A Linchpin Approach to Unsaturated

Fatty Acids: 11,12-

Epoxyeicosatrienoic Acid and

11S,12S-Dihydroxyeicosatrienoic Acid

Ethyl Esters

(Supporting Information)

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General Experimental Procedures

^1H NMR and ^{13}C NMR spectra were obtained as solutions in deuteriochloroform (CDCl_3) at 400 MHz and 100 MHz, respectively, with tetramethylsilane = 0.00 as an internal standard. ^{13}C multiplicities were determined with the aid of a JVERT pulse sequence, differentiating the signals for methyl and methine carbons as "d" from methylene and quaternary carbons as "u". The infrared (IR) spectra were determined as neat oils.

Optical rotations were determined as solutions in dichloromethane unless otherwise noted. R_f values indicated refer to thin layer chromatography (TLC) on 2.5×10 cm, 250 μm analytical plates coated with silica gel GF, unless otherwise noted, and developed in the solvent system indicated. The solvent mixtures used are volume/volume mixtures.

All glassware was oven dried before use. Tetrahydrofuran (THF) was distilled from sodium metal/benzophenone ketyl under dry nitrogen. Dichloromethane (CH_2Cl_2) was distilled from calcium hydride under dry nitrogen. MTBE is methyl t-butyl ether. PE is petroleum ether. All reactions were performed under a nitrogen atmosphere unless otherwise specified.

***trans*-Enyne alcohol (15):** The same procedure was applied as the preparation of 11 except that the *trans*-1,4-dichloro-2-butene 5 was used. *trans*-Enyne alcohol 15 was prepared as a colorless oil (47% yield from 1-heptyne), TLC R_f (MTBE/petroleum ether = 1:4) = 0.45; ^1H NMR δ 0.86 (t, J = 7.1 Hz, 3H), 1.30-1.33 (m, 4H), 1.44-1.47 (m, 2H), 1.95 (t, J = 6.4 Hz, 1H), 2.13 (t, J = 7.2 Hz, 2H), 2.88 (br s, 2H), 2.94 (br s, 2H), 4.25 (d, J = 6.4 Hz, 2H), 5.48-5.52 (m, 2H); ^{13}C NMR δ u 18.7, 21.6, 21.7, 22.1, 28.6, 31.0, 51.2, 76.7, 80.2, 82.6, 83.3; d 13.9, 125.0, 126.9; IR (cm^{-1}); 3152, 2956, 2930; MS m/z (%): 203(M-H, 17), 91(100); HRMS Calcd for $\text{C}_{14}\text{H}_{21}\text{O}$ (M+H) 205.1592, Obsd 205.1594.

***trans*-Triyne (16):** The same procedure was applied as the preparation of 12. *trans*-Triyne 16 was prepared as a colorless oil (40% yield from 15), TLC R_f (MTBE/petroleum ether = 1:10) = 0.35; ^1H NMR δ 0.86 (t, J = 7.1 Hz, 3H), 1.30-1.33 (m, 4H), 1.44-1.49 (m, 2H), 1.79-1.86 (m, 2H), 2.13-2.16 (m, 2H), 2.31-2.33 (m, 2H), 2.47 (t, J = 7.2 Hz, 2H), 2.90 (br s, 4H), 3.13 (br s, 2H), 5.66 (br s, 2H); ^{13}C NMR δ u 9.7, 16.1, 17.8, 18.7, 21.6, 21.7, 22.2, 24.6, 28.7, 31.1, 76.0, 76.5, 76.8, 77.6, 77.8, 82.6, 119.2; d 14.0, 125.4, 126.8; IR (cm^{-1}); 3400, 2930, 1420; MS m/z (%): 279(M^+ , 70), 222(45); HRMS Calcd for $\text{C}_{20}\text{H}_{25}\text{NNa}$ (M+Na) 302.1885, Obsd 302.1871.

***trans*-Ester (17):** The same procedure was applied as the preparation of 13. The *trans*-ester 17 was prepared as a colorless oil (71% yield from 16): TLC R_f (MTBE/petroleum

ether = 1:30) = 0.45; ^1H NMR δ 0.87 (t, J = 6.9 Hz, 3H), 1.23 (t, J = 7.1 Hz, 3H), 1.30-1.36 (m, 4H), 1.43-1.49 (m, 2H), 1.75-1.82 (m, 2H), 2.12-2.17 (m, 2H), 2.19-2.23 (m, 2H), 2.39 (t, J = 7.5 Hz, 2H), 2.89 (br s, 4H), 3.12-3.13 (m, 2H), 4.10 (q, J = 7.5 Hz, 2H), 5.65 (br s, 2H); ^{13}C NMR δ u 9.7, 18.2, 18.7, 21.6, 21.7, 22.2, 23.9, 28.7, 31.1, 33.1, 60.3, 75.2, 76.3, 76.8, 77.6, 79.3, 82.6, 173.2; d 14.0, 14.2, 125.5, 126.7; IR (cm^{-1}); 2932, 1732, 1158; MS m/z (%): 297(M^+ -Et, 10), 155(100); HRMS Calcd for $\text{C}_{22}\text{H}_{30}\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) 349.2144, Obsd 349.2149.

