

Supporting Information

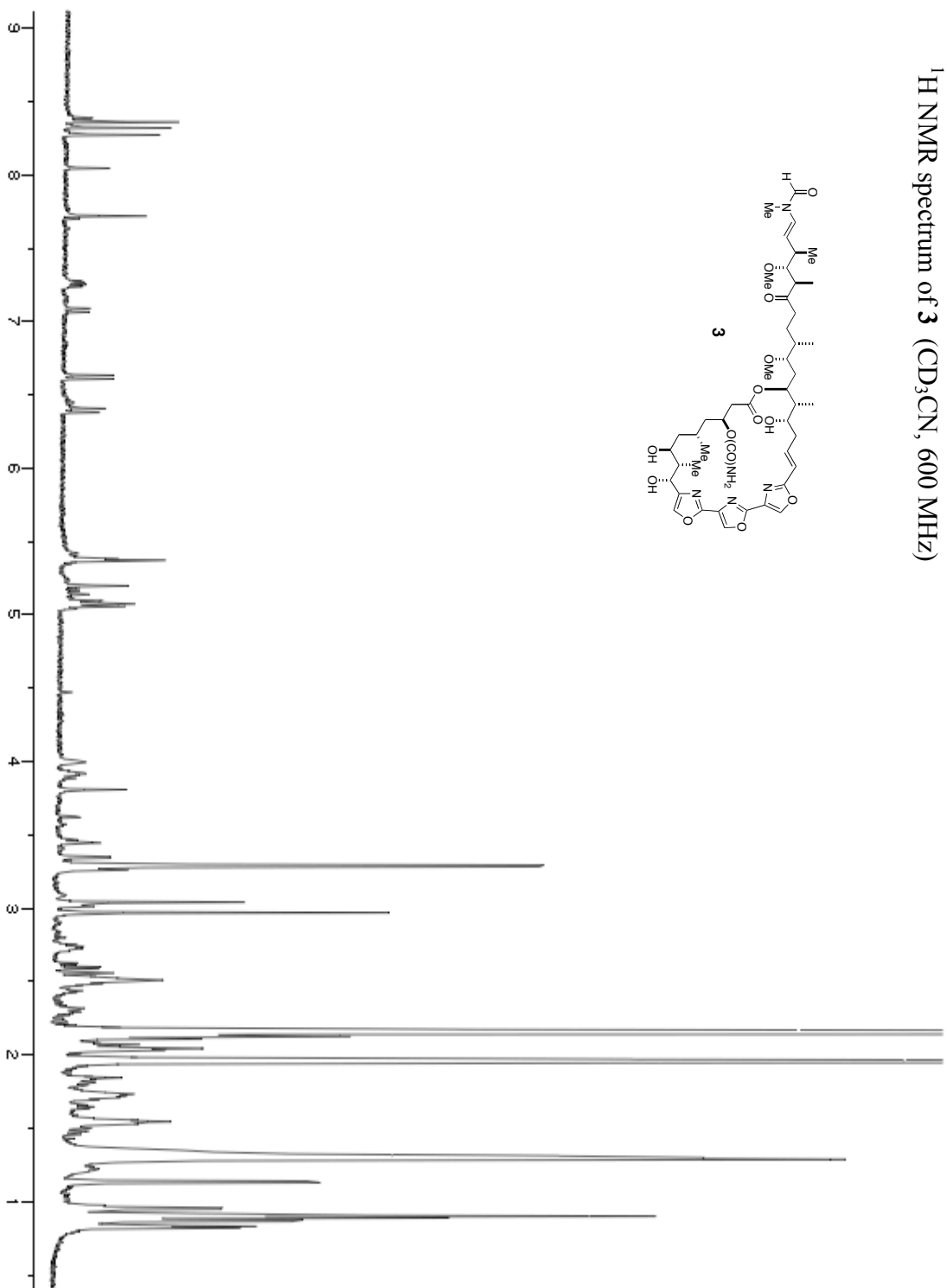
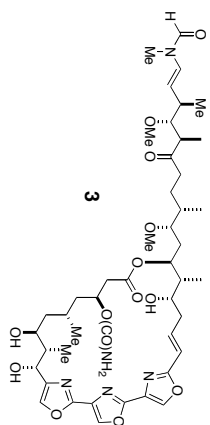
Structure Elucidation at the Nanomole-Scale. 1. Trisoxazole macrolides and Thiazole-containing cyclic peptides from the Nudibranch *Hexabranchnus sanguineus*.

Doralyn S. Dalisay[†], *Evan W. Rogers*[†], *Arthur S. Edison*[‡], *Tadeusz F. Molinski*^{*,†,§}

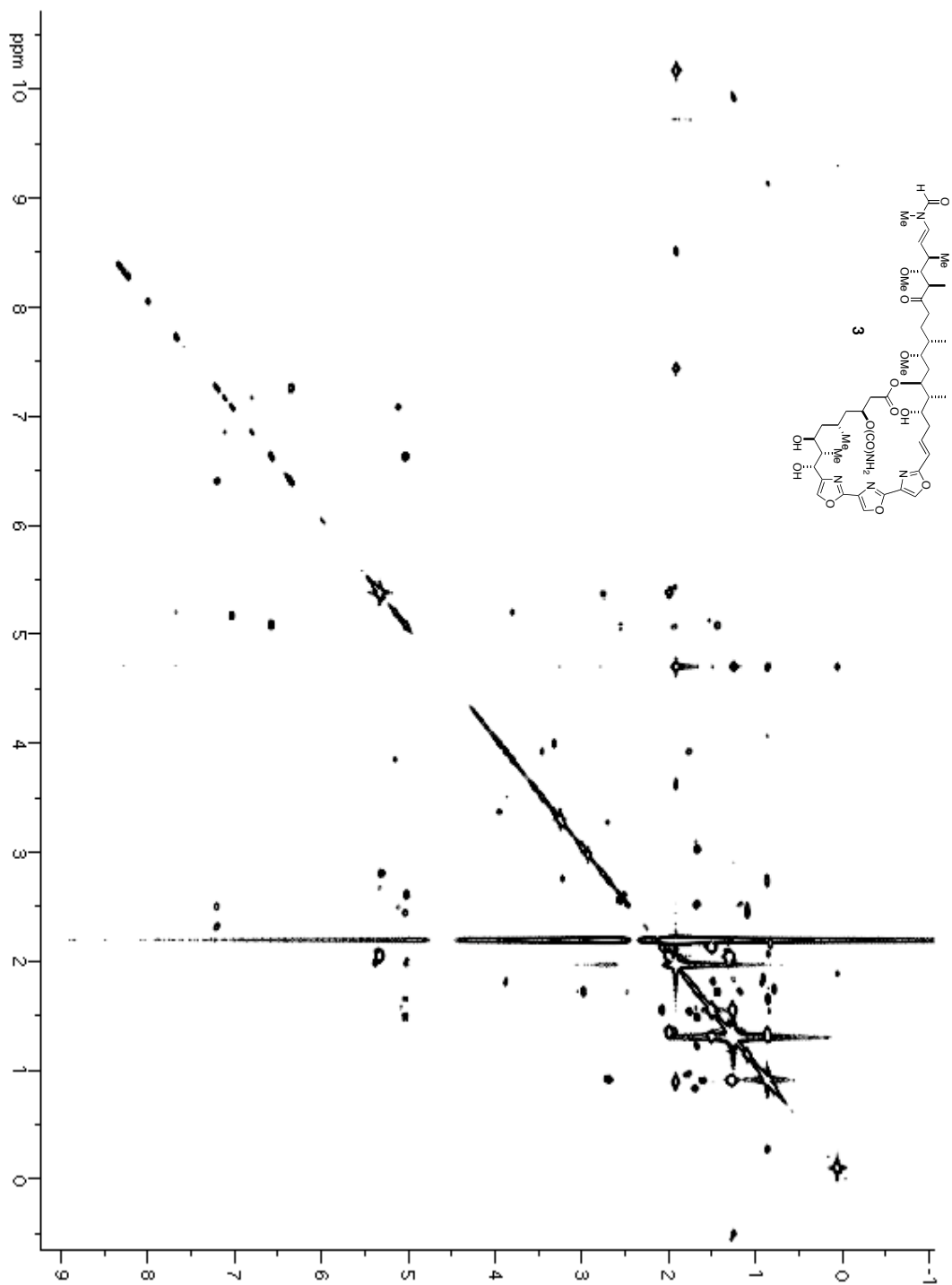
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Pharmaceutical Sciences, MC0358, 9500 Gilman Drive, La Jolla, California 92093-0358,
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¹ H NMR spectrum of 9-des-O-methyl kaabiramide, 3	S2
COSY spectrum of 3	S3
HSQC spectrum of 3	S4
HMBC spectrum of 3	S5
¹ H NMR spectrum of 33-methyl tetrahydrohalichondramide, 5	S6
COSY spectrum of 5	S7
TOCSY spectrum of 5	S8
HSQC spectrum of 5	S9
HMBC spectrum of 5	S10
¹ H NMR spectrum of sanguinamide A, 7	S11
COSY spectrum of 7	S12
TOCSY spectrum of 7	S13
HSQC spectrum of 7	S14
HMBC spectrum of 7	S15
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COSY spectrum of 8	S17
TOCSY spectrum of 8	S18
HSQC spectrum of 8	S19
HMBC spectrum of 8	S20
CD spectra of 7 and 8	S21

