ously been described. Most especially it allows for the convenient synthesis of phosphonate reagents **4a–4d** that can be used to prepare a variety of allyl vinyl sulfides.

Experimental Section

General Methods. ^{1}H NMR spectra were recorded on a Varian T-60 spectrometer. IR spectra were recorded with either a Perkin-Elmer 298 or 457 spectrometer. Alkyl benzenethiosulfonates **4a–4e** were prepared from an alkyl halide and sodium benzenethiosulfonate[7b] in 60–75% yield as previously described.[7] Allyl *p*-toluenethiosulfonate was prepared from allyl chloride and sodium *p*-toluenethiosulfonate in an analogous fashion. Phenyl benzenethiosulfonate was prepared by known methods.[8]

General Procedure A. To a stirred solution of dimethyl methylphosphonate (1.4 mmol) in 3 mL of anhydrous ether at −65 °C under argon was added *n*-butyllithium (1.4 mmol) dropwise. The milky white reaction mixture was stirred 10 min at −65 °C, cooled to −78 °C, and then transferred via syringe to a second flask containing the alkyl benzenethiosulfonate **2** (1 mmol) in 2 mL of ether at −78 °C. The anion was added to **2** over a 5-min interval. The reaction mixture was stirred 10 min at −78 °C and then the reaction was quenched by addition of 1 mL of methanol. The crude phosphonate **4** was isolated by an extractive workup with ether-saturated ammonium chloride solution. Crude products were purified by silica gel column chromatography or distillation at reduced pressure.

General Procedure B. To a stirred solution of dimethyl methylphosphonate (5.0 mmol) in 5 mL of anhydrous ether at −65 °C under argon was added *n*-butyllithium (5.0 mmol) dropwise. The milky white reaction mixture was stirred 10 min at −65 °C and then cooled to −78 °C, and the alkyl or aryl benzenethiosulfonate **2** (1.0 mmol) was added in 1 mL of ether dropwise over 5 min. After the mixture was stirred 10 min at −78 °C, the reaction was quenched with 1 mL of methanol and the phosphonate **4** was isolated and purified as in procedure A.

Dimethyl [(allylthio)methyl]phosphonate (4a): colorless liquid; ^{1}H NMR ($CDCl_3$) δ 2.75 (d, 2 H, PCH_2S, J = 13 Hz), 3.38 (d, 2 H, SCH_2, J = 7 Hz), 3.88 (d, 6 H, CH_3, J = 11 Hz), 5.05–6.20 (m, 3 H, $CH{=}CH_2$); IR (film) 3040, 2920, 2820, 1610, 1430, 1240, 1170, 1030, 910, 870, 830, 810 cm^{-1}; mass spectrum, m/e 196 (M^+), 155, 124 (base), 94, 79; exact mass calcd for $C_6H_{13}O_3PS$ 196.0323, found 196.0337.

Dimethyl [(methallylthio)methyl]phosphonate (4b): colorless liquid; ^{1}H NMR ($CDCl_3$) δ 1.85 (br s, 3 H, CH_3), 2.70 (d, 2 H, PCH_2S, J = 14 Hz), 3.32 (br s, 2 H, SCH_2), 3.85 (d, 6 H, CH_3, J = 11 Hz), 4.95 (br s, 2 H, $=CH_2$); IR (film) 3040, 2920, 2880, 2820, 1625, 1435, 1360, 1240, 1170, 1020, 890, 830, 810 cm^{-1}; mass spectrum, m/e 210 (M^+), 155, 124 (base), 109, 94, 79; exact mass calcd for $C_7H_{15}O_3PS$ 210.0480, found 210.0457.

