

Supplementary Material for

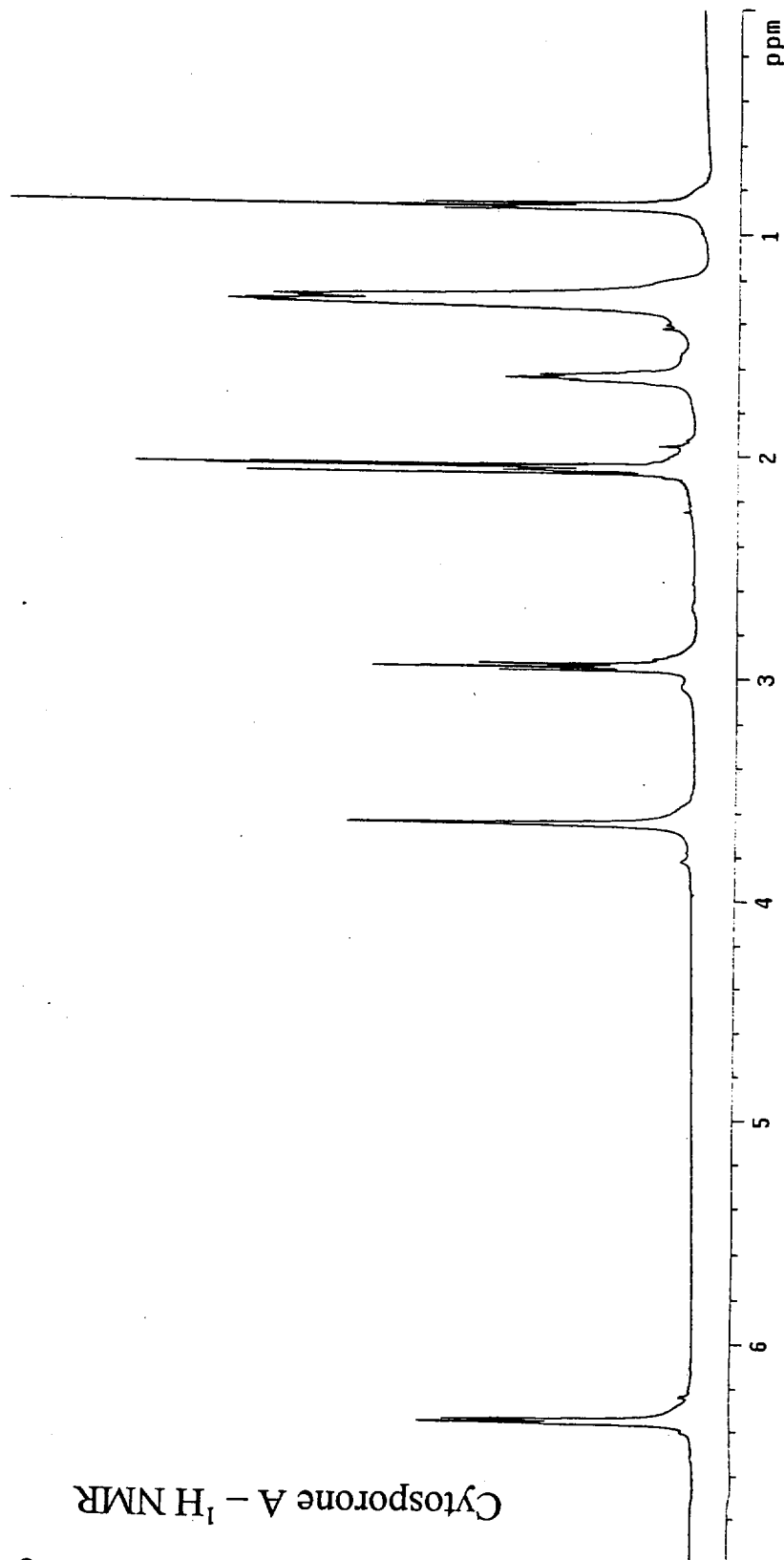
The Cytosporones, New Octaketide Antibiotics Isolated from an Endophytic Fungus.

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2S

STANDARD PROTON PARAMETERS
Solvent: Acetone
Temp. 25.0 C / 298.1 K
File: cr200-s-1-H
UNITY-500 "nmr500"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 45.0 degrees
Acq. time 1.890 sec
Width 4012.0 Hz
Arrayed repetitions
OBSERVE H1, 499.9270431 MHz
DATA PROCESSING
F1 size 16384
Total time 1 minute

Cytosporone A - ^1H NMR

Organic Letters - Cytosporones

ES

new experiment

Solvent: Acetone
 Temp. 25.0 C / 298.1 K
 File: cr200-s-1-c
 UNITY-500 "nmru500"

PULSE SEQUENCE

Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 25.7 degrees
 Acq. time 1.199 sec
 Width 26000.0 Hz

