Supporting Information

for

Synthesis of Enantiopure Termini-Differentiated Heptane Stereotriads Application to Sidechain-Functionalized Tetrahydrofurans of IKD-8344

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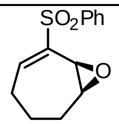
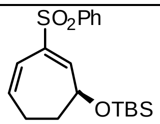
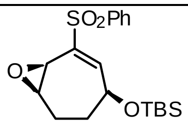
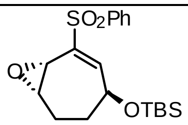
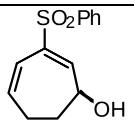
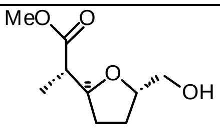
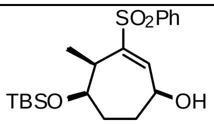
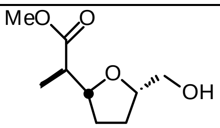
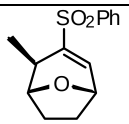
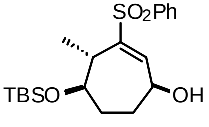
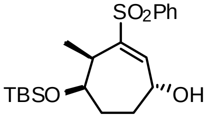
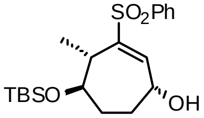
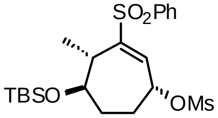
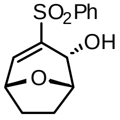
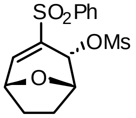
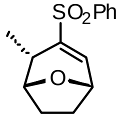
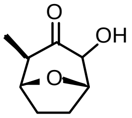
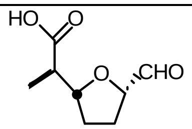
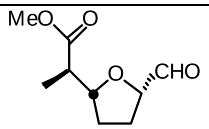
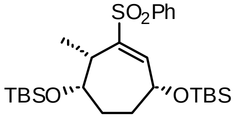
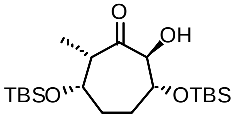
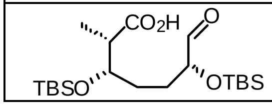
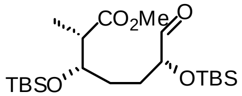
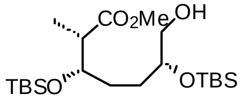
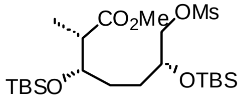
West Lafayette, Indiana 47907

Characterization checklist

Cmpd Structure	Cmpd #	^{1}H NMR	^{13}C NMR	LRMS	HRMS Calcd.	HRMS Found	other
	**5b**	Y	Y	Y	251.0472	251.0471	95% ee
	**6b**	Y	Y	Y	365.1607	365.1607	mp 76°C [α]°(CHCl$_3$) = -20.2
	**syn-7b**	Y	Y	Y	381.1556	381.1554	mp 89°C [α]°(CHCl$_3$) = -15.5 >98% ee & de
	**anti-7b**	Y	Y	Y	381.1556	381.1556	mp 74°C [α]°(CHCl$_3$) = -2.70 >98% ee & de
	**8b**	Y	Y	Y	251.0742	251.0739	mp 61°C [α]°(CHCl$_3$) = -19.6
	**10**	Y	Y	Y	189.1127	189.1126	
	**11**	Y	Y	Y	397.1869	397.1870	mp 84°C [α]°(CHCl$_3$) = +2.40
	**12**	Y	Y	Y	189.1127	189.1127	
	**13**	Y	Y	Y	264.0820	264.0818	mp 47°C [α]°(CHCl$_3$) = +1.94 >99% ee

