The pyrolysis tube was evacuated to remove olefinic products, the white, crystalline residue was triturated with deuteriochloroform, and the filtrate was examined by nmr. The only peaks present were those attributable to the pinacolyl ester. No peaks were observed which could be ascribed to the rearranged tertiary ester, 2,3-dimethyl-2-butyl diphenylphosphinate. Although this analysis is limited by the sensitivity of the method used, it appears to rule out any rearrangement of starting material as a source of rearranged olefin.

Acknowledgment. We thank Mr. Richard Gillespie for much assistance with instrumental problems.

The Structure of the Product Obtained from an Unusual Phosphine Halogenation Reaction[1]

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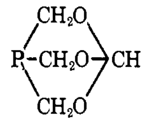
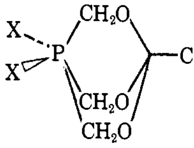
Abstract: Single-crystal X-ray structural analysis of the bromination product of the bicyclic phosphine $P(CH_2O)_3CCH_3$ shows that ring opening has occurred to form $(BrCH_2)_2P(O)CH_2OOCCH_3$. An unusual feature of the structure is that despite the bulkier substituents, the CPC bond angles range from 100 to 104° whereas those in $OP(CH_3)_3$ are 106°. This result is rationalized in terms of the isovalent hybridization hypothesis and the accommodation of the smaller CPC angles by an outward folding of the bromine and acetoxy groups with respect to the P=O axis. Also discussed are the ^1H, ^{31}P, and ^{13}C spectra and the signs and magnitudes of the $^{31}P-^{13}C$ and $^{31}P-^1H$ couplings determined from indor experiments. A plausible mechanism for the reaction is proposed.

Fluorination of trialkylphosphines yields pentacovalent phosphorus derivatives of the type X_2PR_3 in which the fluorines tend to occupy the axial positions of a trigonal bipyramid in the liquid or solution state.[3] Analogous products are probably also formed with chlorine and bromine as shown by their low conductivities in dilute acetonitrile,[4] the strong implication of their presence in the racemization of optically active trialkylphosphines,[5] and the zero dipole moments of X_2AsR_3 systems.[6] Dissociation into XPR_3^+ and X^- ions occurs when X is iodine and evidence has been presented for the presence of XPR_3^+ species in the solid state when X is Cl, Br, and I.[4]

Because the constraint in a bicyclic phosphine such as 1-phospha-4-methyl-3,5,8-trioxabicyclo[2.2.2]octane (I) could give rise to an unusual five-coordinate geometry for phosphorus as shown in II, we have undertaken the halogenation of I under a variety of conditions. We report here the reaction of I with bromine which yields $(BrCH_2)_2P(O)CH_2OOCMe$ (III) in high yield from room temperature to 0°. The formulation of this substance as shown is made definitive by the single-crystal X-ray diffraction study and supported by ^1H, ^{31}P, and ^{13}C nmr experiments.

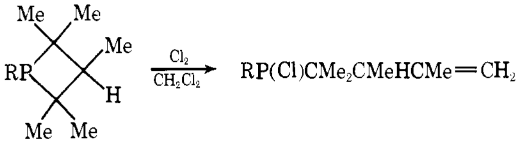


I



II

Recently the reaction of chlorine with phosphetanes in dichloromethane was reported to give chlorophosphine derivatives,[7] an example being



As in our reaction, ring opening is observed but in contrast, a phosphorus–carbon bond is broken, and only one halogen atom is incorporated into the product, while the other apparently removes a proton from a methyl group forming HCl.

Experimental Section

Bis(bromomethyl)acetoxyphosphine Oxide (III). To 10 ml of a 0.5 *M* benzene solution of I[8] kept under a flow of dry nitrogen was slowly added an equimolar amount of bromine dissolved in 20 ml of benzene. The addition was carried out at room temperature and, upon its completion, the clear solution was reduced to half its volume under vacuum. An equivalent volume of pentane was then added and the cloudy solution cooled to 0° for 1 hr. The white crystals collected by filtration were recrystallized from ether in 65% yield (mp 69°).

Anal. Calcd for $C_5H_9O_3Br_2P$: C, 19.49; H, 2.92; P, 10.07; Br, 51.92. Found: C, 19.71; H, 2.87; P, 9.14; Br, 49.76.

