remove most of the alcohol, then water and dilute hydrochloric acid were added. An upper layer separated and crystallized on cooling, mp 71–75°. The combined solids were recrystallized from ethanol, giving 8.0 g, mp 73–75°.

Anal. Calcd for $C_8H_{11}NO_2S$: C, 51.9; H, 6.0; N, 7.6. Found: C, 52.5; H, 6.1; N, 7.3.

N-Methyl-N-phenylsulfamoylacetic Acid (14).—A solution of crude **12** in excess alcoholic potassium hydroxide was kept at room temperature for 3 days. Some inorganic solid which had separated was filtered, and the filtrate was concentrated *in vacuo*. On addition of dilute hydrochloric acid to the residue, an oil separated which was extracted with methylene chloride. The solution was dried and the solvent was removed, leaving a semisolid. This was rinsed with ether and ethyl acetate, after which an ivory solid remained, mp 120–122°.

Anal. Calcd for $C_9H_{11}NO_4S$: C, 47.15; H, 4.84; N, 6.11. Found: C, 47.01; H, 4.83; N, 6.11.

Acknowledgment.—We wish to thank Dr. James W. Wilson for his encouragement and many helpful discussions.

Studies on the Preparation and Rearrangements of Allylic Azides[1]

Calvin A. VanderWerf and Victor L. Heasley[2]

Chemistry Department, University of Kansas, Lawrence, Kansas

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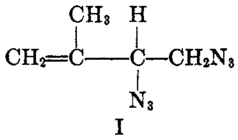
A study of the preparations and rearrangements of the following diazides is reported: 3,4-diazido-1-butene (IV), *trans*-1,4-diazido-2-butene (V), *cis*-1,4-diazido-2-butene (VI), 3,4-diazido-2,3-dimethyl-1-butene (VII), and *trans*-1,4-diazido-2,3-dimethyl-2-butene (VIII). *trans*-1,4-Diazido-2-butene (V) rearranges more slowly than crotyl azide although the former has two potentially mobile groups. 3,4-Diazido-1-butene (IV) rearranges faster than *trans*-1,4-diazido-2-butene (V), and slower than α-methylallyl azide. The decreased rates of rearrangement of IV and V may result from the electron-withdrawing effect of the azide group on the transition state. *cis*-1,4-Diazido-2-butene (VI) rearranges slower than the *trans* isomer. This may result from greater steric hindrance in the transition state of the *cis* isomer. The rate of rearrangement of 3,4-diazido-2,3-dimethyl-1-butene (VII), a tertiary azide, is slower than that of *trans*-1,4-diazido-2,3-dimethyl-2-butene (VIII), a primary azide; in all allylic azide systems that have been studied, the secondary and tertiary azides rearrange more rapidly than the primary ones. Steric interactions between the methyl groups in VII may hinder approach to coplanarity of the double bond and the rearranging azide group in the transition state, and a decreased rate of rearrangement results. The stabilities of the diazides are discussed. The equilibrium mixture of IV and V exploded violently during the course of the investigation. These two compounds are considered to be *extremely* dangerous.

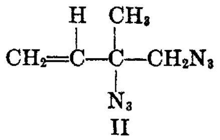
Winstein and co-workers[3] were the first to undertake a study of the rearrangement of allylic azides. They showed that crotyl azide rearranges reversibly to give α-methylallyl azide and that γ,γ-dimethylallyl azide rearranges reversibly to give α,α-dimethylallyl azide. They also demonstrated that the rates of rearrangement increase only slightly with an increase in polarity of the solvent and that the rearrangements exhibit negative entropies of activation. The effect of pressure on the equilibration of α- and γ-methylallyl azide was reported by le Noble.[4] He assumed that the rearrangement occurred through a cyclic transition state.

