Phosphatranes as Unusual Stabilizing Structures for Hypervalent Phosphorus: 10-P-5 Mono- and Divalent Cations

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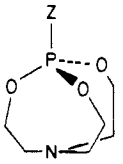
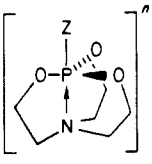
Abstract: ^1H, ^{13}C, and ^{31}P NMR evidence are presented for the formation of the hypervalent phosphorus mono- and divalent cations $[HYP(OCH_2CH_2)_3N]^+$, $[H_2YP(OCH_2CH_2)_3N]^{2+}$, and $[R_2YP(OCH_2CH_2)_3N]^{2+}$ (Y = O, S; R = Me, Et). These new 10-P-5 cations are apparently stabilized by the presence of three five-membered rings chelating the phosphorus and also by the strong basicity of the apical chalcogen arising from its participation in the three-center, four-electron bonding system along the axis of the trigonal-bipyramidal structure.

Phosphatranes constitute a class of compounds which can display pyramidal 8-P-3 (**1**),[3] tetrahedral 8-P-4 (**2**, **3**, **4**, **5**[3]), and trigonal-bipyramidal 10-P-5 (**6**,[3] **7**,[3] **8**,[4,5] **9**,[4,5] **10**,[6] **11**,[6] **12**,[3] **13**[3])



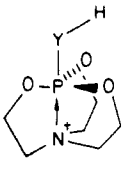


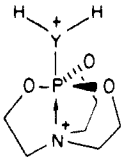
1, Z = lp
2, Z = O
3, Z = S
4, Z = Se
5, Z = BH_3

6, Z = F_3BO, n = 0
7, Z = $OP(OH)_3O$, n = 0
8, Z = H, n = 1+
9, Z = Ph_3C, n = 1+
10, Z = RO, n = 1+
11, Z = RS, n = 1+
12, Z = Et_3SiO, n = 1+
13, Z = $(Et_3Si)_2O$, n = 2+

coordination geometries. Studies of these compounds have elucidated the role of Lewis acids and chelation in stabilizing the 10-P-5 hypervalent structures[3-5] which in the cases of **8**[4] and **11** (R = Et)[6] have been shown to be trigonal bipyramidal (TBP) by X-ray means. The possibility of attack by nucleophiles on the pseudoequatorial carbon in TBP **10** and **11** has also been discussed.[6]

In an earlier publication[7] we predicted on the basis of CNDO/2 calculations that protonation of **2** would give rise to the 10-P-5 species **14** rather than quaternize the nitrogen. Here we present

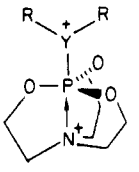


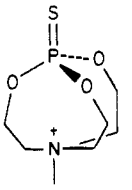


14, Y = O
15, Y = S

16, Y = O
17, Y = S

^1H, ^{13}C, and ^{31}P NMR evidence that **14** is stable at room temperature in CF_3CO_2H (TFA) solutions, and that **3** similarly protonates to give **15**. Moreover, NMR spectral evidence is put forth for the further protonation of the hydroxy and thiohydroxy groups in **14** and **15**, respectively, and for the diethylation of **2** and **3** giving rise to the unusual 10-P-5 dications **16**-**19**. In contrast to the formation of **14** and **16** from **2** in TFA, **2** protonates





18, Y = O, R = Et
19, Y = S, R = Et
20, Y = S, R = Me

21

at the nitrogen in an aqueous $HBF_4/Et_2O/CH_2Cl_2$ mixture to give isolable **21**. Compounds **10** and **11** (R = Me, Et) were reported earlier in a communication, but no experimental or spectral details were given.[6] Here we describe their isolation and NMR spectral properties.

Experimental Section

The standard for the ^1H and ^{13}C NMR spectra was Me_4Si (internal), and for the ^{31}P spectra 85% H_3PO_4 (external) was employed. Chemical shifts downfield of the standard are positive and are reported in parts per million (ppm). Coupling constants are reported in hertz.