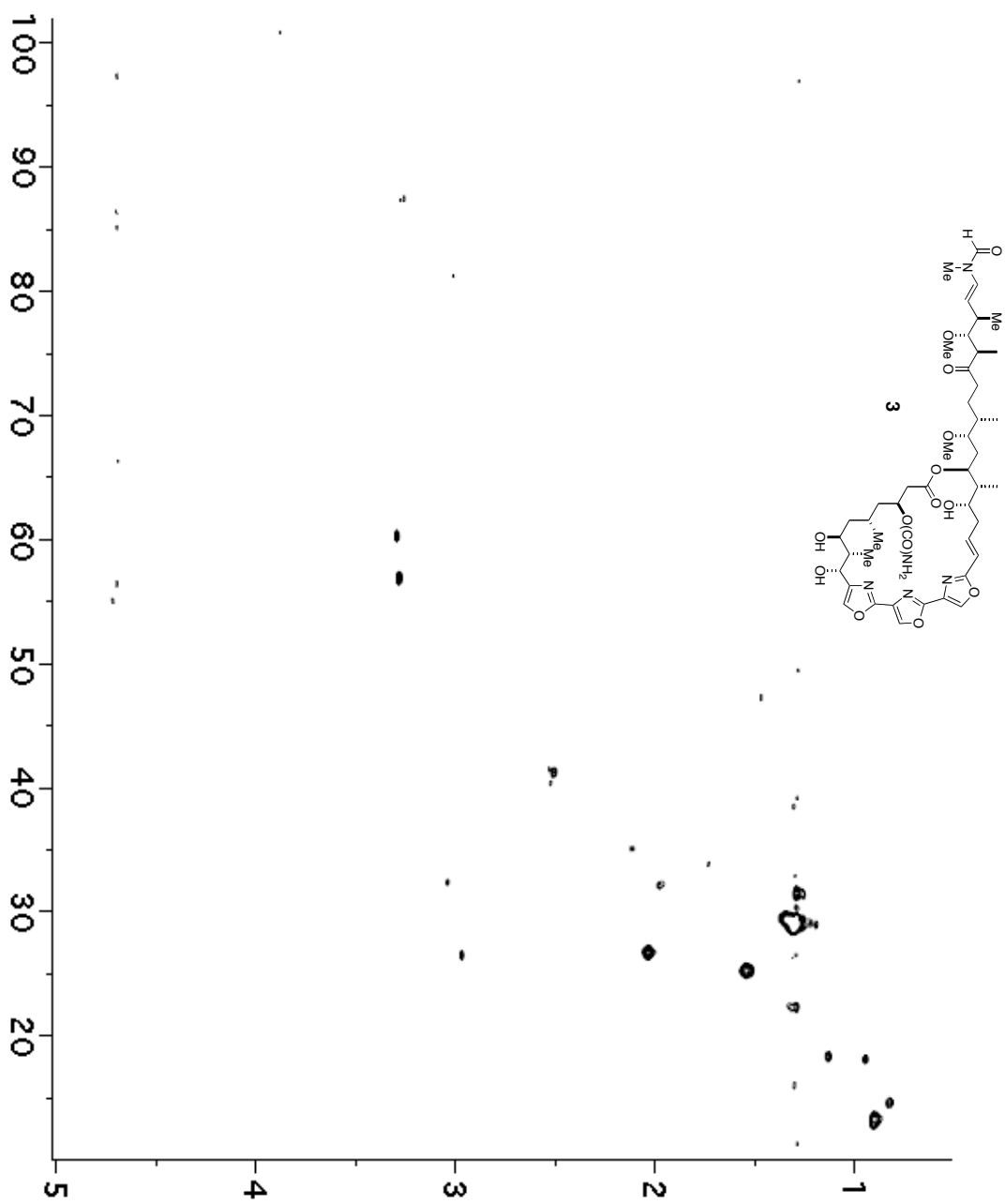
¹H NMR spectrum of **3** (CD₃CN, 600 MHz)



COSY NMR spectrum of **3** (CD₃CN, 600 MHz)



HSQC NMR spectrum of **3** (CD₃CN, 600 MHz)

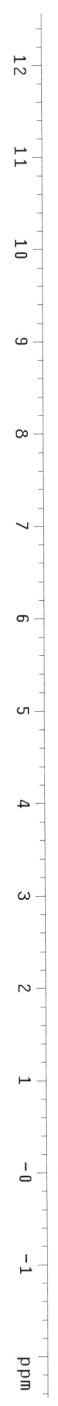
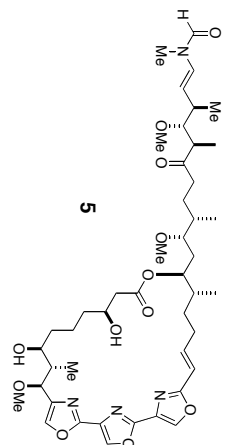


¹H NMR spectrum of **5** (CDCl₃, 400 MHz)

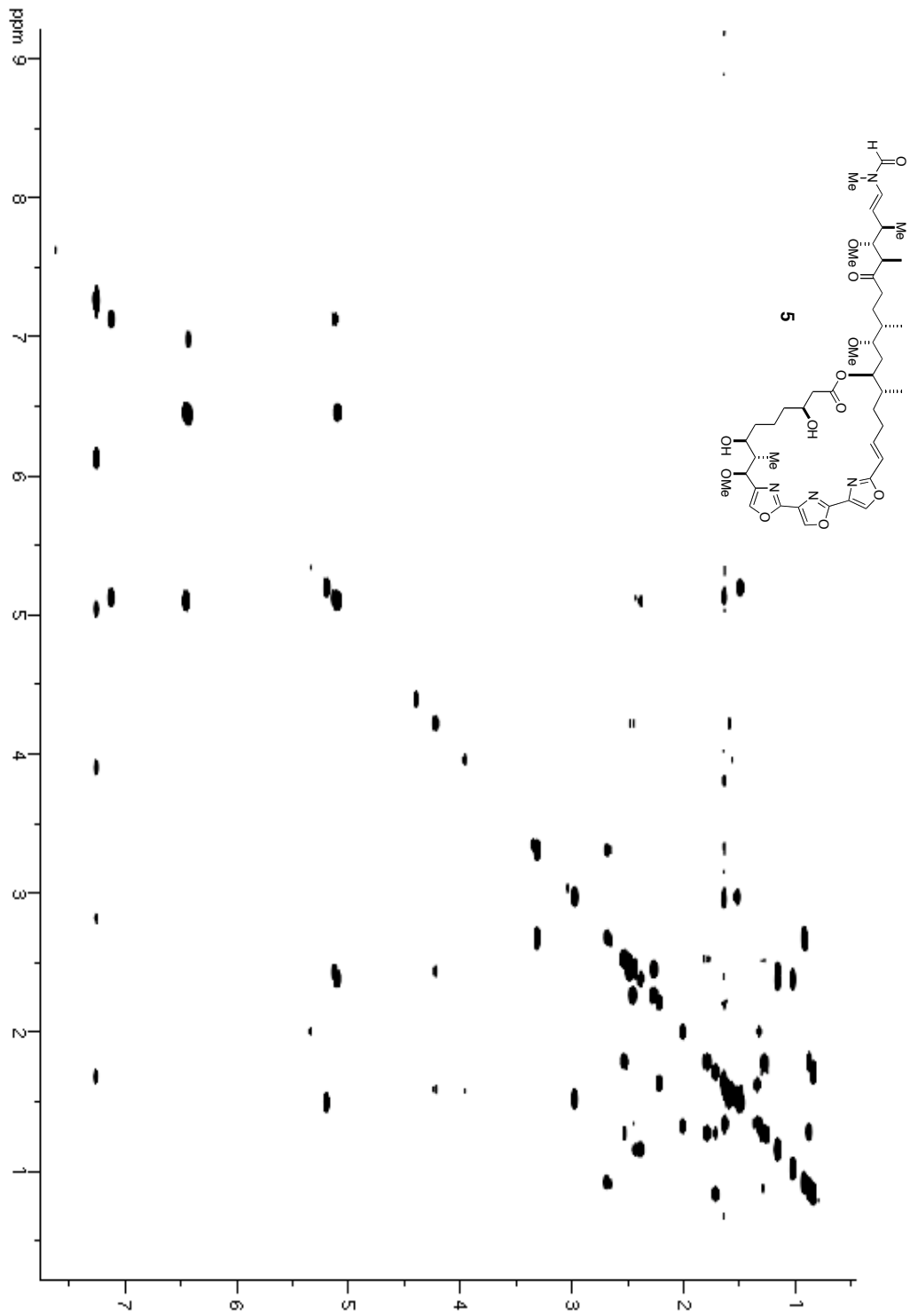
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90.001.5BF10F6F3F3F2.A
exp1 std1h
SAMPLE Dec: & VT
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solvent CDCl3 dnmr 30
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sfrfq 400.125 dm nmn
dn 1.41 dnm
dd 23936 dm wtfilc 200
sw 6006.0 not used proc fi
fb not used fn not used
bs 5.4
dwmr 7.0 weff
dl 1.000 wexp
d1 0 wbs
nt 94 wnt
f 2
atlock n
gain not used
flags n
11 n
11 n
11 n
dp y
DISPLAY
sp -977.2
wp 6005.48
vc 2.48
sc 0
wc 250
hzmm 50.44
fs 0.44
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f2 0
th 20
ins cdc ph
100.000