Arranged repetitions

OBSERVE C13, 100.5037126 MHz
 DECOUPLE H1, 399.8991172 MHz

Power 31 dB

continuously on

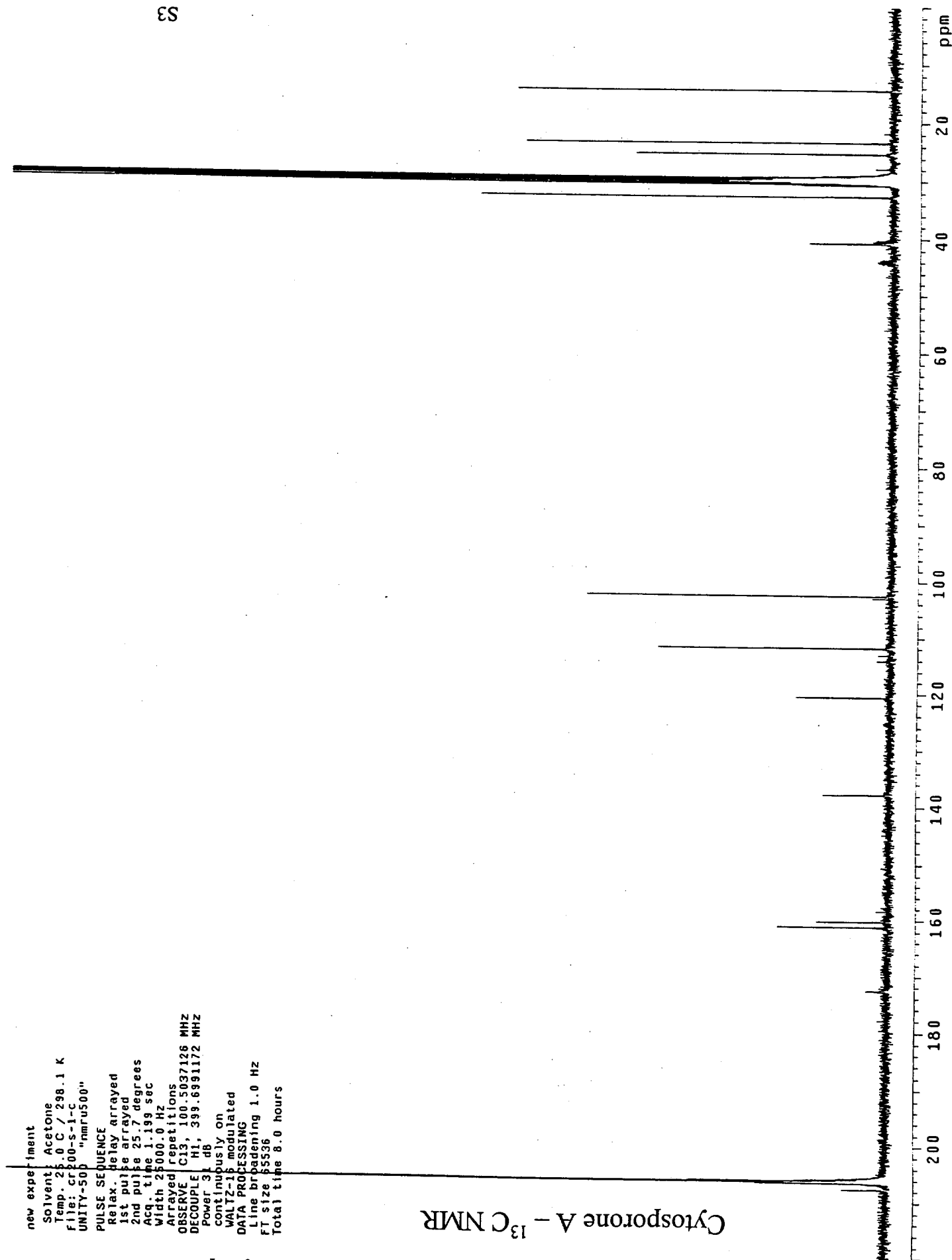
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

