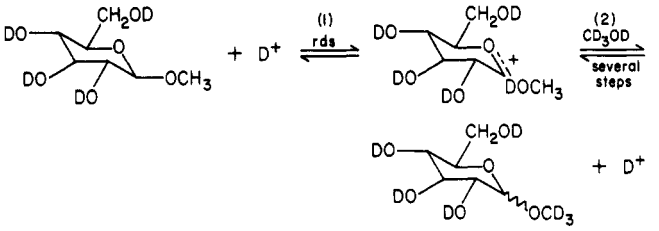
Scheme II

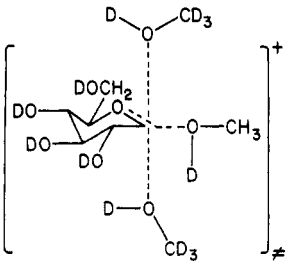


tential formation of the free oxycarbocation; that is, anomerization requires the presence of a nucleophilic agent other than R_1OH. Scheme II[4] summarizes this situation.

At higher methanol concentrations, step 1 would be rate-limiting and only anomerization would be observed; at reduced methanol concentrations, a hydroxyl group of a glucose molecule may react competitively with methanol (step 2) producing maltoside-type products.

Jencks and Sinnott[8] have recently presented evidence that in CH_3CH_2OH/CF_3CH_2OH mixed solvents, solvolysis products of D-glucopyranosyl derivatives cannot be explained on the basis of a mechanism such as Scheme II. One of our "control" experiments supports this conclusion for the Me_2SO/CH_3OH solvent system as well: when the ^1H NMR spectrum of pentamethyl β-D-glucopyranoside[9] was observed in Me_2SO containing 0.92 M D_2SO_4 and 1.2 M CD_3OD, there was no evidence of any reaction over 84.5 hours at 70 °C. This result demonstrates that methylation of the glucoside hydroxyls not only precludes the formation of maltoside-type products, as expected, but it also retards by about two orders of magnitude[10] the rate of anomerization utilizing the 1.2 M CD_3OD. Thus we conclude that not only is the external nucleophile (R_2OH in Scheme I, CD_3OD in Scheme II) involved subsequent to the rate-limiting step in Me_2SO/methanol solutions, but the glucoside hydroxyl groups participate in the reaction.

The mechanism proposed in the extensive study by Jencks and Sinnott[8] proceeds along the course of Scheme II, except the "intermediate" of Scheme II has specific interaction with two solvent molecules and has a lifetime less than 10^{-13} s; i.e., 1 is an activated complex. Our results require the two CD_3OD molecules to be interacting via



1

solvational forces; i.e., these two molecules do not lie inside the solvent cavity, but rather are still part of the solvent cage. Our observation of the rate enhancement due to the glucopyranoside hydroxyls is accounted for, in part, by the interaction of a CD_3OD with the C_2–OD. The complete solvent cage organization established by the four glucopyranoside hydroxyls and how that relates to transition state stabilization is far too complex a subject for studies completed to date; however, the general structure of the transition state for methyl glucopyranoside anomerization seems clear. While reactions of glucopyranosyl derivatives in which the leaving group is poorer than CH_3OD may have more nucleophilic participation by the solvent, the exact mode of anomerization (and probably hydrolysis) of the glucopyranosides appears to involve the specific solvational forces depicted in 1. The recent study of the acetolyses of permethylated glucosides in acetic anhydride,[11] catalyzed by H_2SO_4, may well occur via a mechanism analogous to Scheme II (as proposed) because of the absence of the types of interactions proposed for 1. The results to date support an important interaction between the glycoside hydroxyls and hydroxylic solvent/nucleophiles. The exact details of these interactions (e.g., stereochemical requirements) have yet to be determined.

Acknowledgment. Support by the National Science Foundation, CHE-7907588 and CHE-8421082, is gratefully acknowledged.

Registry No. Methyl α-D-glucopyranoside, 97-30-3; methyl β-D-glucopyranoside, 709-50-2; sulfuric acid, 7664-93-9; methanol, 67-56-1.

Rates of Base-Catalyzed Hydrogen Exchange of Terminal Acetylenes in Aqueous Solution. Absence of a Resonance Interaction

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Rates of detritiation of 13 monosubstituted acetylenes labeled at the acetylenic hydrogen position were measured in aqueous amine buffer solution at 25 °C, and hydroxide ion catalytic coefficients were evaluated. These rate constants, plus a few additional values from the literature, give a good correlation against inductive or field substituent constants: $\log (k_{HO^-}/M^{-1}\,s^{-1}) = 1.46 \pm 0.12 + (8.00 \pm 0.50)\sigma_I$. This correlation is not improved by addition of resonance substituent constants, and the coefficients of the resonance term in two different dual parameter (resonance plus field) treatments of the data are in fact zero.