$[MeOP(OCH_2CH_2)_3N]BF_4$ (10, R = Me). To a solution of 1.3 g of **2** (6.7 mmol) in 10 mL of CH_2Cl_2 at -78 °C was added with stirring a solution of 1.0 g of Me_3OBF_4 (6.7 mmol) in 3 mL of MeCN, and 5 mL of CH_2Cl_2 was added dropwise. As the mixture warmed to room temperature overnight, a white precipitate formed which was filtered off under nitrogen and washed with CH_2Cl_2. After the filtrate and washings were evaporated, a residue remained which was dissolved in a minimum amount of MeCN. After the solution was slowly evaporated to one-quarter of its original volume under a slow stream of dry nitrogen, colorless crystals remained which were isolated by decanting the slightly yellow solution: ^{31}P NMR (CD_3CN) -20.2; ^{13}C NMR (CD_3CN) 62.0 (d, CH_2O $^2J(PC)$ = 5), 48.1 (d, CH_2N, $^2J(PC)$ = 13), 54.0 (d, CH_3O, $^2J(PC)$ = 7); ^1H NMR (CD_3CN) 4.35 (dt, OCH_2, $^3J(PH)$ = 16.6, $^3J(HH)$ = 6.2), 3.39 (dt, CH_2N, $^3J(PH)$ = 4.0, $^3J(HH)$ = 6.2), 3.63 (d, CH_3O, $^3J(PH)$ = 11.5). Anal. Calcd (Found) for $C_8H_{17}NO_4BF_4$: C, 31.09 (30.07); H, 5.55 (5.41); N, 4.53 (4.26).

$[EtOP(OCH_2CH_2)_3N]BF_4$ (10, R = Et). To a stirred solution of 0.50 g of **2** (26 mmol) in 5 mL of CH_2Cl_2 cooled to -78 °C was added dropwise a solution of 0.5 g of Et_3OBF_4 in 5 mL of CH_2Cl_2. After the mixture warmed to room temperature the solvent was evaporated in a stream of dry N_2 to give a white solid. Trituration of the solid with CH_2Cl_2 at 0 °C removed the excess Et_3OBF_4: ^{31}P NMR (CD_3CN) -20.6; ^{13}C NMR (CD_3CN) 61.9 (d, CH_2O, $^2J(PC)$ = 5), 47.9 (d, CH_2N, $^2J(PC)$ = 12), 63.0 (d, OCH_2CH_3, $^2J(PC)$ = 7), 15.3 (d, OCH_2CH_3, $^3J(PC)$ = 9); ^1H NMR (CD_3CN) 4.28 (dt, OCH_2, $^3J(PH)$ = 17.0, $^3J(HH)$ = 6.0), 3.34 (dt, CH_2N, $^3J(PH)$ = 4.0, $^3J(HH)$ = 6.0), 3.91 (dq, (overlapping) OCH_2CH_3, $^3J(PH)$ = $^3J(HH)$ = 7), 1.18 (td, OCH_2CH_3, $^4J(PH)$ = 2.0, $^3J(HH)$ = 7).

$[MeSP(OCH_2CH_2)_3N]BF_4$ (11, R = Me) and $[Me_2SP(OCH_2CH_2)_3N](BF_4)_2$ (20). To a solution of 0.51 g of Me_3OBF_4 (4.3 mmol dissolved in a minimal amount of MeCN was added CH_2Cl_2 until the salt just began to precipitate. This mixture was cooled to -78 °C, and a solution of 0.90 g of **3** (4.3 mmol) was added dropwise. The suspension was stirred while it was allowed to warm to room temperature. The white solid was filtered off and washed with CH_2Cl_2. Upon slow evaporation

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(3) Carpenter, L. E.; Verkade, J. G. *J. Am. Chem. Soc.* **1985**, *107*, 7084.
(4) Clardy, J. C.; Milbrath, D. S.; Springer, J. P.; Verkade, J. G. *J. Am. Chem. Soc.* **1976**, *98*, 623.
(5) Milbrath, D. S.; Verkade, J. G. *J. Am. Chem. Soc.* **1977**, *99*, 6607.
(6) van Aken, D.; Merkelbach, I. I.; Koster, A. S.; Buck, H. M. *J. Chem. Soc., Chem. Commun.* **1980**, 1045.
(7) van Aken, D.; Castelijns, A. M. C. F.; Verkade, J. G.; Buck, H. M. *J. R. Neth. Chem. Soc. (1882-1979)* **1979**, *98*, 12.

