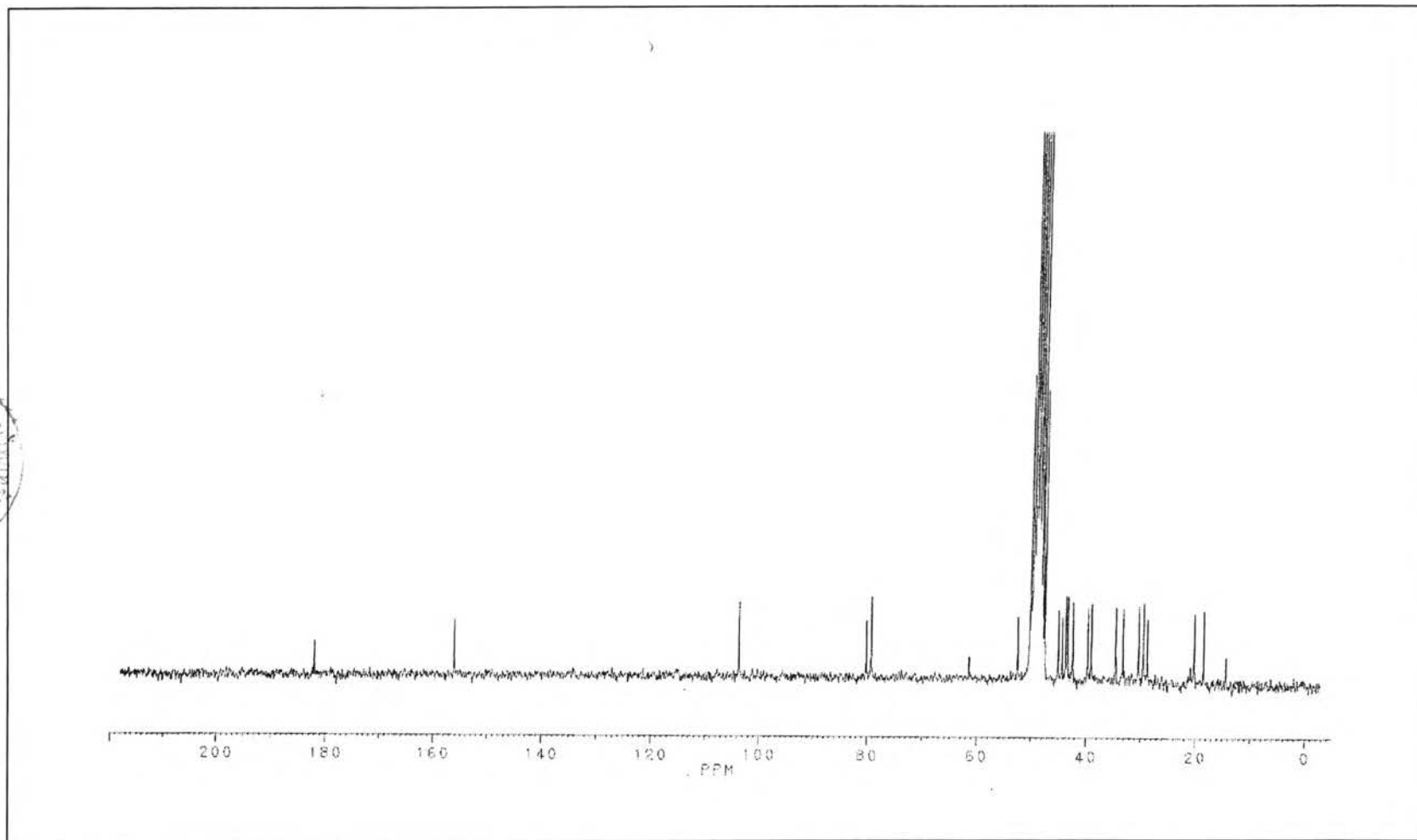


Figure B19. The ^{13}C -NMR spectrum of Metabolite **1a**.

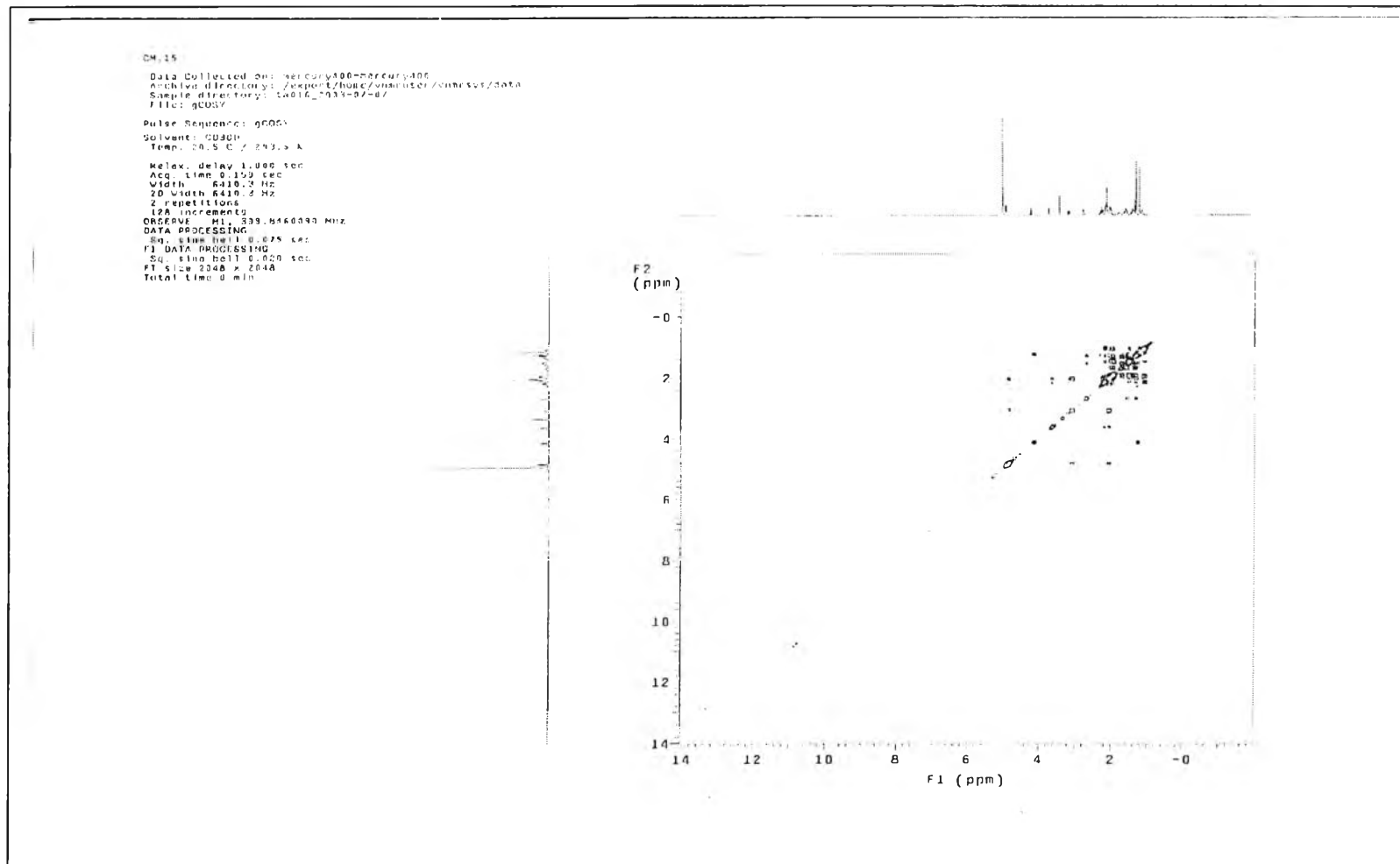


Figure B20. The gCOSY spectrum of Metabolite **1a**.

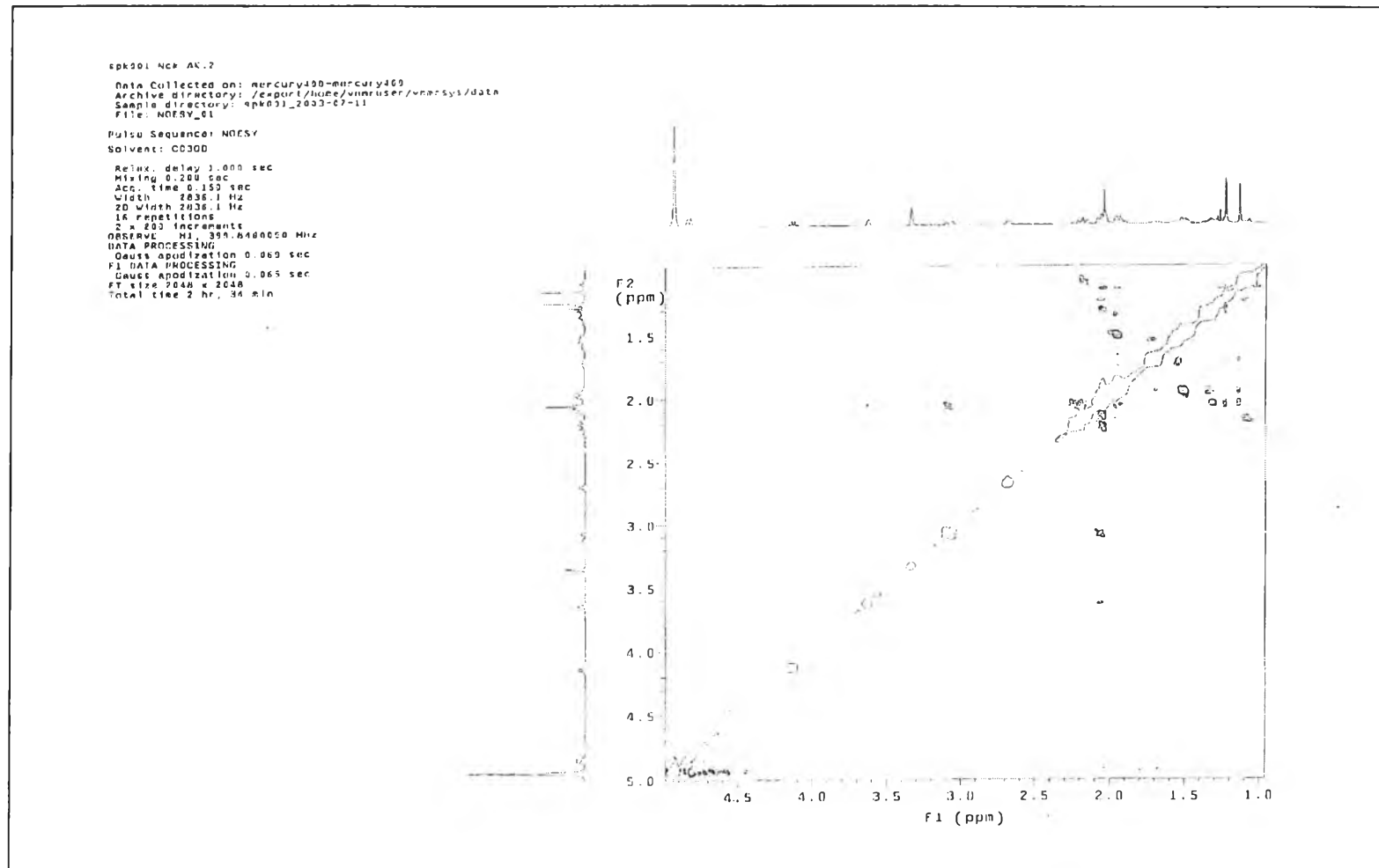


Figure B21. The gNOESY spectrum of Metabolite 1a.

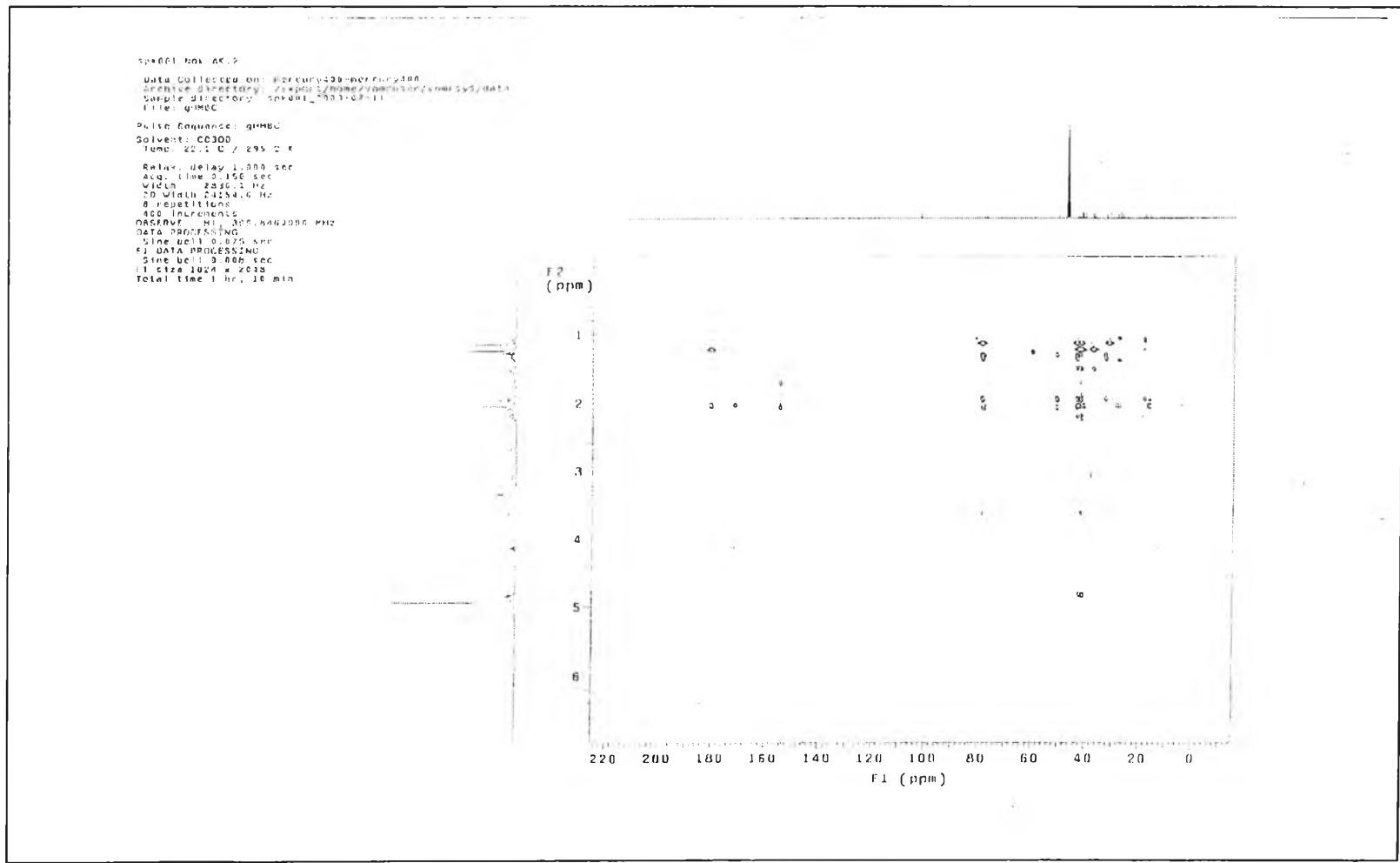


Figure B22. The gHMBC spectrum of Metabolite 1a.

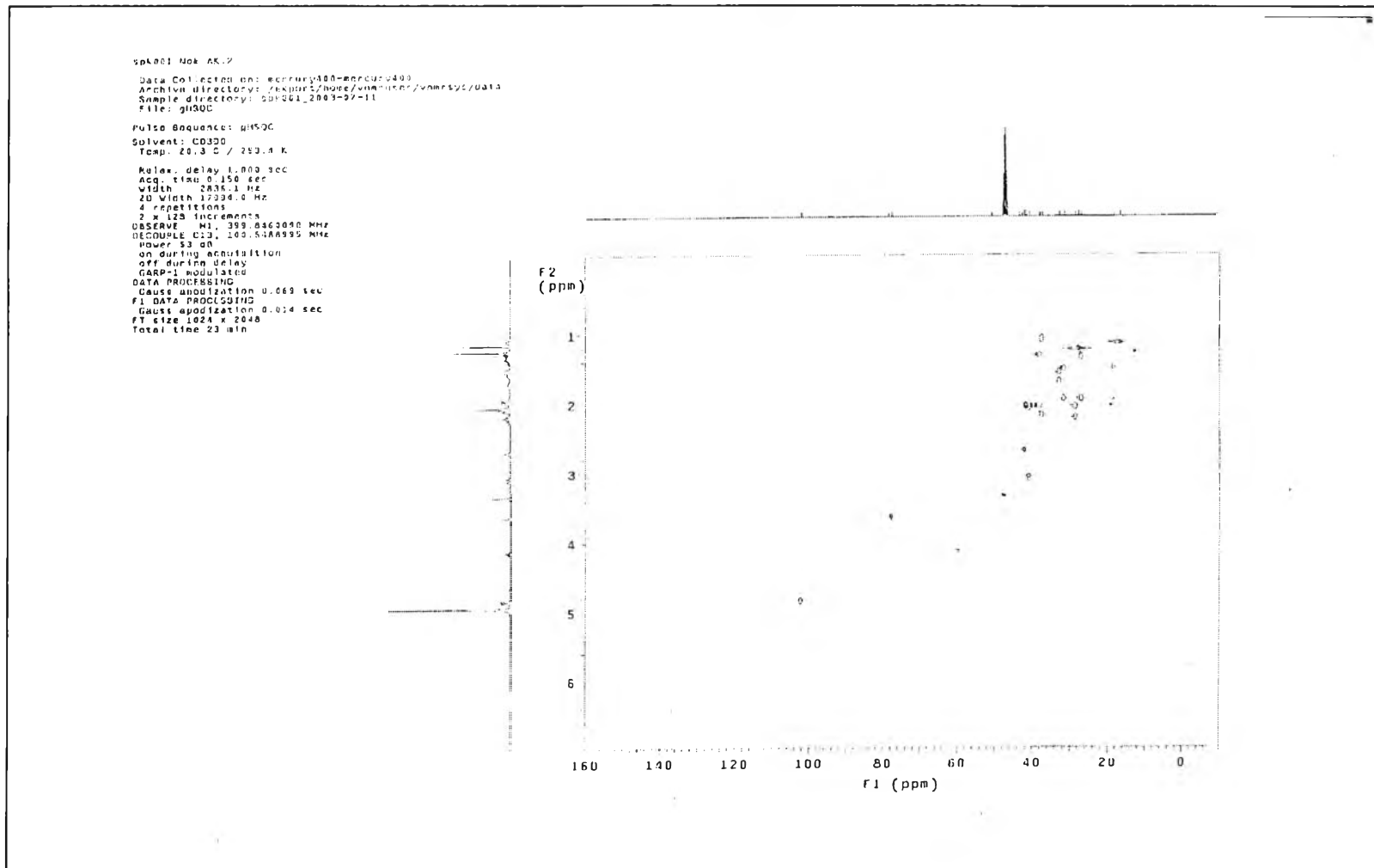


Figure B23. The gHSQC spectrum of Metabolite 1a.

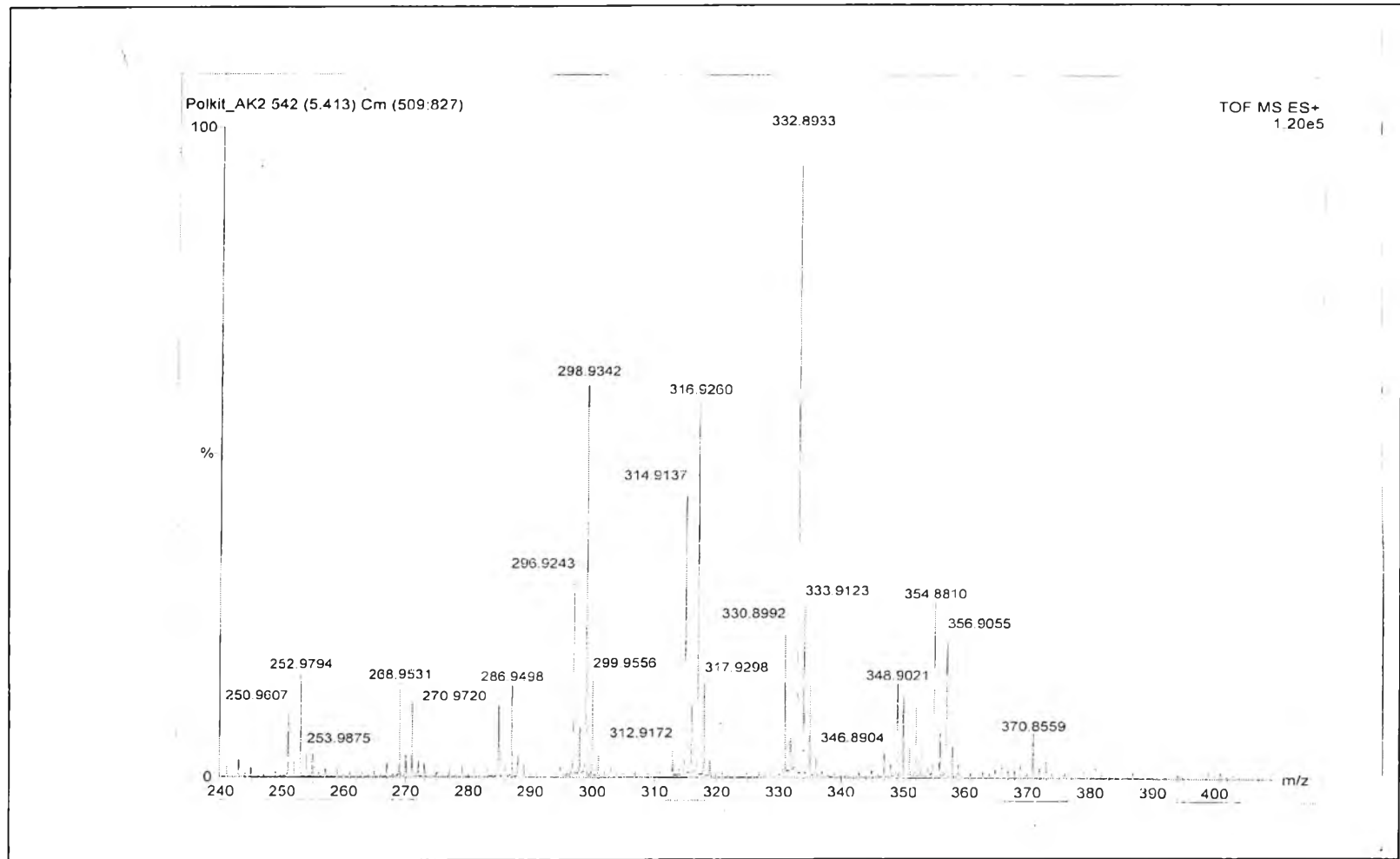


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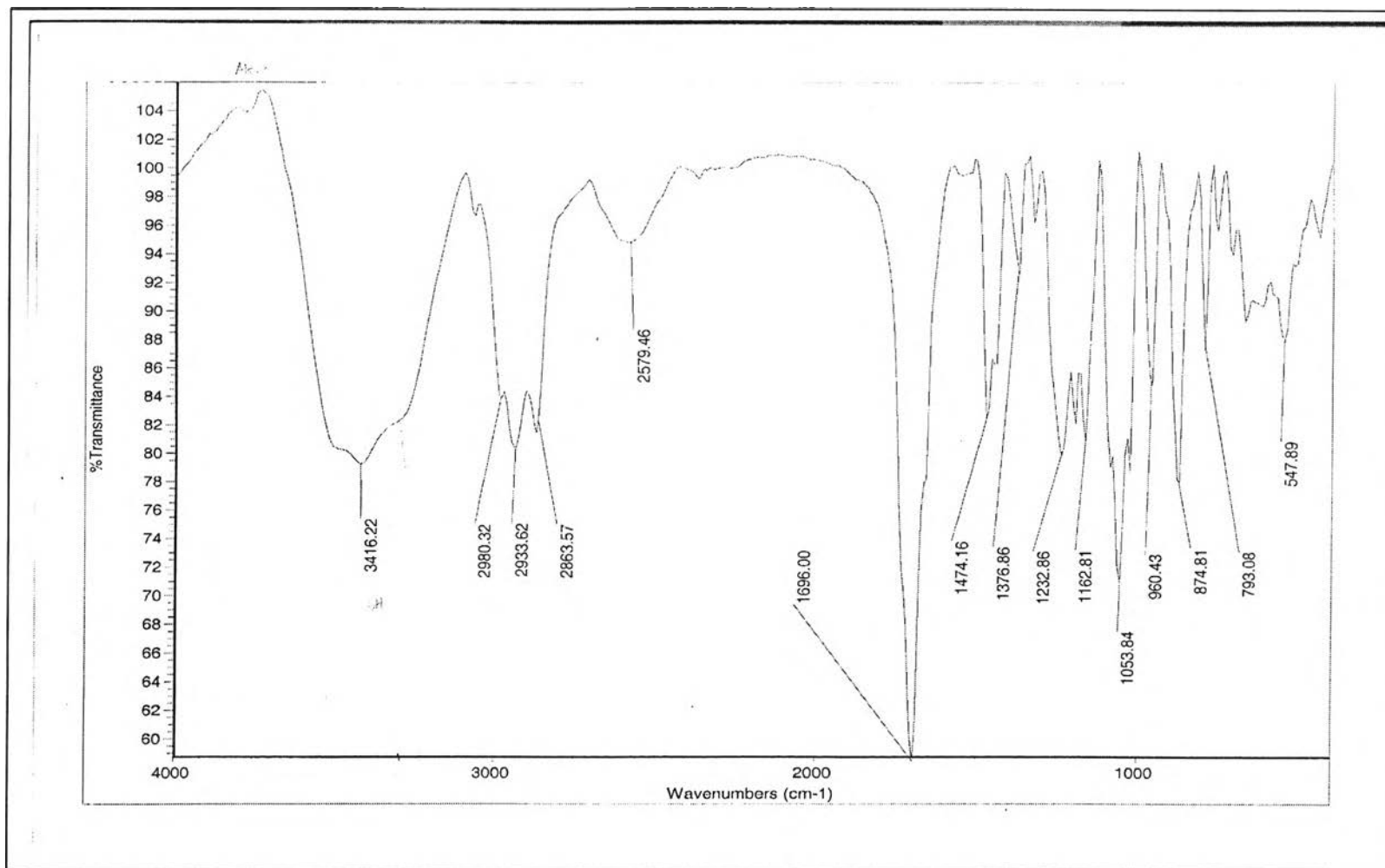


Figure B25. The IR spectrum of Metabolite **1b**.

