TABLE III

ANODIC OXIDATION OF POTASSIUM BUTYRATE AND POTASSIUM PROPANEBORONATE AT PYROLYTIC GRAPHITE

	%	
Products	Potassium butyrate[a]	Potassium propaneboronate[b]
Propylene	66	39
Cyclopropane	33	33
Ethylene	0	28

[a] The aqueous solution of 1 M potassium butyrate is at pH 7. Koehl was the first to obtain this result.[5] We confirm this and have shown also that the form of carbon used (porous carbon rods or either plane of pyrolytic graphite) does not affect the product ratios. [b] This was studied as a saturated aqueous solution of the acid and its potassium salt, 10:1. Some ethylene may be a product of β scission of the propyl cation; some may arise from oxidation of propanol, formed from the propaneboronic acid.[c] Since 28% ethylene is found after only 5-min reaction, ethylene is probably a primary product to the extent of at least 10–20%. Its yield increased with reaction time and with pH, while the cyclopropane to propylene ratio remained about constant. [c] A. G. Davies and B. P. Roberts, *J. Chem. Soc., B*, 17 (1967); T. G. Traylor and J. C. Ware, *J. Amer. Chem. Soc.*, **85**, 3026 (1963).

case and makes its influence felt in the reactions of the carbonium ion. In addition, different rates of oxidation and desorption of the precursors on graphite may be responsible for the variation.[6] In any case, both cations exhibit the characteristic of the free cation, substantial production of cyclopropane.

The similarities in the alkaneboronate and alkanecarboxylate oxidations prompted the study of the anodic oxidation of alkyl halides. Propyl bromide was chosen because of the above-mentioned prominent difference between the free and the encumbered propyl cation. The reaction was run in methanol with sodium perchlorate as the electrolyte. The anode and cathode compartments were separated to prevent cathodic reduction of alkyl halide.[7] Table IV shows that the carbonium ion formed in this reaction can indeed be described as free. It is likely that the anodic oxidations of other alkyl halides also yield free carbonium ions.[8] A possible mechanism is shown in eq 3. Rough calcula-

$$\mathrm{PrBr} \xrightarrow{-e^-} (\mathrm{PrBr})^+ \longrightarrow \mathrm{Pr}^+ + \mathrm{Br}\cdot \qquad (3)$$

tions indicate that the separation in the gas phase of bromine atom from the propyl cation is ~30 kcal mol endothermic. This energy requirement could be ameliorated at the anode surface by adsorption and by charge–charge repulsion of $(\mathrm{PrBr})^+$ and the anode.

TABLE IV

ANODIC OXIDATION OF PROPYL BROMIDE AT BRIGHT PLATINUM

Products	Propyl bromide,[a] %
Propylene	16
Cyclopropane	78
Propane	6

[a] The methanolic solution was 1 M each in propyl bromide and sodium perchlorate monohydrate. The anode and cathode compartments were separated by an agar bridge saturated with potassium chloride. It was not determined if the propylene would be destroyed by the perchlorate under the electrolysis conditions.

The anodic oxidation of 1-nitropropane was attempted in methanol with sodium perchlorate electrolyte. No products attributable to propyl cation intermediacy were detected. Barnes[9] has anodically oxidized n-propylamine at platinum in acetonitrile and has found propylene among the products. No mention is made of cyclopropane but it may not have been sought. A free propyl cation may be formed. Whatever the details of the Barnes reaction, our work shows that nitropropane is not a carbonium ion producing intermediate in the anodic oxidation of n-propylamine.

Experimental Section

Apparatus.—The cell used in the electrolyses has been described.[10] It was modified for the pyrolytic graphite reactions. In place of the platinum, two strips of graphite, $^1/_2 \times 4 \times {}^1/_8$ in., were fixed in the rubber stopper by means of alligator clips inserted through the stopper. The graphite was purchased from High Temperature Materials, Inc., Boston, Mass. The unit for the anodic oxidation of propyl bromide was two cylinders, closed at the bottom, connected by a tube filled with potassium chloride saturated agar gel. The anode compartment of the unit was fitted with a stopper containing the anode, thermometer, and gas inlet and outlet tubes. A stainless steel cathode was put in the other cylinder.

Technique.—The anodic oxidations were typically run for 5 min at 25–35° at a current density of 0.1 A/cm. The gaseous products were swept into liquid nitrogen cooled traps. The material caught was analyzed by glpc. In addition to being identified by comparative retention times, the peaks were trapped as eluted and their ir and mass spectra were obtained.

