on the base of ir and nmr data and gas chromatographic analysis as originally assumed by Winstein and Lucas. cis- and trans-2.3epoxybutane, 4 and 5, each gave only one peak on gas chromatography on a 6-ft 15% Carbowax 20M on Gas-Chrom R column. A mixture of the two compounds was easily separated at 65°, the compounds having retention times of 4.8 and 3.6 min, respectively.

cis-2,3-Epoxybutane (4) had nmr $X_3AA'X_3'$ system; $H_A =$ 2.60-2.94 (two overlapping octets); $H_X = 1.19$ (multiplet with predominating doublet); $J_{AX} = 5.4$ Hz (first-order analysis).²⁴ trans-2,3-Epoxybutane (5) had nmr X₂AA'X₃' system; $H_A =$

2.32-2.66 (multiplet); $H_x = 1.21$ (doublet); $J_{Ax} = 4.5$ Hz (first-order analysis).

General Procedure for Reaction of Epoxides with Nitriles Similar to Procedure of Oda and Coworkers.¹-A 30-ml portion of nitrile (distilled over phosphorus pentoxide) was added to a round-bottom flask and cooled in an ice bath; 15 ml of concentrated sulfuric acid was added slowly with stirring. A mixture of ca. 0.15 mol of the epoxide in 30 ml of the nitrile was added through the reflux condenser over a period of 1 hr. The mixture was then stirred for a period of 3 hr with the ice bath being allowed to melt at its own rate and then poured into 100 ml of ice water. This mixture was then extracted three times with 100 ml of ether and the ether was discarded. The aqueous phase was then neutralized with concentrated NaOH and filtered. Next it was made strongly basic with NaOH and extracted three times with 100 ml of ether. The three ether fractions were combined, dried over anhydrous magnesium sulfate, filtered, and distilled. The oxazoline was isolated by distillation through a short Vigreux column.

A number of attempts were made to seek conditions to improve the yield. These include the use of 60% perchloric acid, trifluoro-acetic acid, and *p*-toluenesulfonic acid in place of the concentrated sulfuric acid, as well as no acid at all. All attempts, in-cluding the use of inverse addition, did not produce better yields.

2,4- and 2,5-Dimethyl-2-oxazolines (2a and 3a).-The general procedure was followed using propylene oxide and acetonitrile.

(24) Compare ref 10, p 224.

The mixture boiling at 112-114° was collected and separated by gas chromatography using a 6-ft column of 15% Carbowax 20M on Gas-Chrom R. Quantitative analysis showed the two isomers 2a and 3a to be in 70:30 proportion. The melting point of the picrate of the 2,4 isomer 2a was 130° (lit.² mp 130-131°).

trans-2,4,5-Trimethyl-2-oxazoline (6a).-The general procedure was followed using 12.0 g (0.166 mol) of cis-2,3-epoxybutane (4). The reaction yielded 1.90 g (10%) of trans-2,4,5-trimethyl-2-oxazoline (6a), bp 115-116°, mp of picrate 152-153°. trans-4,5-Dimethyl-2-phenyl-2-oxazoline (6b).—The general

procedure was followed using 12.0 g (0.166 mol) of the cis epoxide 4 and benzonitrile as solvent. The reaction yielded 0.95 g (5%)of trans-4,5-dimethyl-2-phenyl-2-oxazoline, bp 116-118° (11 mm), mp of picrate 133-134°. Anal. Calcd for $C_{17}H_{16}N_4O_8$: C, 50.5; H, 3.99; N, 13.86. Found: C, 50.40; H, 4.18; N, 13.68.

cis-2.4.5-Trimethyl-2-oxazoline (7a).-This compound was prepared according to the general procedure using 12.0 g (0.166 mol)of trans-2,3-epoxybutane (5).