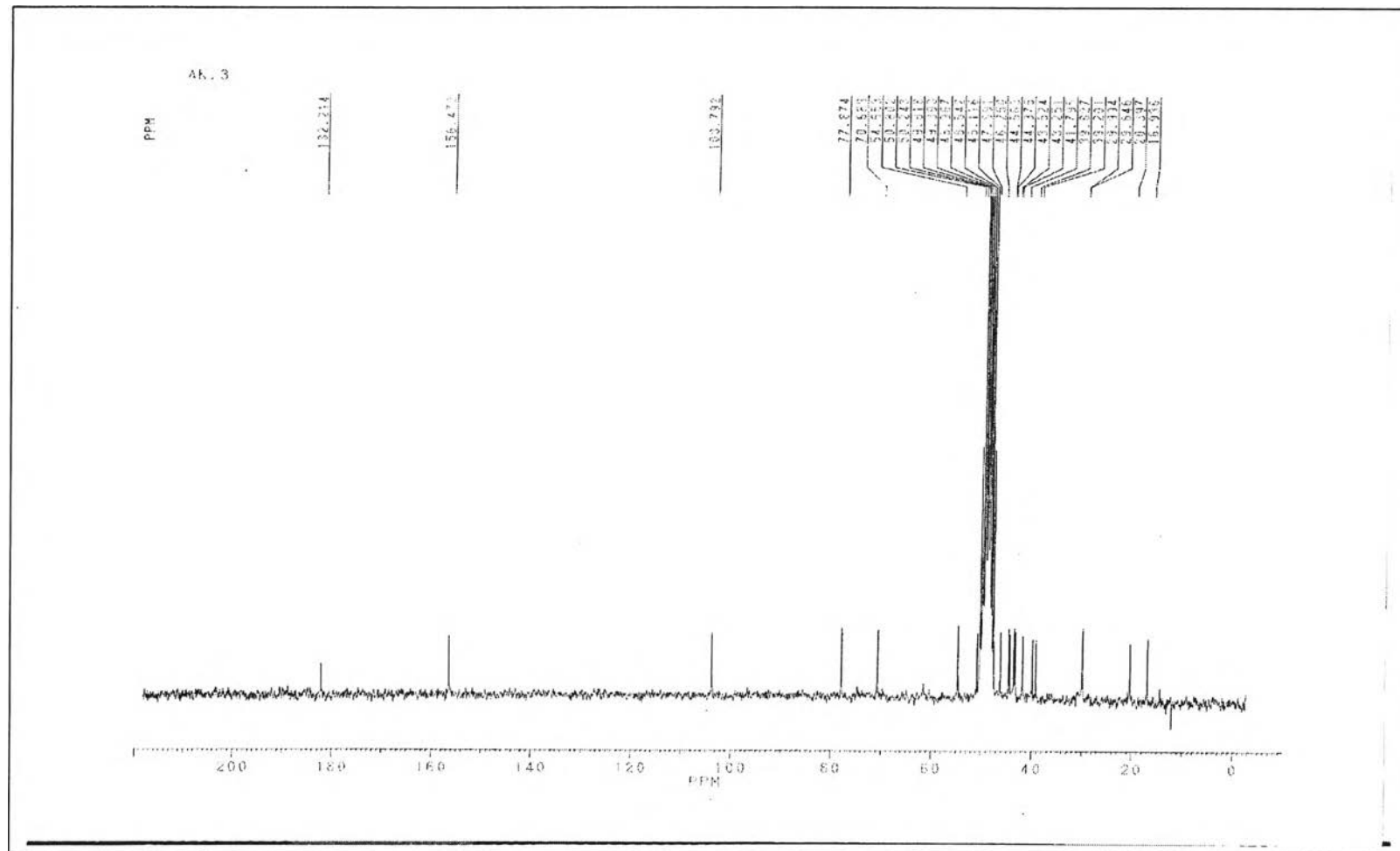


Figure B27. The ^{13}C -NMR spectrum of Metabolite **1b**.

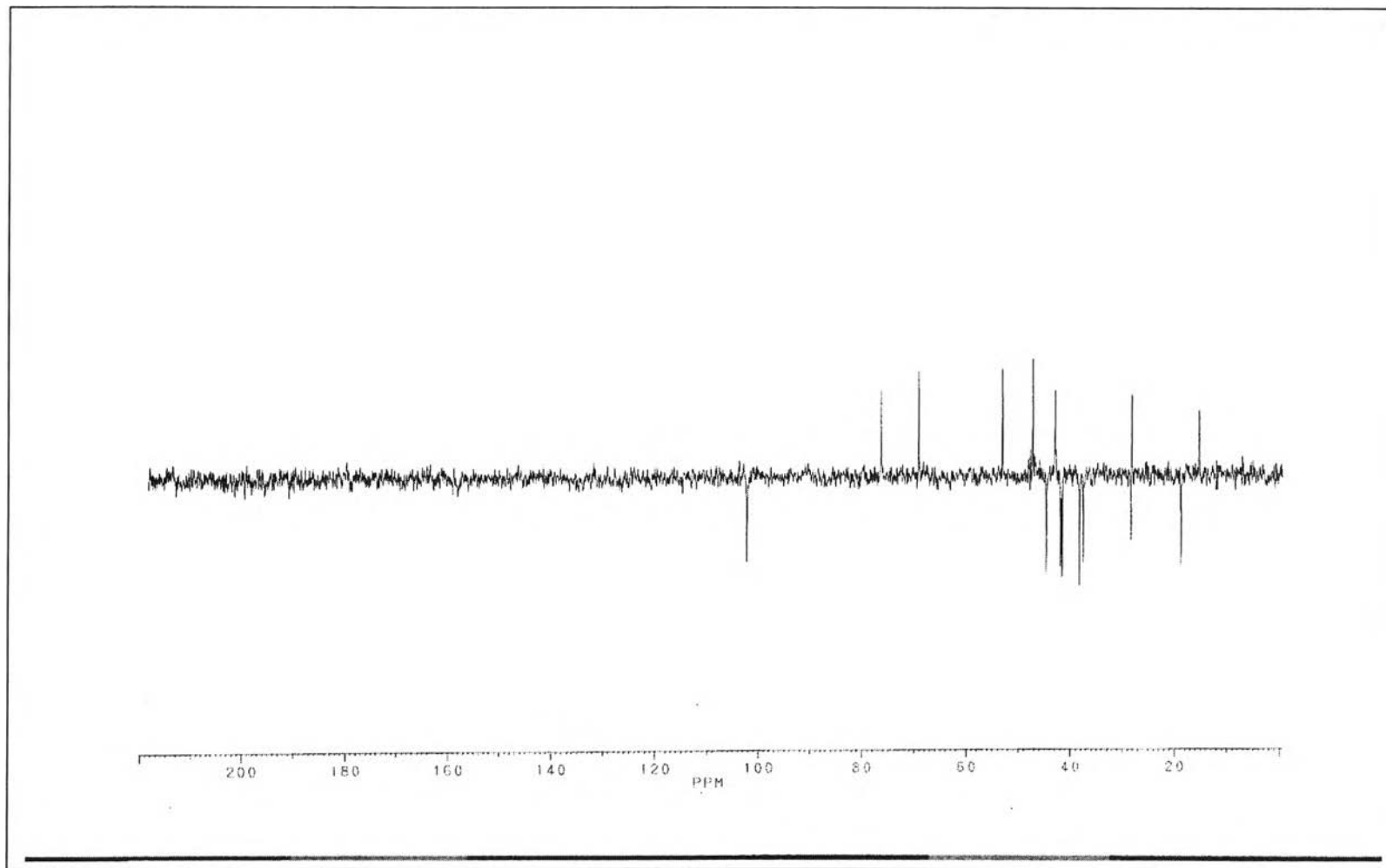


Figure B28. The DEPT spectrum of Metabolite **1b**.

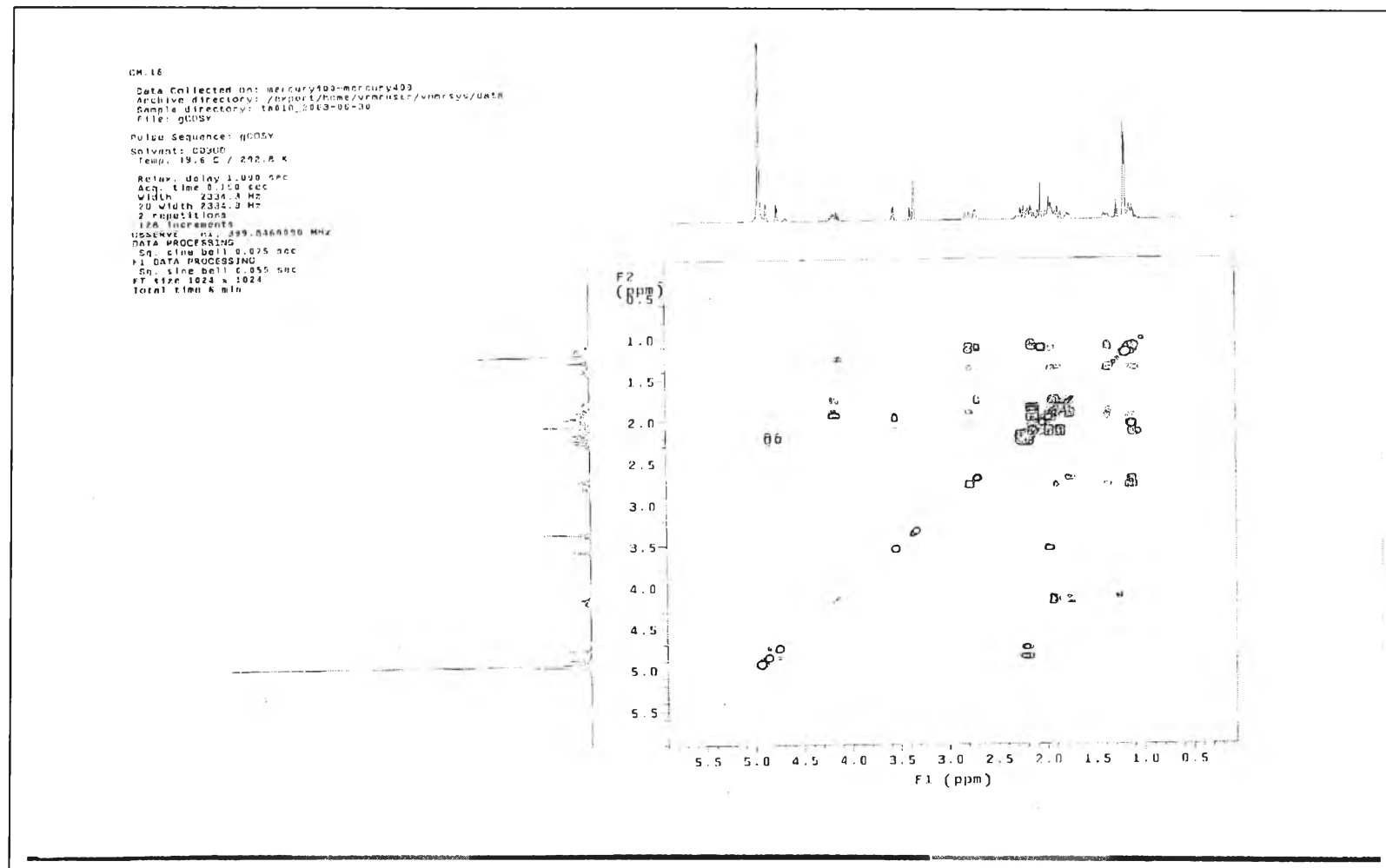


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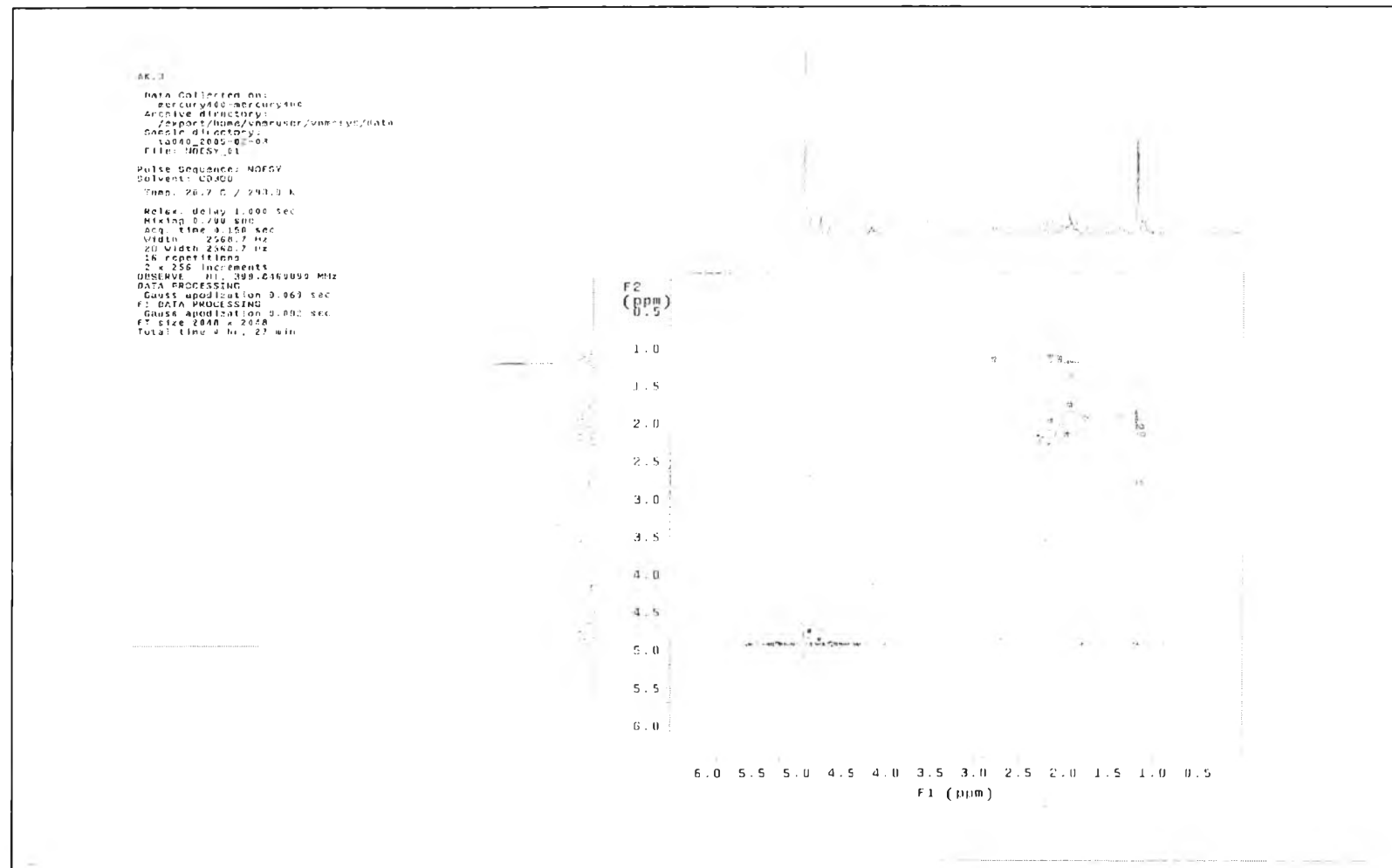


Figure B30. The gNOESY spectrum of Metabolite 1b.

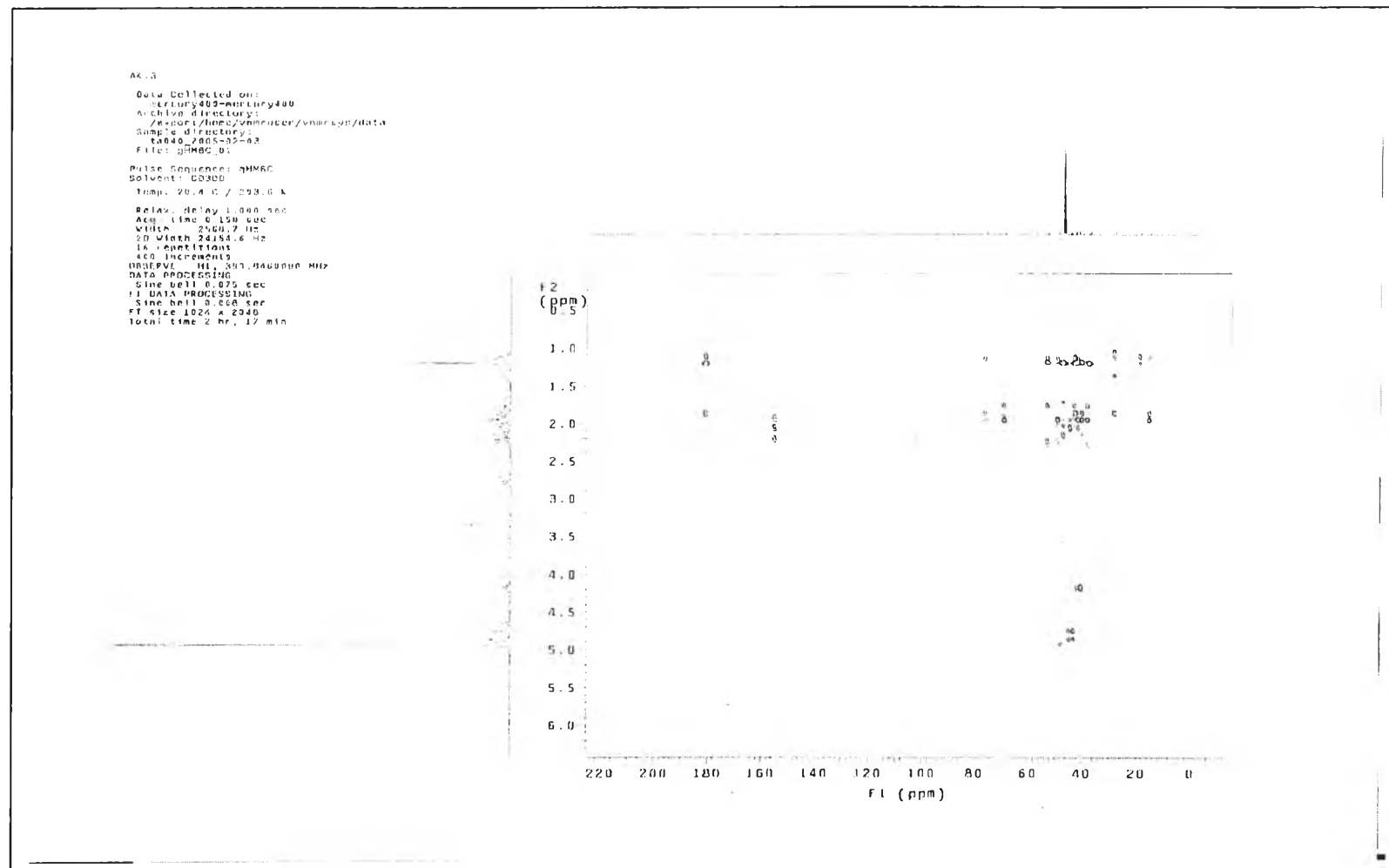


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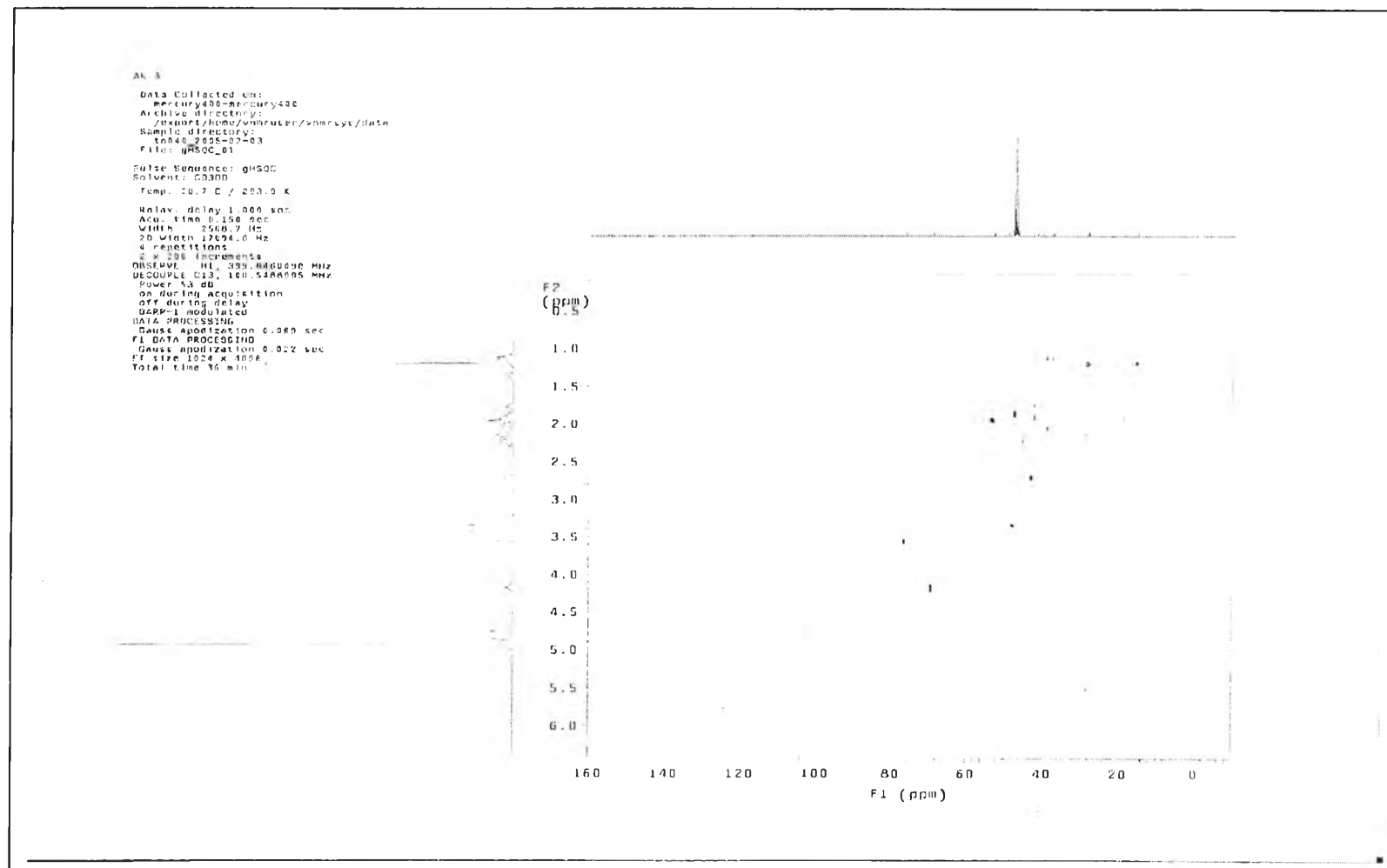


Figure B32. The gHSQC spectrum of Metabolite **1b**.