It is commonly believed that the carbanionic electron pair of acetylide ions resides in an sp hybrid orbital.[1] Since this orbital is orthogonal to the acetylenic π-system, this pair of electrons cannot be delocalized by conjugation with the π-system, and the electron pair is consequently localized on a single atom. In this respect acetylide ions are similar to "normal"[2] oxygen and nitrogen bases, whose

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(1) See: for example, Morrison, R. T., Boyd, R. N. "Organic Chemistry", 4th, ed.; Allyn and Bacon: Boston, 1983; pp 567–568.
(2) Eigen, M. *Angew. Chem., Int. Ed. Engl.* **1964**, *3*, 1–19.

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basic electron pairs are also localized on single atoms, and it is this structural feature of acetylide ions which is thought to be responsible for the normal acid behavior observed for certain acetylenes.[3]

It follows from this idea that the acid strength of monosubstituted acetylenes, ZC≡CH, should be affected only by the inductive or field effects of the substituents, and that resonance interactions should play no role in determining acidity. The acid strengths of acetylenes may be measured by determining rates of acetylenic hydrogen exchange, and there is some evidence that such rates do correlate with inductive substituent constants.[3b] The data, however, are not extensive, and some reservations have been expressed.[4] We have, therefore, examined this issue in greater detail by measuring rates of hydrogen exchange, using tritium as a tracer, for a substantial body of acetylenes. We find that correlations of these data employing inductive substituent constants, and inductive plus resonance substituent constants, provide no evidence of a resonance interaction.

Some of the results detailed here have been reported before in summary form.[5]

Experimental Section

Materials. Propyne was prepared by eliminating hydrogen bromide from 1,2-dibromopropane;[6] 3-fluoro-1-propyne was prepared by displacement of *p*-toluenesulfonate from propargyl tosylate with potassium fluoride;[7] *N,N,N*-trimethylammoniopropyne bromide was prepared by reaction of propargyl bromide with trimethylamine;[8] 3-oxo-1-hexyne was prepared by activated manganese dioxide[9] oxidation of 3-hydroxy-1-hexyne;[10] 3-carbomethoxy-1-propyne was prepared by acid-catalyzed esterification of acetylenecarboxylic acid,[11] which in turn was prepared by decarboxylating acetylenedicarboxylic acid;[12] and cyanoacetylene was prepared by phosphorous pentoxide dehydration of acetylenecarboxamide,[11] which in turn was made by ammonolysis of 3-carbomethoxy-1-propyne.[13] All other materials were best available commercial grades.

Tritiated acetylenes were prepared from the unlabeled materials by exchange with tritiated water in dioxane solution by using either sodium hydroxide or propylamine as catalyst. In the case of ethyne, this reaction was conducted in a metal gas cylinder. Parallel experiments using deuterium oxide, followed by proton NMR examination of the exchanged materials, showed that isotopic labeling under these conditions took place exclusively by replacement of the acetylenic hydrogen. The tritiated substrates were purified by recrystallization or distillation and/or gas chromatography.

Kinetics. Rates of detritiation were measured for the most part as described before,[3d,e] by withdrawing samples of reaction mixture from stoppered flasks or, in the case of moderately volatile substrates, from individual screw-cap vials. Ethyne and propyne, however, were too volatile to be handled in this way, and a special pipetting device,[14] which was an adaptation of a rapid sampling pipet,[15] had to be used. Kinetic samples were quenched in an excess of acid, the substrate was then extracted with toluene, and the toluene extracts were subjected to radiochemical assay. Ethyne, however, escaped from these toluene extracts sufficiently rapidly to give poor kinetic results; in the case of this substrate, therefore, the water remaining after toluene extraction rather than the toluene layer was assayed for radioactivity.

In all cases, the kinetic data conformed to the first-order rate law well, and observed first-order specific rates of detritiation were evaluated by least-squares analysis as slopes of plots of ln CPM vs. time.