We became interested in the rearrangements of azides when it was discovered that 1,4-dibromo-2-methyl-2-butene reacts with sodium azide to give 3,4-diazido-2-methyl-1-butene (I) and another diazide suspected of being 3,4-diazido-3-methyl-1-butene (II), in addition to the expected 1,4-diazido-2-methyl-2-butene (III).[5] In an effort to expand the knowledge of azide rearrangements and to study the preparation and the properties of allylic diazides,[6] an investigation of the following azides was undertaken: 3,4-diazido-1-butene (IV), *trans*-1,4-diazido-2-butene (V), *cis*-1,4-diazido-2-butene (VI), 3,4-diazido-2,3-dimethyl-1-butene (VIII), and *trans*-1,4-diazido-2,3-dimethyl-2-butene (VIII).



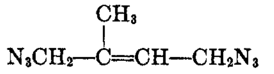
$CH_2{=}C(CH_3){-}CH(N_3){-}CH_2N_3$

I



$CH_2{=}CH{-}C(CH_3)(N_3){-}CH_2N_3$

II

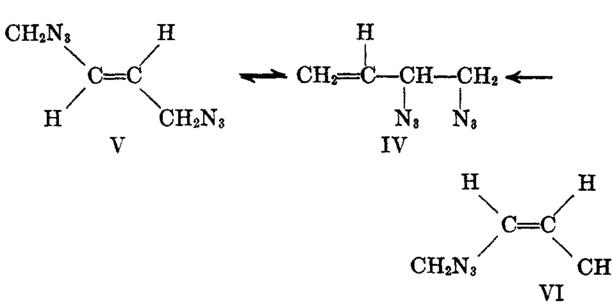


$N_3CH_2{-}C(CH_3){=}CH{-}CH_2N_3$

III

Results and Discussion

The kinetics of the reversible rearrangement of 3,4-diazido-1-butene (IV) to *trans*-1,4-diazido-2-butene (V) were determined by infrared analysis. The rates of



V IV VI

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[1] (a) From the Ph.D. Dissertation of V. L. Heasley; (b) presented in part at the 144th National Meeting of the American Chemical Society, Los Angles, Calif., April 1963; (c) this project was performed as a part of American Petroleum Institute Research Project 52. This work was also supported in part by a research grant from the Petroleum Research Fund of the American Chemical Society (Type C) to C. A. V.

[2] Present address: Department of Chemistry, Pasadena College, Pasadena, Calif.

[3] S. Winstein, A. Gagneux, and W. G. Young, *J. Am. Chem. Soc.*, **82**, 5956 (1960).

[4] W. le Noble, Abstracts, 144th National Meeting of the American Chemical Society, Los Angeles, Calif., April 1963, p 10M.

[5] Studies were not continued on the diazides from 1,4-dibromo-2-methyl-2-butene because of the extreme difficulty involved in an accurate analysis of the product, and because it is still uncertain whether 1,4-dibromo-2-methyl-2-butene has the *cis* or *trans* configuration.

[6] As far as we can determine, this is the first report on the preparation of allylic diazides. By the term "allylic diazides" we refer to those diazides (V, VI, and VIII) in which both azide groups are located on carbon atoms adjacent to a double bond.

rearrangement and the equilibrium concentrations for IV and V are listed in Table I. The infrared spectra of diazides IV and V have, respectively, the following noninterfering absorption bands: 10.68 and 11.38 μ. Vapor phase chromatographic analysis[7] established that no diazide VI was present in the equilibrium mixture of diazides IV and V.

TABLE I

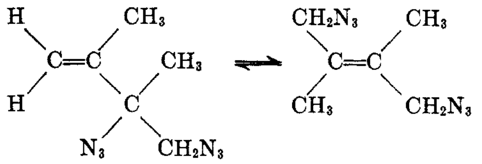
SUMMARY OF THE EQUILIBRIUM CONCENTRATION AND RATES OF REARRANGEMENT OF SOME ALLYLIC AZIDE SYSTEMS IN ACETONE AT 25°