Dimethyl [[(2-chloroallyl)thio]methyl]phosphonate (4c): pale-yellow liquid; ^{1}H NMR ($CDCl_3$) δ 2.70 (d, 2 H, PCH_2S, J = 13 Hz), 3.63 (s, 2 H, SCH_2), 3.80 (d, 6 H, CH_3, J = 11 Hz), 5.27–5.58 (m, 2 H, $=CH_2$); IR (film) 2950, 2850, 1625, 1255, 1055, 1030, 840, 815, 620 cm^{-1}; mass spectrum, m/e 230 (M^+), 232 (M^+ + 2), 195, 124 (base), 109, 94, 79, 58, 45, 43.

Dimethyl [(crotylthio)methyl]phosphonate (4d): colorless liquid; ^{1}H NMR ($CDCl_3$) δ 1.75 (d, 3 H, CH_3, J = 4 Hz), 2.70 (d, 2 H, PCH_2S, J = 14 Hz), 3.32 (d, 2 H, SCH_2, J = 6 Hz), 3.85 (d, 6 H, CH_3, J = 11 Hz), 5.50–5.75 (m, 2 H, $CH{=}CH$); IR (film) 3010, 2975, 2850, 1650, 1450, 1370, 1250, 1180, 1050, 1025, 835, 820 cm^{-1}; mass spectrum, m/e 210 (M^+), 156, 124 (base), 110, 94, 79, 55; exact mass calcd for $C_7H_{15}O_3PS$ 210.0479, found 210.0482.

Dimethyl [(methylthio)methyl]phosphonate (4e): colorless liquid; ^{1}H NMR ($CDCl_3$) δ 2.35 (s, 3 H, CH_3), 2.78 (d, 2 H, PCH_2S, J = 12 Hz), 3.82 (d, 6 H, CH_3, J = 11 Hz); IR (film) 2950, 2850, 1700, 1630, 1450, 1370 cm^{-1}; mass spectrum, m/e 170 (M^+), 124 (base), 109, 94, 79, 61, 45.

Dimethyl [(phenylthio)methyl]phosphonate (4f): colorless liquid; ^{1}H NMR ($CDCl_3$) δ 3.23 (d, 2 H, PCH_2S, J = 14 Hz), 3.82 (d, 6 H, CH_3, J = 12 Hz), 7.14–7.70 (m, 5 H, C_6H_5); IR (film) 3050, 2950, 2840, 1580, 1480, 1435, 1250, 1180, 1050, 1025, 840, 815, 740, 690 cm^{-1}; mass spectrum, m/e 232 (M^+), 231 (base), 123, 121, 110, 109, 93, 77; exact mass calcd for $C_9H_{12}O_3PS$ (M^+ − H) 231.0245, found 231.0233.

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Registry No. 2a, 69530-64-9; **2b**, 69530-65-0; **2c**, 69567-85-7; **2d**, 69530-66-1; **2e**, 1125-25-3; **2f**, 1212-08-4; **3a**, 52713-51-6; **4a**, 84836-03-3; **4b**, 84836-04-4; **4c**, 84836-05-5; **4d**, 84836-06-6; **4e**, 25508-32-1; **4f**, 70369-42-5; $(CH_3O)_2P(O)CH_3$, 756-79-6.

(7) (a) A. P. Kozikowski, A. Ames, and H. Wetter, *J. Organomet. Chem.*, **164**, C33 (1979); (b) S. Hayashi, M. Furukawa, J. Yamamoto, and K. Niigata, *Chem. Pharm. Bull.*, **15**, 1188 (1967).

(8) B. M. Trost and G. S. Massiot, *J. Am. Chem. Soc.*, **99**, 4405 (1977).

HMPA Dehydro Dimer: A Remarkable Complexing Agent of Lithium Cation

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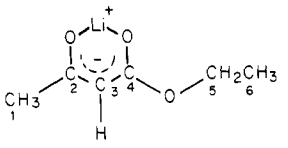
Lithiated enolates are particularly stable. Thus, the Li enolate of ethyl acetoacetate **1** does not react with diethyl



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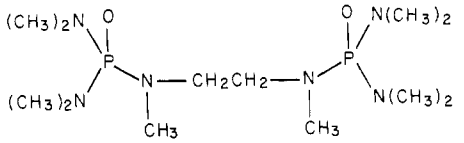

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sulfate at room temperature while the K enolate does.[2] However, in the presence of solvents capable of coordinating cations such as HMPA[2,3] or of macrocyclic coordinates,[4] the anion–cation interaction is weakened so that alkylation does take place.