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small

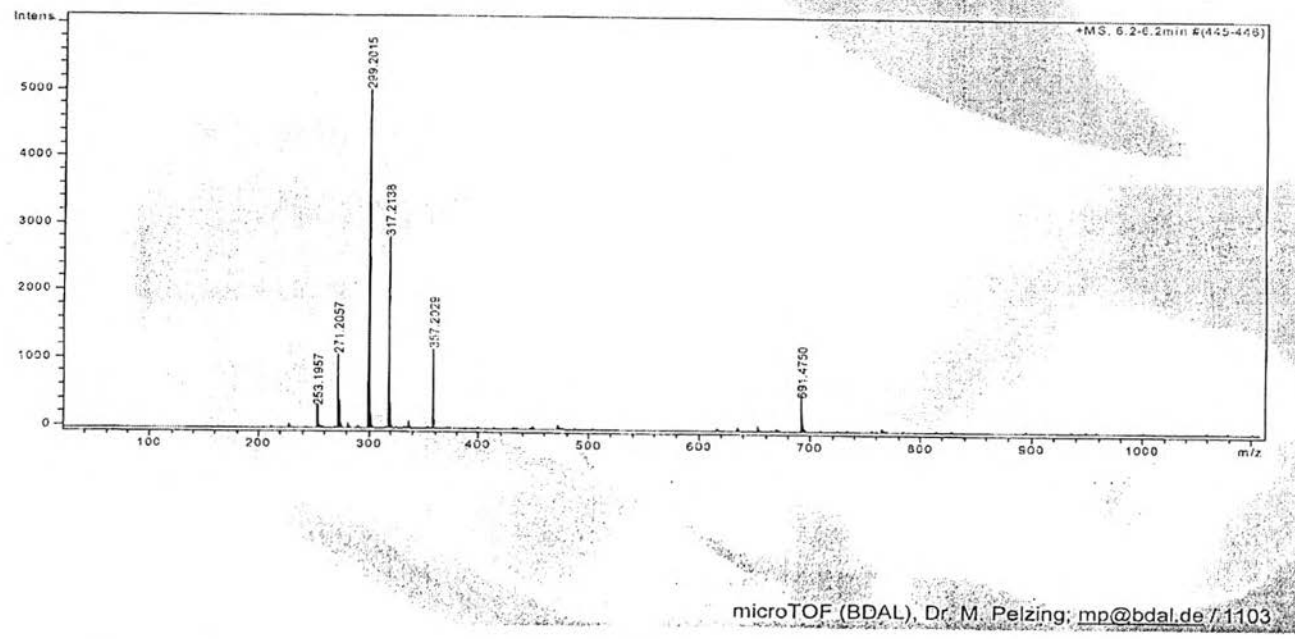


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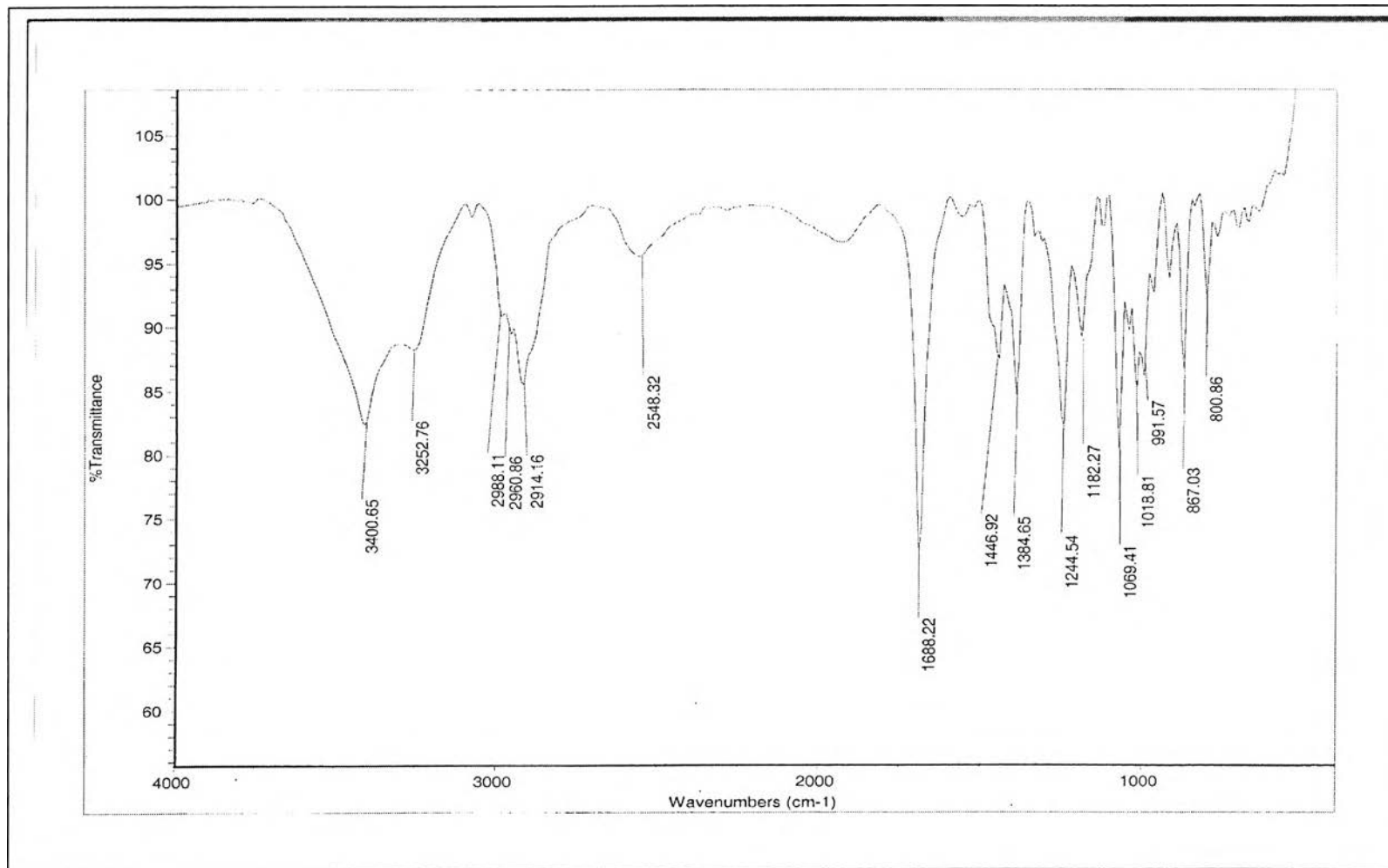


Figure B34. The IR spectrum of Metabolite 1c.

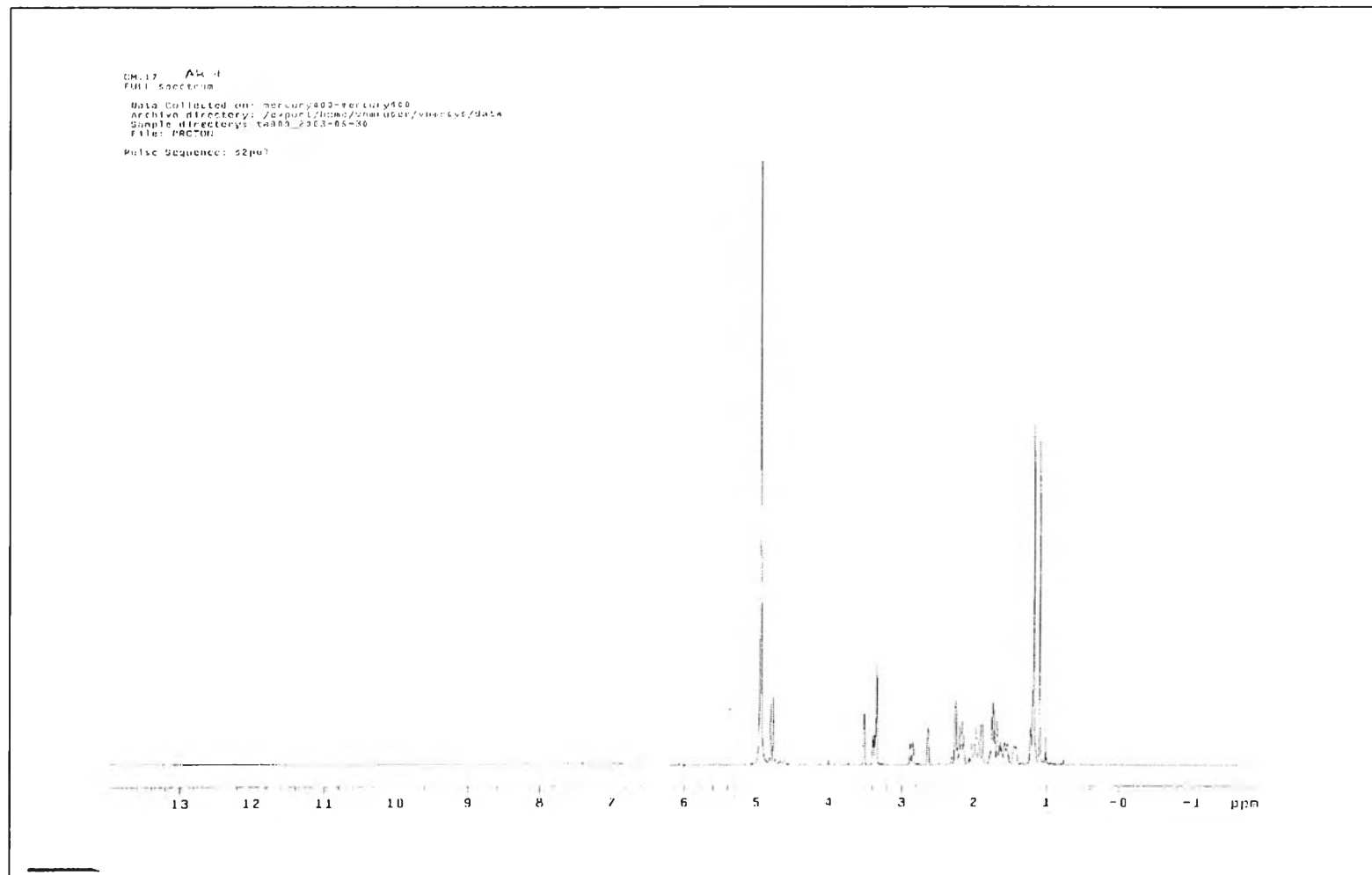


Figure B35. The ^1H -NMR spectrum of Metabolite 1c.

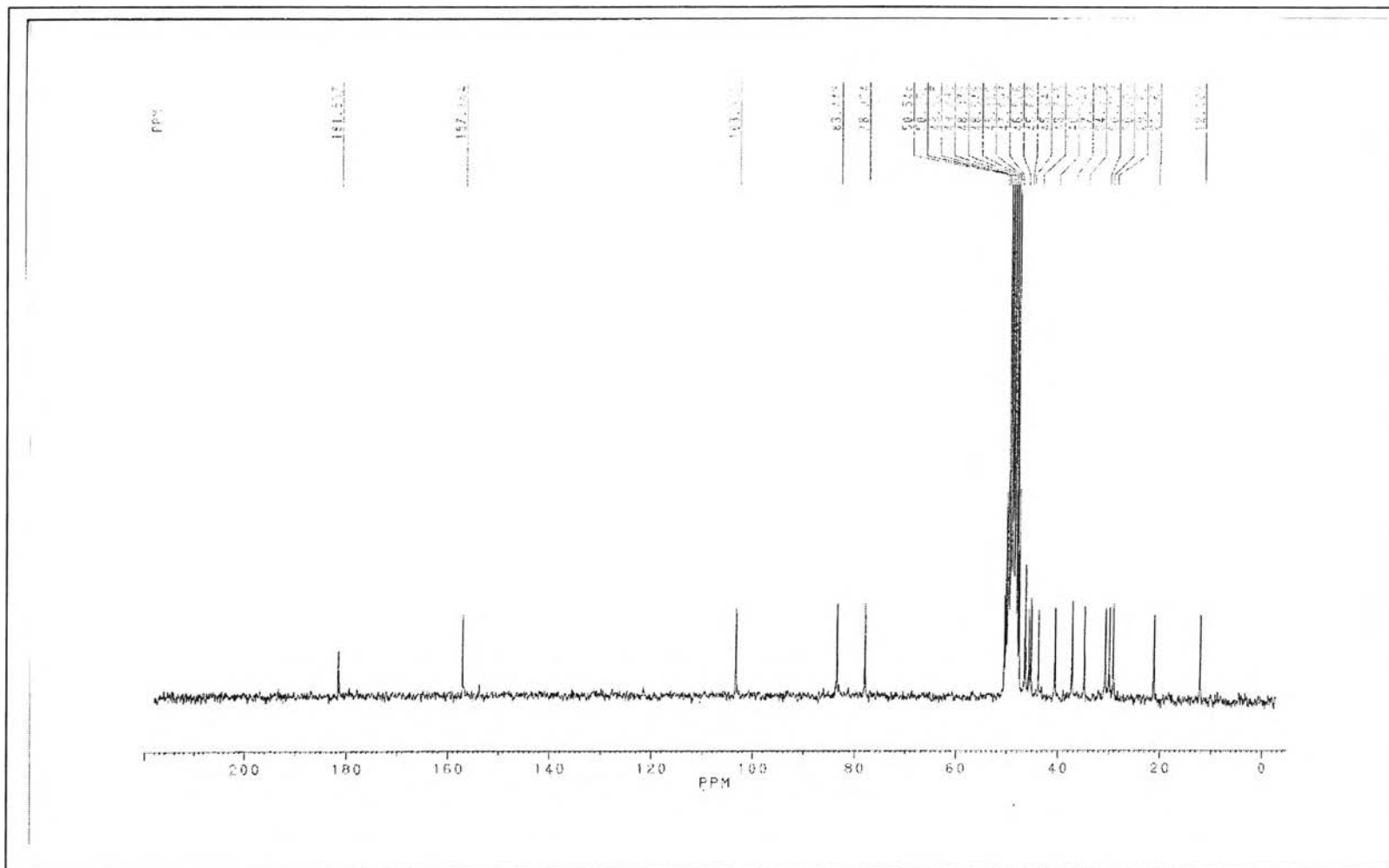


Figure B36. The ^{13}C -NMR spectrum of Metabolite **1c**.

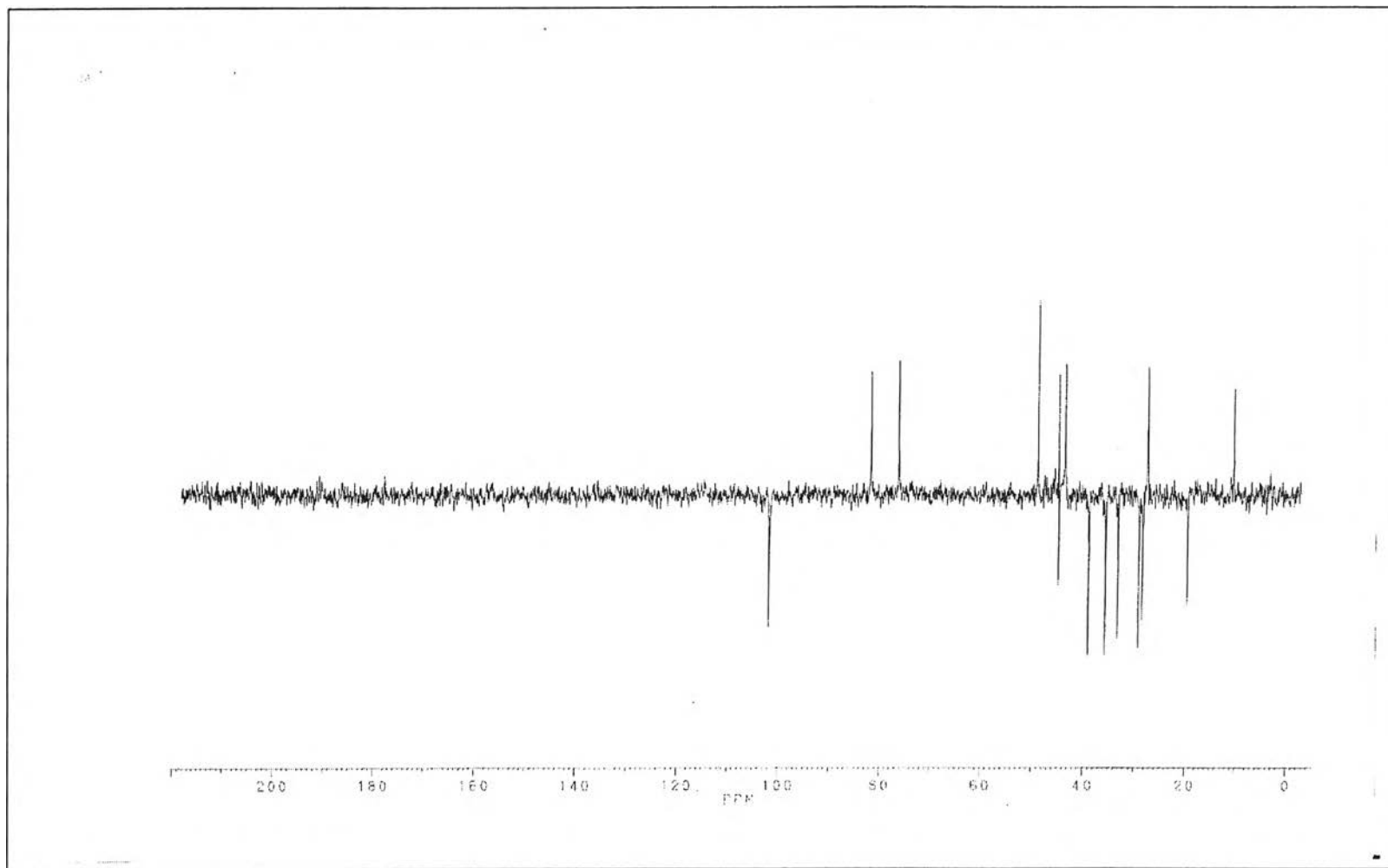


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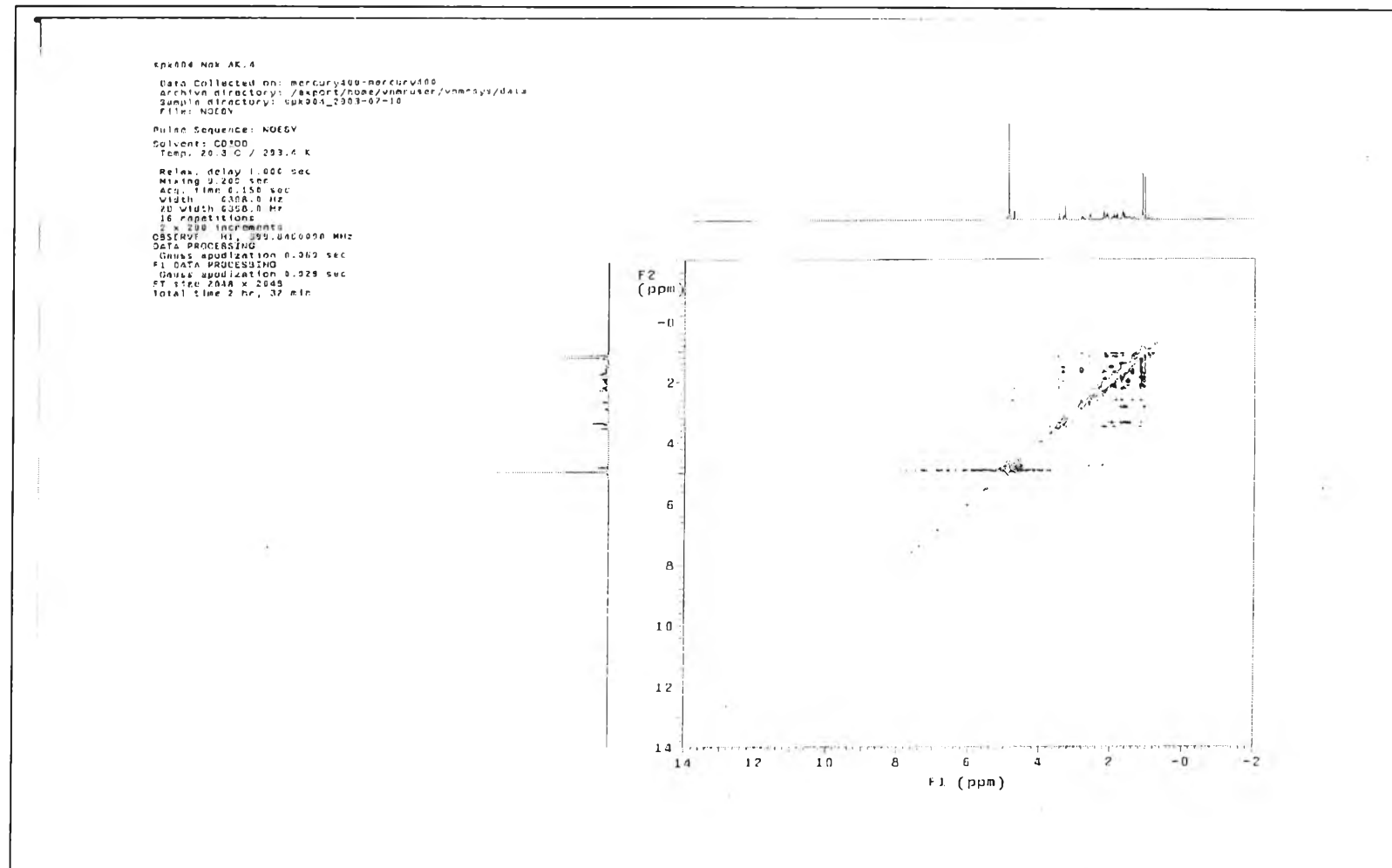


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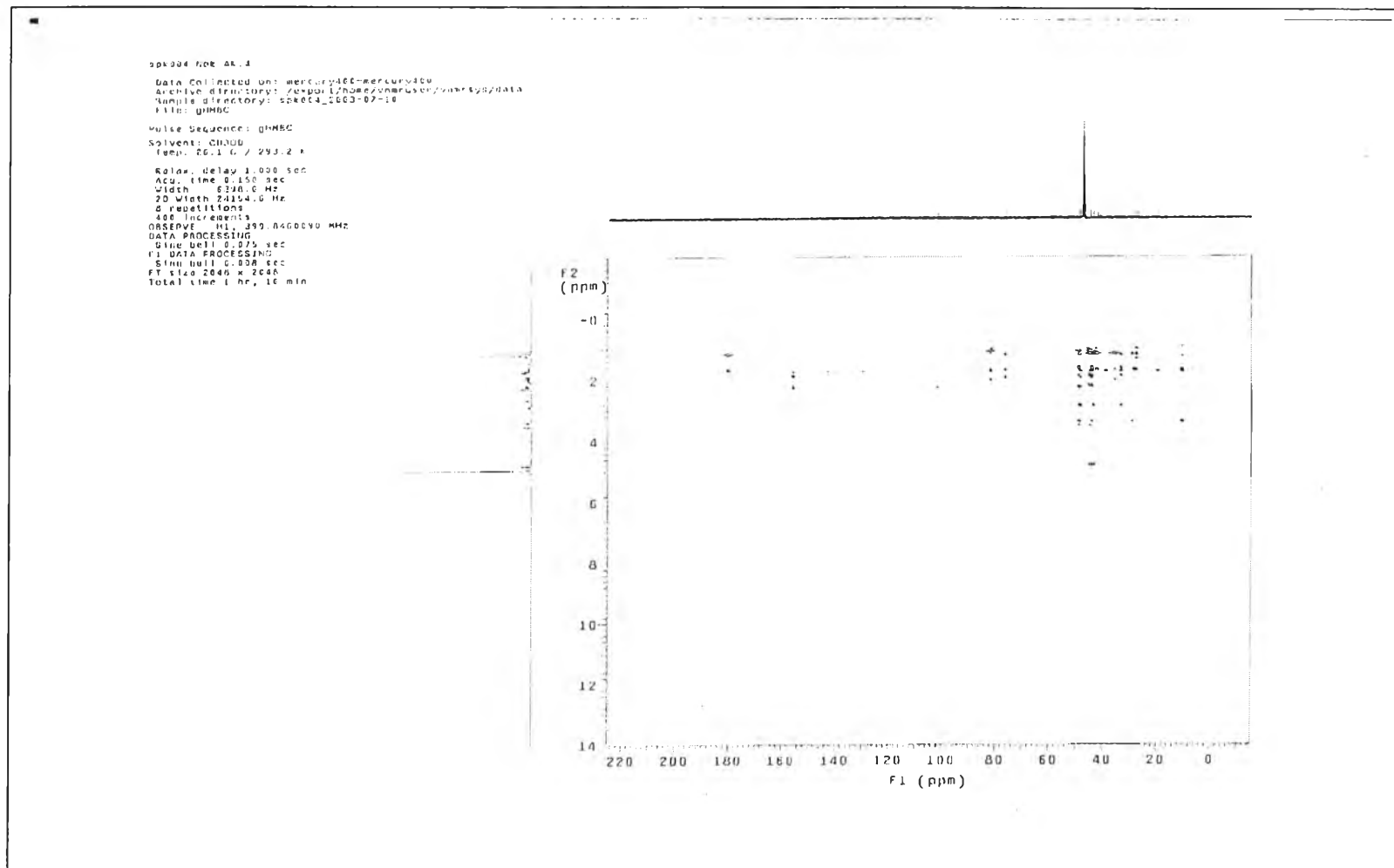


Figure B40. The gHMBC spectrum of Metabolite 1c.