X-Ray Diffraction Study. Clear, colorless crystals of III were grown by slow evaporation of a methylene chloride–hexane solution. Microscopic examination revealed that the crystals were acicular with sharply defined faces. Crystals were selected and mounted on glass fibers. Preliminary Weissenberg photographs exhibited $2/m$ Laue symmetry indicating a monoclinic space group. The following systematic absences were observed: $h0l$ when $l = 2n + 1$, and $0k0$ when $k = 2n + 1$. These absences uniquely

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[1] Contribution No. 3007. Work was performed in the Ames Laboratory of the Atomic Energy Commission and the Chemistry Department of Iowa State University.

[2] NSF Trainee.

[3] E. L. Muetterties and R. A. Schunn, *Quart. Rev., Chem. Soc.*, **20**, 245 (1966).

[4] K. Issleib and W. Seidel, *Z. Anorg. Allgem. Chem.*, **288**, 201 (1956).

[5] L. Horner and H. Winkler, *Tetrahedron Lett.*, 455 (1964).

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[7] J. R. Corfield, M. J. P. Harger, R. K. Oram, D. J. H. Smith, and S. Trippett, *Chem. Commun.*, 1350 (1970).

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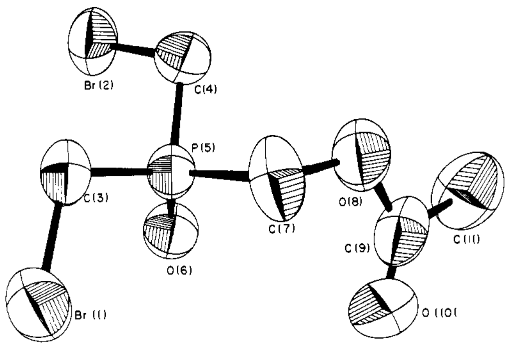

Figure 1. A computer drawing of the final X-ray model of $(BrCH_2)_2P(O)CH_2O_2CCH_3$. The thermal ellipsoids are scaled to enclose 50% probability.

indicate the space group $P2_1/c$. The unit cell parameters are $a = 4.98 \pm 0.01$, $b = 9.25 \pm 0.01$, $c = 22.08 \pm 0.02$ Å, and $\beta = 98.42 \pm 0.08°$ obtained by a least-squares fit to 12 independent reflection angles whose centers were determined by left–right, top–bottom beam splitting on a previously aligned Hilger-Watts four-circle diffractometer (Cu Kα radiation, $\lambda = 1.5418$ Å). Any error in the instrumental zero was eliminated by centering the reflection at both $+2\theta$ and -2θ. A density of 2.01 g/cm^3 was calculated for four molecules per unit cell.

For data collection, a crystal having approximate dimensions of 0.1 mm along each of the crystal axes was used. The crystal was mounted with the *c* axis along the spindle axis. (Approximate cell dimensions had been measured from rotation and Weissenberg photographs taken on a previously mounted crystal with its *b* axis along the spindle axis.) The orientation of the crystal was then modified through a few degrees before commencing to collect data to minimize the occurrence of multiple reflections.

Data were collected at room temperature utilizing a Hilger-Watts four-circle diffractometer equipped with a scintillation counter and using Ni-filtered Cu Kα radiation. Within a 2-θ sphere of 55°, all data in hkl and $\bar{h}kl$ octants were recorded using the stationary-crystal–stationary-counter technique with a take-off angle of 8°. Background counts of 5 sec were taken before and after each scan. Two hundred integrated intensities were collected and used to convert the peak height intensities to integrated values.[9] A total of 1347 reflections were measured in this way.

As a general check on electronic and crystal stability, the intensities of three standard reflections were measured periodically during the data collection period. Since not all of the check reflections declined at the same rate, no correction was made for the 15% reduction in these standard intensities which was detected during data collection.

The absorption coefficient, μ, is 123 cm^{-1}, but no absorption correction was used since it was felt that crystal instability was the limiting factor for accuracy. The standard deviations of the $|F_o|$'s were estimated by the procedure of Stout and Jensen.[10] Of the 1347 reflections, 283 had $F_o^2 < 3.0\sigma(F_o^2)$, and these were considered unobserved and were not used in the refinement.