We decided to examine to what extent HMPA dehydro dimer ("diHMPA") **2**, obtained by radical coupling of HMPA,[5] should present stronger complexing properties of alkali cations, due to chelate formation. In this paper, we examine the behavior of **1**, as well as the K analogue of **1**, in the presence of "diHMPA" (**2**).

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(2) G. Nee, Y. Leroux, and J. Seyden-Penne, *Tetrahedron*, **37**, 1541 (1981), and references therein.

(3) A. L. Kurz, I. P. Beletskaya, A. Macias, and O. A. Reutov, *Tetrahedron Lett.*, 3679 (1968).

(4) C. Cambillau, Thèse, University of Orsay, June 1978.

(5) H. Naarman, M. Beaujean, R. Merenyi, and H. G. Viehe, *Polymer Bull.*, **1980**, *2*, 417. "diHMPA" has been quoted in Normant et al.: (H. Normant, T. Cuvigny, and P. Savignac, *Synthesis*, 805 (1975)), but neither preparation nor properties were given.

0022-3263/83/1948-1111$01.50/0

Table I. Chemical Shift Data of the Enolate Moiety in 1, 3, and 4

	chemical shift, δ										
compd	C_1	C_2	C_3	C_4	C_5	C_6	H_1	H_3	H_5	H_6	Li[a]
1	27.7	186.7	57.9	172.3	84.2	15.1	1.80	4.60	4.01	1.13	0.636
3	27.6	187.7	57.5	171.6	82.8	14.8	1.78	4.57	4.0	1.17	0.76
4	47.9	187.5	56.9	171.1	80.9	14.8	1.77	4.53	4.01	1.17	1.79 (−0.65)

[a] External standard, 1 M LiCl in D_2O; $LiClO_4$ in CD_2Cl_2 + "diHMPA", δ −0.64.

Table II. NMR Parameters of [2.1.1]Cryptand and of "diHMPA" in CD_2Cl_2

		chemical shift, δ		
complexing agent	nucleus	ligand only	ligand in 3 or 4[f]	ligand in 3 or 4 + 1 molar equiv of $LiClO_4$[f]
[2.1.1]cryptand	^{13}C	70.9, 70.3, 70.1, 57.7, 55.9	68.6, 66.8, 65.7, 51.7, 50.4	68.6, 66.8, 65.7, 51.7, 50.4
"diHMPA"	^{1}H	3.08[a] (CH_2), 2.67 (NCH_3), 2.63 ($N(CH_3)_2$)	3.11,[b] 2.66, 2.61	3.07, 2.68, 2.64
	^{13}C[c]	48.0,[d] 36.9, 34.6	48.4,[e] 36.6, 35.1	49.1, 36.7, 35.5
	^{31}P	23.9	25.2	25.3

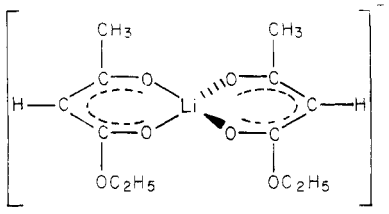
[a] $^3J_{PH}$ are from 9.5 to 11.5 Hz. [b] Integration confirms a ratio of two enolates per one "diHMPA". [c] $^2J_{PC}$ are from 3.4 to 4.7 Hz. [d] Apparent triplet. [e] Doublet. [f] In 3 for "diHMPA" and in 4 for [2.1.1]cryptand.

Results

While a 0.27 M solution of lithium enolate 1 in tetrahydrofuran remains homogeneous upon addition of HMPA, a precipitate (mp 142–144 °C) is formed when diHMPA is added. In contrast, no precipitate is observed when the same experiment is conducted with the K enolate.

