Scheme I^a



^a Reagents: (i) Me₂C(OH)CH=CH₂, Zn(OAc)₂·2H₂O, PhH, Δ ; (ii) KF, MeOH, pH 7.0 buffer; (iii) *t*-BuMe₂SiCl, EtNⁱPr₂, DMF; (iv) O₃, CH₂Cl₂, -78 °C; Me₂S; (v) PhCH₂OCH₂NO₂ (3), *t*-BuOH, THF, *t*-BuOK (10 mol %); (vi) MsCl, Et₃N, CH₂Cl₂; DBU; (vii) *n*-Bu₄NF, THF, THF, THE CH 20 C -55 °C; CH₂Cl₂, O₃, -78 °C; (viii) DBU, CDCl₃, 55 °C.

vacuo. Flash column chromatography [SiO₂, 1:1 Et₂O/hexanes] gave the title compound (0.33 g, 100%): oil; $[\alpha]_D - 92.2^\circ$ (c 1.28, CHCl₃); R_f 0.73 (Et₂O); IR (neat) ν_{max} 2945, 2860, 1755, 1310, 1180, 1065 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 5.87 (dd, 1 H, J = 10.5, 17.7 Hz), 5.18 (dd, 2 H, J = 3, 14.4 Hz), 4.95 (dd, 1 H, J = 3.3, 1.5 Hz), 3.12 (dd, 1 H, J = 3, 15.3 Hz), 2.85 (dd, 1 H, J = 1.5, 15.3 Hz)Hz), 1.31 (s, 3 H), 1.29 (s, 3 H), 0.96 (s, 9 H), 0.26 (s, 3 H), 0.21 (s, 3 H); mass spectrum (CI), m/e 270.2 (M⁺ + H), 202.1, 170.1, 143.1. Anal. Calcd for C₁₄H₂₇NO₂Si: C, 62.40; H, 10.10; N, 5.20. Found: C, 62.02; H, 10.25; N, 5.03.

(4R)-N-(tert-Butyldimethylsilyl)-4-[(2-methyl-3-oxo-2propyl)oxy]-2-azetidinone (6). Ozone was bubbled through a solution of (4R)-N-(tert-butyldimethylsilyl)-4-[(2-methyl-3-buten-2-yl)oxy]-2-azetidinone (0.33 g) in dichloromethane (20 mL) at -78 °C until a blue/purple color persisted. The solution was purged with nitrogen, and dimethyl sulfide (1 mL) was added. The mixure was warmed to room temperature and stirred for 20 h, and then the solvent was evaporated in vacuo. Flash column chromatography [SiO₂, 1:1 hexanes/Et₂O] gave the compound 6 (0.23 g, 70%): oil; $[\alpha]_D$ -123.6° (c 1.12, CHCl₃); R_f 0.66 (Et₂O); IR (neat) ν_{max} 2940, 2870, 1760, 1440, 1315, 1080, 825 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 9.59 (s, 1H), 5.01 (dd, 1 H, J = 1.2, 3.2 Hz), 3.21 (dd, 1 H, J = 3.2, 15.2 Hz), 2.86 (dd, 1 H, J = 0.8,15.2 Hz), 1.33 (s, 3 H), 1.31 (s, 3 H), 0.97 (s, 9 H), 0.29 (s, 3 H), 0.25 (s, 3 H); HRMS (CI) calcd for C13H25NO3Si 272.1682, found (M⁺) 272.1717.

(4R)-N-(tert-Butyldimethylsilyl)-4-[[2-methyl-4-nitro-4-(benzyloxy)-3(Z)-buten-2-yl]oxy]-2-azetidinone (8). To a solution of (benzyloxy)nitromethane (3) (0.17 g, 1.2 equiv) in tert-butyl alcohol and THF (1:1, 10 mL) at 0 °C was added potassium tert butoxide (1.0 M in t-BuOH; 0.085 mL, 0.1 equiv). After 15 min, the β -lactam aldehyde (6) in THF (1 mL) was added, and the stirring was continued for a further 3 h. The solution was diluted with pH 7.0 phosphate buffer (50 mL) and extracted with Et_2O (3 × 25 mL). The combined extracts were dried and evaporated in vacuo. Flash column chromatography $[SiO_2, 1:1]$ hexanes/Et₂O] gave a diastereoisomeric mixture of nitro alcohols 7 (0.311 g, 84%).