Distillation of the product yielded 3.30 g (17%) of cis-2,4,5-trimethyl-2-oxazone (7a), bp 120-122°, mp of picrate 136-137°. Anal. Calcd for $C_{12}H_{14}N_4O_8$: C, 42.11; H, 4.14; N, 16.37. Found: C, 42.19; H, 4.31; N, 16.29.

cis-4,5-Dimethyl-2-phenyl-2-oxazoline (7b).--The general procedure was followed using 12.0 g (0.166 mol) of trans-2,3-epoxy-butane (5) and benzonitrile as solvent. The reaction yielded 0.60 g (3%) of cis-4,5-dimethyl-2-phenyl-2-oxazoline (7b), bp 142-144° (29 mm), mp of picrate 205-207°

Registry No.-2a, 6159-23-5; 3a, 6159-22-4; 4, 1758-33-4; 5, 21490-63-1; 6a, 23336-75-6; 6a picrate, 38898-94-1; 6b, 38898-95-2; 6b picrate, 38898-96-3; 7a, 23236-41-1; 7a picrate, 38898-98-5; 7b, 36746-57-3; 7b picrate, 38899-00-2; propylene oxide, 75-56-9.

Acknowledgments.—The authors wish to thank the Rutgers Research Council for financial support.

2.3-Dimethylcyclopropanecarboxylic Acids from 2,3-Dimethyloxiranes via the Wittig Reaction. Stereochemistry and Mechanism

ROBERT A. IZYDORE AND ROBERT G. GHIRARDELLI*

Paul M. Gross Chemical Laboratory, Duke University, and the U. S. Army Research Office-Durham, Durham, North Carolina 27706

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Triethyl phosphonoacetate anion reacted with (+)-(2R,3R)-2,3-dimethyloxirane to give predominantly (+)-(2S,3S)-2,3-dimethylcyclopropanecarboxylic acid and with cis-2,3-dimethyloxirane to give predominantly cis-2,3-dimethylcyclopropane-trans-carboxylic acid. Inversion of configuration must have occurred at both carbon atoms to account for these products. In each case minor amounts of stereoisomeric acids were produced. The results are discussed in terms of the overall mechanistic scheme.

The reaction of Wittig type reagents with epoxides to form cyclopropanecarboxylic acid derivatives has been well documented. Both carboethoxymethylenephosphoranes^{1,2} and phosphonate anions³⁻⁷ have been successfully utilized. Although certain aspects of the reaction pathway are well understood, there remains some disagreement concerning the overall mechanistic scheme.

(1) D. B. Denney, J. J. Vill, and M. J. Boskin, J. Amer. Chem. Soc., 84, 3944 (1962).

(2) W. E. McEwen, A. Bladé-Font, and C. A. Vander Werf, J. Amer. Chem. Soc., 84, 677 (1962).

(3) W. S. Wadsworth, Jr., and W. D. Emmons, J. Amer. Chem. Soc., 83, 1733 (1961).

(4) I. Tomoskozi, Tetrahedron, 19, 1969 (1963).

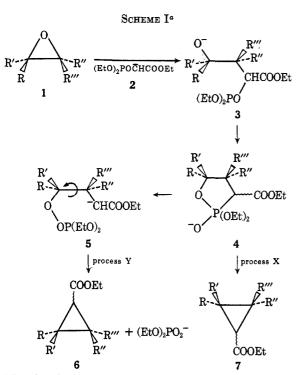
(5) I. Tomoskozi, Tetrahedron, 22, 179 (1966).
(6) I. Tomoskozi, Chem. Ind. (London), 689 (1965).

(7) N. C. Deno, W. E. Billips, D. LaVietes, P. C. Scholl, and S. Schneider, J. Amer. Chem. Soc., 92, 3700 (1970).

Denney¹ postulated a stepwise decomposition of the intermediate 4 (process Y in Scheme I) to give 6 via an intramolecular SN2 displacement. This proposal was based on the observation that carboethoxymethylenetriphenylphosphorane reacted at 200° with cyclohexene oxide to form ethyl 7-norcaranecarboxylate and with optically active styrene oxide to form optically active trans-2-phenylcyclopropanecarboxylate. Denney's inversion mechanism has been supported by Tomoskozi⁴ and Walborsky,⁸ who established the absolute configuration of optically active *trans-2*phenylcyclopropanecarboxylic acid.