	**14**	Y	Y	Y	397.1869	397.1868	mp 132°C [α]°(CHCl$_3$) = -2.72 X-ray
	**15**	Y	Y	Y	397.1869	397.1864	
	**16**	Y	Y	Y	397.1869	397.1868	mp 147°C [α]°(CHCl$_3$) = -3.30
Cmpd Structure	Cmpd #	^{1}H NMR	^{13}C NMR	LRMS	HRMS Calcd.	HRMS Found	other
	**17**	Y	Y	Y	475.1644	475.1646	
	**18**	Y	Y	Y	267.0691	267.0690	mp 114°C [α]°(CHCl$_3$) = -2.00 >99% ee
	**19**	Y	Y	Y	344.0388	344.0386	mp 160°C x-ray
	**20**	Y	Y	Y	265.0898	265.0904	[α]°(CHCl$_3$) = +2.41 >99% ee
	**21**	Y	Y	Y	156.0786	156.0785	
	**22**	Y	Y	Y	173.0814	173.0816	
	**23**	Y	Y	Y	187.0970	187.0968	
	**24**	Y	Y	Y	511.2734	511.2740	mp 88°C
	**25**	Y	Y	Y	403.2700	403.2695	
	**26**	Y	Y	Y	419.2649	419.2643	

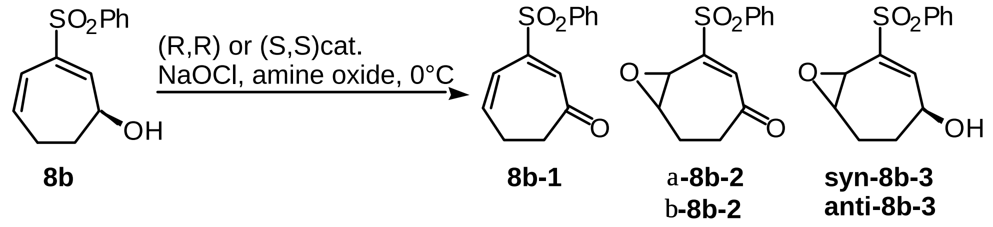
	**27**	Y	Y	Y	433.2806	433.2800	
	**28**	Y	Y	Y	435.2962	435.2961	
	**29**	Y	Y	Y	513.2738	513.2730	

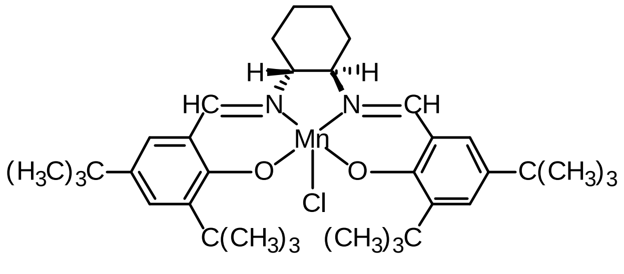
Supplemental Experimental Section

General procedure for asymmetric epoxidation by using Jacobsen’s catalyst

To a solution of substrate (5 mmol) in 25 mL of dichloromethane and 4-(3-phenylpropyl)pyridine N-oxide (P_3NO, 1.2 mmol) at 0°C, Jacobsen’s catalyst (0.3 mmol) was added and the homogeneous solution was mechanically stirred for 5 minutes. Then, 10 mL of commercial bleach (NaOCl 10~15 wt.% in water) was added to this solution. The reaction mixture was vigorously stirred at 0°C and the disappearance of starting material was monitored by TLC check. After 6 to 8 hours of stirring at 0°C, the reaction was completed and the reaction mixture was extracted with dichloromethane (3 × 50 mL). The organic layer was dried over magnesium sulfate, filtered through a short silica gel plug and concentrated. Flash chromatography (hexanes/ethyl acetate, silica gel) provided pure vinyl sulfonyl epoxide with 75~85% yield.