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Table I. NMR Data for **2** and Its Mono- (**14**) and Diprotonation (**16**) Products[a]

	2	CF_3CO_2H, room temp ⇌	**14**	CF_3CO_2H, room temp ⇌	**16**
concn	0.65 M[b]		~0.52 M		~0.13 M
^{31}P NMR	-6.6[3]		-13.7		-28.2
^1H NMR (OCH_2)	4.05 (dt, ^3J(POCH) = 16.1, ^3J(HH) = 5.8[3])		4.79 (dt, ^3J(POCH) = 17.0, ^3J(HH) = 5.5)		4.79 (dt, ^3J(POCH) = 17.0, ^3J(HH) = 5.5)
^1H NMR (NCH_2)	3.02 (t, ^3J(HH) = 5.8[3])		3.88 (td, ^3J(PNCH) = 1.8, ^3J(HH) = 5.5)		3.93 (td, ^3J(PNCH) = 4.5, ^3J(HH) = 5.5)

[a] Shifts are reported in ppm; coupling constants are in Hz. [b] Initial concentration. No **2** is detectable in the ^1H or ^{31}P NMR spectra under these conditions.

Table II. NMR Data for **3** and Its Mono- (**15**) and Diprotonation (**17**) Products[a]

	3	CF_3CO_2H, room temp ⇌	**15**	CF_3CO_2H, room temp ⇌	**17**
concn	0.42 M[b]		~0.21 M		~0.21 M
^{31}P NMR	60.9[3]		24		-6
^1H NMR (OCH_2)	4.10 (dt, ^3J(POCH) = 16.5, ^3J(HH) = 6.2)		4.78 (dt, ^3J(POCH) = 17.0, ^3J(HH) = 6.0)		4.83 (dt, ^3J(POCH) = 17.0, ^3J(HH) = 6.0)
^1H NMR (NCH_2)	3.01 (t, ^3J(HH) = 6.2)		3.87 (dt, ^3J(PNCH) = 2.0, ^3J(HH) = 6.0)		3.98 (td, ^3J(PNCH) = 5.5, ^3J(HH) = 6.0)

[a] Shifts are reported in ppm; coupling constants are in Hz. [b] Initial concentration. No **3** is detectable in the ^1H or ^{31}P NMR spectrum under these conditions. The ^1H NMR data were taken at ambient temperature. At this temperature and down to -10 °C (where solute precipitates) the ^{31}P peaks were broad. At 60 °C, however, reasonably sharp signals could be observed.

of the solution, impure **20** precipitated first. Further slow evaporation did not permit separation of **11** and unreacted **3**. Evaporation to dryness and slow evaporation of an MeCN solution did afford **11** (R = Me) in >90% purity: ^{31}P NMR (CD_3CN) +5.2; ^{13}C NMR (CD_3CN) 64.2 (d, CH_2O, ^2J(PC) = 13), 49.5 (d, CH_2N, ^2J(PC) = 13), 13.8 (d, SCH_3, ^2J(PC) = 6); ^1H NMR (CD_3CN) 4.38 (dt, CH_2O, ^3J(PH) = 17.0, ^3J(HH) = 6.0), 3.43 (dt, CH_2N, ^3J(PH) = 5.0, ^3J(HH) = 6.0, 2.23 (d, SCH_3, ^3J(PH) = 16.0). Compound **20** was obtained in good purity when the reaction was carried out in the absence of MeCN: ^1H NMR (CD_3CN) 4.63 (dt, OCH_2, ^3J(PH) = 16.0, ^3J(HH) = 6.0), 3.77 (dt, CH_2N, ^3J(PH) = 6.5, ^3J(HH) = 6.0), 2.80 (d, SCH_3, ^3J(PH) = 11.4). Anal. Calcd (Found) for $C_8H_{18}O_3NPSB_2F_8$: C, 23.27 (23.04); H, 4.39 (4.35); N, 3.39 (3.27).