In addition to the inversion mechanism the possibility of a competitive direct collapse of 4 (process X) either through a concerted process or through a zwit-

(8) Y. Inouye, T. Sugita, and H. M. Walborsky, Tetrahedron, 20, 1695 (1964).



^a The phosphonate anion 2 is depicted here since it was used in this investigation. The carboethoxymethylenephosphoranes presumably react in an analogous manner.

terion intermediate to yield 7 has been suggested. $^{4,8-10}$ Walborsky argued for the occurrence of the competitive direct collapse based on the low optical yields of trans-2-phenylcyclopropanecarboxylic acid that had been reported by other workers.⁸

In order to clarify the overall mechanistic scheme. the reaction of triethyl phosphonoacetate anion (2) with optically active trans-2,3-dimethyloxirane and with cis-2,3-dimethyloxirane was investigated. The use of these two epoxides was advantageous in that the product ratios of *trans*- to *cis*-2,3-dimethylcyclopropanecarboxylic acids that were formed could be directly related to the extent of occurrence of each of the two competing processes.

Results

Triethyl phosphonoacetate anion (2) reacted slowly with optically pure (+)-(2R,3R)-2,3-dimethyloxirane (8) to give after saponification a 16% yield of an optically active mixture of the isomeric 2,3-dimethylcyclopropanecarboxylic acids. Heating for 8 days at reflux was required. Also formed but not characterized was a considerable quantity of polymeric products which arose from self-condensation of 2 under the reaction conditions. Similar results were obtained with inactive 8.

Assignment of the 2S,3S configuration to the major component 9a was based on the high optical rotation¹¹ of the product mixture, $[\alpha]^{22}D + 19.5^{\circ}$, corrected to $+21.3^{\circ}$ for the presence of the minor components. Structures of the cis-2,3-dimethyl-cis-cyclopropanecarboxylic acid 10 and the cis, trans isomer 11 were

(9) S. Trippett, Quart. Rev., Chem. Soc., 17, 406 (1963).

(10) A. Maercker, Org. React., 14, 387 (1965).
(11) J. M. Walbrick, J. W. Wilson, Jr., and W. M. Jones, J. Amer. Chem. Soc., 90, 2895 (1968), prepared the enantiomer of 9a, $[\alpha]^{25}D = 10.0^{\circ}$, by resolution of the inactive acid 9

assigned by comparing their gas chromatographic retention times with those of authentic samples whose preparation and characterization are discussed below.

The anion 2 reacted with cis-2,3-dimethyloxirane (12) under milder conditions (24 hr at room temperature) to give after saponification an 18% yield of an isomeric mixture of 2,3-dimethylcyclopropanecarboxylic acids and only a small amount of polymeric products. Heating the reaction mixture for 48 hr at reflux increased the yield to 21%.

On standing the major component partially separated from the product mixture as a white solid, mp 79-80.5°. Spectroscopic methods proved less than diagnostic in establishing its structure, but a complete single-crystal X-ray analysis showed its stereochemistry to be cis, trans.¹² The racemic isomer 9 was identified by assuming its gas chromatographic retention time to be equal to that of 9a. The allcis acid 10 could not be readily isolated, but its structure was inferred from the following evidence. A fraction, bp 90.5-91.5° (7 mm), which contained 8%10, 87% 11, and 5% 9 correctly analyzed for $C_{6}H_{10}O_{2}$. Its proton magnetic resonance spectrum was similar to that of pure 11 and did not contain any signals in the vinylic region. This ruled out the possibility of any ring-opened product. The isomeric mixture was converted to its ethyl ester, and then treated with sodium ethoxide in ethanol for 43 hr followed by hydrolysis. Gas chromatographic analysis of the resulting mixture showed the presence of 9 and 11 and only a trace of 10. No additional components were evident. In the proton magnetic resonance spectrum the methyl doublet of 10 appeared 5 Hz upfield from that of 11. These results, from at least three independent experiments in each case, are summarized in Table I.