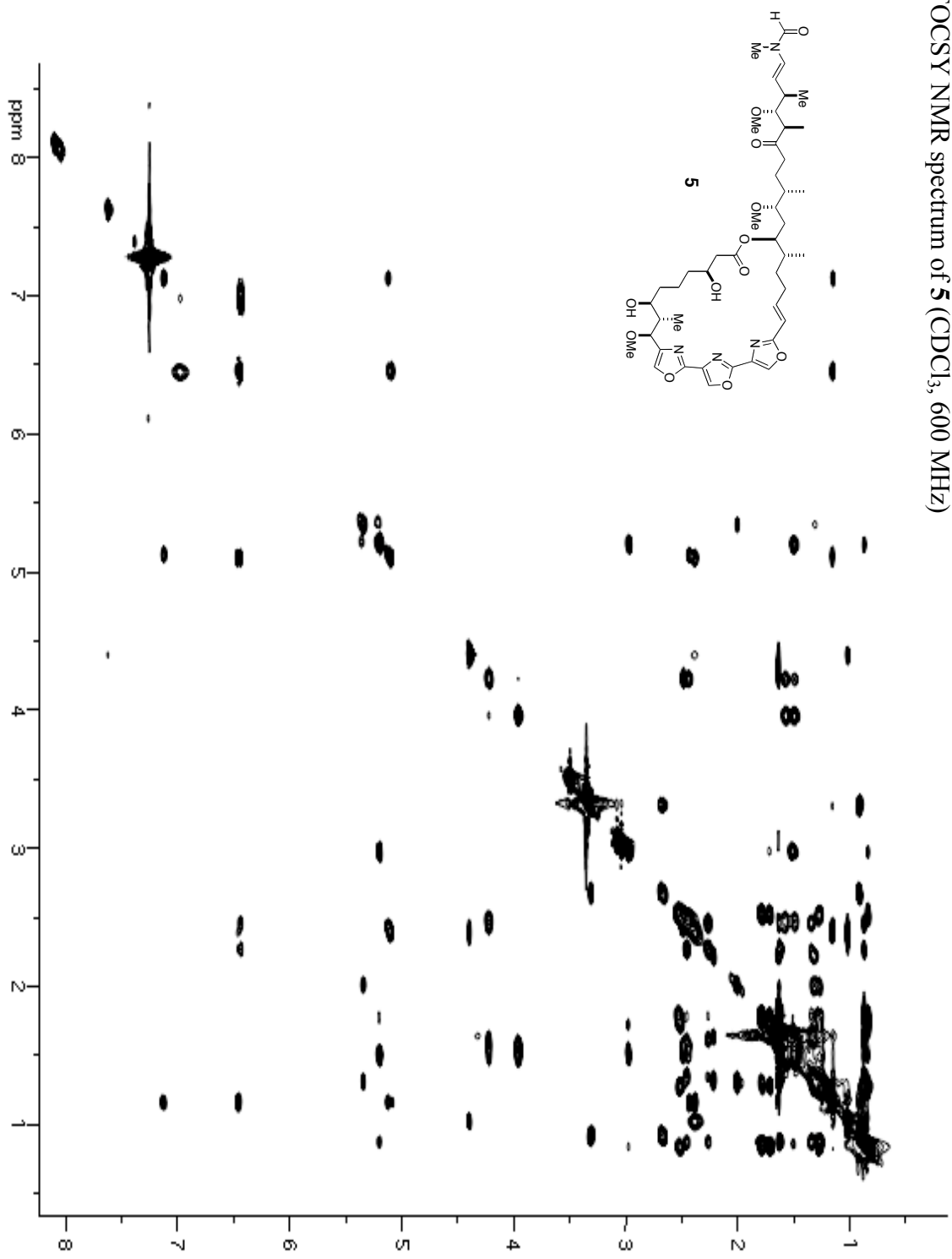
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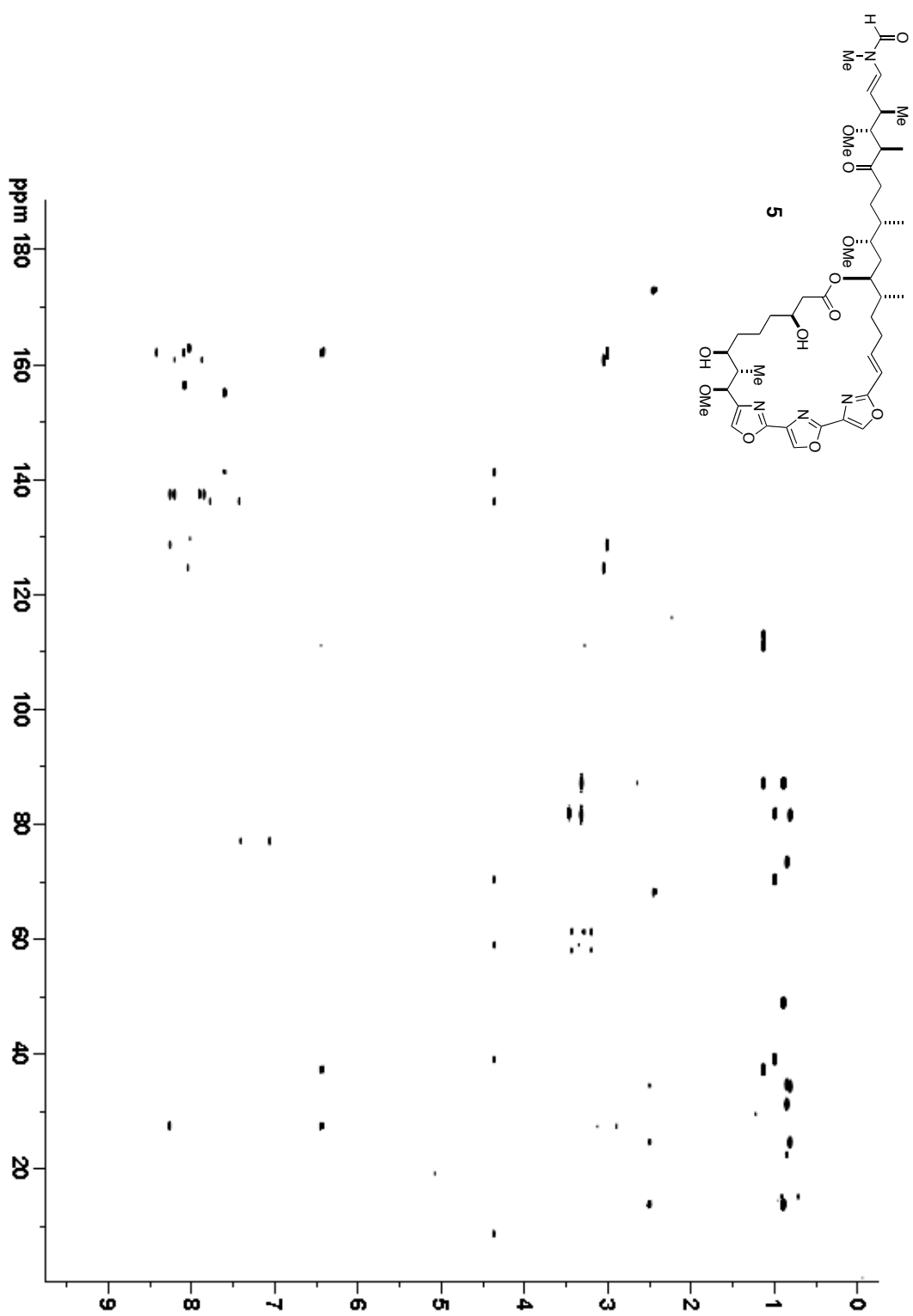
COSY NMR spectrum of **5** (CDCl₃, 600 MHz)



