room temperature. After 10 min a solid precipitated from the initially homogeneous mixture. It was filtered, washed with Et_2O, and dried at 40 °C under reduced pressure: mp 142–144 °C; yield 0.400 g (70%); NMR see Table I. See ref 6 for the analytical data.

Alkylation of 3 and 4. To 0.064 mol of 3 or 4 dissolved in 3 cm^3 CH_2Cl_2 was added 0.128 mol of Et_2SO_4 in a glovebox, and the mixture was stirred in a flask under nitrogen or argon at room temperature for 72 h; CH_2Cl_2 was then evaporated under reduced pressure and the residue dissolved in Et_2O (the unreacted complex 3 or 4 and the inorganic salts precipitate). After filtration, the ethereal solution was analyzed by GC (15% OV-225 column, 5 m, N_2 pressure 3 bars, column temperature 150 °C). The standardization was performed as previously described.[2,14]

Acknowledgment. We are grateful to M. Ourevitch, D. Rousselle, and A. Cordaville and to the CNRS-IRCHA group for recording some NMR spectra, to C. Cambillau, and to the referees for constructive criticisms.

Registry No. 1, 53821-96-8; 2, 51833-57-9; 3, 84850-88-4; 4, 81646-42-6; 6, 607-97-6; 7, 5331-73-7; 8, 57592-45-7; potassium ethylacetoacetate enolate, 25368-78-9; [2.1.1]cryptand, 31250-06-3.

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Preparation of Chiral Substituted Succinic Acids

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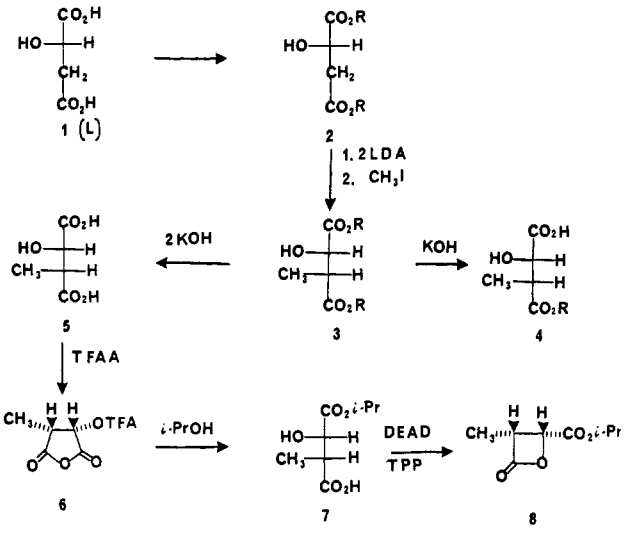
Received June 18, 1982

Only a limited number of small acyclic chiral carbon fragments are available from the natural "chiral pool". However, recent efforts directed toward the synthesis of complex natural products have pointed out the need for a variety of versatile chiral synthetic intermediates.[1] Usually these fragments have been obtained by elaboration of available chiral molecules[1,2] or by chirality transfer from a chiral auxiliary.[3] Described here is a series of synthetic manipulations which utilizes the four-carbon framework of malic acid (1) for the preparation of chiral units with versatile control over the functionality at each carbon.