TABLE I PER CENT COMPOSITION OF PRODUCT MIXTURES FROM REACTION OF OXIDES WITH TRIETHYL PHOSPHONOACETATE ANION

	-2,3-Dimethylcyclopropanecarboxylic acid-			
2,3-Dimethyl-			cis, cis	cis,trans
oxirane	(+) -9a	(±) -9	10	11
2R, 3R (8)	93		6	1
cis (12)		4	6	90

Discussion

The forementioned results are consistent with Denney's inversion mechanism, since the major product formed in each case requires that its epoxide precursor undergo an even number of inversions. The synthesis of 9a from 8 and 2 as described herein is superior to the previously reported preparation¹¹ in that the overall yield is comparable, the tedious process of resolution via diastereomers is eliminated, the optical purity is higher, and the absolute configuration is known.

Convincing evidence that the direct collapse of 4 is competing to some extent was the identification of the minor components formed in each of the two reactions. These are the expected products when the starting epoxides undergo ony one inversion, most likely to have occurred in the opening of the oxide ring. The virtually complete reversal of the relative proportions of 10 to 11 produced in each of the two

(12) A. T. McPhail and P. A. Luhan, J. Chem. Soc., Perkin Trans. 2, 2372 (1972).

reactions would appear to rule out the existence of an intermediate common to the production of cis acids in each as well as ruling out their origin in contamination of the active oxide by the cis isomer. It is thus probable that the 10 and 11 produced from active oxide 8 and the (\pm) -9 from the cis oxide 12 arose through a concerted collapse of the corresponding phosphonate esters 4.¹³

Two factors must be considered in explaining the ratio of 10 to 11 produced *via* Scheme II: the relative amounts of 13a and 13b produced, and the relative probabilities of processes X (collapse) and Y (phosphonate opening, then rear-side attack) for each.

Given the results obtained we must conclude either that 13a is produced in substantially greater amount than 13b, or that the portion which follows path X (X/Y ratio) is substantially greater for the former than for the latter. The ratio of 10 to 11 produced from the cis oxide undoubtedly reflects steric control after the phosphonate ring is opened.

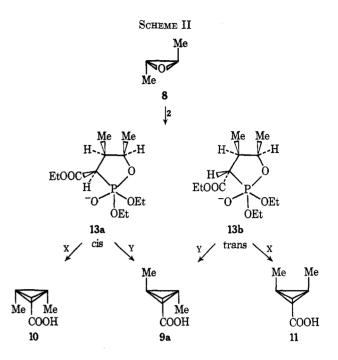
Finally, mention should be made of the low reactivity of 8 and 12 with the phosphonate anion 2. This can be attributed to a steric effect, particularly in view of the reported high yields and facile reactions of 2 with other epoxides.^{3,4,7} We have observed that 8 also reacts with other nucleophiles with difficulty. Despite this shortcoming, 8 has the possibility of being a useful precursor for the synthesis of other types of optically active compounds. We are currently investigating this possibility.

Experimental Section¹⁴

General Procedure for the Reaction of Triethyl Phosphonoacetate Anion (2) with 2,3-Dimethyloxirane.-To a stirred mixture of 9.65 g (0.23 mol) of 57% NaH in mineral oil and 25 ml of dry diglyme was added dropwise a solution of 44.8 g (0.20 mol) of triethyl phosphonoacetate in 25 ml of diglyme. After the evolution of hydrogen had ceased, a solution of 18.0 g (0.25 mol) of 2,3-dimethyloxirane was added. The mixture was stirred at the appropriate temperature. Upon completion of the reaction, a solution of 30 g of NaOH in 50 ml of water was added dropwise The mixture was heated at reflux for 22 hr, cooled with cooling. to room temperature, and diluted with 200 ml of water. The solution was washed with three 150-ml portions of ether, acidified with 50% H₂SO₄, and washed with four 75-ml portions of chloroform. The chloroform washings were dried (Na_2SO_4) and evaporated under reduced pressure to give a liquid residue. The residue was distilled to give a fraction, bp 75-95°, which was dissolved in 40 ml of 10% Na₂CO₃ and washed with four 25-ml portions of chloroform. The carbonate solution was acidified with 50% H_2SO_4 and washed with four 20-ml portions of chloroform. The later chloroform washings were dried (Na₂SO₄) and evaporated under reduced pressure to give a liquid mixture of 2,3-dimethylcyclopropanecarboxylic acids. The mixture could be further purified by distillation to give an analytical sample.