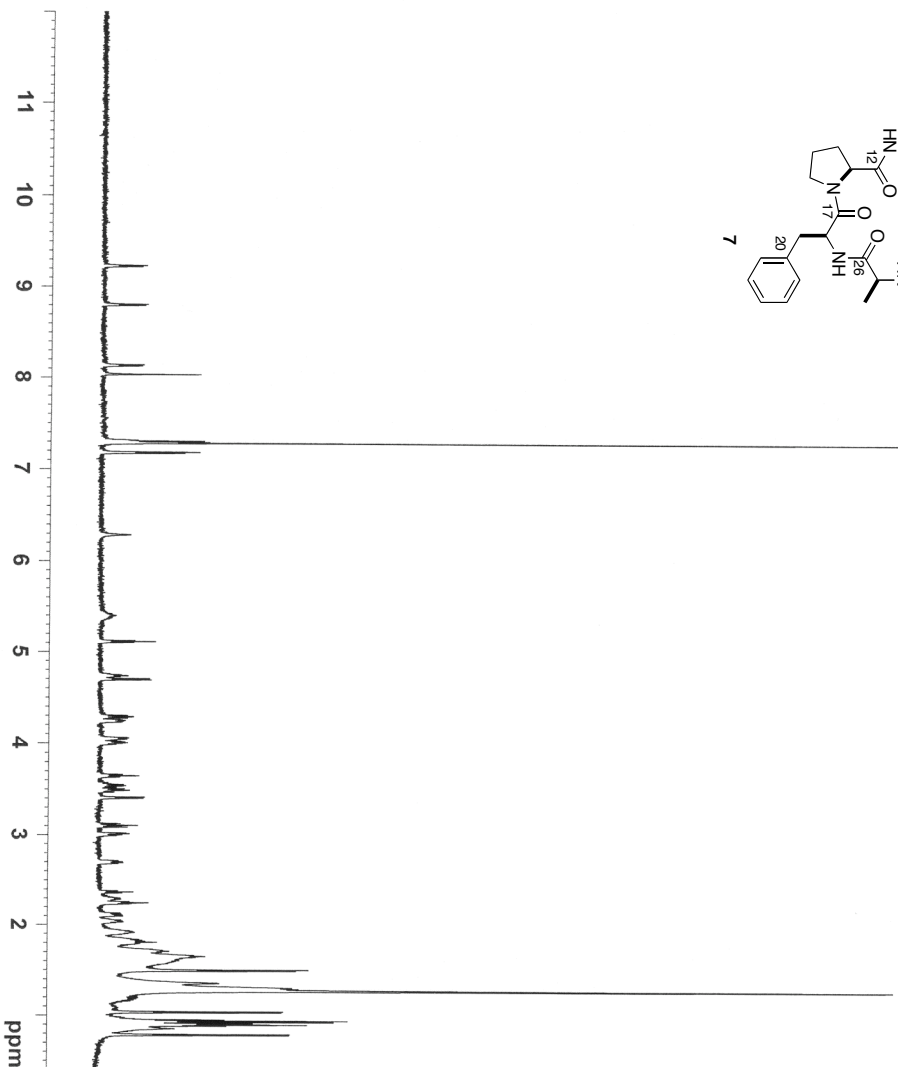
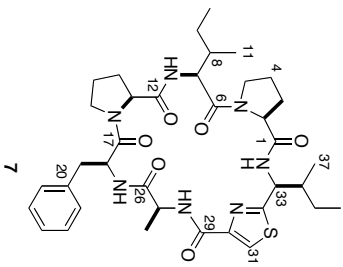
TOCSY NMR spectrum of **5** (CDCl₃, 600 MHz)



HMBC NMR spectrum of **5** (CDCl₃, 600 MHz)

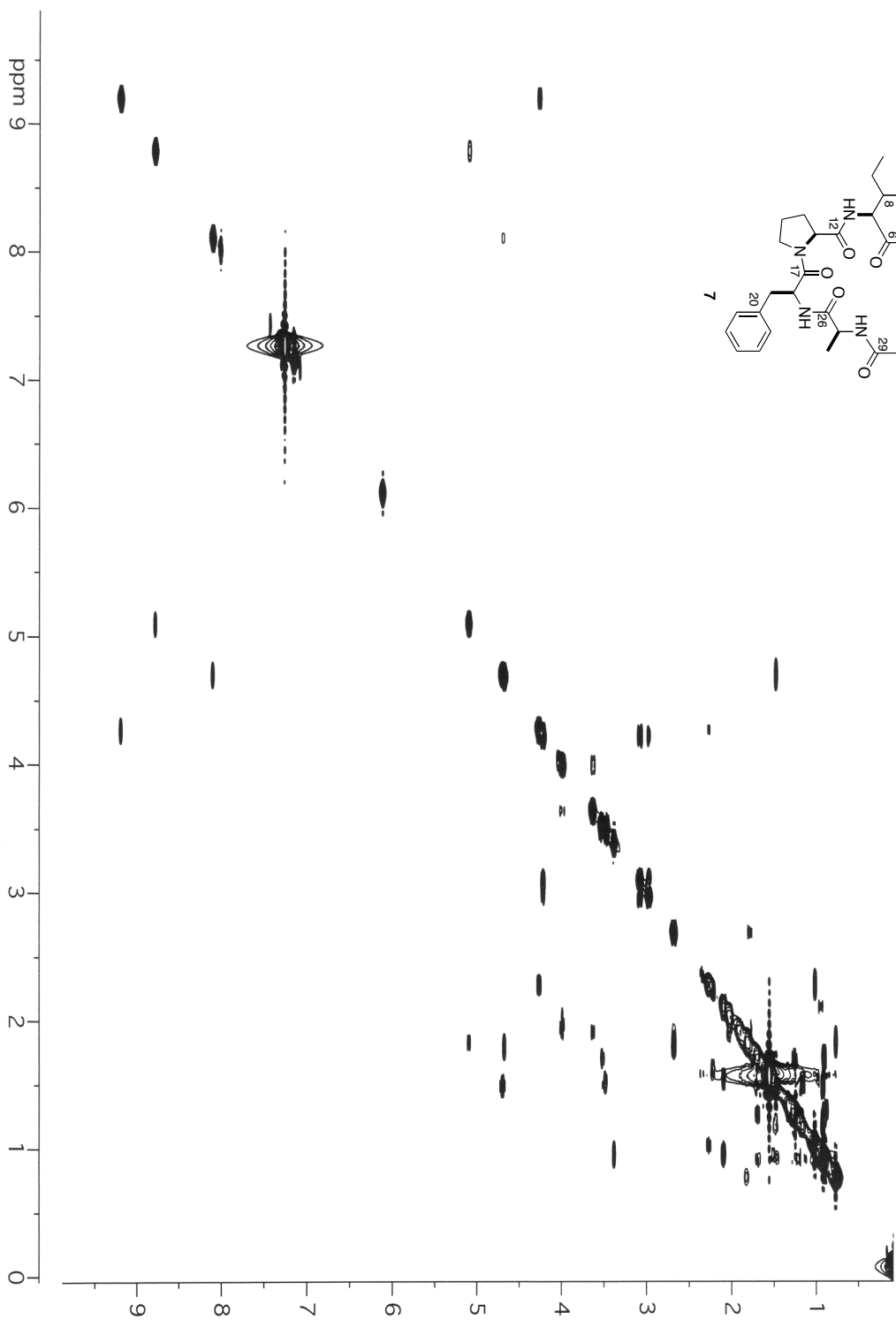
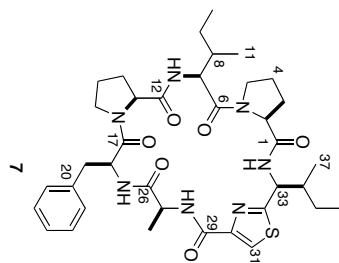