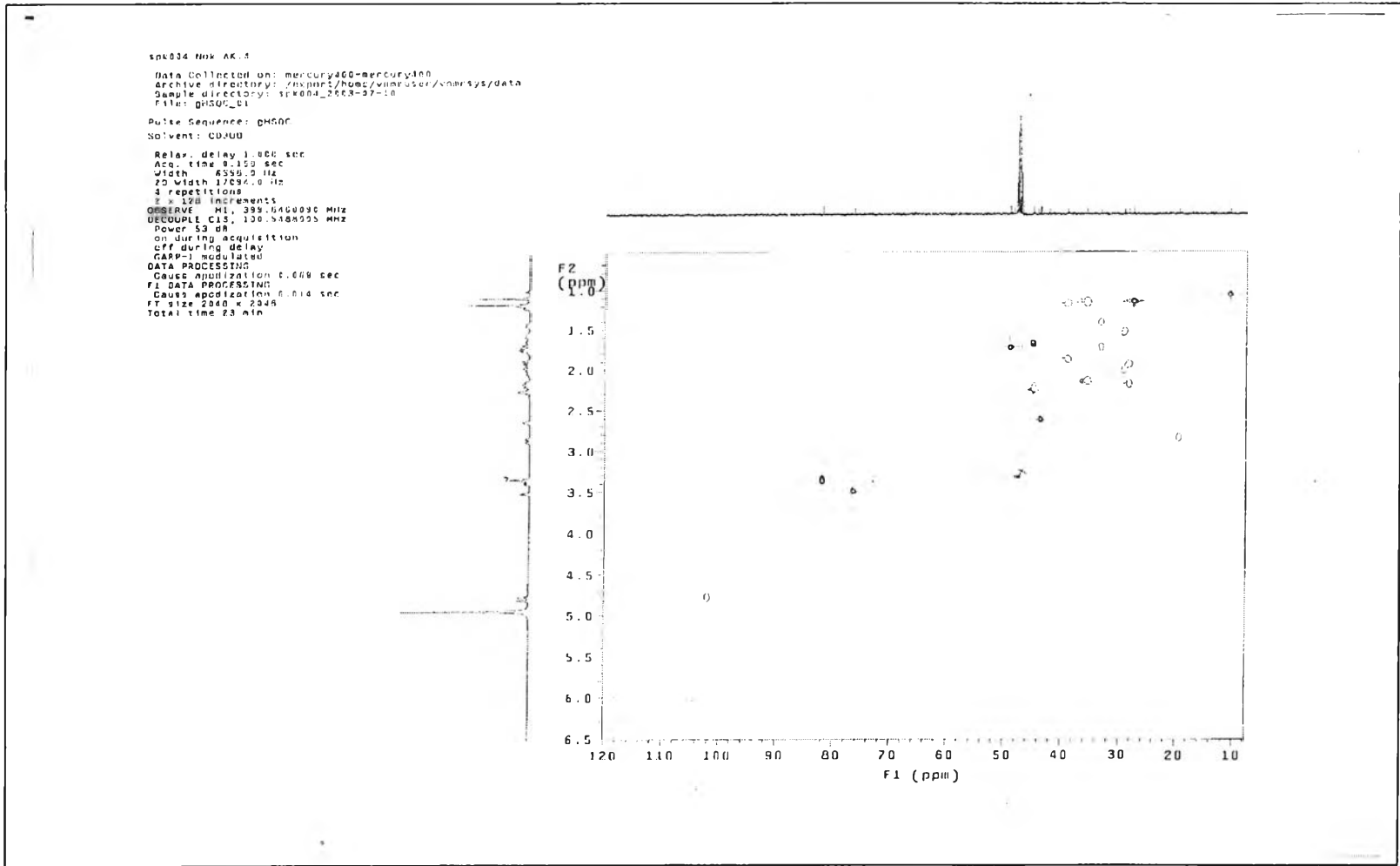


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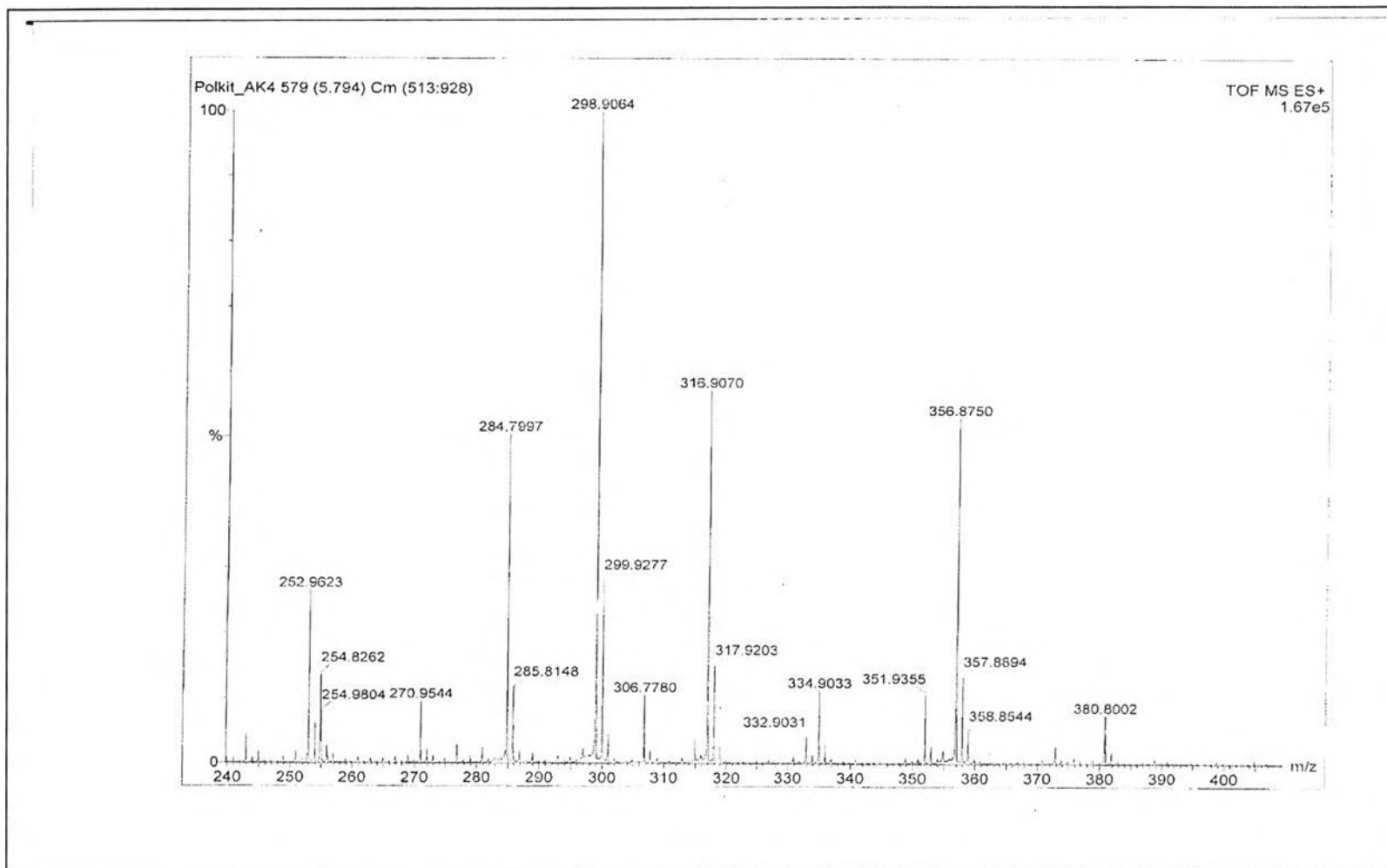


Figure B42. The LC-MS spectrum of Metabolite 1c.

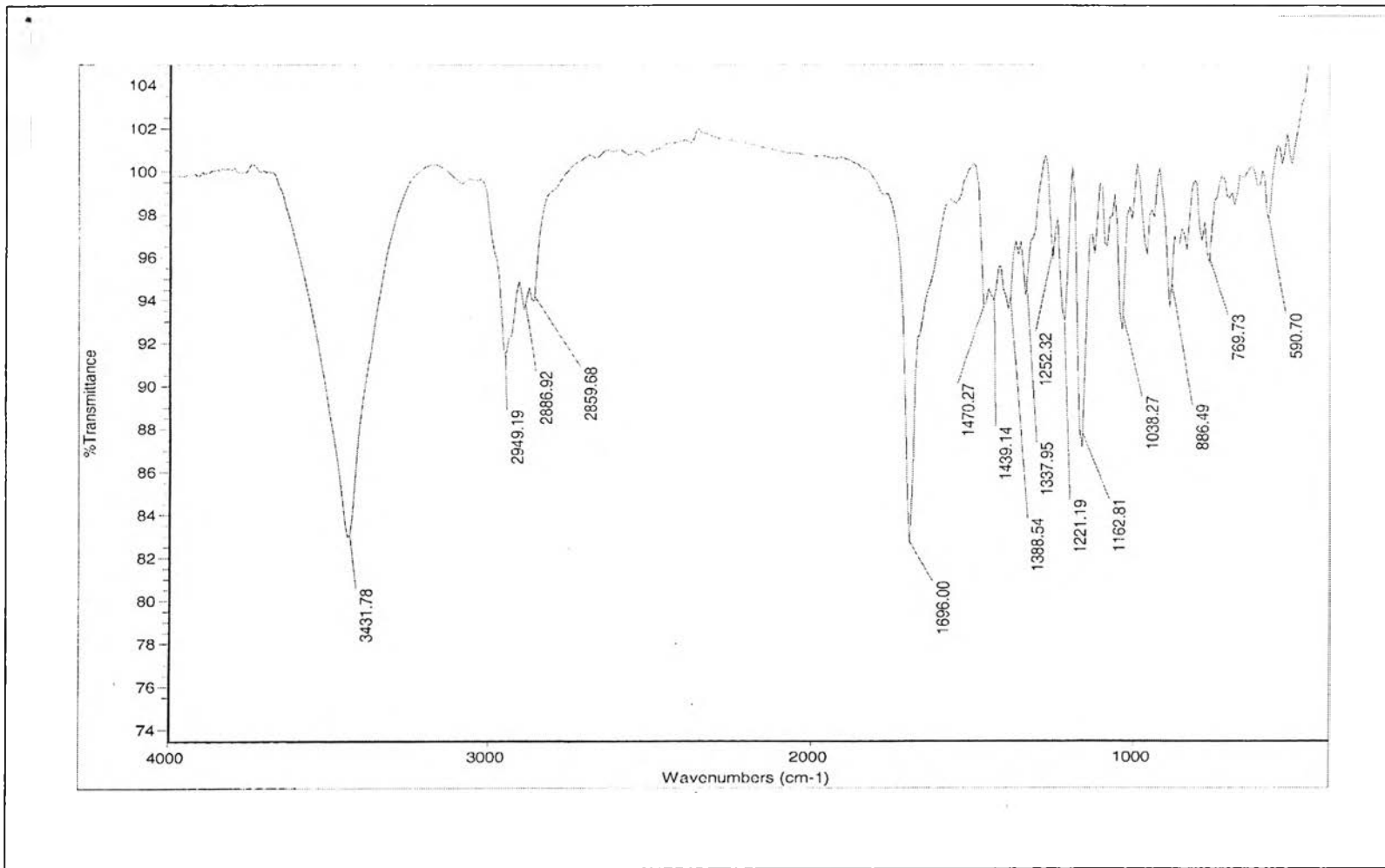


Figure B43. The IR spectrum of Metabolite 1d.

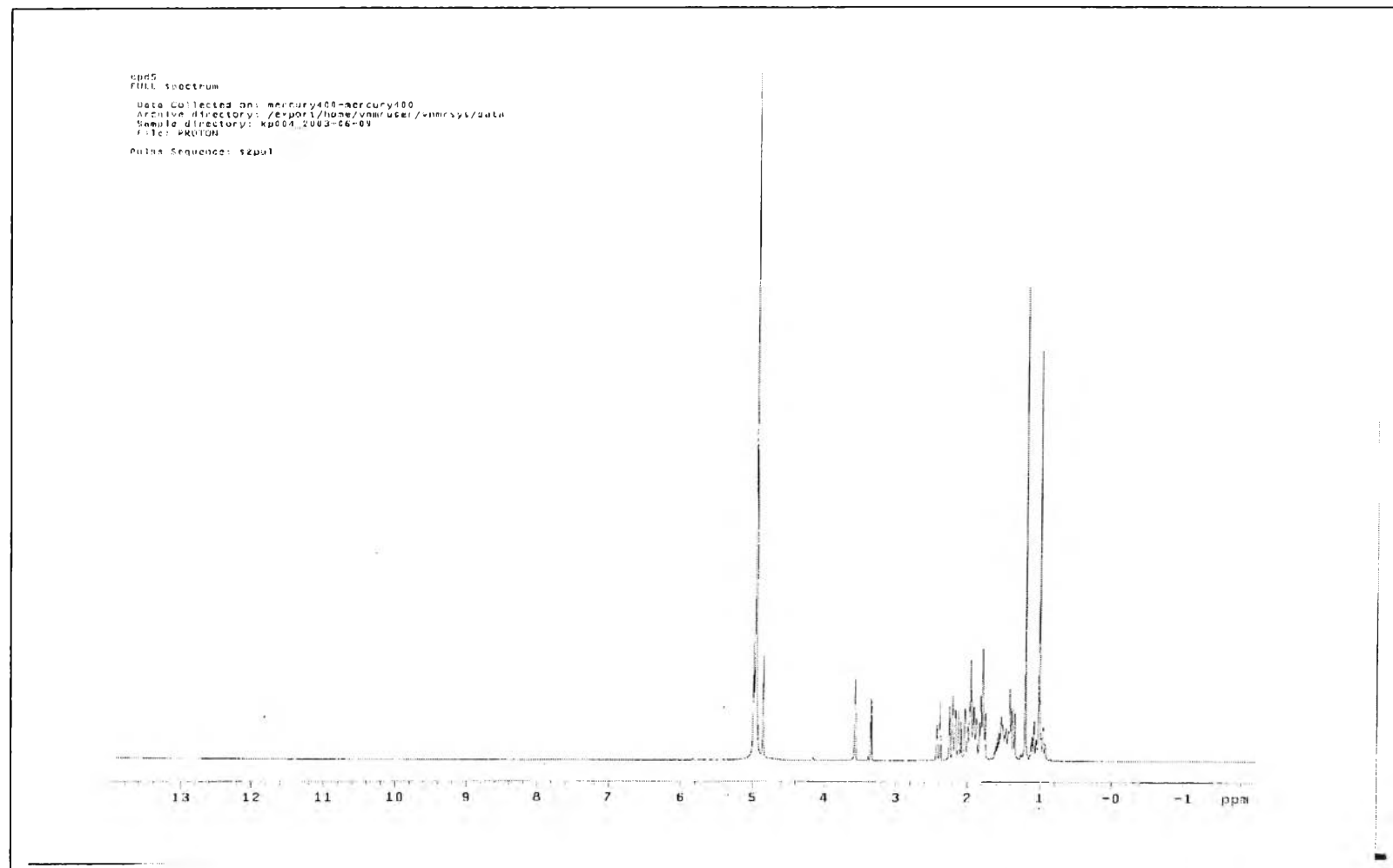


Figure B44. The ^1H -NMR spectrum of Metabolite **1d**.

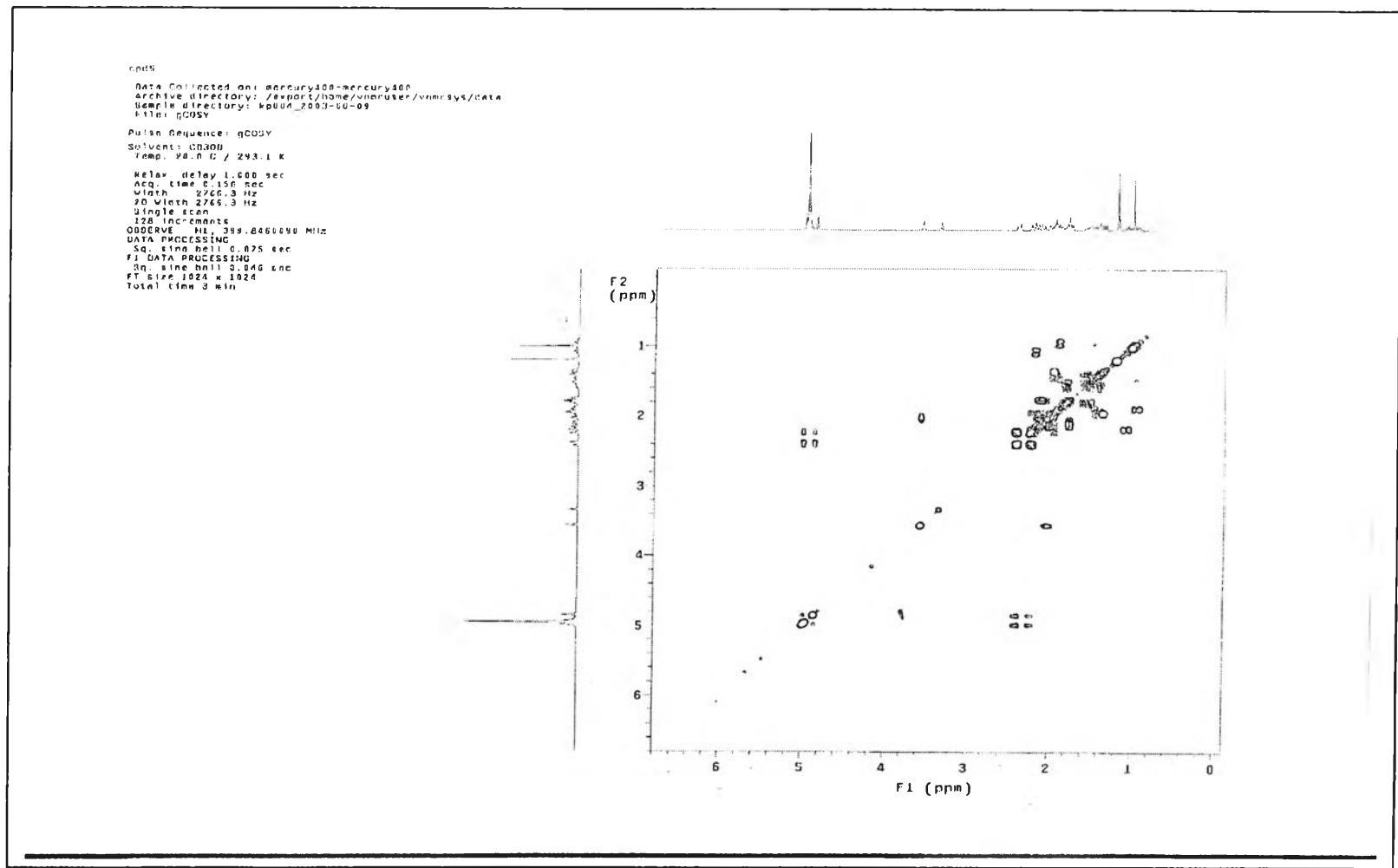


Figure B46. The gCOSY spectrum of Metabolite **1d**.

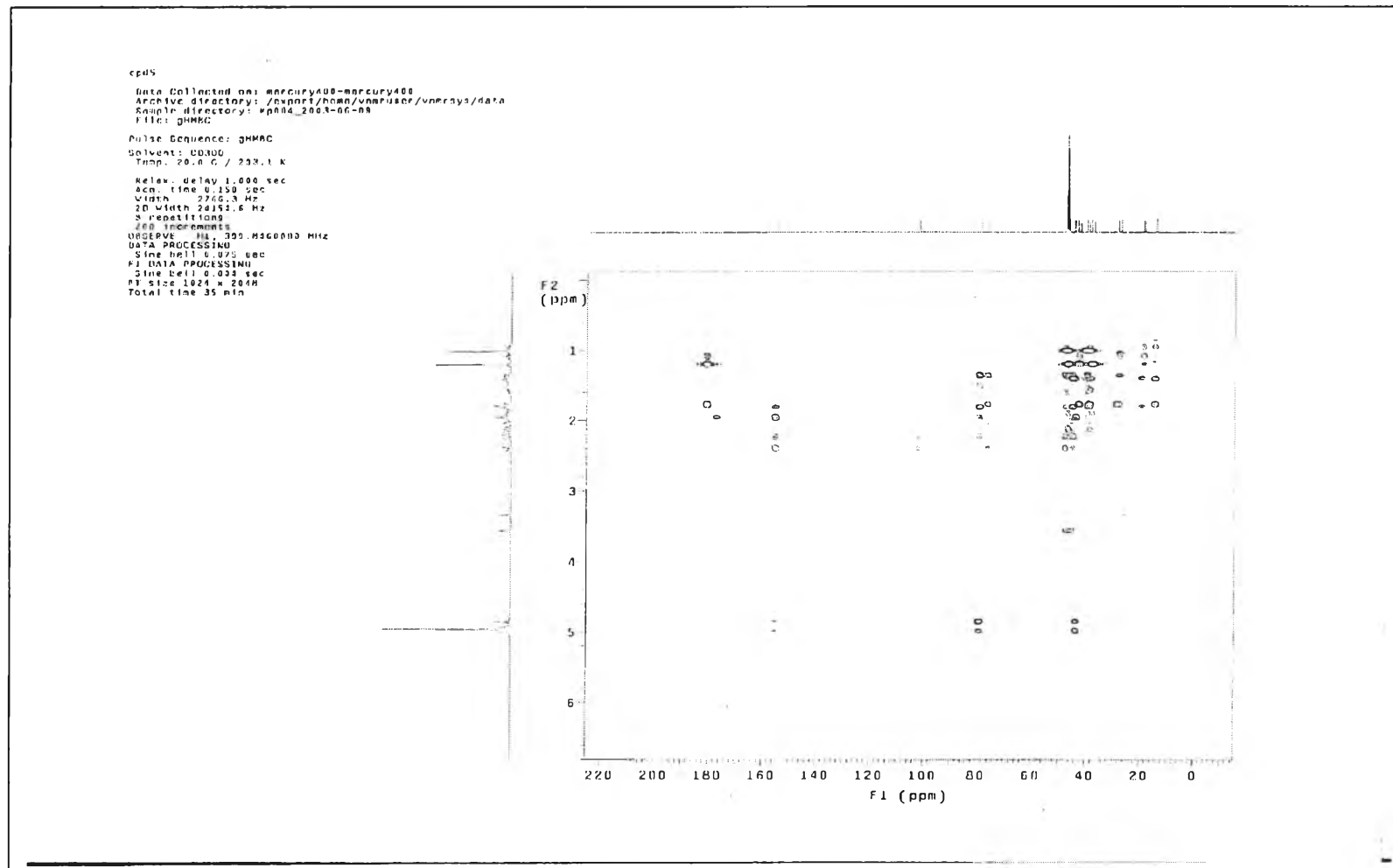


Figure B47. The gHMBC spectrum of Metabolite 1d.

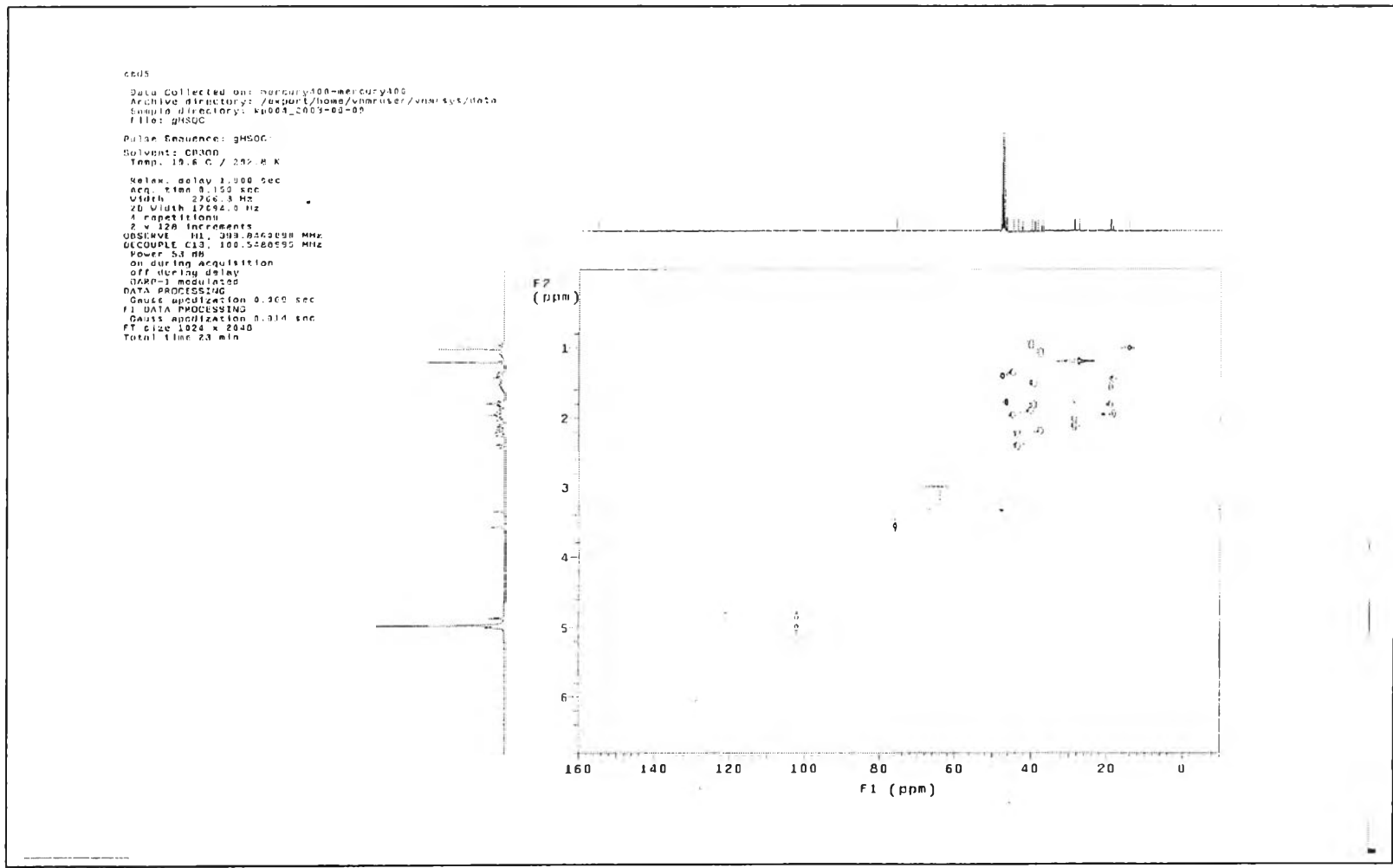


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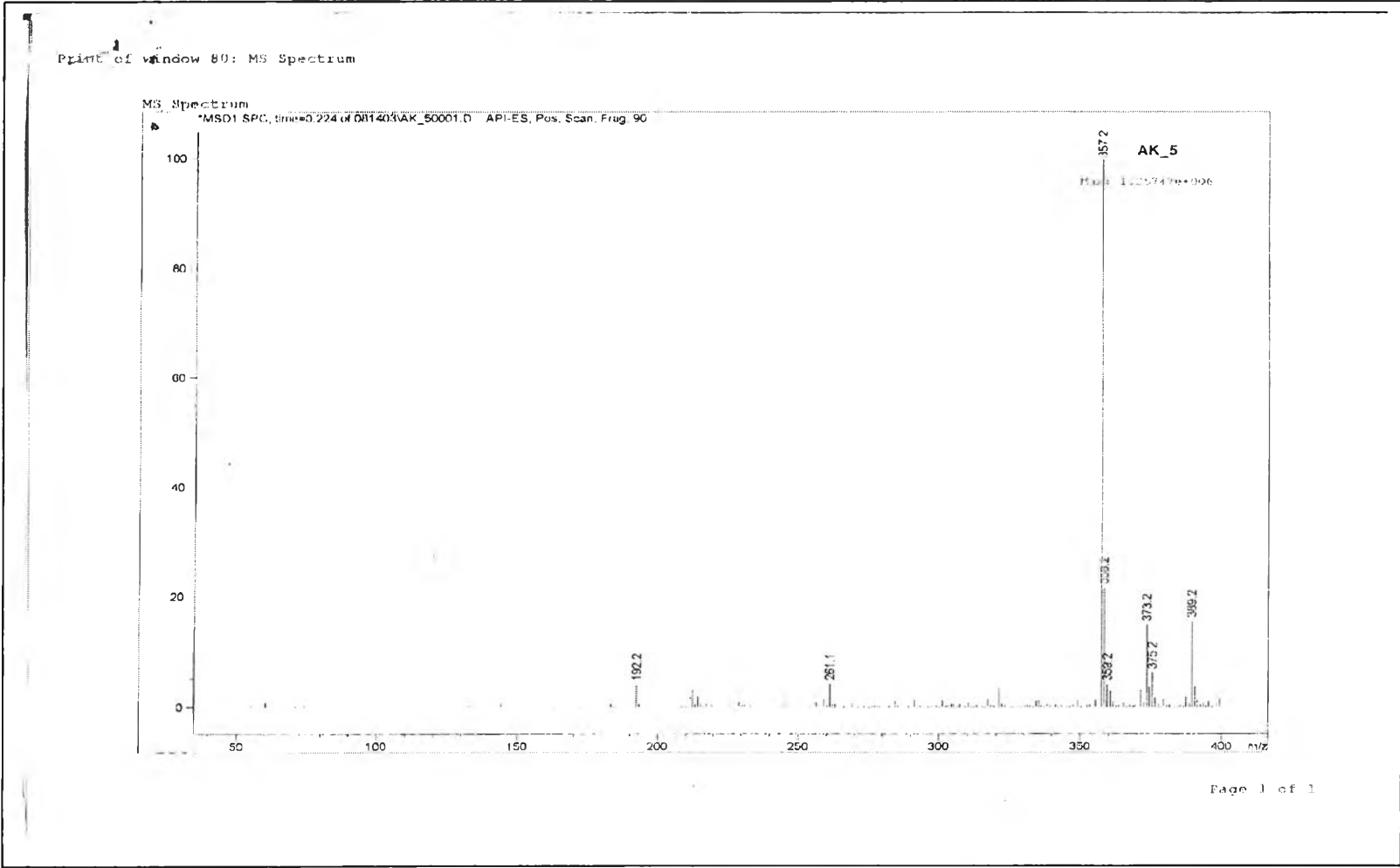


Figure B49. The LC-MS spectrum of Metabolite 1d.