Diol (18): A suspension of AD-mix- α (1.47 g) in *t*-BuOH- H_2O (1:1, v/v, 8 mL) was stirred at rt until both phases were clear. The mixture was cooled to 0°C and methanesulfonamide (70 mg, 0.74 mmol) was added, followed by the *trans*-ester 17 (212 mg, 0.65 mmol) in MTBE (0.5 mL) dropwise over 5 min. The resulting mixture was stirred at 0°C for 49 h. Solid NaHSO_3 (1.52 g) was carefully added to the reaction mixture. The mixture was stirred at rt for 1 h, then partitioned between EtOAc and brine. The combined organic extracts were dried (Na_2SO_4) and concentrated. The residue was chromatographed to give diol 18 as a colorless oil (122 mg, 0.34 mmol, 52% yield). TLC R_f (MTBE/petroleum ether = 1:1) = 0.20; ^1H NMR δ 0.87 (t, J = 7.2 Hz, 3H), 1.23 (t, J = 7.1 Hz, 3H), 1.39-1.33 (m, 4H), 1.42-1.47 (m, 2H), 1.74-1.82 (m, 2H), 2.10-2.15 (m, 2H), 2.18-2.23 (m, 2H), 2.39 (t, J = 7.5 Hz, 2H), 2.43-2.53 (m, 4H), 3.09-3.11 (m, 2H), 3.70-3.77 (m, 2H), 4.10 (q, J = 7.5 Hz, 2H); ^{13}C NMR δ u 9.8, 18.1, 18.7, 22.2, 23.8, 24.2,

24.3, 28.6, 31.1, 33.1, 60.4, 74.9, 75.4, 76.3, 77.2, 79.5, 83.5, 173.3; d 14.0, 14.2, 71.1, 71.2; IR (cm⁻¹); 3224, 2932, 1736; MS *m/z* (%): 361(M⁺+H, 100), 343(30); HRMS Calcd for C₂₂H₃₂O₄Na (M+Na) 383.2198, Obsd 383.2193; [α]_D = +3.1 (c 1.31, CH₂Cl₂).

Triene (9): To a solution of Ni(OAc)₂•4H₂O (34 mg, 0.14 mmol) in ethanol (0.5 mL) was added a solution of NaBH₄ (1M solution in ethanol, 0.12 mL, 0.12 mmol). The black mixture was evacuated and backfilled with H₂ three times. Ethylenediamine (9 mg, 0.15 mmol, 0.01 mL) was added, followed by a solution of diol **18** (100 mg, 0.28 mmol) in ethanol (0.5 mL). The flask was evacuated and backfilled with H₂ three times. The reaction mixture was stirred at rt for 10 h under H₂. The black suspension was filtered through a short column packed with flash silica gel. The column was eluted with MTBE (50 mL). The solvent was removed to give the triene **9** as a colorless oil (82 mg, 0.22 mmol, 80% yield). TLC *R_f*(MTBE/petroleum ether = 3:1) = 0.15; ¹H NMR δ 0.86 (t, J = 7.0 Hz, 3H), 1.23 (t, J = 7.1 Hz, 3H), 1.25-1.37 (m, 6H), 1.62-1.70 (m, 2H), 2.00-2.11 (m, 4H), 2.26-2.32 (m, 6H), 2.78-2.81 (m, 2H), 3.49-3.52 (m, 2H), 4.10 (q, J = 7.1 Hz, 2H), 5.34-5.53 (m, 6H); ¹³C NMR δ 22.5, 24.8, 25.8, 26.6, 27.4, 29.3, 31.5, 31.8, 31.9, 33.7, 60.3, 173.8; d 14.0, 14.2, 73.1, 73.2, 124.6, 125.3, 128.5, 129.1, 131.1, 133.6; IR (cm⁻¹); 3224, 2926, 1736; MS *m/z* (%): 367(M⁺+H, 100), 242(20); HRMS Calcd for C₂₂H₃₈O₄Na (M+Na) 389.2668, Obsd 389.2666; [α]_D = -16.6 (c 0.51, CH₂Cl₂).