¹H NMR spectrum of 7 (CDCl₃, 600 MHz)

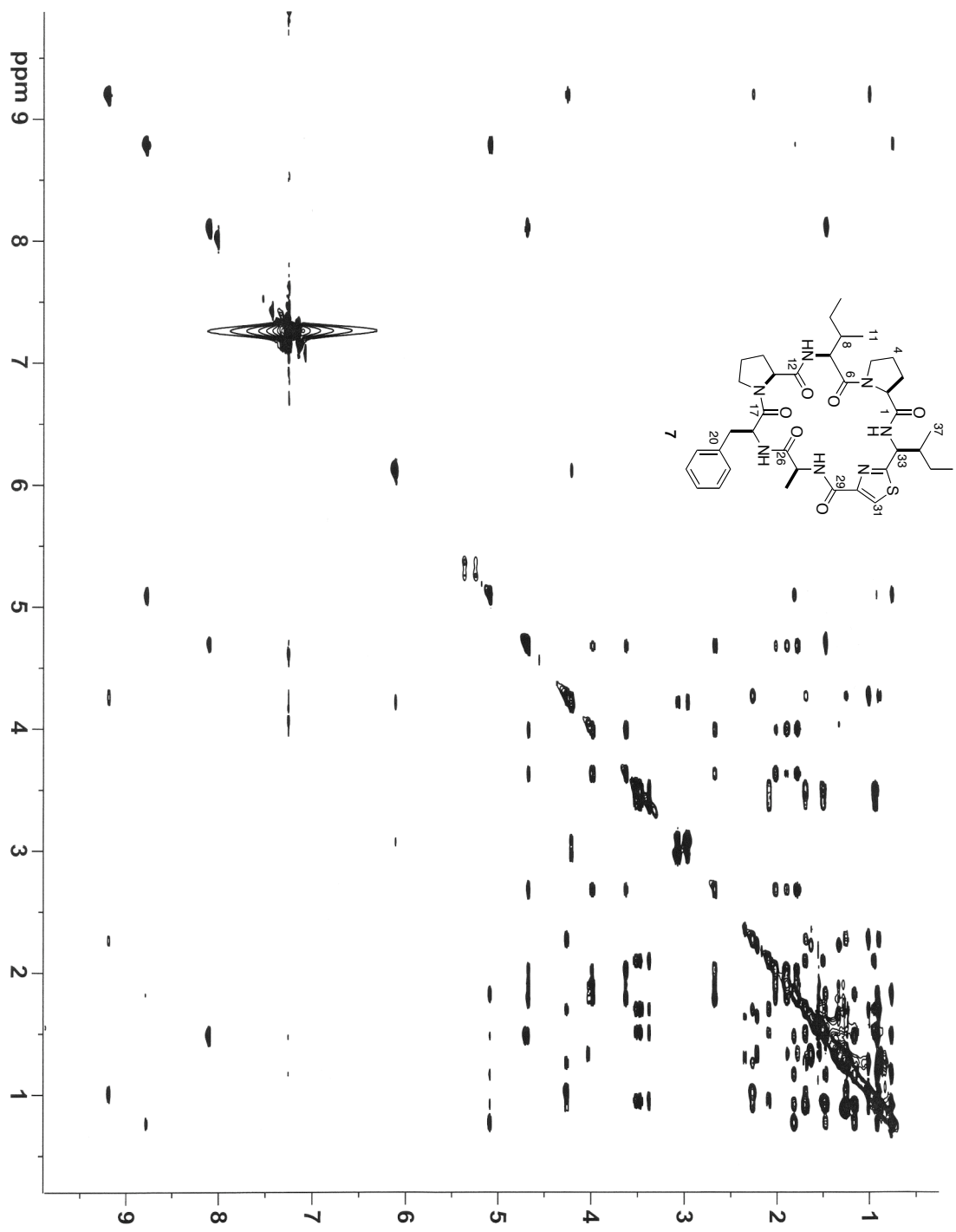


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 Time 6.36
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 F2PROB zgpg30
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1
 DS 0
 SWH 12335.526 Hz
 AQ 0.189228 Hz
 RG 2.6204429
 DE 40.533 usec
 TE 298.0 K
 T1 1.00000000 sec
 T10 1
 CHANNEL f1
 =====
 NUC1 1H
 P1 8.55 usec
 PL1 17.10 dB
 SFO1 600.1337060 MHz
 F2 - Processing parameters
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 SF 600.1300000 MHz
 WDW EM
 SSB 0
 GB 0
 PC 1.00

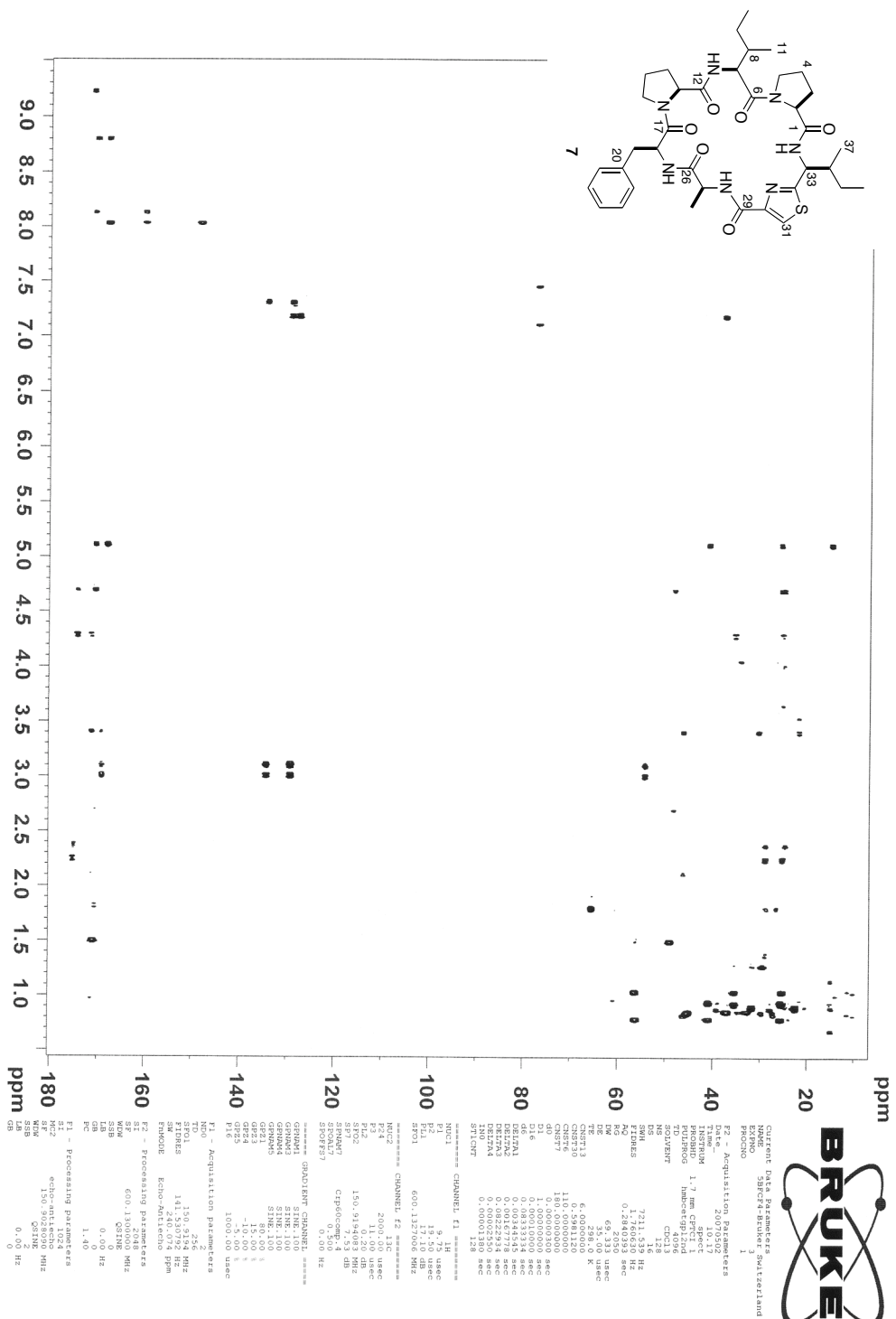
COSY NMR spectrum of 7 (CDCl₃, 600 MHz)