Reaction of 2 with (+)-(2R,3R)-2,3-Dimethyloxirane (8). The reaction of 0.185 mol of 2 with 18.0 g (0.25 mol) of 8^{15,16}

(15) H. J. Lucas and H. K. Garner, J. Amer. Chem. Soc., 70, 990 (1948).
 (16) S. Winstein and H. J. Lucas, J. Amer. Chem. Soc., 61, 1576 (1939).



was carried out by heating at reflux over an 8-day period to give 3.65 g (16%) of an isomeric liquid mixture of 2,3-dimethylcyclopropanecarboxylic acids. Also isolated as the residue from the distillation was a considerable quantity of polymeric material which arose from self-condensation of 2. This was verified by a separate experiment in which 2 was heated in the absence of 8 to give the same polymeric material. The isomeric mixture of 2,3-dimethylcyclopropanecarboxylic acids was distilled under reduced pressure to give an analytical sample: bp 84-87° (3.6 mm); $[\alpha]^{22}$ D +19.5° (c 0.2002, 95% ethanol); ir (neat) 2.9-4.3 (broad, -OH), 5.95 (C=O), 7.70, 8.14, 9.20, 9.37, and 10.7 μ (broad); nmr (CDCl₃) δ 12.06 (s, 1, CO₂H), 1.18 (d, J = 5.8 Hz), and 1.5-0.9 (m, 9).

Anal. Calcd for C₆H₁₀O₂: C, 63.14; H, 8.83. Found: C, 62.92; H, 8.80.

Vpc analysis of the cyclopropanecarboxylic acid mixture at 175° showed the presence of three components in the ratio of 6:93:1 in the order of increasing retention time. The major component was assigned as (+)-(2S,3S)-trans-2,3-dimethylcyclopropanecarboxylic acid (9a) on the basis of the high optical rotation of the mixture.¹¹ The two minor components were identified as cis-2,3-dimethyl-cis-cyclopropanecarboxylic acid (10) and cis-2,3-dimethylcyclopropane-trans-carboxylic acid (11) on the basis of their retention times.

Reaction of 2 with inactive 8 under the same reaction conditions gave similar results.

Reaction of 2 with cis-2,3-Dimethyloxirane (12).—From the reaction of 0.185 mol of 2 with 14.7 g (0.20 mol) of 12¹⁶ at room temperature for 24 hr was obtained 3.8 g (18%) of an isomeric liquid mixture of 2,3-dimethylcyclopropaneearboxylic acids. Heating the reaction mixture at reflux for 38 hr increased the yields to 21%. Distillation of the isomeric mixture under reduced pressure gave an analytical sample: bp 88.5–94.0° (7 mm); ir (neat) 2.9–4.3 (broad, -OH), 5.95 (C=O), 7.65, 7.70, 7.80, 8.14, 9.25, and 10.6 μ (broad); nmr (CDCl₃) δ 11.62 (s, 1, CO₂H), 1.12 (d, J = 5.0 Hz), 1.03 (d, J = 5.0 Hz), and 1.7–0.7 (m, 9).

Anal. Calcd for $C_6H_{10}O_2$: C, 63.14; H, 8.83. Found: C, 63.00; H, 8.80.

Vpc analysis of the mixture at 175° showed the presence of three components in the ratio of 6:4:90 in order of increasing retention time. On standing, the major component partially crystallized from the mixture as a white solid: mp 79-80.5°; ir (Nujol) 2.9-4.3 (broad, -OH), 5.95 (C=O), 7.65, 7.70, 8.14, 9.25, and 10.6 μ (broad); nmr (CDCl₃) δ 11.75 (s, 1, CO₂H), 1.12 (d, J = 5.0 Hz), 1.8-0.9 (m, 9).