The nitro alcohols 7 (0.362 g) were dissolved in dichloromethane (20 mL), and dimethylaminopyridine (0.05 equiv) was added. The solution was cooled to -78 °C, and then methanesulfonyl chloride (0.189 g, 2 equiv) and diisopropylethylamine (0.319 g, 2 equiv) were added simultaneously. The solution was warmed to room temperature and stirred for 19 h. 1,8-Diazabicyclo[5.4.0]undec-7-ene (0.5 g, 4 equiv) was added, and the solution was stirred for a further 1 h. The solution was washed with water (10 mL), dilute hydrochloric acid (2 M, 10 mL), and aqueous sodium bicarbonate (saturated, 10 mL), dried, and evaporated in vacuo. Flash column chromatography $[SiO_2, 1:1 \text{ hexanes}/Et_2O]$ gave the title compound (8) (0.282 g, 81%) as an oil: $[\alpha]_D -53.4^\circ$ (c 1.06, CHCl₃); R_f 0.6 (3:1 hexanes/Et₂O); IR (neat) ν_{max} 2940, 2870, 1760, 1685, 1545, 1075 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.41 (br s. 5 H), 6.54 (s, 1 H), 5.01 (s, 2 H), 4.77 (dd, 1 H, J = 3.3, 1.2 Hz), 3.08 (dd, 1 H, J = 3.3, 15.3 Hz), 2.78 (dd, 1 H, J = 0.9, 15.3 Hz), 1.35 (s, 3 H), 1.34 (s, 3 H), 0.95 (s, 9 H), 0.26 (s, 3 H), 0.21 (s, 3 H); ¹³C NMR (75 MHz, CDCl₃) δ 170.4, 154.4, 133.8, 129.4, 129.2, 128.8,

121.2, 76.2, 75.3, 74.4, 49.4, 27.7, 26.0, 25.8, 18.1; mass spectrum (CI), m/e 382.2, 292.1, 274.1, 184.1, 142.1, 115.0.

(2R,5R)-3,3-Dimethyl-2-[(benzyloxy)carbonyl]-4-oxa-1azabicyclo[3.2.0]heptan-7-one (9). To solution of the β -lactam nitroalkene 8 (0.28 g, 0.67 mmol) in THF (2 mL) at -55 °C was added tetrabutylammonium fluoride (1.0 M in THF, 0.67 mL, 1 equiv). After 10 min the solution was diluted with dichloromethane (20 mL) and cooled to -78 °C, and ozone was bubbled through. The solution was purged with nitrogen and then washed with water (10 mL) and dried, and evaporated in vacuo to give a 1:1 mixture of diastereoisomeric oxapenams. The mixture was dissolved in CDCl₃ (2 mL), and 1,8-diazabicyclo[5.4.0]undec-7-ene (0.02 mL, 2 equiv) was added. The solution was heated to 55 °C for 8 h. Flash column chromatography [SiO₂, 3:1 hexanes/Et₂O] gave the title compound (9) (0.093 g, 52%): oil; $[\alpha]_D + 134^\circ$ (c 1.06, CHCl₃); R_f 0.33 (1:1 hexanes/Et₂O); IR (neat) ν_{max} 2995, 1790, 1755, 1165, 700 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.37 (s, 5 H), 5.42 (d, 1 H, J = 2.7 Hz), 5.10 (s, 2 H), 4.28 (s, 1 H), 3.32 (dd, 1 H, J = 2.7, 16.2 Hz), 2.88 (d, 1 H, J = 16.2 Hz), 1.51 (s, 3 H),1.19 (s, 3 H); ¹³C NMR (75 MHz, CDCl₃) δ 176.7, 168.3, 134.8, 128.7, 90.3, 84.1, 68.6, 67.3, 45.0, 28.8, 22.5; mass spectrum (EI), 276 (M⁺ + H), 247, 234, 200, 98, 91; HRMS (EI) calcd for C₁₅-H₁₇NO₄ 275.1157, found (M^{•+}) 275.1163.