(1R,4S,7R)-2-benzenesulfonyl-8-oxa-bicyclo[5,1,0]oct-2-en-4-ol(syn-8b-3), (1R,7R)-2-benzenesulfonyl-8-oxa-bicyclo[5,1,0]oct-2-en-4-one(b-8b-2), 3-benzenesulfonyl-cyclohepta-2,4-dienone(8b-1)





(R,R) Jacobsen's catalyst

Phenylsulfonyl diene **8b** was epoxidized by using general asymmetric epoxidation procedure. (S,S)-Jacobsen’s catalyst was used. After 5 hours, the substrate was consumed. Flash chromatography (hexanes/ethyl acetate = 3:2, silica gel) afforded 30% of **syn-8b-3** as a white solid (mp = 71 °C), 35% of b**-8b-2** as a colorless oil and 12% of **8b-1** as a colorless oil.

8b-1: Rf = 0.5 in Hexane/EA = 1:1; colorless oil; ^{1}H NMR (300MHz, $CDCl_3$): δ 2.37~2.43ppm (m, 2H), δ 2.65~2.69ppm (m, 2H), δ 6.34~6.39ppm (dd, $^3J_{H,H}$ = 0.98Hz, $^3J_{H,H}$ = 11.47Hz, 1H), δ 6.61~6.69ppm (m, 1H), δ 7.10ppm (s, 1H), δ 7.59~7.70ppm (m, 3H), δ 7.88~7.98ppm (m, 2H); ^{13}C NMR (75MHz, $CDCl_3$, APT): δ 22.49ppm(e), δ 41.36ppm (e), δ 121.98ppm (o), δ 128.96ppm (o), δ 130.04ppm (o), δ 132.31ppm (o), δ 134.69ppm (o), δ 138.81ppm (e), δ 143.93ppm (o), δ 149.80ppm (e), δ 199.45ppm (e); Mass spectrum (CI): 249 (M + H$^+$, 1.000); HRMS calcd for $C_{13}H_{12}O_4S$ 249.0585, found 249.0583.

b**-8b-2:** Rf = 0.4 in Hexane/EA = 1:1; colorless oil; ^{1}H NMR (300MHz, $CDCl_3$): δ 2.21~2.33ppm (m, 2H), δ 2.46~2.55ppm (m, 1H), δ 2.67~2.79ppm (m, 1H), δ 3.76~3.80ppm (dd, $^3J_{H,H}$ = 1.46Hz, $^3J_{H,H}$ = 4.15Hz, 1H), δ 7.09ppm (dd, $^3J_{H,H}$ = 1.46Hz, $^3J_{H,H}$ = 1.46Hz, 1H), δ 7.56~7.76ppm (m, 3H), δ 7.90~7.98ppm (m, 2H); ^{13}C NMR (75MHz, $CDCl_3$, APT): δ 21.38ppm (e), δ 37.72ppm (e), δ 50.75ppm (o), δ 57.77ppm (o), δ 129.11ppm (o), δ 130.25ppm (o), δ 135.08ppm (o), δ 135.75ppm (o), δ 138.39ppm (e), δ 148.54ppm (e), δ 200.15ppm (e); Mass spectrum (CI): 265 (M + H$^+$, 1.000); HRMS calcd for $C_{13}H_{12}O_4S$ 265.0535, found 265.0532.

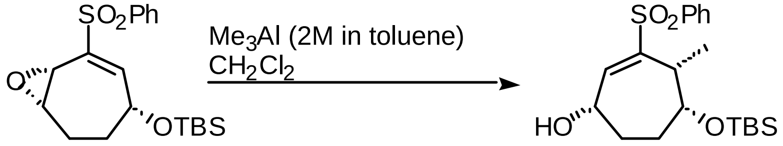
syn-8b-3: Rf = 0.3 in Hexane/EA = 1:1; colorless oil; [α]°($CHCl_3$) = -12.0; ^{1}H NMR (300MHz, $CDCl_3$): δ 1.76~1.86ppm (m, 2H), δ 1.92~2.05ppm (m, 1H), δ 2.12~2.17ppm (d, $^3J_{H,H}$ = 3.62Hz, 1H, OH), δ 2.22~2.32ppm (m, 1H), δ 3.40~3.42ppm (m, 1H), δ 3.70ppm (d, $^3J_{H,H}$ = 3.91Hz, 1H), δ 4.31~4.37ppm (m, 1H), δ 7.41ppm (d, $^3J_{H,H}$ = 1.00Hz, 1H), δ 7.52~7.68ppm (m, 3H), δ 7.9~8.00ppm (m, 2H); ^{13}C NMR (75MHz, $CDCl_3$, APT): δ 24.91ppm (e), δ 31.79ppm (e), δ 51.61ppm (o), δ 58.78ppm (o), δ 71.42ppm (o), δ 128.71ppm (o), δ 129.86ppm (o), δ 134.24ppm (o), δ 137.26ppm (e), δ 139.71ppm (e), δ 150.51ppm (o); Mass spectrum (CI): 249 (M + H$^+$ - H_2O, 1.000), 267 (M + H$^+$, 0.216); HRMS calcd for $C_{13}H_{14}O_4S$ 267.0691, found 267.0691.