Propaneboronic Acid.—This was prepared according to the method of Snyder, Kuck, and Johnson.[11]

Registry No.—Sodium butyrate, 156-54-7; potassium propaneboronate, 19581-69-2; potassium pentanoate, 19455-21-1; sodium butaneboronate, 19581-70-5; potassium butyrate, 589-39-9; propyl bromide, 106-94-5.

Acknowledgment.—We are grateful to the National Science Foundation for a graduate fellowship for J. T. K., and to the Air Force Office of Scientific Research and the Research Office of the U. S. Army for support of this work.

[6] A referee suggested this possibility.

[7] J. W. Sease, F. G. Burton, and S. L. Nickol, *J. Amer. Chem. Soc.*, **90**, 2595 (1968).

[8] L. L. Miller and A. K. Hoffman, *ibid.*, **89**, 593 (1967).

[9] K. K. Barnes, *Dissertation Abstr.*, 3805-B, (1967).

[10] P. H. Reichenbacher, M. Y.-C. Liu, and P. S. Skell, *J. Amer. Chem. Soc.*, **90**, 1816 (1968).

[11] H. R. Snyder, J. A. Kuck, and J. R. Johnson, *ibid.*, **60**, 105 (1938).

The Wolff Rearrangement of 1,3-Bisdiazo-2-decalones

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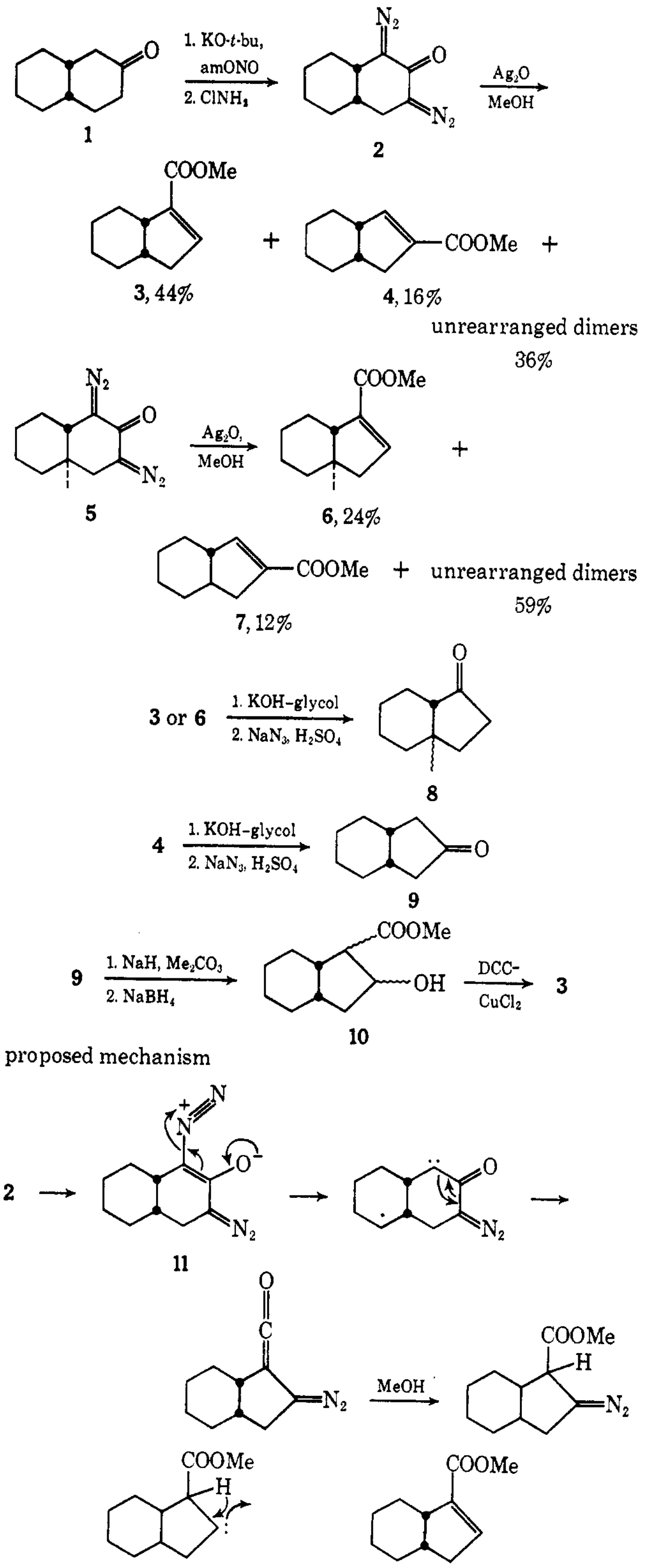
Our interest in the stereospecific synthesis of functionalized perhydroindans led us to an examination of the rearrangement of *cis*- and *trans*-1,3-bisdiazo-2-decalone with the aim of discovering (a) whether the ring contraction would be directionally selective, (b) whether the product composition would be dependent upon ring junction stereochemistry, and (c) whether the stereochemistry of the ring junction would be retained. In addition, it was hoped that the results would shed some light on the mechanism of the Wolff rearrangement.