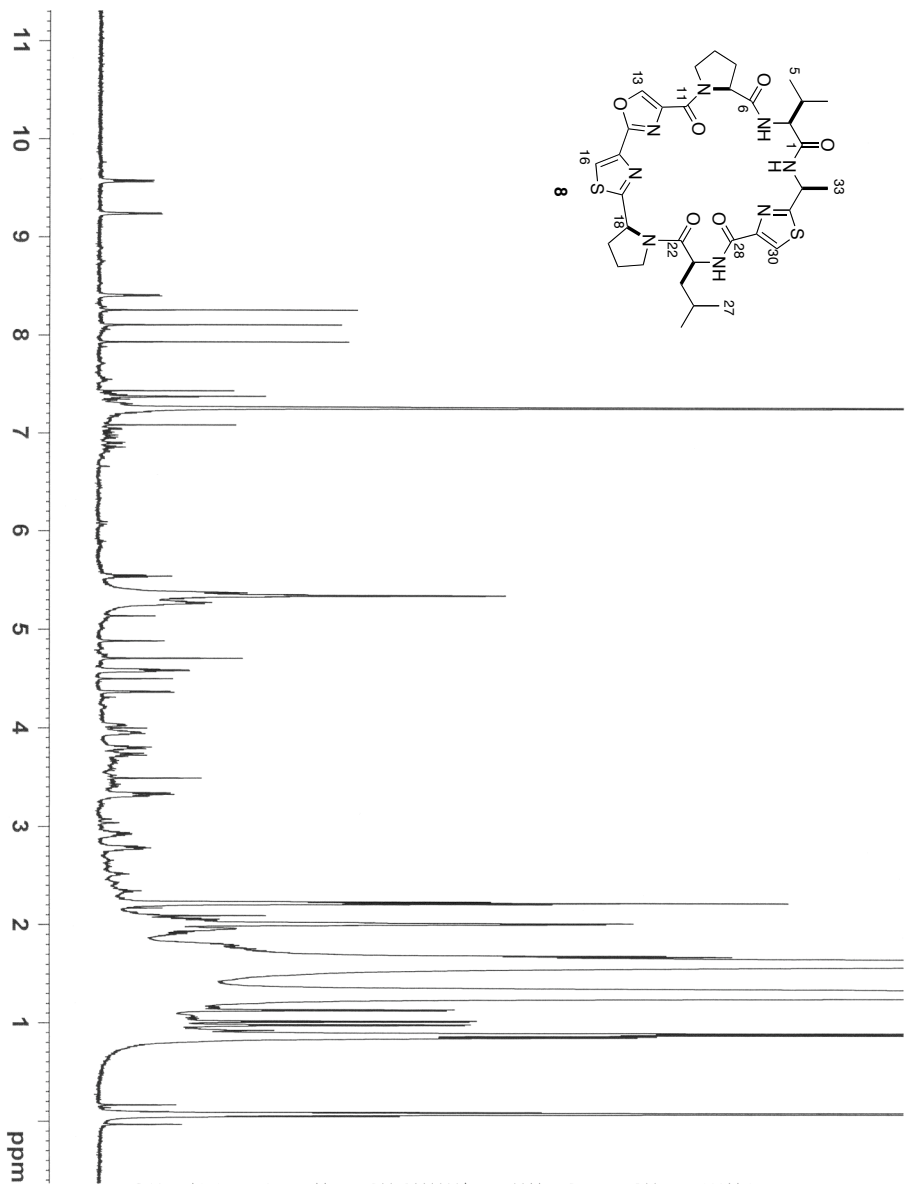
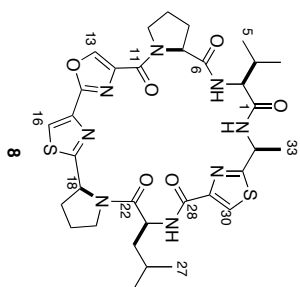
TOCSY NMR spectrum of **7** (CDCl₃, 600 MHz)



HMBC NMR spectrum of 7 (CDCl₃, 600 MHz)



¹H NMR spectrum of **8** (CDCl₃, 600 MHz)



Current Data Parameters
 NAME 5bfct3jucsd
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 PROCNO 1

F2 - Acquisition Parameters
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 Time_ 19.04

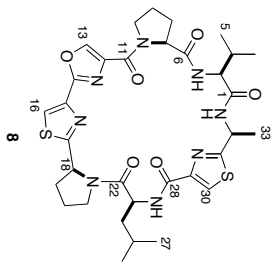
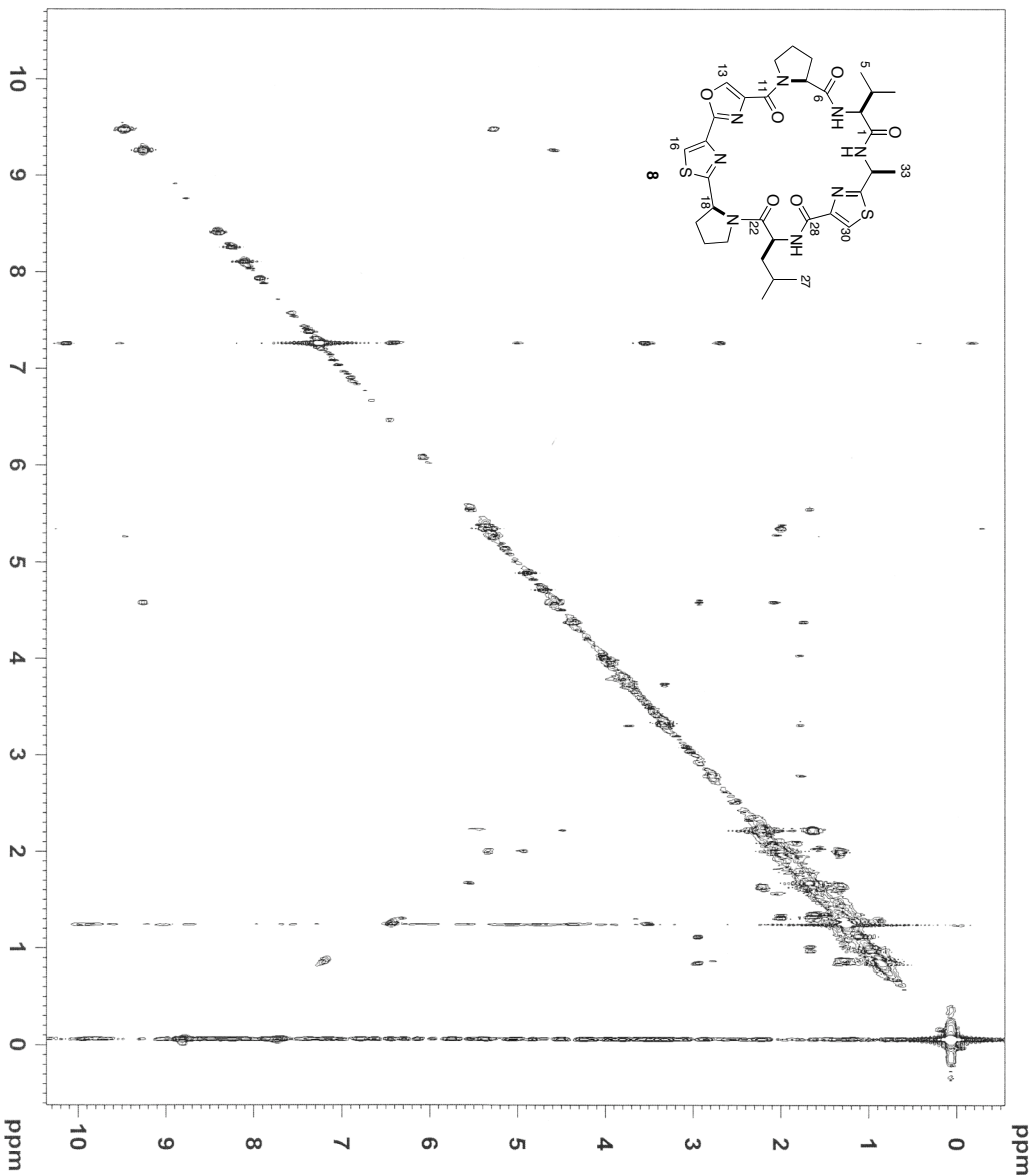
INSTRUM spect
 PROBD 1.7 mm CPTCL1
 PULPROG zg30
 TD 57690
 SOLVENT CDCl3
 NS 128
 DS 2