System	Rates of rearrangement, respectively ($k \times 10^5$ sec^{-1})	Equilibrium concn, respectively (%)
α-Methylallyl azide and crotyl azide[a]	7.18,[b] 3.83[b]	34.8, 65.2[c]
α,α-Dimethylallyl azide and γ,γ-dimethylallyl azide[a]	23.9,[b] 7.13[b]	23.0, 77.0[c]
3,4-Diazido-1-butene (IV) and *trans*-1,4-diazido-2-butene (V)	1.2, 0.85[e]	42, 58[d]
3,4-Diazido-2,3-dimethyl-1-butene (VII) and *trans*-1,4-diazido-2,3-dimethyl-2-butene (VII)	2.3, 5.3[g]	70, 30[f]

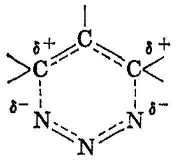
[a] See ref 4. [b] ±2%. [c] ±0.2. [d] Although extreme instability made the analysis of IV and V very difficult, we feel that the "true" values for the equilibrium concentrations lie within the range ±1.5 (see the Experimental Section for a discussion of the determination of the range). These values for the equilibrium concentrations are substantiated by analysis of the diamines resulting from reduction of the equilibrium mixture of IV and V. Also, an independent determination of the equilibrium concentration of IV in carbon tetrachloride gave 42 ± 1.6. An attempt was made to determine the equilibrium concentrations and rate constants by nmr analysis; however, a violent explosion prevented continuation of this work. [e] These rate constants are average values obtained by using the extremes of the equilibrium concentrations mentioned in footnote *d*. [f] The standard deviation is approximately 2. [g] The standard deviation is 0.1.

The kinetics of the rearrangement of *cis*-1,4-diazido-2-butene (VI) were also determined by infrared analysis. The rate constant (2.6×10^{-6} sec^{-1}) for this first-order rearrangement[8] was obtained by plotting the logarithm of the concentration of VI against time. Since the infrared spectrum of VI has a peak at 7.15 μ which is not present in the spectrum of the equilibrium mixture of IV and V, the conclusion can be made that the rearrangement of VI to give IV and V is irreversible. Since VI decomposes rather rapidly, the actual rate of rearrangement is certainly not greater than the calculated value and may be considerably less.

Nmr spectroscopy provided an excellent method for determining the equilibrium concentrations and the rates of rearrangement of 3,4-diazido-2,3-dimethyl-1-butene (VII) and *trans*-1,4-diazido-2,3-dimethyl-2-butene (VIII).[9] The results of the study of the rearrangement of VII and VIII are given in Table I. The nmr spectrum of VIII has a peak at τ 6.17 (methylene hydrogens) and the nmr spectrum of VII has a peak at τ 4.90 (vinyl hydrogens), both of which were used in the calculations of the equilibrium concentrations and the rates of rearrangement.


VII VIII

The data in Table I show that the rates of rearrangement of 3,4-diazido-1-butene (IV) and *trans*-1,4-diazido-2-butene (V) are considerably slower than the rates of rearrangement of α-methylallyl azide and crotyl azide even though V has two azide groups that are situated for rearrangement. The following explanation may account for the decreased rates of rearrangement of IV and V compared to α-methylallyl azide and crotyl azide. Winstein and co-workers[3] have shown that there is a small solvent effect in azide rearrangements which indicates that there is charge formation in the transition state. On the other hand, the small solvent effect and the negative entropy point to covalent bonding in the transition state. Perhaps the following structure describes the transition state. The electron-withdrawing effect[10] of the azide group, not involved in rearrangement, would destabilize this transition state and the rate of rearrangement of IV and V would be decreased.



It seemed possible that *trans*-1,4-diazido-2-butene (V) with its two potentially mobile groups would show a greater rate of rearrangement than crotyl azide. Apparently the destabilizing effect of the additional azide group in V is great enough to offset the effect of having two azide groups present.

Greater steric hindrance in the transition state in the rearrangement of *cis*-1,4-diazido-2-butene (VI) than in the rearrangement of *trans*-1,4-diazido-2-butene (V) may account for the fact that VI rearranges more slowly than V. This result is in agreement with recent studies on the Claisen rearrangement[11] which show that the *trans*-allyl aryl ethers rearrange more rapidly than the *cis* isomers.