The elemental analysis of the precipitate 3[6] corresponds to formula $C_{24}H_{52}Li_2N_6O_8P_2$ (2.e., one diHMPA residue for two enolate moieties) and is confirmed by ^1H NMR (vide infra). Thie complex is poorly soluble in THF but very soluble in CH_2Cl_2 or $CHCl_3$, while 1 is insoluble in these two solvents.

It has been shown that lithium salts frequently lead to (Li^+, Li^+ triple ion) couples,[7] especially in the case of acetoacetates[4,8,9] or acetoacetonates.[10] The structure of the K^+ analogue 5 has been determined by X-ray crys-



[Li, 2.1.1]$^+$, 4

[K, 2.2.2]$^+$, 5

tallography.[8] The stoichiometry of the isolated complex should indicate such a structure. For this reason, we decided to compare the spectroscopic and reactivity properties of 3 with those of complex 4.

(1) NMR Studies. We indicate in Table I the ^1H, ^{13}C, and ^7Li NMR chemical shifts of the lithium enolate 1 in deuterated THF (1 is insoluble in CD_2Cl_2) and those of complexes 3 and 4 in CD_2Cl_2. The ^7Li NMR of 4 has been described previously.[9] The complementary NMR parameters of the complexing agents in 3 and 4, as well as those of the ligands alone and in the presence of 1 equiv $LiClO_4$, are listed in Table II.

Table III. Alkylation of the Li Enolate by Et_2SO_4[a]

			distribution, %		
reagent	solvent	yield, %	6	7 + 8	7/8 ratio
1[b]	THF	<5			
3[c]	CH_2Cl_2	53	54	46	92/8
4[c]	CH_2Cl_2	66	45	55	91/9

[a] At room temperature for 72 h. [b] Concentration of 0.27 M for each reagent. [c] Concentration of 3 or 4 of 0.064 M; concentration of $EtSO_4$ of 0.128 M. In all cases, completion to 100% yield consumed all starting material.

From Tables I and II the following comments can be made.

(a) While 4 exhibits two signals in the ^7Li NMR, indicating thus that the exchange between the two Li cations complexed either by the two enolate moieties or by [2.2.2.]cryptand is slow,[9] a single signal is observed in 3, even at −40 °C (at lower temperatures the compound is not soluble). This signal is located at 0.76 ppm, which does not correspond to the signal expected if a rapid exchange were taking place between a Li cation complexed by two enolate moieties (1.79 ppm) and a Li cation complexed by diHMPA (0.64 ppm) i.e., 0.57 ppm (vide infra).

(b) The ^{13}C signals of the enolate moiety of 3 and 4 are different: C_3 and C_4 are shielded in 3 and 4 relative to those in 1, the shielding being slightly stronger in 4. The greatest difference is observed for C_5: in 3 this carbon is moved upfield by 1.4 ppm relative to that in 1 while in 4 this upfield shift is 3.3 ppm.

Furthermore (Table II), the identical values of the ^{13}C chemical shifts of [2.1.1]cryptand in 4 and in the $LiClO_4$–[2.1.1.]cryptand complex confirms the presence of the cation inside the macrocyclic coordinate.[4]

A slight difference in ^{13}C chemical shifts of diHMPA is observed in 3 and in $LiClO_4$–diHMPA: in both cases, a small downfield shift is observed relative to pure diHMPA. A similar observation can be made from ^{31}P chemical shifts.

(c) The ^1H NMR does not show any significant difference between 1, 3, and 4.

