mide 7789-60-8; 1,3-dithiane, 505-23-7; (+)-(S)-2-methylbutanoic acid, 1730-91-2.

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(16) An attempt to obtain 12 by direct cyclization of 9 afforded a very low yield of the expected alkylpyridine and other unidentified products.
(17) A sample containing ~80% of 5 was obtained by preparative GLC. The rotatory power of this sample ([α]²⁵0 ~+30°) showed that 5 is also optically active. On this basis, and taking into account the reaction intermediates used to the sufficiency are consequently with the sufficiency are consequently. mediates suggested for the cyclization of 1,4-dioxo compounds with hydroxylamine, ¹⁸ a possible pathway for the formation of **5** from **10** can be formulated as follows.

18) See ref 8, p 279

- (19) See ret 8, p. 279.
 (19) The optical purity was determined in case of chiral 2-sec-butylpyridine (2) by NMR analysis in the presence of tris[3-(trifluoromethylhydroxymethylene)-d-camphorato]europium(III).²⁰ From the NMR results obtained on a solution containing 35.3 mg of (--)-(R)-2, [α]²⁵D 12.8 (ethanol), prepared by resolution of racemic compound with dibenzoyl-(+)-tartaric acid, 1 and 59.9 mg of the europium complex, [α]o + 196° (carbon tetrachloride), in 0.7 ml of carbon tetrachloride) looking at the classe of the 12 rice retent and 5 the methyl group restates at the classe of the 12 rice retent and 5 the methyl group restates at the classe of the property and the classe of the property and the classe of the property and the property an signals of the H-2 ring proton and of the methyl group protons nearer to signals of the H-2 hit priority and of the merty group priority hears the asymmetric center, a value of 29 (ethanol) for the maximum rotatory power of 2 was extrapolated. This value is in satisfactory agreement with that obtained through the cleavage method, $[\alpha]^{25}$ D +32.2° (ethanol) (Scheme IV), considering that the precision of the NMR method is about $\pm 10\%$.
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Reaction of Azomethine Ylides with Sulfur Ylides. A Novel Azetidine Synthesis

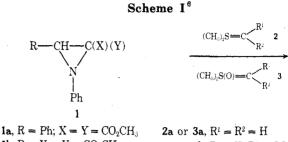
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Aziridines represent versatile substrates which can serve as azomethine ylide precursors. We have shown an unusual feature of these ylides: they are able to react with both electrophilic and nucleophilic reagents. 1-4 Not the least interesting of these last are sulfur ylides which lead to azetidines in a novel way.⁵ As a result of a continuing study of this last reaction we wish to report some new information and experimental details which were not given in the preliminary note.5

The various azetidines which were prepared are shown in Scheme L



$$\begin{array}{lll} \textbf{1b}, \ R = X = Y = CO_2CH_3 & \textbf{2b}, \ R^1 = H; \ R^2 = CO_2CH_3 \\ \textbf{1c}, \ R = Ph; \ X = H; \ Y = CO_2C_2H_5 & \textbf{2c}, \ R^1 = H; \ R^2 = CO_2C_2H_5 \\ \textbf{2d} \ \ \text{or} \ \ \textbf{3d}, \ R^1 = H; \ R^2 = COPh \\ \textbf{2e}, \ R^1, \ R^2 = C_{13}H_{18}{}^a \\ \textbf{2f}, \ R^1 = Cl; \ R^2 = CO_2C_2H_5 \end{array}$$

4d and 4'd, $R = X = Y = CO_2CH_3$

6b or 6'b; 6d or 6'd; 6e or 6'e; 6f or 6'f

2g, $R^1 = Br$; $R^2 = CO_2C_2H_5$

^a Fluorenylidene.