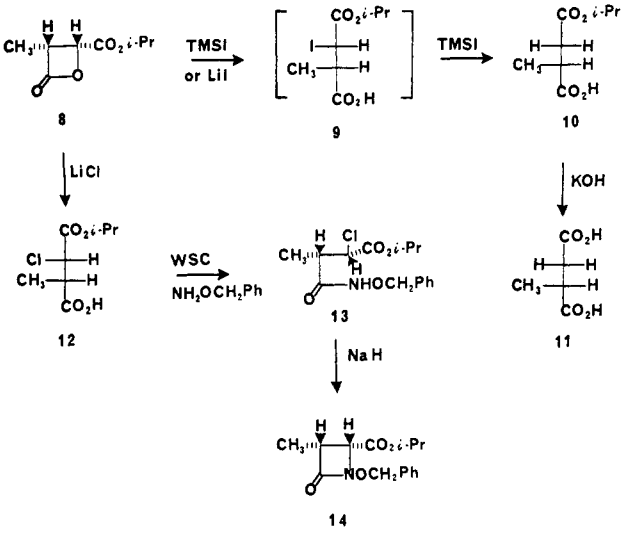
As previously reported,[4,5] alkylation of the dianion of L-malic acid diesters 2 gave predominantly (>10:1) erythro product 3 (Scheme I).[6] Treatment of 3 with 100 mol % of KOH resulted in selective formation of the α-hydroxy acid 4, thus differentiating the two carboxyl groups of the substituted malic acid. Alternatively, complete hydrolysis to the diacid 5 followed by treatment with 200 mol % of trifluoroacetic anhydride (TFAA) provided the anhydride 6. Solvolysis of 6 with any of a variety of alcohols[7] gave the β-hydroxy acid 7 cleanly. Conceptually, β-lactones, like 8, can be prepared by activation of either the carboxyl or hydroxyl groups of 7.[8] Carboxyl activation retains all the stereochemistry from 7, whereas hydroxyl group activation results in inversion at the hydroxyl-bearing carbon. In this study, hydroxyl activation was utilized. Thus, cis β-lactone 8 was prepared in 62% yield by reaction of 7 with diethyl azodicarboxylate and triphenylphosphine (DEAD/TPP).

We have previously used β-lactones similar to 8 as precursors to chiral β-hydroxy carboxylic acids which are useful for the preparation of optically pure β-lactams.[5,9]

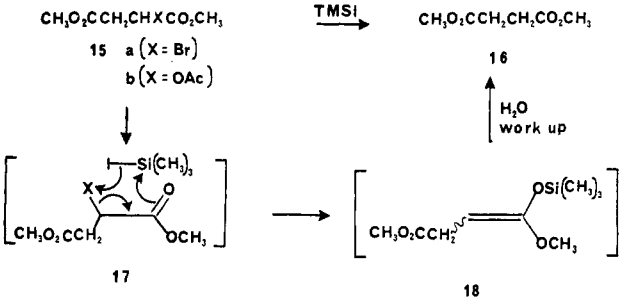
Scheme I



Scheme II



Scheme III



However, the β-lactam synthesis is experimentally simplified if β-halo carboxylic acids can be used instead of the

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0022-3263/83/1948-1114$01.50/0

β-hydroxy acids. Literature analogy[10] suggested that treatment of β-lactone **8** with trimethylsilyl iodide (Me_3SiI) should result in ring opening with inversion to give the erythro iodide **9** (Scheme II). However, when the reaction was attempted with an excess (300 mol %) of Me_3SiI, only small amounts of a mixture of erythro and threo iodide **9** were obtained. Instead, the major product was (*R*)-2-methylsuccinic acid moniisopropyl ester (**10**). Control reactions indicated that **10** was obtained by subsequent reaction of the iodide **9** with the excess Me_3SiI. For example, when the isolated diastereomeric mixture of iodides **9** was resubmitted to the reaction with Me_3SiI, **10** was obtained cleanly. Similarly, dimethyl α-bromosuccinate (**15a**) was reduced directly to dimethyl succinate with Me_3SiI. However, the corresponding acetate (**15b**) was not reduced (Scheme III). Scheme III presents a plausible mechanism for reduction. This process appears to be directly analogous to that described for the reduction of α-halo ketones with Me_3SiI.[11]

In order to determine if any racemization at the remaining chiral center had occurred, **10** was saponified to give optically pure (*R*)-2-methylsuccinic acid. To our knowledge, optically pure (*R*)-2-methyl succinic acid (**11**) has been previously prepared only by resolution,[12] although samples enriched in the *R* isomer have been obtained by asymmetric homogeneous hydrogenation of itaconic acid.[13] The *S* isomer is available by biohydrogenation of 2-methylfumaric acid with resting cells of *Proteus mirabilis* under a hydrogen atmosphere.[14] (*S*)-(−)-Methylsuccinic acid has also been prepared by reaction of (*S*)-(−)-ethyl lactate with ethyl cyanoacetate and DEAD/TPP followed by hydrolysis.[15]