cis- and *trans*-bisdiazodecalones **2** and **5** were prepared by treatment of the ketones with amyl nitrite and potassium *t*-butoxide[1] and reaction of the dioximino ketones formed with chloramine.[2] The bisdiazo ketones, isolated in 25–40% yield, showed characteristic ir absorption at 2084, 2062, and 1580 cm^{-1} and strong ultraviolet absorption at 323 mμ (ε 25,000). Photolysis[3] of **2** in aqueous THF or in absolute methanol led to a complex mixture of at least 12 products. When the rearrangement was carried out by refluxing with silver oxide in absolute methanol, a 73% yield of crude product was obtained. Glpc analysis revealed that 64% of the product was composed of three monomeric compounds, present in yields of 4, 44, and 16%. The two major components were isolated by preparative glpc and proved to be the two unsaturated esters **3** and **4** (Scheme I). The remaining 36% of the product was composed of a mixture of three compounds of long retention time, which spectroscopic and mass spectral analysis indicated were unrearranged *dimeric* methoxydecalones; no effort was made to elucidate the detailed structures of these isomeric dimers.

Unequivocal structural assignment of **3** and **4** was made on the basis of the following data. Ester **3** was saponified with KOH–ethylene glycol and then subjected to Schmidt rearrangement[4] to give an equilibrium mixture (75:25)[5] of *cis*- and *trans*-perhydroindan-1-one **8**; no perhydroindan-2-one was detected. Similar degradation of **4** contaminated with a small amount of **3** afforded *cis*-perhydroindan-2-one **9** as the major product. Because of the equilibrating conditions of the Schmidt rearrangement, the ring-junction stereochemistry of **3** was confirmed by independent synthesis. *cis*-Perhydroindan-2-one[6] was treated with sodium hydride–dimethyl carbonate[7] and the resulting β-keto ester reduced with sodium borohydride to give hydroxy ester **10** in 77% yield. Dehydration with cupric chloride–DCC[8] afforded a 73% yield of an ester identical with **3** by glpc peak enhancement on two columns. Thus the *cis* stereochemistry was preserved in both of the unsaturated esters.

Silver oxide–methanol rearrangement of 1,3-bisdiazo-*trans*-2-decalone **5** led to an 89% yield of crude product in which the unrearranged methoxy ketone dimers predominated (59% of the total product). The monomeric products, obtained in 24 and 12% yields, respectively, were assigned structures **6** and **7** on the following basis. Although the basic spectral features of **6** and **7** were identical with those of **3** and **4** (see Experimental Section), glpc analysis revealed that all four compounds were indeed different. Schmidt degradation of **6** afforded the same mixture of stereoisomeric ketones **8** as that obtained from **3**; similar degration of **7** afforded a ketone different from **8** or **9** and thus was assumed to be *trans*-2-perhydroindanone.

SCHEME I



The increased propensity for dimerization in the *trans*-fused system presumably results from the angle strain imposed on the adjacent six-membered ring in the transition state. When bond formation leads to a *cis*-fused perhydroindan, the angle strain can be relieved by the six-membered ring taking on a slightly twisted

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(1) H. Rapoport and J. B. Lavigne, *J. Amer. Chem. Soc.*, **75**, 5329 (1953).

(2) M. O. Forster, *J. Chem. Soc.*, **107**, 260 (1915).