Table I. Hydroxide Ion Catalytic Coefficients for Loss of Tritium from Acetylenes Labeled at Acetylenic Hydrogen in Aqueous Solution at 25 °C (Ionic Strength = 0.10 M)

substrate	$k_{HO^-}/M^{-1}\ s^{-1}$
HC≡CT	109
$CH_3C{\equiv}CT$	14.9
$CH_3CH_2C{\equiv}CT$	21.7
$CH_3(CH_2)_2C{\equiv}CT$	20.9
$CH_3(CH_2)_3C{\equiv}CT$	20.4
$CH_3(CH_2)_5C{\equiv}CT$	16.9[a]
$(CH_3)_3CC{\equiv}CT$	40.8
$CH_3(CH_2)_2CHOHC{\equiv}CT$	77.8
$CH_3(CH_2)_2COC{\equiv}CT$	2260
$CH_2FC{\equiv}CT$	364
$CF_3C{\equiv}CT$	89 800
$CH_3O_2CC{\equiv}CT$	46 800
$CNC{\equiv}CT$	3 930 000
$C_6H_5C{\equiv}CT$	244[b]
$4\text{-}NO_2C_6H_4C{\equiv}CT$	830[a]
$(CH_3)_3N^+CH_2C{\equiv}CTBr^-$	1290
$C_6H_5N^+(CH_3)_2CH_2C{\equiv}CTBr^-$	2050[a]

[a] Reference 3e. [b] Reference 3d.

Results

All of the acetylenes studied here were too reactive to permit detritiation rate measurements to be made in sodium hydroxide solutions, and determinations were, therefore, performed in amine buffer solutions. Series of solutions of constant buffer ratio and constant ionic strength (μ = 0.10 M) but varying buffer concentrations were used. Four or five concentrations, whose extremes differed by factors of 5–10, were generally employed. The results are summarized in Table S1.[16]

None of the buffer amines employed (benzylamine, 2,2-dimethoxyethylamine, tris(hydroxymethyl)methylamine, and 3,3,3-trifluoroethylamine) were strongly basic, and hydroxide ion concentrations, therefore, remained constant along each of the buffer solution series. Rate contributions due to hydroxide ion were, therefore, evaluated by extrapolating the data, by using linear least-squares analysis, to zero buffer concentration. Buffer catalysis was generally quite weak, and in some cases, notably for *tert*-butylacetylene, it could not even be detected; the extrapolations were, therefore, along lines of shallow slope, and accurate values of the zero-buffer-concentration intercepts could be obtained. These intercepts were converted into hydroxide-ion catalytic coefficients by dividing them by hydroxide ion concentrations; the latter were obtained by calculation using literature values of amine conjugate acid pK_a's and activity coefficients rec-

(3) (a) Charman, H. B.; Tiers, G. V. D.; Kreevoy, M. M.; Filipovich, G. *J. Am. Chem. Soc.* **1959**, *81*, 3149. (b) Charman, H. B.; Vinard, D. R.; Kreevoy, M. M. *J. Am. Chem. Soc.* **1962**, *84*, 347–352. (c) Halevi, E. A.; Long, F. A. *J. Am. Chem. Soc.* **1961**, *83*, 2809–2814. (d) Lin, A. C.; Chiang, Y.; Dahlberg, D. B.; Kresge, A. J. *J. Am. Chem. Soc.* **1983**, *105*, 5380–5386. (e) Kresge, A. J.; Powell, M. F. *J. Org. Chem.* following paper found in this issue.

(4) Dessy, R. A.; Okuzumi, Y.; Chen, A. *J. Am. Chem. Soc.* **1962**, *84*, 2899–2904. Eaborn, C.; Skinner, G. A.; Walton, D. R. M. *J. Chem. Soc. B* **1966**, 922–925, 989–990.

(5) Powell, M. F.; Peterson, M. R.; Csizmadia, I. G. *THEOCHEM* **1983**, 323–335.

(6) Kobrich, G.; Buck, P. In "Chemistry of Acetylenes"; Viehe, H. G., Ed.; Dekker: New York, 1969; p 24.

(7) Edgell, W. F.; Parts, L. *J. Am. Chem. Soc.* **1955**, *77*, 4899–4902.

(8) Marszak, I.; Guermont, J. P.; Epsztein, R. *Mem. Services Chim. Etat. (Paris)* **1951**, *36*, 301–308; *Chem. Abstr.* **1954**, *48*, 8724g–8725d.

(9) Stork, G.; Tomasz, M. *J. Am. Chem. Soc.* **1964**, *86*, 471–478.