Although only the few azides reported in Table I have been studied, it appears that, with the exception of 3,4-diazido-2,3-dimethyl-1-butene (VII), both secondary and tertiary azides rearrange more rapidly than the primary azides, and α,α-dimethylallyl azide (a tertiary azide) rearranges more rapidly than α-methylallyl azide (a secondary azide). One explanation for the slower rate of rearrangement of VII (a tertiary azide) compared to VIII (a primary azide) involves

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[7] The analyses of the diazides were made with a Beckman GC-2 chromatograph on a 6 ft × 0.25 in. diameter column packed with 2.5% SE-30 on 60–80 mesh Chromosorb W at 60°. The helium pressure was 25 psi.

[8] The rearrangement of VI was studied neat. The rate of rearrangement of *trans*-1,4-diazido-2-butene (V) under identical conditions was found to be 0.92×10^{-5} sec^{-1}; the equilibrium constant from the determination in acetone was used here. This seems justifiable inasmuch as the equilibrium concentrations do not change greatly with solvent. (See Table I, footnote *d*.)

[9] The possibility that 3,4-diazido-2,3-dimethyl-1-butene (VII) rearranges to *cis*-1,4-diazido-2,3-dimethyl-2-butene in addition to *trans*-1,4-diazido-2,3-dimethyl-2-butene (VIII) was not eliminated. If this occurs, the rate of rearrangement reported for VIII would be a composite of the rates for VIII and the *cis* isomer. The value for VII, however, would remain unchanged.

[10] For a discussion on the electronic effects of azide groups see P. Smith, J. Hall, and R. Kan [*J. Am. Chem. Soc.*, **84**, 485 (1962)] and J. Boyer and F. Canter [*Chem. Rev.*, **54**, 1 (1954)].

[11] (a) W. N. White and B. E. Norcross, *J. Am. Chem. Soc.*, **83**, 1968 (1961); (b) *ibid.*, **83**, 3265 (1961).

Table II

Preparation of the Equilibrium Mixture of the Allylic Azides

Bromide[a]	Solvent, ml	NaN_3 and H_2O, moles, ml	Azide	Bp, °C (mm)	Yield, %
trans-1,4-Dibromo-2-butene	EtOH, 15 Me_2CO, 15	0.20, 40	IV and V	35–50 (0.5)	88
cis-1,4-Dibromo-2-butene	EtOH, 16 Me_2CO, 16	0.24, 40	VI[b]	. . .	. .
trans-1,4-Dibromo-2,3-dimethyl-2-butene	Me_2CO, 30	0.20, 41	VII and VIII	45–53 (0.1)	86

[a] The quantities of reagents are based on 0.10 mole of bromide. [b] *cis*-1,4-Diazido-2-butene was flash distilled because of suspected instability. The yield was nearly quantitative.

considerations of the increasingly severe steric interactions between the hydrogens of the α- and β-methyl groups in VII as the system proceeds from the ground state to a transition state which is best described as a nearly planar, six-membered ring.[12]

Experimental Section[13]

Discussion on the Rate Calculations.—The rate equation for a reversible, first-order rearrangement,[14] $\ln (A_0 - A_{eq})/(A - A_{eq}) = (k_1 + k_{-1})t$, was used to determine the rate of rearrangement of IV to V. In this expression the concentrations of IV at the start of the reaction, at any time, t and at equilibrium are represented by A_0, A, and A_{eq}, respectively; k_1 and k_{-1} represent the rate of rearrangement of IV and V, respectively.

Since VII contained some VIII as an impurity, it was necessary to modify the above rate equation in order to determine the rearrangement of VII. If the concentrations of VII at the start and at any time (t) are represented by A_0 and A and the concentrations of impurity VIII, and VIII present during the rearrangement are represented by B_0 and B, then the following equation can be written: $A_0 - A + B_0 = B$. From this equation the following rate expression for the rearrangement of VII to VIII can be derived.

$$\ln \frac{(KA_0 - B_0)}{[A(K + 1) - (A_0 + B_0)]} = (K + 1)k_{-1}t$$

In this equation k_{-1} refers to the rate of rearrangement of VIII.