(3) During the course of this work the photolysis of 1,6-bisdiazo-4-*t*-butylcyclohexanone was reported: R. Tasovac, M. Stefanović, and A. Stojiljković, *Tetrahedron Lett.*, 2729 (1967).

(4) E. W. Garbisch, Jr., and J. Wohllebe, *J. Org. Chem.*, **33**, 2157 (1968).

(5) H. O. House and G. H. Rasmusson, *ibid.*, **28**, 31 (1963).

(6) Prepared by rhodium–alumina reduction of 2-indanone; upon Wolff-Kishner reduction *cis*-perhydroindan was obtained, thus confirming that the ring junction was indeed *cis*.

(7) S. J. Rhoads, J. C. Gilbert, A. W. Decora, R. T. Garland, R. J. Spangler, and J. M. Urbigkit, *Tetrahedron*, **19**, 1625 (1963).

(8) E. J. Corey, N. H. Andersen, R. M. Carlson, J. Paust, E. Vedejs, I. Vlattas, and R. E. K. Winter, *J. Amer. Chem. Soc.*, **90**, 3245 (1968).

conformation. There is no possibility for strain relief in the *trans*-fused perhydroindane, however, and thus dimerization becomes energetically more favorable. This effect is also reflected in the fact that *cis*-perhydroindan-1-one is more stable than the *trans* form by 0.65 kcal/mol.[5]

The directional selectivity of the rearrangement is not so easily explained. It is possible that steric crowding accelerates the removal of the diazo group adjacent to the ring junction, thus leading to perferential formation of the carbene in the 1 position, although such crowding is not readily apparent from an examination of models. We feel that the best explanation of the results is provided by a transition state for keto-carbene formation in which the ketone exists largely in the enolic form, **11**, as in the mechanism outlined below. As nitrogen is expelled from the diazonium enolate, C-1 becomes less negative; the increase in the positive character at this position is favored by the inductive effect of the alkyl group attached to C-1. Thus the inductive effect promotes preferential loss of nitrogen from C-1, and the directional selectivity should be independent of ring-junction stereochemistry. This is confirmed by the similar product ratios obtained from the *cis* and the *trans* compounds. Therefore, it appears that the stabilization of increased positive character at the α carbon plays an important role in the mechanism and stereospecificity of the Wolff rearrangement.

Experimental Section

Infrared spectra were recorded on a Perkin-Elmer Model 257 spectrophotometer. Ultraviolet spectra were determined with a Carey 11 spectrophotometer. Nmr spectra were recorded on a Varian A-60 spectrometer in CCl_4 solutions using tetramethylsilane as an internal standard. The glpc analyses were obtained with an Aerograph 90-P3 gas chromatograph using a 10 ft × 0.25 in. column packed with 10% FFAP on Chromosorb W and with helium as the carrier gas.

1,3-Bisdiazo-*cis*-2-decalone (2).—To a solution of potassium *t*-butoxide (from 8.3 g of K, 0.21 mol) in 200 ml of dry *t*-butyl alcohol was added 12.8 g (0.084 mol) of *cis*-2-decalone[9] at 25°. Isoamyl nitrite (30 g, 0.26 mol) was then added dropwise, and the resulting solution was allowed to stand for 18 hr at 25°. Water (1 l.) was added, and the resulting solution was extracted with five 200-ml portions of ether. The aqueous layer was acidified with dilute HCl and extracted with three 100-ml portions of chloroform. The combined chloroform extracts were dried ($MgSO_4$) and evaporated, and the residue was taken up in 30 ml of hot methanol and poured into 250 ml of ether. The resulting precipitate was collected and dried to give 9.88 g (50%) of dioximino ketone: ν_{max} (Nujol) 3215, 1715, 1610, 1585 cm^{-1}; λ_{max}^{EtOH} 273 nm (ε 12,000).

The dioximino ketone (500 mg, 2.38 mmol) was dissolved in 9 ml of 2 *N* NaOH and 10 ml of concentrated ammonium hydroxide. Ether (100 ml) was added, and sodium hypochlorite (30 ml of 5% solution) was added dropwise with stirring over 2 hr. After stirring 18 hr at 25°, the ether layer was separated and the aqueous layer was extracted with three 50-ml portions of ether. The combined extracts were dried ($MgSO_4$) and evaporated to give 344 mg (69%) of **2** as a bright yellow oil: ν_{max} ($CHCl_3$) 2084, 2062, 1580 cm^{-1}; λ_{max}^{EtOH} 323 mμ (ε 25,000).