(10) Barvelle, M.; Glenat, R. *Bull. Chim. Soc. Fr.* **1967**, 453–458.

(11) Moureu, M. C.; Bangrand, J. C. *Ann. Chim. (Paris)* **1920**, *14*, 47–58.

(12) Perkin, W. H.; Simonsen, J. L. *J. Chem. Soc.* **1907**, *91*, 816–854.

(13) Zoss, A. O.; Hennion, G. F. *J. Am. Chem. Soc.* **1941**, *63*, 1151–1153.

(14) Powell, M. F. Ph.D. Thesis, University of Toronto, 1981, pp 108–112.

(15) Kresge, A. J.; Mylonakis, S. G.; Sato, Y.; Vitullo, V. P. *J. Am. Chem. Soc.* **1971**, *93*, 6181–6188.

(16) Supplementary material; see paragraph at the end of this paper.

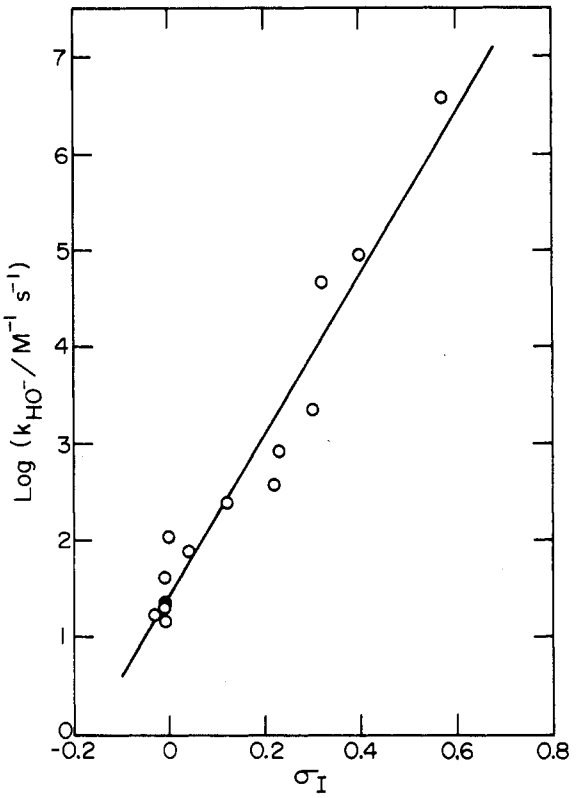

Figure 1. Correlation of specific rates of hydroxide-ion catalyzed detritiation of acetylenes labeled at the acetylenic hydrogen position.

ommended by Bates.[17] The results are listed in Table I.

Discussion

The specific rates of detritiation of monosubstituted acetylenes collected in Table I show a general pattern of reactivity much as expected for an acid ionization reaction: electron-supplying substituents retard the rate of reaction, and electron-accepting groups accelerate it. The strongest electron-accepting groups, moreover, such as cyano and trifluoromethyl, show the greatest effects.

These substituent effects are correlated well by field or inductive substituent constants, σ_I. Values of σ_I are available[18] for 15 of the substituents represented in Table I, and Figure 1 shows the correlation which they provide: log (k_{HO^-}/M^{-1} s^{-1}) = 1.44 ± 0.13 + (8.40 ± 0.56)σ_I, correlation coefficient = 0.972, and standard deviation of log (k_{HO^-}/M^{-1} s^{-1}) = 0.40. This suggests that these substituents are affecting the rates of this reaction through their inductive or field effects and that resonance interactions are playing only a minor role at best.

A more positive indication that this is so may be obtained by fitting the data to a dual parameter relationship, such as that of eq 1, which contains resonance substituent

$$\log k = a + L\sigma_I + D\sigma_R \quad (1)$$

constants, σ_R, in addition to field or inductive constants, σ_I. The regression coefficients L and D of this expression measure the localized (field or inductive) and delocalized (resonance) electrical effects of the substituents, respectively. The resonance substituent constants applicable in the present case are σ_R^-, and values of these are available[18] for 14 of the substituents represented in Figure 1.[19] Least-squares fitting of the 14 data points to eq 1 gives log (k_{HO^-}/M^{-1} s^{-1}) = 1.43 ± 0.14 + (8.65 ± 0.89)σ_I − (0.95 ± 0.99)σ_R^-, correlation coefficient = 0.972, and standard deviation of log (k_{HO^-}/M^{-1} s^{-1}) = 0.39. Comparison of this result with that obtained by using σ_I alone shows that introduction of the resonance substituent constant does not improve the quality of the correlation at all. The magnitude of the regression coefficient D which measures the resonance effect, moreover, is indistinguishable from zero, and the magnitude of the coefficient L, which measures the inductive or field effect, is the same as the coefficient of σ_I obtained in the single parameter treatment.