F1 size 65536

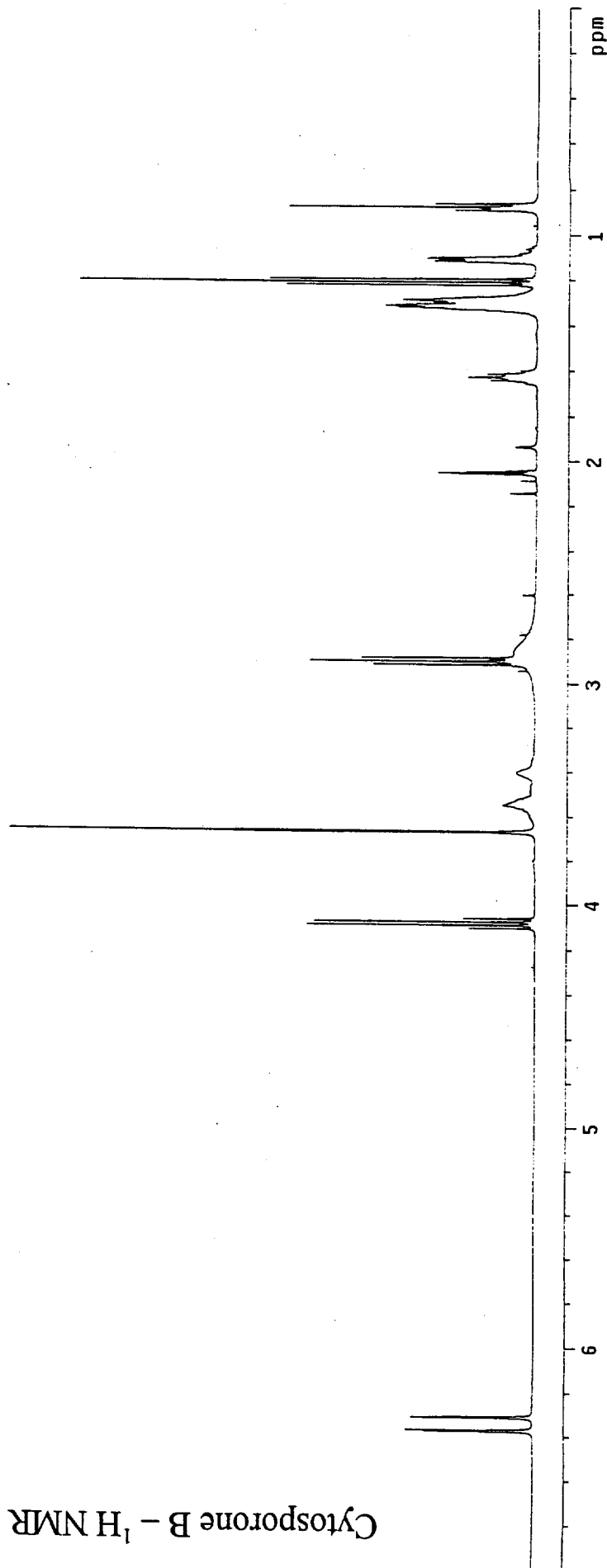
Total time 8.0 hours

Cytosporone A - ^{13}C NMR

S4

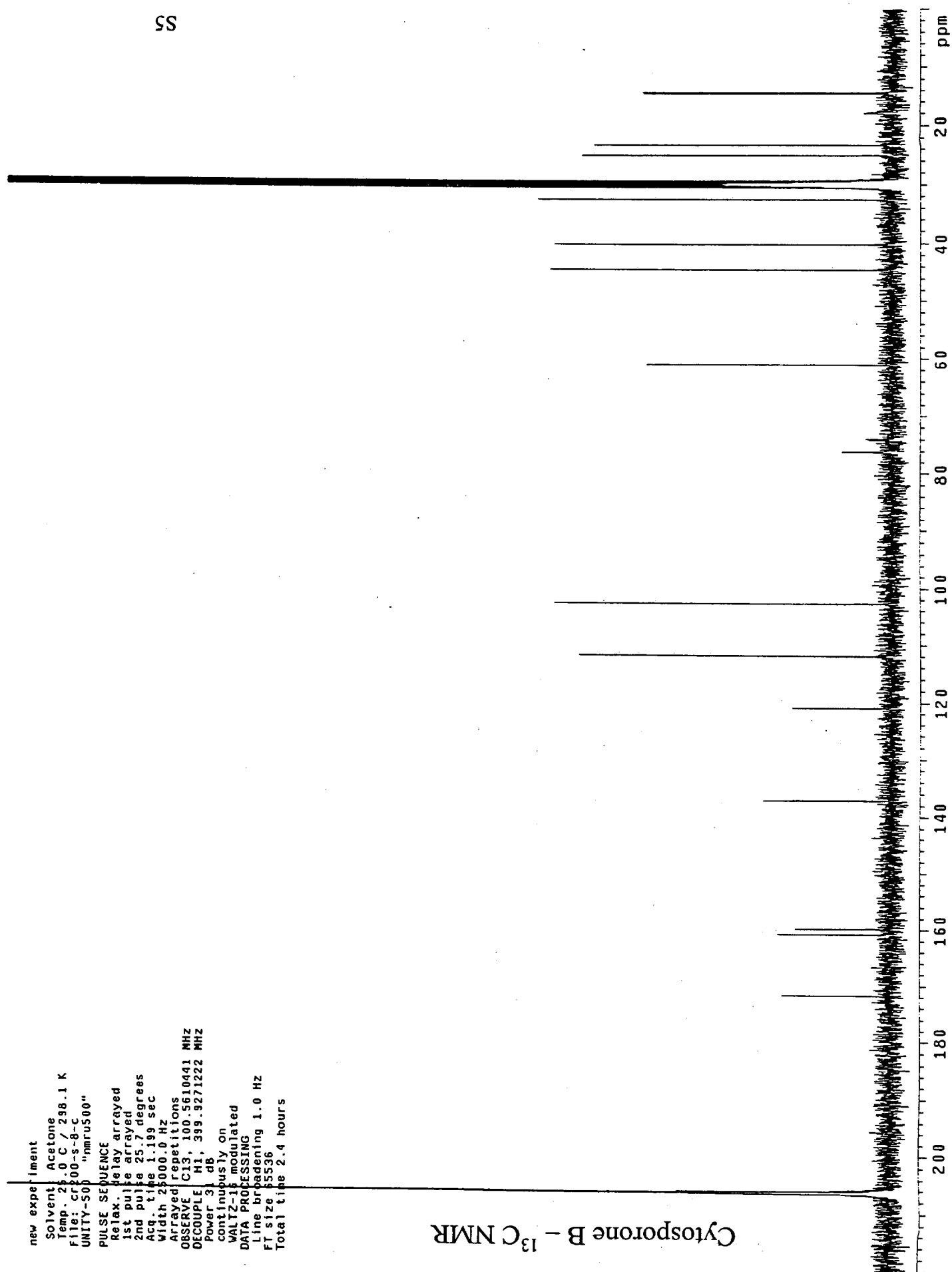
STANDARD PROTON PARAMETERS
 Solvent: Acetone
 Temp. 25.0 C / 298.1 K
 File: cr200-s-8-H
 UNITY-500 "nmru500"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 51.1 degrees
 Acq. time 1.888 sec
 Width 3999.6 Hz
 Arrayed repetitions
 OBSERVE Hz 499.9270436 MHz
 DATA PROCESSING
 Line broadening 0.1 Hz
 FT size 16384
 Total time 3 minutes

Cytosporone B - ¹H NMR



S5

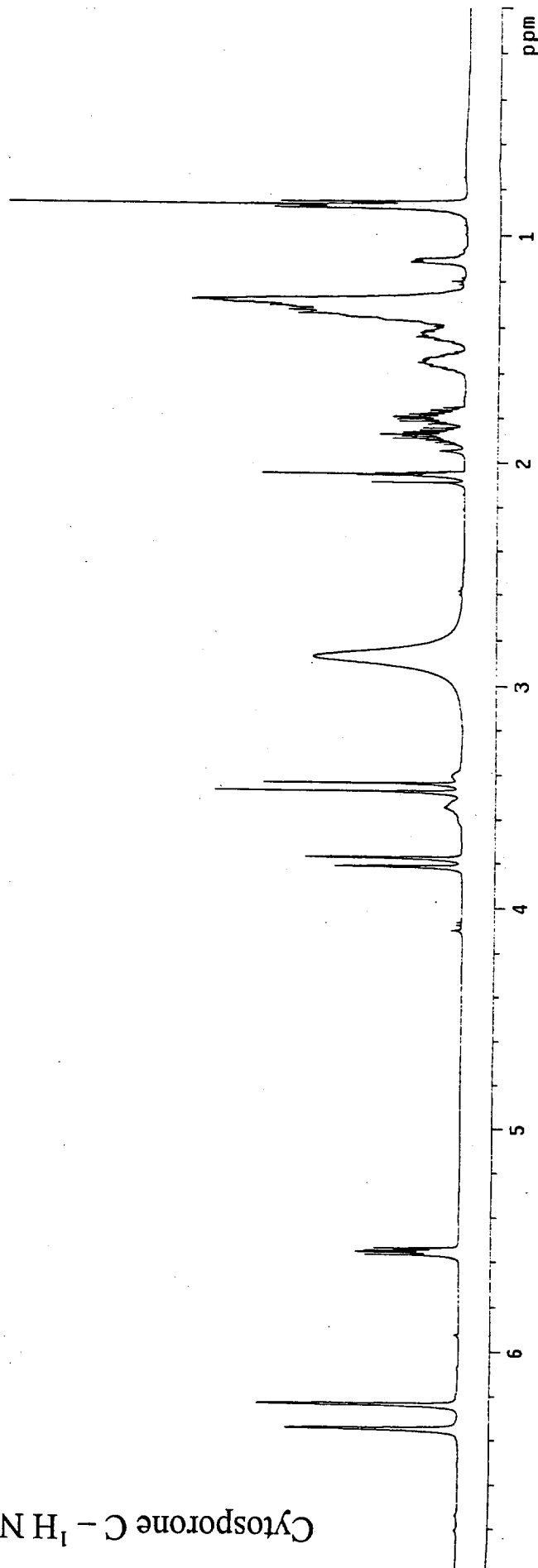
new experiment
 Solvent Acetone
 Temp. 21.0 C / 298.1 K
 File: cr200-s-8-c
 UNITY-S00 "nmrUS00"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 25.7 degrees
 Acq. time 1.199 sec
 Width 25000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 100.5610441 MHz
 DECOUPLE H1, 399.9271222 MHz
 Power 31 dB
 continuously on
 VALTZ-16 modulated
 DATA PROCESSING
 line broadening 1.0 Hz
 FT size 65536
 Total time 2.4 hours