This "modified" equation was also used to determine one of the limits in the rearrangement of IV to V. (See footnote *e* in Table I.) The maximum amount of impurity V in IV was estimated, and the "modified" rate equation, with the correct extreme equilibrium concentrations for IV and V (40.5 and 59.5%, respectively), was used. The other limit was determined by assuming that IV contained no V. Then the rate equation, $\ln (A_0 - A_{eq})/(A - A_{eq}) = (k_1 + k_{-1})t$, was used with the following equilibrium concentrations: IV, 43.5% and V, 56.5%.

Substitution of decreasing diazide concentrations and the corresponding time in the preceding rate equations gave the rate constants.

Preparation of the Equilibrium Mixtures of the Allylic Azides Reported in Table II. General Procedure.—Sodium azide in water was added to the bromide in the appropriate solvent and the reaction mixture was stirred at room temperature for approximately 16 hr. The azide was isolated by pouring the reaction mixture into water, separating the crude azide, extracting the aqueous solution with ether, combining the ether extracts with the crude diazide, removing the solvent, and distilling.

Procedure for the Isolation of *trans*-1,4-Diazido-2-butene (V), *cis*-1,4-Diazido-2-butene (VI), and *trans*-1,4-Diazido-2,3-dimethyl-2-butene (VIII).—These diazides were prepared as described in the general procedure and Table II. Their formation was followed with gas chromatography and when the reaction was completed (*ca.* 0.5 hr), the product was poured into a separatory funnel containing 300 ml of water. The heavy diazide which settled to the bottom was removed, flash distilled, and collected in a flask surrounded by Dry Ice. The diazides were stored at Dry Ice temperatures to prevent rearrangement.

Procedure for the Isolation of 3,4-Diazido-1-butene (IV) and 3,4-Diazido-2,3-dimethyl-1-butene (VII).—It is possible to convert equilibrium mixtures of IV and V and of VII and VIII to 3,4-diazido-1-butene (IV) and 3,4-diazido-2,3-dimethyl-1-butene (VII), respectively, by careful vacuum distillation through a 25-cm column packed with glass helices. The pot temperature was held approximately 20° above the boiling points and the column temperature was maintained, by circulating water of the proper temperature through the jacketed column, so that most of the refluxing diazide was returned to the pot. In this manner the 1,4-diazides can be converted to the vicinal diazides in nearly quantitative yield.

***trans*-1,4-Dibromo-2-butene.**—1,4-Dibromo-2-butene was obtained from Aldrich Chemical Co., Inc. Gas chromatography showed the presence of considerable quantities of 3,4-dibromo-1-butene. Three recrystallizations from 95% ethanol gave pure *trans*-1,4-dibromo-1-butene.

***cis*-1,4-Dibromo-2-butene.**—This compound was prepared according to the procedure of Valette.[15]

***trans*-1,4-Dibromo-2,3-dimethyl-2-butene.**—This dibromide was prepared essentially according to the procedure of Sweeting and Johnson.[16]

Establishment of the Structures and Purities of the Diazides.—Owing to extreme instabilities, the elemental analyses of the diazides were not determined. The structures and purities are based on infrared analyses, nmr analyses, and reduction to amines. Table III contains the principal peaks in the nmr spectra of the dibromides and diazides reported in this study.

The structure of *cis*-1,4-diazido-2-butene (VI) is based on its infrared spectrum which confirms the presence of azide groups, and on its nmr spectrum which is not only nearly identical with that of the starting dibromide, but also contains only methyl and vinyl hydrogens which are different from those of *trans*-1,4-diazido-2-butene (V).