[EtSP(OCH$_2$CH$_2$)$_3$N]BF$_4$ (11, R = Et). To a stirred solution of **3** (1 g, 4.7 mmol) in CH_2CL_2 cooled to -78 °C was added dropwise a solution of 1 g of Et_3OBF_4 in CH_2Cl_2. After the mixture warmed to room temperature, the precipitate which formed was filtered and washed with CH_2Cl_2. Slow evaporation of a MeCN solution of the solid under N_2 afforded crystalline **11** (R = Et) as needles: mp 164.6 °C; ^{31}P NMR (CD_3CN) 5.6; ^{13}C NMR (CD_3CN) 64.1 (d, CH_2O, ^2J(PC) = 13), 49.3 (d, CH_2N, ^3J(PH) = 14), 25.9 (d, SCH_2, ^2J(PC) = 6), 15.3 (d, SCH_2CH_3, ^3J(PC) = 9); ^1H NMR (CD_3CN) 4.28 (dt, CH_2O, ^3J(PH) = 16, ^3J(HH) = 5.8), 3.40 (dt, CH_2N, ^3J(PH) = 5.0, ^3J(HH) = 5.8), 2.77 (dq, SCH_2CH_3, ^3J(PH) = 14.5, ^3J(HH) = 7.2), 1.23 (td, SCH_2CH_3, ^4J(PH) = 1.6, ^3J(HH) = 7.2). Anal. Calcd (Found) for $C_8H_{17}NO_3SBF_4$: C, 29.56 (29.30); H, 5.27 (5.37); N, 4.31 (4.26).

[SP(OCH$_2$CH$_2$)$_3$NH]BF$_4$ (21). To a stirred room temperature solution of 0.5 g of **3** in 15 mL of CH_2Cl_2 and 5 mL of Et_2O was added all at once 1 mL of a 50% aqueous solution of HBF_4. During 1 h of continued stirring, a white solid precipitated. After 10 mL of Et_2O was added the solid was filtered off and washed with CH_2Cl_2. The crude product was recrystallized from hot acetonitrile: mp 205 °C; ^{31}P NMR (CD_3CN) 57.4; ^1H NMR (CD_3CN) 3.44 (m, CH_2N), 4.40 (dm, CH_2O, ^3J(PH) = 16.4) 7.5 (br s, NH). Anal. Calcd (Found) for $C_6H_{13}NO_3PSBF_4$: C, 24.26 (24.60); H, 4.41 (4.51); N, 4.72 (4.91).

Discussion

In Table I the ^{31}P chemical shift of the phosphate **2** is seen to move substantially upfield upon mono- and diprotonation. Such ^{31}P chemical shift changes were previously reported for the monoalkylated cations **10** and **11**[6] and also for the mono- and disilylation products **12** and **13**,[3] respectively. It should be mentioned in this connection that protonation of $OP(OR)_3$ compounds leads to only small (~0–1 ppm) ^{31}P chemical shifts.[8] Transannulation of the nitrogen in **2** to form a TBP structure protonated at the phosphoryl oxygen in **14** and **16** is further supported by (a) a TBP structure for **11** (R = Et) confirmed by X-ray means,[6] (b) coupling of the NCH_2 protons to phosphorus in **14** and **16** which is absent in **2**,[4-6] (c) coupling of phosphorus to the NC carbons in **14** (^{13}C NMR (CF_3CO_2D) 62.8 (d, CH_2O, ^2J(PC) = 6.5), 50.7 (d, CH_2N, ^2J(PC) = 6.3)), (d) the disappearance of the O=P stretching frequency of **2**[5] in TFA, and (e) the lack of detectable change in the OCH_2 proton NMR resonance pattern from **14** to **16**, suggesting that the second protonation does not occur on an endocyclic oxygen (i.e., in a fluxional manner). The increase in coupling of the NCH_2 protons to phosphorus from monopositive **14** to dipositive **16** can be ascribed to the Fermi contact spin–spin coupling term, if this contribution can be assumed to dominate in this three-bond coupling. In the Fermi term, coupling rises with effective positive nuclear charge on the coupling atoms.[9] The additional proton in **16** is expected to induce a higher P–N bond order in **16** than in **14**. To the extent that this process is accompanied by greater s character in the P–N bond, the Fermi contribution to the coupling term is also expected to augment.

The concentrations of **14** and **16** in Table I were approximated from the ^{31}P NMR peak integrals. The high-field ^{31}P NMR resonance is the only one remaining upon dilution to 0.09 M. Similarly, the triplet of doublets at 3.88 ppm in the ^1H NMR spectrum assigned to the NCH_2 protons disappears in favor of the 3.93 ppm triplet of doublets. That the protonations in Table I are entirely reversible is demonstrated by the fact that evaporation of the CF_3CO_2H under vacuum permits the quantitative recovery of **2**.