(1R,4S,7R)-2-benzenesulfonyl-8-oxa-bicyclo[5,1,0]oct-2-en-4-ol (anti-9b-3), (1S,7S)-2-benzenesulfonyl-8-oxa-bicyclo[5,1,0]oct-2-en-4-one (a-8b-2), 3-benzenesulfonyl-cyclohepta-2,4-dienone (8b-1)

Phenylsulfonyl diene **8b** was epoxidized by using general asymmetric epoxidation procedure. (R,R)-Jacobsen's catalyst was used to make **anti-8b-3**. After 5 hours, substrate was consumed. Flash chromatography (hexanes/ethyl acetate = 3:2, silica gel) afforded 26% of **anti-8b-3** as a colorless oil, 33% of a**-8b-2** as a colorless oil and 14% of **8b-1** as a colorless oil.

anti-8b-3: Rf = 0.3 in Hexane/EA = 1:1; colorless oil; $[\alpha]^{\circ}(CHCl_3) = +2.5$; ^{1}H NMR (300MHz, $CDCl_3$): δ 1.71~1.82ppm (m, 2H), δ 1.88~1.97ppm (m, 1H), δ 2.20~2.25ppm (d, $^3J_{H,H}$ = 5.32Hz, 1H, OH), δ 2.28~2.40ppm (m, 1H), δ 3.37~3.42ppm (m, 1H), δ 3.61~3.65ppm (dd, $^3J_{H,H}$ = 1.05Hz, $^3J_{H,H}$ = 4.43Hz, 1H), δ 4.50~4.65ppm (m, 1H), δ 7.41ppm (dd, $^3J_{H,H}$ = 1.05Hz, $^3J_{H,H}$ = 5.13Hz, 1H), δ 7.54~7.66ppm (m, 3H), δ 7.90~7.94ppm (m, 2H); ^{13}C NMR (75MHz, $CDCl_3$, APT): δ 22.59ppm (e), δ 28.69ppm (e), δ 50.00 ppm (o), δ 57.98 ppm (o), δ 68.07ppm (o), δ 128.27ppm (o), δ 129.48ppm (o), δ 133.92ppm (o), δ 138.71ppm (e), δ 139.09ppm (e), δ 144.68ppm (o); Mass spectrum (CI): 249 (M + H+ - H_2O, 1.000), 267 (M + H+, 0.294); HRMS calcd for $C_{13}H_{14}O_4S$ 267.0691, found 267.0693.

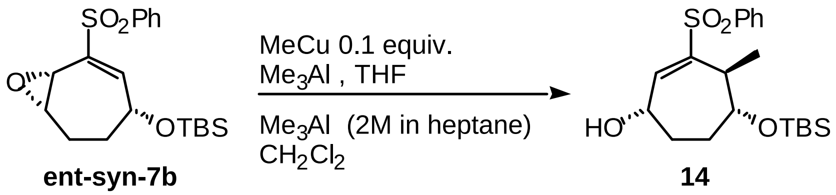
(1S,4R,5R)-3-benzenesulfonyl-5-(tert-butyldimethylsilanyl)oxy-4-methyl-cyclohept-2-enol (11)