Cytosporone B - ^{13}C NMR

Organic Letters - Cytosporones

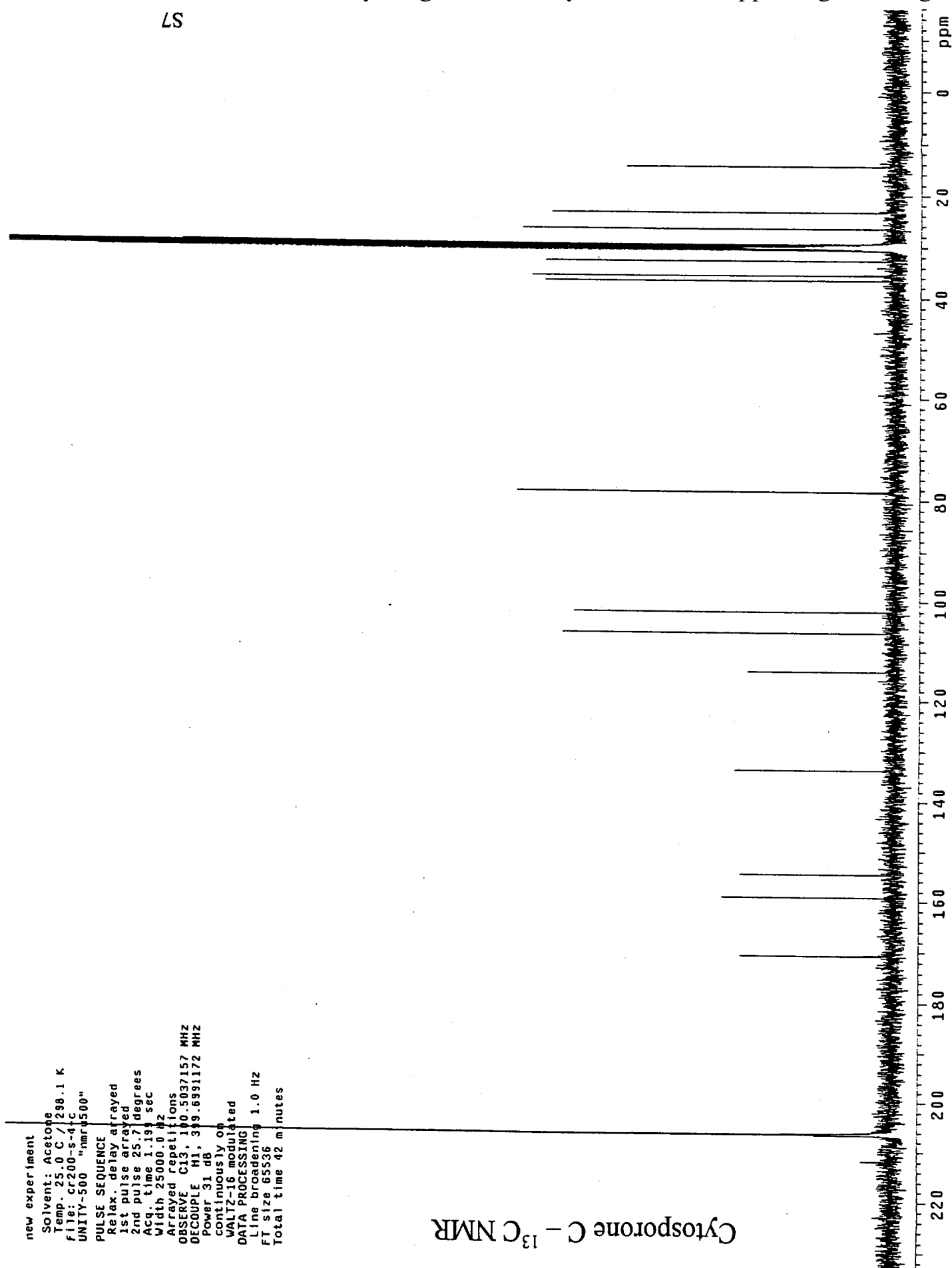
9S

STANDARD PROTON PARAMETERS
Solvent: Acetone
Temp. 25.0 C / 298.1 K
File: cr200-s-438-H
UNITY-500 "nmru500"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 51.1 degrees
Acq. time 1.888 sec
Width 3999.6 Hz
Arrayed repetitions
OBSERVE H1, 499.9270436 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 16384
Total time 27 minutes