A complete single-crystal X-ray analysis determined its configuration as $11.^{12}$ The minor component 9 (4% of mixture) was assumed to display the same vpc retention time as the active isomer. The minor component 10 (6% of mixture) was identified on the basis of the spectral, vpc, and elemental analysis data.

⁽¹³⁾ A zwitterionic intermediate has been proposed⁸ for the case where the cationic center is benzylic, but would be less likely in the present case.

⁽¹⁴⁾ Melting points and boiling points are uncorrected. Infrared spectra were obtained with a Beckman Model 137 infrared spectrophotometer. Nuclear magnetic resonance spectra were recorded on a Varian A-60 using deuteriochloroform as a solvent and tetramethylsilane as an internal reference. Optical rotations were measured in a 0.1-dm cell with a Jasco Model ORD/UV5 recording spectropolarimeter. Concentrations are given in g/ml. Analytical gas-liquid partition chromatography was performed on a Hewlett-Packard Model 700 gas chromatograph using a 6 ft \times 0.125 in. column containing 10% Carbowax 20M on 80-100 mesh Chromosorb W, acid washed and DMCS treated. Peak areas were obtained with a disk integrator. Elemental analyses were performed by Atlantic Microlab, Inc., Atlanta, Ga.

(Dialkylamino)methyloxosulfonium Methylides

Preparation of Ethyl 2,3-Dimethylcyclopropanecarboxylates (Mixed Isomers) (14).—To 2.6 g (0.022 mol) of thionyl chloride was added dropwise a solution of 2.0 g (0.017 mol) of a mixture of 2,3-dimethylcyclopropanecarboxylic acids consisting of 4% 9, 6% 10, and 90% 11 in 5 ml of benzene. The reaction mixture was stirred at room temperature for 90 min and then heated at reflux for 90 min. After cooling to room temperature, 10 ml of absolute ethanol was added dropwise. The solution was evaporated under reduced pressure to give a liquid residue. Distillation gave 1.60 g (67%) of isomeric ethyl 2,3-dimethylcyclopropanecarboxylate (14): bp 53–56° (7 mm); ir (neat) 5.82 (C=O), 7.65, and 8.50 μ (COC); nmr (CDCl₃) δ 4.03 (m, 2, OCH₂CH₃, J = 7 Hz), 1.7–0.8 (m, 12).

Anal. Calcd for C₈H₁₄O₂: C, 67.57; H, 9.92. Found: C, 67.37; H, 9.99.

A solution containing 1.35 g of 14 and 0.015 mol of sodium ethoxide in 40 ml of absolute ethanol was heated at reflux for 43 hr. A solution of 10 g of NaOH in 15 ml of water was added dropwise, and the reflux was continued for 4 hr. The ethanol was removed under reduced pressure and the remaining aqueous solution was washed with three 15-ml portions of chloroform, acidified with 50% H₂SO₄, and washed with three additional 15ml portions of chloroform. The latter chloroform washings were dried (Na₂SO₄) and evaporated under reduced pressure to give 1.0 g of 2,3-dimethylcyclopropanecarboxylic acids. Vpc analysis showed the presence of 9 and 11 and only a trace of 10. No additional component was evident.

Registry No.—2, 38868-10-9; 8, 1758-32-3; (±)-9, 20431-63-4; 9a, 20431-72-5; 10, 34669-52-8; 11, 34669-51-7; 12, 1758-33-4; 14, 17214-87-8.

Acknowledgment.—The authors are grateful to Dr. A. T. McPhail and Mrs. P. A. Luhan for the X-ray crystallographic analysis.

Preparation and Applications of (Dialkylamino)methyloxosulfonium Methylides. Synthesis of Cyclopropanes and Oxiranes^{1a}

CARL R. JOHNSON* AND PETER E. ROGERS^{1b}

Department of Chemistry, Wayne State University, Detroit, Michigan 48202

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Dimethylsulfoximine, prepared from dimethyl sulfoxide, was dialkylated to give (N,N-dimethylamino)- and (N,N-diethylamino)dimethyloxosulfonium fluoroborate. Reaction of these salts with sodium hydride in a variety of aprotic solvents gave methylides. These ylides are effective as nucleophilic methylene transfer reagents; reactions with electrophilic alkenes yield cyclopropanes, while aldehydes and ketones react to give oxiranes.