The conversion of β-lactone **8** to iodide **9** was also attempted with LiI. Treatment of a THF solution of **8** with 300 mol % of LiI at room temperature for 30 min again yielded a diastereomeric mixture of erythro and threo iodides **9**, which were clearly distinguishable by ^1H NMR. We had anticipated inversion in opening the β-lactone **8** and suspected that racemization occurred after formation of the iodide. This could proceed by reaction of the iodide first obtained with the excess LiI. Indeed, when the reaction was repeated with portionwise addition of only 100 mol% of LiI, only one diastereomer was obtained in 85% yield. Similarly, treatment of **8** with LiCl in THF for longer times (24 h) at room temperature gave the erythro chloride **12**.

The optical integrity of **12** was verified by its conversion to the hydroxamate **13** and subsequently to the β-lactam **14**. As described in the Experimental Section, a ^1H NMR chiral shift study of **14**, as with similar β-lactams prevously described,[5] revealed that **14** was optically pure.

In conclusion, we have demonstrated ready access to a number of substituted and functionalized optically active succinic acid fragments from malic acid. In addition to the synthesis of β-lactams, these and related chiral units should have considerable utility in the synthesis of several natural products.

Experimental Section

General Methods. Melting points were taken on a Thomas-Hoover melting point apparatus and are uncorrected. Infrared spectra were recorded on a Perkin-Elmer 727b spectrometer. ^1H NMR spectra were obtained in chloroform-*d* with tetramethylsilane as a reference on Varian EM 390 and Nicolet NB 300 instruments. Mass spectra were recorded on a Du Pont DP 102 spectrometer. Optical rotations were determined with a Rudolf Model 574 polarimeter. Elemental analyses were performed by Midwest Microlabs, Indianapolis, IN, or MHW Laboratories, Phoenix, AZ.

Diethyl 2(*S*)-hydroxy-3(*R*)-methylsuccinate (3, R = Et) was obtained as reported[4] by treatment of L-diethyl malate (78.95 mmol) with LDA (210 mol %) followed by quenching the dianion with CH_3I (150 mol %). Pure erythro isomer **3** was obtained in 87% yield after chromatography on silica gel with ethyl acetate–hexanes (15:85).

Isopropyl 2(*S*)-Hydroxy-3(*R*)-methylsuccinate (7). To a solution of **3** (14 g, 68.63 mmol) in 250 mL of dioxane–water (1:1) was added a 20% solution of KOH (48.5 mL, 151 mmol, 220 mol %). The solution was stirred at reflux for 12 h. After the reaction mixture was cooled to room temperature, it was passed through Dowex resin (SO_3H form) and the resin further eluted with water. The eluant was evaporated to dryness under reduced pressure to give the crude diacid **5** as a viscous oil which was used without further purification. To the crude **5** cooled to 0 °C was added excess trifluoroacetic anhydride (TFAA; 33 mL, 223 mmol). The mixture was stirred at 0 °C for 30 min, allowed to warm to room temperature, and stirred for an additional 3 h. The excess TFAA and TFA were evaporated under reduced pressure at room temperature. Isopropyl alcohol was added, and the resulting solution was stirred at room temperature for 12 h. The entire reaction mixture was dissolved in several volumes of ethyl acetate and extracted with three portions of aqueous 1 M $NaHCO_3$. The combined aqueous solutions were washed with ethyl acetate and then acidified to pH 2 with 1.2 N HCl. The aqueous layer was extracted with several portions of ethyl acetate. These latter organic extracts were combined, washed with brine, dried over $MgSO_4$, filtered, and evaporated to give crude **7** as an oil. Chromatography on silica gel with 40% ethyl acetate in hexanes gave 9.5 g (73% from **3**) of **7** as an oil: IR (neat oil) 1700–1740 cm^{-1}; ^1H NMR (90 MHz) δ 1.27 (d, 6 H), 1.30 (d, 3 H), 3.10 (m, 1 H), 4.4 (d, 1 H), 5.13 (m, 1 H), 9.3 (s, 2 H), CO_2H + OH).