A three-dimensional Patterson synthesis was computed from sharpened data[11] and unambiguously revealed the positions of the bromine atoms. The remaining atoms were found in the successive bromine-phased electron density syntheses.[12] These positions were then refined by full-matrix least-squares techniques with isotropic thermal parameters to a conventional discrepancy index ($R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$) of 0.174 and a weighted R factor of $\omega R = (\Sigma\omega(|F_o| - |F_c|)^2/\Sigma\omega|F_o|^2)^{1/2} = 0.225$. The scattering factors were those of Hanson, *et al.*[13] A difference electron density map at this stage showed that all the nonhydrogen atoms had been accounted for, but some anisotropic motion, particularly of the heavier atoms, was quite evident. Accordingly, anisotropic refinement was begun and after four cycles the final values of R and ωR of 0.110 and 0.142, respectively, were obtained. A final electron density difference map showed no peaks higher than 0.5 e/Å^3. A final statistical analysis of the F_o and F_c values as a function of the indices, the scattering angle, and the magnitude of F_o showed no unusual trends and suggests that the relative weighting scheme used is a reasonable one. The relatively high R is undoubtedly due to crystal decomposition.

In Table I we give the final values of the positional parameters, along with their standard deviations as derived from the inverse least-squares matrix. Table II gives the values of F_o and F_c in electrons times ten for the 1064 reflections above background.[14] The values of F_c for the unobserved reflections in no case exceeded 3.0σ (F_o). An indication of the directions and root-mean-square amplitudes of vibration for the atoms refined anisotropically is provided by the computer drawing of the final X-ray model in Figure 1. Table III lists selected bond lengths and Table IV lists selected bond angles.

Table I. Final Atomic Positions[a]

Atom	x/a	y/b	z/c
Br(1)	0.7335 (5)	0.3766 (2)	0.0523 (1)
Br(2)	0.7543 (4)	−0.1921 (3)	0.0668 (1)
C(3)	0.857 (3)	0.178 (2)	0.0649 (7)
C(4)	0.890 (2)	−0.071 (1)	0.1376 (8)
P(5)	0.7306 (8)	0.1017 (5)	0.1300 (2)
O(6)	0.439 (2)	0.101 (1)	0.1271 (6)
C(7)	0.920 (3)	0.196 (2)	0.1953 (7)
O(8)	0.880 (2)	0.118 (1)	0.2488 (5)
C(9)	0.671 (4)	0.166 (3)	0.2804 (7)
O(10)	0.550 (3)	0.270 (2)	0.2681 (6)
C(11)	0.634 (5)	0.057 (3)	0.3323 (9)

[a] The estimated standard deviations as given by the inverse least-squares matrix appear in parentheses and apply to the last significant digit in each case.

Table III. Intramolecular Distances[a]

Atoms	Distance, Å	Atoms	Distance, Å
Br(1)–C(3)	1.94 (2)	P(5)–O(6)	1.45 (1)
Br(2)–C(4)	1.96 (2)	C(7)–O(8)	1.42 (2)
P(5)–C(4)	1.78 (2)	C(9)–C(11)	1.56 (3)
P(5)–C(7)	1.82 (2)	C(9)–O(8)	1.41 (2)
P(5)–C(3)	1.79 (1)	C(9)–O(10)	1.15 (2)

[a] See footnote *a*, Table I.

Table IV. Bond Angles[a]

Atoms	Angle, deg	Atoms	Angle, deg
C(4)–P(5)–C(7)	100 (1)	P(5)–C(7)–O(8)	107 (1)
C(4)–P(5)–C(3)	103 (1)	C(11)–C(9)–O(8)	109 (2)
C(4)–P(5)–O(6)	115 (1)	C(11)–C(9)–O(10)	127 (2)
C(7)–P(5)–C(3)	104 (1)	O(8)–C(9)–O(10)	123 (2)
C(3)–P(5)–O(6)	116 (1)	Br(1)–C(3)–P(5)	110 (1)
C(7)–P(5)–O(6)	116 (1)	C(7)–O(8)–C(9)	117 (2)
Br(2)–C(4)–P(5)	110 (1)		

[a] See footnote *a*, Table I.

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(9) L. E. Alexander and G. S. Smith, *Acta Crystallogr.*, 17, 1195 (1964).

(10) G. H. Stout and L. H. Jensen, "X-Ray Structure Determination," Macmillan, London, 1968, pp 454–458.