Cytosporone C - ^1H NMR

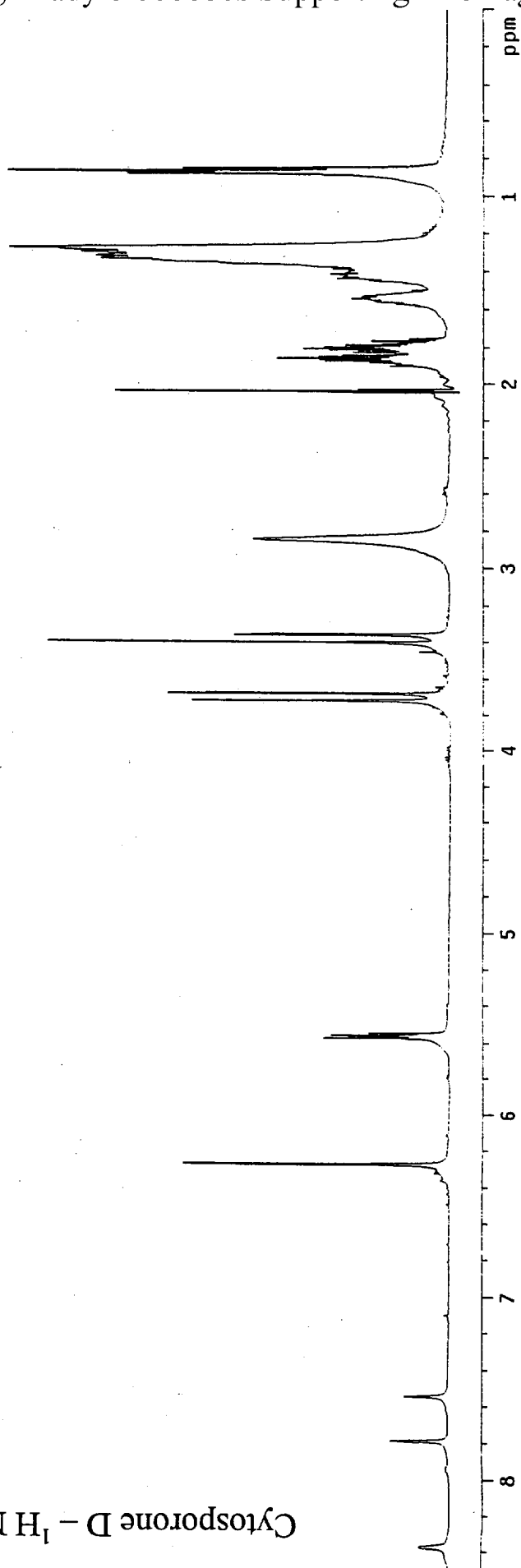
Organic Letters - Cytosporones

LS



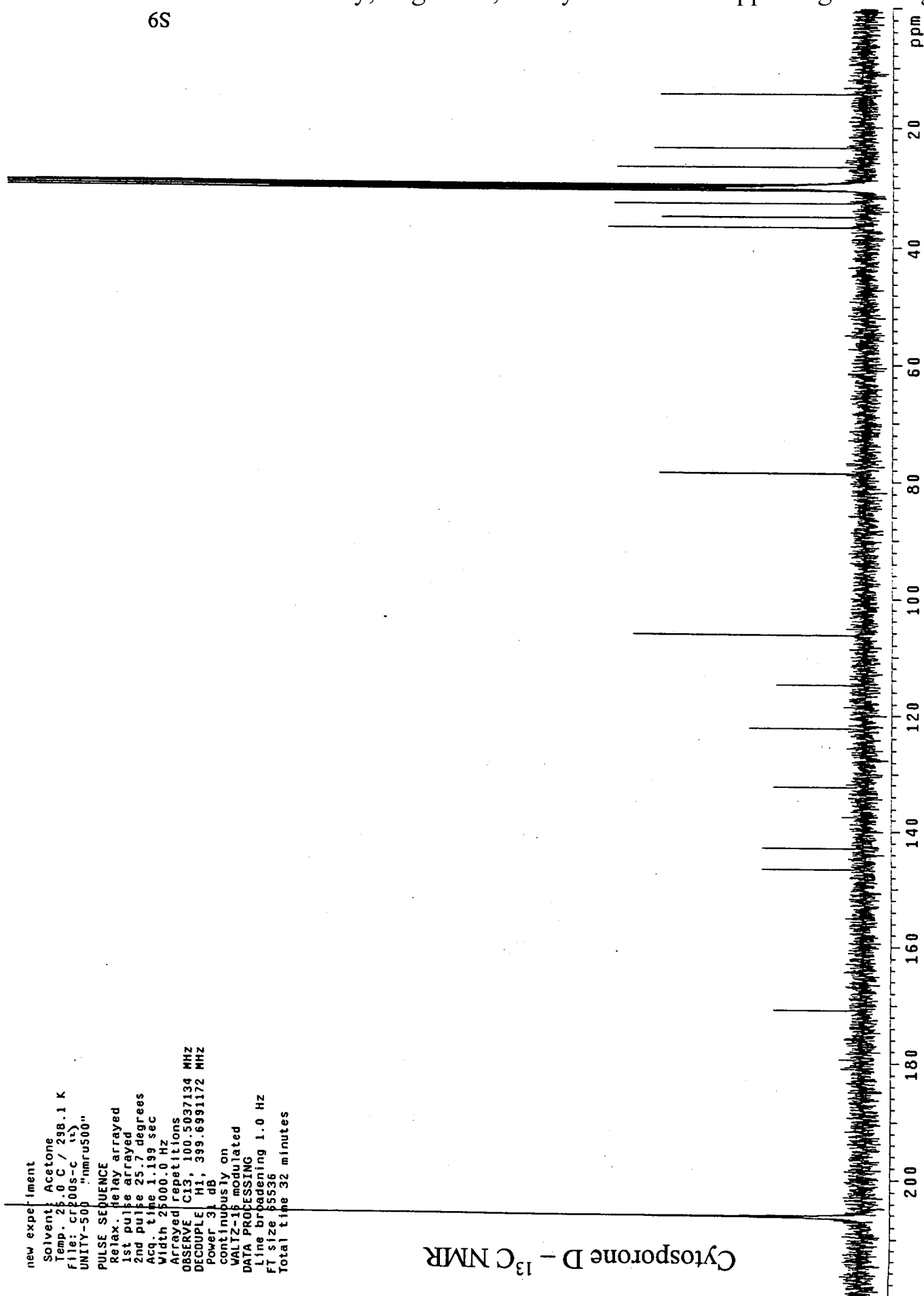
8S

STANDARD PROTON PARAMETERS
Solvent: Acetone
Temp. 25.0 C / 298.1 K
File: CR200s-2-H-Ace
UNITY-500 "nmru500"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 51.1 degrees
Acq. time 0.640 sec
Width 12001.2 Hz
Arrayed repetitions
OBSERVE H1, 499.9270491 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 16384
Total time 17 minutes

