

Xylocarpins A-I, Limonoids from Chinese Mangrove Plant *Xylocarpus granatum*

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HMBC spectrum

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NOESY spectrum

ESIMS spectrum

HRSIMS data

10 Xylocarpin I (10)

IR spectrum

^1H NMR spectrum

^{13}C and DEPT spectra

HMQC spectrum

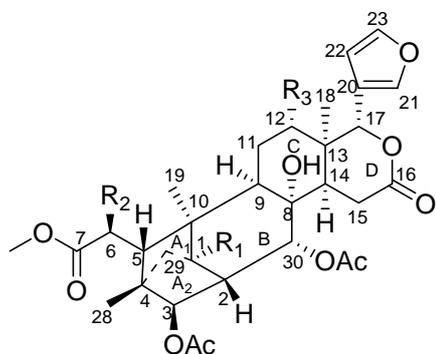
HMBC spectrum

NOESY spectrum

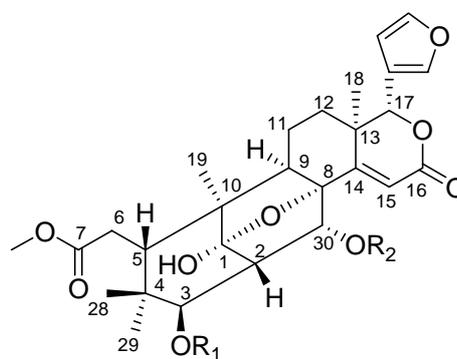
ESIMS spectrum

HRSIMS data

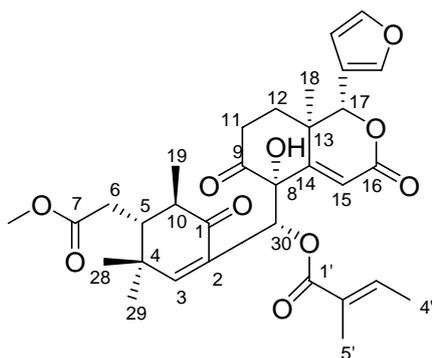
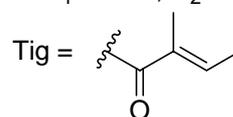
Structures of xylocarpins A-I (1-5, 7-10)



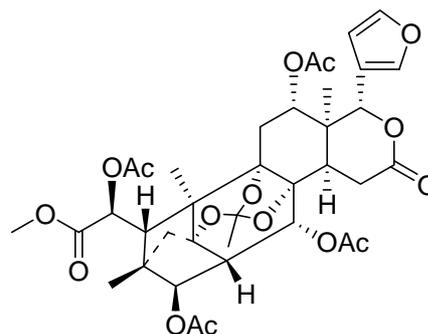
1. $R_1 = R_2 = \text{OAc}$, $R_3 = \text{H}$
2. $R_1 = \text{OAc}$, $R_2 = R_3 = \text{H}$
3. $R_1 = \text{OH}$, $R_2 = \text{H}$, $R_3 = \text{OAc}$
4. $R_1 = R_3 = \text{OAc}$, $R_2 = \text{OH}$
5. $R_1 = R_2 = \text{OAc}$, $R_3 = \text{OH}$
6. $R_1 = R_3 = \text{OAc}$, $R_2 = \text{H}$



7. $R_1 = R_2 = \text{Ac}$
8. $R_1 = \text{OAc}$, $R_2 = \text{Tig}$



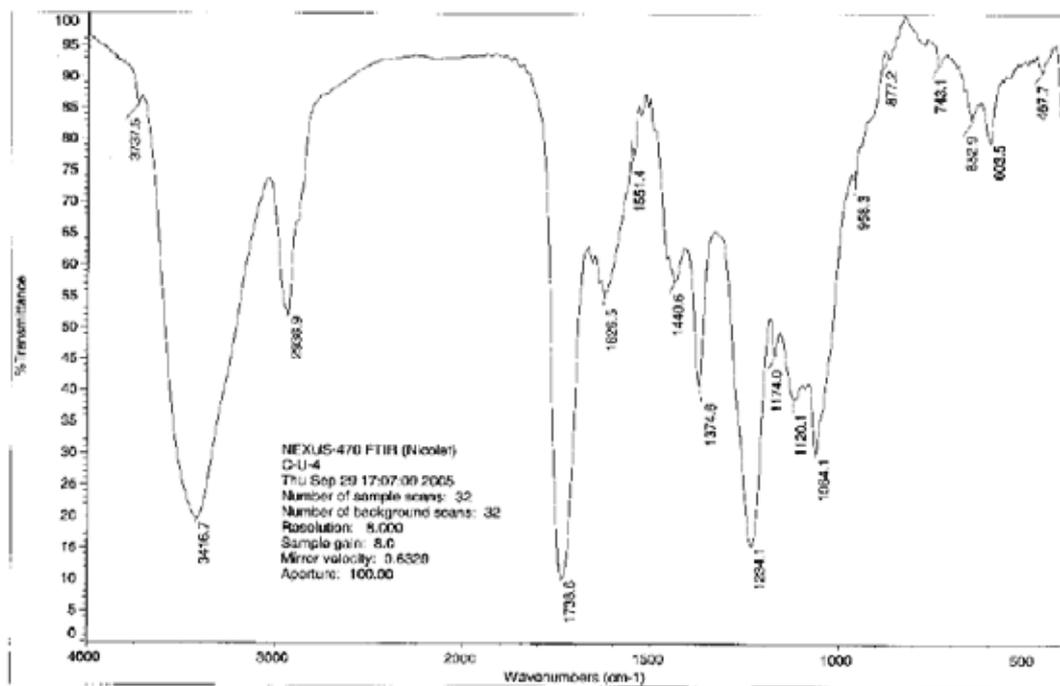
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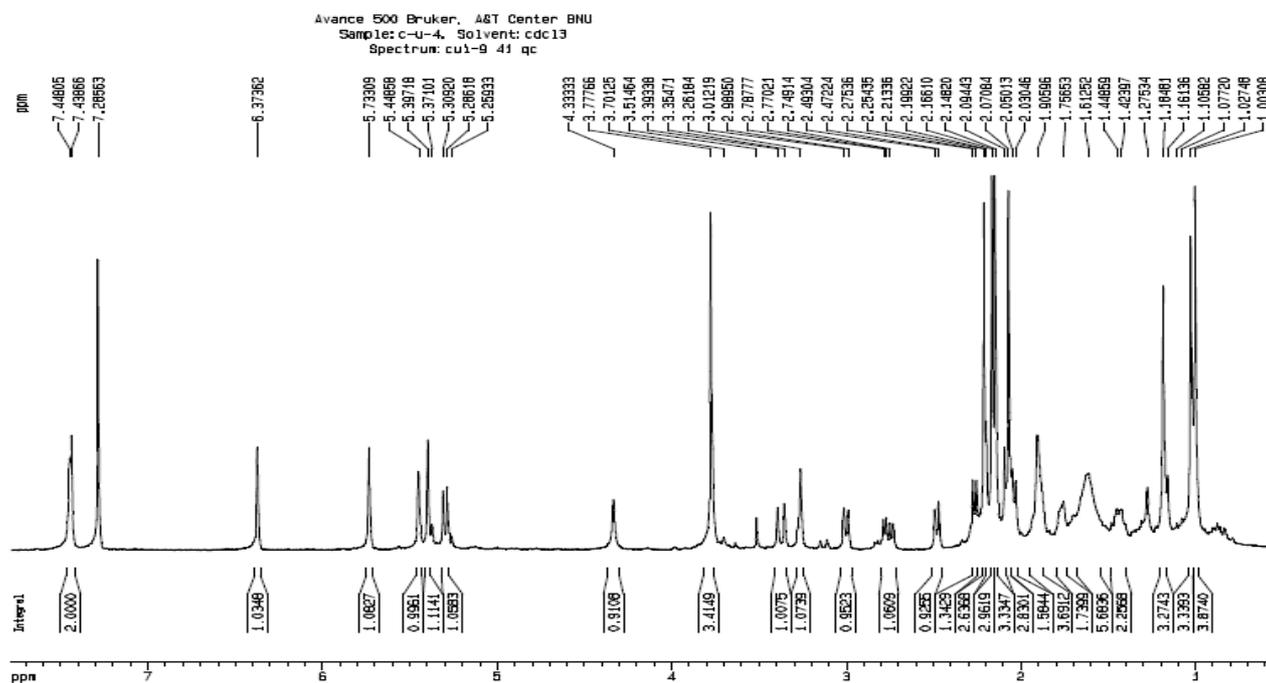
10

1 Xylocarpin A (1)

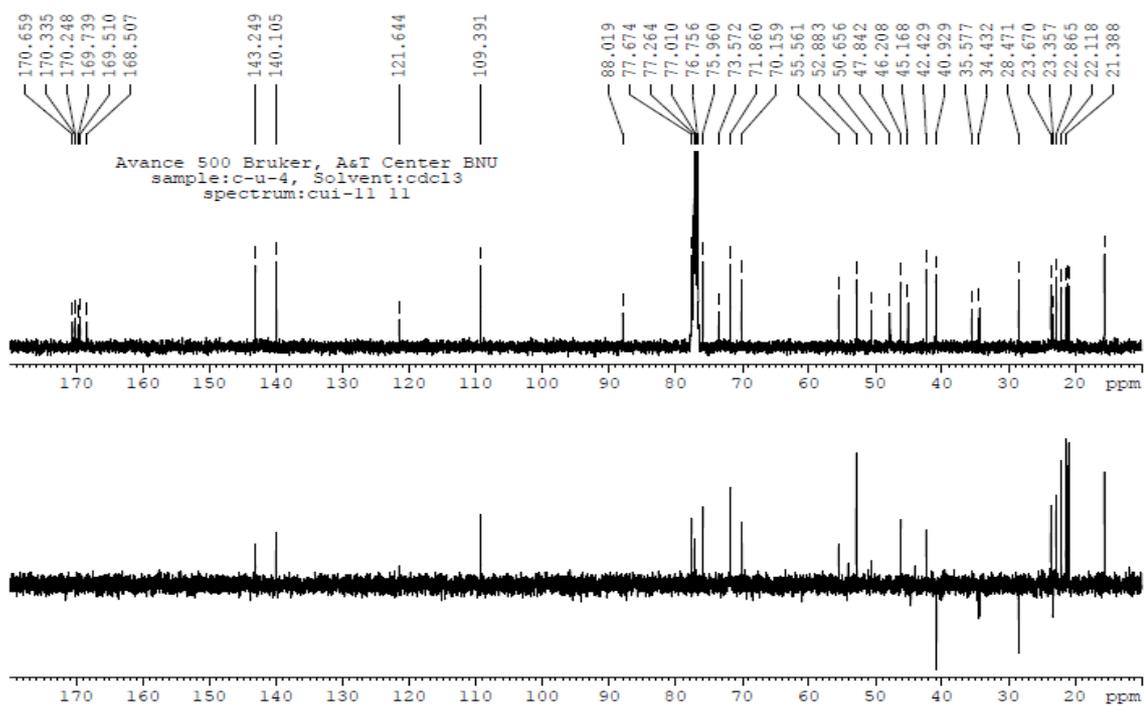
IR spectrum



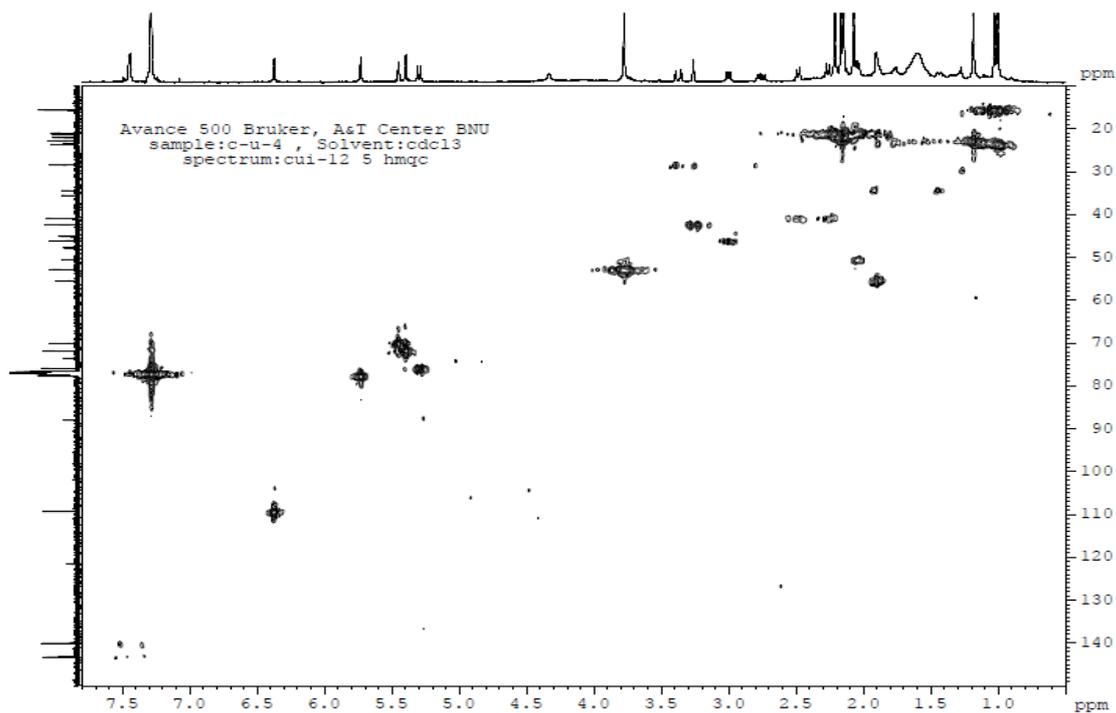
¹H NMR spectrum



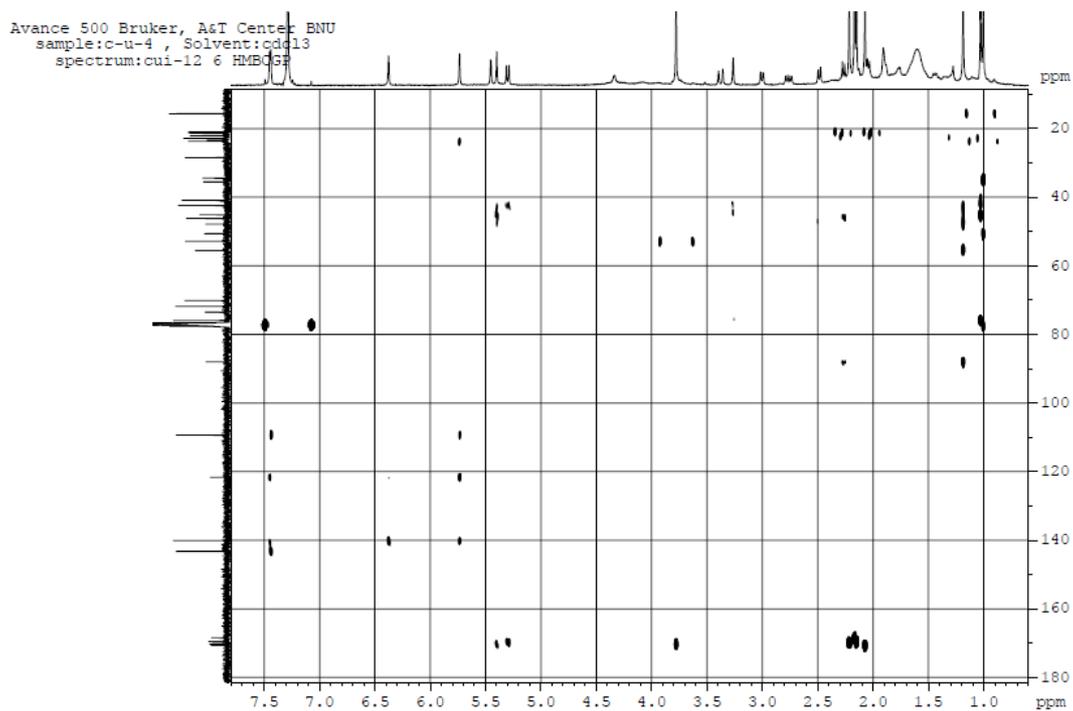
¹³C and DEPT NMR spectra



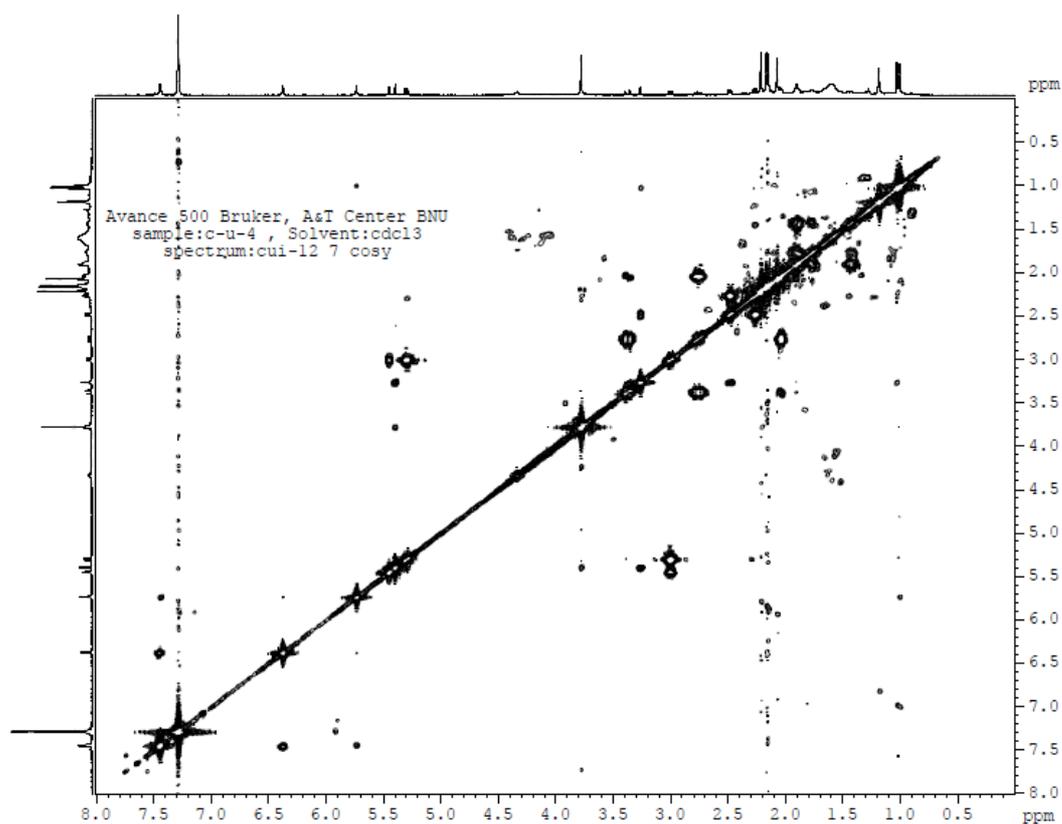
HMQC spectrum



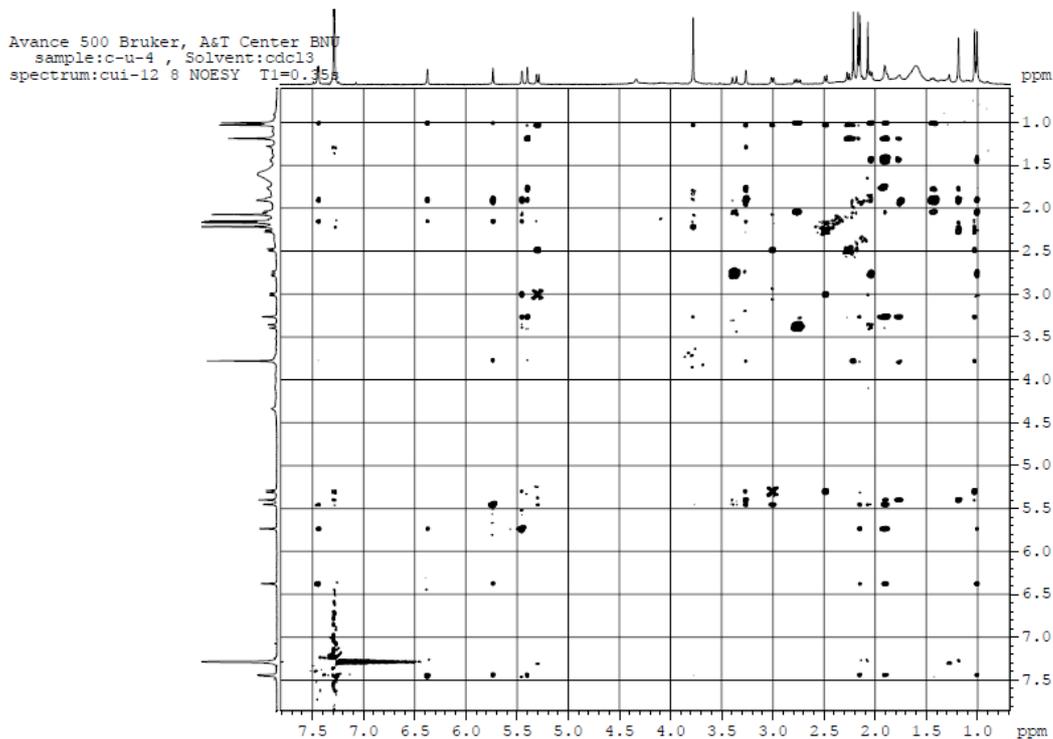
HMBC spectrum



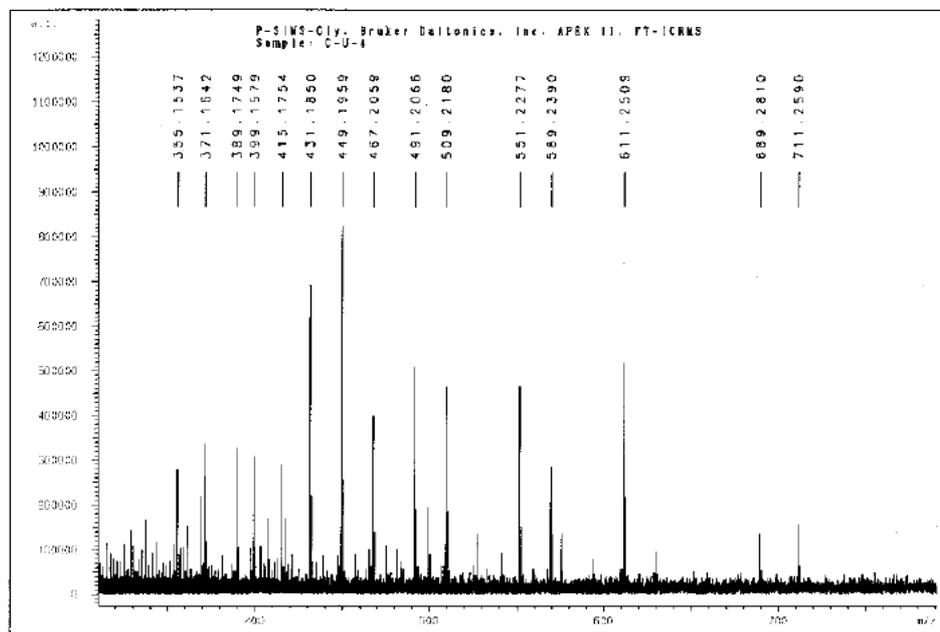
DQFCOSY spectrum



NOESY spectrum



HRSIMS data



Z:\BRUKER\POS-SIMS\AP-SIMS (A)\12\Xylocarp\REFLAV1 810202 Refl Av1 12 05 41:36 2006

XMASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-CuiJianXin/06/pdata/1/massan
XMASS Mass Analysis Constraints

Ion mass [1] = 711.2589540
Ion mass [2] = 689.2809730

Charge = +1
Tolerance = 0.0100000

DBR min = -2
DBE max = 200

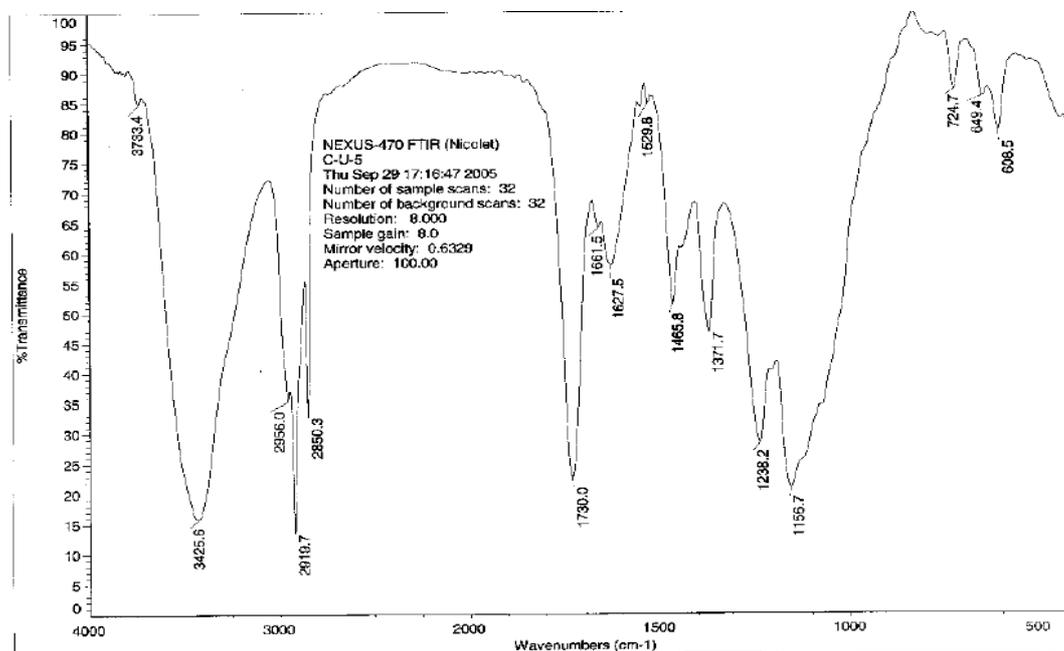
Max Candidates = 100

Atom	#(min, max)	Wt%(min, max)
C	25 38	0.00 100.00
H	25 60	0.00 100.00
O	5 20	0.00 100.00
Na	0 1	0.00 100.00