In the reactions of ethyl chloro- (or bromo-) (dimethylsulfuranylidene)acetate (2f or 2g) with aziridine 1a and 4oxazoline 5, we expected competition between dimethyl sulfide (DMS) and bromide or chloride elimination. However, in each case, DMS is a better leaving group than the halide and we observe only formation of the epimeric halogenated azetidines 4f, 4'f; 4g, 4'g; and 6f, 6'f. In an attempt to prepare the C2 monosubstituted azetidines, the sulfonium ylides were heated with aziridine 1c in boiling benzene (under these conditions, 1c is in equilibrium with the corresponding azomethine ylide^{2,3,7}) but no reaction occurs, except the isomerization of 1c and the partial decomposition of the sulfur ylide. We have no explanation for this failure; it is not due to instability of the sulfonium ylide, which is still present after 8 hr of reaction. Nevertheless it is possible to get the C2 monosubstituted azetidines in good yield by demethoxycarbonylation of compounds 4 with piperi-

Compd	R ¹									
		${\mathtt R}^2$	Ir, cm ⁻¹ VC=0	NMR, $\delta(Me_4Si)$						
				н ₂	н ₃	н ₄	J ₂₃ , Hz	J ₃₄ , Hz	со ₂ сн ₃ (со ₂ сн ₂ сн ₃)	Rei %
7a	H	Н	1744	4.45	$2.42^a \ 2.97$	4.82	8.8 7.4	$\substack{8.4\\7.6}$	3.83	90
7'a	H	Н		4.14	2.58 2.30	5.36	b	b	3.69	10
7b	Н	CO_2CH_3	1744	4.68 d	3.50 t	4. 93 d	7.0	7.0	$\frac{3.84}{3.74}$	≥90°
7c	Н	$CO_2C_2H_5$	1740	4.76 d	3.50 t	5.03 d	7.0	7.0	$3.92 \\ (1.27)$	≥90°
7 d	H	COPh	1678 1740	4.91 d	4.42 t	5.00 d	7.0	7.0	3.86	≥90°
7e	d		1754	5.01 s		5.51 s			3.24	91
7'e	d			4.85 s		5.39 s			3.20	9

Table I Ir and NMR Data of 7 and 7'

 $^aJ_{\text{gem}}=11.0$ Hz. b The mixture 7a + 7'a has a satisfactory elementary analysis. It is not possible to measure the coupling constants of 7'a on the spectra of this mixture. The given δ is the position of the multiplet's center. c Near 7 it is possible to detect (NMR) the presence of some impurities bearing ester groups. The integration of the methoxycarbonyl methyl protons allows us to give the ratio of 7. These impurities are not identified. d Azetidine resulting from the demethoxycarbonylation of 4e.

dine in boiling toluene or xylene. Here this reaction, which gives fairly good results for five-membered heterocycles, successfully applied to four-membered rings.

In the resulting mixtures, compounds 7 are always the major products (≥90%); they are isolated and purified by recrystallization. Compounds 7'a and 7'e are identified by NMR (Table I); 7'b, 7'c, and 7'd, which are formed in small quantities (≤10%), are not identified with certainty.

The values of J_{23} and J_{34} in compounds 7 (\leq 7, 6 Hz) indicate that H_2 and $R^1 = H_3$ are trans and the same for H_4 and $R^1 = H_3$.

The assignment of formula 7e to the major product arising from 4e is realized by assuming that this compound behaves as the other azetidines 4.

Compounds 7a-c and 7e are recovered unchanged after boiling with piperidine in toluene. When treated with N-deuteriopiperidine, 7b and 7c are unchanged; there is no isomerization or deuterium exchange with H_2 or H_3 . Furthermore, the N-deuteriopiperidine converts 4b to 7b with a deuterium on C_2 only, providing a simple method to deuterate the azetidines on C_2 . These results indicate that the demethoxycarbonylation of 4a-c and 4e is under kinetic control and takes place without changing the configuration at carbon 3.

4d (R = Ph) is a special case since H_3 is acidic enough to be replaced by deuterium, owing to the presence of a benzoyl group. Thus the reaction of 4d with N-deuteriopiperidine in boiling toluene leads to a deuterated compound on C_3 and not on C_2 . Furthermore, demethoxycarbonylation of pure 4'd gives the same mixture as 4d. Demethoxycarbonylation of 4d is slower than the isomerization of 4'd to 4d.

These results may be rationalized as follows: the demethoxycarbonylation proceeds through the intermediate carbanion 8, which undergoes a kinetic protonation. These carbanions with an α nitrogen atom are likely to be pyramidal. There are two carbanions epimeric on C_2 which are easily interconverted and which are protonated to give 7 and 7' in a ratio which depends on the relative stabilities of the epimeric carbanions.