ent-syn-7b **11**

To a solution of ent-syn-7b (1.90 g, 5 mmol) in 50 mL of freshly distilled dichloromethane at -78°C under argon, trimethylaluminum (2.0M solution in toluene, 6.25 mL, 12.5 mmol) was added slowly. The temperature was raised to 25°C over 2h. After 6 hours of stirring at 25°C, the reaction was cooled to -78°C and quenched with 25 mL of 5% aqueous HCl. The mixture was extracted with ethyl acetate (3 × 30 mL). The extract was dried over magnesium sulfate. Filtration, evaporation and flash chromatography (hexanes/ethyl acetate = 5:2, silica gel) gave 1.82 g (92%) of 11 as a white solid (mp = 84°C; Rf = 0.3 in Hexane/EA = 2:1; $[\alpha]^{\circ}(CHCl_3) = +2.4$). ^{1}H NMR (300MHz, $CDCl_3$): δ -0.319ppm (s, 3H), δ -0.203ppm (s, 3H), δ 0.752ppm (s, 9H), δ 1.21ppm (d, $^3J_{H,H}$ = 7.08Hz, 3H), δ 1.58~2.10ppm (m, 4H), δ 2.51~2.59ppm (m, 1H), δ 3.42~3.49ppm (m, 1H), δ 4.48~4.54ppm (m, 1H), δ 7.28ppm (d, $^1J_{H,H}$ = 4.64Hz, 1H), δ 7.51~7.66ppm (m, 3H), δ 7.84~7.88ppm (m, 2H); ^{13}C NMR (75MHz, $CDCl_3$): δ -5.32ppm, δ -5.13ppm, δ 13.63ppm, δ 17.89ppm, δ 25.67ppm, δ 28.21ppm, δ 31.41ppm, δ 40.47ppm, δ 67.00ppm, δ 71.66ppm, δ 128.27ppm, δ 129.34ppm, δ 133.48ppm, δ 139.04ppm, δ 143.94ppm, δ 146.07ppm; Mass spectrum (CI): 265 (M + H^+ - HOTBS, 0.710), 397 (M + H^+, 1.000); HRMS calcd for $C_{20}H_{32}O_4SSi$ 397.1869, found 397.1870.

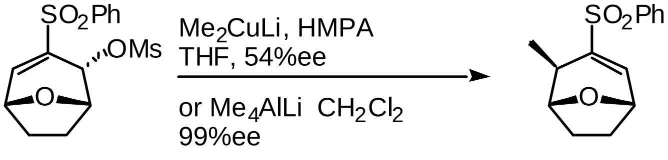
(1S,4S,5R)-3-benzenesulfonyl-5-(tert-butyldimethylsilanyl)oxy-4-methyl-cyclohept-2-enol (14)