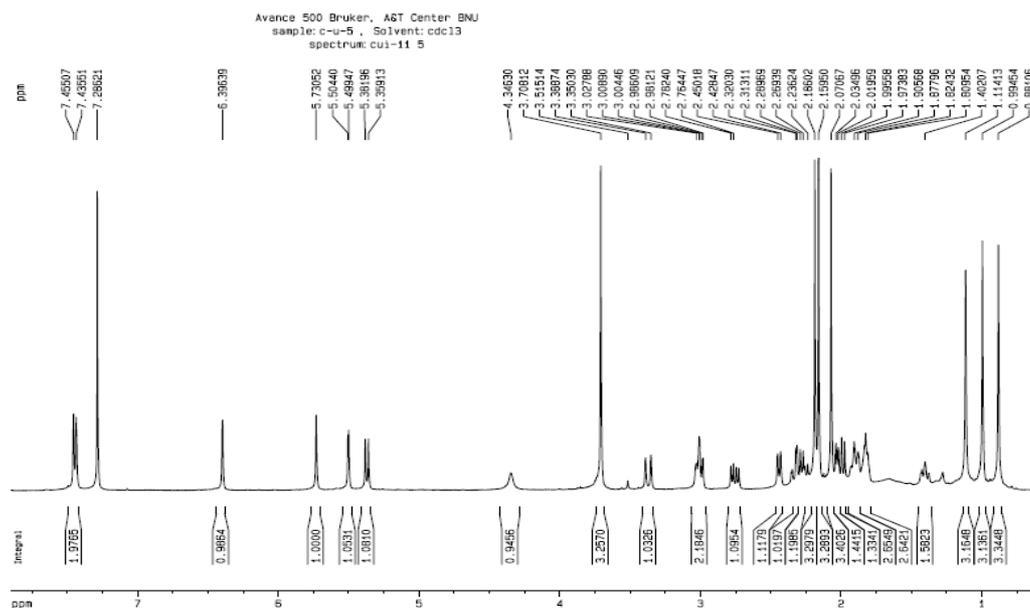
#	C	H	O	Na	mass	error
*** Mass Analysis for mass 711.2589540						
1	35	44	14	1	711.2623012	4.706e-06
*** Mass Analysis for mass 689.2809730						
1	35	45	14	0	689.2803558	8.954e-07
2	26	50	19	1	689.2838193	4.129e-06
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2 Xylocarpin B (2)

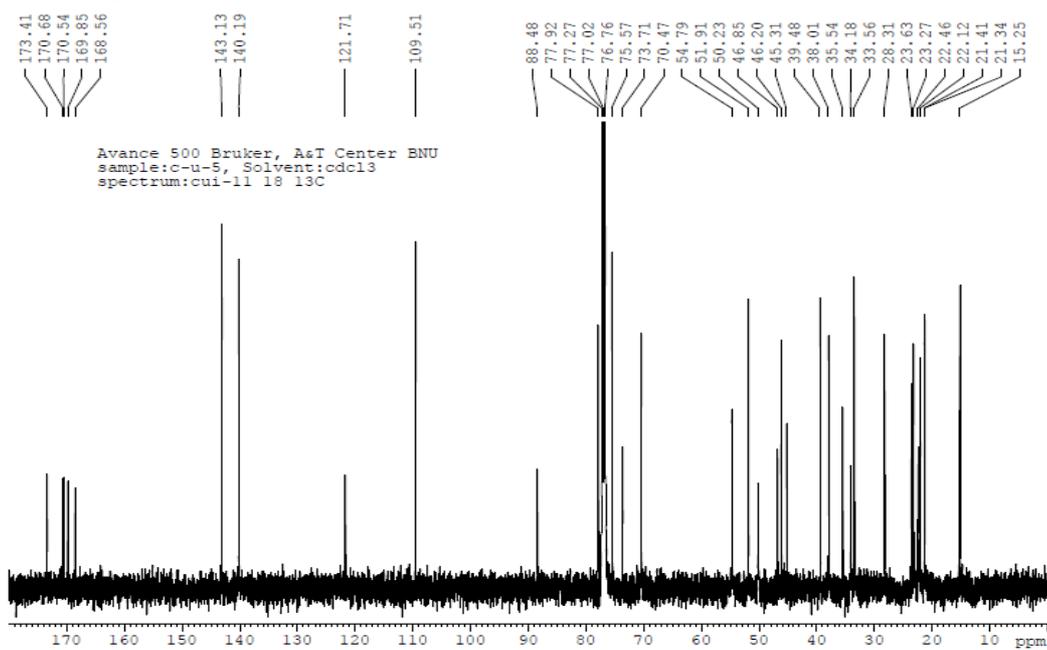
IR spectrum



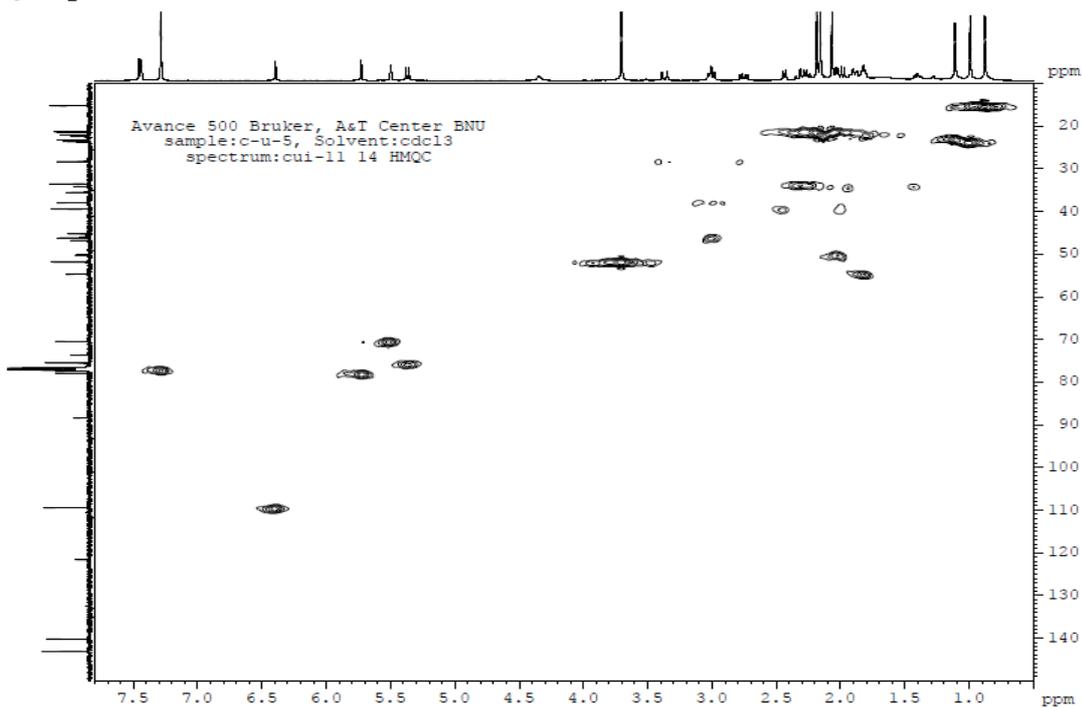
¹H NMR spectrum



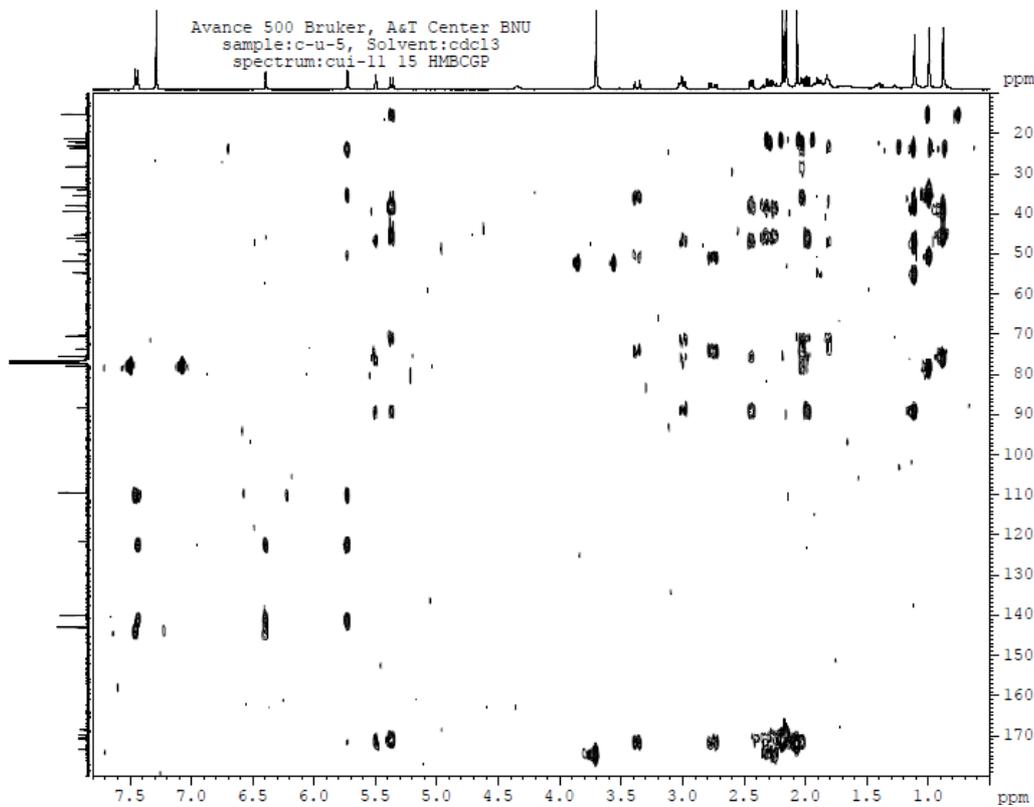
¹³C NMR spectrum



HMQC spectrum

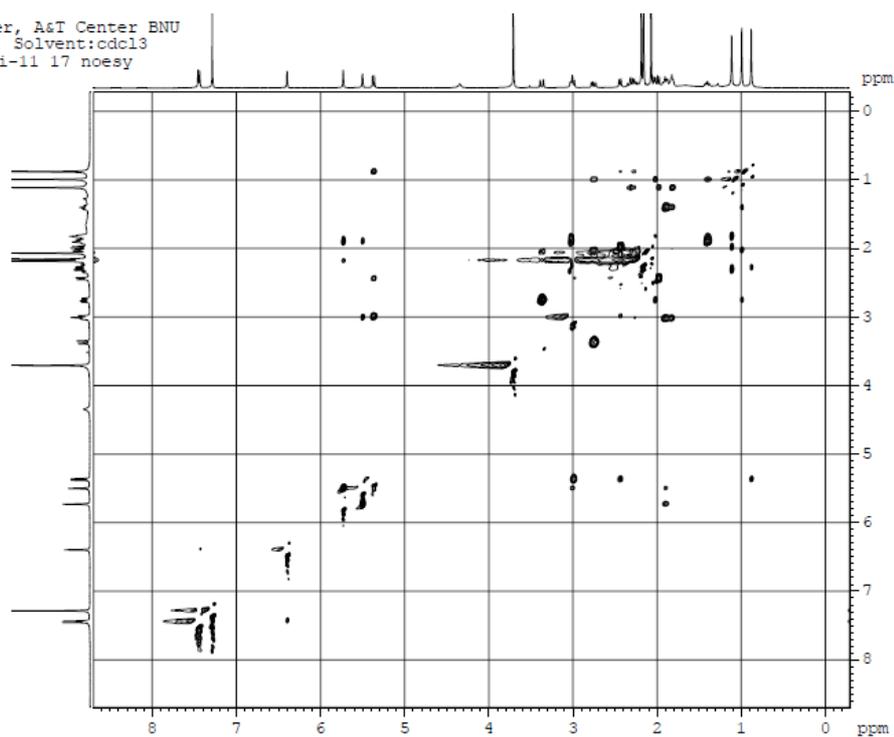


HMBC spectrum



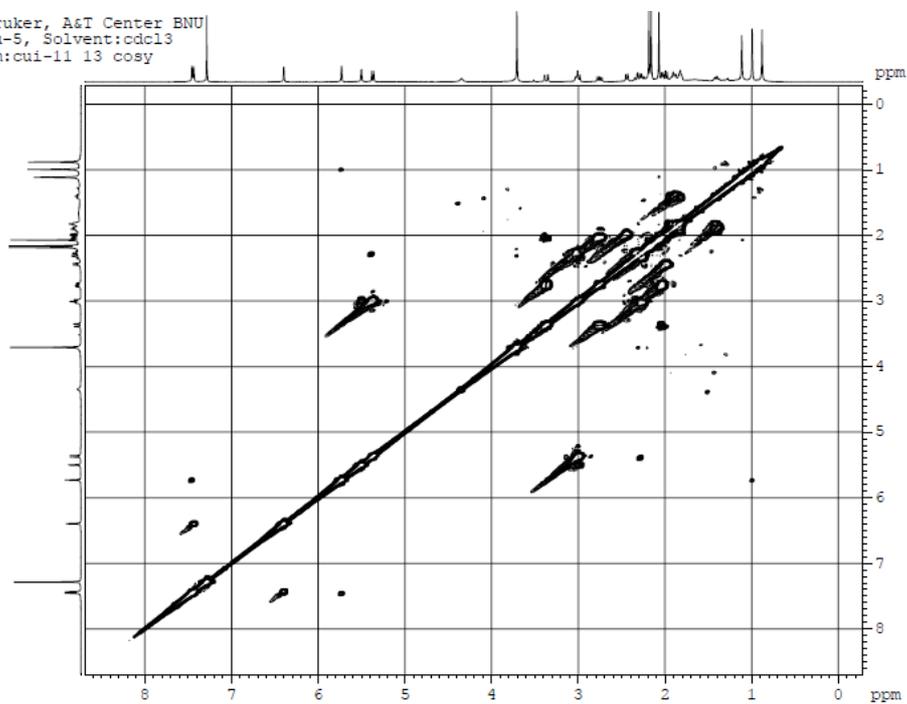
NOESY spectrum

Avance 500 Bruker, A&T Center BNU
sample:c-u-5, Solvent:cdcl3
spectrum:cui-11 17 noesy

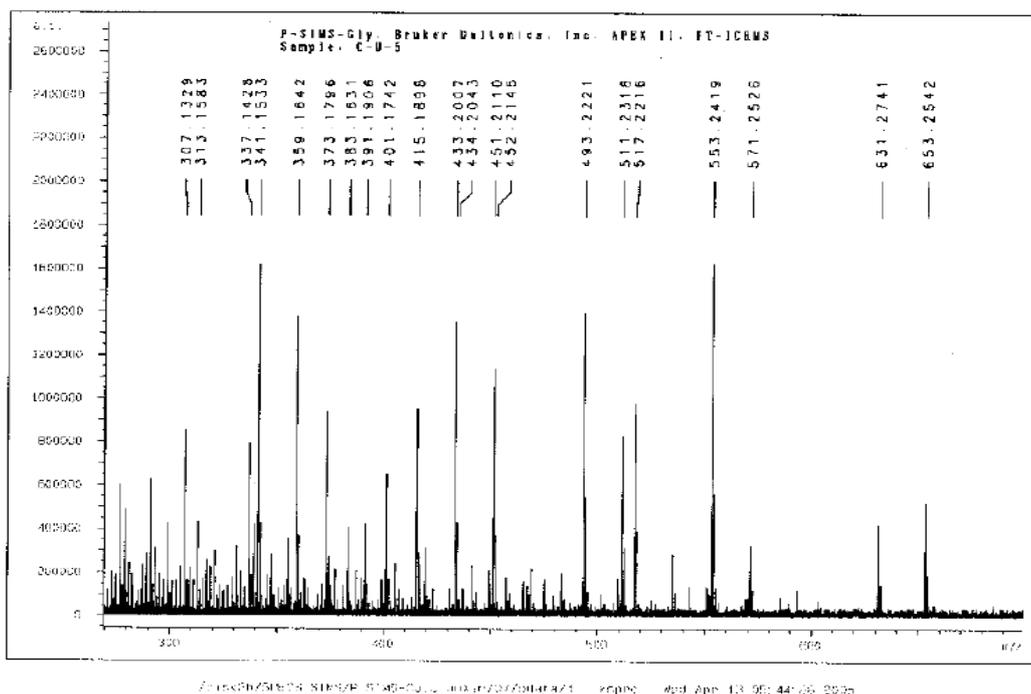


DQFCOSY spectrum

Avance 500 Bruker, A&T Center BNU
sample:c-u-5, Solvent:cdcl3
spectrum:cui-11 13 cosy



HRSIMS data



XMASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-CuiClanXin/07/pdata/1/ma:
 XMASS Mass Analysis Constraints

Ion mass [1] = 653.2542210
 Ion mass [2] = 631.2741490

Charge = +1
 Tolerance = 0.0100000

DBE min = -2
 DBE max = 200

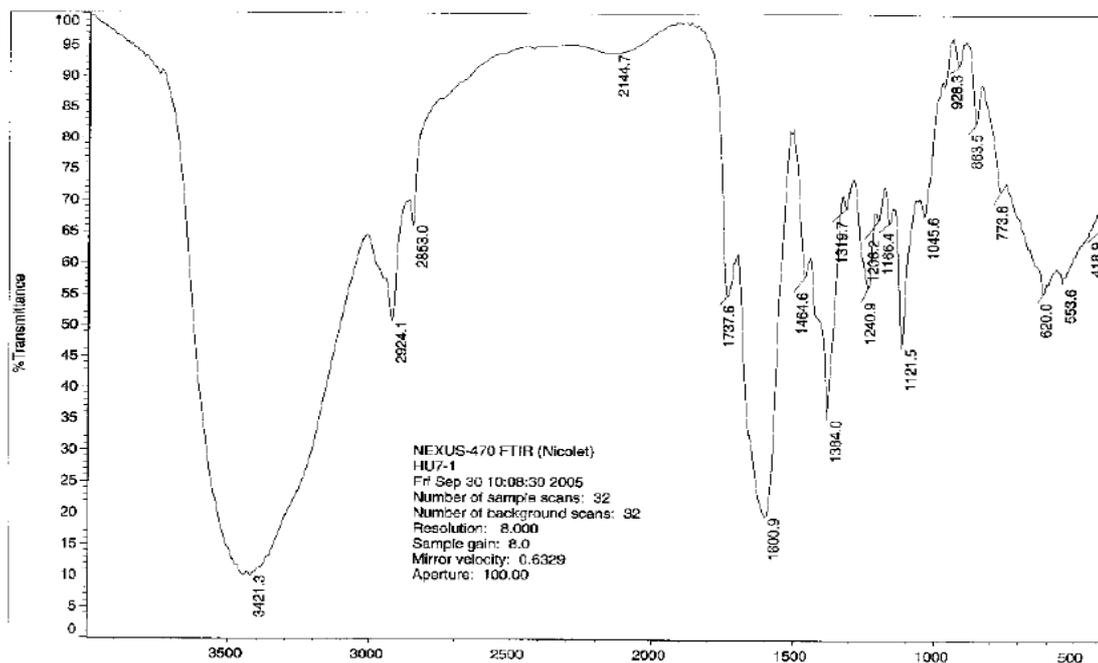
Max Candidates = 100

Atom	#(min, max)		Wt%(min, max)	
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H	25	60	0.00	100.00
O	5	20	0.00	100.00
Na	0	1	0.00	100.00

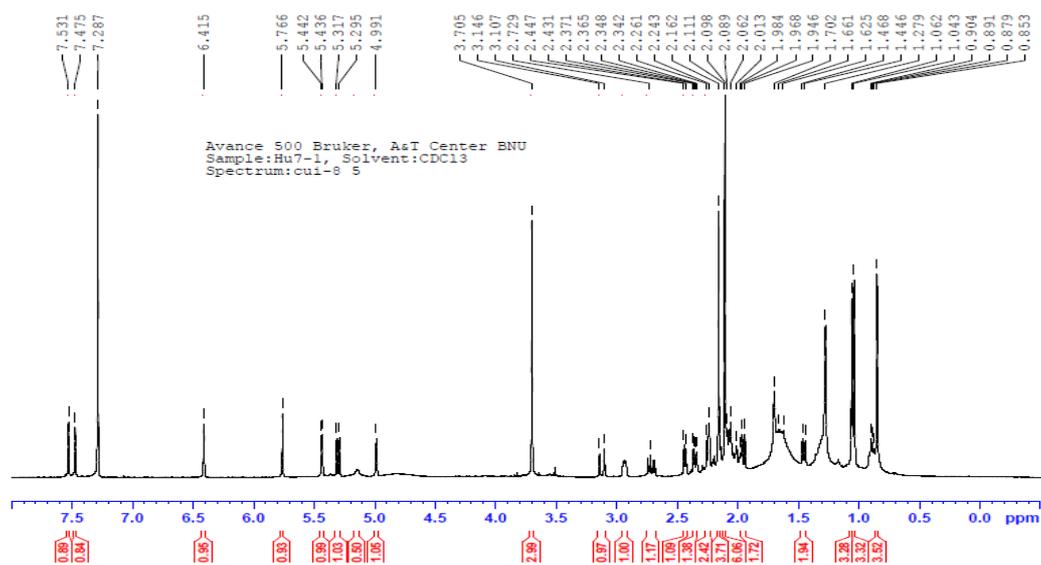
#	C	H	O	Na	mass	error
*** Mass Analysis for mass 653.2542210						
1	33	42	12	1	653.2568238	3.984e-06
*** Mass Analysis for mass 631.2741490						
1	33	43	12	0	631.2746784	1.155e-06
2	31	44	12	1	631.2724730	2.655e-06