In view of the ease of disilylation[3] and diprotonation of **2**, we sought evidence for its dialkylation. The ^{31}P NMR spectrum of **2** in CD_3CN in the presence of 1 equiv of Et_3OBF_4 displays peaks at -19 and -9 ppm. Further addition of alkylating agent to a molar equivalent ratio of 65:1 results in preservation of the peak at -19 ppm but an upfield movement of the low-field resonance to -27 ppm, at which point the -19 ppm peak disappears. We thus assign the -19 ppm resonance to monoethylated **10** (R = Et) and the -27 ppm peak to diethylated **18**. These values are also consistent with those recently reported for silylated species **12** (-18.7 ppm) and **13** (-25.6 ppm).[3]

In Table II it is seen that the structures depicted for **15** and **17** can be deduced from the ^{31}P and ^1H NMR data in a manner analogous to that used to arrive at the structures for **14** and **16**. At a concentration of 0.70 M **3**, resonances for **3** (66.3 ppm), **15** (25.9 ppm), and **17** (-5.8 ppm) were seen in a ratio of 1:12:4, respectively. At a concentration of 0.18 M **3**, only **17** (-6.5 ppm) was detected. It is noteworthy that the ^{31}P chemical shift changes for **3** on mono- and diprotonation are substantially larger than those for **2**. This accords with the observation that protonation

(8) Olah, G. A.; McFarland, C. W. *J. Org. Chem.* **1971**, *36*, 1474.

(9) Grant, D. M.; Litchman, W. M. *J. Am. Chem. Soc.* **1965**, *87*, 3994.

of $SP(OR)_3$ leads to ^{31}P shift changes of ~20 ppm.[10] As with CF_3CO_2H solutions of **2**, unreacted **3** can be recovered upon evaporation of the solvent. Interestingly, **21**, in which the nitrogen is protonated, apparently forms as the main product when **3** is allowed to react with aqueous $HBF_4/Et_2O/CH_2Cl_2$.[11] The reason for the difference in behavior of **3** in this medium and in TFA is not clear, though it may be related to stabilizing solvation effects of water on the ammonium cation **21**.

In a 1:1 mixture of CD_3CN/CH_2Cl_2, addition of 1 equiv of Et_3OBF_4 to **3** produces a precipitate which redissolves on addition of a second equivalent of ethylating agent. The two peaks at ca. +6 and −8 ppm are assigned to **11** (R = Et) and **19**, respectively. Interesting in this respect is the isolation of **20**.

The upfield shifts in the ^{31}P NMR resonance from **10** to **18**, **11** to **19**, **12** to **13**, **14** to **16**, and **15** to **17** are consistent with the observation on isoelectronic/isostructural $M[P(OR)_3]_x^{n+}$ complexes wherein $\delta(^{31}P)$ moves upfield linearly with increasing positive charge.[12,13] Another possible rationale for the upfield shift with increasing positive charge in the hypervalent TBP phosphorus cations reported here is that the second proton increases the P–N bond order at the expense of the apical P–O bond order, resulting in a more "balanced" TPB configuration for the phosphorus.[14] This polarization process is related to that observed in 10-S-4 and 10-I-3 species possessing varying electronegativity differences in the apical ligands.[15,16] Thus, the upfield progression of the ^{31}P chemical shift from four- to five-coordinate phosphorus may be maximized by balanced bond orders in the apical bonds.

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Registry No. 2, 10022-55-6; **3**, 60028-25-3; 8-P-4-**10** (R = Me), 76570-87-1; 10-P-5-**10** (R = Me), 81041-54-5; 8-P-4-**10** (R = Et), 76570-89-3; 10-P-5-**10** (R = Et), 81041-56-7; 8-P-4-**11** (R = Me), 76570-91-7; 10-P-5-**11** (R = Me), 81041-58-9; 8-P-4-**11** (R = Et), 76570-93-9; 10-P-5-**11** (R = Et), 81041-60-3; 8-P-4-**14**, 102632-36-0; 10-P-5-**14**, 102632-37-1; 8-P-4-**15**, 102632-39-3; 10-P-5-**15**, 102632-40-6; 8-P-4-**16**, 102632-38-2; 10-P-5-**16**, 102733-94-8; 8-P-4-**17**, 102632-41-7; 10-P-5-**17**, 102733-95-9; 8-P-4-**20**, 102632-34-8; 10-P-5-**20**, 102648-84-0; **21**, 102632-35-9.