Method A: CuI (98%, 97 mg, 0.5 mmol) and methyllithium (1.4M solution in diethyl ether, 0.36 mL, 0.5 mmol) in 20 mL of THF were stirred for 10 minutes at -30°C under argon. To this solution, trimethylaluminum (2.0M solution in toluene, 3.0 mL, 6 mmol) was added at -78°C followed by the addition of **ent-syn-7b** (1.90 g, 5 mmol) in 5 mL of THF. The temperature was raised to 0°C over 2h and the reaction was stirred for another 4 hours. The reaction was cooled to -78°C and quenched with 15 mL of 5% aqueous HCl. The reaction was extracted with ethyl acetate (3 × 30 mL). The extract was dried over magnesium sulfate. Filtration, evaporation and flash chromatography (hexanes/ethyl acetate = 5:2, silica gel) gave 1.92 g (97%) of **14** as a crystalline solid (mp = 132°C; Rf = 0.3 in Hexane/EA = 2:1; [α]°(CHCl$_3$) = -2.7). The absolute and relative stereochemistry was confirmed by X-ray crystallography of **ent-14**. ^{1}H NMR (300MHz, CDCl$_3$): δ -0.204ppm (s, 3H), δ -0.076ppm (s, 3H), δ 0.746ppm (s, 9H), δ 0.973ppm (d, $^3J_{H,H}$ = 7.33Hz, 3H), δ 1.65~1.97ppm (m, 3H), δ 2.04~2.20ppm (m, 1H), δ 2.68ppm (b, 1H, OH), δ 2.80~2.89ppm (m, 1H), δ 3.77~3.82ppm (m, 1H), δ 4.10~4.53ppm (m, 1H), δ 7.20ppm (d, $^3J_{H,H}$ = 3.42Hz, 1H), δ 7.48~7.62ppm (m, 3H), δ 7.84~7.88ppm (m, 2H); ^{13}C NMR (75MHz, CDCl$_3$, APT): δ -5.01ppm (o), δ -4.45ppm (o), δ 16.86ppm (o), δ 18.56ppm (e), δ 26.34ppm (o), δ 28.52ppm (e), δ 29.04ppm (e), δ 40.55ppm (o), δ 70.47ppm (o), δ 71.49ppm (o), δ 129.03ppm (o), δ 129.67ppm (o), δ 133.69ppm (o), δ 139.97ppm (e), δ 142.92ppm (e), δ 146.12ppm (o); Mass spectrum (CI): 265 (M + H$^+$ - HOTBS, 1.000), 397 (M + H$^+$, 0.749); HRMS calcd for $C_{20}H_{32}O_4SSi$ 397.1869, found 397.1868.

Method B: To a solution of **ent-syn-7b** (1.90 g, 5 mmol) in 50 mL of freshly distilled dichloromethane at -78°C under argon, trimethylaluminum (2.0M solution in heptane, 6.25 mL, 12.5 mmol) was added slowly. The temperature was raised to 25°C over 2h. After 6h of stirring at 25°C, the reaction was cooled to -78°C and quenched with 25 mL of 5% aqueous HCl. The mixture was extracted with ethyl acetate (3 × 30 mL). The extract was dried over magnesium sulfate. Filtration, evaporation and flash chromatography gave 1.82 g (92%) of **14**.

(1S,4R,5R)-3-benzenesulfonyl-4-methyl-8-oxa-bicyclo[3,2,1]oct-2-ene (13)

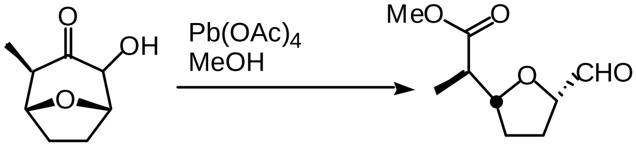


19 **13**

Method A: To a solution of **19** (0.35 g, 1 mmol) and HMPA (0.35 mL, 2 mmol) in 100 mL of THF at -78°C under argon, one equivalent of fresh made Me_2CuLi in 5 mL of THF was added and the reaction was stirred for 10 hours at -78°C under argon. The reaction was quenched with 10 mL of saturated NH_4Cl/concentrated NH_3 (9:1) at -78°C. The reaction was warmed to 25°C and extracted with ethyl acetate (3 × 20 mL). Evaporation and flash chromatography (hexanes/ethyl acetate = 2:1, silica gel) gave 0.22 g (85%) of **13** as a colorless oil (54%ee, Rf = 0.4 in Hexane/EA = 3:2). ^{1}H NMR (300MHz, $CDCl_3$): δ 1.14ppm (d, $^3J_{H,H}$ = 6.84Hz, 3H), δ 1.25~1.42ppm (m, 1H), δ 1.91~2.15ppm (m, 4H), δ 4.20~4.23ppm (m, 1H), δ 4.67~4.71ppm (m, 1H), δ 7.12ppm (d, $^3J_{H,H}$ = 3.95Hz, 1H), δ 7.50~7.65ppm (m, 3H), δ 7.83~7.90ppm (m, 2H); ^{13}C NMR (75MHz, $CDCl_3$, APT): δ 19.41ppm (o), δ 28.71ppm (e), δ 33.99ppm (e), δ 38.80ppm (o), δ 73.61ppm (o), δ 79.88ppm (o), δ 128.29ppm (o), δ 129.67ppm (o), δ 133.81ppm (o), δ 140.60ppm (e), δ 141.49ppm (e), δ 141.92ppm (o); Mass spectrum (CI): 265 (M + H^+, 1.000); HRMS (EI) calcd for $C_{14}H_{16}O_3S$ 264.0820, found 264.0818.