The structures of *trans*-1,4-diazido-2-butene (V) and 3,4-diazido-1-butene (IV) are based on their nmr spectra, and on the fact that reduction with hydrogen and platinum gives 1,4- and 1,2-butanediamines, respectively.[17]

The structures of *trans*-1,4-diazido-2,3-dimethyl-2-butene (VIII) and 3,4-diazido-2,3-dimethyl-1-butene (VII) are based on their nmr spectra. The spectrum of *trans*-1,4-diazido-2,3-dimethyl-2-butene (VIII) is nearly identical with that of the starting dibromide. The structure of VII is confirmed by its nmr spectrum which shows absorptions for the hydrogens of two different methyl groups, methylene hydrogens and vinyl hydrogens. Additional evidence for the molecular structures of VII and VIII was obtained by reduction of the equilibrium mixtures of these diazides with hydrogen and platinum to give

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[12] It has also been determined that the equilibrium concentrations of 3-azido-2,3-dimethyl-1-butene and 1-azido-2,3-dimethyl-2-butene are 72 and 28%, respectively. Conceivably the high equilibrium concentrations of 3-azido-2,3-dimethyl-1-butene (a tertiary azide) results from steric interactions similar to those proposed for VII.

[13] Melting points and boiling points are uncorrected. A Varian Associate A-60 instrument was used to make the nmr spectra. The infrared spectra were recorded with a Perkin-Elmer Model 21.

[14] For a discussion of the derivation of this equation see A. Frost and R. Pearson, "Kinetics and Mechanism," 2nd ed, John Wiley and Sons, Inc., New York, N. Y., 1961, p 186.

[15] A. Valette, *Ann. Chim.*, **3**, 644 (1948).

[16] O. Sweeting and J. Johnson, *J. Am. Chem. Soc.*, **68**, 1057 (1946).

[17] The 1,4- and 1,2-butanediamine obtained from V and IV, respectively, were identified by vapor phase chromatographic analysis on a 9.5 ft × 0.25 in. diameter column packed with 10% Carbowax 5M on Fluoropak 80, using authentic diamines for comparison purposes. An Aerograph 90-P chromatograph was used.

TABLE III

NMR DATA FOR THE DIBROMIDES AND DIAZIDES IN τ UNITS (CARBON TETRACHLORIDE)

Compd	Position			
	CH_3	CH_2	CH	CH=C
cis-1,4-Dibromo-2-butene	. . .	5.94, 6.06	. . .	4.02, 4.12, 4.21
cis-1,4-Diazido-2-butene	. . .	6.08, 6.17	. . .	4.09, 4.17, 4.25
trans-1,4-Dibromo-2-butene	. . .	6.05, 6.15	. . .	4.04[a]
trans-1,4-Diazido-1-butene	. . .	6.15, 6.20 (shoulder)	. . .	4.19[a]
3,4-Diazido-2-butene	. . .	6.69, 6.79	5.83, 5.93, 6.03	*b*
trans-1,4-Dibromo-2,3-dimethyl-2-butene	8.10	6.08	. . .	. . .
trans-1,4-Diazido-2,3-dimethyl-2-butene	8.13	6.17	. . .	. . .
3,4-Diazido-2,3-dimethyl-1-butene	8.18, 8.55	6.71	. . .	4.90

[a] This is the principal peak; other peaks are also present. [b] This terminal vinyl group is very complicated and contains too many peaks to list.

a diamine[18] whose dipicrate derivative gave the correct analysis for $C_{18}H_{22}N_8O_{14}$, mp 197.5°.

Anal. Calcd for $C_{18}H_{22}N_8O_{14}$: C, 37.64; H, 3.86; N, 19.51. Found: C, 37.21; H, 4.14; N, 19.67.

The purities of the diazides were determined to be approximately 95%, with the exception of 3,4-diazido-2,3-dimethyl-1-butene (VII) which contained 10% of VIII.

Stabilities of the Diazides.—Although equilibrium mixtures of IV and V had been distilled many times and were found to be stable to 130°, distillation of a sample for the final experiment of this study resulted in a violent explosion. No explanation can be given for the explosion. These compounds should be considered *extremely* hazardous. The equilibrium mixture of VII and VIII did not explode when heated to 190°. A violent explosion did occur, however, when they were heated in an open flame.