(2) Reactivity with Et_2SO_4. Et_2SO_4 has been selected as an alkylating agent, as it is well-known that the reaction

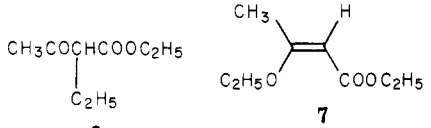
(6) Anal. Calcd: C, 45.88; H, 8.28; N, 13.37; P, 9.87; Li, 2.2. Found: C, 44.94; H, 8.25; N, 13.73; P, 9.66; Li, 1.84.
(7) G. Fraenkel and M. P. Hallden-Abberton, *J. Am. Chem. Soc.*, **103**, 5657 (1981). A. Regis and J. Corset, *Chem. Phys. Lett.*, **32**, 462 (1975).
(8) C. Cambillau, G. Bram, J. Corset, and C. Riche, *N. J. Chim.*, **3**, 9 (1979).
(9) C. Cambillau and M. Ourevitch, *J. Chem. Soc., Chem. Commun.* 996 (1981).
(10) M. Raban and D. Haritos, *J. Am. Chem. Soc.*, **101**, 5178 (1979).
(11) A. Reutov and A. L. Kurts, *Russ. Chem. Rev.*, **46**, 1040 (1977).

Table IV. Alkylation of the K Enolate by Et_2SO_4[a]

addend	yield, %	distribution, % 6	7 + 8	7/8 ratio
b[2]	63	88	12	
2HMPA[a,2]	85	45	55	
1 "diHMPA"	95	30	70	15/85

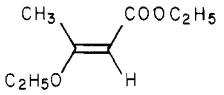
[a] At room temperature for 24 h; the concentration of each reagent in THF was 0.27 M. [b] Concentration of 0.33 M; completion to 100% yield consumed all starting material.

of enolates such as 1 with this electrophile is highly sensitive to cation–anion interactions, whereas the effects are less important with other reagents. The data related to alkylation of 1, 3, and 4 are indicated in Table III, and those related to the alkylation of the K^+ analogue of 1 are in Table IV. These reactions lead to a mixture of C-alkylated compound 6 and of *Z* or *E* O-alkylated compounds 7 and 8.[2]



6

7



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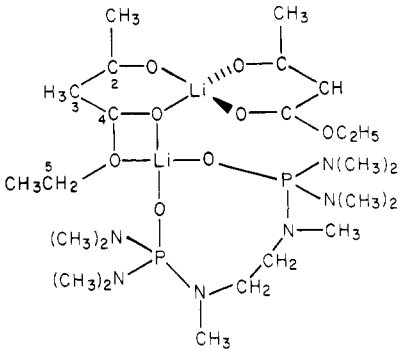
From Table III it appears that 3 and 4 not only have similar solubility properties but also have very similar behavior both concerning the alkylation extent at a given reaction time and the product distribution. Different results are observed when 1 is reacted in THF in the presence of 2 molar equiv of HMPA[2] (6, 34%; 7 + 8; 66%), but the solvent and concentrations are different. Table IV shows that the influence of diHMPA on the rate and product ratios is different from what one observes when using 2 molar equiv of HMPA in THF.

Discussion

(1) Lithium Complexation. The comparison of the ^{13}C NMR parameters of 3 and 4 shows that the environment of the enolate moieties is not identical in the two cases. However, the solubility properties, the parallel reactivities, and the C/O alkylations ratios observed under identical experimental conditions (which is a good indication of the enolate–cation association[2,3]) strongly suggest that 3 has a triple ion structure.

The most affected ^{13}C chemical shifts of 3 and 4 are at carbon 5 and, to a lesser extent, at carbons 3 and 4. This would suggest that a Li^+ cation is in proximity to these atoms.

^7Li NMR also can give some indications: as $LiClO_4$ is totally insoluble in CD_2Cl_2 unless 1 equiv of diHMPA is added, it appears that the cation is strongly interacting with the complexing agent. The fact that the ^7Li resonance in the $LiClO_4$–diHMPA and [Li, 2.1.1] complexes are the same (–0.64 ppm) is in agreement with this proposal. The fact that one signal is observed for 3 indicates a fast exchange between the two Li cations. Its position (0.76 ppm) is not the mean value between the $[Li, diHMPA]^+$ and the $[Li, enolate\ triple\ ion]^-$ complexes in 4: this indicates a possible interaction of Li^+ inside the triple ion structure with the complexing agent which lies in its vicinity. The sensitivity of ^7Li in such an environment to the solvent has been previously quoted.[9] The model shown as structure 3 accounts for these observations.