Cytosporone D - ^1H NMR

6S

new experiment
 Solvent: Acetone
 Temp. 25.0 C / 298.1 K
 File: cr200s-c 11
 UNITY-500 "nmru500"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 25.7 degrees
 Acq. time 1.199 sec
 Width 25000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 100.5037134 MHz
 DECOUPLE H1, 399.6991172 MHz
 Power 31 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 32 minutes

Cytosporone D - ^{13}C NMR

Organic Letters - Cytosporones

01S

STANDARD PROTON PARAMETERS

Solvent: Acetone
 Temp. 25.0 C / 298.1 K
 File: cr200-s-3t1c-h
 UNITY-500 "nmrus00"

PULSE SEQUENCE

Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 51.1 degrees
 Acq. time 1.892 sec
 Width 8000.0 Hz

Arrayed repetitions

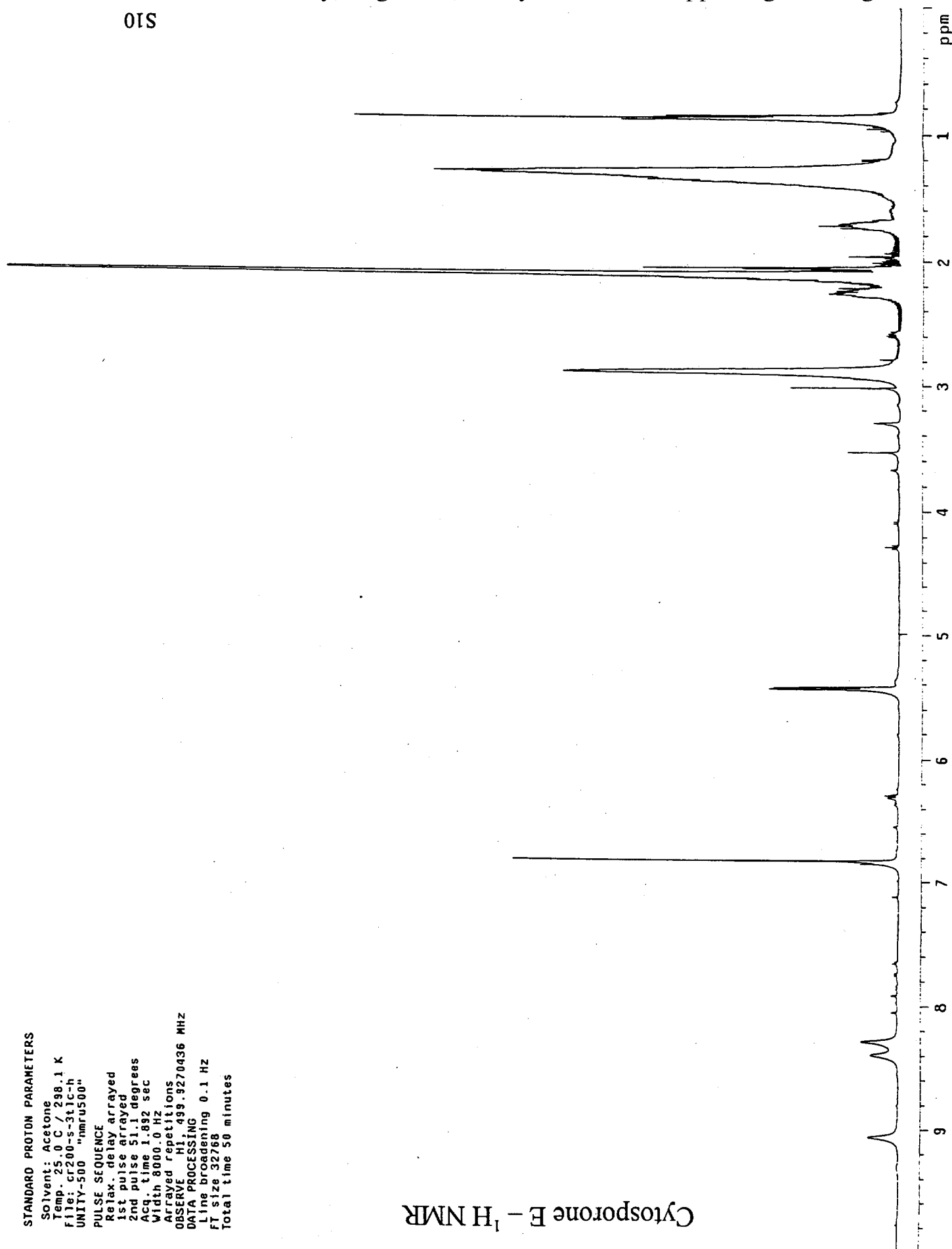
OBSERVE H1 499.9270436 MHz

DATA PROCESSING

line broadening 0.1 Hz

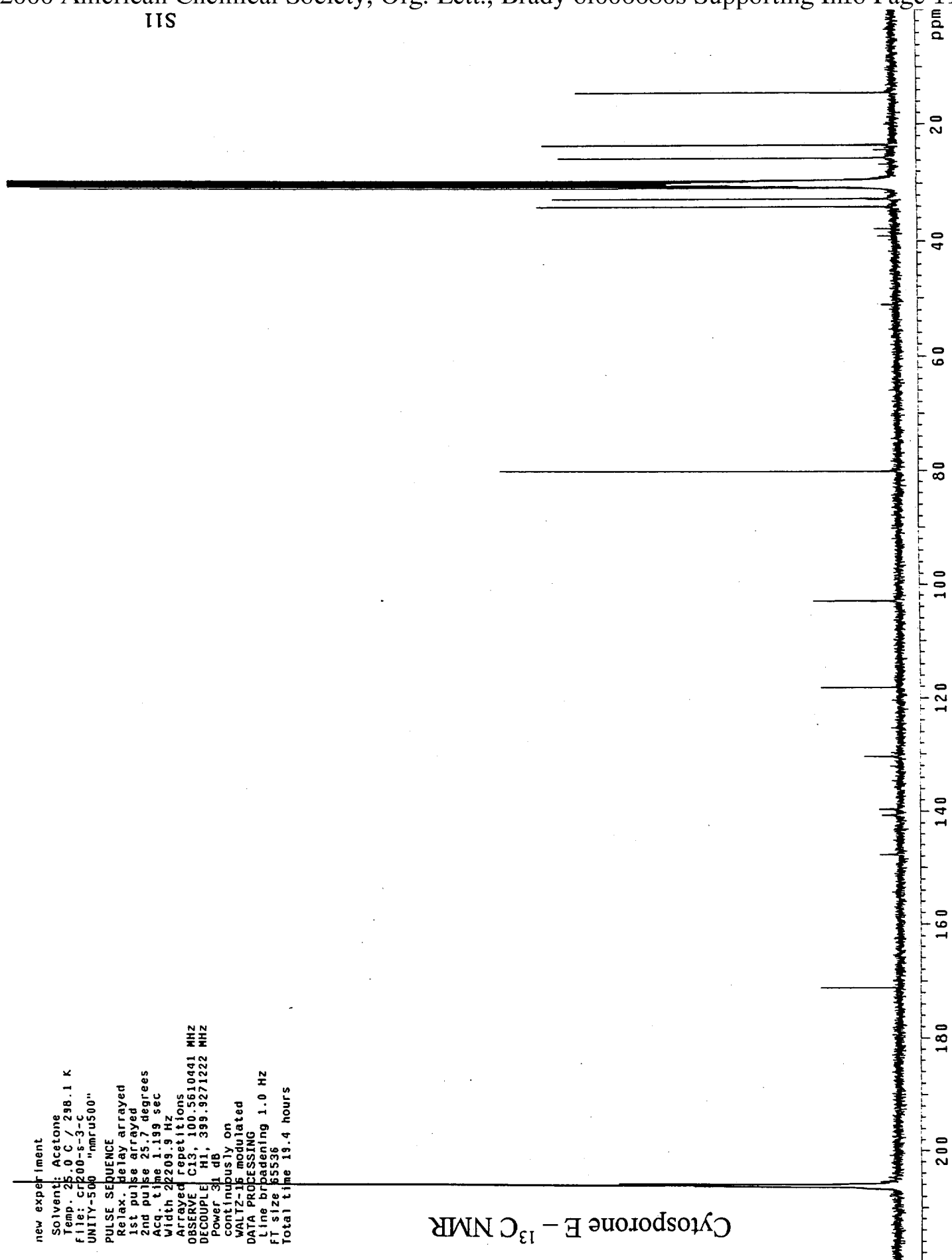
FT size 32768

Total time 50 minutes

Cytosporone E - ^1H NMR

IIS

new experiment
 Solvent: Acetone
 Temp. 25.0 C / 298.1 K
 File: cr200-s-3-c
 UNITY-500 "nmru500"
 PULSE SEQUENCE
 Relax. Delay arrayed
 1st pulse arrayed
 2nd pulse 25.7 degrees
 Acq. time 1.199 sec
 Width 20209.9 Hz
 Arrayed repetitions
 OBSERVE C13, 100.5610441 MHz
 DECOUPLE H1, 399.3271222 MHz
 Power 31 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 19.4 hours

Cytosporone E - ^{13}C NMR

Organic Letters - Cytosporones

Table 1. Crystal data and structure refinement for Cytosporone C.

Identification code	Cytosporone C	
Empirical formula	C ₁₆ H ₂₄ O ₅	
Formula weight	296.35	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 4.8430(6) Å	α = 86.5170(4)°.
	b = 8.6725(10) Å	β = 89.810(2)°.
	c = 19.681(2) Å	γ = 77.518(2)°.