Rearrangement of 1,3-Bisdiazo-*cis*-2-decalone (2).—To a solution of **2** (1.19 g, 6.6 mmol) in 15 ml of refluxing absolute methanol was added 1 g of freshly prepared silver oxide in small portions over 2 hr. The mixture was refluxed for 12 hr, then filtered through Hy-Flo and evaporated to give 1.10 g of crude product. Distillation through a short-path column afforded 737 mg (73%) of yellow oil, bp 150–165° (bath temperature) (16 mm). Glpc analysis (120°) showed the presence of six products, three low-boiling products in 4, 44, and 16% yields, and approximately equal amounts of three high-boiling products in 36% total yield. Preparative glpc afforded pure samples of **3** [44% yield, ν_{max} (neat) 1715 cm^{-1}; λ_{max} 227 mμ (ε 14,000); nmr δ 6.61 (t, 1), 3.62 (s, 3); molecular ion *m/e* 180. (*Anal.* Found: C, 73.2; H, 9.1)] and **4** [16% yield; ν_{max} (neat) 1720 cm^{-1}; λ_{max} 227 mμ (ε 10,000); nmr δ 6.61 (m, 1), 3.62 (s, 3); molecular in *m/e* 180 (*Anal.* Found: C, 73.2; H, 9.1)].

Schmidt Degradation of 3.—A solution of **3** (26.4 mg, 0.15 mmol) and 100 mg of potassium hydroxide in 2 ml of ethylene glycol was refluxed for 10 min. The solution was poured into 25 ml of water and extracted with two 5-ml portions of ether. The aqueous solution was acidified and extracted with three 5-ml portions of benzene. The extracts were concentrated to 2 ml, and 50 mg of sodium azide and 2 ml of concentrated H_2SO_4 were added. After heating at 45° for 30 min, the solution was poured into 20 ml of water and refluxed for an additional 30 min. The solution was extracted with three 5-ml portions of ether, and the extracts were dried ($MgSO_4$) and evaporated to give 14.0 mg of crude product. Glpc analysis indicated a 3:1 mixture of two compounds which were shown to be *cis* and *trans* **8** by peak enhancement with authentic samples.[5]

Schmidt Degradation of 4.—Reaction of 10.4 mg of **4** containing some **3** with KOH and then HN_3 as described above afforded 6.7 mg of product. Glpc showed the major product to be *cis*-2-perhydroindanone by peak enhancement with an authentic sample.[6]

Synthesis of Authentic 3.—A solution of *cis*-2-perhydroindanone[6] (2.0 g, 14.5 mmol) in 9.5 ml of dimethyl carbonate containing 5 drops of methanol was added dropwise to a stirred suspension of sodium hydride (3.0 g of 50% dispersion, freed of mineral oil by washing with petroleum ether) in 43 ml of dimethyl carbonate; stirring was then continued for 1 hr. The mixture was cooled to 0°, and a solution of 3 ml of glacial acetic acid in 25 ml of ether was added. Water (100 ml) was added, and the solution was extracted with three 50-ml portions of ether. The combined extracts were washed with sodium bicarbonate, dried ($MgSO_4$), and evaporated to give 2.60 g (92%) of product. The crude β-keto ester (2.60 g, 13.3 mmol) was dissolved in 150 ml of ethanol and 25 ml of water at 0°. Sodium borohydride (1.5 g) was added in portions to the cooled, stirred solution over 1 hr. The solution was stirred 1.5 hr at 25°, and most of the solvent was removed at reduced pressure. Ice was added, and the mixture was extracted with three 50-ml portions of ether. The combined extracts were dried ($MgSO_4$) and evaporated to give 2.20 g (84%) of hydroxy ester **10** as a mixture of diastereomers: ν_{max} (neat) 3400, 1730 cm^{-1}.

To a solution of hydroxy ester **10** (100 mg, 0.51 mmol) and N,N′-dicyclohexylcarbodiimide (110 mg, 0.53 mmol) in 25 ml of ether was added 50 mg of cuprous chloride;[8] the resulting mixture was stirred for 18 hr at 25°. The solution was filtered through a short column of silica gel to remove the urea; evaporation of the eluate afforded 67 mg (73%) of ester **3**. This product was identical with the major product from the Wolff rearrangement in spectral properties and by glpc peak enhancement on FFAP and SE-30 columns.