3 Xylocarpin C (3)

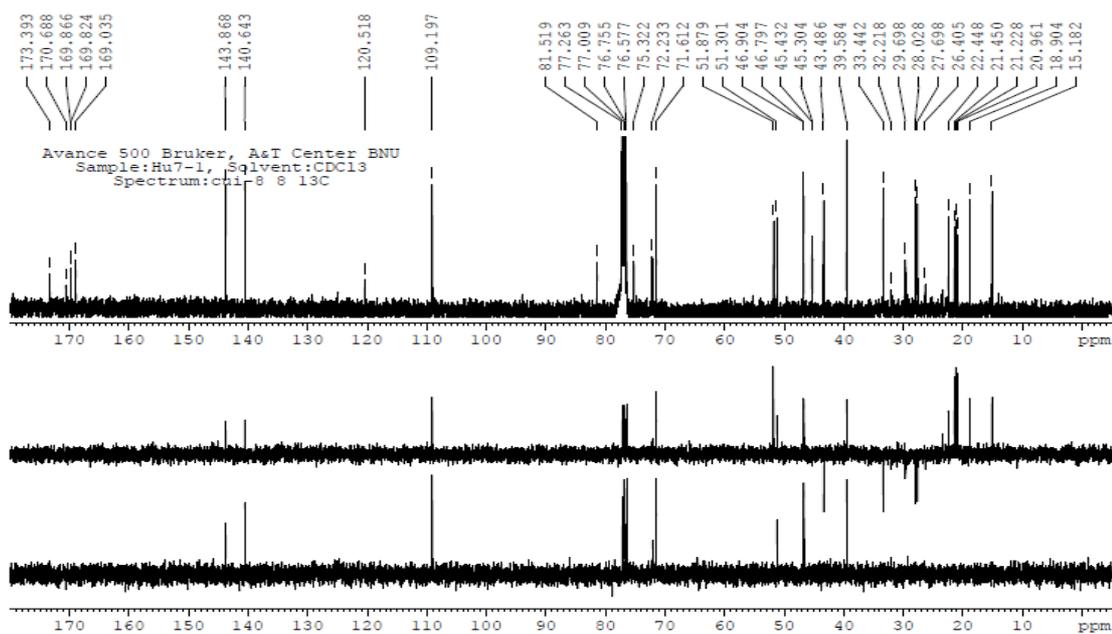
IR spectrum



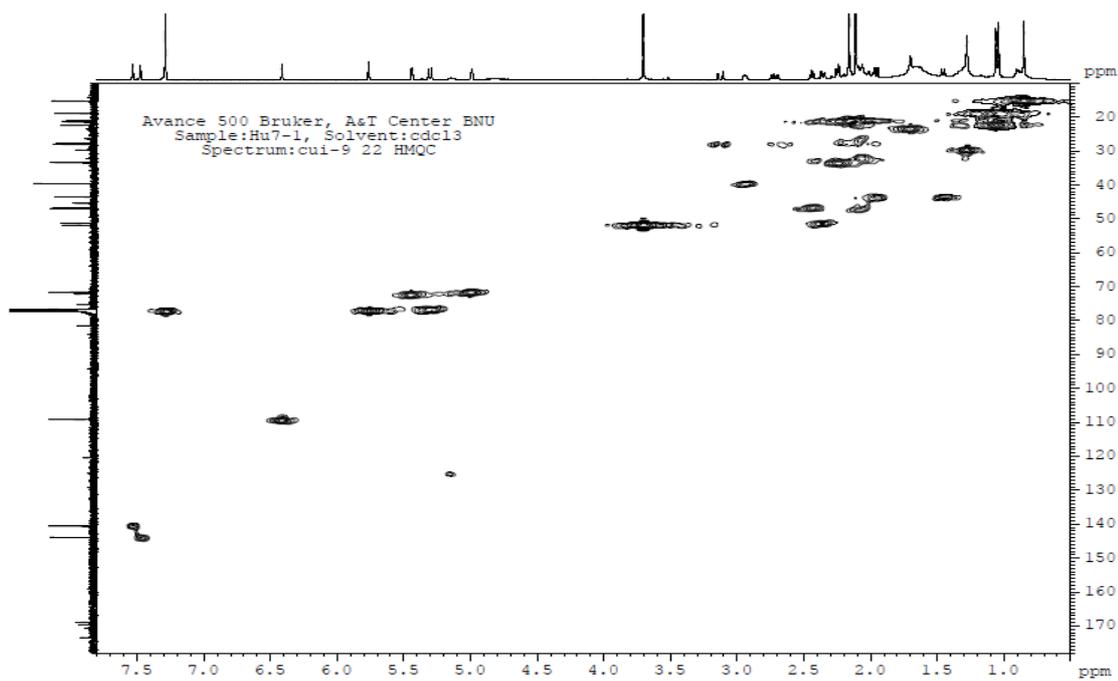
¹H NMR spectrum



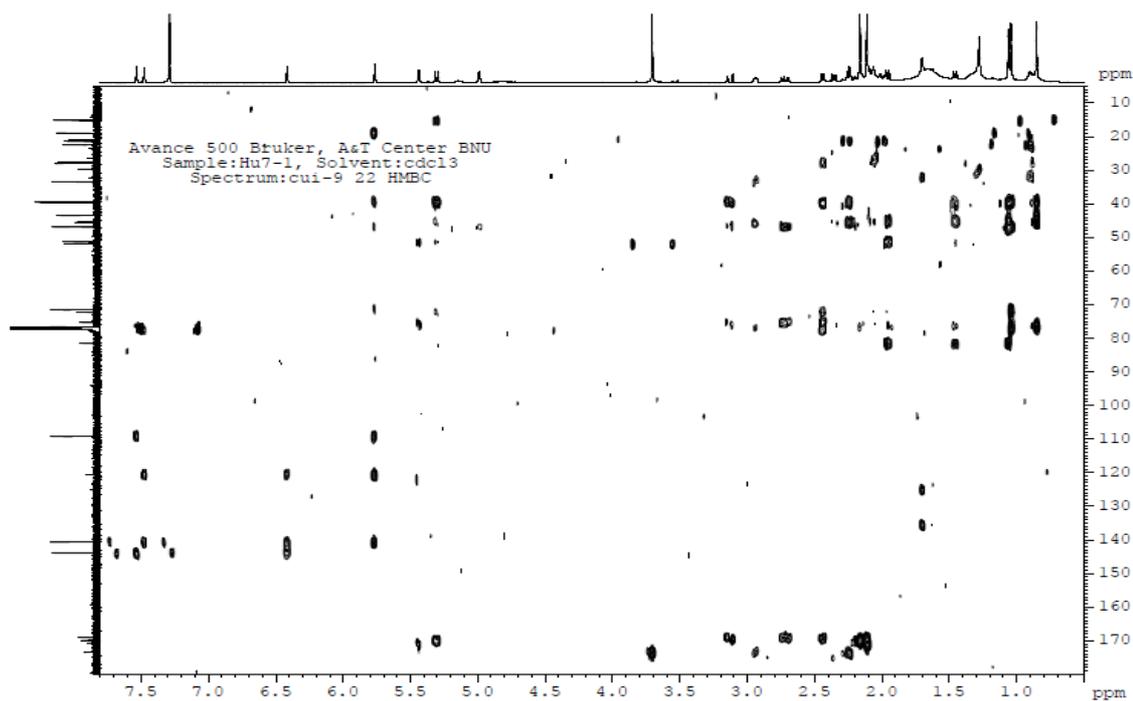
^{13}C and DEPT NMR spectra



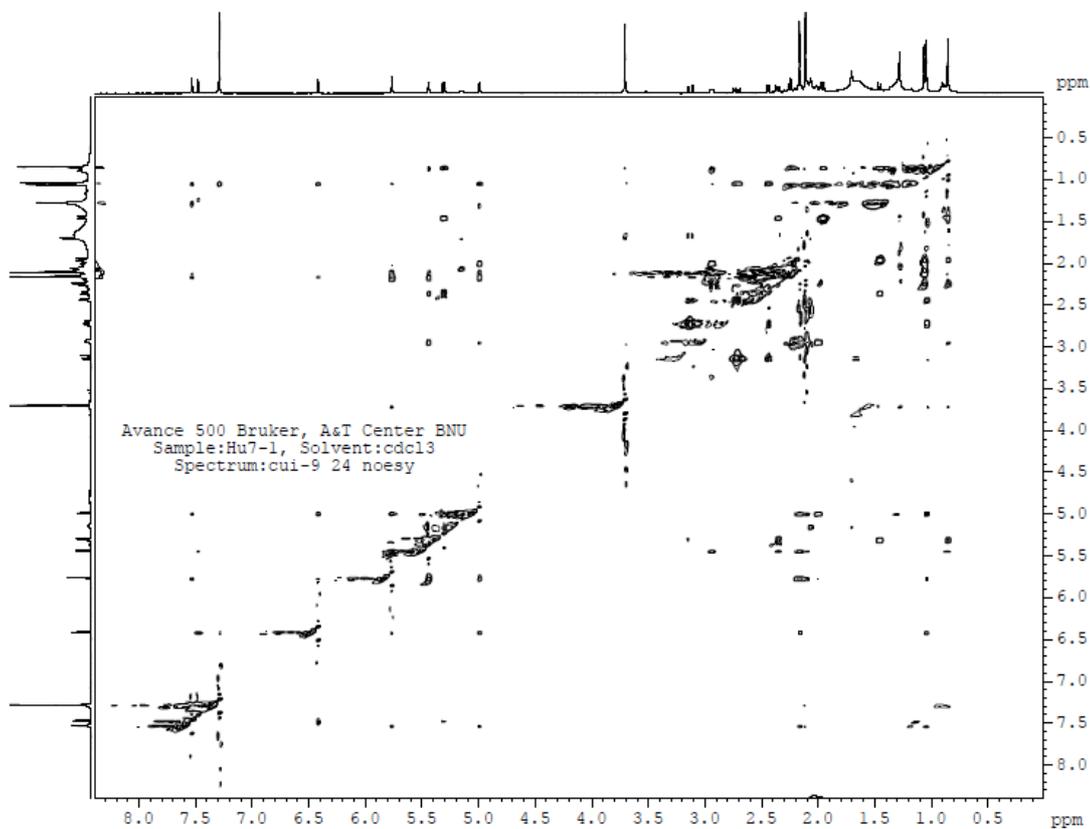
HMQC spectrum



HMBC spectrum



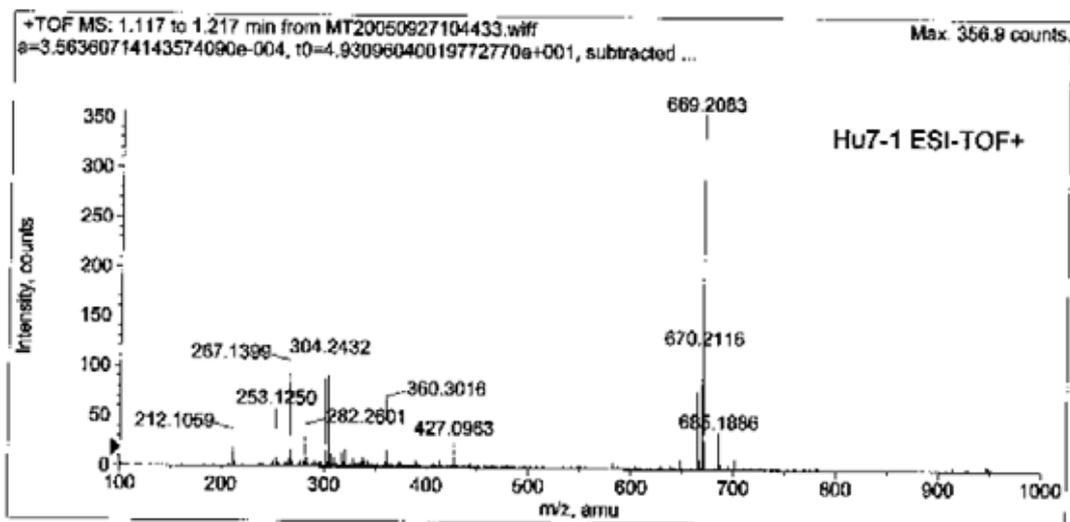
NOESY spectrum



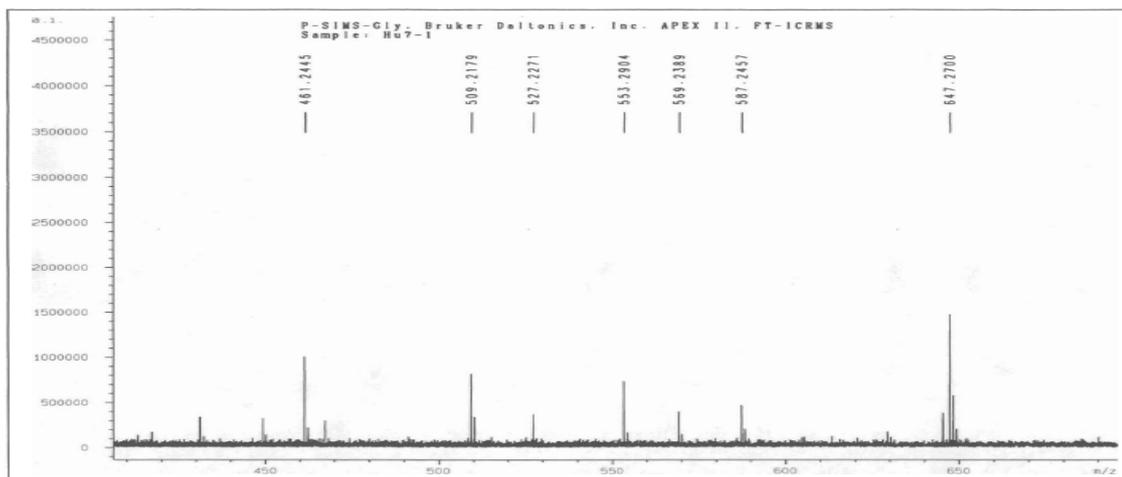
ESIMS spectrum

Workstation: QSTAR

Printing Date: Tuesday,
 September 27, 2005



HRSIMS data



XMASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-wangYan/111/pdata/1/
 XMASS Mass Analysis Constraints

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Tolerance = 0.0030000

DBE min = -2

DBE max = 200

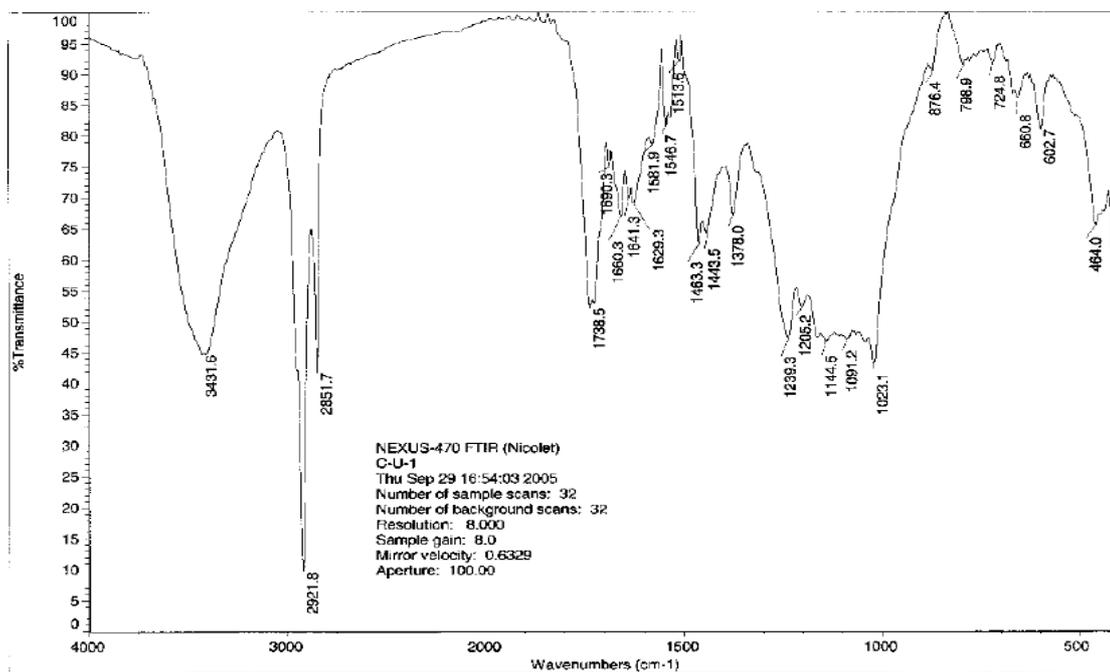
Max Candidates = 100

Atom	#(min, max)	Wt%(min, max)
C	30 36	0.00 100.00
H	30 50	0.00 100.00
O	5 15	0.00 100.00

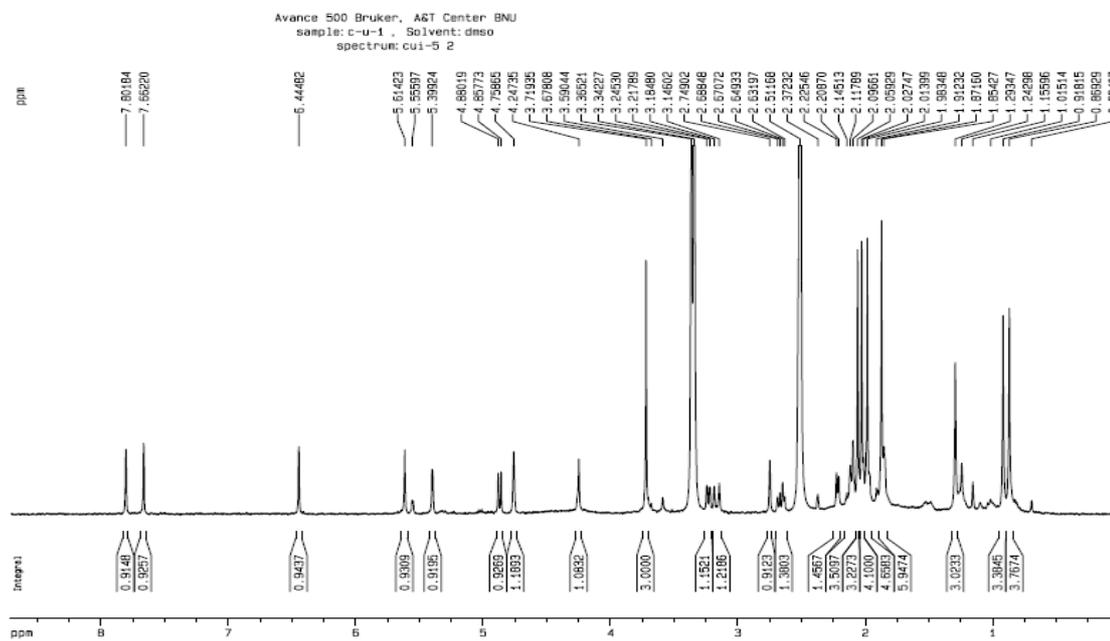
#	C	H	O	mass	error
***	Mass Analysis for mass 647.2699700				
1	33	43	13	647.2697925	2.742e-07

4 Xylocarpin D (4)

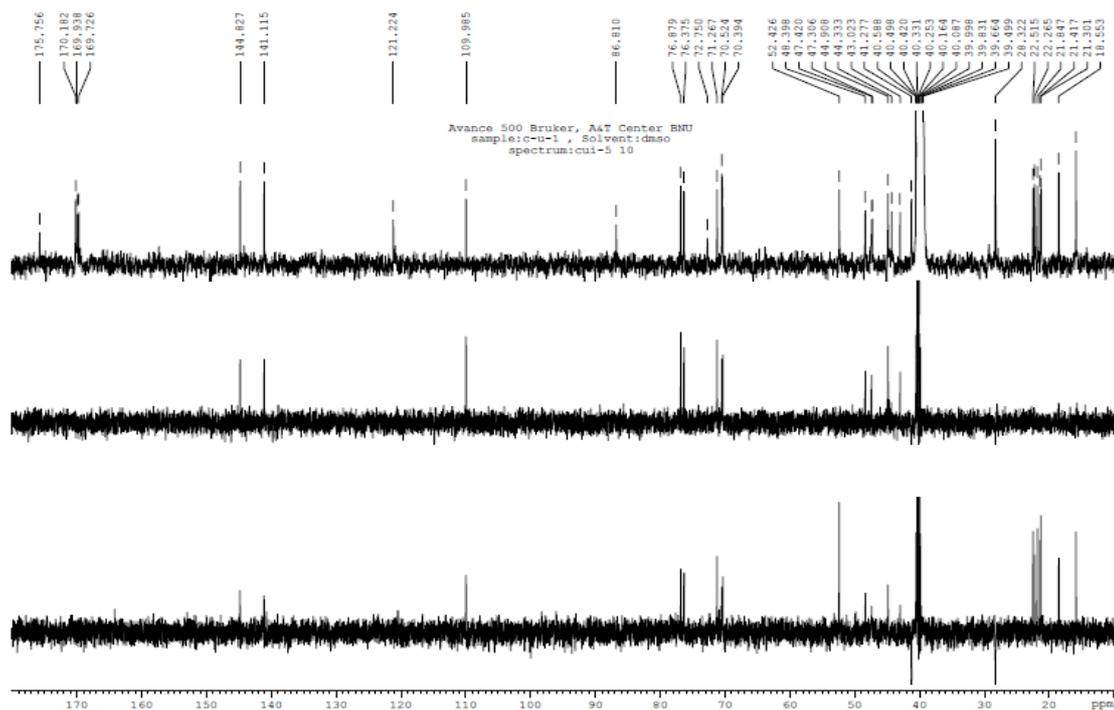
IR spectrum



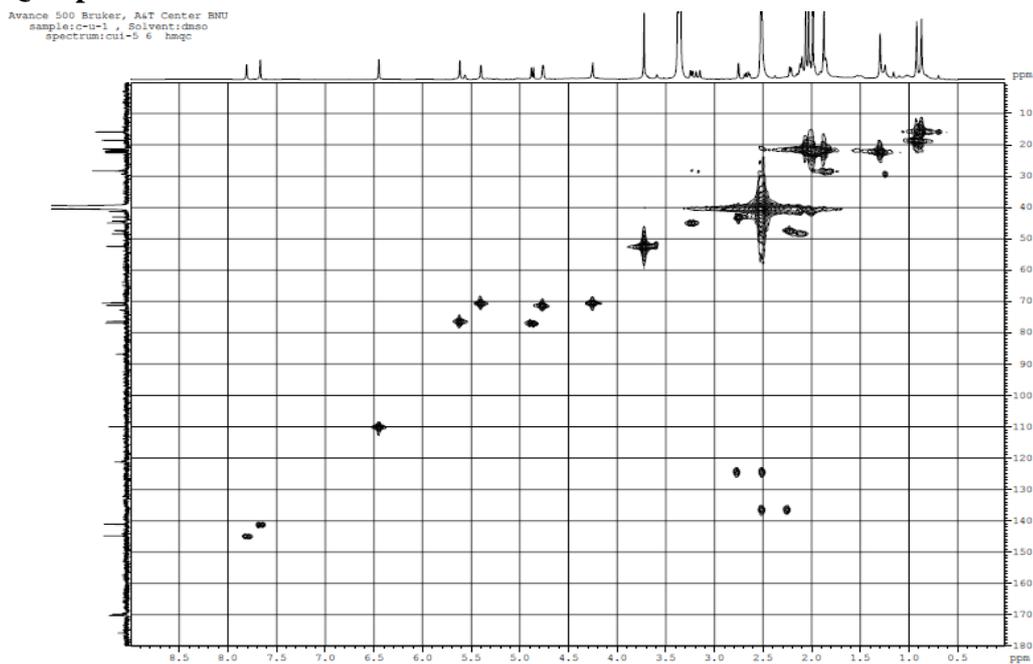
¹H NMR spectrum



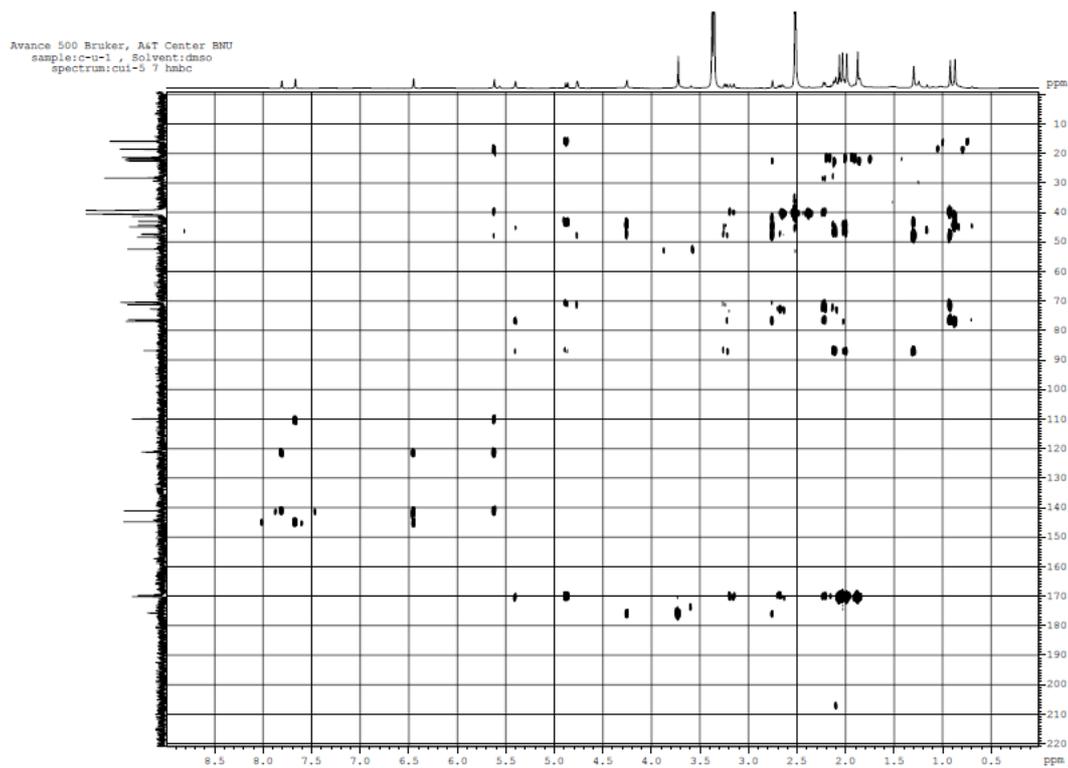
¹³C and DEPT NMR spectra



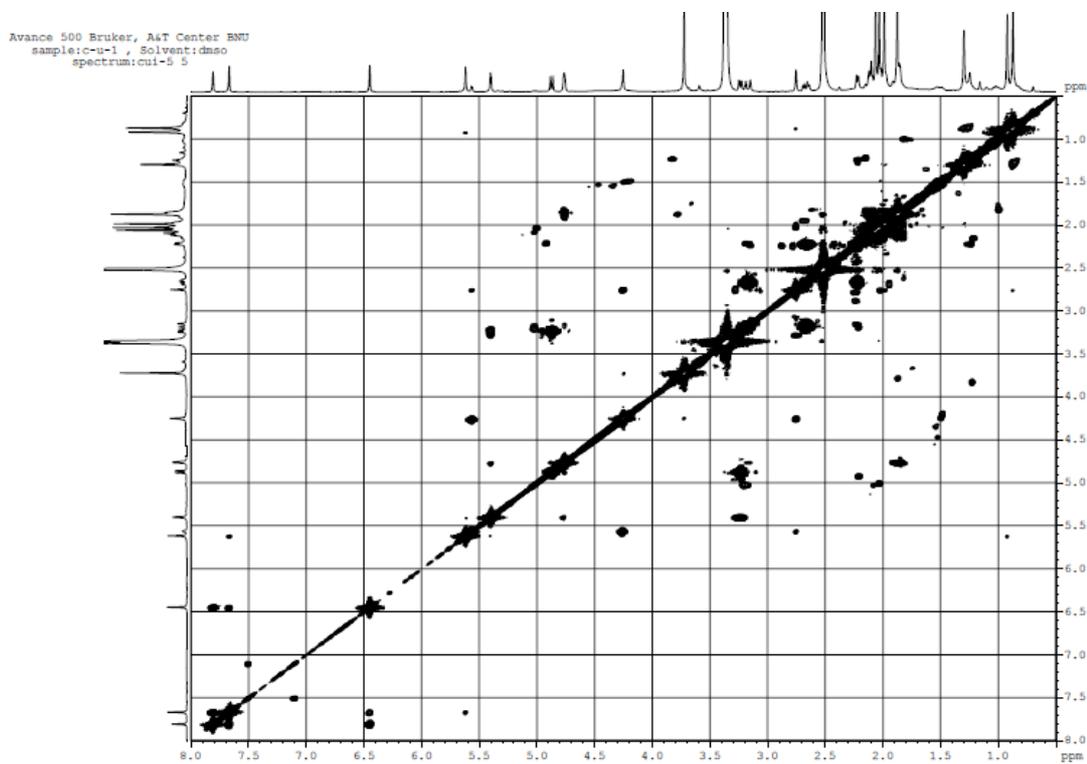
HMQC spectrum



HMBC spectrum



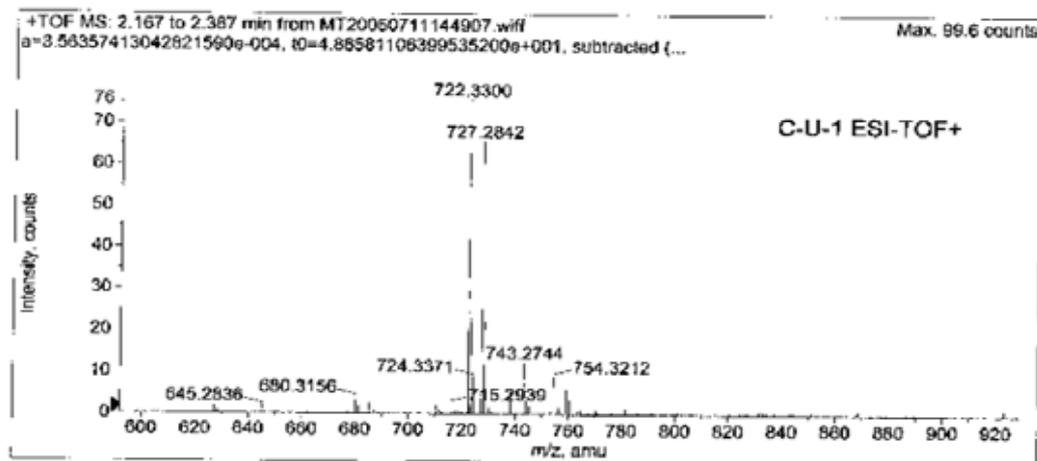
DQF-COSY spectrum



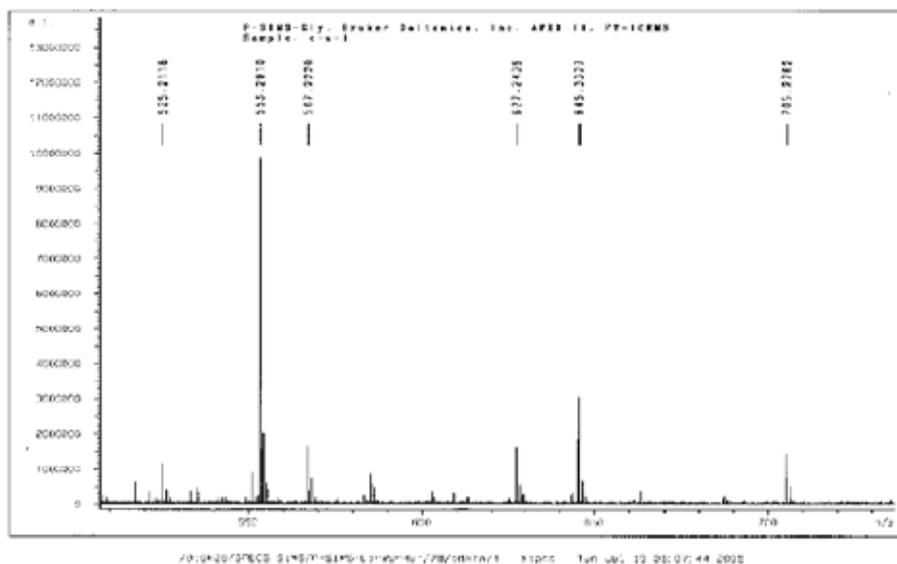
ESIMS spectrum

Workstation: QSTAR

Printing Date: Monday,
 July 11, 2005



HRSIMS data



\MASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-LinWenHan/72/pdata/1/massana
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Ion mass = 705.2761620