Volume	805.57(17) Å ³	
Z	2	
Density (calculated)	1.222 Mg/m ³	
Absorption coefficient	0.090 mm ⁻¹	
F(000)	320	
Crystal size	0.4 x 0.4 x 0.05 mm ³	
Theta range for data collection	2.07 to 20.82°.	
Index ranges	-4 ≤ h ≤ 4, -8 ≤ k ≤ 7, -17 ≤ l ≤ 19	
Reflections collected	2714	
Independent reflections	1638 [R(int) = 0.0498]	
Completeness to theta = 20.82°	97.3 %	
Absorption correction	SADABS	
Max. and min. transmission	1 and 0.434045	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	1638 / 0 / 283	
Goodness-of-fit on F ²	1.284	
Final R indices [I > 2σ(I)]	R1 = 0.0769, wR2 = 0.1969	
R indices (all data)	R1 = 0.1051, wR2 = 0.2170	
Largest diff. peak and hole	0.205 and -0.512 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Cytosporone C. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3803(10)	9593(6)	3215(3)	68(2)
O(2)	-3320(12)	3771(8)	4629(3)	60(2)
O(3)	3231(10)	2746(6)	2813(3)	65(2)
O(4)	4376(9)	7357(5)	2749(2)	45(1)
C(1)	2968(14)	8377(10)	3151(4)	52(2)
C(2)	461(17)	8066(10)	3517(6)	62(2)
C(3)	322(12)	6347(7)	3615(3)	37(2)
C(4)	-1388(13)	5854(8)	4112(3)	44(2)
C(5)	-1588(13)	4291(8)	4155(3)	40(2)
C(6)	-89(14)	3238(10)	3731(3)	47(2)
C(7)	1639(12)	3746(8)	3246(3)	41(2)
C(8)	1803(11)	5324(8)	3173(3)	37(2)
C(9)	3491(15)	5873(8)	2605(3)	44(2)
C(10)	1916(15)	6150(11)	1931(3)	46(2)
C(11)	3595(16)	6630(11)	1328(4)	53(2)
C(12)	1913(18)	6904(12)	672(4)	60(2)
C(13)	3428(18)	7485(13)	72(4)	64(2)
C(14)	1690(20)	7845(15)	-574(4)	72(3)
C(15)	3140(20)	8508(15)	-1168(5)	85(3)
C(16)	1370(30)	8960(20)	-1786(6)	109(5)
O(1S)	-4000(20)	10790(10)	4482(5)	168(4)
H(1S)	-5000	10000	5000	149(4)

Table 3. Bond lengths [Å] and angles [°] for Cytosporone C.