3(*R*)-(Isopropoxycarbonyl)-2(*R*)-methyl-β-propiolactone (8). To a solution of the β-hydroxy acid **7** (9.0 g, 47.4 mmol) and PPh_3 (12.41 g, 47.4 mmol) in 150 mL of dry THF at 0 °C was added a solution of diisopropyl azodicarboxylate (DIAD; 9.32 mL, 47.4 mmol) in 30 mL of THF with a double-tipped needle under N_2. After the mixture was stirred at 0 °C for 30 min, the ice bath was removed, and the reaction mixture was stirred at room temperature for 12 h. The solvent was removed under reduced pressure, and the resulting yellow oil was triturated with diethyl ether. Most of the H_2DIAD and Ph_3PO crystallized out and were removed by filtration. The filtrate was concentrated and purified by chromatography over silica gel with CH_2Cl_2–hexanes (3:2) to give 5.52 g (62%) of pure β-lactone **8** as an oil: IR (neat) 1730, 1840 cm^{-1}; ^1H NMR (90 MHz) δ 1.30 (d, 6 H), 1.32 (d, 3 H), 4.1 (m, 1 H), 4.93 (d, 1 H, $J \simeq 6.5$ Hz), 5.18 (m, 1 H); mass spectrum (CI with CH_4), *m/e* 173 (M + 1). Anal. Calcd for $C_8H_{12}O_4$: C, 55.81; H, 7.03. Found: C, 55.49; H, 7.25.

Isopropyl 2(*S*)-Iodo-3(*S*)-methylsuccinate (9). To a solution of the β-lactone **8** (500 mg, 2.91 mmol) in 15 mL of dry THF was added LiI (1.17 g, 8.72 mmol, 300 mol %). The reaction mixture was stirred at room temperature under N_2 for 30 min and then poured into a separatory funnel containing 100 mL of ether. The organic layer was extracted with two 25-mL portions of 10% Na_2CO_3. The basic aqueous layers were combined, acidified to pH ~2 with 6 N HCl, and extracted with three 25-mL portions of ethyl acetate. The combined ethyl acetate layers were washed with 25 mL of water and 25 mL of brine, dried over $MgSO_4$, filtered, and evaporated to give 740 mg (85%) of the desired product as an oil and a mixture of diastereomers ((2*R*,*S*)-**9**): IR (neat) 1705–1745 cm^{-1} (br); ^1H NMR (90 MHz) δ 1.23 (dd, 6 H, diastereomeric isopropyl methyls), 1.45 (d, 3 H), 3.03 (m, 1 H), 4.48 (dd, J = 10.5 Hz), 4.51 (dd, J = 10.5 Hz), corresponding

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to a total of 1 H (diastereomeric C_2 H's, which are clearly distinguishable so that ≤5% of either diastereomer can be deleted), 5.05 (m, 1 H, apparent double heptuplet), 11.13 (s, 1 H, CO_2H).

When the reaction was carried out by adding only 1 equiv of LiI portionwise, a single diastereomer (2*S*,3*S*) of **9** was obtained as an oil in 85% yield: IR (neat) 1705–1745 cm^{-1} (br); ^1H NMR (90 MHz) δ 1.28 (d, 6 H), 1.33 (d, 3 H), 3.18 (m, 1 H), 4.51 (d, 1 H, J = 10.5 Hz), 5.08 (m, 1 H), 11.13 (s, 1 H, CO_2H).

Isopropyl 2(*S*)-Chloro-3(*S*)-methylsuccinate (12). A solution of the β-lactone **8** (500 mg, 2.91 mmol) and LiCl (366 mg, 8.72 mmol) in 15 mL of dry THF was stirred under N_2 at room temperature for 24 h. The reaction was worked up in the same manner as the corresponding iodide **9** to give 430 mg (71%) of **12** as an oil: IR (neat) 1705–1745 cm^{-1} (br); ^1H NMR (90 MHz) δ 1.30 (d, 6 H), 1.32 (d, 3 H), 3.20 (m, 1 H), 4.52 (d, 1 H, J = 7.5 Hz) 5.10 (m, 1 H), 11.5 (s, 1 H, CO_2H); mass spectrum, m/e 208 (^{35}Cl), 210 (^{37}Cl).