Charge = +1

Tolerance = 0.0030000

DBE min = -2

DBE max = 200

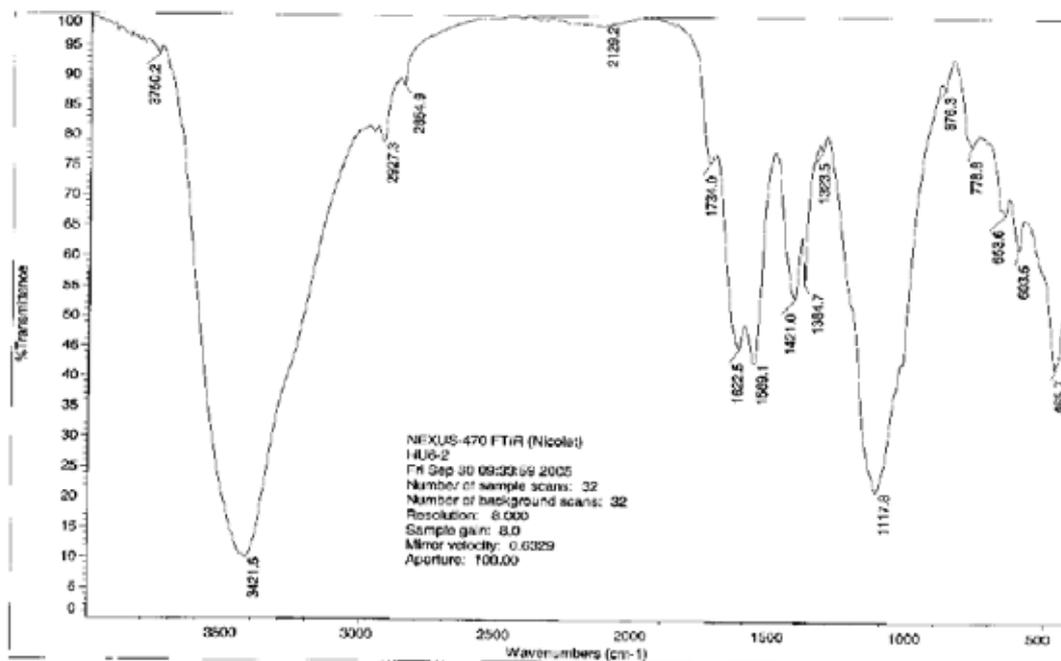
Max Candidates = 100

Atom	#(min, max)	Wt%(min, max)
C	30 40	0.00 100.00
H	30 70	0.00 100.00
O	10 20	0.00 100.00

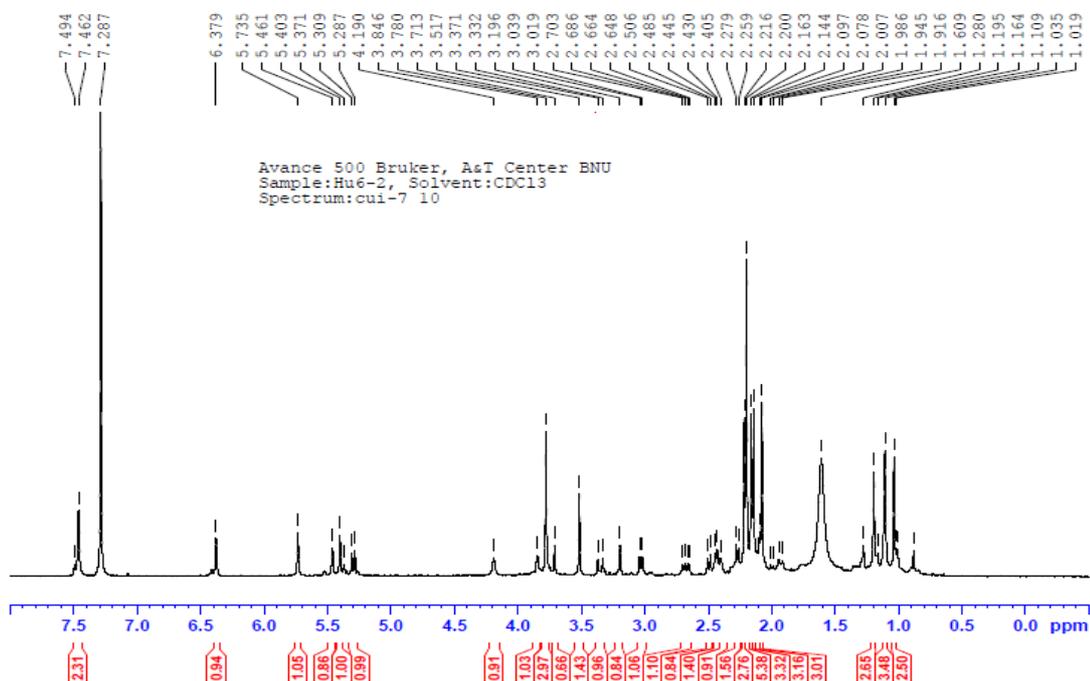
C H O mass error
 *** Mass Analysis for mass 705.2761620
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5 Xylocarpin E (5)

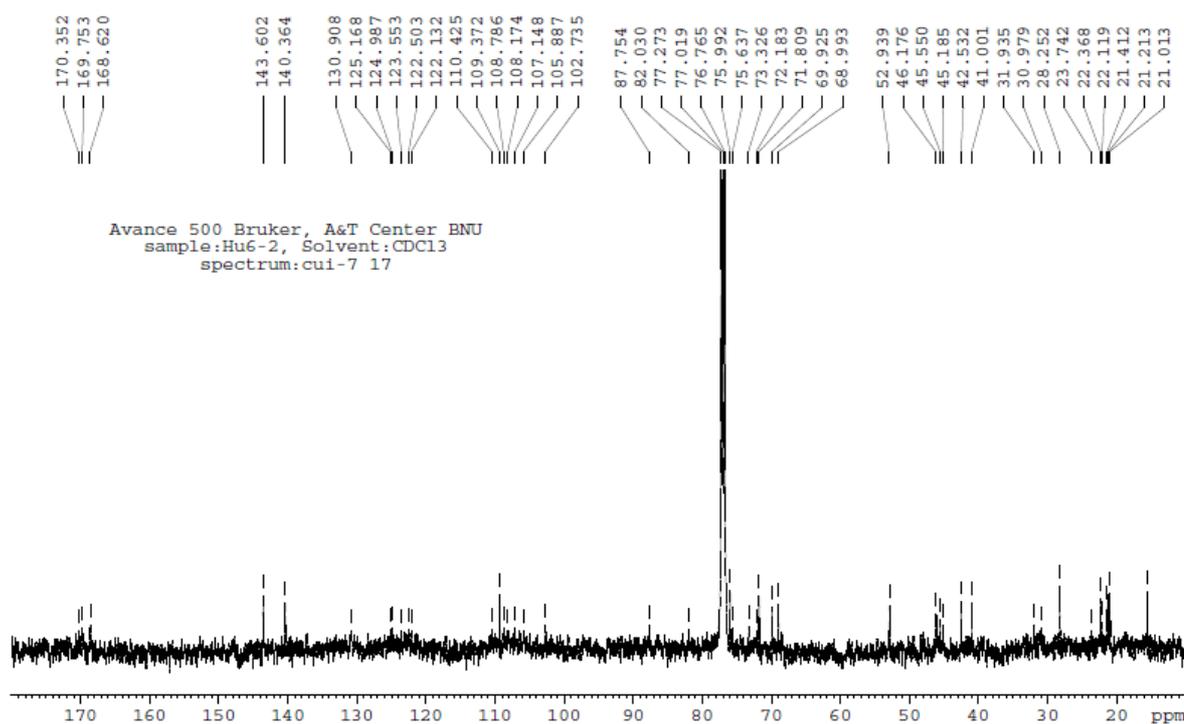
IR spectrum



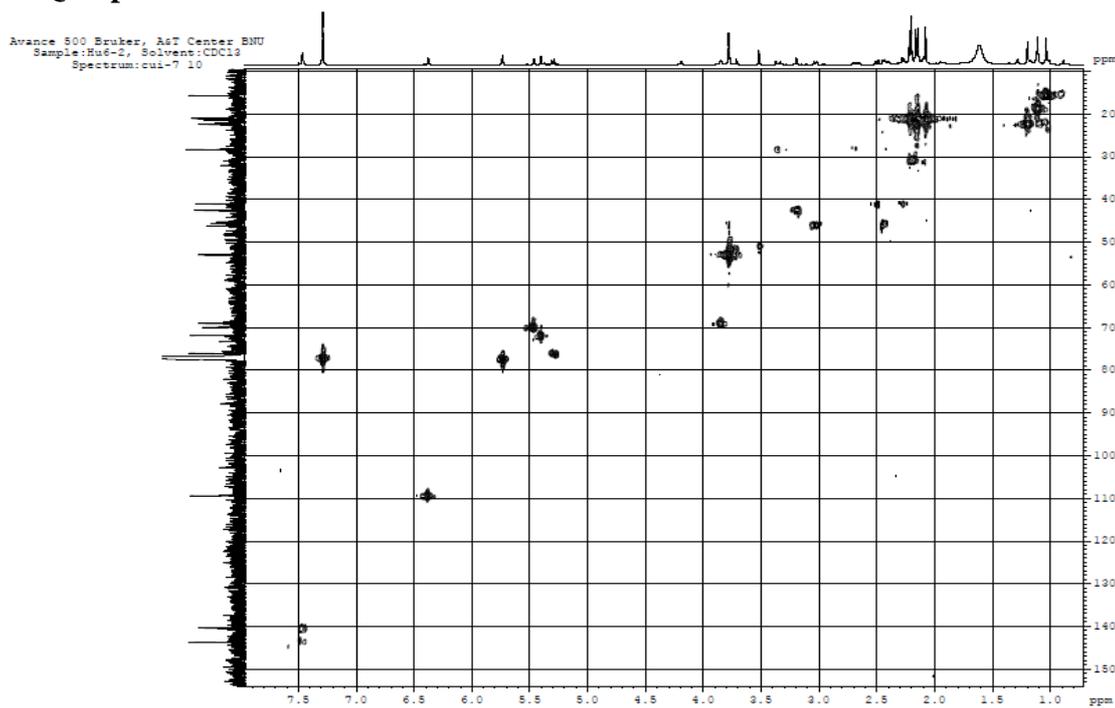
¹H NMR spectrum



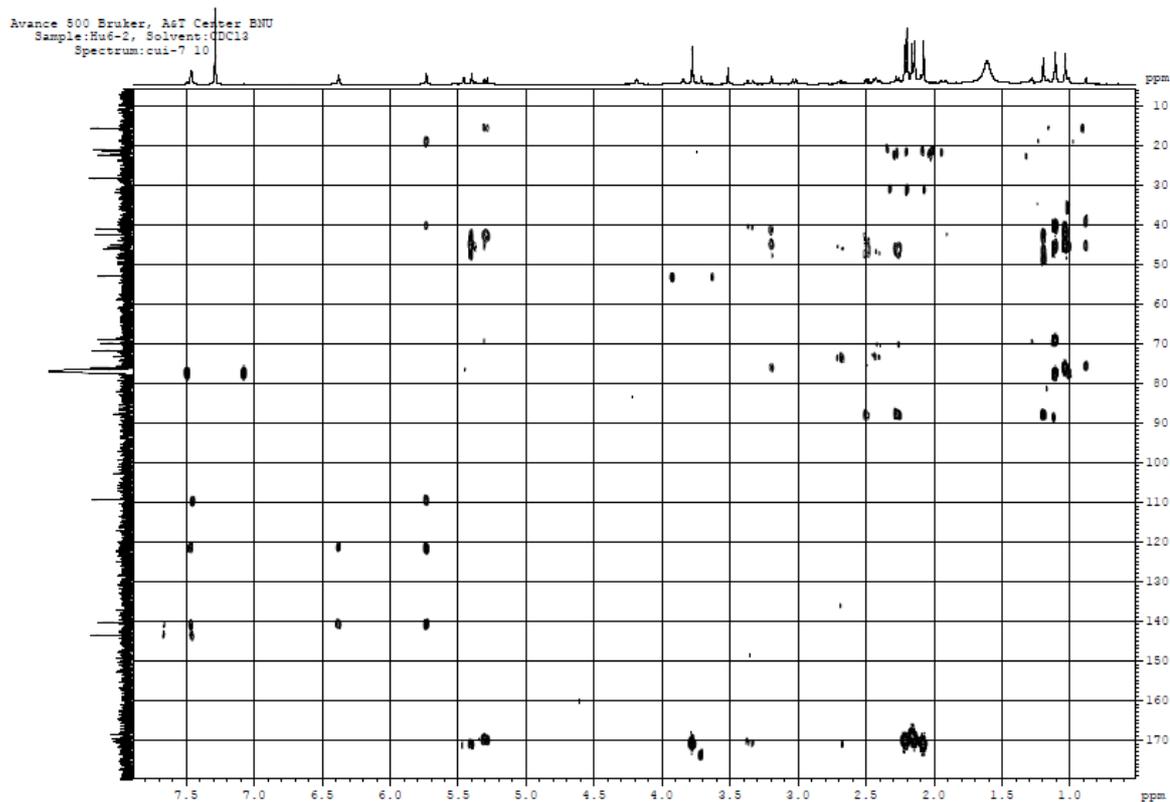
¹³C NMR spectrum



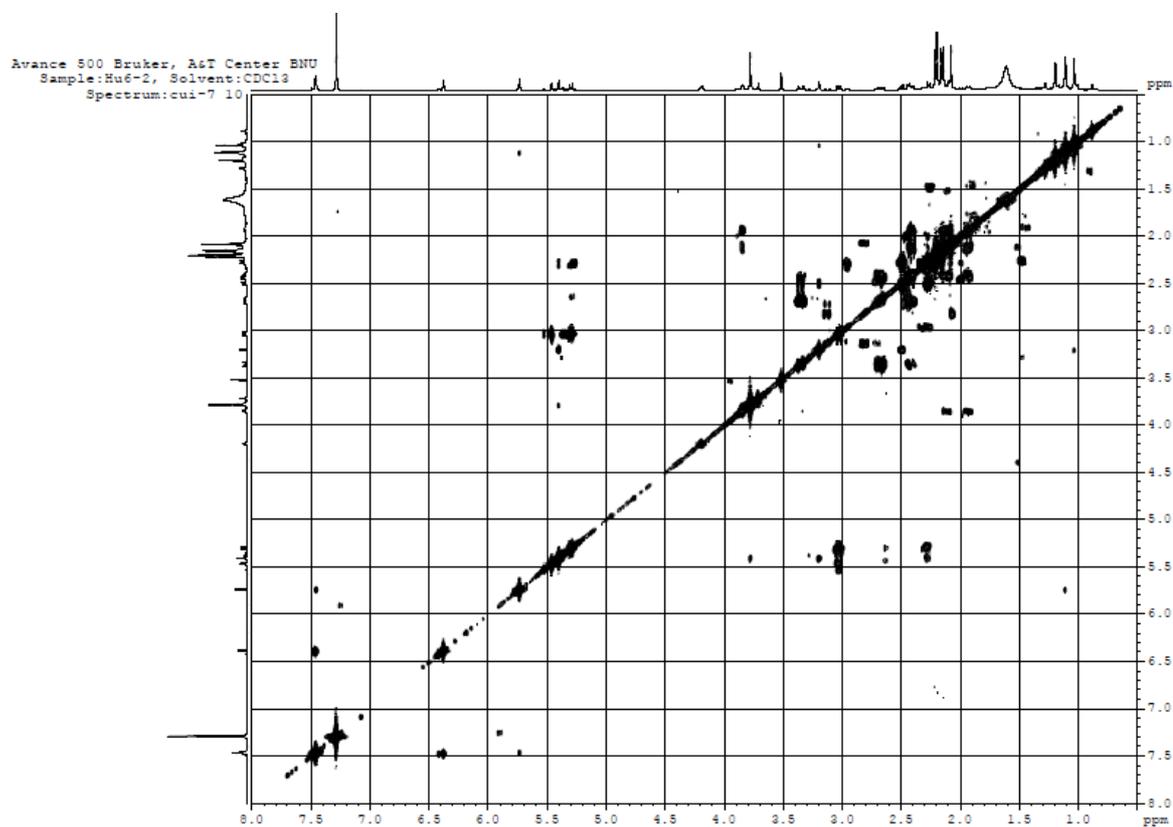
HMQC spectrum



HMBC spectrum



DQF COSY



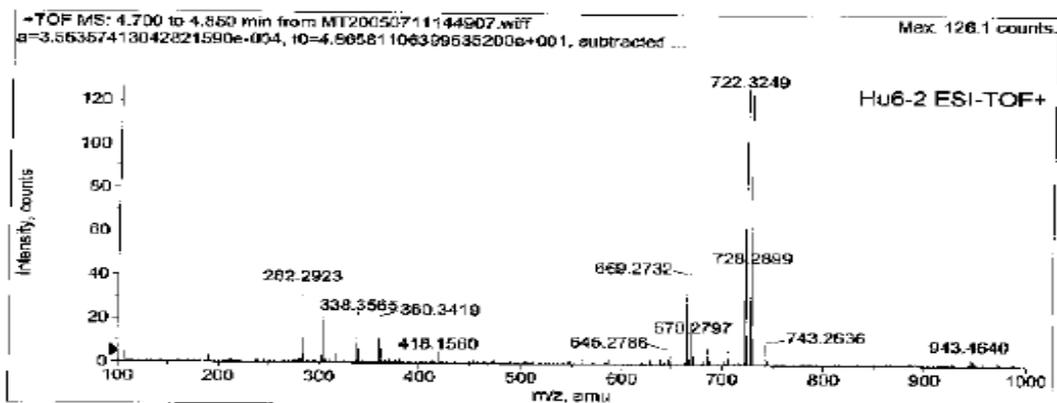
ESIMS spectrum

Acq. File:
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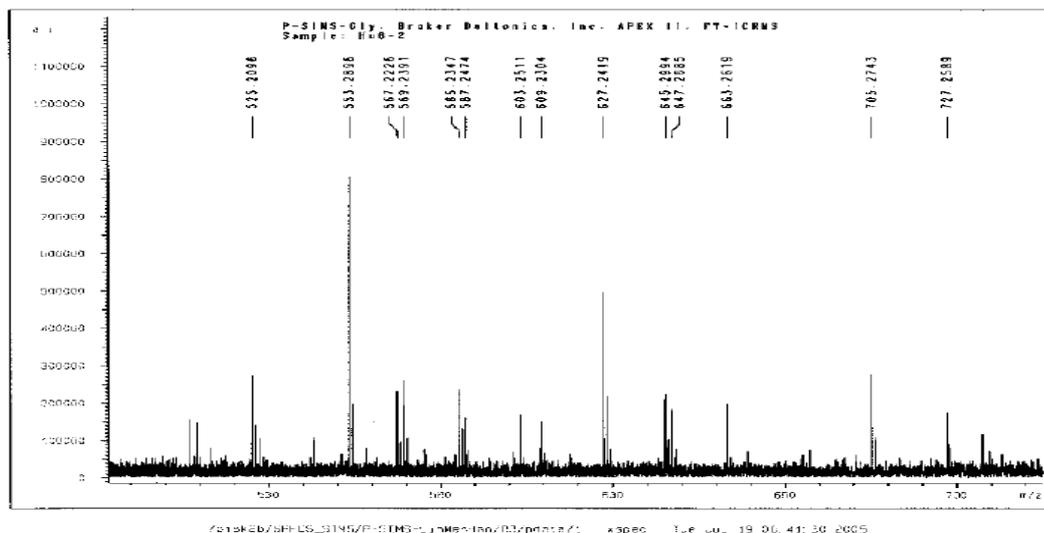
Sample Name: 20050711

Workstation: QSTAR

Printing Date: Monday,
 July 11, 2005



HRSIMS data



XMAS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-LinWenHan/83/pdata/...
 XMAS Mass Analysis Constraints

Ion mass = 705.2743130

Charge = +1
 Tolerance = 0.0050000

DBE min = -2
 DBE max = 200

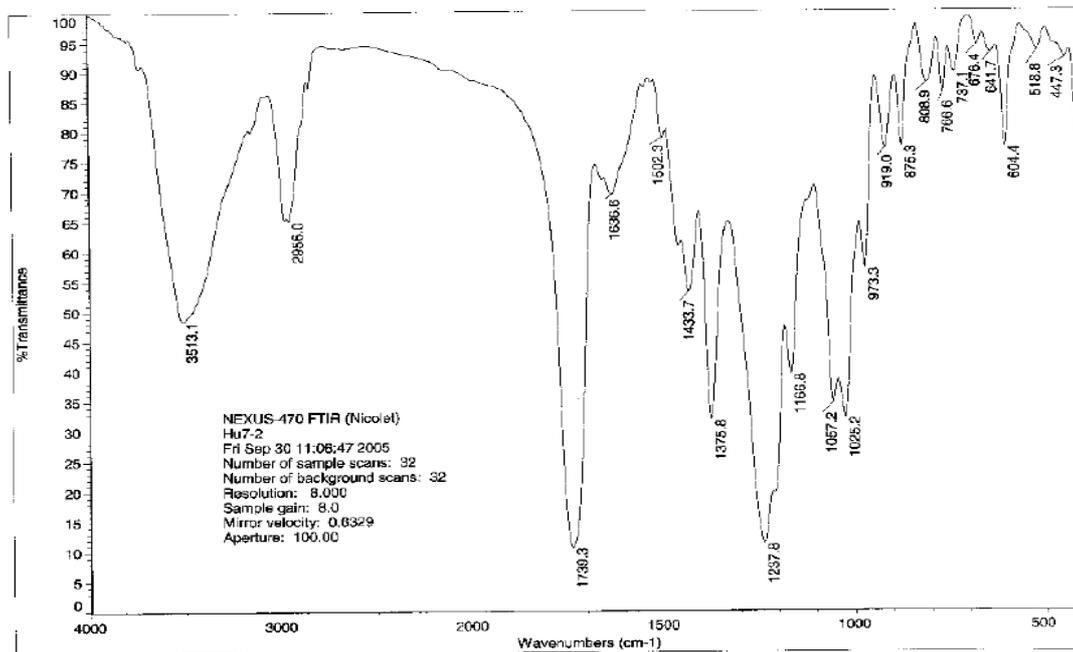
Max Candidates = 100

Atom	# (min, max)	Wt% (min, max)
C	30 40	0.00 100.00
H	30 70	0.00 100.00
O	10 20	0.00 100.00

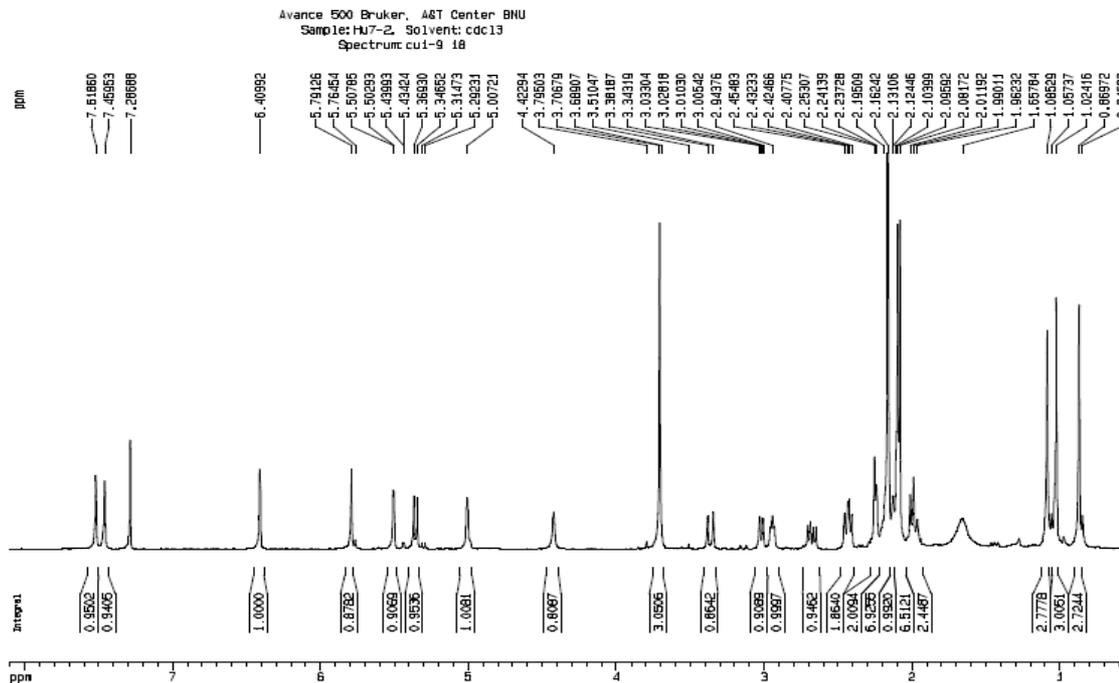
#	C	H	O	mass	error
***	Mass Analysis for mass 705.2743130				
1	35	45	15	705.2752699	1.357e-06