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(14) Table II will appear following these pages in the microfilm edition of this volume of the journal. Single copies may be obtained from the Reprint Department, ACS Publications, 1155 Sixteenth St., N. W., Washington, D. C. 20036, by referring to author, title of article, volume, and page number. Remit $3.00 for photocopy or $2.00 for microfiche.

Nmr Studies. The instrumentation used to carry out the normal nmr experiments as well as the indor studies is described elsewhere[15] and pertinent data are given in Table V. The ^{1}H spectrum consists of a singlet CH_3, a doublet CH_2Br, and a doublet CH_2O resonance in the ratio 3:4:2. ^{13}C satellites on these resonances were also observed. Although the ^{31}P resonance is expected to be a quintet of triplets, the small $^2J_{\mathrm{PH}}$ couplings of *ca.* 5 Hz allowed only the center of the ^{31}P chemical shift to be located upon irradiating at the ^{31}P frequency while observing the center of a peak in either of the ^{1}H doublets. Similar indor experiments involving the natural abundance ^{13}C satellites in the ^{1}H spectra of the CH_2Br and CH_2O doublets while irradiating in the ^{31}P or ^{13}C frequency regions allowed the determination of the absolute signs of the various couplings using the assumption that $^1J_{\mathrm{CH}}$ is positive. That $^1J_{\mathrm{CH}}$ is very likely to be positive in all cases is strongly indicated from both theoretical and experimental evidence.[16]

Table V. Nmr Data for Bis(bromomethyl)acetoxyphosphine Oxide[a]

Nucleus	δ^b	$^2J_{\mathrm{PH}}$	$^1J_{\mathrm{PC}}$	$^1J_{\mathrm{CH}}$
CH_2Br	-3.12^c	-6.5		$+152$
^{13}CH$_2$Br	-0.398^d		$+67$	
^{31}P	-33.55^e			
CH_2O	-4.37^c	-4.2		$+150$
^{13}CH$_2$O	-37.51^d		$+88$	
CH_3	-2.22^c			

[a] In CD_3CN solution. [b] Negative values imply downfield shift with respect to the standard used. [c] TMS internal standard. [d] With respect to naturally abundant ^{13}C in external $^{13}CH_3COOH$. [e] With respect to external 85% H_3PO_4.

The connected transitions as revealed by the indor spectra obtained on either the CH_2Br or CH_2O protons are shown schematically in Figure 2. Since the upfield ^{13}C proton satellite doublet (b) in the ^{1}H spectrum and the upfield member of the ^{31}P doublet (a) arise from the same ^{13}C spin state, $^1J_{\mathrm{PC}}$ is positive as is $^1J_{\mathrm{CH}}$. The low-field ^{13}C proton satellite doublet (b) is connected to the low-field member (a) of the ^{31}P doublet for the same reason. Because the low-field member (c) of each ^{13}C proton satellite doublet (b) is connected to the upfield members (d) of each doublet in the ^{13}C triplet of doublets, the same ^{31}P spin state is involved. The same is true for the lines labeled c which are connected to those labeled e. Thus $^1J_{\mathrm{PC}}$ and $^2J_{\mathrm{PH}}$ are opposite in sign, rendering the latter coupling negative since $^1J_{\mathrm{PC}}$ was already found to be positive.

The ^{13}C and ^{31}P chemical shifts were experimentally determined relative to the protons in TMS (the internal locking reference) which were caused to resonate at 60.002 MHz. The ^{13}C and ^{31}P shifts were converted to the more conventional δ values relative to external $^{13}CH_3COOH$ and 85% H_3PO_4, respectively, *via* the relationship $\delta = (\nu_r - \nu_s)10^6/\nu_r$, where ν_r and ν_s are the frequencies of the reference and sample, respectively, measured with respect to the proton frequency in TMS.