2(*R*)-Methyl-4-isopropylsuccinic Acid Monoester 10. To a solution of the cis β-lactone **8** (0.5 g, 2.91 mmol) and NaI (1.3 g, 8.72 mmol) in 30 mL of acetonitrile at room temperature was added 1.10 mL (8.72 mmol) of $(CH_3)_3SiCl$ with continuous stirring under N_2. The reaction mixture was then heated to reflux and maintained at reflux for 24 h. It was then cooled to room temperature, poured into a separatory funnel containing ethyl acetate, and extracted with two 25-mL portions of 10% $NaHCO_3$. The combined aqueous layers were acidified to pH 2 with 6 N HCl and then extracted with several portions of ethyl acetate. The ethyl acetate layers were combined, washed with brine, dried over $MgSO_4$, filtered, and evaporated to give 304 mg (60%) of pure **10** as an oil. Before measuring the optical rotation a sample was chromatographed on silica gel with ethyl acetate-hexanes (3:7). $[\alpha]^{20}_D$ +4.50 (*c* 2.5, CH_3OH); IR (neat) 1700–1730 cm^{-1} (br); ^1H NMR (90 MHz) δ 1.23 (d, 6 H), 1.24 (d, 3 H), 2.23–3.17 (m, 3 H for 1 C_2 H and 2 C_3 H's), 5.03 (m, 1 H), 11.67 (s, 1 H, CO_2H); mass spectrum (CI with CH_4), m/e 175 (M + 1).

Similar treatment of α-iodo ester **9** gave **10** in 74% yield. The spectroscopic data of this sample were identical with those obtained above.

Reduction of Dimethyl 2-Bromosuccinate (15a). A 50-mL flask was charged with NaI (933 mg, 6.67 mmol), dimethyl bromosuccinate **15a** (0.50 g, 2.22 mmol), and CH_3CN (25 mL). The suspension was stirred under N_2, and $(CH_3)_3SiCl$ (0.85 mL, 6.67 mmol) was added. The reaction mixture was heated at reflux for 16 h and then worked up in the same manner as for **10** above (except that a 10% sodium thiosulfate wash was added during the extraction). The dimethyl succinate obtained (74% yield) was identical in all respects with an authentic sample.

2(*R*)-Methylsuccinic Acid (11). To a solution of the monoisopropyl ester **10** (0.7 g, 4.02 mmol) in 5 mL of dioxane was added 2.9 mL of a 20% KOH solution (aqueous). Dioxane–water (1:1) was added portionwise until the reaction mixture became homogeneous. The mixture was then heated at reflux for 12 h. After cooling to room temperature the solution was passed through an ion-exchange resin (Dowex, SO_3H) by eluting with water. The eluant was evaporated under reduced pressure to give a white solid which was recrystallized from ethyl acetate–hexanes to yield 465 mg (88%) of **11** as white crystals: mp 111–112 °C (lit.[12] mp 115 °C); $[\alpha]^{22}_D$ +16.2° (*c* 2.14, absolute ethanol) [lit.[12] $[\alpha]^{20}_D$ +16.59° (*c* 4.136, absolute ethanol); ^1H NMR (Me_2SO-d_6, 300 MHz) δ 1.11 (d, 3 H, J = 7.2 Hz), 2.25–2.33 (m, apparent q, 1 H), 2.47–2.55 (m, apparent q, 1 H), 2.61–2.71 (m, 1 H), 12.25 (s, 2 H, CO_2H's).