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Preparation of Optically Active 2-Thienylcarbinols by Kinetic Resolution Using the Sharpless Reagent

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After the discovery of the Sharpless asymmetric epoxidation² and kinetic resolution of allylic alcohols,^{2b,3} the kinetic resolution of various other types of substrates having a hydroxyl group at the chiral center and a prox-

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Table I. H	Kinetic	Resolution	of 1	Using	твнр,	L-(+)-DIPT	, and	Ti(O- <i>i</i> -	-Pr).	ť
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	substrate 1				(<i>R</i>)-	1 ^b
run		R ¹	\mathbb{R}^2	time, h	yield,° %	% ee ^d
1 ^e	a	н	n-Bu	18	39	>95
2	b	Н	<i>i</i> -Pr	18	30	91
3	с	н	t-Bu	18	25 ^f	47 ^g
4	d	н	\mathbf{Ph}	45	30	>95
5	е	Me	<i>n</i> -Am	20	35	>95

^a The reaction was carried out with TBHP (3 equiv), L-(+)-DIPT (1.2 equiv), and $Ti(O-i-Pr)_4$ (1.0 equiv) in CH_2Cl_2 at 0 °C. ^bAbsolute configurations were proven by chemical correlation (see the Experimental Section). ^cIsolated yield based on racemic 1 except for 1c. ^dEnantiomeric excesses were determined by conversion to the MTPA ester followed by ¹H NMR analysis and/or by conversion to the acetate (Ac₂O/pyridine) followed by ¹H NMR analysis in the presence of (+)-Eu(DPPM)₃. ^eThe resolution using 20% catalyst in the presence of molecular sieves^{2b} afforded (R)-1a with 43% ee at 50% conversion, indicating that the reaction must be carried out to 87% conversion to obtain (R)-1a with 95% ee. ^fDetermined by ¹H NMR analysis. ^gAbsolute configuration was not determined.

imate locus capable of accepting an oxygen atom have been investigated. To date β -hydroxy amines⁴ and 2-furyl-carbinols⁵ have been shown to be good substrates for the kinetic resolution.

Metal-catalyzed oxidation of substituted thiophenes to thiophene 1-oxides or 1,1-dioxides has been reported,⁶ and hence we were interested in the kinetic resolution of 2thienylcarbinols (1). At first, we were concerned about the efficiency of kinetic resolution of 1, since it has been reported that β -hydroxy sulfides are not good kinetic resolution substrates, and in the reaction with *trans*-2-(9anthrylthio)cyclohexanol, the enantiomeric excess (ee) of the recovered starting alcohol was only 20% at 60% conversion (eq 1).⁷



In practice, however, our concern was unfounded, and we found that the kinetic resolution of 1 proceeds highly effectively to provide a practical method for preparation of optically active 1^8 (eq 2), which is the subject of this paper.



A preliminary experiment revealed that the oxidation of 1a with 2 equiv of TBHP proceeded to nearly 50% conversion but not beyond. It was also found that the unidentified polymeric compound was the only product that resulted from the oxidation and was readily separable from the remaining 1a.⁹ The kinetic resolution of various 1, therefore, was carried out by using 3 equiv of TBHP, and the remaining chiral 1 was isolated by filtration through a short silica gel column after usual workup; the results are summarized in Table I. It can be seen from the table that the efficiency of the kinetic resolution depends on the steric bulk of the substituent (R^2) at carbinol carbon and decreases with increasing bulk of R^2 . When \mathbf{R}^2 is a primary alkyl or an aromatic group, highly efficient kinetic resolution occurs to afford the chiral alcohols 1 of more than 95% ee and in good yields, while the kinetic resolution of 1c in which R^2 is a tertiary alkyl group resulted in the recovery of the chiral alcohol with only 47% ee in 25% yield. When L-(+)-DIPT is employed, the slow-reacting enantiomer is always that shown in eq 2, which was confirmed by converting into the corresponding α -benzyloxy acids or α -hydroxy acids of known absolute configuration. Thus, the present kinetic resolution occurs in the same sense as observed in that of secondary allylic alcohols,³ β -hydroxy amines,⁴ α -acetylenic alcohols,⁷ and 2-furylcarbinols.⁵ Noteworthy also is the fact that the kinetic resolution by using a catalytic amount (20%) of $Ti(O-i-Pr)_4/L-(+)-DIPT$ proceeds with rather lower efficiency compared to the stoichiometric reaction (see footnote e in Table I).

As the starting racemic alcohols 1 can be readily prepared in large quantity from 2-thiophenecarbaldehyde and Grignard reagents or 2-thienyllithium and aldehydes, the optically active compounds 1 in which \mathbb{R}^2 is a primary alkyl, secondary alkyl, or an aromatic group are now readily available asymmetric starting materials. Application of the optically active 1 in organic synthesis and material science are in progress in our laboratory.