Experimental Section¹⁰

The sulfur ylides were prepared by known procedures: 2a and 3a, 11 2b and 2e, 12 2d, 13 2e, 14 2f and 2g, 15 3d. 16

The aziridines 1a and 1b and 4-oxazoline 5 were obtained by thermolysis of the corresponding triazolines under nitrogen.^{3,17} The neat triazoline was thermolyzed under nitrogen (1a, 180° for 15 min; 1b, 145° for 20 min; 5, 155° for 20 min). When the evolution of N_2 had ceased, the resulting aziridines or 4-oxazoline were dissolved in anhydrous benzene or THF.

The addition of the sulfur ylides to aziridines 1 or 4-oxazolines 5 was always realized under nitrogen and anhydrous solvents were used.

Azetidine Synthesis. As an example, we will describe the reaction between aziridine 1a and dimethylphenacylidene sulfurane (2d). All other reactions were done following a similar procedure and we will indicate only the modifications of experimental conditions in each case. Furthermore, we will report the experimental and spectroscopic data related to the compounds which were not already published, 5 i.e., reactions with halogenated sulfuranes and also reactions with the trimethoxycarbonyl aziridine 1b.

3-Benzoyl-2,2-dimethoxycarbonyl-1,4-diphenylazetidines 4d and 4'd ($R = Ph; X = Y = CO_2CH_3$). Aziridine 1a, resulting from the thermolysis of 2.1 g (6 mmol) of the corresponding triazoline, was dissolved in 40 ml of benzene. Then 1.08 g (6 mmol) of the sulfurane 2d in 20 ml of benzene was added dropwise. When the addition was complete, the reaction mixture was allowed to stand for 2 hr at room temperature before removing the solvent in vacuo. The reaction led to a quantitative yield (NMR) of the mixture 4d + 4'd. Recrystallization from methanol gave 85% (2.3 g) of 4d + 4'd (79:21). Fractional crystallization from benzene-petroleum ether afforded 0.6 g of 4d, mp 152°, and 0.2 g of 4'd, mp 200°.

Compounds 4b, 4'b, 4c, and 4'c were obtained by this procedure. For compounds 4a and 4e the reaction was carried out in the mixture THF-Me₂SO (4:1) at room temperature. All these products

were recrystallized in boiling methanol except for compound 4e (benzene-petroleum ether).

3-Chloro-2-2-dimethoxycarbonyl-1,4-diphenyl-3-ethoxycarbonylazetidines 4f and 4'f. Using the same procedure, the re action led to a quantitative yield (NMR) of the mixture 4f + 4'f

Recrystallization from methanol afforded 4f in a 72% yield: mp 154°; ir 1736, 1756 cm⁻¹ (ester C=O); NMR (CDCl₃) δ 5.94 (1 H, s), 3.97 (3 H, s), 3.54 (3 H, s), 1.30 (3 H, t), 4.30 (2 H, q), 6.40–7.60 (10 H, m). 4'f (isomer only identified by NMR): δ 5.68 (1 H, s), 3.81 (3 H, s), 3.64 (3 H, s), 0.82 (3 H, t), 3.70 (2 H, q), 6.40-7.60 (10 H,

The chemical shift of the methyl of the ethoxycarbonyl group on carbon 3 indicates that 4f has the trans configuration (phenyl and ester group on C3 and C4). It is the same for azetidine 4g (see ref

3-Bromo-2,2-dimethoxycarbonyl-1,4-diphenyl-3-ethoxycarbonylazetidine 4g. A solution containing 1.36 g (6 mmol) of ethyl bromo(dimethylsulfuranylidene)acetate (2g) in 25 ml of chloroform was added at 0-5° to a solution of 6 mmol of 1a in 10 ml of chloroform. After standing for 4 hr at room temperature, the solution was washed with water and dried (Na₂SO₄). Removal of CHCl3 in vacuo led to 2.5 g of an oily residue which contained (NMR) only the isomer 4g (85%), benzaldehyde, and methyl anilinomalonate (these two last products result from the hydrolysis of unreacted 1a, 15%). Recrystallization from methanol led to azetidine 4g: 1.5 g (52%); mp 150°; ir 1738, 1760 cm⁻¹ (C=O); NMR (CDCl₃) δ 5.88 (1 H, s), 4.04 (3 H, s), 3.61 (3 H, s), 1.33 (3 H, t), 4.36 (2 H, q), 6.60-7.70 (10 H, m).