O(1)-C(1)	1.222(8)
O(2)-C(5)	1.372(8)
O(3)-C(7)	1.368(7)
O(4)-C(1)	1.303(8)
O(4)-C(9)	1.484(7)
C(1)-C(2)	1.476(10)
C(2)-C(3)	1.507(10)
C(3)-C(8)	1.369(8)
C(3)-C(4)	1.386(9)
C(4)-C(5)	1.377(9)
C(5)-C(6)	1.364(9)
C(6)-C(7)	1.381(9)
C(7)-C(8)	1.387(9)
C(8)-C(9)	1.497(9)
C(9)-C(10)	1.513(10)
C(10)-C(11)	1.524(9)
C(11)-C(12)	1.508(10)
C(12)-C(13)	1.506(10)
C(13)-C(14)	1.507(13)
C(14)-C(15)	1.509(12)
C(15)-C(16)	1.472(13)
O(1S)-H(1S)	1.340(8)
H(1S)-O(1S)#1	1.340(8)

C(1)-O(4)-C(9)	122.5(5)
O(1)-C(1)-O(4)	117.6(6)
O(1)-C(1)-C(2)	121.5(7)
O(4)-C(1)-C(2)	120.9(7)
C(1)-C(2)-C(3)	115.3(7)
C(8)-C(3)-C(4)	121.7(6)
C(8)-C(3)-C(2)	117.6(6)
C(4)-C(3)-C(2)	120.5(6)
C(5)-C(4)-C(3)	118.6(6)
O(2)-C(5)-C(4)	120.1(6)
O(2)-C(5)-C(6)	118.9(7)
C(4)-C(5)-C(6)	121.0(6)
C(7)-C(6)-C(5)	119.6(7)
O(3)-C(7)-C(6)	122.5(6)
O(3)-C(7)-C(8)	116.7(6)
C(6)-C(7)-C(8)	120.7(6)
C(3)-C(8)-C(7)	118.3(6)
C(3)-C(8)-C(9)	121.9(6)
C(7)-C(8)-C(9)	119.8(6)
O(4)-C(9)-C(8)	112.2(5)
O(4)-C(9)-C(10)	107.8(6)
C(8)-C(9)-C(10)	112.9(5)
C(9)-C(10)-C(11)	115.0(6)
C(12)-C(11)-C(10)	112.7(6)
C(11)-C(12)-C(13)	114.4(7)
C(14)-C(13)-C(12)	114.4(7)
C(13)-C(14)-C(15)	114.7(8)
C(16)-C(15)-C(14)	114.6(9)
O(1S)#1-H(1S)-O(1S)	179.998(5)

Symmetry transformations used to generate equivalent atoms:

#1 -x-1, -y+2, -z+1

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Cytosporone C. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
O(1)	65(3)	41(3)	104(4)	-3(3)	15(3)	-29(3)
O(2)	70(4)	56(5)	62(4)	-12(3)	31(3)	-28(3)
O(3)	82(4)	40(4)	77(4)	-9(3)	40(3)	-22(3)
O(4)	44(3)	45(3)	55(3)	-5(2)	10(2)	-27(2)
C(1)	41(4)	46(5)	69(5)	4(4)	2(4)	-15(4)
C(2)	49(5)	41(5)	101(7)	-20(5)	30(5)	-19(4)
C(3)	29(4)	30(4)	53(4)	-7(3)	9(3)	-9(3)
C(4)	41(4)	45(5)	47(4)	-12(4)	13(3)	-12(4)
C(5)	41(4)	43(5)	40(4)	3(3)	11(3)	-20(4)
C(6)	55(5)	34(5)	57(5)	1(4)	9(4)	-18(4)
C(7)	44(4)	35(5)	45(4)	-7(3)	14(3)	-12(3)
C(8)	31(4)	38(5)	42(4)	3(3)	2(3)	-12(3)
C(9)	42(5)	39(5)	55(5)	-1(3)	8(4)	-19(4)
C(10)	45(5)	52(5)	48(5)	-2(4)	8(4)	-25(5)
C(11)	50(5)	62(6)	49(5)	-3(4)	9(4)	-21(5)
C(12)	61(5)	74(7)	51(5)	-1(4)	7(4)	-31(5)
C(13)	59(5)	82(7)	54(5)	2(4)	14(4)	-24(5)
C(14)	64(7)	98(8)	56(6)	-3(5)	9(5)	-24(6)
C(15)	68(6)	126(9)	57(6)	11(6)	9(5)	-19(7)
C(16)	97(9)	151(14)	66(7)	23(7)	22(7)	-7(8)
O(1S)	231(9)	117(7)	174(8)	-1(5)	77(8)	-77(6)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Cytosporone C.

	x	y	z	U(eq)
H(2O)	-3410(140)	4360(80)	4790(30)	40(20)
H(3O)	3310(110)	1720(80)	2960(30)	34(17)
H(2B)	-1220(170)	8730(90)	3220(40)	70(30)
H(2A)	80(160)	8460(90)	3910(40)	70(30)
H(4A)	-2420(120)	6620(70)	4420(30)	45(17)
H(6A)	-190(110)	2260(80)	3780(30)	26(18)
H(9A)	5490(150)	5230(80)	2610(30)	50(20)
H(10B)	60(150)	7040(80)	1960(30)	50(20)
H(10A)	1410(110)	5260(80)	1820(30)	42(18)
H(11B)	5410(170)	5760(100)	1290(40)	80(30)
H(11A)	3960(160)	7650(110)	1450(40)	90(30)
H(12B)	160(180)	7770(100)	700(40)	90(30)
H(12A)	1250(140)	5970(90)	550(30)	60(20)
H(13B)	5230(160)	6720(90)	-40(30)	70(20)
H(13A)	4210(190)	8200(110)	120(40)	110(40)
H(14B)	-30(170)	8500(90)	-580(40)	100(30)
H(14A)	1290(170)	6920(110)	-750(40)	100(30)
H(15B)	5000(200)	7710(110)	-1260(40)	110(30)
H(15A)	4500(200)	9170(120)	-1130(50)	140(40)
H(16C)	900(400)	8100(200)	-1900(80)	170(100)
H(16B)	2490(170)	9320(100)	-2120(50)	100(30)
H(16A)	-600(200)	9510(120)	-1660(50)	110(40)
H(2S)	-1840(100)	10520(60)	5120(30)	42(15)