Experimental Section

General. ¹H NMR spectra were measured on either a HI-TACHI R-40 (90 MHz) or a JEOL FX-90Q (90 MHz) spectrometer. Optical rotations were measured on a YANAKO OR-50 porarimeter using a 20-cm³ capacity (0.5-dm path length) cell. Dichloromethane was distilled from calcium hydride. Titanium isopropoxide and L-(+)-diisopropyl tartrate were distilled under vacuum and stored under an argon atmosphere. Stock solution of TBHP in dichloromethane was prepared and stored as described by Sharpless.^{2b} Racemic 1-(2-thienyl)pentan-1-ol (1a),¹⁰ 1-(2-thienyl)-2-methylpropan-1-ol (1b),¹¹ 2-thienylbenzyl alcohol

⁽⁹⁾ A referee raised the possibility that the oxidation of eq 1 proceeds via epoxidation as is the case of 2-furylcarbinols⁵ and the resulting oxidation product polymerizes readily as shown below.



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(1d),¹² and 1-[2-(5-methylthienyl)]hexan-1-ol (1e)¹⁰ were prepared by the literature methods. 1-(2-Thienyl)-2,2-dimethylpropan-1-ol (1c) was prepared from 2-thiophenecarbaldehyde and t-BuLi.

General Procedure for Kinetic Resolution of 1. A typical experimental procedure is represented by preparation of (R)-1-(2-thienyl)pentan-1-ol (1a). To a solution of $Ti(O-i-Pr)_4$ (1.96) mL, 1.87 g, 6.57 mmol) in CH₂Cl₂ (5 mL) was added L-(+)-DIPT (1.66 mL, 1.85 g, 7.88 mmol) at -20 °C. After the mixture was stirred for 10 min, a solution of 1a (1.12 g, 6.57 mmol) in CH_2Cl_2 (2 mL) was added, and the resulting solution was stirred for 20 min at -20 °C. To this solution was added TBHP (6.08 mL, 19.7 mmol, 3.24 M in CH_2Cl_2). The solution was stirred at 0 °C for 18 h, and Me_2S (5.0 mL) was added at -20 °C. After being stirred for 30 min at -20 °C, the solution was poured into a mixture of 10% tartaric acid solution (0.5 mL), Et₂O (20 mL), NaF (1.5 g), and Celite (1.5 g). The mixture was stirred vigorously for 1 h at room temperature, yielding a precipitate, which was filtered through a pad of Celite. The filtrate was concentrated to give an oil, which was dissolved in Et_2O (50 mL) and treated with 1 N NaOH (25 mL) for 15 min at 0 \degree C with vigorous stirring.² The ethereal solution was washed with brine, dried (Na_2SO_4) , and concentrated to give an oil, which was chromatographed on a silica gel column ($2^{\phi} \times 8$ cm) with Et₂O-hexane as an eluent to afford 436 mg of (R)-1a (78% yield of theory, >95% ee determined by ¹H NMR analysis of the derived MTPA ester and also by ¹H NMR shift analysis of the corresponding acetate in the presence of (+)-Eu(DPPM)₃): $[\alpha]^{25}_{D}$ +20.7° (c 1.11, CHCl₃).

The absolute configuration was proven by transformation to (R)-2-(benzyloxy)hexanoic acid: To a suspension of oil-free NaH (108 mg, 4.50 mmol) in DMF (10 mL) was added (R)-1a (506 mg, 2.97 mmol) at 0 °C. After the mixture was stirred for 30 min. benzyl bromide (0.44 mL, 3.70 mmol) was added at 0 °C. The resulting mixture was stirred for 30 min at 0 °C, and the reaction was quenched by addition of H_2O (15 mL). The mixture was extracted with hexane, and the hexane solution was dried $(MgSO_4)$ and concentrated. Chromatography of the residue on silica gel gave an oil, which was dissolved in H₂O-CCl₄-CH₃CN (3:2:2, 35 mL). To this mixture was added $NaIO_4$ (4.72 g, 22.1 mmol) and RuCl₃·3H₂O (35 mg, 0.13 mmol),¹³ and the resulting mixture was stirred vigorously at room temperature for 1 h. The precipitate was filtered through a pad of Celite with ethyl acetate (15 mL). The mixture was extracted with ethyl acetate repeatedly, and the extracts were dried (MgSO₄) and concentrated. Chromatography of the residue on silica gel provided (R)-2-(benzyloxy)hexanoic acid (444 mg, 67%): $[\alpha]^{25}_{D}$ +68.0° (c 1.02, EtOH) [lit.¹⁴ S enantiomer, $[\alpha]_{D}$ -73° (c 1.19, EtOH)].