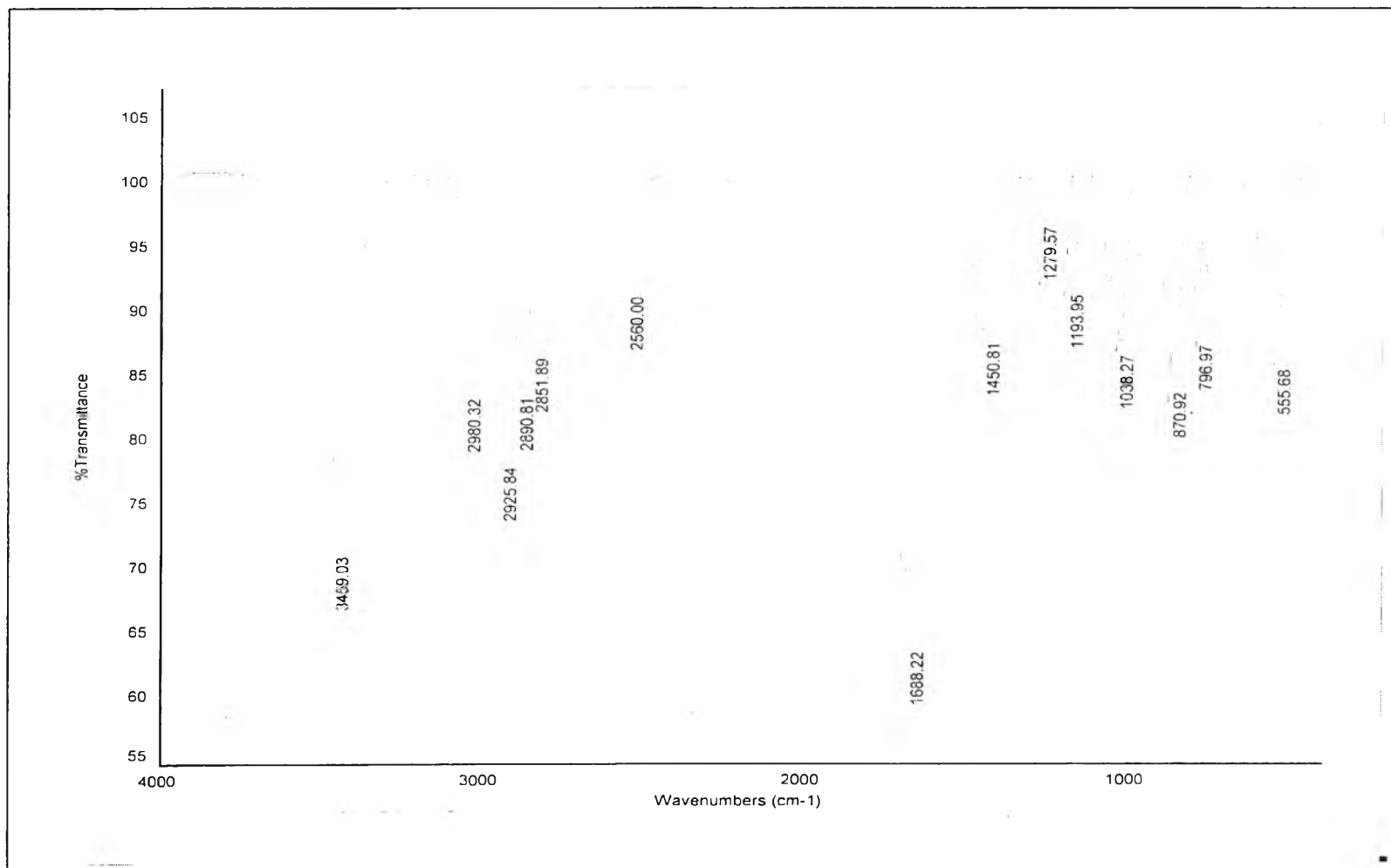


Figure B50. The IR spectrum of Metabolite **1e**.

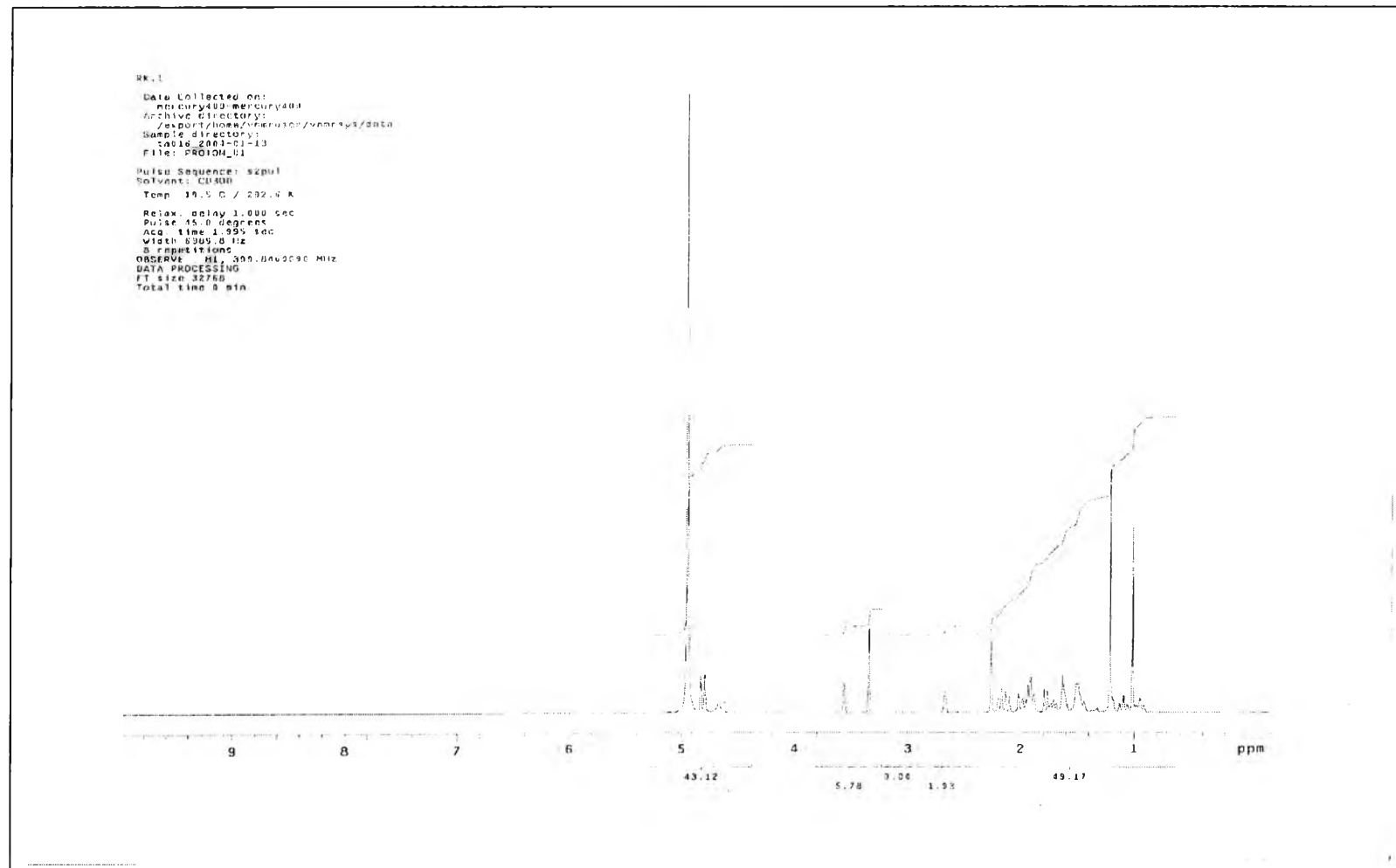


Figure B51. The ^1H -NMR spectrum of Metabolite 1e.

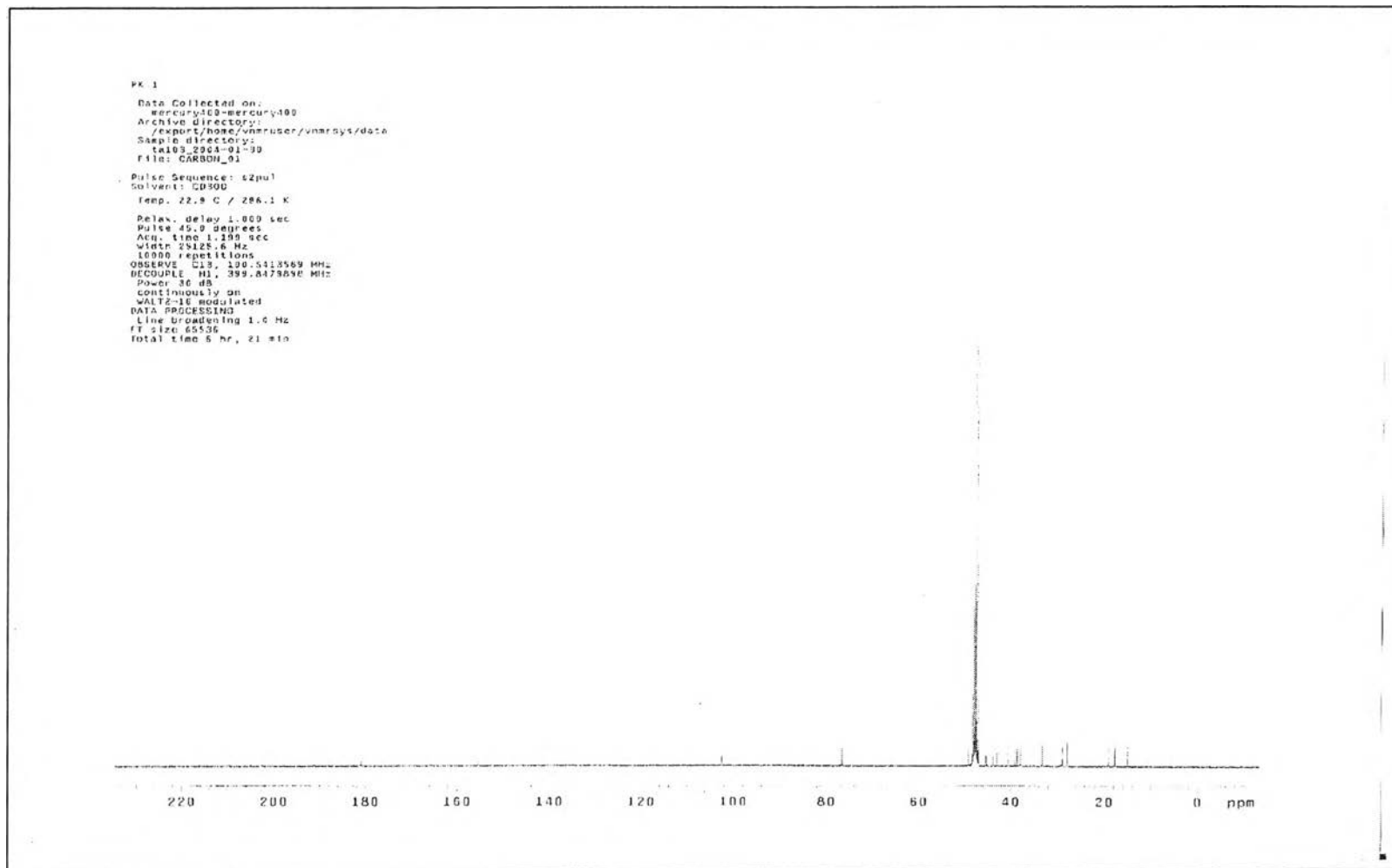


Figure B52. The ¹³C-NMR spectrum of Metabolite 1e.

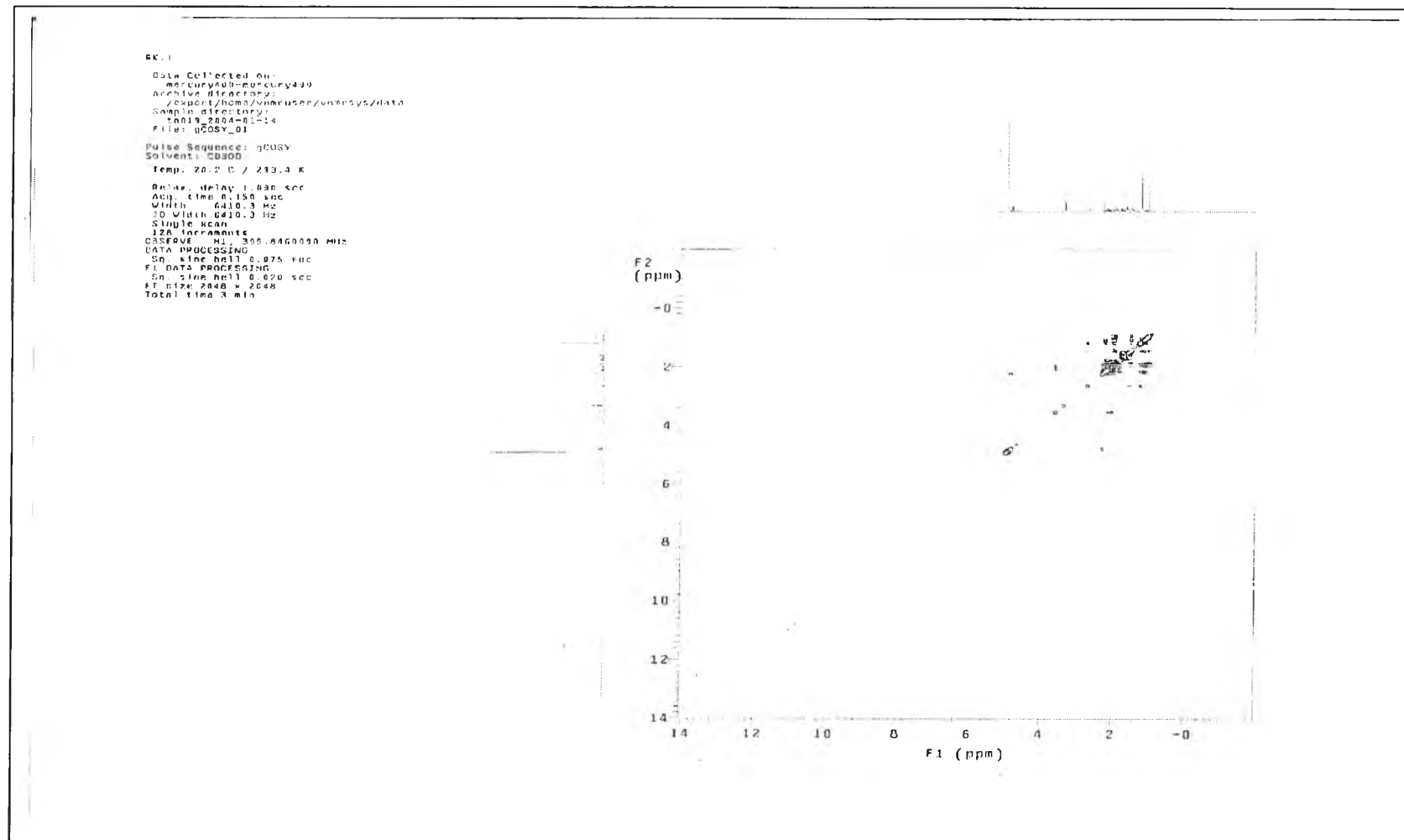


Figure D53. The gCOSY spectrum of Metabolite **1e**.

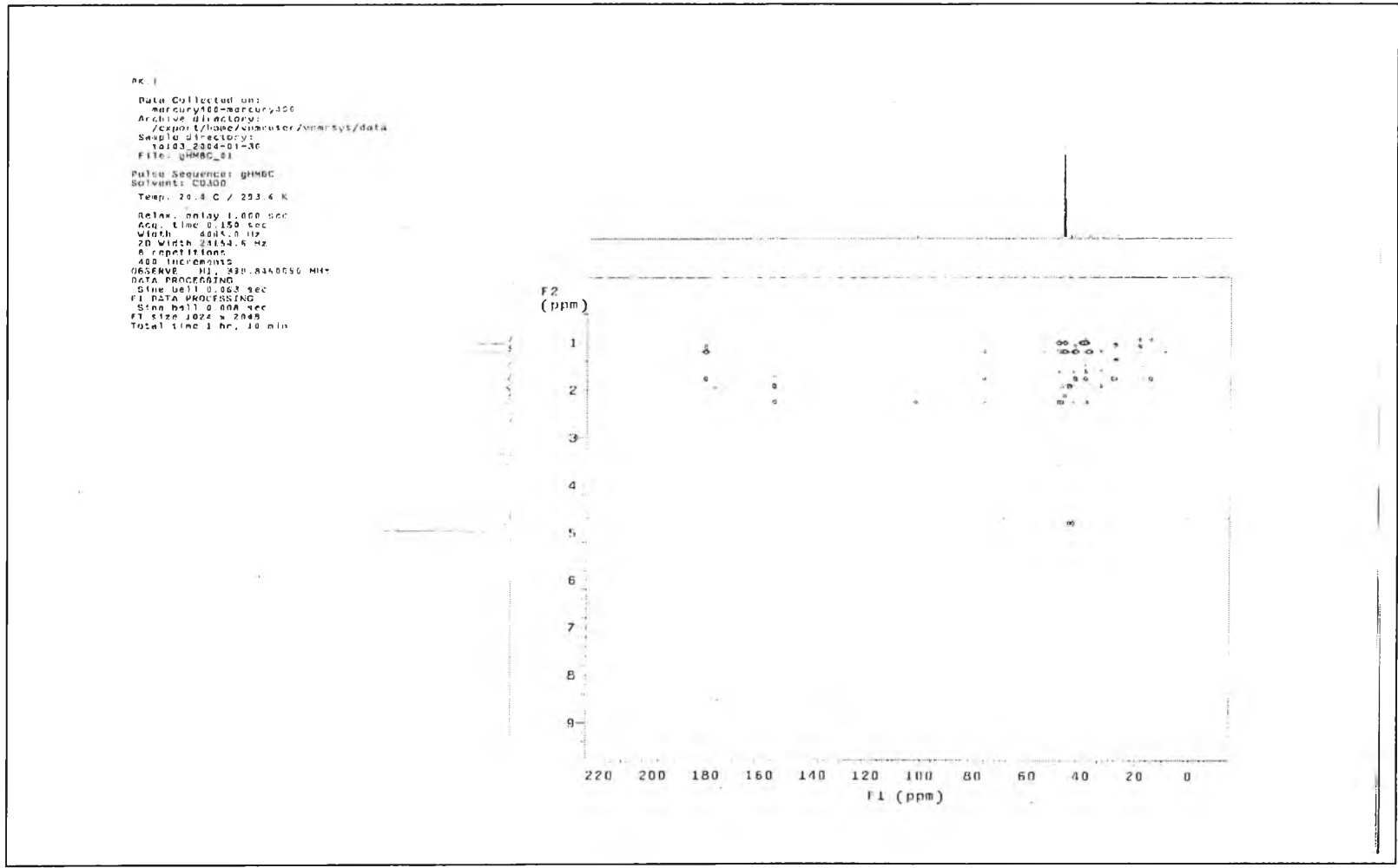


Figure B54. The HMBC spectrum of Metabolite **1e**.

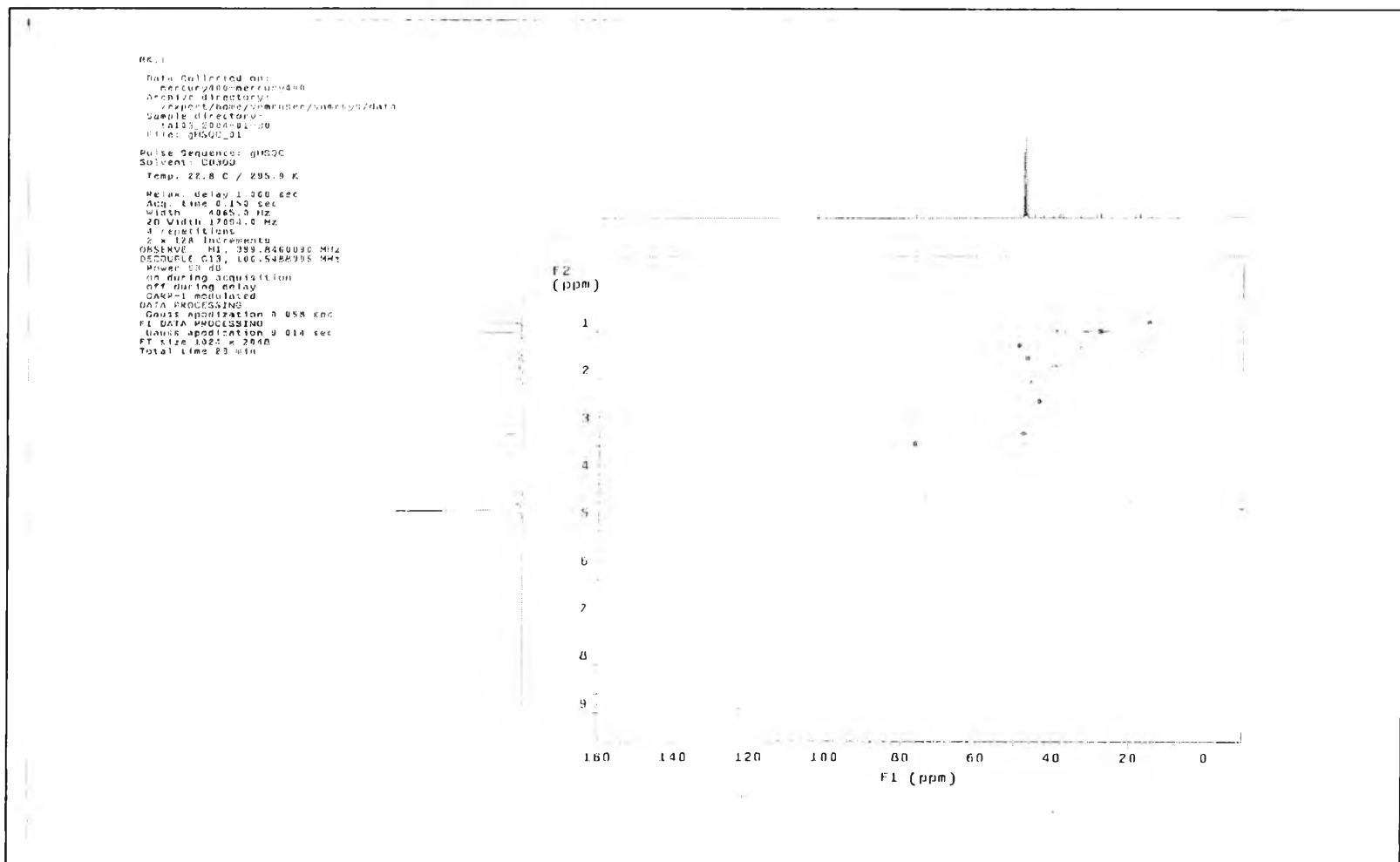
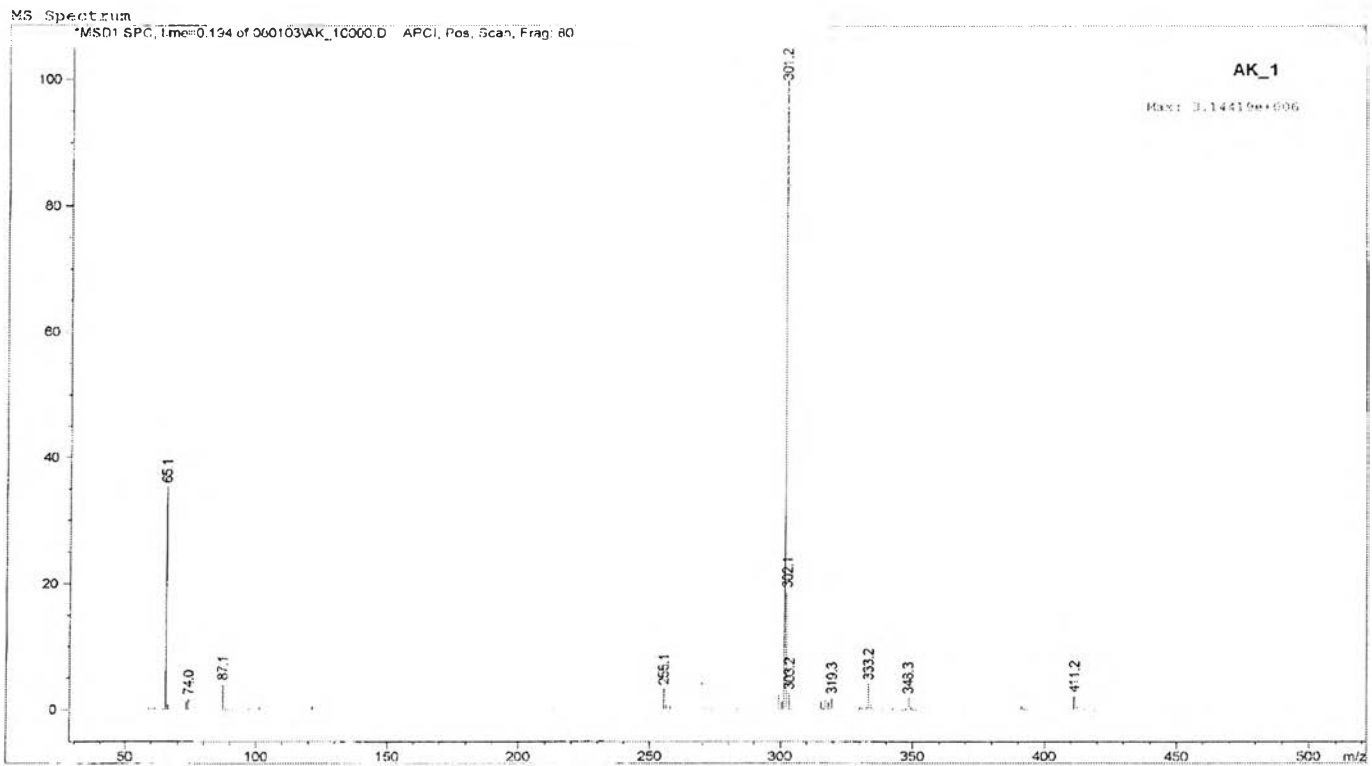


Figure B55. The gHSQC spectrum of Metabolite 1e.