***N*-Benzyloxy *O*-isopropyl 2(*S*)-methyl-3(*S*)-chlorosuccinamate (13)** was prepared by the previously reported procedure.[9] Thus, 414 mg (1.986 mmol) of **12** was dissolved along with 380 mg (2.4 mmol) of OBHA·HCl in 20 mL of THF–H_2O (1:1) at an apparent pH of 4.5. A solution of 760 mg (3.97 mmol) of water-soluble carbodimide [*N*-ethyl-*N*^{1}-[3-(dimethylamino)-propyl]carbodiimide] was added and the pH maintained at 4.5 by addition of either 1.0 N NaOH or 1.0 N HCl as required. After 30 min, the solution was extracted with three 25-mL portions of ethyl acetate. The combined ethyl acetate was washed with two 20-mL portions of 1 M citric acid, 20 mL of H_2O, and 20 mL of brine, dried over $MgSO_4$, filtered, and evaporated to give a solid. Recrystallization from ether–hexanes gave 436 mg (70%) of **13** as white crystals: mp 131–131.5 °C; $[\alpha]^{23}_D$ +6.3° (*c* 2.96, CH_3OH); IR ($CHCl_3$) 3410, 1730, 1690 cm^{-1}; ^1H NMR (90 MHz) δ 1.17 (d, 3 H, J = 7.5), 1.28 (d, 6 H), 2.67 (m, 1 H), 4.40 (d, 1 H, J = 10.5 Hz), 4.95 (s, 2 H), 5.07 (m, 1 H), 7.45 (s, 5 H), 8.78 (s, NH); mass spectrum, m/e 255 [M − 58 (OC_3H_7)]. Anal. Calcd for $C_{15}H_{20}NO_4Cl$: C, 57.42; H, 6.38; N, 4.47; Cl, 11.32. Found: C, 57.29; H, 6.16; N, 4.53; Cl, 11.36.

1-(Benzyloxy)-3(*R*)-methyl-4(*R*)-(isopropoxycarbonyl)-2-azetidinone (14). A solution of 100 mg (0.319 mmol) of the hydroxamate **13** in 8 mL of DMF–CH_2Cl_2 (3:5) was added to 15 mg of a 50% mineral oil suspension of NaH under N_2 at room temperature and stirred for 1.5 h. Ether (100 mL) was added, and the resulting solution was washed with water and brine. The ether was then dried ($MgSO_4$), filtered, and evaporated to give 96 mg of an oil. The crude product was purified by chromatography on silica gel by eluting with 20% ethyl acetate in hexanes to provide 86 mg (98%) of pure **14** as an oil: $[\alpha]^{23}_D$ +27.7 ± 1.5 (*c* 1.77, CH_3OH); IR (neat) 1780, 1730 cm^{-1}; ^1H NMR (300 MHz) δ 1.19 (d, 3 H, J = 7.5 Hz), 1.295 (2 overlapping d, 6 H), 3.23 (m, 1 H), 4.24 (d, 1 H, J = 6.3 Hz), 5.09–5.19 (m, 1 H), 5.13 (d, 2 H, diastereotopic protons of OCH_2Ph), 7.35–7.43 (m, 5 H); mass spectrum m/e 277. The ^1H NMR (300 MHz) was also run in the presence of a chiral shift reagent (0.25 M solution of **14** in $CDCl_3$ which was also 0.125 M in tris[3-[(heptafluoropropyl)hydroxymethylene]-*d*-camphorato]europium(III). As described earlier,[5] these conditions clearly distinguish the diastereotopic benzylic protons of *N*-(benzyloxy)-2-azetidinones.

Acknowledgment. We are grateful to the National Institutes of Health and Eli Lilly & Co. for their support. The 300-MHz NMR system used was made available by grants from the NIH and the University of Notre Dame.

Efficient Synthesis of C-Pivot Lariat Ethers. 2-(Alkoxymethyl)-1,4,7,10,13,16-hexaoxacyclooctadecanes[1]

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Received July 26, 1982

Catalysis of ester aminolysis by cyclic and acyclic polyethers reported from this laboratory[2] has served as an encouragement to examine polyether catalysis in other reactions of amines as well as to develop new polyethers. Recently, syntheses of crown ethers with functionalized side chains (lariat ethers[3]) have been reported.[3-7] In particular, alkoxymethyl-substituted crown ethers have attracted considerable attention as synthons for more complex macrocycles and polymer-supported crowns.[4,7-11]

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