1,3-Bisdiazo-*trans*-2-decalone (5).—Bisdiazo ketone **5** was prepared from *trans*-2-decalone[10] in 25% yield as described for the *cis* compound **2**: ν_{max} (Nujol) 2080, 2062, 1580 cm^{-1}; λ_{max}^{EtOH} 324 mμ (ε 24,000).

Rearrangement of 1,3-Bisdiazo-*trans*-2-decalone (5).—To a solution of **5** (530 mg, 2.6 mmol) in 15 ml of refluxing absolute methanol was added 1 g of freshly prepared silver oxide in portions over 1 hr. After refluxing 2 hr more, the uv spectrum of an aliquot showed X2% of **5** remaining. The suspension was filtered through Hy-Flo and evaporated to give 435 mg of crude product. Glpc analysis showed the presence of two low-boiling products (12 and 24% yield) and two higher boiling products (59% total yield). Preparative glpc afforded pure samples of **6** [24% yield; ν_{max} (neat) 1720 cm^{-1}; λ_{max}^{EtOH} 227 mμ (ε 11,000); nmr δ 6.61 (t, 1), 3.62 (s, 3); molecular ion *m/e* 180] and **7** [12% yield, ν_{max} (neat) 1720 cm^{-1}; λ_{max}^{EtOH} 227 mμ (ε 10,000); nmr δ 6.61 (m, 1), 3.62 (s, 3); molecular ion *m/e* 180]. Both **6** and **7** were different from **3** and **4** on simultaneous injection glpc analysis.

Schmidt Degradation of 6 and 7.—Degradation was carried out on 20 mg of **6** and 15 mg of **7** exactly as described for **3**. The crude product (11 mg) from **6** upon glpc analysis proved to be a 75:25 mixture of *cis*- and *trans*-1-perhydroindanones **8**, identical with

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[9] R. L. Augustine, *J. Org. Chem.*, **23**, 1853 (1958).

[10] E. E. van Tamelen and W. C. Proost, Jr., *J. Amer. Chem. Soc.*, **76**, 3632 (1954).

that obtained from **3**. Degradation of **7** afforded 7 mg of crude product which was similar but not identical with **8** or **9** on glpc analysis.

Registry No.—**2**, 19614-40-5; **5**, 19614-41-6.

Acknowledgment.—We are grateful to E. I. du Pont de Nemours and Co. for financial assistance in the form of a summer fellowship for D. L. F.

Tetramethyl Acetylenediphosphonate and Dimethyl Chloroacetylenephosphonate and Their Reactions with Cyclopentadiene, 1,3-Cyclohexadiene, and Diazomethane[1]

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Our previous work on Diels–Alder reactions of alkynyl derivatives of the group IVb elements, mainly of bis(trimethyltin)acetylene[1a–c] and bis(trimethylsilyl)acetylene,[1d] made an assessment of the Diels–Alder reactivity of acetylenes with group Vb organic substituents of interest to us. Especially noteworthy in our previous work was the formation of *m*-bis(trimethylsilyl)benzene as almost the sole product in the reaction of α-pyrone with bis(trimethylsilyl)acetylene,[1d] and it was especially of interest to investigate if a similar isomerization might occur in suitable Diels–alder reactions of phosphorus acetylenes of type $(RO)_2P(O)C{\equiv}CP(O)(OR)_2$.

The preparation of tetraethyl acetylenediphosphonate has been reported previously by Ionin and Petrov[3] (eq 1 and 2, R = Et).

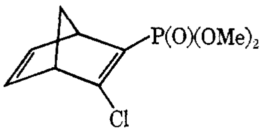
$$(RO)_3P + ClC{\equiv}CCl \xrightarrow[0^\circ]{Et_2O} \underset{\text{I}}{(RO)_2P(O)C{\equiv}CCl} + RCl \qquad (1)$$

$$(RO)_3P + (RO)_2P(O)C{\equiv}CCl \longrightarrow \underset{\text{II}}{(RO)_2P(O)C{\equiv}CP(O)(OR)_2} + RCl \qquad (2)$$

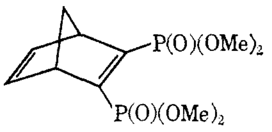
While some displacement reactions of the C—Cl bond of I (R = Et) were studied,[3,4] the reactivity of the C≡C bond in compounds such as I and II appears not to have been investigated. In the present study we have prepared tetramethyl acetylenediphosphonate by the Ionin–Petrov two-step procedure; in the first step $(MeO)_2P(O)C{\equiv}CCl$ was obtained in 88% yield and in the second step the $(MeO)_2P(O)C{\equiv}CP(O)(OMe)_2$ yield was 72%.