Methyl ester: To a solution of triene **9** (50 mg, 0.14 mmol) in MeOH (0.5 mL) was added K_2CO_3 (19 mg, 0.14 mmol) and the mixture was stirred at rt for 1.5 h, then partitioned between CH_2Cl_2 and brine. The combined organic extracts were dried (Na_2SO_4) and concentrated. The residue was chromatographed to give the methyl ester as a colorless oil (40 mg, 0.11 mmol, 83% yield). TLC R_f (MTBE/petroleum ether = 3:1) = 0.15; 1H NMR δ 0.86 (t, J = 7.0 Hz, 3H), 1.26-1.35 (m, 6H), 1.62-1.70 (m, 2H), 2.02-2.07 (m, 4H), 2.16-2.32 (m, 6H), 2.78-2.80 (m, 2H), 3.48-3.51 (m, 2H), 3.64 (s, 3H), 5.35-5.53 (m, 6H); ^{13}C NMR δ u 22.5, 24.7, 25.7, 26.6, 27.2, 29.3, 31.5, 31.8, 31.9, 33.4, 174.0; d 14.0, 51.5, 73.1, 73.2, 124.6, 125.3, 128.6, 129.0, 131.1, 133.6; IR (cm^{-1}); 3216, 2926, 1740; MS m/z (%): 353($M^+ + H$, 30), 225(50); HRMS Calcd for $C_{21}H_{36}O_4Na$ ($M + Na$) 375.2511, Obsd 375.2515; $[\alpha]_D = -6.1$ (c 1.05, $CHCl_3$), lit: $[\alpha]_D = -6.3$ (c 0.97, $CHCl_3$).

S-7

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[illegible]

S-14

Integral

ppm

Current Data Parameters

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20050412

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ppm

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CHANNEL f2 waitz16

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SI 32768
SF 100.6127749 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

10 MHz plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -5.000 ppm
F2 -503.07 Hz
PPMCH 10.25000 ppm/cm
HZCM 1031.28101 Hz/cm

9

Current Data Parameters
NAME 22365
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050420
Time 12.54
INSTRUM spect
PROBHD 5 mm DPX 3H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
DS 16
AQ 0
RG 327.641 Hz
FIDRES 0.176660 Hz
AQRES 2.025092 sec
RG 57
NUC1 61,400 MHz
NUC2 13,76.8 MHz
TE 300.2 K
D1 5.00000000 sec

CHANNEL f1
NUC1 31
P1 18.00 sec
PL1 0.00 dB
SFO1 400.1326739 MHz

F2 - Processing parameters
SI 32768
SF 400.130625 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

90 MHz pulse parameters
CT 20.00 cm
CF 20.00 MHz
F1 5.000 MHz
F2 350.117 Hz
F3 1.000 MHz
F4 400.13 Hz
PC 4.00000000 Hz/cm
HCH1 200.00000 Hz/cm

9

Current Data Parameters
NAME ZZ368
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050421
Time 0.04
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG 1vert
TD 65536
SOLVENT CDCl3
NS 4040
DS 0
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 9193.2
DM 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
D11 0.03000000 sec
D13 0.00000300 sec
D20 0.00700000 sec

CHANNEL f1

NUC1 13C
P1 13.00 usec
P2 25.00 usec
PL1 0.00 dB
SF01 100.6242995 MHz

CHANNEL f2

CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 17.00 dB
SF02 400.1320007 MHz

F2 - Processing Parameters

SI 32768
SF 100.6127741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -5.000 ppm
F2 -503.07 Hz
PPMCM 10.25000 ppm/cm
HZCM 1031.28101 Hz/cm

ppm

Date: 20050506
 Time: 17.45
 INSTR: spect
 PROG: 5 m exp m1
 PULPROG: zg
 TO: 32768
 SOLVENT: CDCl3
 NS: 16
 DS: 6
 SWH: 5592.841 Hz
 FIDRES: 0.17650 Hz
 AQ: 2.925952 sec
 RG: 57
 AG: 88.400 us/c
 DE: 6.00 us/c
 TE: 300.0 K
 D1: 5.00000000 sec
 CHANNEL F1
 NUC1: 1H
 P1: 18.00 us/c
 PL1: 0.00 dB
 SFO1: 400.1362718 MHz
 F2 - Processing parameters
 SI: 32768
 SF: 400.1362718 MHz
 SW: 5592.841 Hz
 LB: 9.30 Hz
 GB: 0
 PC: 1.00
 IN phase parameters
 CX: 20.00 cm
 CR: 9.000 ppm
 F1: 3601.17 Hz
 F2: -600.13 Hz
 FREQ: 9.50000 ppm/cm
 HZCM: 200.16502 Hz/cm

Current Data Parameters
NAME Z2388
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

174.202

133.599
133.295
131.106
129.042
128.563
125.315
124.821
124.608

77.317
77.000
76.681
73.207
73.108
73.094
51.541
51.474
34.016
33.381
31.873
31.789
31.485
29.269
28.923
28.806
27.356
27.238
26.557
25.749
24.811
24.702
22.535
14.037

Date_ 20050506
Time 22:04
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2141
DS 0
SMH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 11585.2
DW 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 6.00000000 sec
D11 0.03000000 sec
D13 0.00000300 sec
D20 0.00700000 sec

CHANNEL f1

MU1 13C
P1 13.00 usec
P2 26.00 usec
PL1 0.00 dB
SF01 100.6242995 MHz

CHANNEL f2

CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 17.00 dB
SF02 400.1320007 MHz

F2 - Processing parameters

SI 32768
SF 100.6127749 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
FIP 200.000 ppm
F1 20122.55 Hz
F2 -503.07 Hz
F2 10.25000 ppm/cm
P1MCH 1031.26101 Hz/cm