In the past decade the chemistry of sulfur ylides has been an area of substantial interest.² The dimethylsulfonium and dimethyloxosulfonium methylides introduced by Corey and Chaykovsky are very useful synthetic reagents.³ These ylides have been used to transfer a methylene group in a stepwise fashion across the double bond of a carbonyl or an electrophilic olefin to yield an epoxide or a cyclopropane, respectively. The transfer of more complex groups has also been achieved.⁴

The observation in this laboratory that ylides derived from (dimethylamino)alkylaryloxosulfonium fluoroborates⁵ were capable of transferring alkylidene groups prompted us to undertake a study of the preparation and chemistry of an ylide derived from dimethylsulfoximine. This ylide would be accessible to the synthetic organic chemist and could serve as a model for ylides derived from other symmetrical dialkyl sulfoximines.

The first goal of this work was to prepare an ylide from dimethyl sulfoxide (DMSO) in as few steps as possible. Dimethyl sulfoximine (1) could be pre-

(1) (a) Part XXXIX in the series "Chemistry of Sulfoxides and Related Compounds." We gratefully acknowledge support by the National Science Foundation (GP 19623). (b) National Science Foundation Graduate Trainee, 1968-1971.

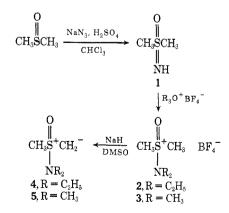
(2) A. W. Johnson, "Ylid Chemistry," Academic Press, London and New York, 1966.

(3) (a) E. J. Corey and M. Chaykovsky, J. Amer. Chem. Soc., 84, 867
(1962); (b) ibid., 3782 (1962); (c) ibid., 86, 1640 (1964); (d) ibid., 87, 1353
(1965); (e) H. Konig, Fortschr. Chem. Forsch., 9, 487 (1968).

(4) (a) E. J. Corey and W. Oppolzer, J. Amer. Chem. Soc., 86, 1899 (1964);
(b) E. J. Corey, M. Jautelat, and W. Oppolzer, Tetrahedron Lett., 2325 (1967);
(c) G. B. Payne, J. Org. Chem., 32, 3351 (1967);
(d) ibid., 33, 1284 (1968);
(e) G. B. Payne and M. R. Johnson, ibid., 33, 1285 (1968);
(f) G. B.

(5) (a) C. R. Johnson, E. R. Janiga, and M. Haake, J. Amer. Chem. Soc.,
 90, 3890 (1968); (b) C. R. Johnson, M. Haake, and C. W. Schroeck, *ibid.*,
 92, 6594 (1970).

pared in 85% yield from DMSO using 1.1 equiv of hydrazoic acid, generated in a chloroform slurry from sodium azide and sulfuric acid.⁶ The N,N-diethyl salt (2)⁷ was chosen as the model ylide precursor. The choice of the N,N-diethyl derivative was prompted by the fact that triethyloxonium fluoroborate, the alkylating agent of choice, requires one less step in its preparation than trimethyloxonium fluoroborate. The dialkylation of the crude sulfoximine was accomplished in one flask using excess sodium carbonate as a base to give 2 in 81% yield. A similar procedure gave (dimethylamino)dimethyloxosulfonium fluoroborate (3) in 85% yield. Both salts were stable, white, crystalline solids.



(Diethylamino)methyloxosulfonium methylide (4) was readily prepared by dissolving the salt 2 in DMSO

⁽⁶⁾ H. R. Bentley and J. K. Whitehead, J. Chem. Soc., 2081 (1950).

⁽⁷⁾ H. Schmidbaur and G. Kammel, *Chem. Ber.*, **104**, 3241 (1971), have recently described the preparation of salts **2** and **3** and the corresponding yildes **4** and **5**. Their interest was largely in the preparation and study of spectral properties.