3-Benzoyl-1-phenyl-2,2,4-trimethoxycarbonylazetidines 4d and 4'd ($R = X = Y = CO_2CH_3$). The equilibrium between aziridine 1b and the corresponding azomethine ylide is established only at the reflux of toluene. So the reaction between 1b and sulfurane 2d was run in boiling toluene for 3 hr. The reaction led to a quantitative yield (NMR) of the mixture 4d + 4'd (48:52). Recrystallization from methanol gave pure 4'd (38%): mp 164°; ir 1670 (C=O ketone), 1724, 1734, 1760 cm⁻¹ (C=O esters); NMR (CDCl₃) δ 5.00 (1 H, d, J = 8.9 Hz), 5.27 (1 H, d, J = 8.9 Hz), 3.60 (3 H, s) 3.64 (3 Hz)H, s), 3.70 (3 H, s), 6.60-7.90 (10 H, m); 4d (isomer only identified by NMR) δ 5.15 (1 H, d, J = 7.0 Hz), 5.39 (1 H, d, J = 7.0 Hz), 3.71 (3 H, s), 3.79 (3 H, s), 4.03 (3 H, s).

The stereochemical assignment is based on J_{34} values:⁵ 4'd is the cis compound ($J_{34} = 8.9 \text{ Hz}$) and 4d the trans compound ($J_{34} = 7.0$ Hz).

2-Acetyl-3-chloro-1,4-diphenyl-3-ethoxycarbonyl-2-methoxycarbonylazetidines 6f and 6'f. The sulfonium ylide 2f (1.1 g, 6 mmol) was added to a 30-ml benzene solution of 6 mmol of 4oxazoline 5 (obtained from 2 g of the corresponding triazoline). After standing for 18 hr at room temperature the benzene was removed in vacuo. The reaction led (NMR) to a mixture 6f + 6'f (75:25). Recrystallization from methanol gave 0.3 g of 6f + 6'f (83: 17). Fractional crystallization in benzene-petroleum ether led to 6f (or 6'f), mp 126°. A second crop of 0.4 g, containing 6f + 6'f (35: 65), was obtained from methanol, overall yield 28%: 6f (or 6'f) ir 1702, 1740, 1756 cm⁻¹ (C=O); NMR (CDCl₃) δ 5.94 (1 H, s), 4.03 (3 H, s), 2.33 (3 H, s), 1.33 (3 H, t), 4.27 (2 H, q), 6.40-7.70 (10 H, m); 6'f (or 6f), NMR (CDCl₃) δ 5.90 (1 H, s), 3.59 (3 H, s), 2.57 (3 H, s), 1.36 (3 H, t), 4.42 (2 H, q), 6.40-7.70 (10 H, m).

6d and 6'd were obtained by the same procedure. For compounds 6e and 6'e the reaction was carried out in the mixture THF-Me₂SO (4:1) at room temperature. For 6b or 6'b the reaction was run in boiling benzene (2 hr). All these compounds were recrystallized in methanol except for 6e or 6'e (acetone).

Demethoxycarbonylation of Azetidines 4. Piperidine (1 ml) was added to 1 g of azetidine 4 in 20 ml of anhydrous toluene or xylene and the mixture was usually refluxed for 24 hr (the reaction was followed by NMR). When 4 had completely disappeared, the solvent and excess of piperidine were distilled under reduced pressure (3 mm)