3

The crystal structure of the lithium enolate of acetylacetone[12] shows that one oxygen atom can be coordinated to two lithiums which is in agreement with our proposal. An X-ray study would strengthen this hypothesis, but we have not been able to obtain single crystals.

If one compares (Table III) the alkylation results obtained in the presence of diHMPA (46%) and HMPA (66%), it appears that, although the solvent is different, the amount of O-alkylation is larger when HMPA is used. This suggests that a triple ion species is not involved in this latter process.

(2) Potassium Complexation. Although no definite complex has been isolated, it appears that the chelation with diHMPA induces an increase in the rate of alkylation as well as an enhancement of the amount of O-alkylated compound, relative to the reaction run with 2 mol of HMPA. This is indicative of a weaker enolate–cation interaction in the former case.[2,3] Furthermore, in the presence of 2 mol of HMPA, different species in equilibrium appear to participate in the alkylation process.

Conclusion

We have shown that "diHMPA" is a good complexing agent of K^+ and Li^+. In the latter case, it shows peculiar behavior which is analogous to that of macrocyclic complexing agents. Furthermore, we have observed that diHMPA is inert to strong bases (*n*-BuLi, Ph_3CK in DME),[13] so that further applications can be envisioned.

Experimental Section

NMR spectra were run on a Brucker WH 90 spectrometer at 34.97 MHz (^7Li; external standard 1 M LiCl in D_2O), a CFT20 Varian spectrometer at 20 MHz (^{13}C; internal standard Me_4Si), and an A 60 Varian spectrometer at 60 MHz (^1H; internal standard Me_4Si). All solutions were prepared in a glovebox and kept under argon.

The solvent purification and some reactions were conducted as previously described.[2] GC analyses were run on HYFI 600 D Aerograph equipment.

Synthesis of "diHMPA". HMPA (75 g) and 3.06 g of *tert*-butyl peroxide were introduced in a glass tube. After being carefully degassed, the tube was sealed and placed in an oil bath at 140 °C for 12 h. After cooling at room temperature, the glass tube was immersed in a liquid nitrogen bath, opened, and then raised to room temperature. Acetone and t-BuOH were evaporated under reduced pressure, and the residue was carefully fractionated: bp 165–175 °C (5×10^{-3} mmHg); yield 78%.

Preparation of 3. To 0.351 g of dry lithiated enolate 1 dissolved in 6.9 cm^3 of anhydrous THF, was added 0.332 g of diHMPA. This mixture was stirred under nitrogen or argon at

(12) F. A. Schroder and H. P. Weber, *Acta Crystallogr., Sect. B* **B31**, 1745 (1975).
(13) G. Nee, unpublished results.

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.

(14) G. Nee and J. Seyden-Penne, *Tetrahedron*, 38, 3485 (1982).

Preparation of Chiral Substituted Succinic Acids

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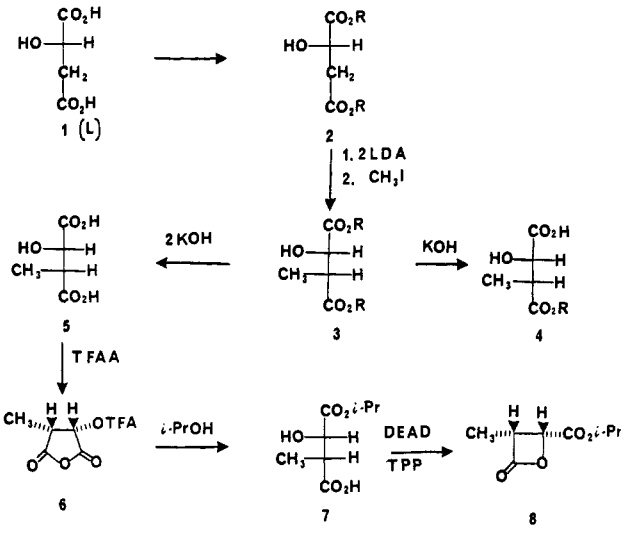
Only a limited number of small acyclic chiral carbon fragments are available from the natural "chiral pool". However, recent efforts directed toward the synthesis of complex natural products have pointed out the need for a variety of versatile chiral synthetic intermediates.[1] Usually these fragments 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.