SWH 9615.385 Hz
 FIDRES 0.166673 Hz
 AQ 2.9999299 sec
 RG 1440
 DW 52.000 usec
 DE 6.50 usec
 TE 303.0 K
 D1 1.50000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.75 usec
 PL1 15.00 dB
 SFO1 599.8241987 MHz

F2 - Processing Parameters
 SI 32768
 SF 599.8200117 MHz
 WDM EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

COSY NMR spectrum of **8** (CDCl₃, 600 MHz)



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Current Data Parameters
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EXPNO    24
PROCNO    1

F2 - Acquisition Parameters
Date_     20080629
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INSTRUM   spect
PROBHD    1.7 mm Cryo
PULPROG   cosympfepb
TD         2048
SOLVENT   CDCl3
NS        8
DS         8
SWH        7211.539 Hz
FIDRES     3.521259 Hz
AQ         0.1420181 sec
RG         512
DM         69.333 usec
DE         6.50 usec
dD         2.00 usec
dE         0.00005738 sec
dL1        1.48689198 sec
dL3        0.00000400 sec
dL6        0.00120000 sec
dL20       0.00120000 sec
IN0        0.00013895 sec
STICNT     128

===== CHANNEL f1 =====
NUC1       1H
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P2         19.00 usec
PC         15.00 usec
SFO1       599.8239991 MHz

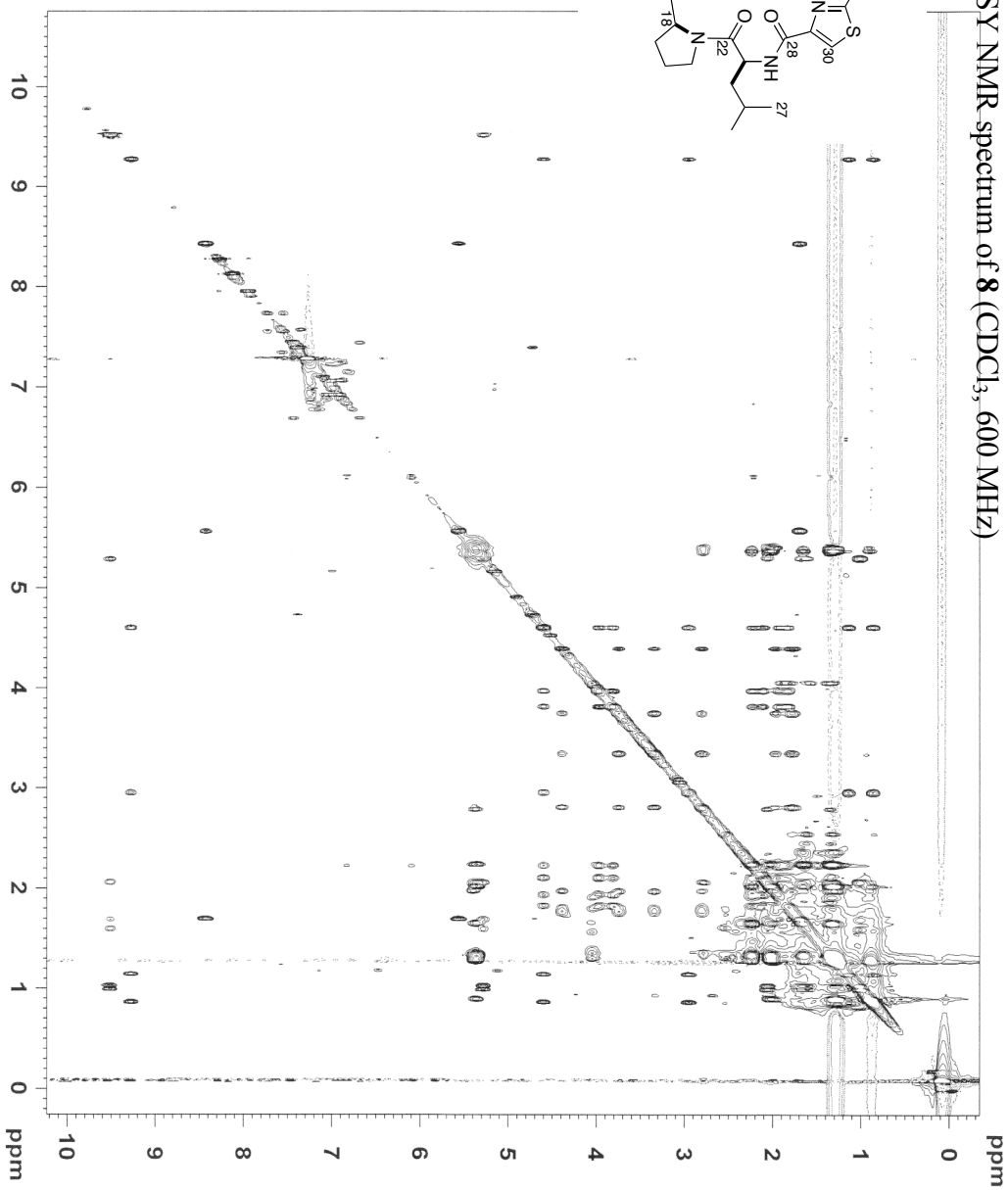
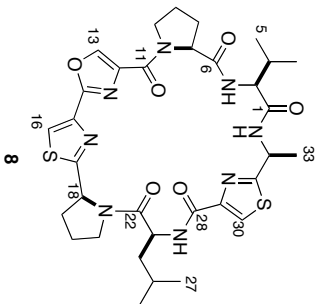
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GPNAM2     SINE.100
GPZ1       10.00 %
GPZ2       10.00 %
F16        1000.00 usec

F1 - Acquisition Parameters
IND0       1
NUC1       13C
P1         25.00 usec
P2         599.823 MHz
PC         15.00 usec
SFO1       599.8201000 MHz
SFO2       28.112631 Hz
SW         11.998 ppm
FMODE      States-TPPI

F2 - Processing parameters
SI         1024
SF         599.8201000 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
SF         599.8201000 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0
PC         0
  
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TOCSY NMR spectrum of 8 (CDCl₃, 600 MHz)



Current Data Parameters
 NAME 5frc3jucsd
 EXPNO 23
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080629
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 INSTRUM 1.7 mm QNP5 spect
 FIDPROC mlevph
 TD 2048
 SOLVENT CDCl3
 NS 24
 DS 2
 SWH 7211.539 Hz
 FIDRES 3.521259 Hz
 AQ 0.1420447 sec
 DE 69.333 usec
 TE 295.10 K
 D1 2.0000000 sec
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 D9 0.3000001 sec
 FACTOR1 0.0001320
 FWD 110
 SCALES 180
 STICNT 6
 128