Discussion

The computer drawing of the determined structure of III shown in Figure 1 clearly shows the presence of an acetoxymethyl group, two bromomethyl groups, and an oxygen atom bound to a central phosphorus. The planar acetoxy moiety exhibits bond angles and lengths which are typical of this functional group,[17] and the same is true for the bromomethyl group.[17] Further evidence for the presence of the acetoxy group are the infrared bands at 1252 and 1752 cm^{-1} observed in KBr which typically have ranges of 1220–1280 and 1735–1750 cm^{-1}, respectively.[18a] The P=O stretching frequency at 1220 cm^{-1} is somewhat higher than that found for $OPMe_3$ (1160 cm^{-1})[18b] in the solid state and compares more favorably with the bands reported[19] for $OP(CH_2OH)_3$ (1190, 1130), $OP(CH_2Cl)_3$ (1245, 1190), $OP(CH_2Br)_3$ (1205, 1190, 1150), and $OP(CH_2I)_3$ (1190). The P=O and P—C bond lengths are very similar to those in $OPMe_3$ (1.48 and 1.82 Å, respectively[20]) and the average CPC bond angle of 102° is significantly smaller than that found in $OPMe_3$ (106°).[20] The smaller bond angles in III could well reflect the tendency of the more electronegative phosphorus substituents to induce more p character in the phosphorus hybrids in order to maintain isovalent hybridization. Mitigating against smaller CPC angles in III are the larger steric requirements of the phosphorus substituents but this problem appears to be circumvented by the folding of the bromines and the acetoxy group outward with respect to the O=P axis.

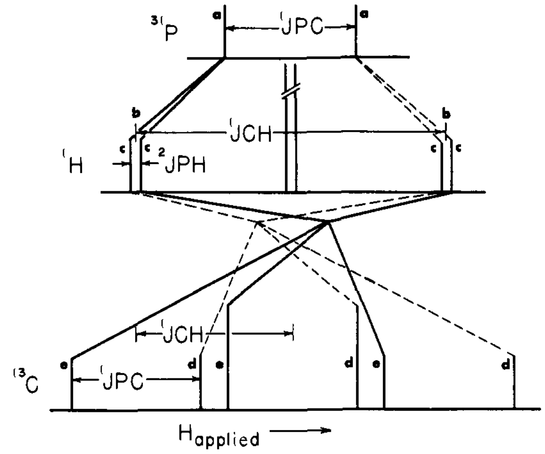

Figure 2. Schematic representations of the ^{31}P, ^{1}H, and ^{13}C spectra showing the lines having nuclear spin states in common as revealed by indor experiments. To save space, scales were not preserved among the spectra shown. The labels a through e are discussed in the text.

The proton chemical shifts are in keeping with the supposition that inductive effects are dominant, thus yielding increasingly downfield values from CH_3COO to CH_2Br to CH_2O owing to the increasingly electronegative substituent on the proton-bearing carbon (Table V). The sizable downfield shift in the ^{13}C resonance from CH_2Br to CH_2O (37 ppm) for the most part probably also reflects the increase in electronegativity of the carbon substituent.[21] The ^{31}P chemical shift compares more favorably with $OP(CH_2Cl)_3$ (−38.1 ppm) and $OPMe_3$ (−36.2 ppm) than it does with $OP(CH_2Br)_3$ (−42.5) or $P(CH_2OH)_3$ (−45.4 ppm)[22] but the interplay of factors influencing ^{31}P chemical shifts is not understood in sufficient detail to interpret our results.

In a study of the systematic trends of coupling constants between directly bonded nuclei, Jameson and

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(15) R. D. Bertrand, D. A. Allison, and J. G. Verkade, *J. Amer. Chem. Soc.*, 92, 71 (1970).

(16) C. J. Jameson and H. S. Gutowsky, *J. Chem. Phys.*, 51, 2790 (1969).

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(18) (a) R. M. Silverstein and G. C. Bassler, "Spectrometric Identification of Organic Compounds," 2nd ed, Wiley, New York, N. Y., 1967, p 92; (b) V. J. Goubeau and W. Berger, *Z. Anorg. Allgem. Chem.*, 304, 147 (1960).

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(21) J. W. Emsley, J. Feeney, and L. H. Sutcliffe, "High Resolution Nuclear Magnetic Resonance Spectroscopy," Vol. 2, Pergamon Press, New York, N. Y., 1966, p 988.

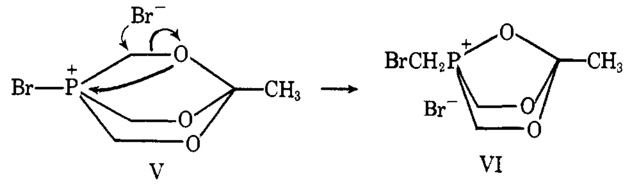
(22) V. Mark, C. Dungan, M. Crutchfield, and J. Van Wazer, *Top. Phosphorus Chem.*, 5, 227 (1967).