Print of window 80: MS Spectrum



Instrument 1 8/1/2003 8:53:53 AM TA

Figure B56. The LC-MS spectrum of Metabolite 1e.

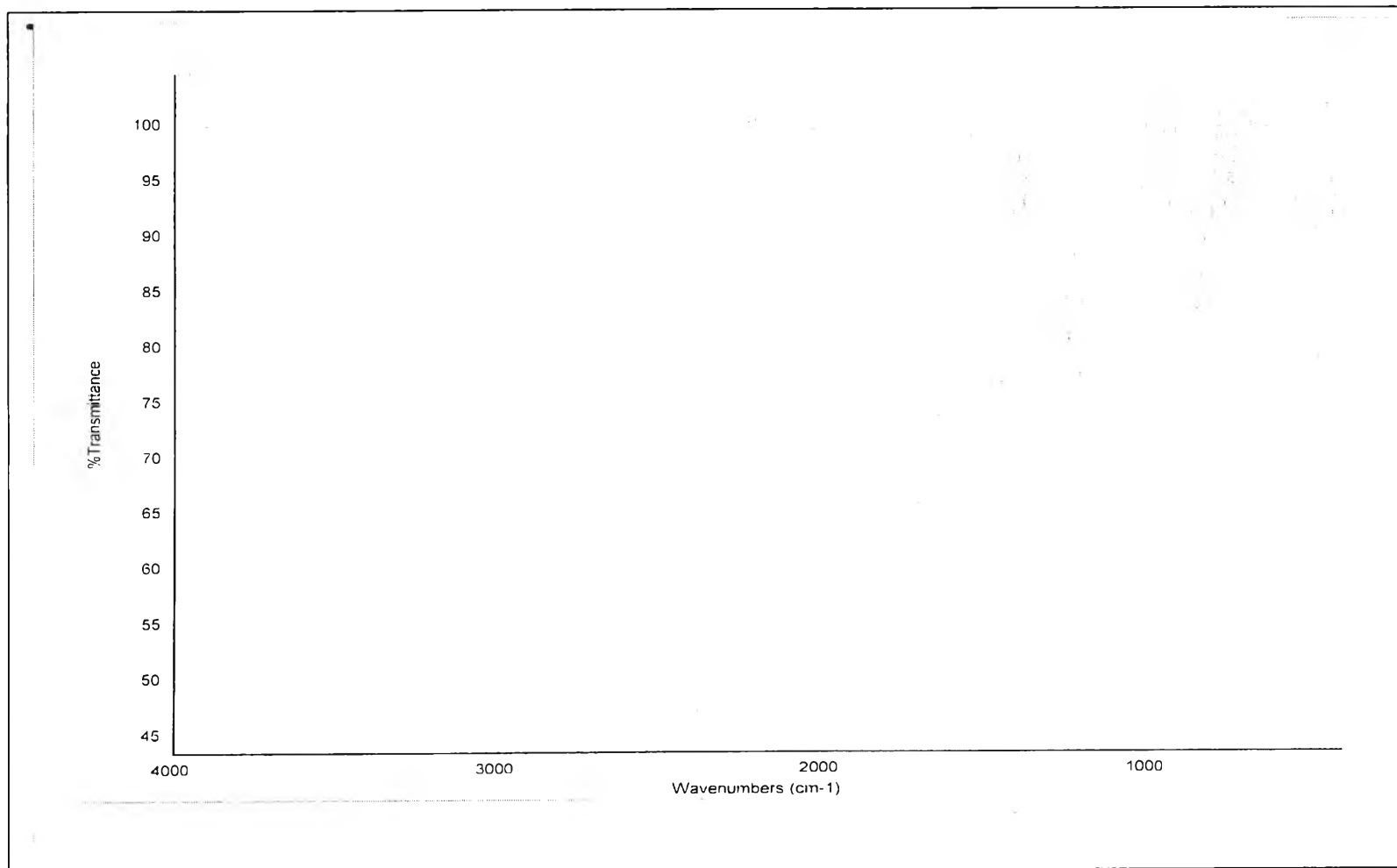


Figure B57. The IR spectrum of Metabolite 1f.

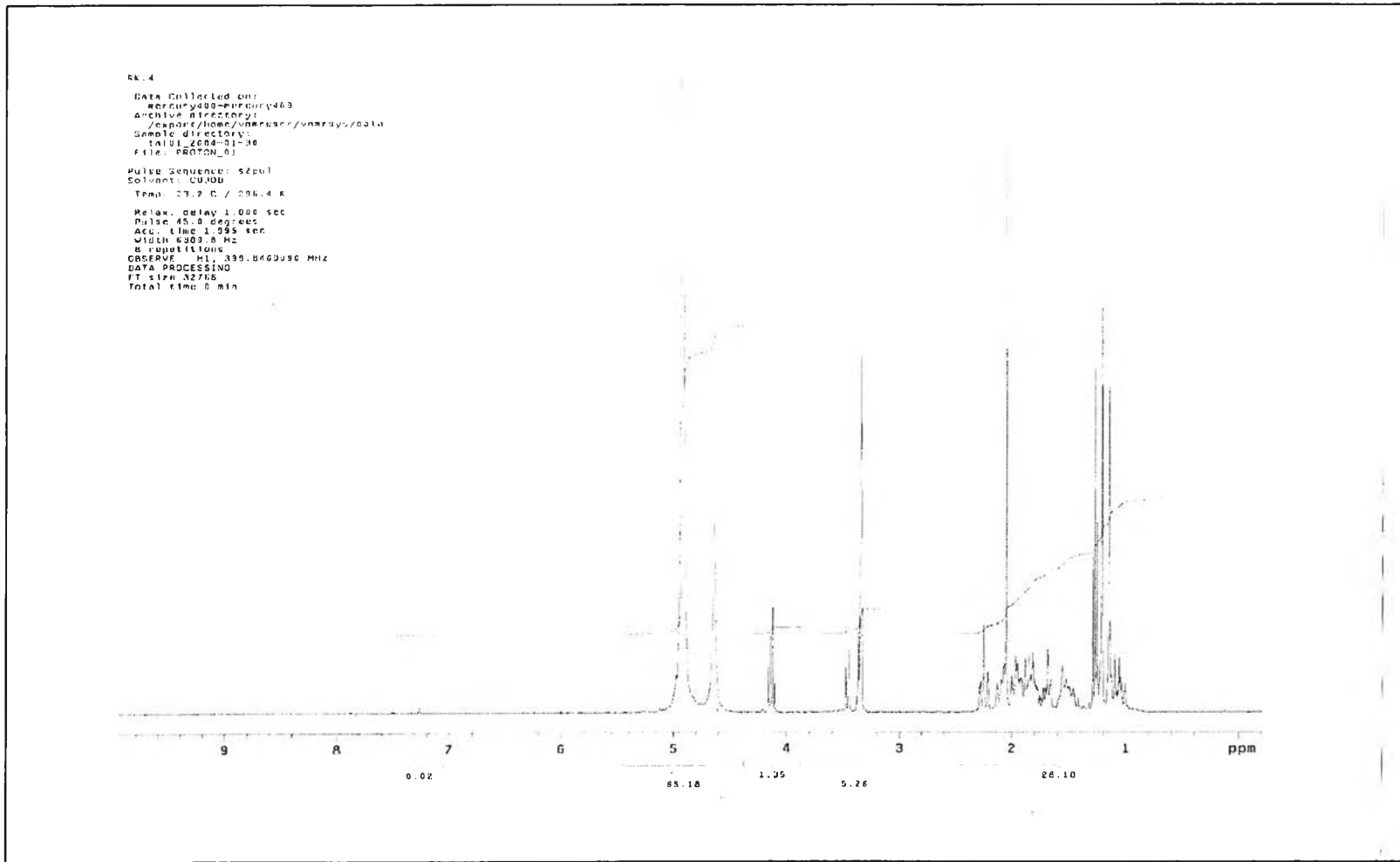


Figure B58. The ¹H-NMR spectrum of Metabolite **1f**.

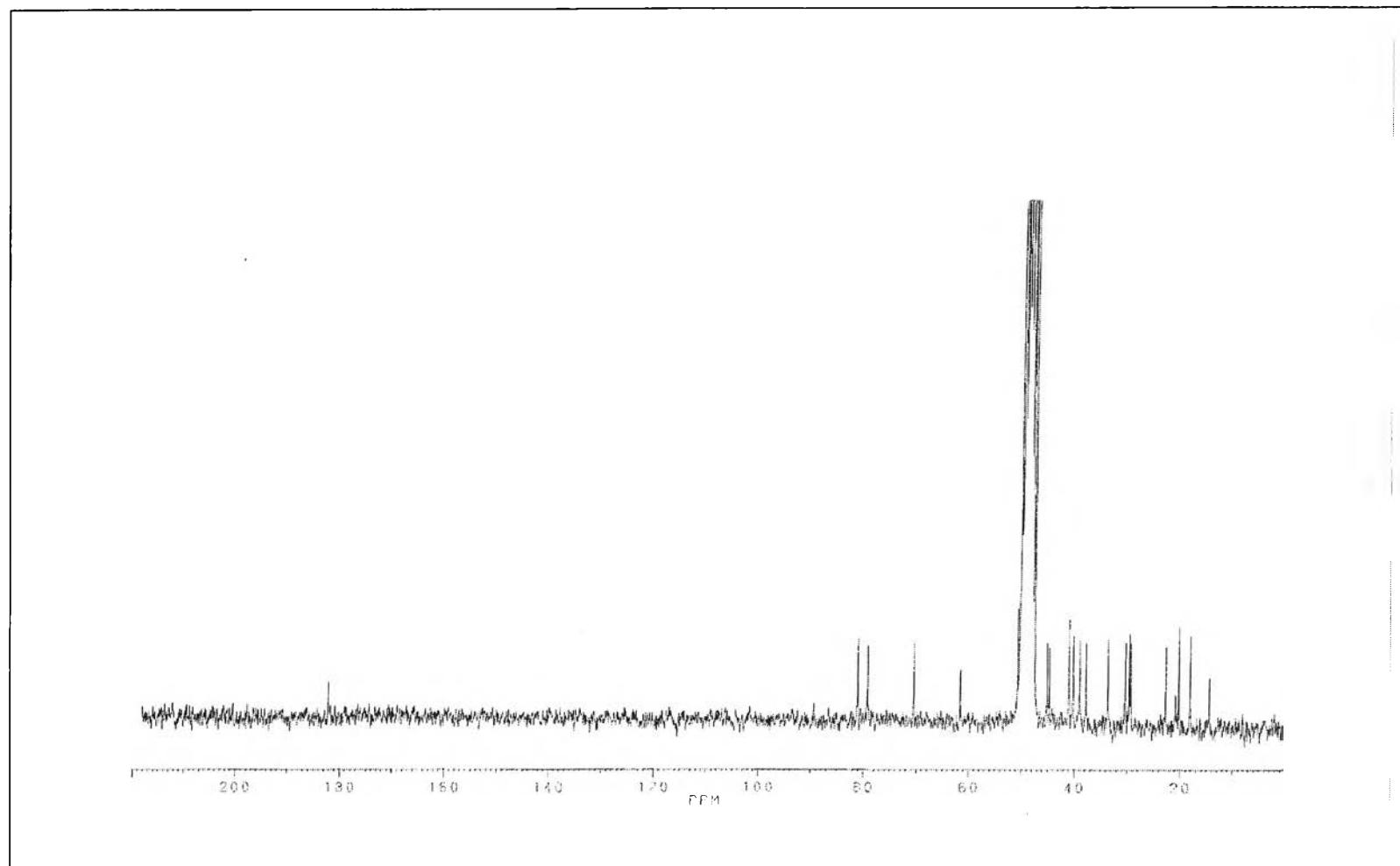


Figure B59. The ^{13}C -NMR spectrum of Metabolite **1f**.

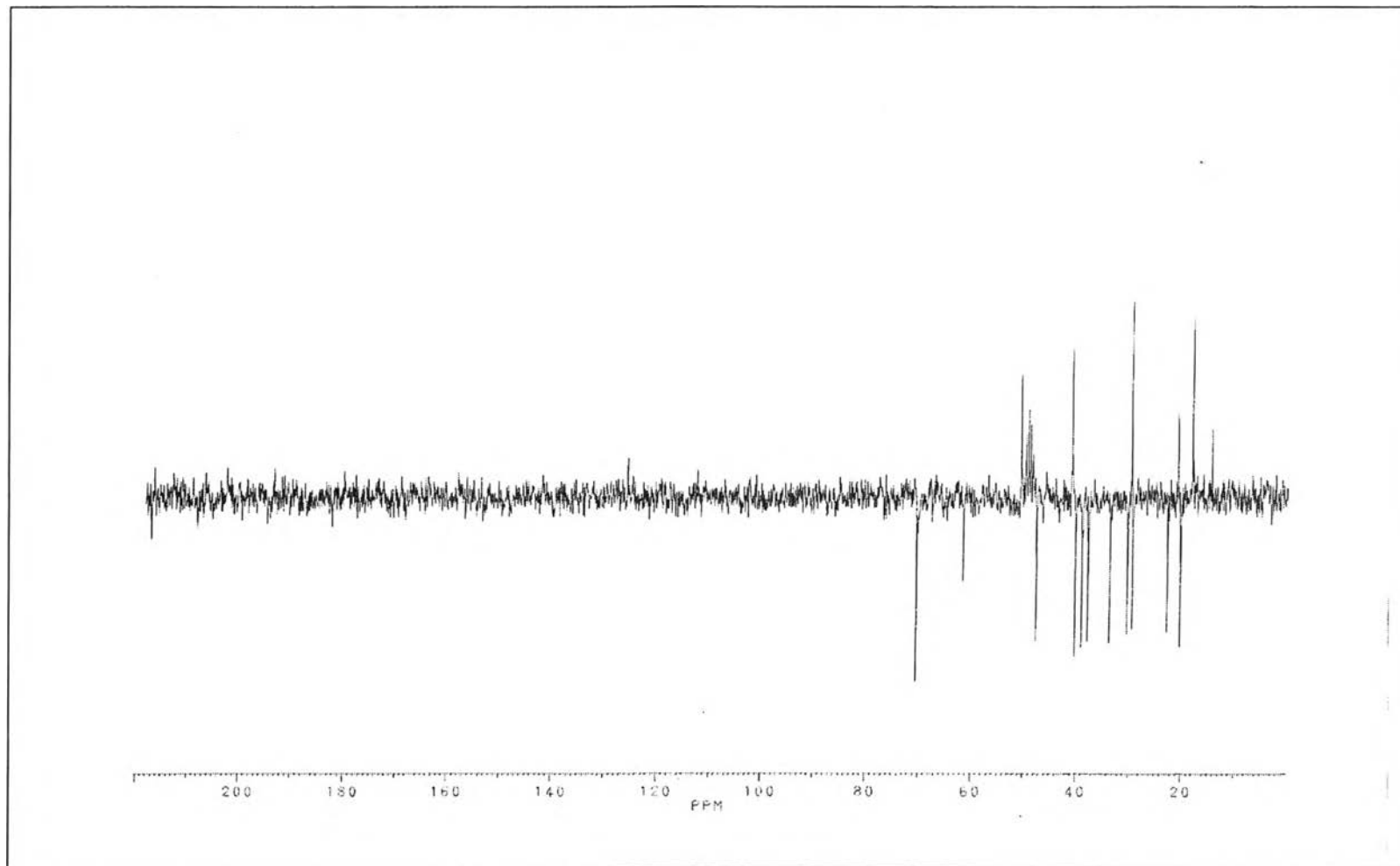


Figure B60. The DEPT spectrum of Metabolite **1f**.

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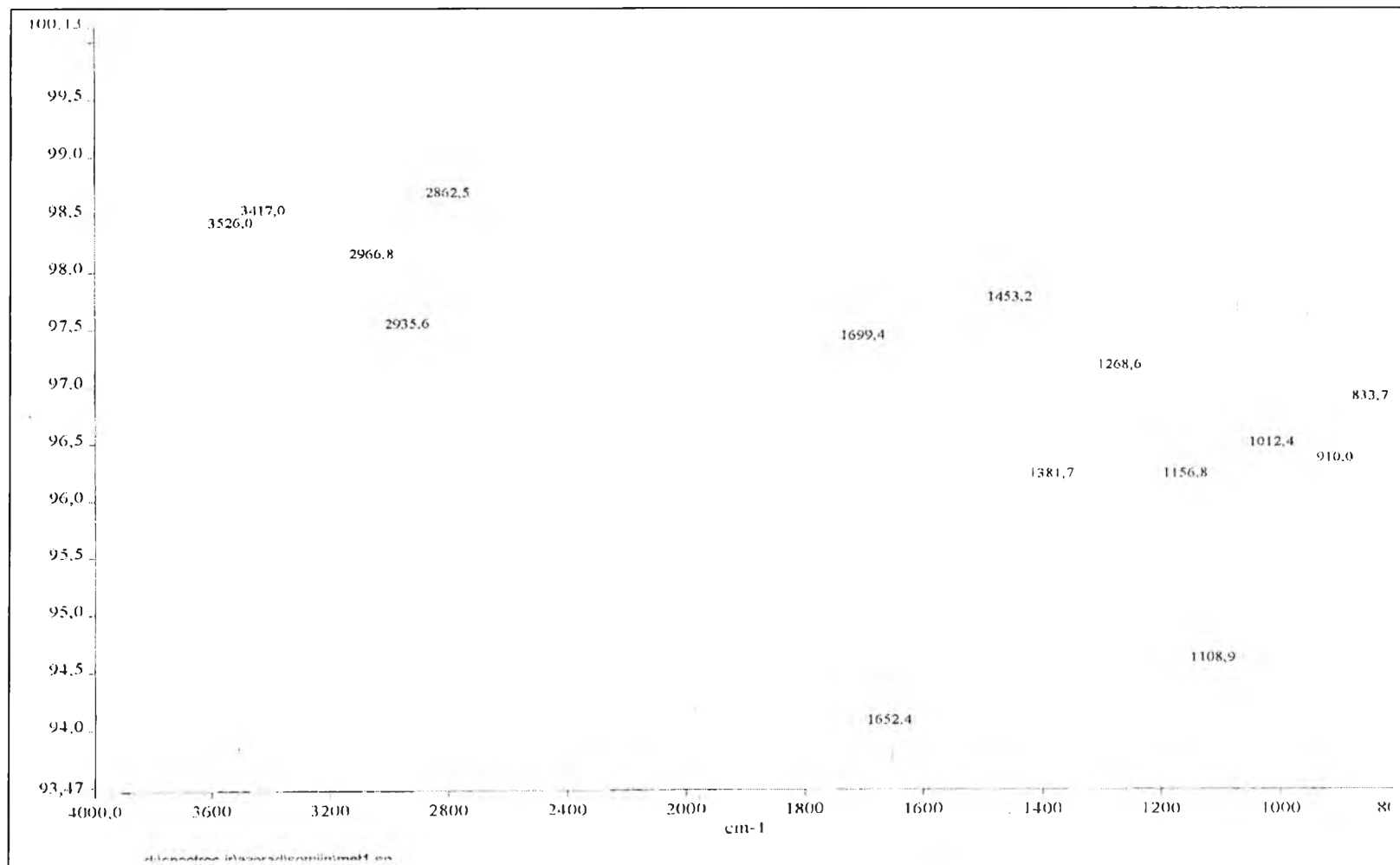


Figure B66. The IR spectrum of Metabolite 2a.

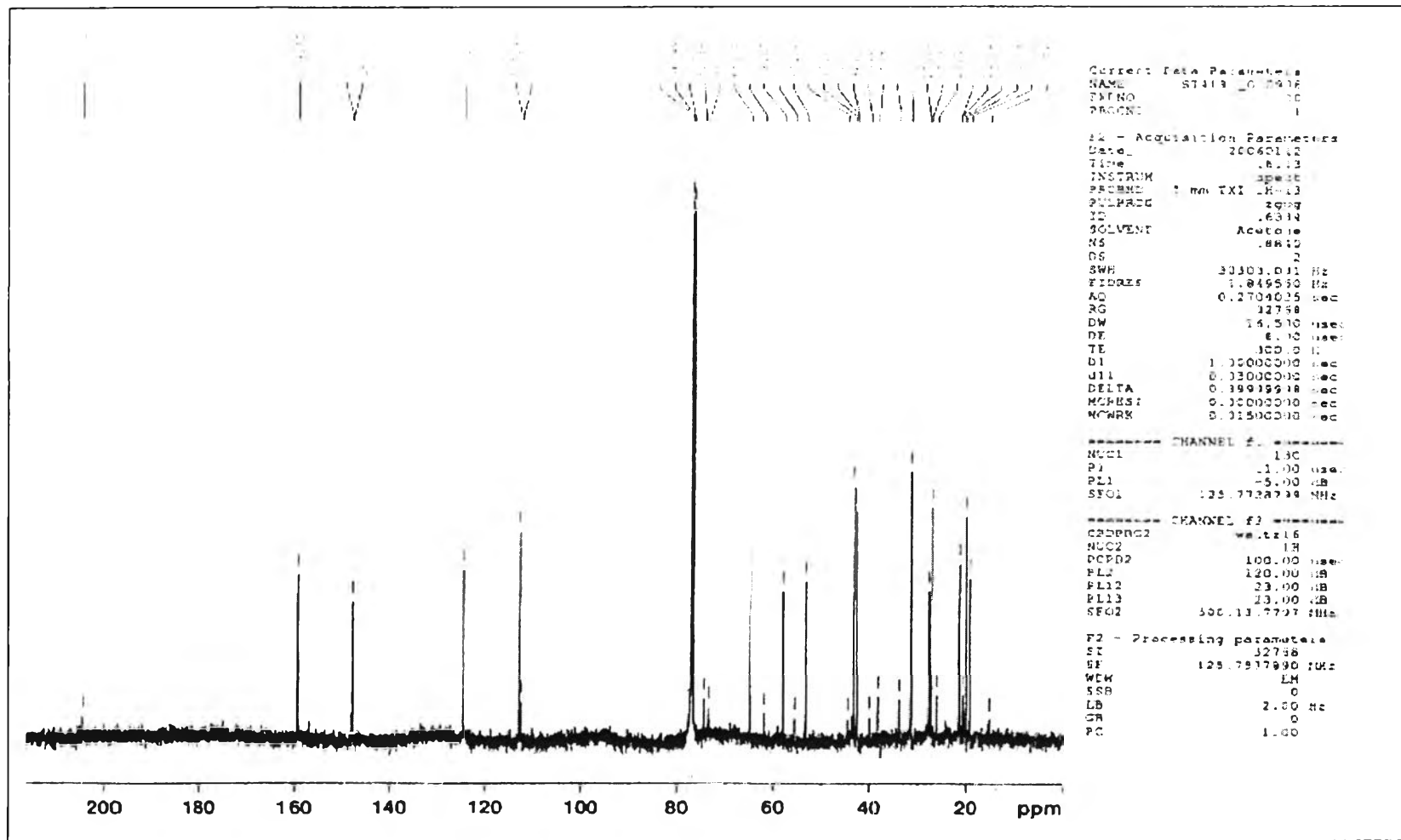


Figure B68. The ^{13}C -NMR spectrum of Metabolite 2a.

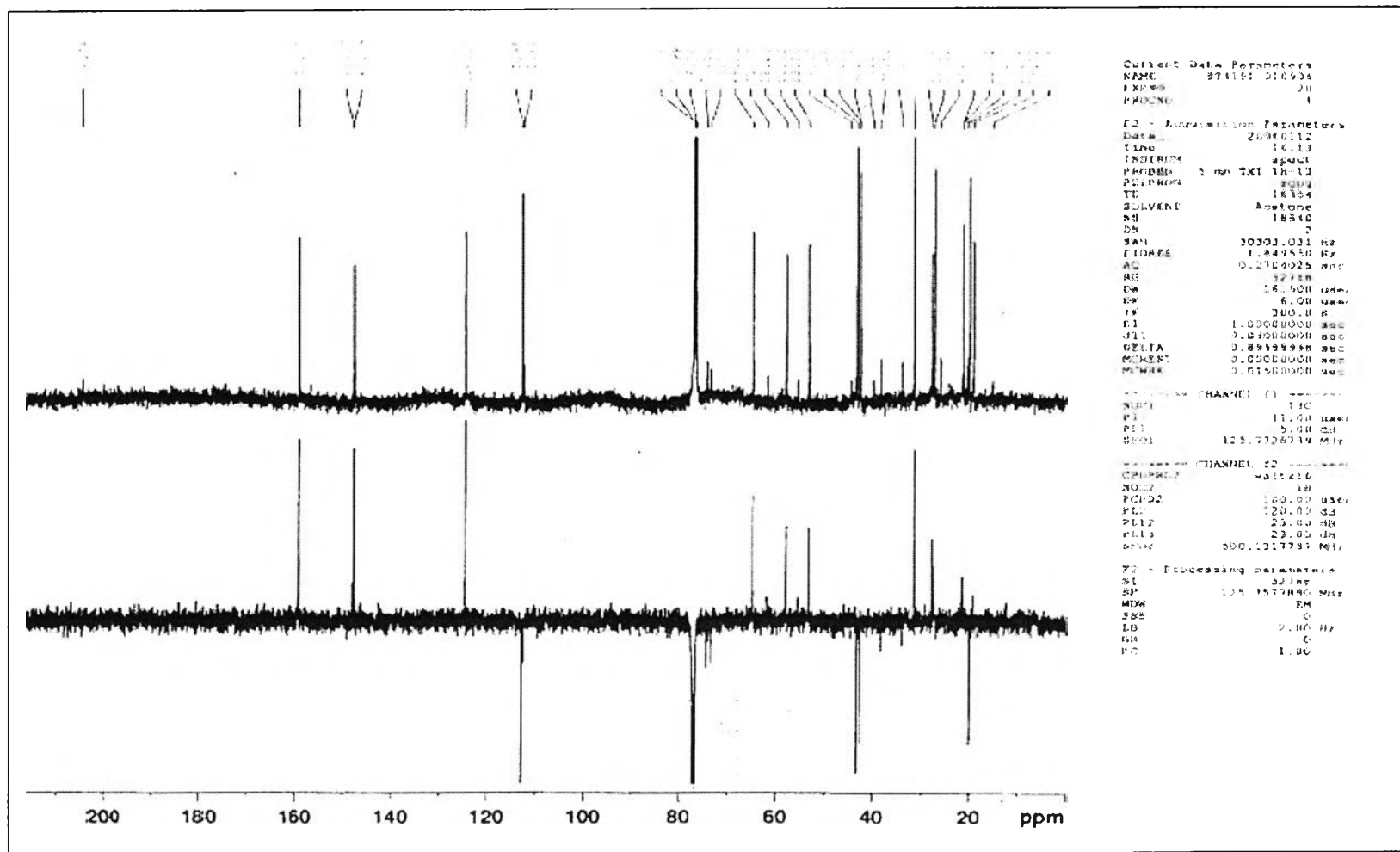


Figure B69. The DEPT spectrum of Metabolite 2a.