A brief study of the reactions of I and II (R = Me) showed that they readily undergo Diels–Alder reactions and 1,3 dipolar additions and thus organophosphorus-substituted acetylenes are potentially useful precursors for the introduction of organophosphorus substituents into diverse organic structures.

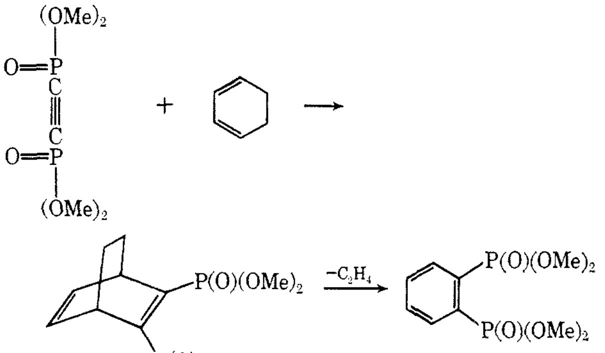
Reaction of cyclopentadiene with I and II (R = Me) gave the norbornadienephosphosphonates IIIa and IIIb in good yield. Tetramethyl acetylenediphosphonate also was allowed to react with 1,3-cyclohexadiene since in the case of this diene it was expected that the initial bicyclic Diels–Alder adduct would undergo thermolysis with loss of the dimethylene bridge to form a benzene derivative.[5,6] Such thermolysis would be analogous to the loss of carbon dioxide from an acetylene–α-pyrone Diels–Alder adduct, and the structure of the benzene derivative formed would provide further useful information concerning the factors which are important in determining whether or not isomerization occurs in Diels–Alder reactions of this type. The reaction of II (R = Me) with 1,3-cyclohexadiene at 150° gave a phenylenediphosphonate in 93% yield (eq 3), and the melting point and spectral properties of this product were in complete agreement with those of the known[7] tetramethyl *o*-phenylenediphosphonate (IV). The ethylene evolved in the thermolytic decomposition of the initial adduct was identified by means of its conversion into 1,2-dibromoethane. The reaction of I (R = Me) with 1,3-cyclohexadiene similarly gave dimethyl *o*-chlorobenzenephosphonate, which also has been prepared previously.[7] Although the reaction of II (R = Me) with α-pyrone remains to be studied, it would appear that this acetylene reacts normally in Diels–Alder reactions of this type and thus bis(trimethylsilyl)acetylene remains the only acetylene which we have examined thus far which reacts anomalously.



IIIa



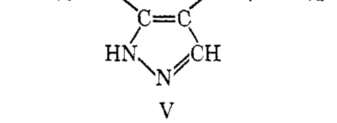
IIIb



IV

(3)

The addition of a solution of diazomethane to II (R = Me) resulted in formation of the 2-pyrazoline V.



V

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[1] Part V of a series of papers on "The Diels-Alder Reaction in Organometallic Chemistry." Parts I-IV are unnumbered and are listed below: (a) part I, D. Seyferth, C. Sarafidis, and A. B. Evnin, *J. Organometal. Chem.*, **2**, 417 (1965); (b) part II, A. B. Evnin and D. Seyferth, *J. Amer. Chem. Soc.*, **89**, 952 (1967); (c) part III, D. Seyferth and A. B. Evnin, *ibid.*, **89**, 1468 (1967); (d) part IV, D. Seyferth, D. R. Blank, and A. B. Evnin, *ibid.*, **89**, 4793 (1967).

[2] Postdoctoral Research Associate, 1967–1968.

[3] B. I. Ionin and A. A. Petrov, *Zh. Obshch. Khim.*, **35**, 1917 (1965).

[4] B. I. Ionin and A. A. Petrov, *ibid.*, **35**, 2255 (1965).

[5] K. Alder and H. Rickert, *Ann. Chem.*, **524**, 180 (1937).

[6] K. Alder and H. Rickert, *Ber.*, **70**, 1354 (1937).

[7] R. Obrycki and C. E. Griffin, *Tetrahedron Lett.*, 5049 (1966).