6 6-dehydroxyxylocarpin D (6)

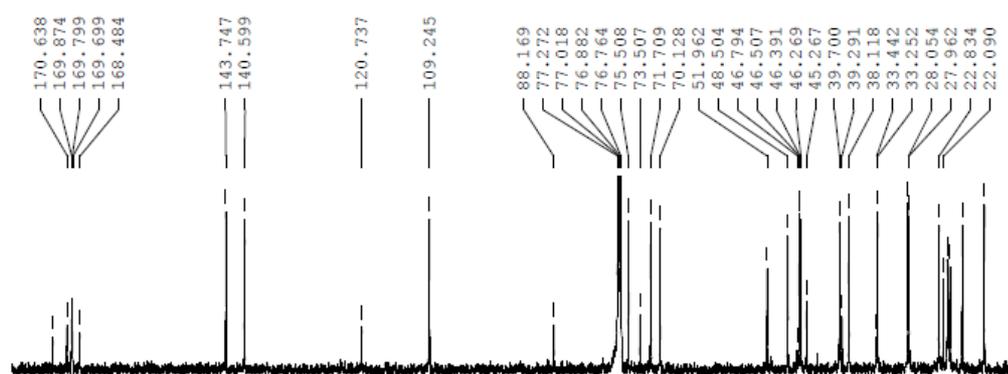
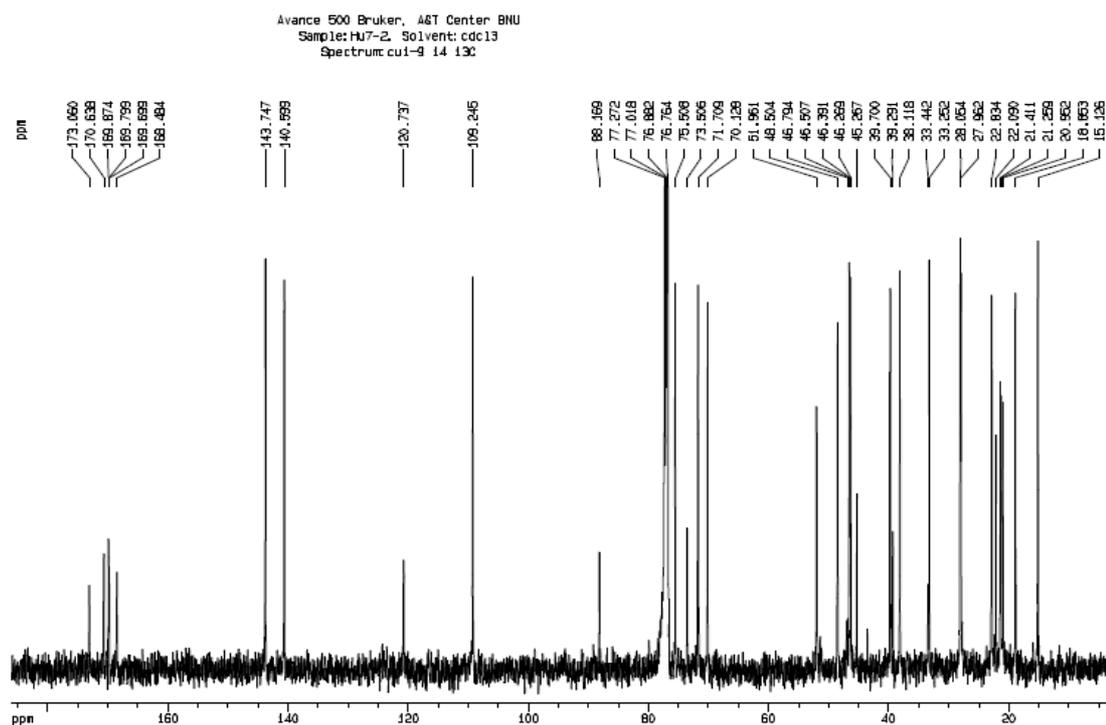
IR spectrum



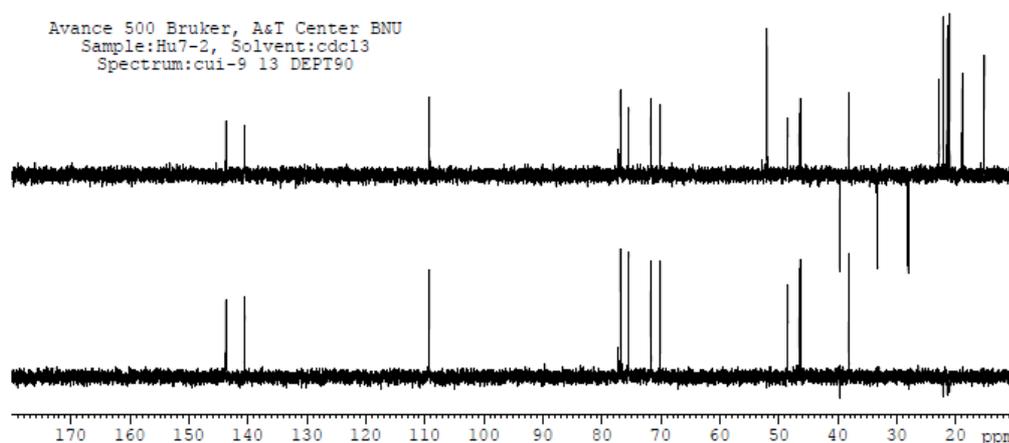
¹H NMR spectrum



¹³C and DEPT NMR spectra

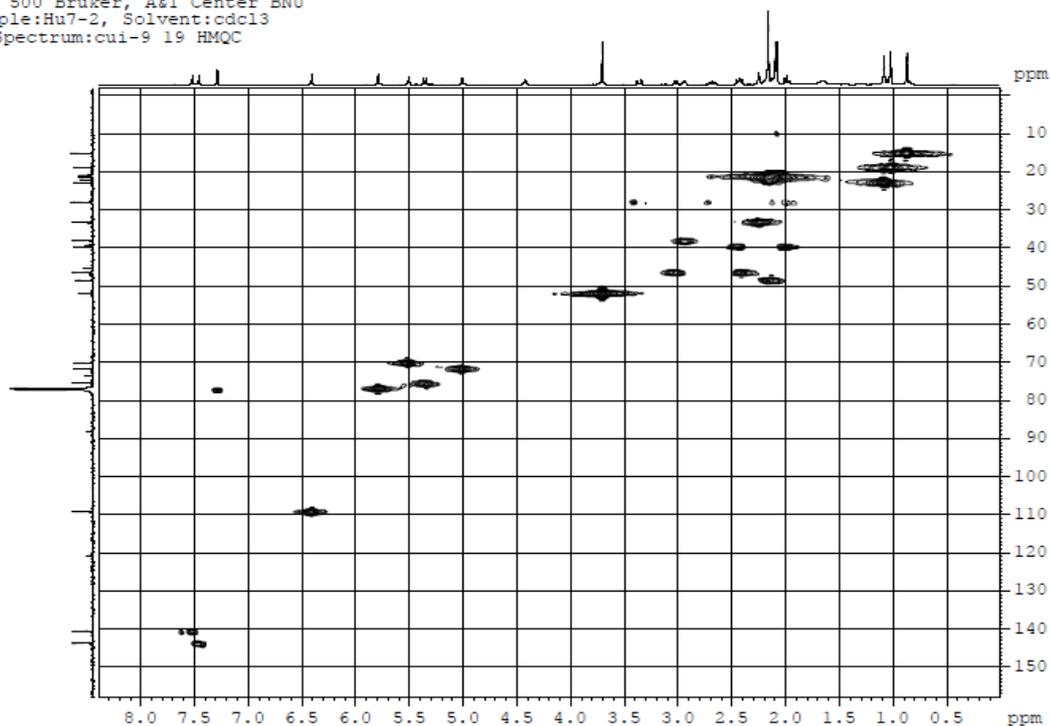


Avance 500 Bruker, A&T Center BNU
Sample:Hu7-2, Solvent:cdcl3
Spectrum:cui-9 13 DEPT90



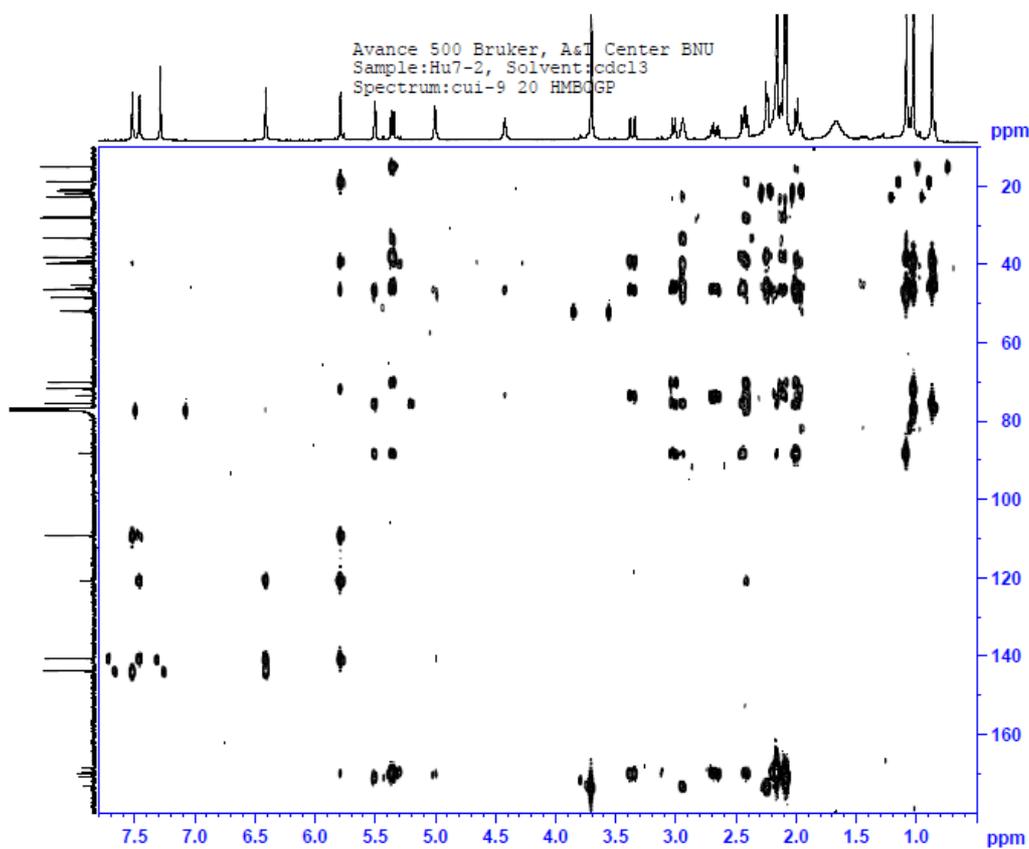
HMQC spectrum

Avance 500 Bruker, AsT Center BNU
Sample:Hu7-2, Solvent:cdcl3
Spectrum:cui-9 19 HMQC

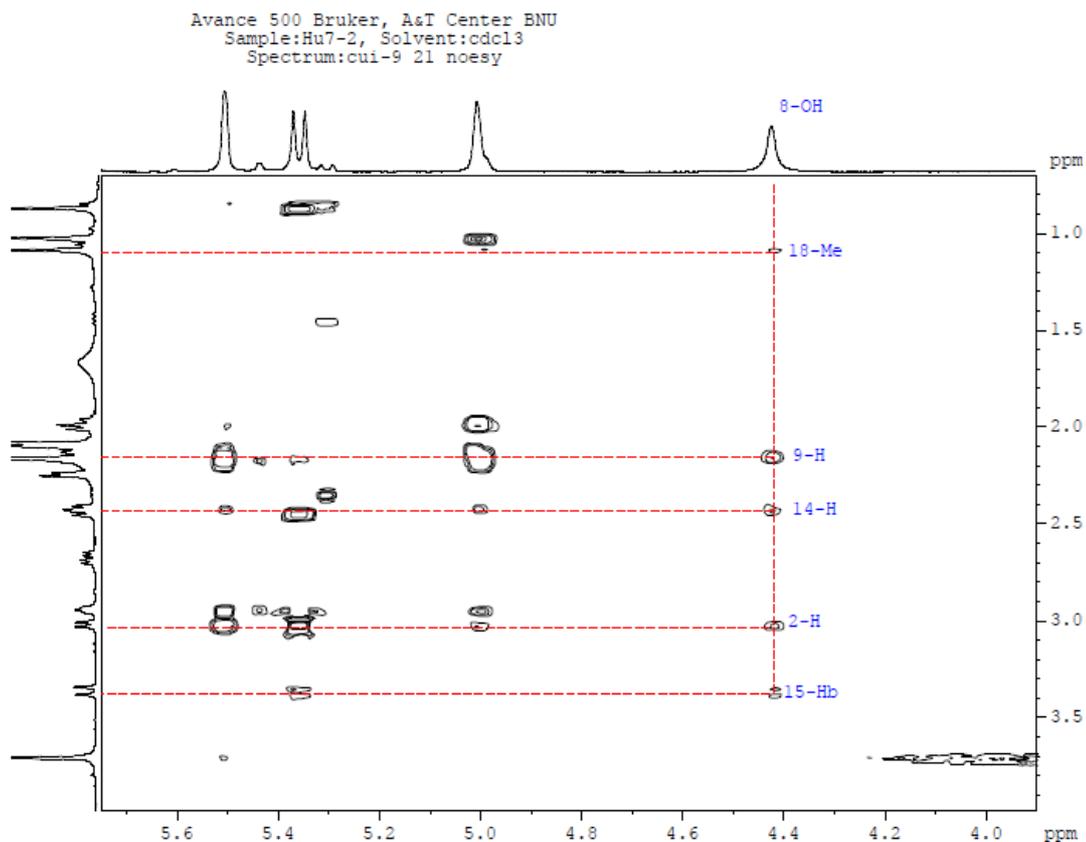


HMBC spectrum

Avance 500 Bruker, AsT Center BNU
Sample:Hu7-2, Solvent:cdcl3
Spectrum:cui-9 20 HMBCGP



NOESY spectrum



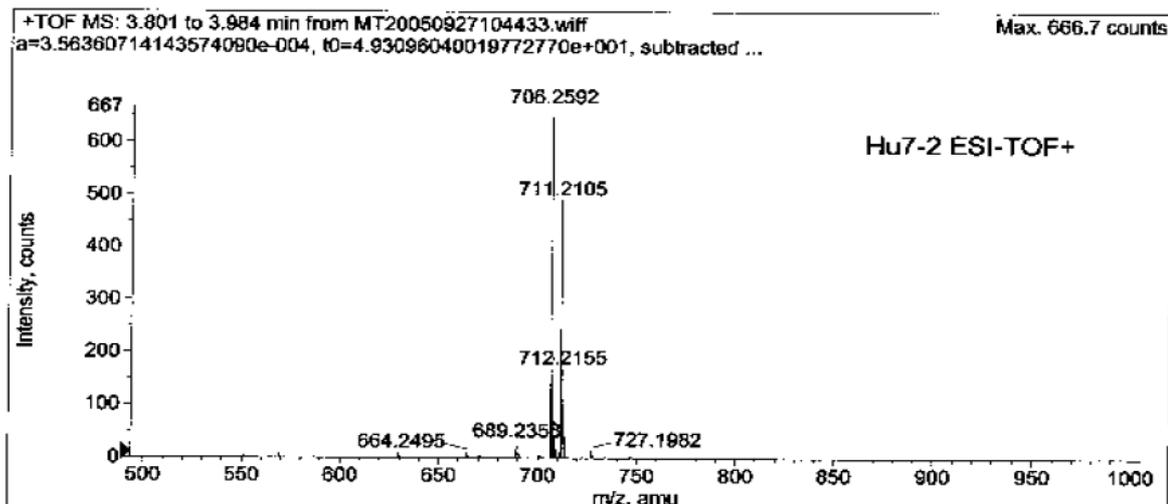
ESIMS spectrum

Acq. File:
MT20050927104433.wiff

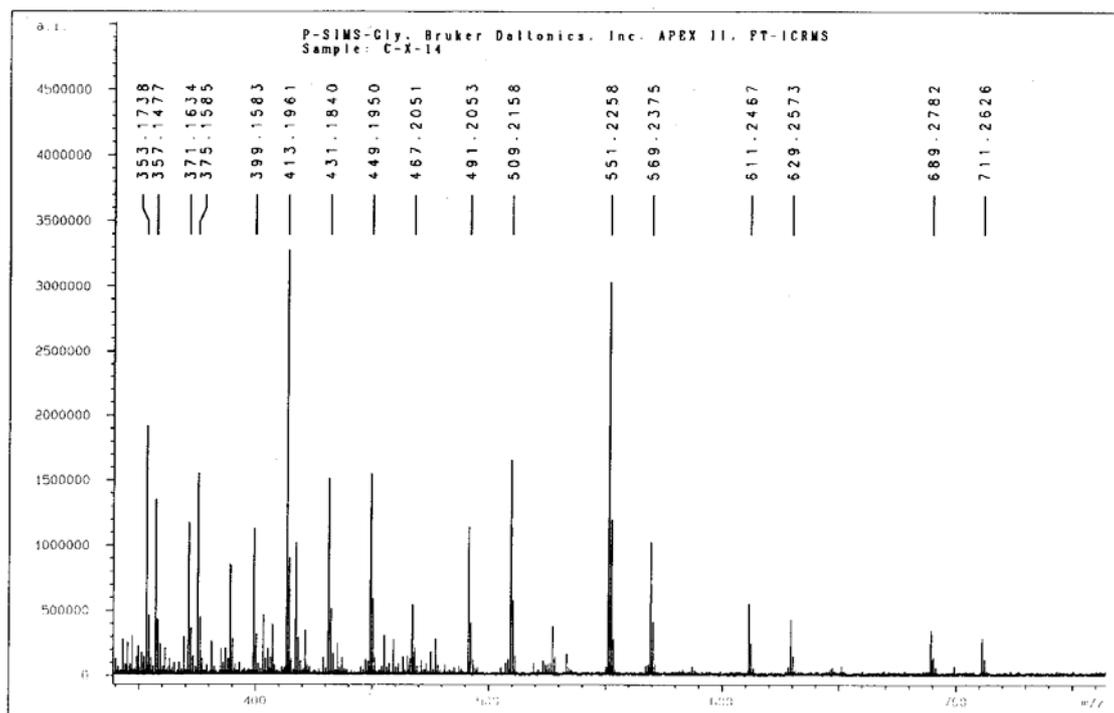
Sample Name:

Workstation: QSTAR

Printing Date: Tuesday,
September 27, 2005



HRSIMS data



XMASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-LinWenHan/27,
 XMASS Mass Analysis Constraints

Ion mass [1] = 689.2782480
 Ion mass [2] = 711.2626170

Charge = +1
 Tolerance = 0.0050000

DBE min = -2
 DBE max = 200

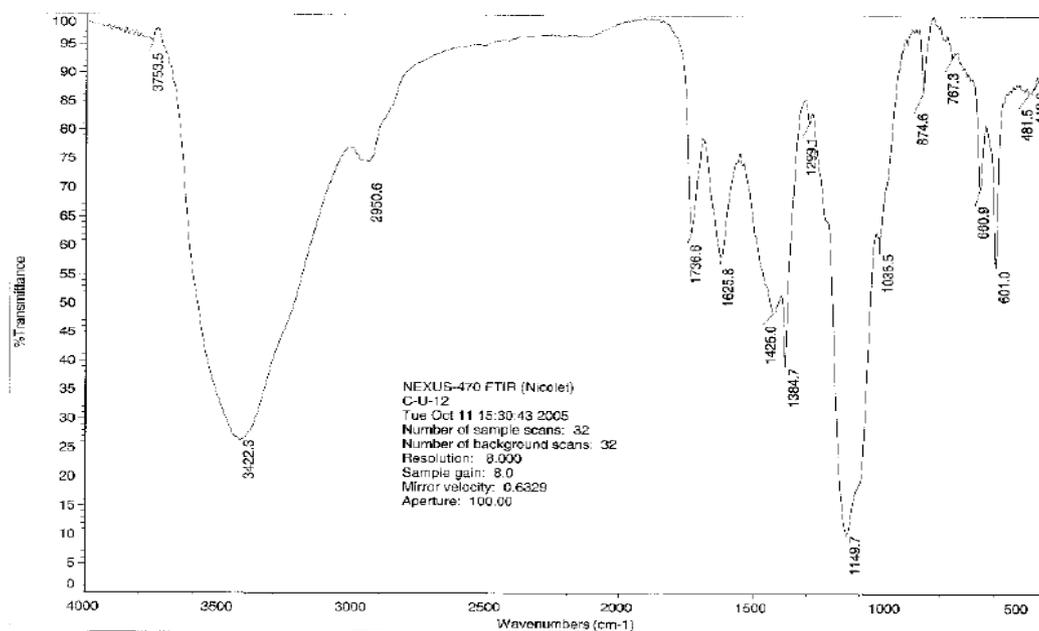
Max Candidates = 100

Atom	#(min, max)		Wt%(min, max)	
C	30	40	0.00	100.00
H	25	70	0.00	100.00
O	10	20	0.00	100.00
Na	0	1	0.00	100.00

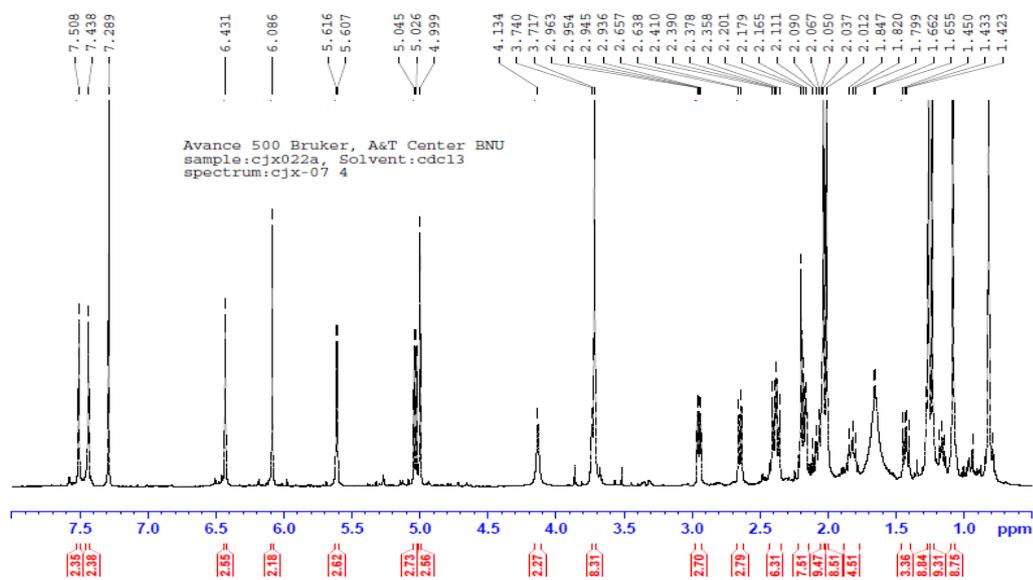
#	C	H	O	Na	mass	error
*** Mass Analysis for mass 689.2782480						
1	33	46	14	1	689.2779504	4.317e-07
2	35	45	14	0	689.2803558	3.058e-06