(10) Skvortsov, N. K.; Ionin, B. I.; Petrov, A. A. *Z. Obshch. Khim.* **1974**, *44*, 220.
(11) The ^1H NMR spectrum of **21** (see Experimental Section) does not display a clean doublet of triplets (OCH_2) and a triplet (CH_2N) as was observed earlier for the related cation $OP(OCH_2CH_2)_3NMe^+$.[4] Contamination by $(HOCH_2CH_2)_3NH^+$ arising from the hydrolysis of **3** or **21** could explain this observation.
(12) Coskran, K. J.; Bertrand, R. D.; Verkade, J. G. *J. Am. Chem. Soc.* **1967**, *89*, 4535.
(13) Yarbrough, L. W.; Verkade, J. G., unpublished results.
(14) We thank a referee for suggesting this possible explanation.
(15) Livant, P.; Martin, J. C. *J. Am. Chem. Soc.* **1977**, *99*, 5761.
(16) Lam, W. Y.; Duesler, E. N.; Martin, J. C. *J. Am. Chem. Soc.* **1981**, *103*, 127.

Zwitterionic Tetramethylenes as the Common Intermediates in the Cycloaddition and Polymerization Reactions of *N*-Vinylcarbazole with Electrophilic Tetrasubstituted Ethylenes: A New Explanation for "Charge-Transfer" Initiation

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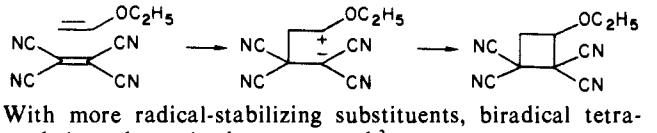
Contribution from the C. S. Marvel Laboratories, Chemistry Department, University of Arizona, Tucson, Arizona 85721. *Received January 7, 1985*

Abstract: The reactions of *N*-vinylcarbazole (NVCZ) with electrophilic tetrasubstituted ethylenes were studied in detail as an example of a reaction whose outcome can be manipulated by changes in concentration, structure, and working procedure to form either small molecules (cyclobutanes, 1-butenes) or poly(vinylcarbazole). Equivalent concentrations and evaporating workup (organic chemists's conditions) lead to small molecules; a large excess of NVCZ and precipitative workup give polymer. The mechanism involves gauche and trans zwitterionic tetramethylenes as intermediates. The former gives cyclobutane reversibly. The latter gives 1-butenes intramolecularly or adds monomers to form cyclohexanes or eventually polymer. The organic chemistry and polymer chemistry is unified on this basis. Extensive stereochemical and kinetics support for these propositions is given. Two other proposed mechanisms for these "charge-transfer" initiations are excluded.

The spontaneous, thermal reaction of electron-rich olefins with electron-poor olefins leads to a rich diversity of both small organic molecules and polymers. The most often encountered small molecules are cyclobutanes. These kinetically favored products isomerize to more thermodynamically favored 1-butene derivatives in more vigorous reaction conditions.

As regards mechanism, organic chemists have proven that tetramethylene intermediates, arising from bond formation between the β-carbons of the reacting olefins, are the keys to small molecules formation. For strongly polar olefins, the penetrating studies of Huisgen[1,2] identified the tetramethylene zwitterion as the intermediate in the cycloaddition reactions, for example,



With more radical-stabilizing substituents, biradical tetramethylenes have also been proposed.[3]

On the polymer side, homopolymers of one or both of the reaction partners or alternating copolymers are formed. To explain these results, Hall recently proposed the Bond-Forming Initiation

(1) Huisgen, R. *Acc. Chem. Res.* **1977**, *10*, 177.
(2) Huisgen, R. *Acc. Chem. Res.* **1977**, *10*, 199.
(3) Bartlett, P. D. *Q. Rev. Chem. Soc.* **1979**, *24*, 473.

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