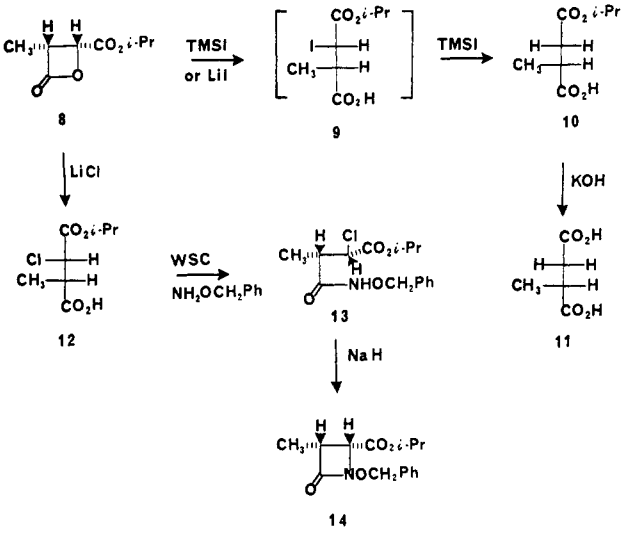
As previously reported,[4,5] alkylation of the dianion of L-malic acid diesters 2 gave predominantly (>10:1) erythro product 3 (Scheme I).[6] Treatment of 3 with 100 mol % of KOH resulted in selective formation of the α-hydroxy acid 4, thus differentiating the two carboxyl groups of the substituted malic 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]

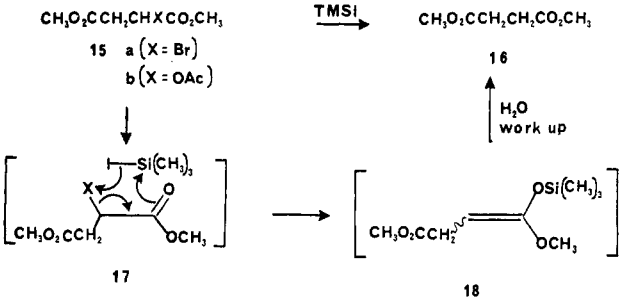
Scheme I



Scheme II



Scheme III



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

† Fellow of the Alfred P. Sloan Foundation 1981–1983.

(1) For a nice review see: Bartlett, P. A. *Tetrahedron* 1980, *36*, 2.
(2) Collum, D. B.; McDonald, J. H., III; Still, W. C. *J. Am. Chem. Soc.* 1980, *102*, 2118.
(3) (a) Evans, D. A.; Nelson, J. V.; Vogel, E.; Taber, T. R. *J. Am. Chem. Soc.* 1981, *103*, 3099 and references therein. (b) Evans, D. A.; Takacs, J. M. *Tetrahedron Lett.* 1980, *21*, 4233.
(4) Seebach, D.; Wasmuth, D. *Helv. Chim. Acta* 1980, *63*, 197.
(5) Miller, M. J.; Bajwa, J. S.; Mattingly, P. G.; Peterson, K. *J. Org. Chem.* 1982, *47*, 4928.
(6) For an alternative preparation of 3 see: Mori, K.; Iwasawa, H. *Tetrahedron* 1980, *36*, 87.
(7) While isopropyl alcohol was used in the sequence reported here, the reaction works just as well with methanol or benzyl alcohol.

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