New Hemiketal Steroid from the Soft Coral *Cladiella* sp.

Guang-Wen Zhang, Xiang-Quan Ma, Hiroshi Kurihara, Xin-Sheng Yao,
Jing-Yu Su, and Long-Mei Zeng*

TABLE OF CONTENTS

	Page
¹ H NMR of Compound 1 -----	2
¹³ C NMR of Compound 1 -----	2
HSQC of Compound 1 -----	3
HMBC of Compound 1 -----	4
TOCSY of Compound 1 -----	5
¹ H- ¹ H COSY of Compound 1 -----	6
¹ H NMR of Compound 1a -----	7
¹ H NMR of Compound 1b -----	8

fukami750_2_6.1.1:TXI
ZE-25/CD3OD/298K
1H(zg0pr)

H NMR of compound 1

Current Data Parameters
NAME fukami750_2_6
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040811
Time 11.31
INSTRUM dmh750
PROBHD 5 mm TXI XYZ
PULPROG zg0pr
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8333.333 Hz
FIDRES 0.127157 Hz
AQ 3.9322102 sec
RG 1024
DW 60.000 usec
DE 4.50 usec
TE 298.0 K
D1 2.00000000 sec
d12 0.00002000 sec
d13 0.00003000 sec

===== CHANNEL f1 =====
NUC1 1H
P0 8.00 usec
PL1 -2.00 dB
PL3 80.00 dB
SFO1 750.1336577 MHz

F2 - Processing parameters
SI 65536
SF 750.1299730 MHz
WDW EM
SSB 0

C NMR of compound 1

fukami500_25.1000.1:BBO
ZE-25/CD3OD/298K
13C(zgpg30)

Current Data Parameters
NAME fukami500_25
EXPNO 1000
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040818
Time 7.01
INSTRUM dmh500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT MeOH
NS 16384
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 8192
DW 15.900 usec
DE 4.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
d13 0.00003000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.72 usec
PL1 5.00 dB
SFO1 125.7703148 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 18.80 dB
PL13 18.80 dB
SFO2 500.1323533 MHz

F2 - Processing parameters
SI 65536
SF 125.7575936 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

fukami750_2_6.105.1:TXI
ZE-25/CD3OD/298K
HSQC(invigtp2)

HSQC of compound 1

Current Data Parameters
NAME fukami750_2_6
EXPNO 105
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040812
Time 17.48
INSTRUM dmw750
PROBHD 5 mm TXI XZ
PULPROG invigtp2
TD 2048
SOLVENT MeOH
NS 64
DS 16
SWH 8333.333 Hz
FIDRES 4.069010 Hz
AQ 0.1229300 sec
RG 16384
DW 60.000 usec
DE 4.50 usec
TE 298.0 K
CNST2 145.0000000
d0 0.00000300 sec
d1 2.00000000 sec
d4 0.00172414 sec
d11 0.03000000 sec
d13 0.00000300 sec
d16 0.00020000 sec
d20 0.00120300 sec
d21 0.00051714 sec
IN0 0.00000829 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.50 usec
P2 25.00 usec
PL1 -2.00 dB
SFO1 750.1336577 MHz

===== CHANNEL f2 =====
CPDPRG2 p5m4sp180
NUC2 13C
P3 15.00 usec
P4 30.00 usec
PCPD2 1429.00 usec
PL2 -5.00 dB
PL12 11.50 dB
SFO2 188.6353536 MHz
SFNAM15 Wurst50K_1403

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 15.00 %
GPZ2 40.00 %
GPZ3 10.05 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 4
TD 256
SFO1 188.6354 MHz
FIDRES 117.835594 Hz
SW 159.917 ppm
FNAME undefined

F2 - Processing parameters
SI 1024
SF 750.1300143 MHz
WDW QSINE
SSB 2
LE 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 TPPI

fukami750_2_6.105.1:TXI
ZE-25/CD3OD/298K
HSQC(invigtp2)

Current Data Parameters
NAME fukami750_2_6
EXPNO 105
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040812
Time 17.48
INSTRUM dmw750
PROBHD 5 mm TXI XZ
PULPROG invigtp2
TD 2048
SOLVENT MeOH
NS 64
DS 16
SWH 8333.333 Hz
FIDRES 4.069010 Hz
AQ 0.1229300 sec
RG 16384
DW 60.000 usec
DE 4.50 usec
TE 298.0 K
CNST2 145.0000000
d0 0.00000300 sec
d1 2.00000000 sec
d4 0.00172414 sec
d11 0.03000000 sec
d13 0.00000300 sec
d16 0.00020000 sec
d20 0.00120300 sec
d21 0.00051714 sec
IN0 0.00000829 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.50 usec
P2 25.00 usec
PL1 -2.00 dB
SFO1 750.1336577 MHz

===== CHANNEL f2 =====
CPDPRG2 p5m4sp180
NUC2 13C
P3 15.00 usec
P4 30.00 usec
PCPD2 1429.00 usec
PL2 -5.00 dB
PL12 11.50 dB
SFO2 188.6353536 MHz
SFNAM15 Wurst50K_1403

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 15.00 %
GPZ2 40.00 %
GPZ3 10.05 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 4
TD 256
SFO1 188.6354 MHz
FIDRES 117.835594 Hz
SW 159.917 ppm
FNAME undefined

F2 - Processing parameters
SI 1024
SF 750.1300143 MHz
WDW QSINE
SSB 2
LE 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 TPPI

fukami750_2_6.106.1:TXI
ZE-25/CD3OD/298K
HMBC(inv4gplplrnd)