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[18] Vapor phase chromatographic analysis of this diamine product, on the same column as was used for the 1,4- and 1,2-butanediamines, indicated only one compound, whereas the chromatogram of the reduction product of VIII contained two peaks. These results indicate that reduction of the equilibrium mixture of VII and VIII gives pure 2,3-dimethyl-1,2-butanediamine and reduction of VIII gives a mixture of the isomeric diamines. Perhaps the rate of reduction of VIII is slow compared to that of VII, and VIII rearranges to VII which is rapidly reduced.

The Addition of O,O′-Dimethylphosphorylsulfenyl Chloride to Unsaturated Hydrocarbons

W. H. MUELLER, R. M. RUBIN,[1] AND P. E. BUTLER[2]

The Central Basic Research Laboratory, Esso Research and Engineering Company, Linden, New Jersey

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O,O′-Dimethylphosphorylsulfenyl chloride was added to ethylene, propylene, pentene-1, isobutylene, 1,3-butadiene, isoprene, methylacetylene, and allene. The exclusive formation of O,O′-dimethyl S-chloroalkylthiophosphates was observed. Both Markovnikov- and anti-Markovnikov-oriented adducts were obtained from terminal olefins and conjugated dienes. Addition to the dienes produced exclusively 1,2-addition products. Allene and methylacetylene, however, afforded only anti-Markovnikov adducts.

Many O,O,S-trialkyl thiophosphates have been found to be of potential interest as pesticides[3] and, therefore, their chemistry and their preparation *via* displacement reactions[4] have been studied extensively. However, addition reactions to unsaturated hydrocarbons, an equally interesting approach to such compounds, have received little attention. In a previous study we have determined the scope and limitation of the cationic addition of dialkyldithiophosphoric acid to unsaturates.[5] Our present paper is primarily concerned with the adduct orientation obtained from dimethylphosphorylsulfenyl chloride, $(CH_3O)_2P(O)SCl$, and unsaturated hydrocarbons. Michalski and co-workers have reported the first additions of dialkylphosphorylsulfenyl chlorides to terminal olefins.[6,7] A similar addition to allene was described in the patent literature,[8] and most recently the addition of O,O-diethylphosphorylsulfenyl chloride to butadiene has been reported.[9]

Publication of the latter work on butadiene prompts us to report our results in this area. With the aid of nmr spectroscopy we were able to reveal a more complex picture of the adduct orientation. In contrast to the earlier claimed[6] exclusive formation of Markovnikov-oriented products we find little or no selectivity. An exception is the selective anti-Markovnikov addition to allene and methylacetylene.

In this study, O,O′-dimethylphosphorylsulfenyl chloride was chosen as a convenient model reagent for the important nmr analyses of the products. As typical olefin substrates, ethylene, propylene, pentene-1, isobutylene, 1,3-butadiene, isoprene, and allene were selected. Methylacetylene was used as a representative acetylenic compound.

Results

O,O′-Dimethylphosphorylsulfenyl chloride was prepared in high yield (>90%) by treating trimethyl phosphorothionate with 1 mole equiv of sulfuryl chloride.

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[1] Present address: Yale University, New Haven, Conn., summer employee, 1965.

[2] Analytical Research Division, Esso Research and Engineering Co.

[3] G. Schrader, "Die Entwicklung neuer Insektizider Phosphorsäure-Ester," Verlag Chemie GMBH, Weinheim, Germany, 1963.

[4] K. Sasse, "Methoden der Organischen Chemie," Houben-Weyl, Vol. XII/2, E. Müller, Ed., Georg Thieme Verlag, Stuttgart, 1964, pp 652–681.

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[6] B. Borecka, T. Kapecka, and J. Michalski, *Roczniki Chem.*, **36**, 87 (1962).

[7] J. Michalski, B. Pliszka-Krawiecka, and A. Skowronska, *ibid.*, **37**, 1479 (1963).

[8] Y. G. Gololobov, V. V. Semidetko, and L. Z. Soborovskii, Russian Patent 166692 (1964).

[9] A. N. Pudovik and E. A. Ishmaeva, *Zh. Obshch. Khim.*, **35**, 2080 (1965).