Method B: To a solution of **19** (0.28 g, 0.8 mmol) in 4 mL of freshly distilled dichloromethane at -78°C under argon, one equivalent of fresh made Me_4AlLi in 2 mL of dichloromethane was added and the reaction was stirred for 15 minutes. Then, reaction temperature was allowed to rise to 25°C during 4 hour span and the reaction was stirred for another 4 hours at 25°C. The reaction was quenched with 5% aqueous HCl at 0°C. Extraction, evaporation and flash chromatography gave 0.21 g (98%) of **13** as a white solid or colorless oil (99%ee, mp = 47°C, [α]°($CHCl_3$) = +1.94).

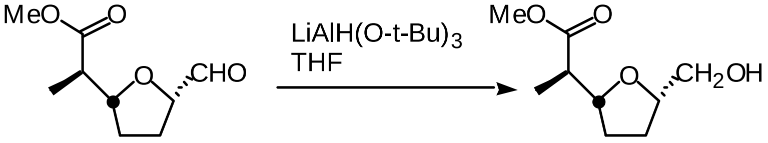
(2R,2'R,5'S)-2-(*cis*-5'-formyl-tetrahydro-furan-2'-yl)-propionic acid methyl ester (23)



21 **23**

To a solution of α-hydroxyketone **21** (0.26 g, 1 mmol) in 30 mL of methanol at 25°C, $Pb(OAc)_4$ (95%, 0.70 g, 1.5 mmol) was added. After stirring 1 hour at 25°C, the solution was concentrated, and the residue was partitioned between Et_2O/H_2O (1:1), extracted with ethyl acetate (3 × 20 mL), and dried over magnesium sulfate. Filtration, evaporation and flash chromatography (hexanes/ethyl acetate = 65:35, silica gel) gave 0.14 g (76%) of **23** as a colorless oil (Rf = 0.3 in Hexane/EA = 1:1). ^{1}H NMR (300MHz, $CDCl_3$): δ 1.14ppm (d, $^3J_{H,H}$ = 7.08Hz, 3H), δ 1.49~1.59ppm (m, 1H), δ 1.99~2.18ppm (m, 3H), δ 2.52~2.63ppm (m, 1H), δ 3.71ppm (s, 3H), δ 4.18~4.29ppm (m, 2H), δ 9.65ppm (d, $^3J_{H,H}$ = 1.43Hz, 1H); ^{13}C NMR (75MHz, $CDCl_3$, APT): δ 13.84ppm (o), δ 27.64ppm (e), δ 28.74ppm (e), δ 45.20ppm (o), δ 51.90ppm (o), δ 82.35ppm (o), δ 83.16ppm (o), δ 175.08ppm (e), δ 203.06ppm (o); Mass spectrum (CI): 157 (M + H^+ - HCHO, 0.332), 187 (M + H^+, 1.000); HRMS calcd for $C_9H_{14}O_4$ 187.0970, found 187.0968.