7 Xylocarpin F (7)

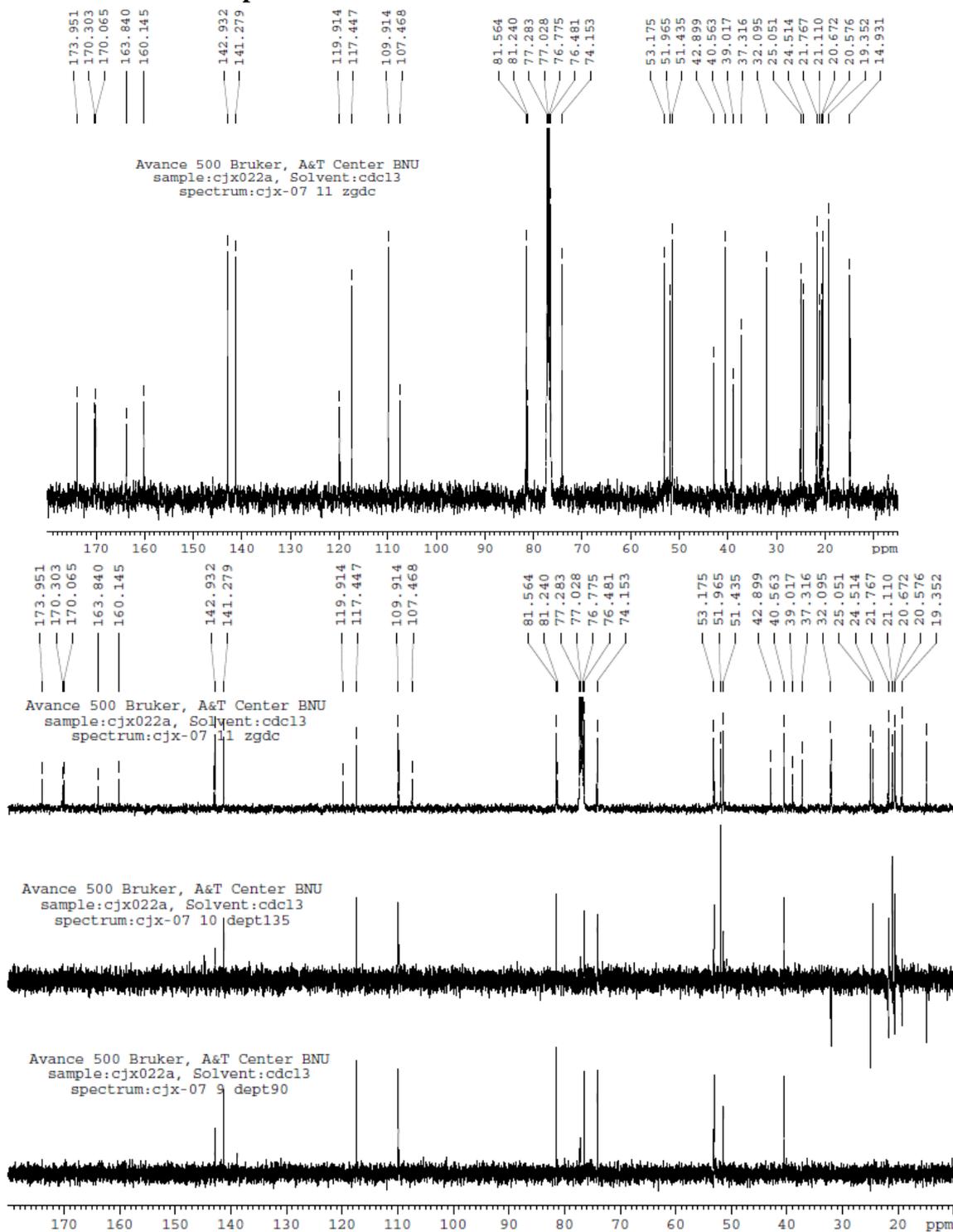
IR spectrum



¹H NMR spectrum

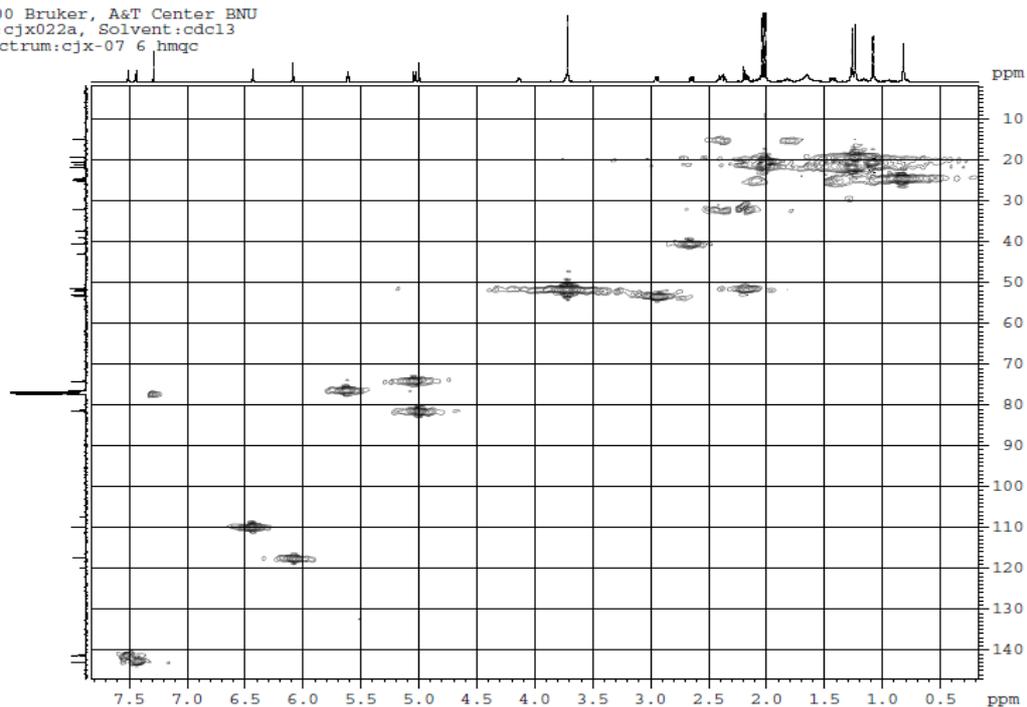


¹³C and DEPT NMR spectra



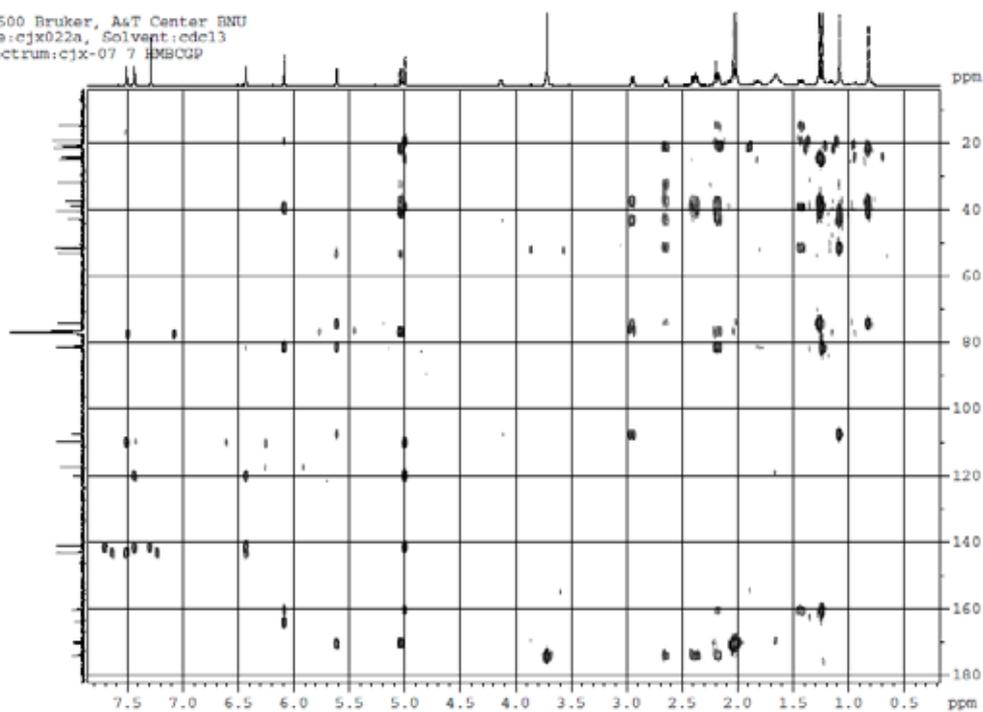
HMQC spectrum

Avance 500 Bruker, A&T Center BNU
sample:cjx022a, Solvent:cdcl3
spectrum:cjx-07 6 hmqc

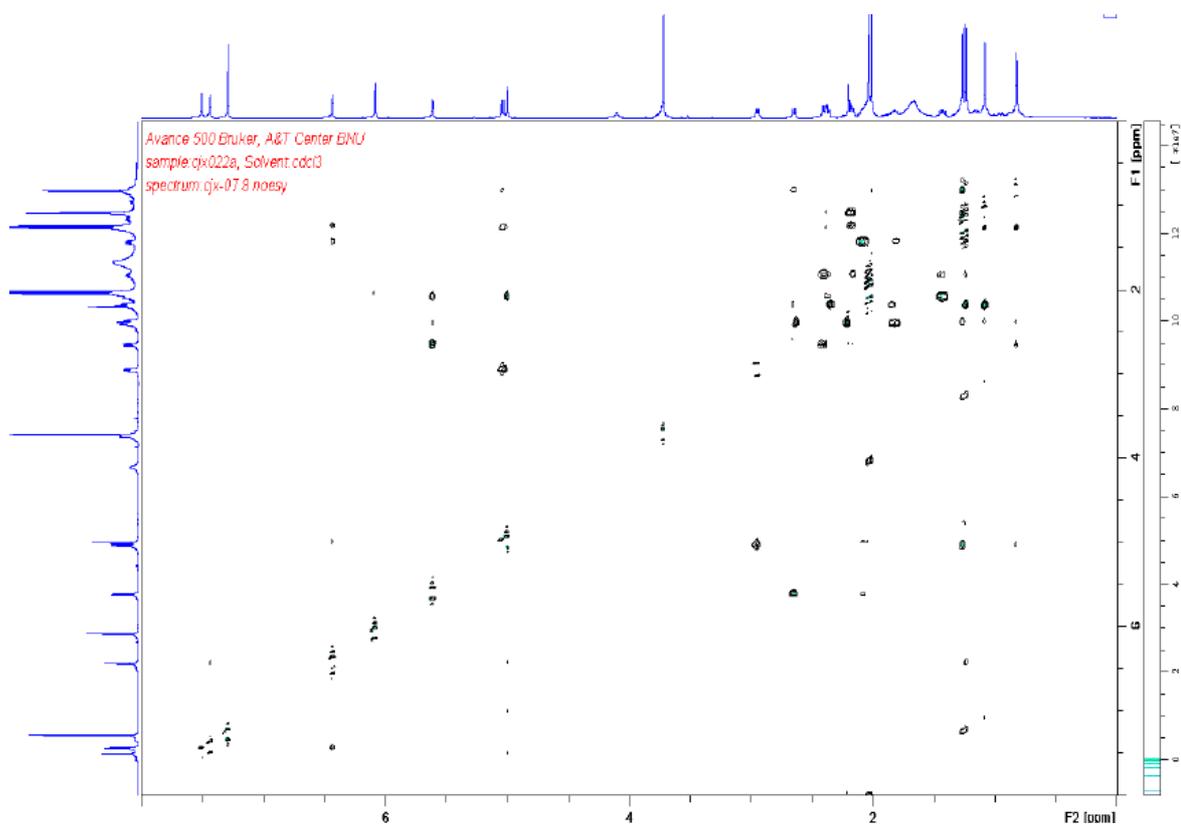


HMBC spectrum

Avance 500 Bruker, A&T Center BNU
sample:cjx022a, Solvent:cdcl3
spectrum:cjx-07 7 HMBCGP



NOESY spectrum

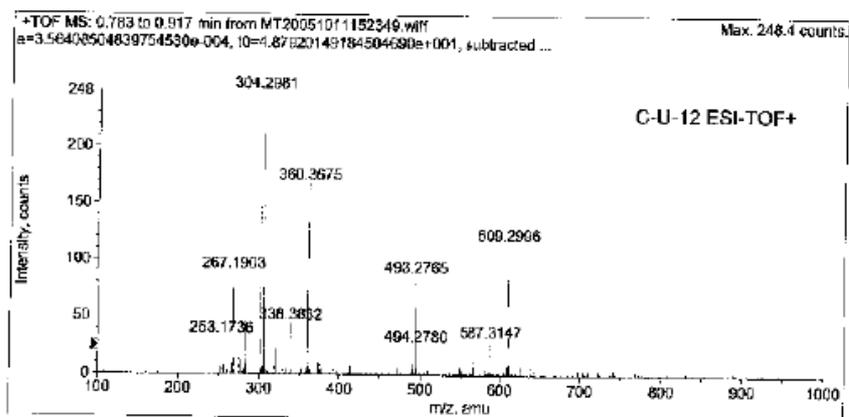


ESIMS spectrum

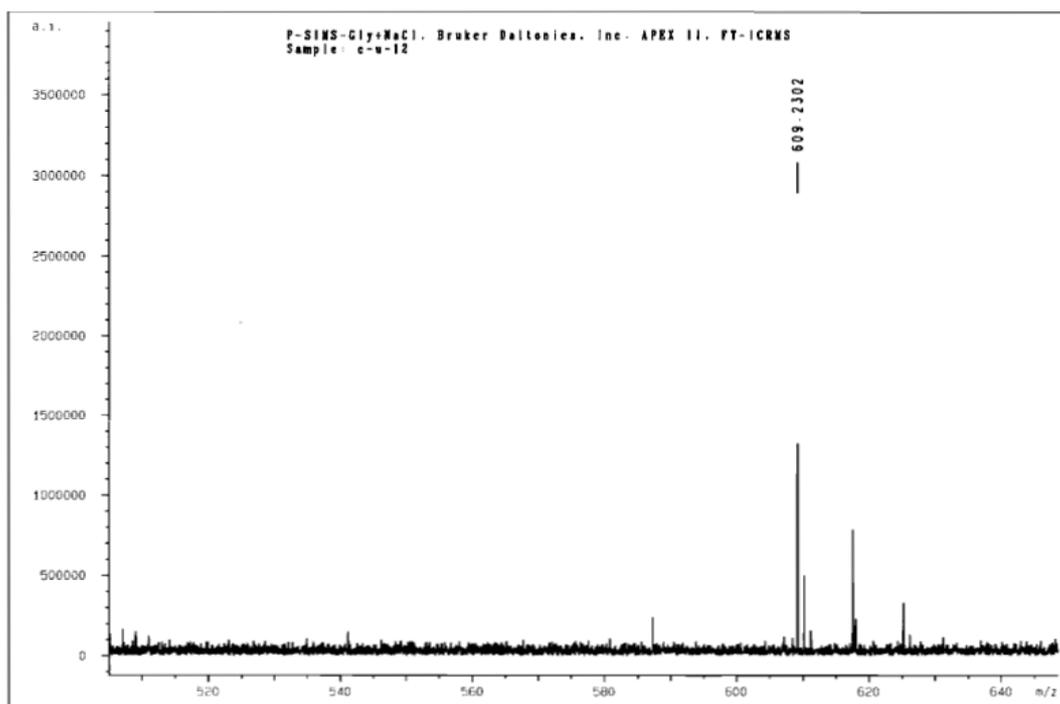
Acq. File: MT20051011152349.wiff

Printout Date: Tuesday, October 11, 2005

WORKALATION: Q577B



HRSIMS data



XMASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-LinWenHan/34/pdata/1
XMASS Mass Analysis Constraints

Ion mass = 609.2301500

Charge = +1

Tolerance = 0.0030000

DBE min = -2

DBE max = 200

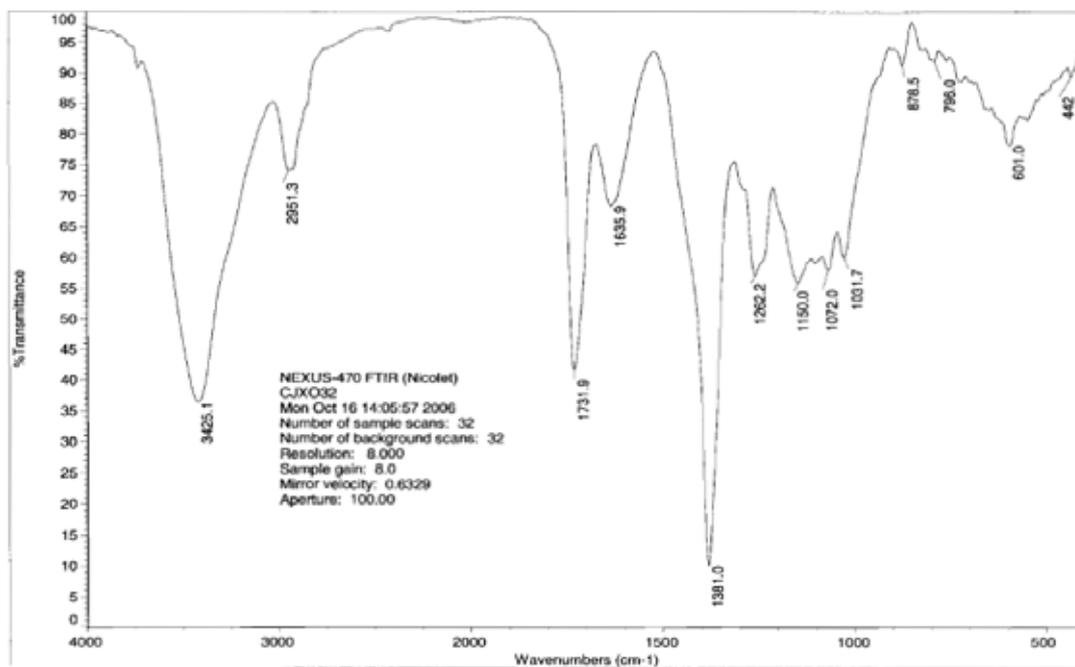
Max Candidates = 100

Atom	#(min, max)		Wt%(min, max)	
C	20	40	0.00	100.00
H	20	55	0.00	100.00
O	4	20	0.00	100.00
Na	0	1	0.00	100.00

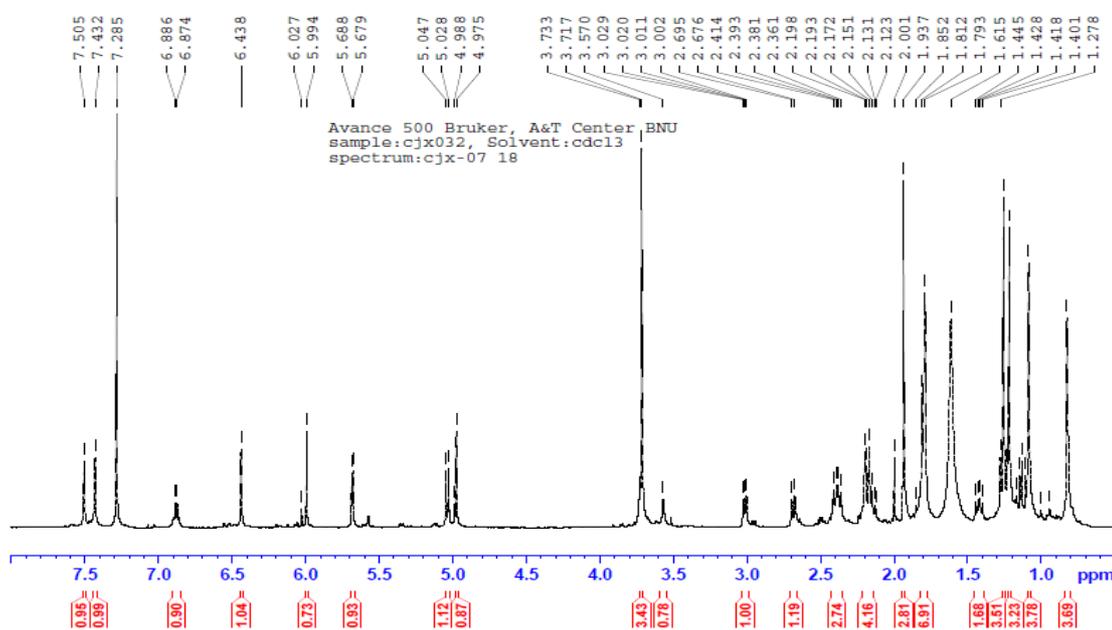
#	C	H	O	Na	mass	error
*** Mass Analysis for mass 609.2301500						
1	31	38	11	1	609.2306113	7.572e-07

8 Xylocarpin G (8)

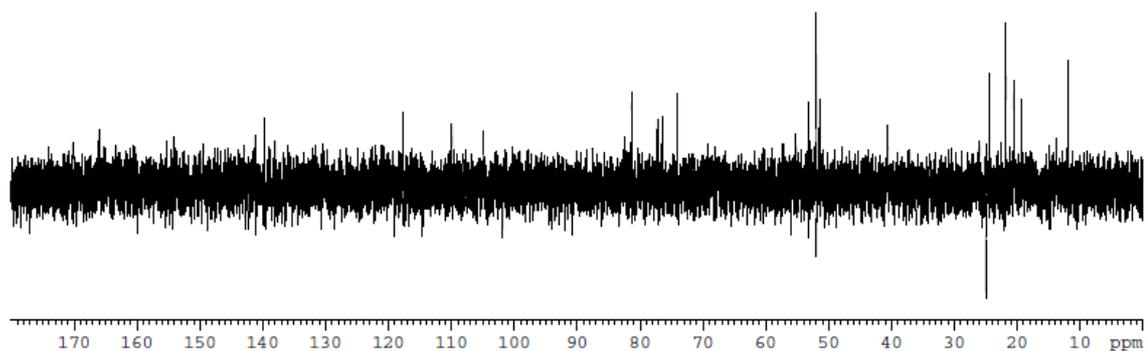
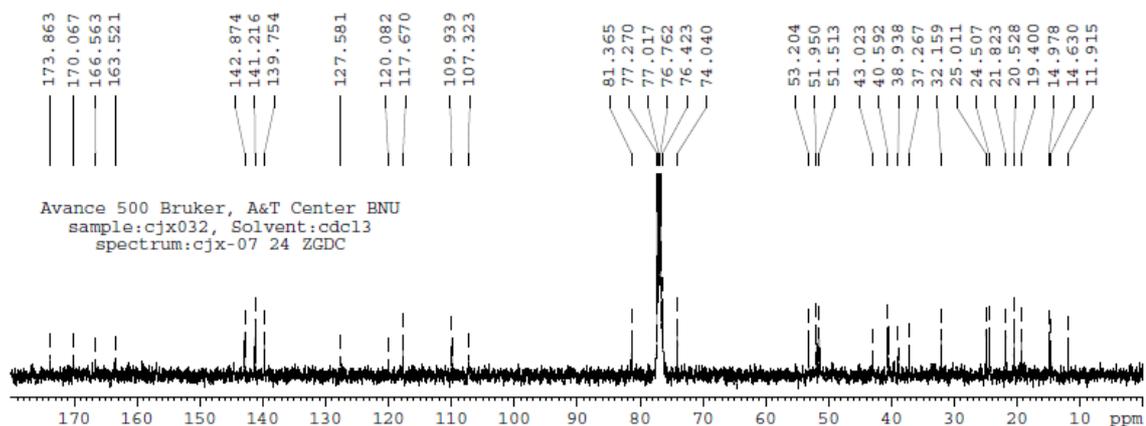
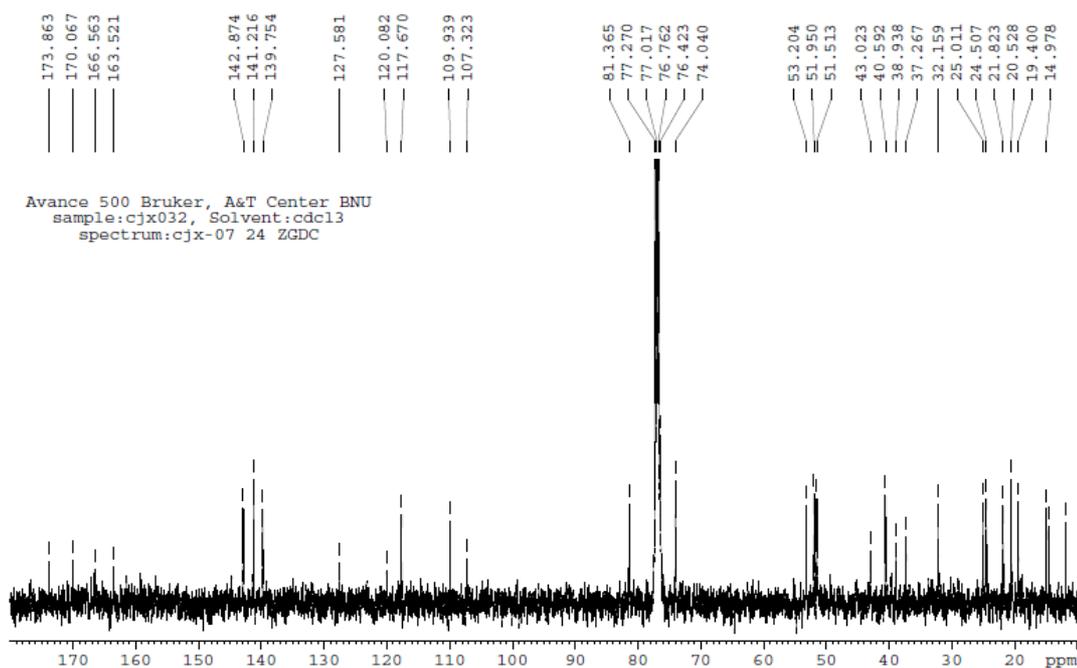
IR spectrum