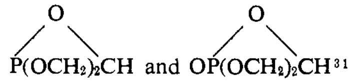
Gutowsky[16] formulated a model which predicted both positive and negative coupling constants for $^{31}P-^{13}C$ coupling. Jameson later showed[23] that the transition in the sign of the coupling constant in compounds of phosphorus is in agreement with intuitive arguments based on hybridization and the effect of ligand electronegativity on hybridization. Thus, for trivalent phosphorus compounds, the P–C coupling constant is small and negative, becoming positive in phosphonium salts and pentavalent phosphorus molecules, and increasingly positive as more electronegative atoms are bonded to phosphorus. At the same time, the geminal PCH coupling constant is positive for trivalent compounds and becomes negative in phosphonium and pentavalent systems. The finding that both P–C couplings are positive with $^1J_{PCO}$ being larger than $^1J_{PCBr}$ is in accord with the electronegativity arguments given by Jameson.[23] The positive increase in $^2J_{PH}$ couplings for the PCH_2Br (−6.5 Hz) and PCH_2O (−4.2 Hz) systems in III also can be rationalized on isovalent hybridization arguments[24] which yield such a coupling trend when more electronegative substituents are attached to carbon.[15,24] These couplings compare well with $^2J_{PH}$ values found for $OP(CH_2Br)_3$ (7 Hz)[25] and $OP(CH_2OH)_3$ (3.1 Hz)[26] and this suggests that the latter couplings are also negative. The $^1J_{CH}$ couplings are typical for carbon groups which utilize very nearly sp^3 hybrids in bonding to hydrogen.[27]

Although the detailed mechanism for this reaction cannot be deduced from the data presently available, it apparently does not proceed primarily by a free radical path since an atmosphere of oxygen results in the same yield of product as a control reaction run under nitrogen. Thus, a plausible pathway involves nucleophilic attack of phosphorus on bromine to form V from IV directly or *via* II

$$\begin{tikzcd}[column sep=small] \mathrm{Br_2 + I} \arrow[r] & \underset{\mathrm{IV}}{\mathrm{Br{-}Br{\leftarrow}P(CH_2O)_3CCH_3}} \arrow[rr] \arrow[dr] & & \underset{\mathrm{V}}{\mathrm{[BrP(CH_2O)_3CCH_3]Br}} \\ & & \mathrm{II} \arrow[ur] & \end{tikzcd}$$

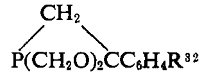
The possibility of at least a transient existence of IV is made reasonable by the characterization of similar halogen adducts involving a variety of group VI bases as well as R_3N.[28] Although no indication of the presence of V has been found even at 0°, the reaction of bromine with $P(OCH_2)_3CCH_3$ under these conditions has permitted the acquisition of nmr and conductivity evidence[29] strongly indicating the stable presence of $[BrP(OCH_2)_3CCH_3]Br$ prior to the formation of the Michaelis–Arbuzov product[30] at higher temperatures. Nucleophilic attack of bromide on a methylene carbon in V could allow ring contraction to form VI. Whether this step takes place *via* ring opening to form a phosphonium salt or *via* a three-center intermediate is open to question. The plausibility of VI as a possible intermediate is given credence by the isolation and characterization of





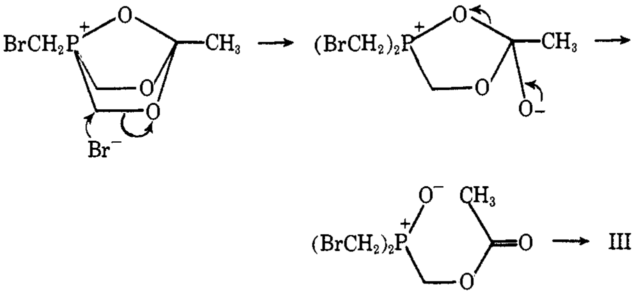
$P(OCH_2)_2CH$ and $OP(OCH_2)_2CH$[31]

as well as



$P(CH_2O)_2CC_6H_4R$[32]

Repetition of the bromide attack on a methylene carbon could then complete the sequence in which a strong driving force is the formation of a P=O link.



Acknowledgment. The National Science Foundation is thanked for a grant to John G. Verkade in partial support of this research and funding to the University toward the purchase of the nmr instrumentation employed.

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