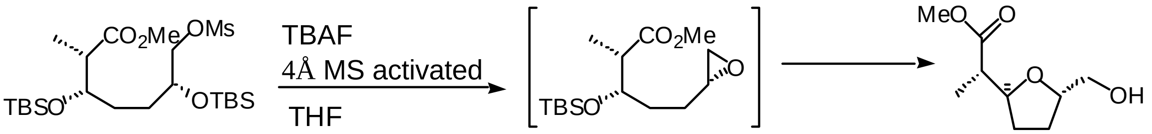
(2R,2’R,5’S)-2-(*cis*-5’-hydroxymethyl-tetrahydro-furan-2’-yl)-propionic acid methyl ester (12)



23 **12**

To a solution of **23** (186 mg, 1 mmol) in 10 mL THF at 0°C under argon, $LiAlH(O\text{-}t\text{-}Bu)_3$ (1.0M solution in THF, 1.05 mL, 1.05 mmol) was added, and the reaction was kept at 0°C for 2h of stirring. The reaction was quenched with 10 mL of 5% aqueous HCl and extracted with ethyl acetate (3 × 40 mL). The extract was washed with saturated sodium bicarbonate and brine, and dried over magnesium sulfate. Filtration, evaporation and flash chromatography (hexanes/ethyl acetate = 85:15, silica gel) gave 0.43 g (100%) of **12** as a colorless oil (Rf = 0.3 in hexane/EA = 1:1). ^{1}H NMR (300MHz, $CDCl_3$): δ 1.13ppm (d, $^3J_{H,H}$ = 6.90Hz, 3H), δ 1.65~1.76ppm (m, 1H), δ 1.82~1.91ppm (m, 2H), δ 1.97~2.10ppm (m, 1H), δ 1.97~2.10ppm (m, 1H), δ 2.27ppm (b, 1H, OH), δ 2.45~2.56ppm (m, 1H), δ 3.40~3.46ppm (m, 1H), δ 3.68~3.77ppm (m, 1H), δ 3.71ppm (s, 3H), δ 4.01~4.09ppm (m, 2H); ^{13}C NMR (75MHz, $CDCl_3$, APT): δ 14.29ppm (o), δ 26.38ppm (e), δ 29.94ppm (e), δ 46.04ppm (o), δ 52.35ppm (o), 64.52ppm (e), δ 80.54ppm (o), δ 81.69ppm (o), δ 175.96ppm (e); Mass spectrum (CI): 189 (M + H^+, 1.000); HRMS calcd for $C_9H_{16}O_4$ 189.1127, found 189.1126.

(2S,2’S,5’S)-2-(*trans*-5’-hydroxymethyl-tetrahydro-furan-2’-yl)-propionic acid methyl ester (10)



29 **30** **10**

To a solution of **29** (256 mg, 0.5 mmol) in 4 mL of THF, 4Å molecular sieve (powfered, activated, 200 mg) and TBAF (1.0M solution in THF, water content ~5%, 0.50 mL) were added respectively at 25°C. The reaction m was stirred for 4h. Evaporation and flash chromatography (hexanes/ethyl acetate = 3:2, silica gel) afforded 84 mg (89%) of **10** as a colorless oil (Rf = 0.3 in Hexane/EA = 1:1). This procedure would give a mixture of **10** and intermediate **30** if molecular sieve was not well activated. The mixture of **10**/**30** could be treated with $BF_3 \bullet OEt_2$ in dichloromethane at 0°C for 2 hours of stirring and gave **10** in quantitative yield. ^1H NMR (300MHz, $CDCl_3$): δ 1.12ppm (d, $^3J_{H,H}$ = 7.08Hz, 3H), δ 1.24ppm (b, 1H, OH), δ 1.56~1.79ppm (m, 2H), δ 1.88~2.13ppm (m, 2H), δ 2.50~2.60ppm (m, 1H), δ 3.44~3.50ppm (m, 1H), δ 3.61~3.67ppm (m, 1H), δ 3.71ppm(s, 3H), δ 4.07~4.18ppm (m, 2H); ^{13}C NMR (75MHz, $CDCl_3$, APT): δ 13.54ppm (o), δ 27.29ppm (e), δ 29.77ppm (e), δ 45.16ppm (o), δ 51.84ppm (o), δ 64.66ppm (e), δ 79.59ppm (o), δ 80.69ppm (o), δ 175.53ppm (e); Mass spectrum (CI): 189 ($M+H^+$, 1.000); HRMS calcd for $C_9H_{16}O_4$ 189.1127, found 189.1126.