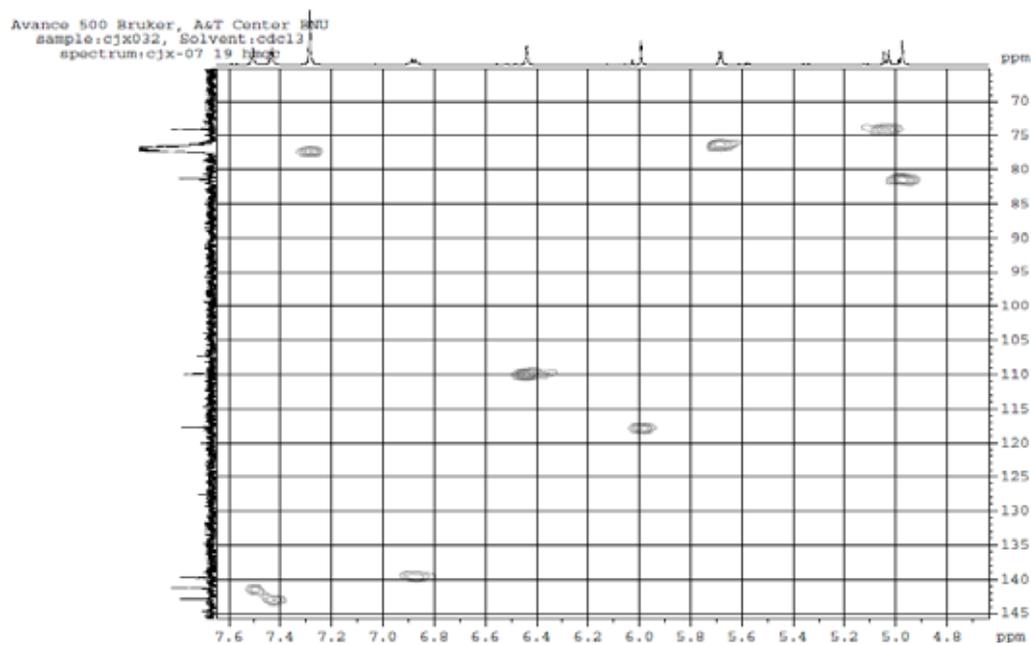
¹H NMR spectrum



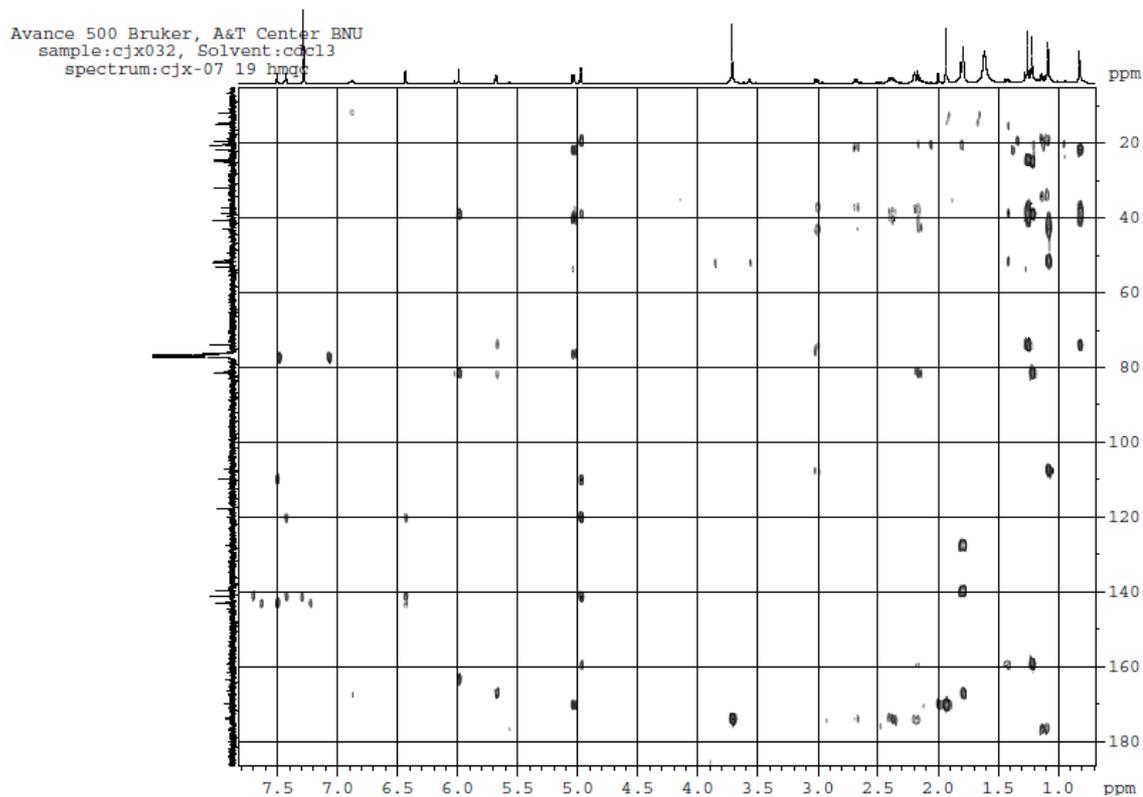
¹³C and DEPT NMR spectra



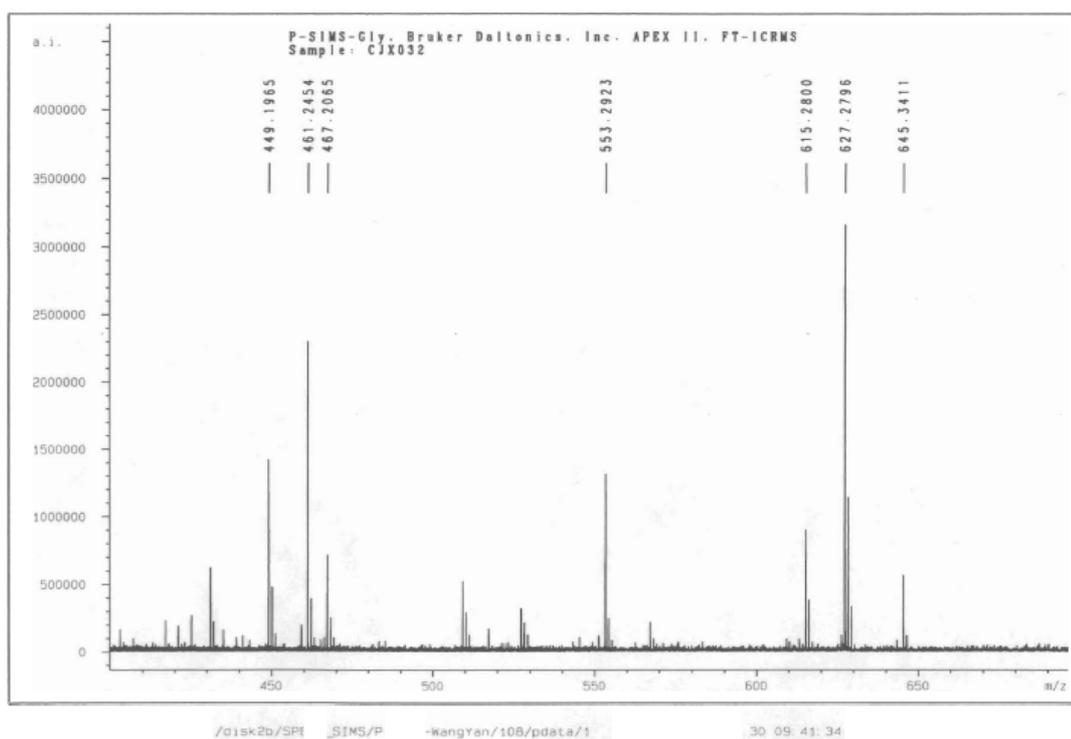
HMQC spectrum



HMBC spectrum



HRSIMS spectrum



XMASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-WangYan/108/pdata/1/massanal
XMASS Mass Analysis Constraints

Ion mass = 627.2796360

Charge = +1

Tolerance = 0.0030000

DBE min = -2

DBE max = 200

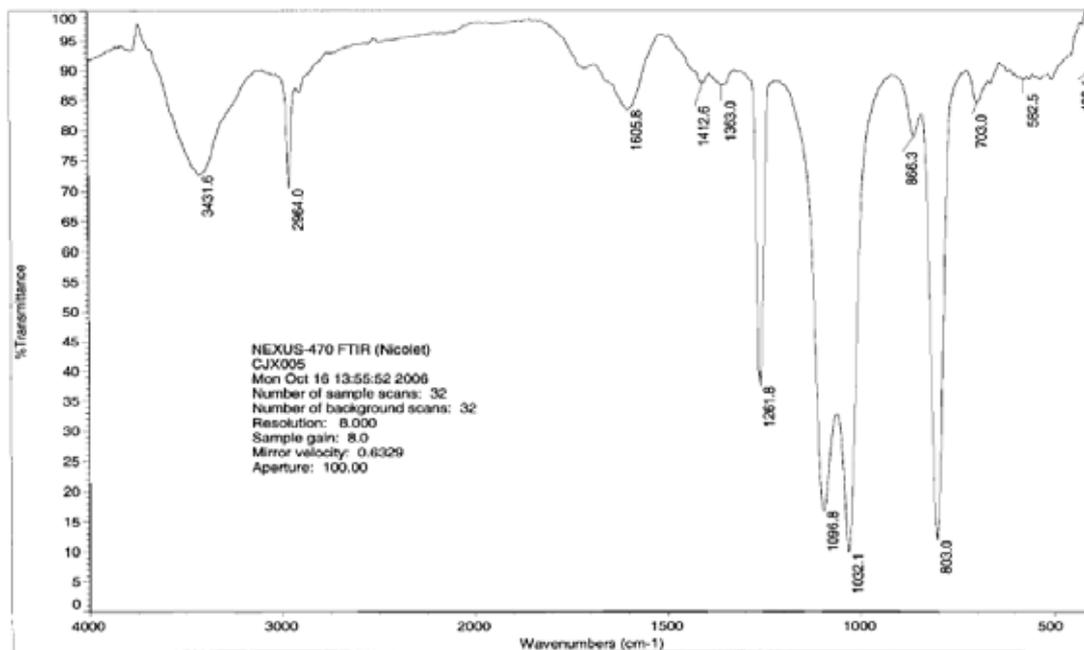
Max Candidates = 100

Atom	#(min, max)		Wt%(min, max)	
C	30	36	0.00	100.00
H	30	50	0.00	100.00
O	5	15	0.00	100.00

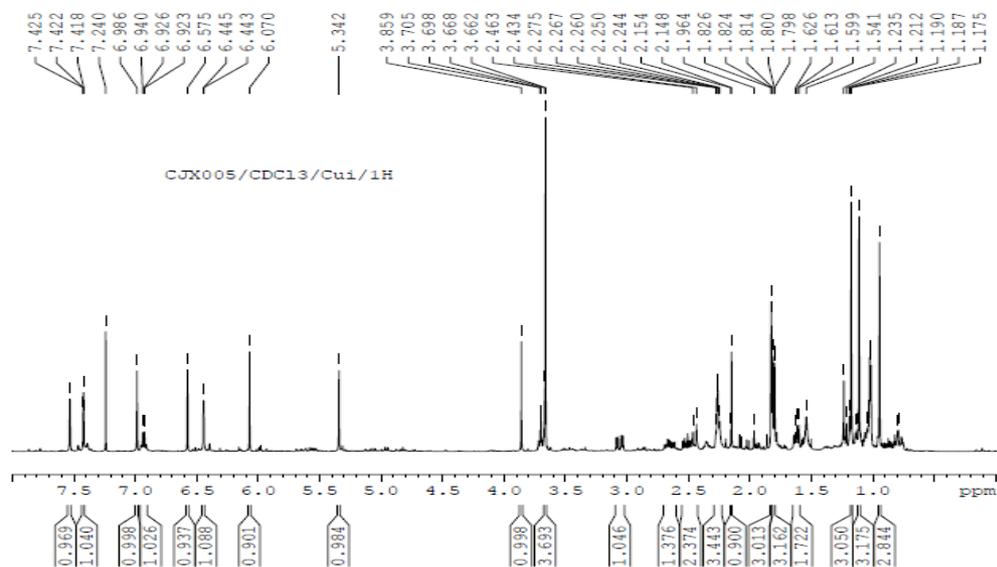
#	C	H	O	mass	error
*** Mass Analysis for mass 627.2796360					
1	34	43	11	627.2799643	5.234e-07

9. Xylocarpin H (9)

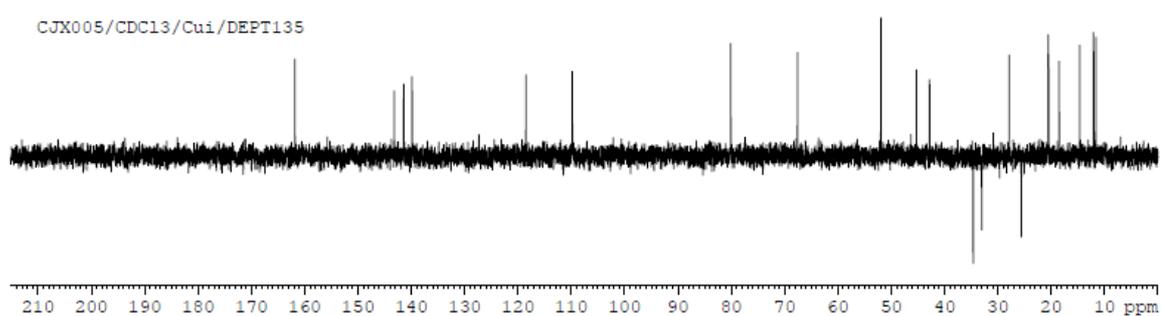
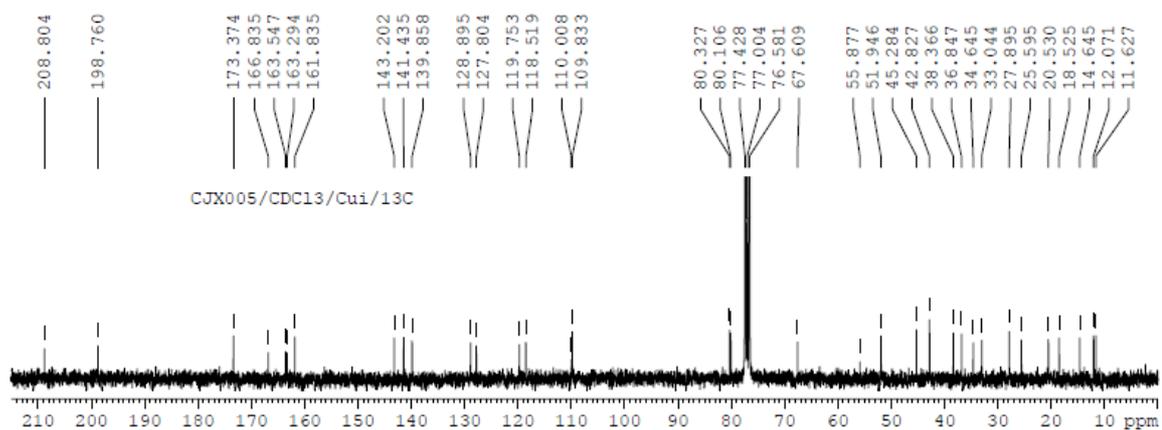
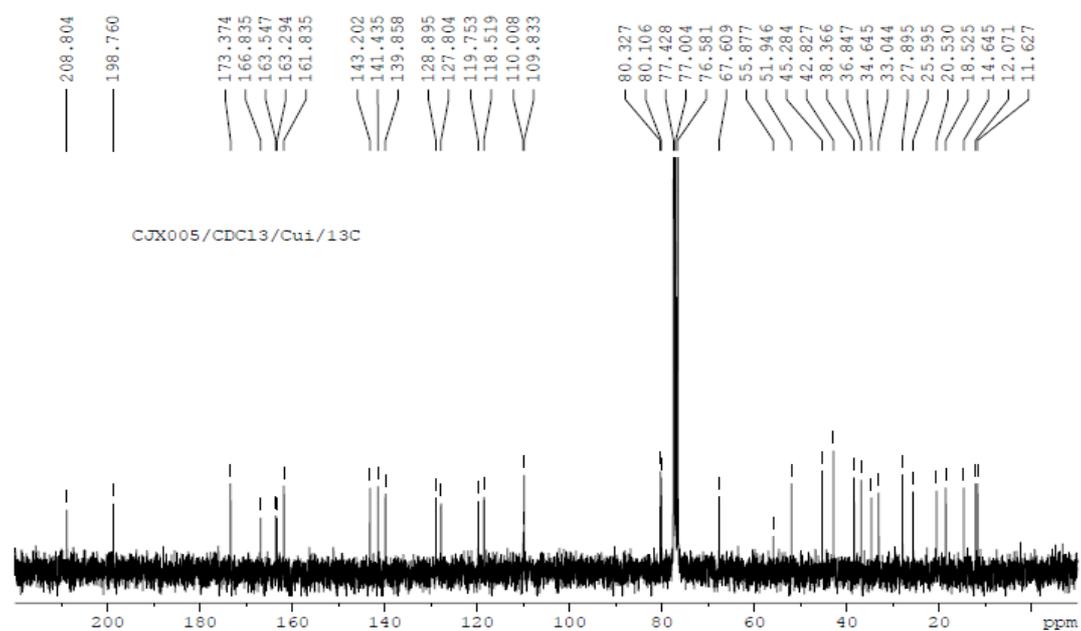
IR spectrum



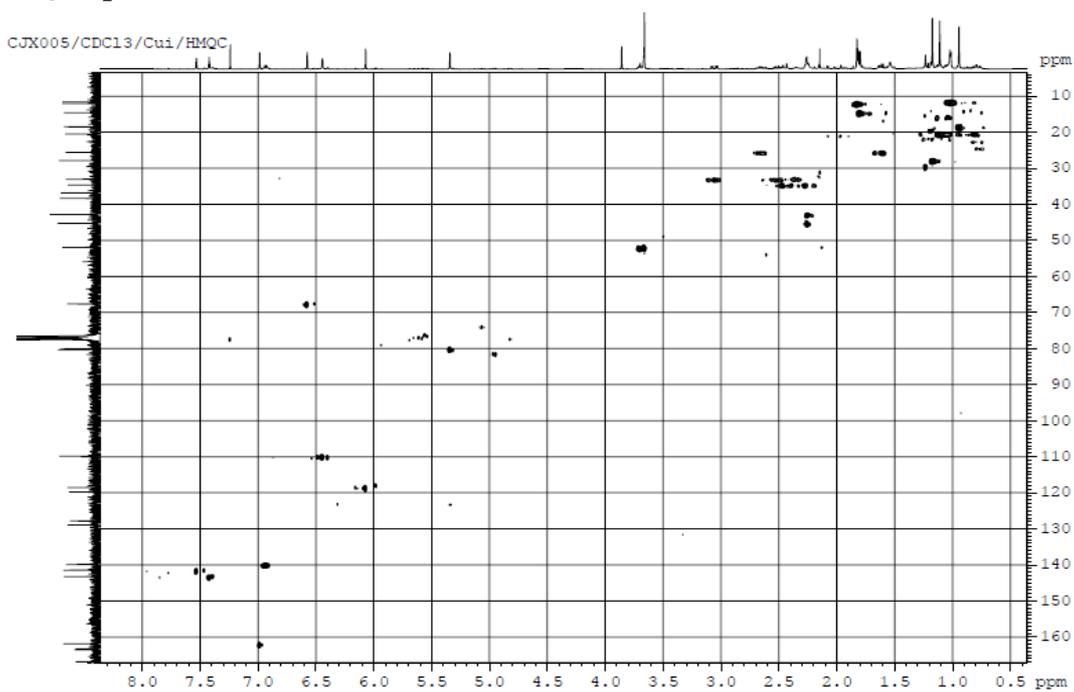
¹H NMR spectrum



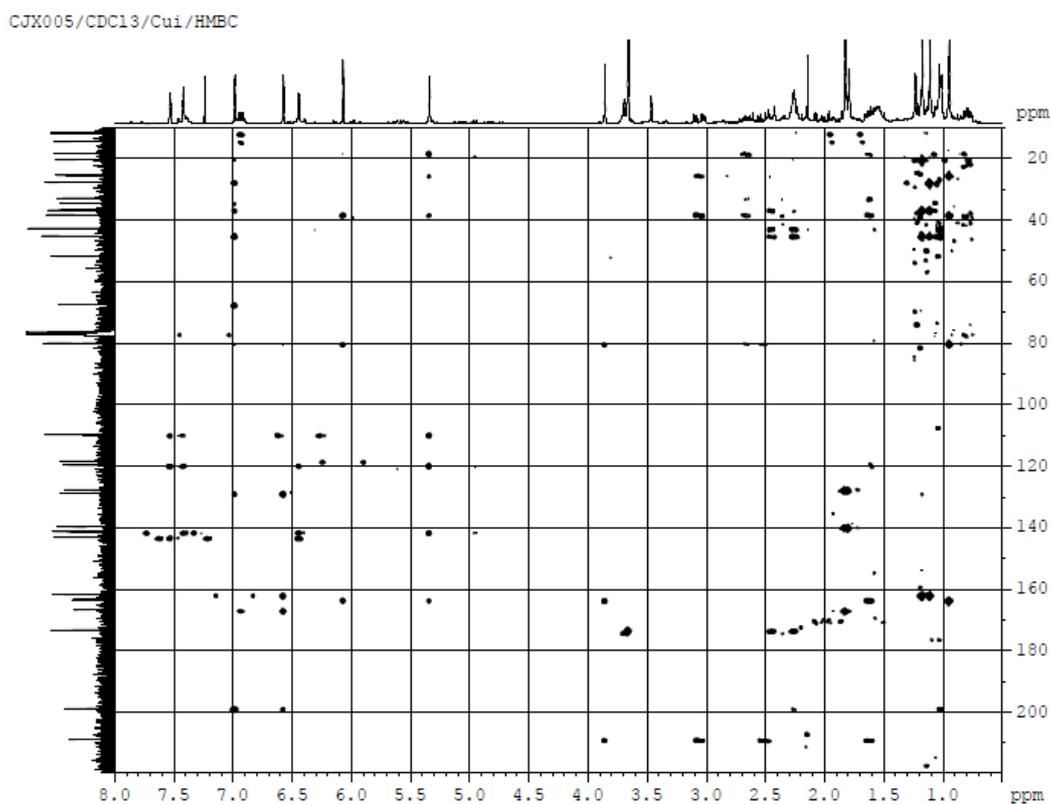
¹³C and DEPT NMR spectra



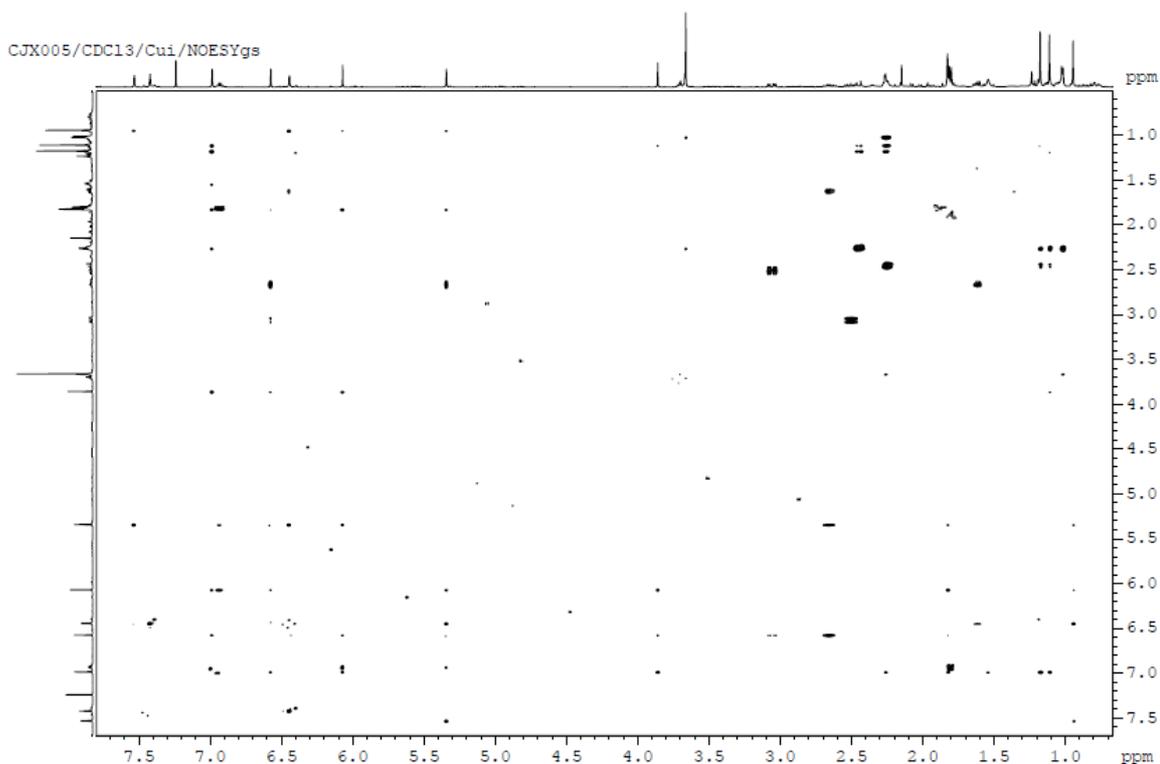
HMQC spectrum



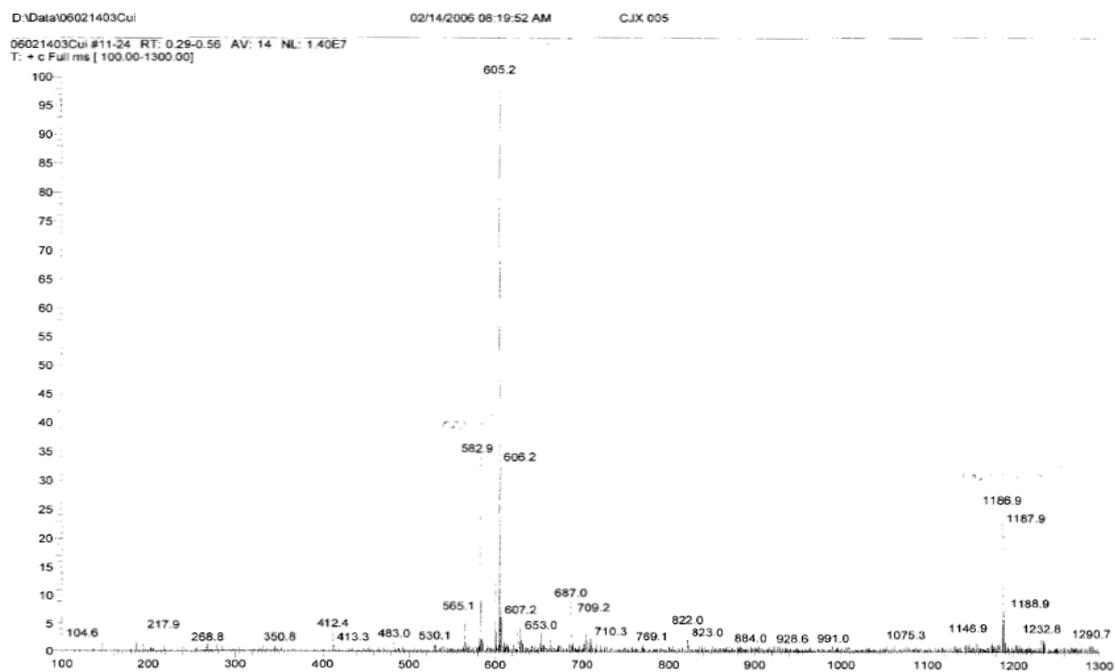
HMBC spectrum



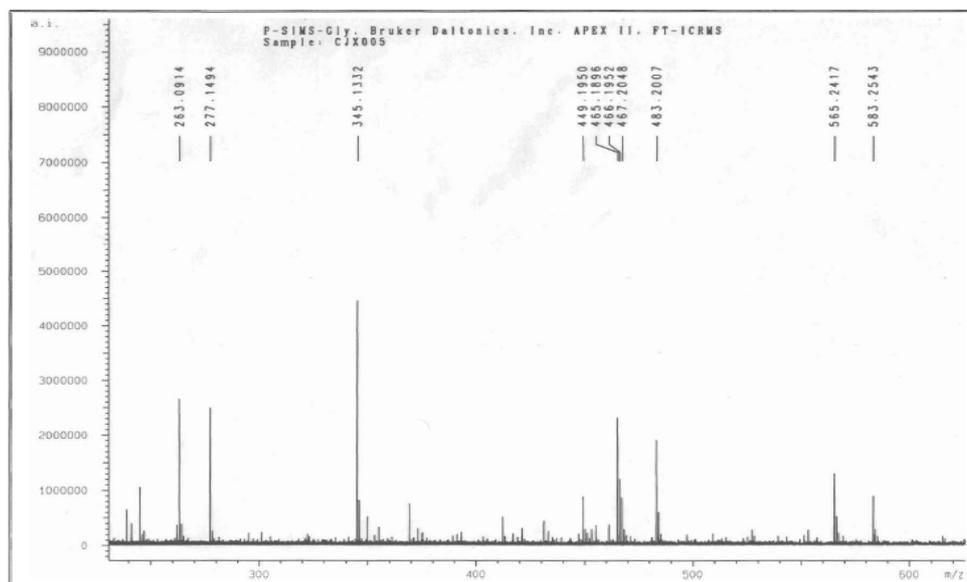
NOESY spectrum



ESIMS spectrum



HRSIMS spectrum



XMASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-WangYan/113/pdata/1/massana:
XMASS Mass Analysis Constraints