HMBC of compound 1

Current Data Parameters
NAME fukami750_2_6
EXPNO 106
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040813
Time 1.32
INSTRUM dmw750
PROBHD 5 mm TXI XYZ
PULPROG inv4gplplrnd
TD 2048
SOLVENT MeOH
NS 32
DS 16
SWH 8333.333 Hz
FIDRES 4.069010 Hz
AQ 0.1229300 sec
RG 16384
DN 60.000 usec
DE 4.50 usec
TE 298.0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 2.00000000 sec
d2 0.00344828 sec
D6 0.06500000 sec
d13 0.00000300 sec
D16 0.00020000 sec
IN0 0.00001205 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.50 usec
P2 25.00 usec
PL1 -2.00 dB
SFO1 750.1336577 MHz

===== CHANNEL f2 =====
NUC2 13C
P3 15.00 usec
PL2 -5.00 dB
SFO2 188.6410122 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100

GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 25.00 %
GPZ2 15.00 %
GPZ3 20.05 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 2
TD 512
SFO1 188.641 MHz
FIDRES 81.042534 Hz
SW 219.962 ppm
FMODE undefined

F2 - Processing parameters
SI 1024
SF 750.1300143 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
HC2 0F
SF 188.6200877 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

fukami750_2_6.106.1:TXI
ZE-25/CD3OD/298K
HMBC(inv4gplplrnd)

Current Data Parameters
NAME fukami750_2_6
EXPNO 106
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040813
Time 3.32
INSTRUM dmw750
PROBHD 5 mm TXI XYZ
PULPROG inv4gplplrnd
TD 2048
SOLVENT MeOH
NS 32
DS 16
SWH 8333.333 Hz
FIDRES 4.069010 Hz
AQ 0.1229300 sec
RG 16384
DN 60.000 usec
DE 4.50 usec
TE 298.0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 2.00000000 sec
d2 0.00344828 sec
D6 0.06500000 sec
d13 0.00000300 sec
D16 0.00020000 sec
IN0 0.00001205 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.50 usec
P2 25.00 usec
PL1 -2.00 dB
SFO1 750.1336577 MHz

===== CHANNEL f2 =====
NUC2 13C
P3 15.00 usec
PL2 -5.00 dB
SFO2 188.6410122 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100

GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 25.00 %
GPZ2 15.00 %
GPZ3 20.05 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 2
TD 512
SFO1 188.641 MHz
FIDRES 81.042534 Hz
SW 219.962 ppm
FMODE undefined

F2 - Processing parameters
SI 1024
SF 750.1300143 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
HC2 0F
SF 188.6200877 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

TOCSY of compound 1

H-H COSY of compound 1

fukami750_2_6.101.1:TXI
ZE-25/CD3OD/298K
COSY(cosygpmpfprtp)

H NMR of compound 1a

SAMPLE SPECIAL
 date Mar 14 2003 temp 28.0
 solvent CDC13 gain not used
 file exp spin 20
 ACQUISITION test 0.008
 sw 7.000 7.000
 at 4.800 6.600
 np 7.400 6.600
 fb 3.000 6.600
 bs 4.000 6.600
 dl 4.000 6.600
 nt 8 6.600
 ct 8 6.600
 TRANSMITTER fn 69536
 tn H1
 sfrq 499.777 sp
 tof -29.4 wp
 tpwr 54 rfl
 pw 3.800 rfp
 DECOUPLER H1
 dn H1
 dof -176.6
 dm nnn wc
 dnm C sc
 dpwr 38 vs
 dmf 20202 th
 al ph

7.081
7.061

6.247
6.244
6.227
6.223

6.077
6.074
6.071

3.871
3.966
3.949
3.945

3.362
3.359
3.341
3.338

2.495
 2.485
 2.468
 2.461
 2.455
 2.435
 2.378
 2.370
 2.346
 2.338
 2.168
 2.156
 2.137
 2.130
 2.116
 2.102
 2.085
 2.088
 2.072
 2.055
 2.042
 2.025
 2.010
 1.993
 1.980
 1.967
 1.954
 1.944

fukami750_2_4.1.1:TXI
E-25/Pyr-d5/298K
1H(zg0)

H NMR of compound 1b

Current Data Parameters
NAME fukami750_2_4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040528
Time 13.52
INSTRUM dmw750
PROBHD 5 mm TXI XYZ
PULPROG zg0
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8333.333 Hz
FIDRES 0.127157 Hz
AQ 3.9322100 sec
RG 512
DW 60.000 usec
DE 4.50 usec
TE 298.0 K
D1 2.00000000 sec
NUC1 1H
PO 8.00 usec
PL1 -2.00 dB
SFO1 750.1336944 MHz

F2 - Processing parameters
SI 65536
SF 750.1299730 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

fukami750_2_4.1.1:TXI
E-25/Pyr-d5/298K
1H(zg0)

Current Data Parameters
NAME fukami750_2_4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040528
Time 13.52
INSTRUM dmw750
PROBHD 5 mm TXI XYZ
PULPROG zg0
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8333.333 Hz
FIDRES 0.127157 Hz
AQ 3.9322100 sec
RG 512
DW 60.000 usec
DE 4.50 usec
TE 298.0 K
D1 2.00000000 sec
NUC1 1H
PO 8.00 usec
PL1 -2.00 dB
SFO1 750.1336944 MHz

F2 - Processing parameters
SI 65536
SF 750.1299730 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00