File : C:\HPCHEM\1\DATA\MM4560.D
Operator : MM
Acquired : 3 Jan 80 5:31 am using AcqMethod DEF350
Instrument : 5972 - In
Sample Name: ST 005 116H 19R Pr3
Misc Info : Somji
Vial Number: 1

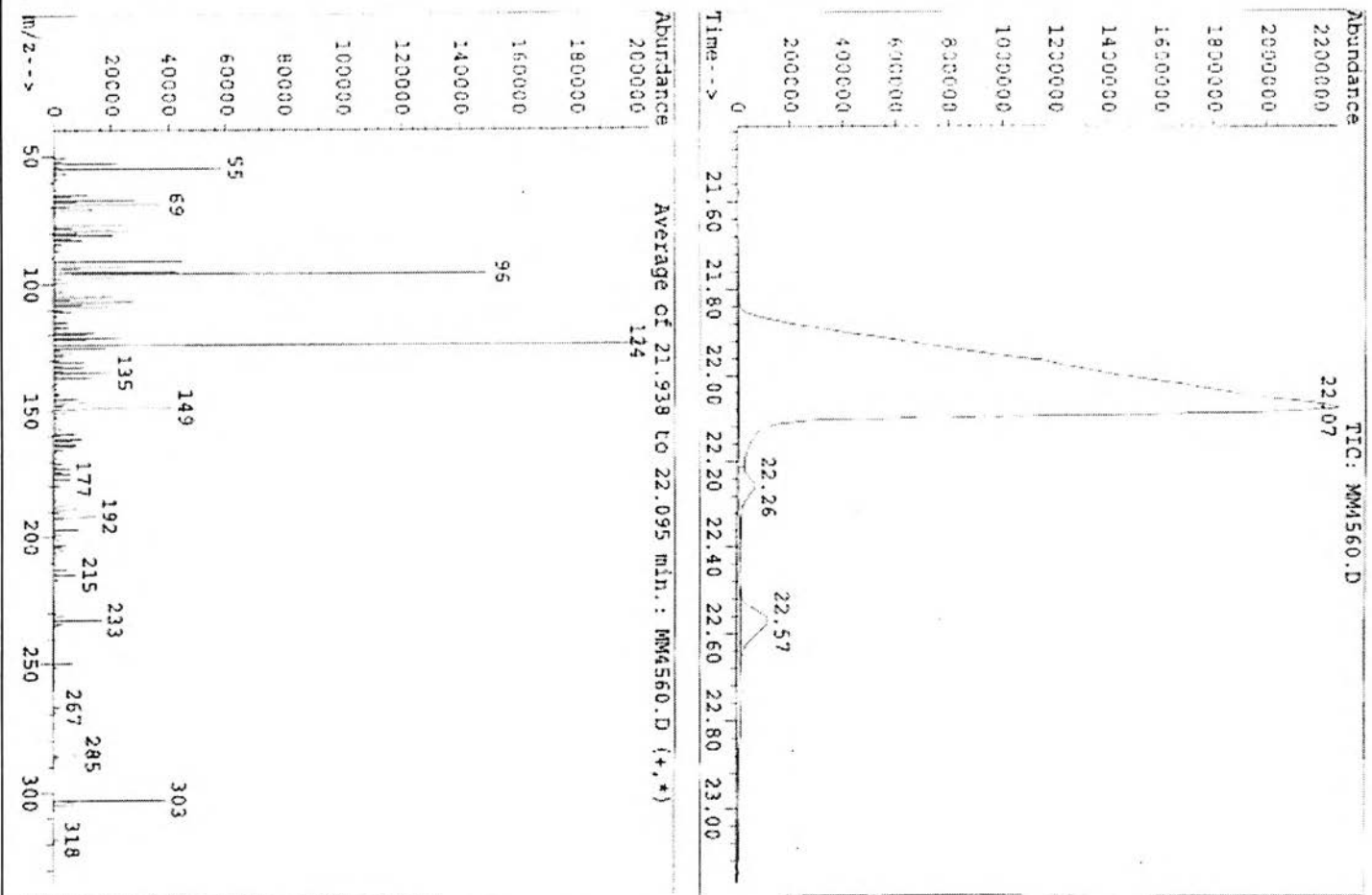


Figure B73. The GC-MS spectrum of Metabolite 2a.

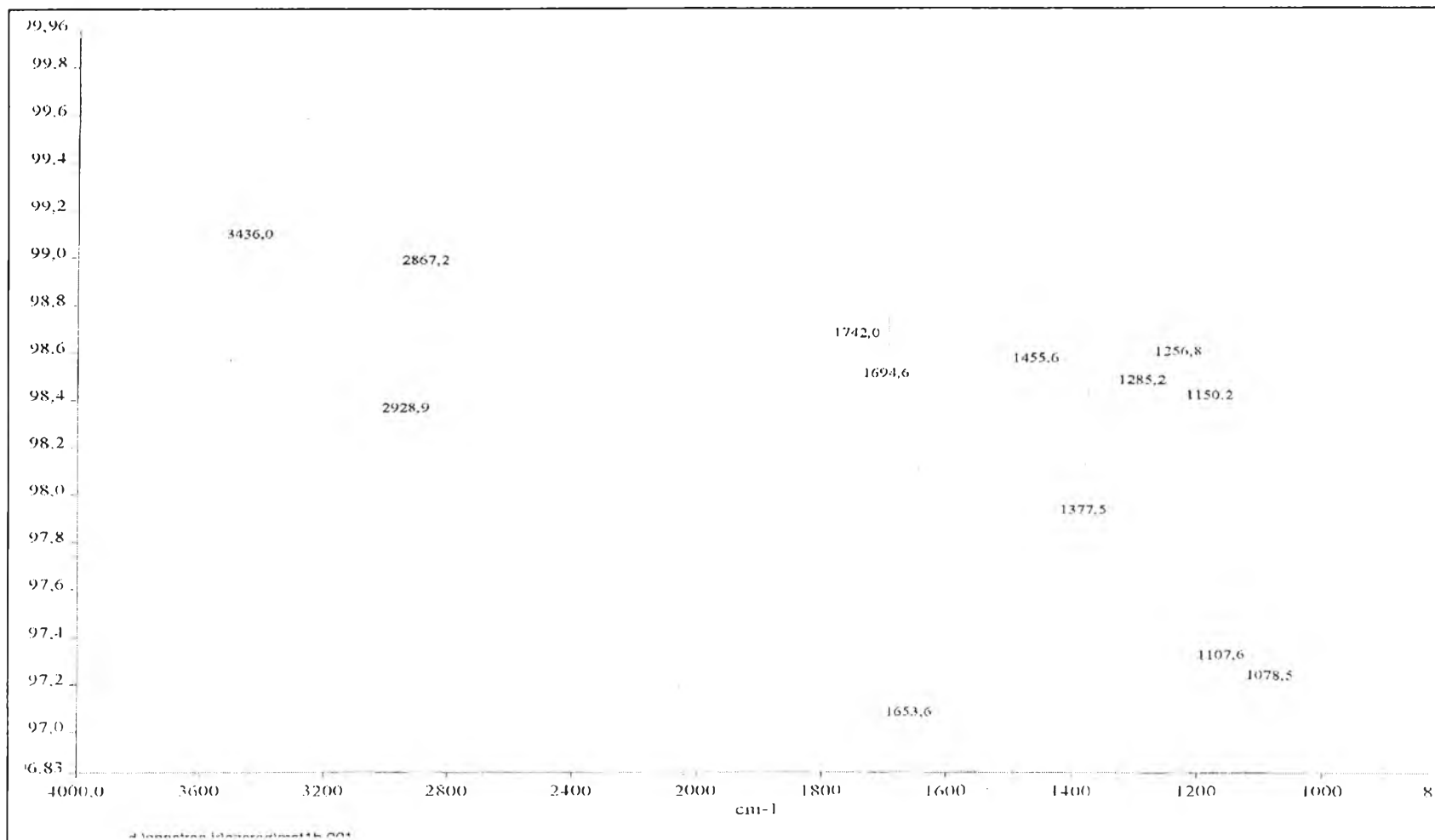


Figure B74. The IR spectrum of Metabolite 2b.

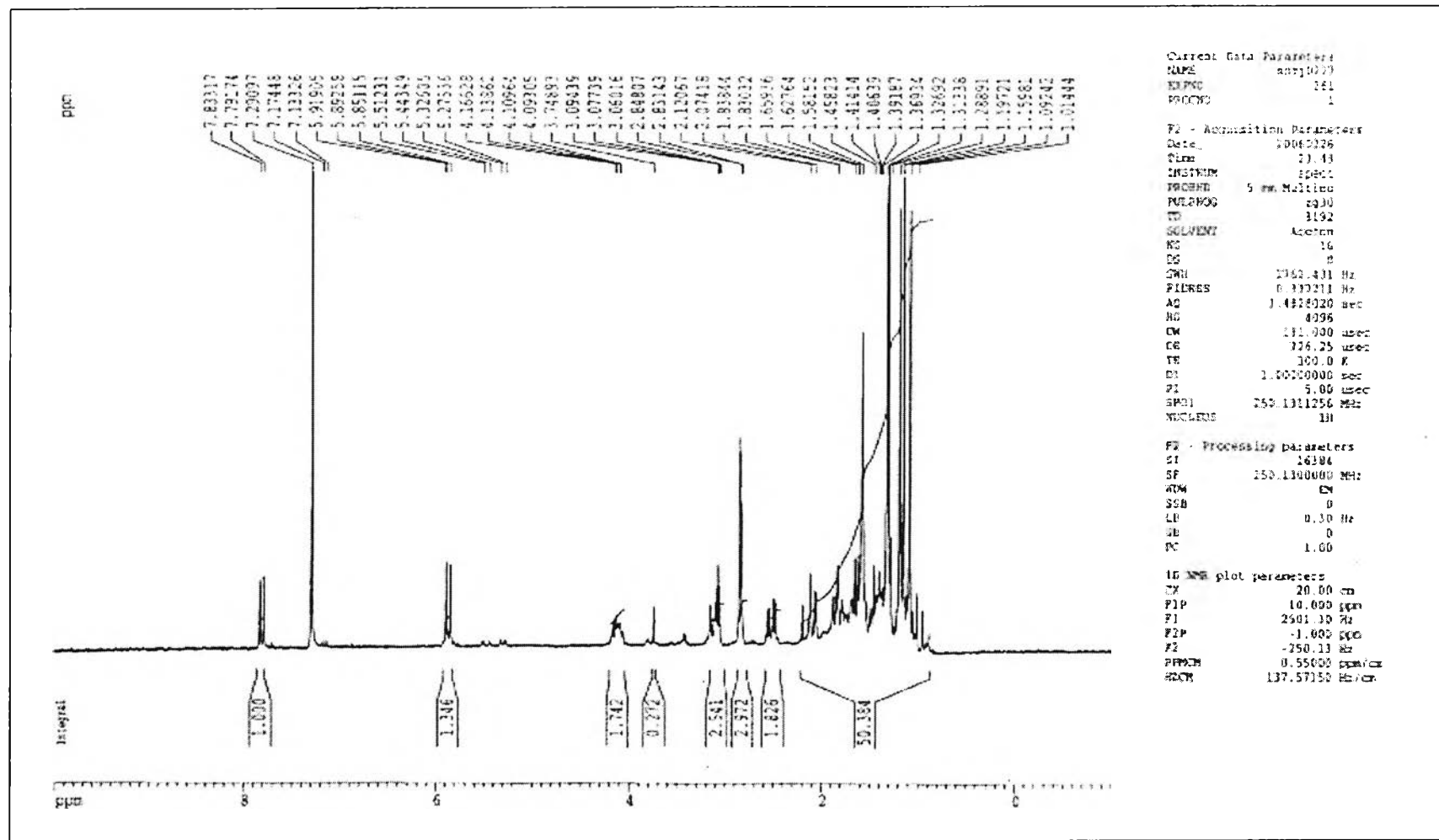


Figure B75. The $^1\text{H-NMR}$ spectrum of Metabolite **2b**.

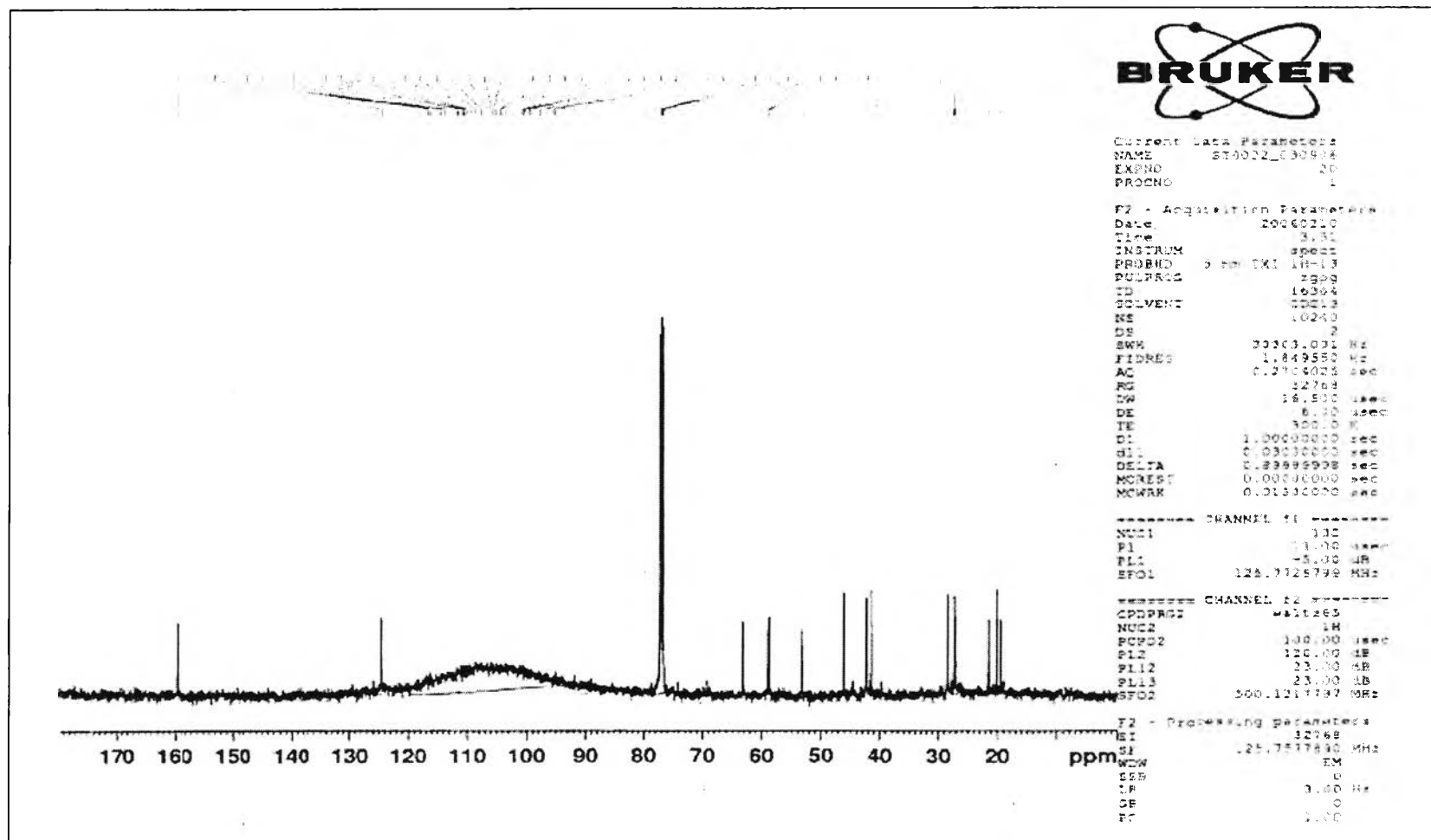


Figure B76. The ^{13}C -NMR spectrum of Metabolite 2b.

File : C:\HPCHEM\1\DATA\MM4994.D
Operator : MM
Acquired : 11 Feb 80 10:56 pm using AcqMethod DPF350
Instrument : 5972 - In
Sample Name: ST 3 4r8
Misc Info : Sonji
Vial Number: 1

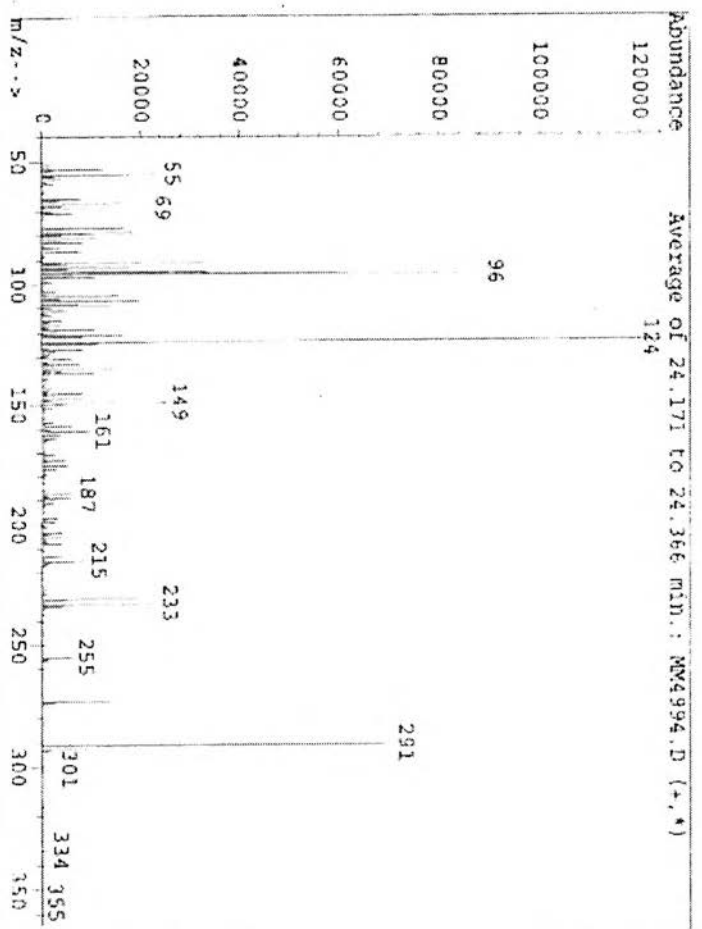
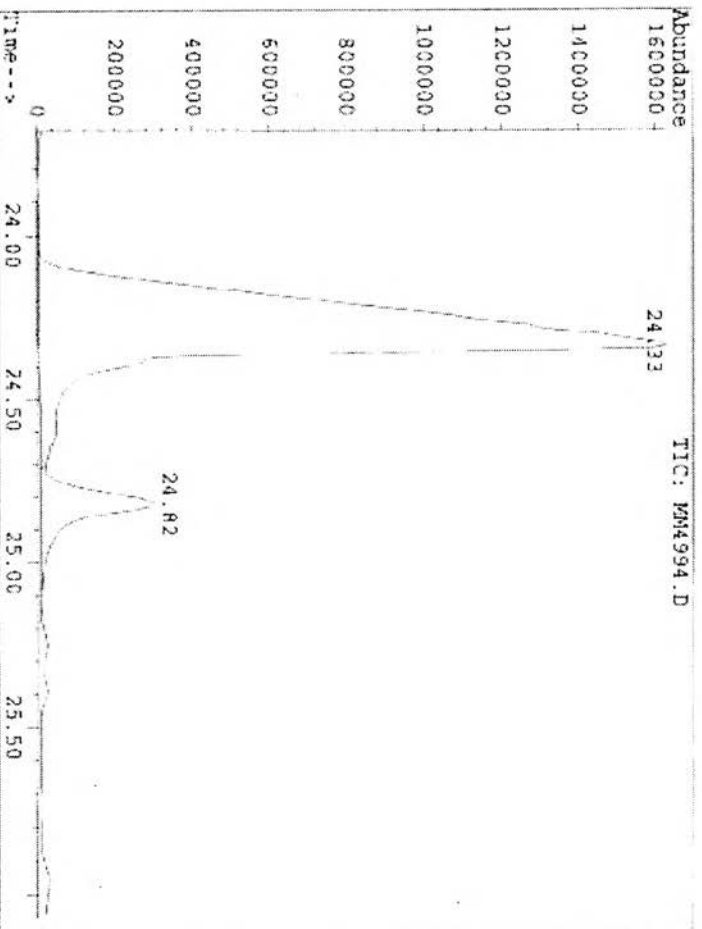


Figure B82. The GC-MS spectrum of Metabolite 2b.

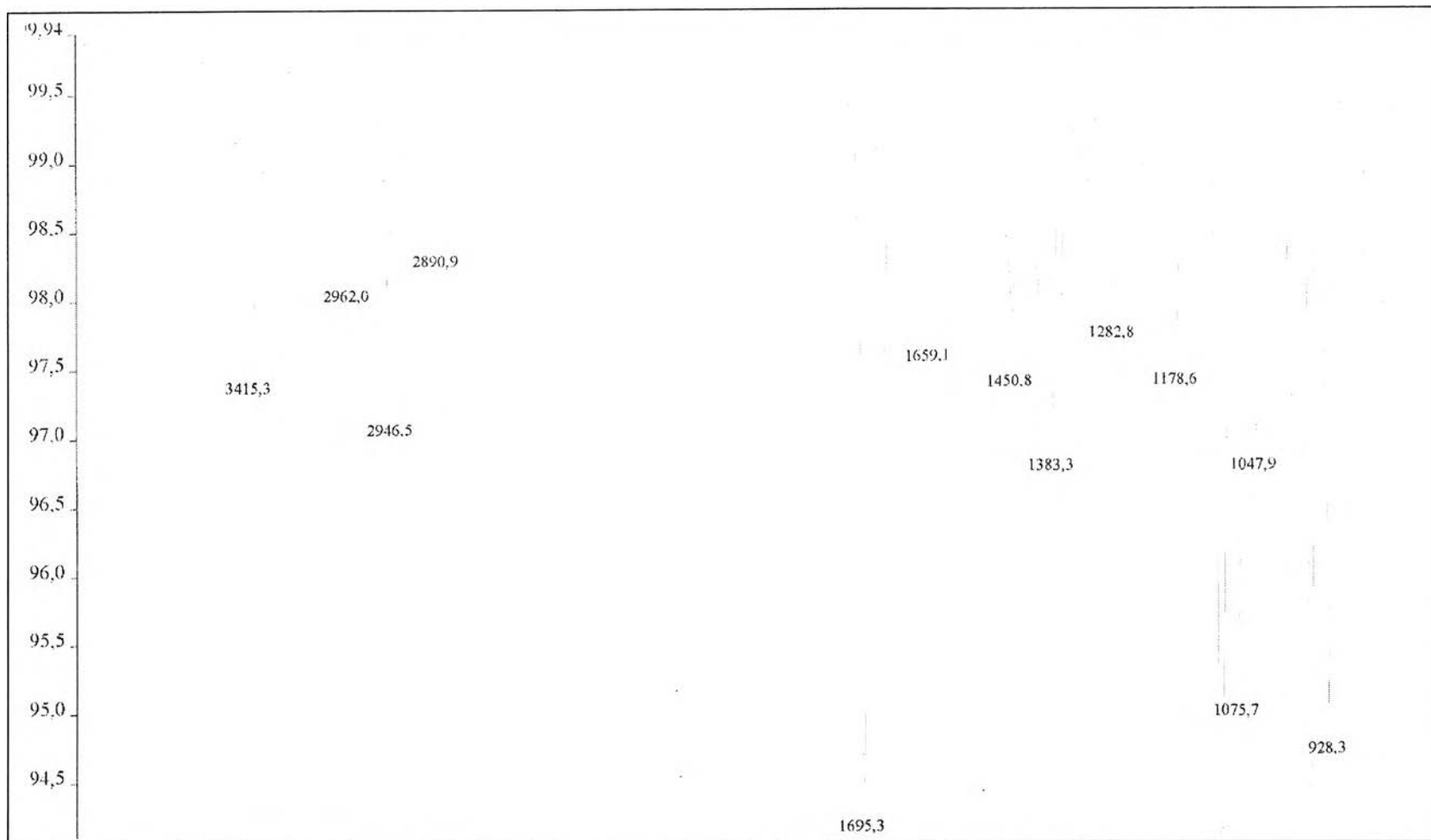


Figure B83. The IR spectrum of Metabolite 2c.