Ion mass [1] = 565.2417020
Ion mass [2] = 583.2542500

Charge = +1
Tolerance = 0.0030000

DBE min = -2
DBE max = 200

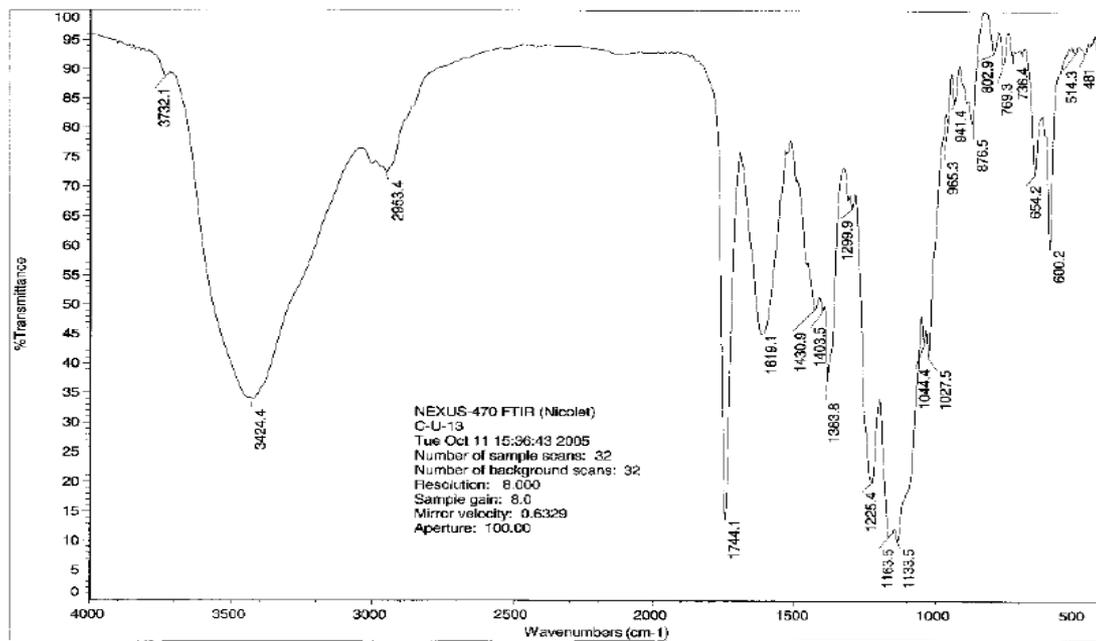
Max Candidates = 100

Atom	#(min, max)		Wt%(min, max)	
C	30	36	0.00	100.00
H	30	50	0.00	100.00
O	5	15	0.00	100.00

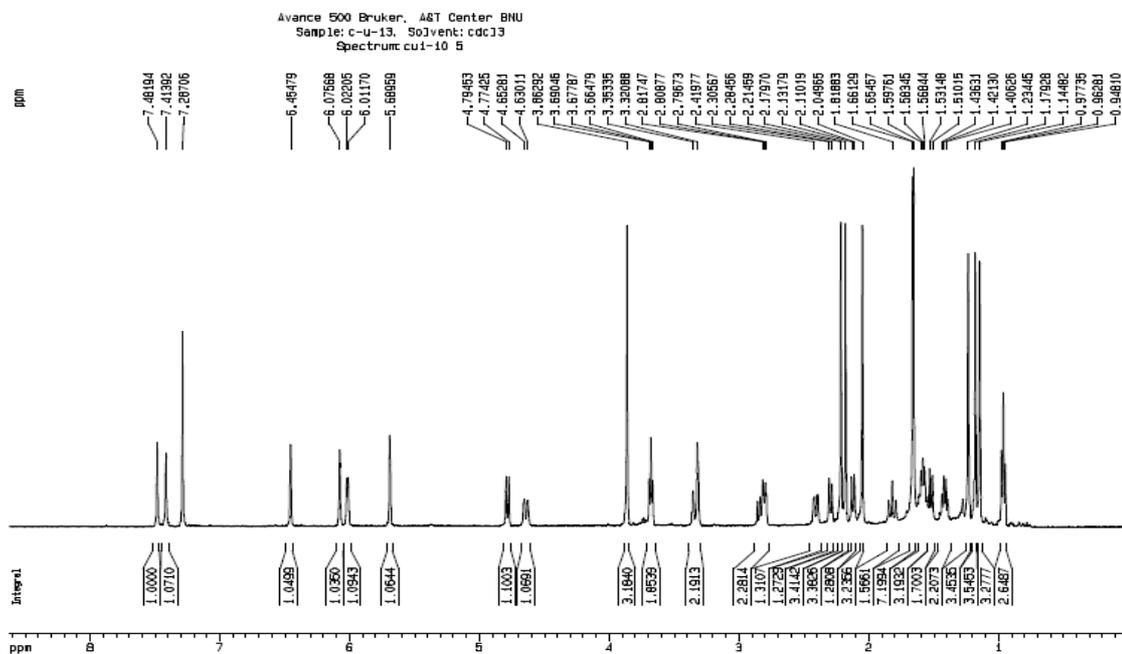
#	C	H	O	mass	error
***	Mass Analysis for mass 565.2417020				
1	32	37	9	565.2431885	2.630e-06
***	Mass Analysis for mass 583.2542500				

10. Xylocarpin I (10)

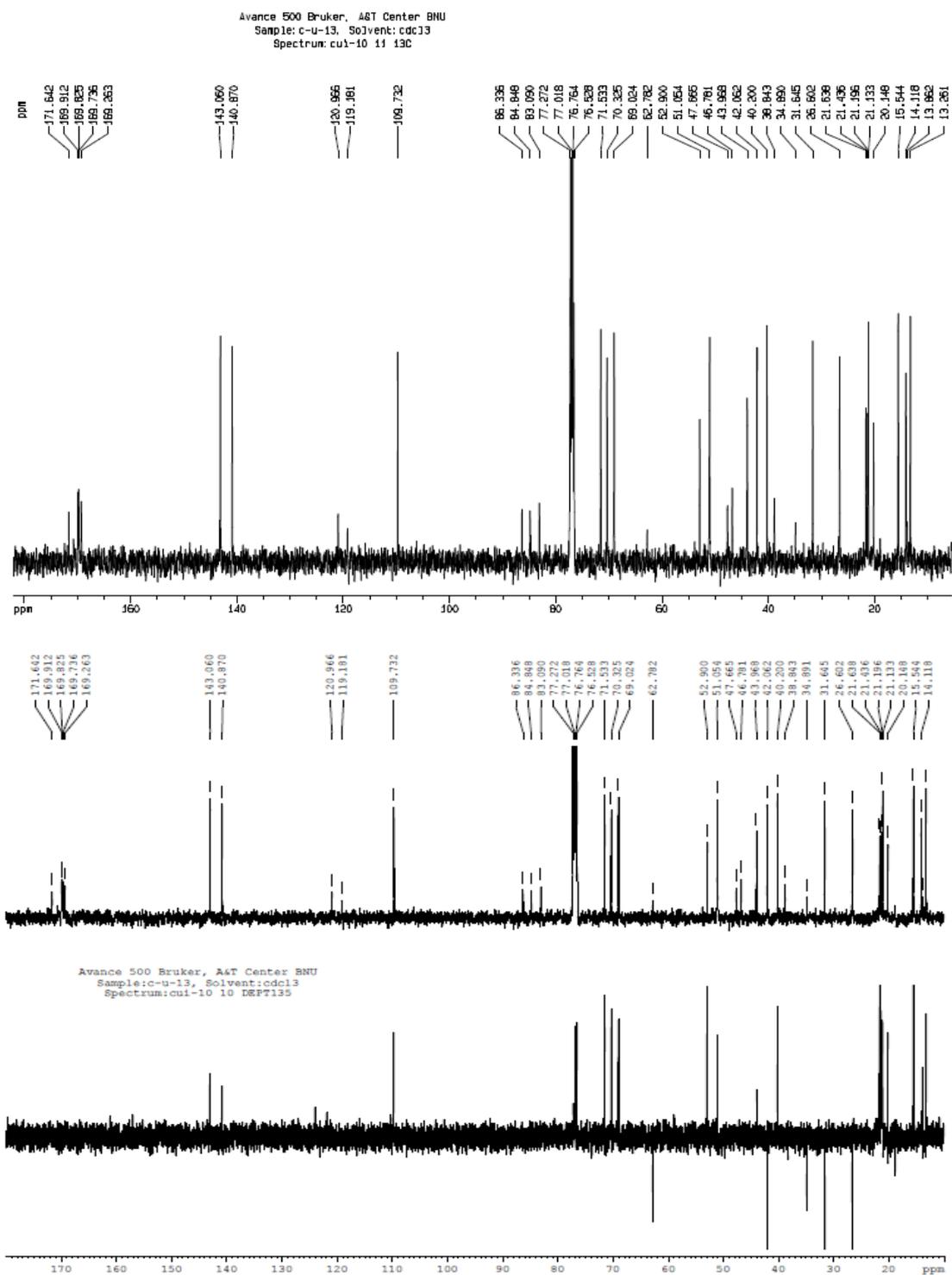
IR spectrum



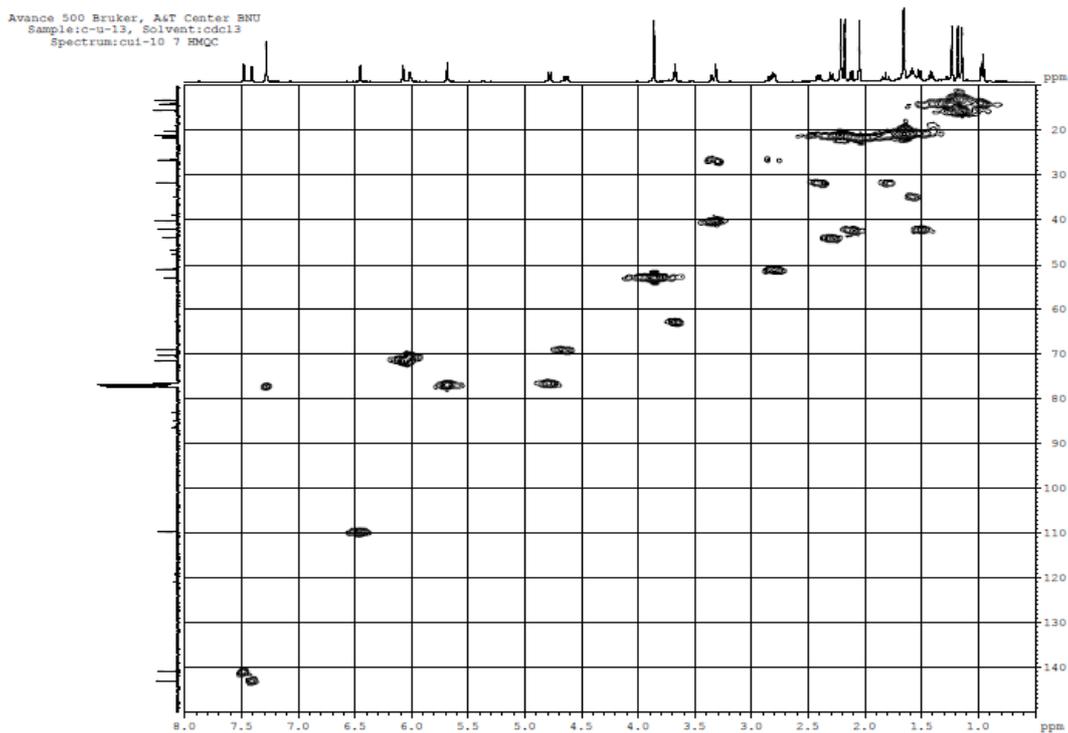
¹H NMR spectrum



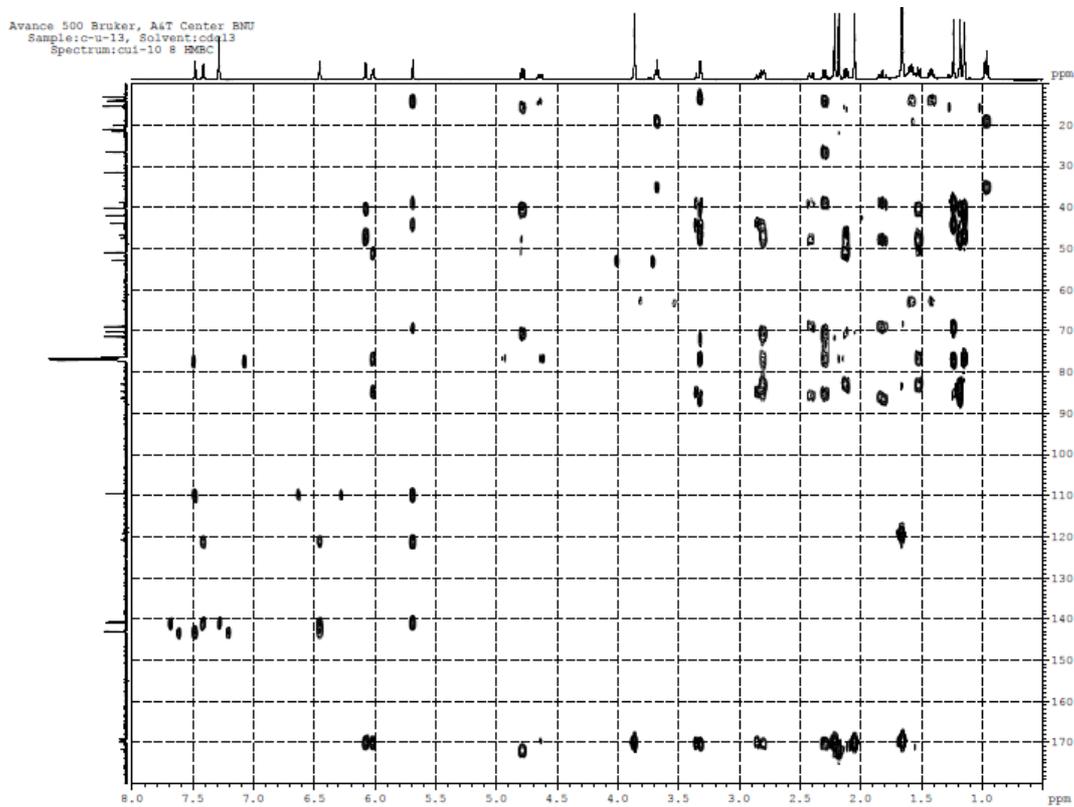
¹³C and DEPT NMR spectra



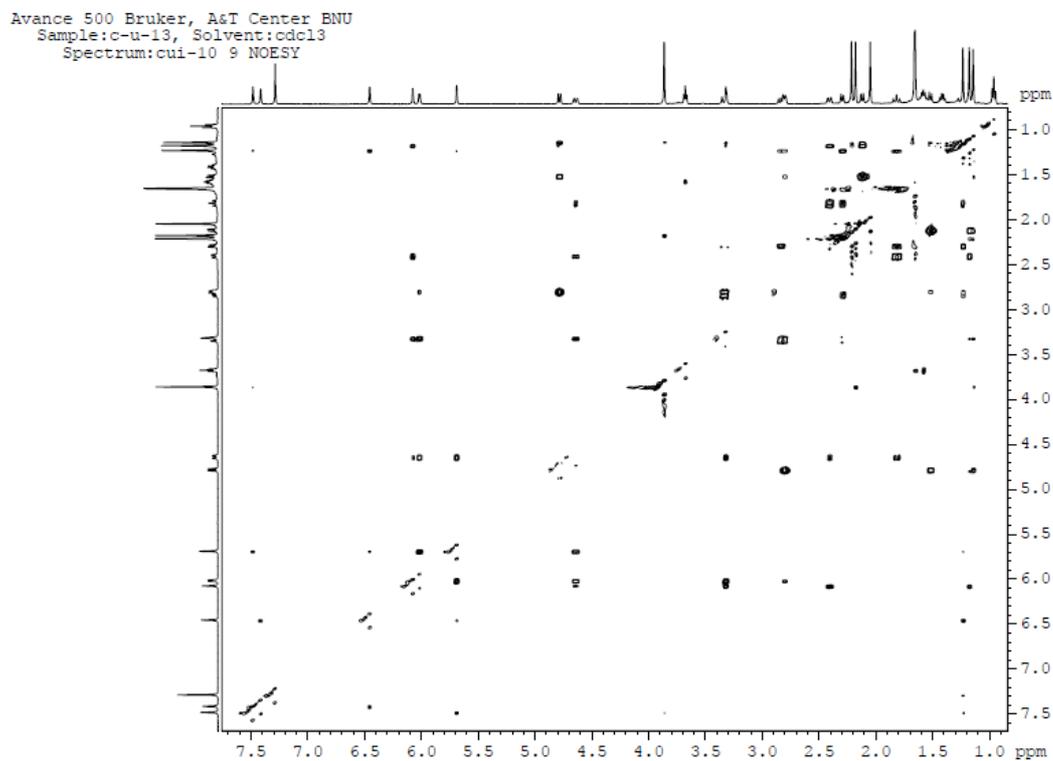
HMQC spectrum



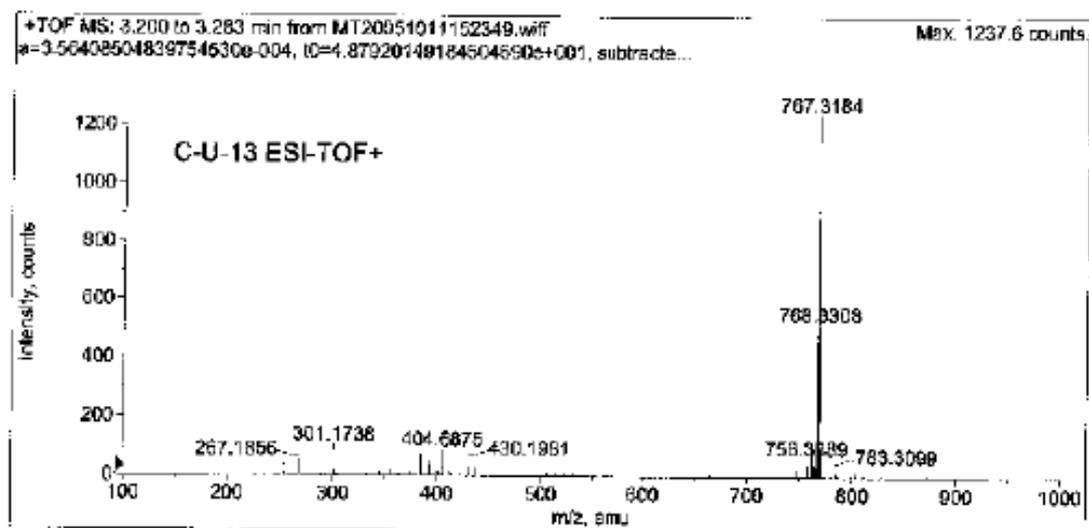
HMBC spectrum



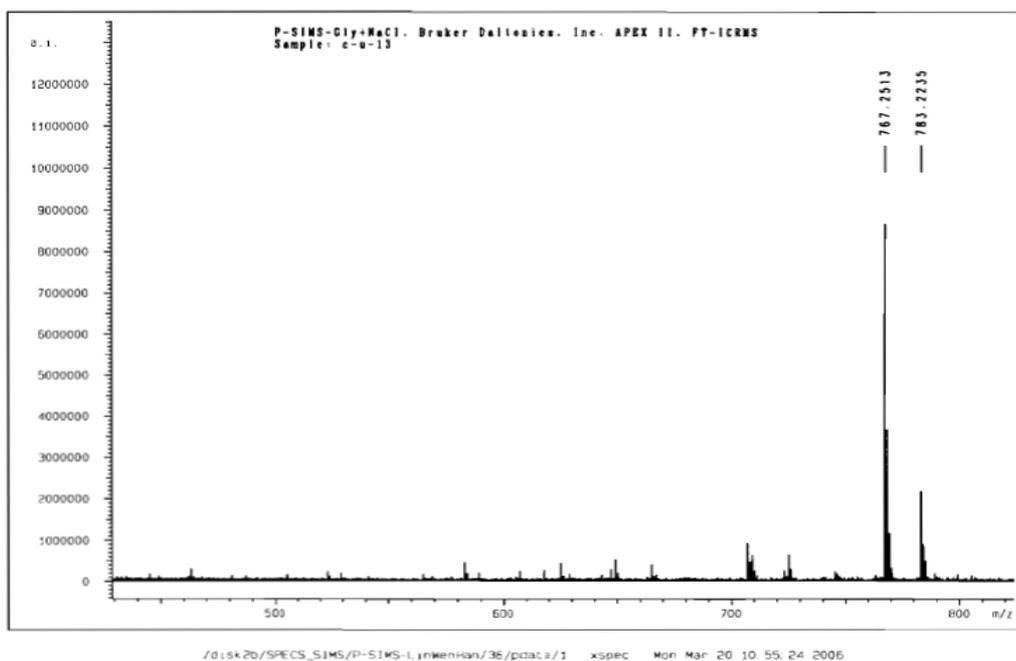
NOESY spectrum



ESIMS spectrum



HRSIMS data



XMASS Mass Analysis for /disk2b/SPECS_SIMS/P-SIMS-WangYan/113/pdata/1/massana1
XMASS Mass Analysis Constraints

Ion mass [1] = 565.2417020
Ion mass [2] = 583.2542500

Charge = +1
Tolerance = 0.0030000

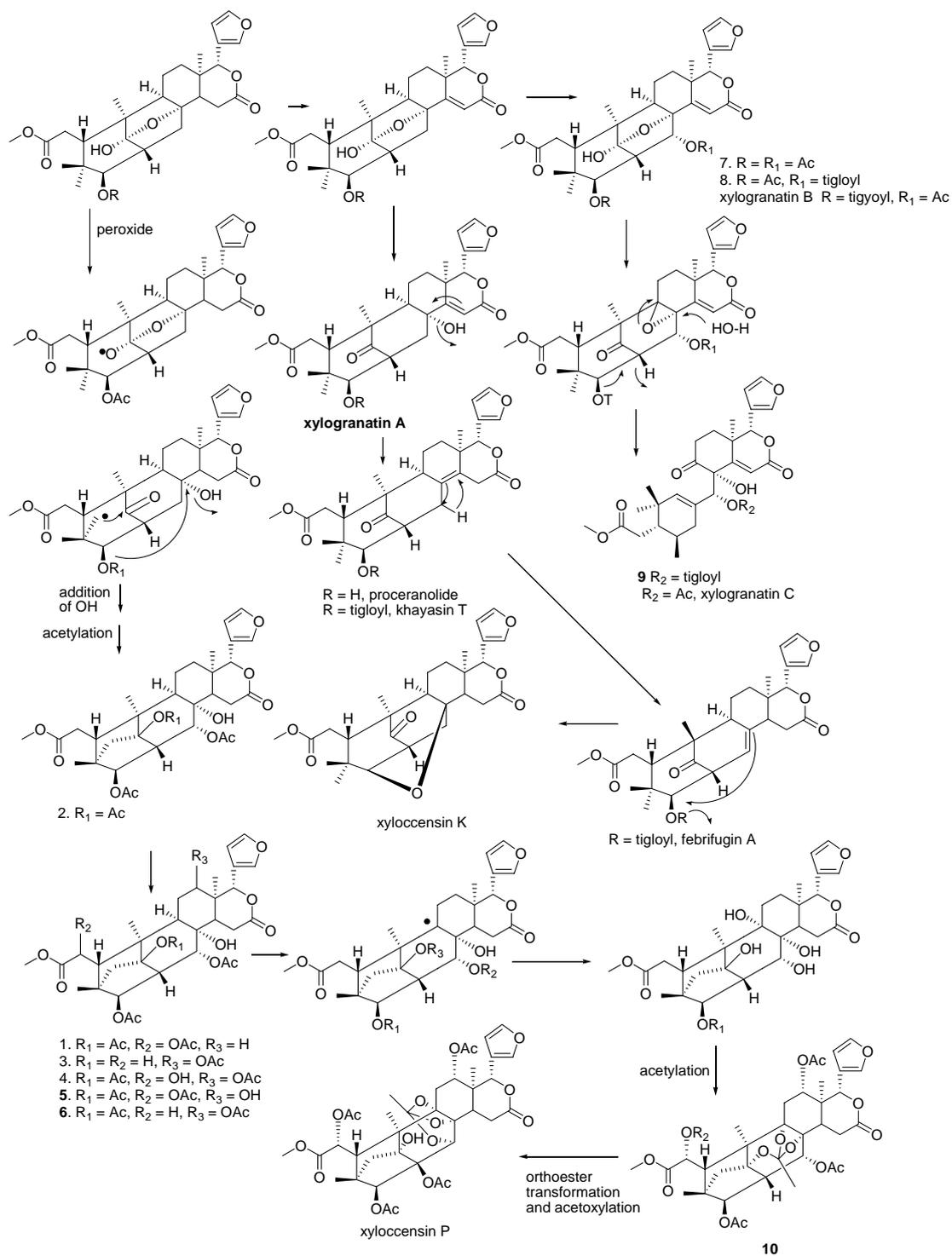
DBE min = -2
DBE max = 200

Max Candidates = 100

Atom	#(min, max)	Wt%(min, max)
C	30 36	0.00 100.00
H	30 50	0.00 100.00
O	5 15	0.00 100.00

#	C	H	O	mass	error
***	Mass Analysis for mass 565.2417020				
1	32	37	9	565.2431885	2.630e-06

*** Mass Analysis for mass 583.2542500



Scheme 1 The hypothesis of biotransformation of the isolated limonoids