- I. Demethoxycarbonylation of 4a. 4a gave a quantitative yield of 7a + 7'a (90:10) after 140 hr of reaction in boiling xylene. The mixture 7a + 7'a was purified by distillation under vacuum, bp 150-155° (0.015 mm). Addition of ethanol yielded 7a (30%), mp
- II. Demethoxycarbonylation of 4b-e. The crude reaction mixtures were analyzed by NMR. Compounds 7 were isolated and purified: 7b, mp 102° (ether), 74% yield; 7c, mp 60° (ether-petroleum ether), 58% yield; 7d, mp 118° (éthanol), 74% yield; 7e, mp 192° (ether-petroleum ether), 68% yield.
 - III. Demethoxycarbonylation of 4'd. Piperidine (0.2 ml) was

added to the solution of 0.1 g of 4'd in 10 ml of anhydrous toluene. After 8 hr of reflux, the NMR indicated that 52% of 7d was formed and that the remaining 48% was 4d (and not the starting 4'd). This shows that 4'd is isomerized to 4d in the reaction. After 24 hr the reaction was complete and gave the same mixture as 4d.

IV, Evidence for Kinetic Control of the Reaction, A. Reaction of 7a-c and 7e with Piperidine. Piperidine (0.1 g) was added to the solution of 0.1 g of azetidine in 10 ml of anhydrous toluene. After distillation of toluene in vacuo, the starting azetidine was recovered without any change.

B. Preparation of N-Deuteriopiperidine. Piperidine (10 ml) distilled over KOH was mixed with 10 ml of D2O (98%). After standing for 0.5 hr under nitrogen the piperidine was extracted with ether (saturated with D2O). The ether layer was dried over anhydrous K₂CO₃ and then distilled at atmospheric pressure, giving 8 ml of piperidine deuterated to the extent of 65% (NMR).

Piperidine N-deuterated to 95% was obtained in the following way. In a two-neck flask fitted with a dropping funnel and a Dean-Stark trap, 50 ml of anhydrous toluene, 1 g of N-deuteriopiperidine (65%), and 1 g of D₂O (99.5%) were introduced under nitrogen. The mixture was heated to reflux and water was eliminated by azeotropic distillation with toluene and then the compound to be demethoxycarbonylated was added in toluene solution.

- C. Reaction of 4b with N-Deuteriopiperidine. 4b (1 g) in toluene solution (10 ml) was added under nitrogen to 1 g of piperidine-1-d in toluene (10 ml). After 24 hr of reflux, distillation under reduced pressure gave an oily residue which crystallized by addition of hot methanol. 7b deuterated on C₂ to the extent of 95% (mp 102°, 70% yield) was obtained.
- D. Reaction of 7d with N-Deuteriopiperidine. 7d (1 g) was treated with 1 g of piperidine-1-d (65%) in boiling toluene for 24 hr. Distillation of toluene and excess of piperidine in vacuo gave 7d deuterated on C3. 7d was recrystallized from boiling methanol. The NMR showed that it was a mixture of deuterated and nondeuterated compounds in equivalent amount.

Registry No.—1a, 34671-09-5; 1b, 51597-29-6; 1c, 49790-76-3; 2a, 6814-64-8; 2b, 18915-90-7; 2c, 7380-81-6; 2d, 5633-34-1; 2e, 55800-52-7; 2f, 16980-47-5; 2g, 55800-53-8; 3a, 5367-24-8; 3d, 20718-17-6; 4a, 43113-13-9; 4b, 43113-14-0; 4c, 43113-16-2; 4d, 55800-57-2; 4e, 5580-54-9; 4f, 55800-55-0; 4g, 55800-56-1; 4'b, 43113-15-1; 4'c, 43113-17-3; 4'd, 55800-58-3; 4'f, 55800-59-4; 4'g, 55800-60-7; 5, 4311-18-4; 6b, 55869-69-7; 6d, 55869-70-0; 6e, 55800-61-8; 6f, 55800-62-9; 6'b, 55869-71-1; 6'd, 55869-72-2; 6'e, 55800-63-0; 6'f, 55869-73-3; 7a, 55800-64-1; 7b, 55800-65-2; 7b deuterated on C2, 55869-74-4; 7c, 55800-66-3; 7d, 55800-67-4; 7d deuterated on C₃, 55869-75-5; 7e, 55800-68-5; 7'a, 55800-69-6; 7'e, 55800-70-9; piperidine, 110-89-4; *N*-deuteriopiperidine, 694-58-6.

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