The kinetic resolution of 1b-d was carried out in essentially the same manner as that of 1a. The yields and physical data of Notes

optically active 1b-d are as follows.

(R)-1-(2-Thienyl)-2-methylpropan-1-ol (1b): yield 30%; 91% ee (determined by ¹H NMR analysis of the derived MTPA ester); $[\alpha]_{D}^{25}$ +14.2° (c 1.02, CHCl₃). The absolute configuration was proven by transformation to (R)-2-(benzyloxy)-3-methylbutanoic acid. By use of the same procedure as described for conversion of (R)-1a into (R)-2-(benzyloxy)hexanoic acid, (R)-1b (678 mg, 4.34 mmol) was converted into (R)-2-(benzyloxy)-3methylbutanoic acid (494 mg, 55%): $[\alpha]^{25}_{D}$ +66.0° (c 2.60, EtOH) [lit.¹⁴ S enantiomer, $[\alpha]_{D}$ -74° (c 1.01, EtOH)].

(R)-1-(2-Thienyl)-2,2-dimethylpropan-1-ol (1c): yield 25% (determined by ¹H NMR analysis using DIPT as an internal standard); 47% ee (determined by ¹H NMR analysis of the derived MTPA ester); IR (neat) 3420, 2960, 1362, 1005, 693 cm⁻¹; ¹H NMR (CCl₄) δ 0.89 (s, 9 H), 2.25 (br s, 1 H), 4.36 (s, 1 H), 6.64–6.81 (m, 2 H), 6.90–7.02 (m, 1 H). Anal. Calcd for $C_9H_{14}OS$: C, 63.49; H, 8.29; S, 18.83. Found: C, 63.24; H, 8.30; S, 19.18.

(R)-2-Thienylbenzyl alcohol (1d): yield 30%; >95% ee (determined by ¹H NMR analysis of the derived MTPA ester); $[\alpha]^{25}_{D}$ -9.8° (c 0.98, CHCl₃); mp 60.0-60.5 °C (recrystallized from n-C₆H₁₄-CHCl₃). The absolute configuration was proven by transformation to (*R*)-mandelic acid: The above mentioned carbinol (R)-1d (253 mg, 1.33 mmol) was treated with pyridine (1 mL) and acetic anhydride (1 mL) to give the acetate (278 mg, 90%). A mixture of this acetate, NaIO₄ (2.57 g, 12.0 mmol), and RuCl₃·3H₂O (15 mg, 0.06 mmol) in H₂O-CCl₄-CH₃CN (3:2:2, 14 mL) was stirred vigorously at room temperature for 1 h. The precipitate was filtered off through a pad of Celite with ethyl acetate (10 mL). The filtrates were extracted with ethyl acetate $(2 \times 5 \text{ mL})$. The extracts were dried (MgSO₄) and concentrated to give an oil, which was treated with K_2CO_3 (940 mg, 6.8 mmol) in MeOH-H₂O (4:1, 5 mL) at room temperature for 2 h. The solution was neutralized by addition of dilute HCl, and the solvents were removed under reduced pressure. Chromatography of the residue on silica gel provided (R)-mandelic acid (146 mg, 80%): $[\alpha]^{20}_{D}$ -145.1° (c 0.68, EtOH) [lit.¹⁵ $[\alpha]_{D}$ -144.7° (c 0.43, EtOH)].

(R)-1-[2-(5-Methylthienyl)]hexan-1-ol (1e): yield 35%; $>\!95\%$ ee (determined by ¹H NMR analysis of the derived MTPA ester); $[\alpha]^{25}_{D}$ +14.9° (c 1.22, CHCl₃). The absolute configuration was proven by transformation to (R)-2-hydroxyheptanoic acid. By use of the same procedure as described for conversion of (R)-1d into (R)-mandelic acid, (R)-1e (693 mg, 3.49 mmol) was converted into (R)-2-hydroxyheptanoic acid (383 mg, 75%): $[\alpha]^{27}$ D -5.62° $(c 5.16, CHCl_3)$ [lit.¹⁶ S enantiomer, $[\alpha]^{27}_{D} + 5.55 \pm 0.05^{\circ}$ (c 5.5, CHCl₃)].

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