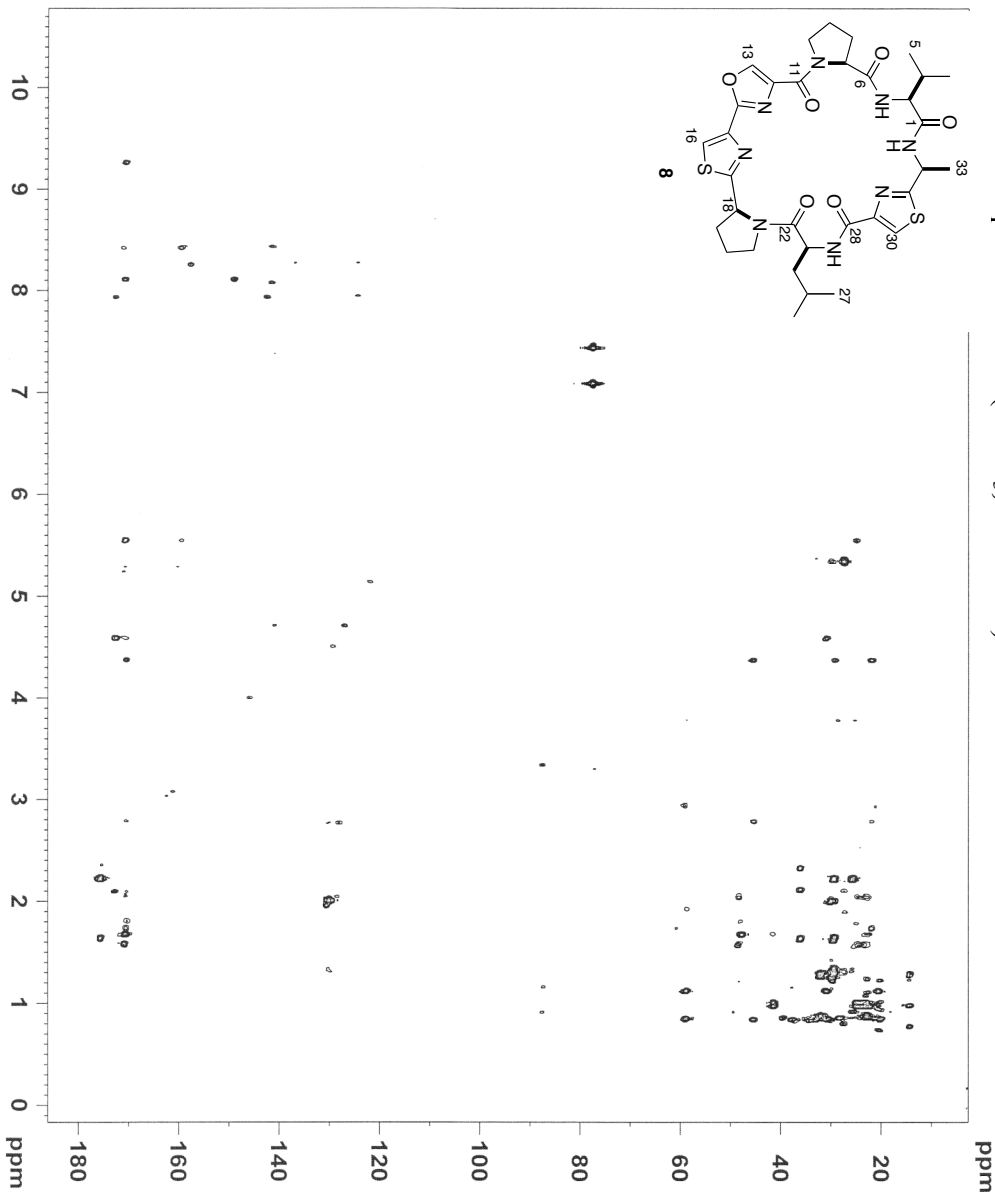
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 P17 2500.00 usec
 P2 11.00 usec
 P6 25.00 usec
 P7 50.00 usec
 FL1 15.00 dB
 FL2 20.00 dB
 SFO1 599.822991 MHz

F1 - Acquisition parameters
 TD 25
 SFO1 599.823 MHz
 FIDRES 28.112631 Hz
 SW 11.998 ppm
 FWHODE States-1PFI

F2 - Processing parameters
 SI 1024
 SF 599.822991 MHz
 SSB 2
 LB 0.00 Hz
 GB 0
 EC 1.40

F1 - Processing parameters
 SI 1024
 SF 599.820000 MHz
 SSB 2
 WDW GSIINE
 GB 0.00 Hz
 CB 2

HMBC NMR spectrum of **8** (CDCl₃, 600 MHz)



Current Data Parameters
 NAME SPC12UCSD
 EXPTNO 13
 PROCNO 1

F2 - Acquisition Parameters
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 INSTRUM spect
 PULPROG hmcpg1prdtf
 TD 4096
 NS 384
 DSF 64
 SFO2 150.8361700 MHz
 AQ 0.2849393 sec
 FIDRES 1.760630 Hz
 EQ 62.333 usec
 DE 6.50 usec
 TE 300.2 K
 C13ST13 145.0000000
 CNST2 1.0000000 sec
 D0 1.0000000 sec
 D16 0.0020000 sec
 G2 0.0034828 sec
 G3 0.0001490 sec
 INO 0.0001490 sec

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 PL1 0.00 dB
 P12 15.00 dB
 SFO1 599.822991 MHz

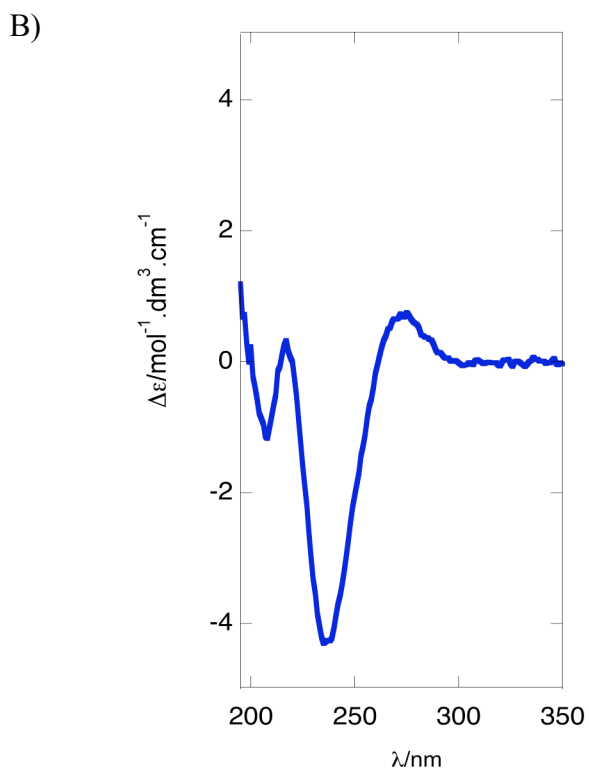
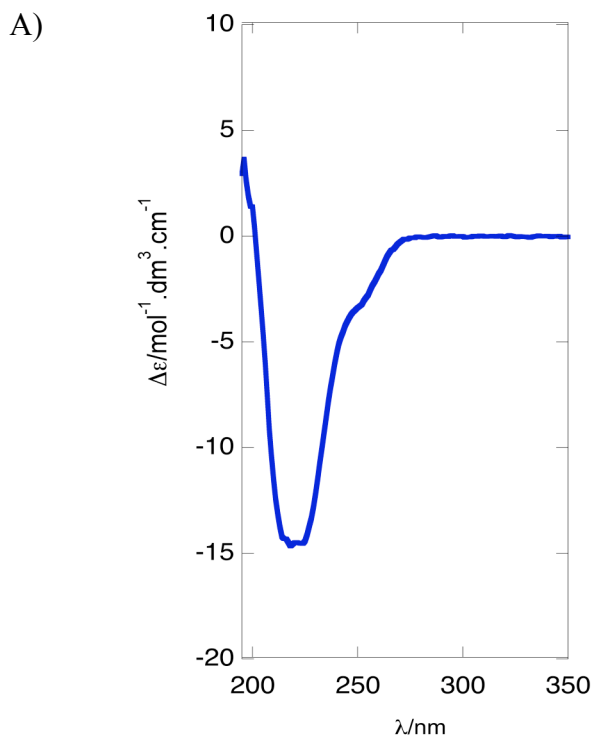
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 PL2 0.50 dB
 SFO2 150.8361700 MHz

***** GRADIENT CHANNEL *****
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 GNUM2 SINE:100
 GNUM3 SINE:100
 GPZ1 56.00 %
 GPZ2 48.10 %
 GPZ3 48.10 %
 P16 1000.00 usec

F1 - Acquisition parameters
 NFO 562
 SFO1 150.836 MHz
 FIDRES 131.082214 Hz
 SFO2 222.473 ppm
 SFOXIDE QF

F2 - Processing parameters
 SF 599.820074 MHz
 SINE SINE
 LA 0.00 Hz
 GB 1.40
 PC 0

F1 - Processing parameters
 OF 150.8248258 MHz
 SF 599.820074 MHz
 SINE SINE
 SSB 0.00 Hz
 GB 0



Circular Dichroism spectra of A) sanguinamide A (3.8×10^{-4} M, MeOH) and B) sanguinamide B (2.1×10^{-4} M, MeOH)