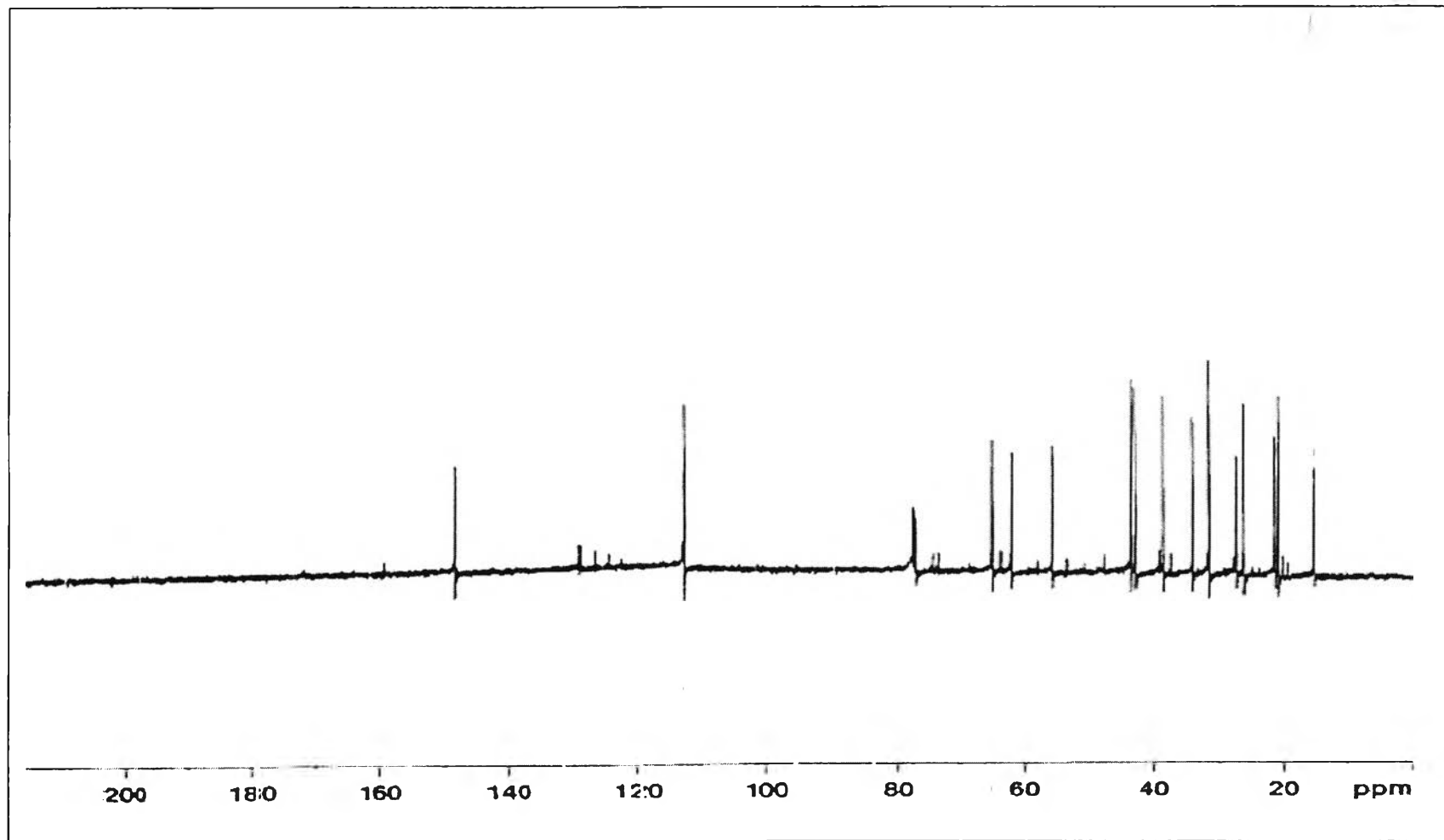


Figure B85. The ^{13}C -NMR spectrum of Metabolite 2c.

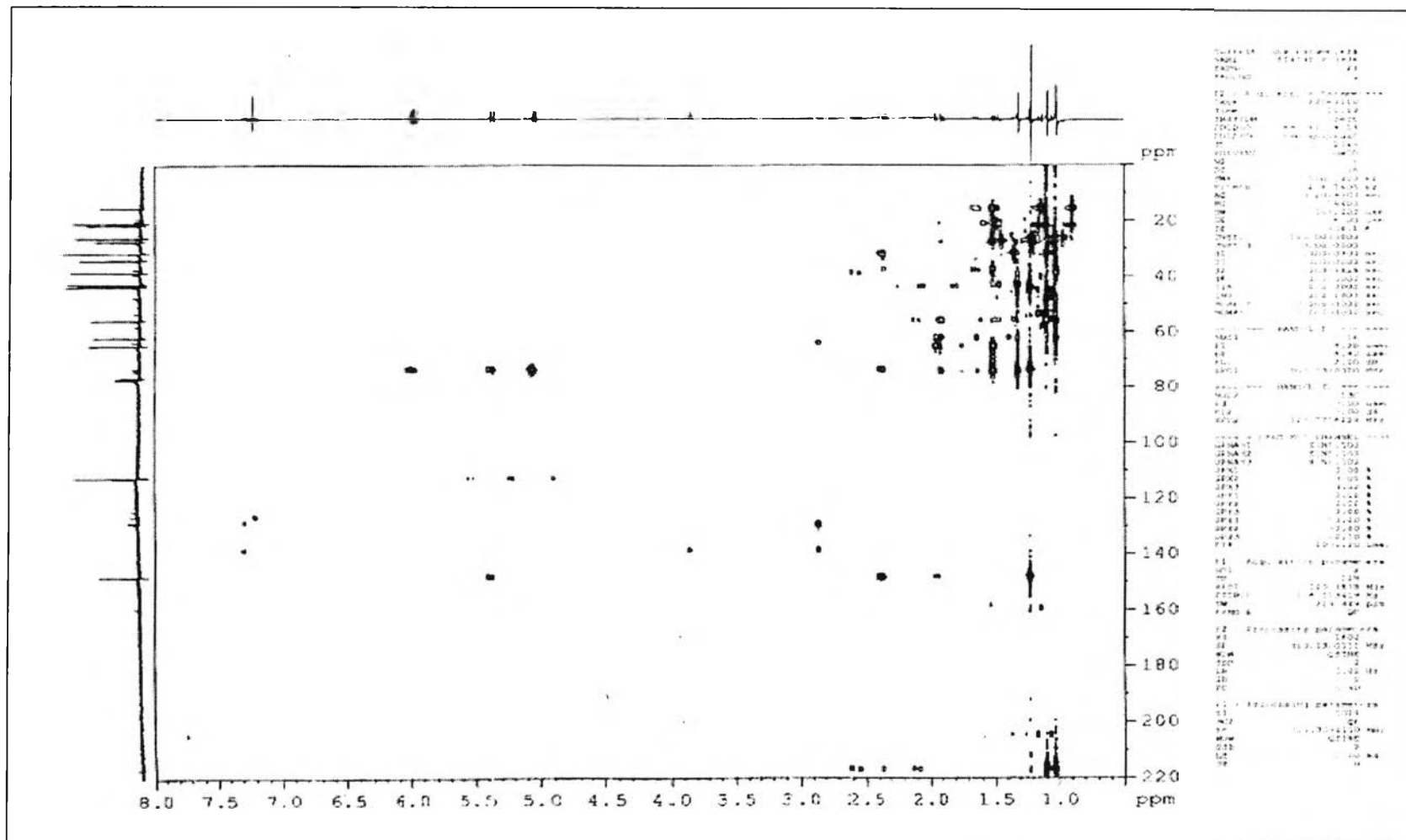


Figure B88. The gHMBC spectrum of Metabolite 2c.

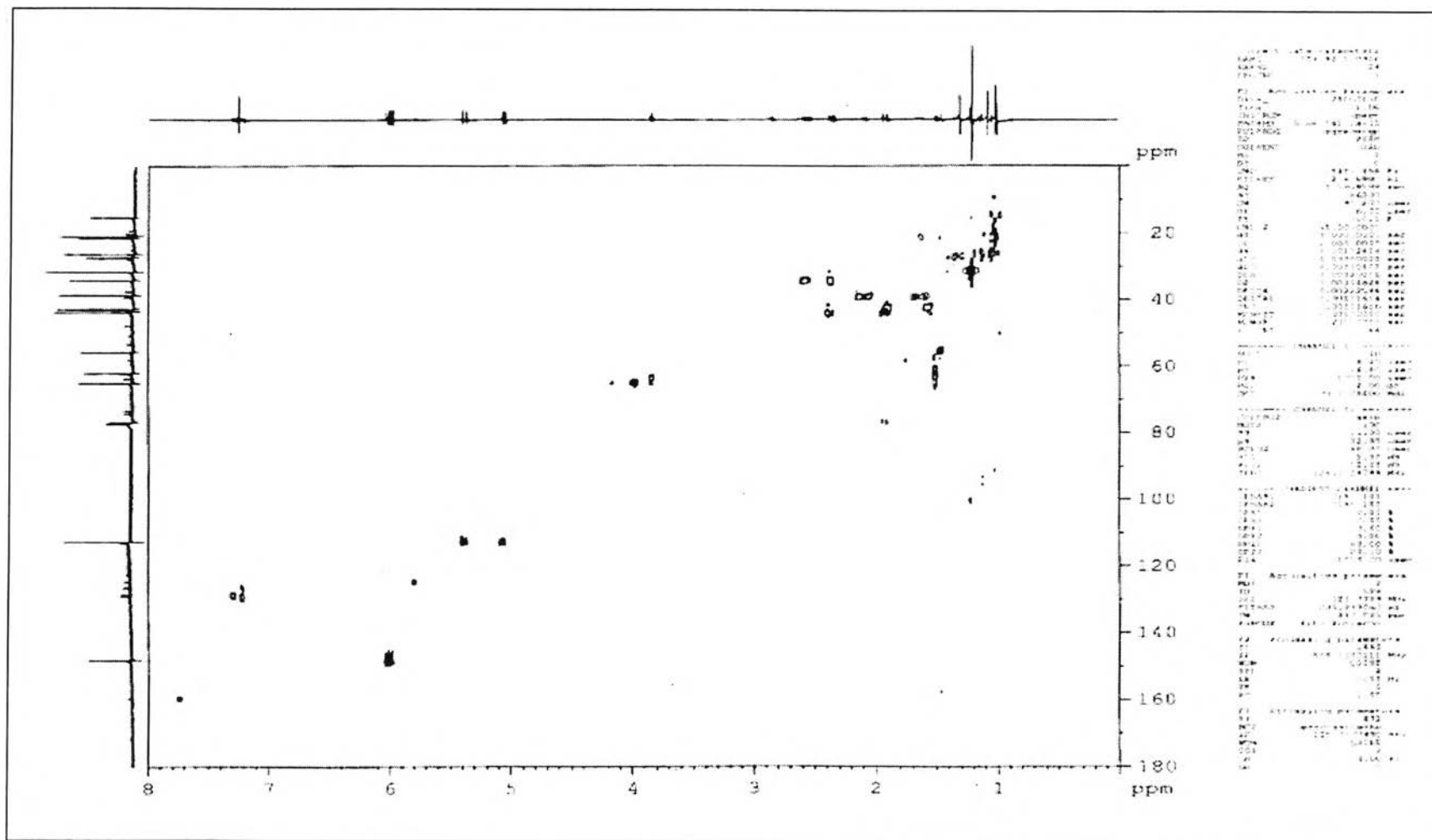


Figure B89. The gHSQC spectrum of Metabolite **2c**.

File : C:\HPCHEM\1\DATA\MM4561.D
Operator : MM
Acquired : 3 Jan 80 6:04 am using AcqMethod DEPR350
Instrument : 5972 In
Sample Name : ST 005 116H 192 F14
Misc Info : Somj1
Vial Number: 1

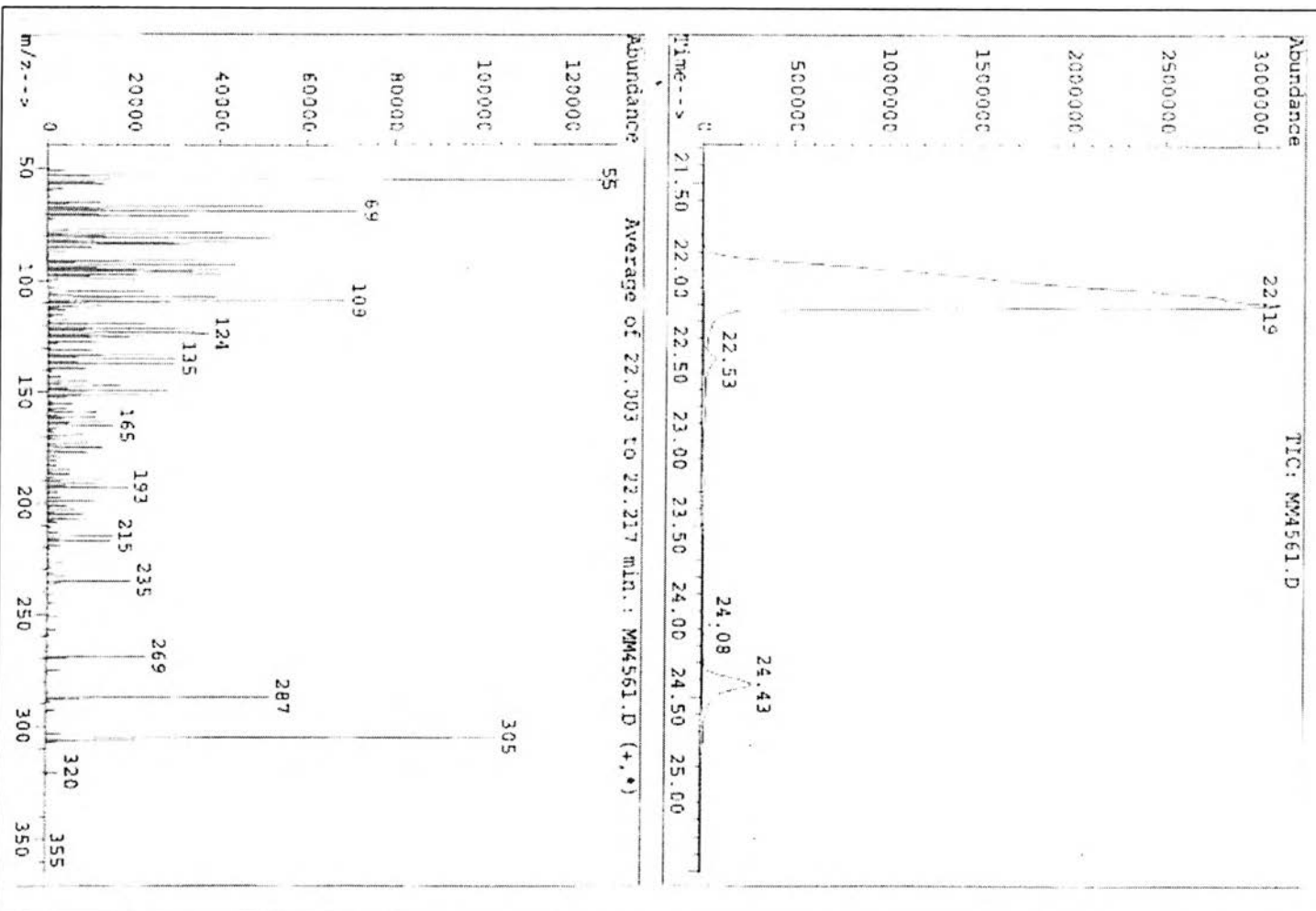


Figure B90. The GC-MS spectrum of Metabolite 2c.

APPENDIX C

Table C1. Crystal data and structure refinement for metabolite **1c**.

Empirical formula	$C_{20}H_{30}O_4$	
Formula weight	334.44	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	orthorhombic, $P2_{(1)}2_{(1)}2_{(1)}$	
Unit cell dimensions	$a = 7.8825(10)$ Å	alpha = 90 deg.
	$b = 11.1507(15)$ Å	beta = 90 deg.
	$c = 21.621(3)$ Å	gamma = 90 deg.
Volume	$1900.4(4)$ Å ³	
Z, Calculated density	4, 1.169 Mg/m ³	
Absorption coefficient	0.080 mm ⁻¹	
F(000)	728	
Theta range for data collection	2.05 to 26.37 deg.	
Index ranges	$-9 \leq h \leq 9, -13 \leq k \leq 13, -27 \leq l \leq 26$	
Reflections collected / unique	15283 / 3877 [R(int) = 0.0202]	
Completeness to theta	26.37 100.0%	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3877/ 0 / 330	
Goodness-of-fit on F^2	1.056	
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0431, wR2 = 0.1129	
R indices (all data)	R1 = 0.0452, wR2 = 0.1148	
Absolute structure parameter	0.00	
Extinction coefficient	0.0033(15)	
Largest diff. peak and hole	0.285 and -0.244 e. Å ⁻³	

Table C2. Bond lengths (Å) for metabolite **1c**.

Bond lengths	(Å)
C(1) - C(2)	1.536 (2)
C(1) - C(3)	1.5611 (19)
C(1) - C(9)	1.566 (2)
C(1) - C(13)	1.575 (2)
C(2) - H(21)	1.00 (2)
C(2) - H(22)	0.92 (2)
C(2) - H(23)	0.99 (2)
C(3) - O(1)	1.4355 (19)
C(3) - C(4)	1.519 (2)
C(3) - H(3)	0.985 (17)
C(4) - C(5)	1.506 (3)
C(4) - H(41)	0.93 (2)
C(4) - H(42)	0.92 (2)
C(5) - C(6)	1.530 (2)
C(5) - H(51)	0.99 (2)
C(5) - H(52)	0.96 (2)
C(6) - C(8)	1.526 (2)
C(6) - C(7)	1.544 (2)
C(6) - C(9)	1.558 (2)
C(7) - H(71)	1.02 (2)
C(7) - H(72)	0.98 (2)
C(7) - H(73)	0.93 (2)
C(8) - O(3)	1.214 (2)
C(8) - O(2)	1.297 (2)

Table C2. Bond lengths (Å) for metabolite **1c**. (continued)

Bond lengths	(Å)
C(9) - C(10)	1.533 (2)
C(9) - H(9)	0.97 (2)
C(10) - C(11)	1.511 (2)
C(10) - H(101)	1.01 (2)
C(10) - H(102)	0.94 (2)
C(11) - O(4)	1.417 (2)
C(11) - C(12)	1.519 (2)
C(11) - H(11)	0.94 (2)
C(12) - C(17)	1.547 (3)
C(12) - C(18)	1.545 (3)
C(12) - C(13)	1.559 (2)
C(13) - C(14)	1.550 (2)
C(13) - H(13)	0.96 (2)
C(14) - C(15)	1.533 (4)
C(14) - H(141)	0.94 (3)
C(14) - H(142)	0.96 (3)
C(15) - C(16)	1.524 (4)
C(15) - H(151)	1.06 (3)
C(15) - H(152)	0.95 (3)
C(16) - C(19)	1.511 (5)
C(16) - C(17)	1.517 (4)
C(16) - H(16)	1.04 (3)
C(17) - H(171)	0.92 (2)
C(17) - H(172)	0.92 (2)

Table C2. Bond lengths (Å) for metabolite 1c. (continued)

Bond lengths	(Å)
C(18) - C(19)	1.516 (4)
C(18) - H(181)	0.93 (3)
C(18) - H(182)	0.97 (3)
C(19)- C(20)	1.318 (4)
C(20) - H(20A)	0.93 (2)
C(20) - H(20B)	0.93 (2)
O(1) - H(1)	0.72 (2)
O(2) - H(2)	0.87 (3)
O(4) - H(4)	0.76 (2)

Table C3. Bond angles (deg.) for metabolite **1c**.

Angles	(deg.)	Angles	(deg.)
C(2) - C(1) - C(3)	108.48 (12)	C(4) - C(5) - C(6)	114.01 (14)
C(2) - C(1) - C(9)	112.79 (12)	C(4) - C(5) - H(51)	109.50 (12)
C(3) - C(1) - C(9)	105.90 (12)	C(6) - C(5) - H(51)	112.90 (11)
C(2) - C(1) - C(13)	114.35 (13)	C(4) - C(5) - H(52)	109.10 (12)
C(3) - C(1) - C(13)	108.72 (12)	C(6) - C(5) - H(52)	103.90 (13)
C(9) - C(1) - C(13)	106.21 (11)	H(51) - C(5) - H(52)	107.00 (16)
C(1) - C(2) - H(21)	112.10 (14)	C(8) - C(6) - C(5)	113.22 (13)
C(1) - C(2) - H(22)	111.80 (14)	C(8) - C(6) - C(7)	105.00 (15)
H(21) - C(2) - H(22)	107.50 (19)	C(5) - C(6) - C(7)	107.43 (15)
C(1) - C(2) - H(23)	110.90 (11)	C(8) - C(6) - C(9)	113.17 (13)
H(21) - C(2) - H(23)	111.30 (17)	C(5) - C(6) - C(9)	107.81 (13)
H(22) - C(2) - H(23)	102.90 (19)	C(7) - C(6) - C(9)	110.04 (14)
O(1) - C(3) - C(4)	108.42 (12)	C(6) - C(7) - H(71)	110.10 (17)
O(1) - C(3) - C(1)	112.57 (13)	C(6) - C(7) - H(72)	109.20 (15)
C(4) - C(3) - C(1)	113.99 (12)	H(71) - C(7) - H(72)	113.00 (2)
O(1) - C(3) - H(3)	103.80 (10)	C(6) - C(7) - H(73)	113.90 (16)
C(4) - C(3) - H(3)	108.30 (9)	H(71) - C(7) - H(73)	101.00 (2)
C(1) - C(3) - H(3)	109.20 (10)	H(72) - C(7) - H(73)	109.00 (2)
C(5) - C(4) - C(3)	113.21 (13)	O(3) - C(8) - O(2)	123.13 (17)
C(5) - C(4) - H(41)	109.30 (13)	O(3) - C(8) - C(6)	120.87 (16)
C(3) - C(4) - H(41)	106.90 (13)	O(2) - C(8) - O(6)	115.97 (15)
C(5) - C(4) - H(42)	110.60 (11)	C(10) - C(9) - C(6)	114.23 (13)
C(3) - C(4) - H(42)	109.90 (12)	C(10) - C(9) - C(1)	111.13 (13)
H(41) - C(4) - H(42)	106.70 (17)	C(6) - C(9) - C(1)	116.56 (12)

Table C3. Bond angles (deg.) for metabolite **1c**. (continued)

Angles	(deg.)	Angles	(deg.)
C(10) - C(9) - H(9)	105.40 (12)	C(14) - C(13) - H(13)	107.30 (12)
C(6) - C(9) - H(9)	103.30 (11)	C(12) - C(13) - H(13)	103.00 (12)
C(1) - C(9) - H(9)	104.70 (11)	C(1) - C(13) - H(13)	104.10 (12)
C(11) - C(10) - C(9)	111.08 (13)	C(15) - C(14) - C(13)	116.40 (2)
C(11)-C(10)-H(101)	109.60 (14)	C(15) -C(14) - H(141)	108.40 (15)
C(9) - C(10) - H(101)	111.50 (13)	C(13) -C(14) - H(141)	105.60 (16)
C(11)-C(10)-H(102)	110.30 (12)	C(15) -C(14) - H(142)	104.70 (16)
C(9) - C(10) - H(102)	108.50 (13)	C(13) -C(14) - H(142)	114.10 (15)
H(101)-C(10)-H(102)	105.60 (17)	H(141)-C(10)-H(142)	107.00 (2)
O(4) - C(11) - C(10)	110.46 (14)	C(16) - C(15) - C(14)	112.00 (2)
O(4) - C(11) - C(12)	113.38 (14)	C(16) -C(15) - H(151)	111.00 (16)
C(10) - C(11) - C(12)	111.66 (14)	C(14) -C(15) - H(151)	109.10 (15)
O(4) - C(11) - H(11)	103.20 (13)	C(16) -C(15) - H(152)	110.10 (17)
C(10) - C(11) - H(11)	110.80 (12)	C(14) -C(15) - H(152)	104.20 (16)
C(12) - C(11) - H(11)	107.00 (12)	H(151)-C(15)- H(152)	110.00 (2)
C(11) - C(12) - C(17)	111.38 (16)	C(19) - C(16) - H(17)	101.30 (2)
C(11) - C(12) - C(18)	110.44 (15)	C(19) - C(16) - C(15)	110.00 (3)
C(17) - C(12) - C(18)	100.82 (18)	C(17) - C(16) - C(15)	108.00 (2)
C(11) - C(12) - C(13)	111.94 (13)	C(19) -C(16) - H(16)	111.60 (18)
C(17) - C(12) - C(13)	111.72 (15)	C(17) -C(16) - H(16)	115.70 (17)
C(18) - C(12) - C(13)	110.01 (16)	C(15) -C(16) - H(16)	109.90 (19)
C(14) - C(13) - C(12)	108.10 (14)	C(16) -C(17) - C(12)	102.40 (2)
C(14) - C(13) - C(1)	116.38 (13)	C(16) -C(17) - H(171)	110.10 (14)
C(12) - C(13) - C(1)	116.64 (13)	C(12) -C(17) - H(171)	113.90 (14)

Table C3. Bond angles (deg.) for metabolite **1c**. (continued)

Angles	(deg.)	Angles	(deg.)
C(16)- C(17) -H(172)	109.40 (14)	C(20) - C(19) - C(16)	126.20 (3)
C(12)- C(17) -H(172)	108.40 (14)	C(20) - C(19) - C(18)	125.50 (3)
H(171)-C(17)-H(172)	112.00 (2)	C(16) - C(19) - C(18)	108.30 (2)
C(19) - C(18) - C(12)	104.80 (2)	C(19) -C(20)- H(20A)	120.00 (0)
C(19)-C(18)-H(181)	110.30 (16)	C(19) -C(20)- H(20B)	120.00 (0)
C(12)-C(18)-H(181)	111.60 (17)	H(20A)-C(20)-H(20B)	120.00 (0)
C(19)-C(18)-H(182)	107.90 (17)	C(3) -O(1) - H(1)	110.00 (2)
C(12)-C(18)-H(182)	110.70 (17)	C(8) -O(2) - H(2)	107.40 (18)
H(181)-C(18)-H(182)	111.00 (3)	C(11) -O(4) - H(4)	112.00 (18)

BIOGRAPHY

Miss Somjintana Taveepanich was born on March 2, 1976 in Bangkok Province, Thailand. She graduated with Bachelor Degree of Chemistry in 1997 from the Faculty of Science, Khon Khan University and Master Degree of Science in Organic Chemistry in 2000 from the Faculty of Science, Chulalongkorn University. Since 2001, she has studied for a Doctor of Philosophy Degree in Organic Chemistry at Department of Chemistry, Faculty of Science, Chulalongkorn University. She had received financial support from University Development Commission (UDC) scholarship and The Royal Golden Jubilee Ph.D. scholarship and the Graduate school, Faculty of Science, Chulalongkorn University.