A similar result is produced when another dual parameter equation, which is the principal alternative to eq 1, is used. In this expression, eq 2,[20] F is a nonresonance or

$$\log k = h + fF + rR \quad (2)$$

"field" substituent constant, R is a resonance substituent constant, and f and r are the regression coefficients which measure the magnitude of the nonresonance and resonance effects, respectively. Values of F and R are not available for as many substituents as are values of σ_I and σ_R^-, and only eight of the groups of Table I are represented.[21] Least-squares analysis of these eight data points gives log (k_{HO^-}/M^{-1} s^{-1}) = 1.58 ± 0.32 + (5.02 ± 1.15)F + (0.005 ± 0.730)R, correlation coefficient = 0.953, and standard deviation of log (k_{HO^-}/M^{-1} s^{-1}) = 0.59. Once again the resonance interaction is of zero magnitude.

These considerations lead to the conclusion that rates of hydrogen exchange of acetylenes, at least the group of acetylenes investigated here, are controlled essentially completely by inductive or field effects of the substituents they contain. This reinforces the idea that these acetylenes, though carbon acids, are, nevertheless, "normal" and not "pseudo" acidic species. Their thermodynamically uphill proton-transfer reactions are, therefore, processes in which the proton-transfer stage is rapid and reversible, and separation of the proton-transfer products is rate-determining, eq 3.

$$ZC{\equiv}CH + B \rightleftarrows ZC{\equiv}C^-{\cdot}HB^+ \xrightarrow[\text{r.d.}]{} ZC{\equiv}C^- + HB^+ \quad (3)$$

It follows from this conclusion that the acidities of these acetylenes are expressed fully in the catalytic coefficients of their hydrogen-exchange reactions, such as the hydroxide ion catalytic coefficients of Table I. These hydroxide ion catalytic coefficients are then at least a relative measure of the acid dissociation constants of the acetylenes, and, if the absolute value of the acid dissociation constant for one of these acetylenes were known, they could be turned into absolute aqueous solution pK_a's. An upper limit for the pK_a of phenylacetylene has recently been estimated: pK_a ≤ 20.0,[3d] and on this basis the upper limit for the most reactive of the acetylenes examined here, cyanoacetylene, is pK_a ≤ 15.8. A value as low as this might be determined directly in aqueous solution; unfortunately

(17) Bates, R. G. "Determination of pH. Theory and Practice"; Wiley: New York, 1973; p 49. Roy, R. N.; Robinson, R. A.; Bates, R. G. *J. Am. Chem. Soc.* **1973**, *95*, 8231–8235.

(18) (a) Charton, M. *Prog. Phys. Org. Chem.* **1981**, *13*, 119–251. The ionic acetylenes (last two entries of Table I) were not included in the correlation, and substituent constants for CH_3CHOH^- and CH_3CO^- were taken as those for $CH_3(CH_2)_2CHOH^-$ and $CH_3(CH_2)_2CO^-$, respectively. (b) σ_I (= 0.22) for CH_2F^- was estimated from the pK_a of CH_2FCO_2H by using a correlation of σ_I and pK_a for monosubstituted acetic acids.

(19) The missing value is for the *n*-butyl group; its absence is unlikely to have an appreciable effect on the correlation because a number of other alkyl groups with similar values of σ_R^- are represented.

(20) Swain, C. G.; Lupton, E. C., Jr. *J. Am. Chem. Soc.* **1968**, *90*, 4328–4337. Swain, C. G.; Unger, S. H.; Rosenquist, N. R.; Swain, M. S. *J. Am. Chem. Soc.* **1983**, *105*, 492–502.

(21) The missing values are for *n*-Pr, *n*-Bu, *n*-hex, RCHOH, CH_2F, CH_3O_2C and *p*-$NO_2C_6H_4$; most of these missing substituents have only weak electron-accepting resonance properties.

cyanoacetylene is highly unstable in the concentrated basic solutions which would be required.

Acknowledgment. We are grateful to the Natural Sciences and Engineering Research Council of Canada and the donors of the Petroleum Research Fund, administered by the American Chemical Society, for their financial support of this research.

Supplementary Material Available: Table S1 (6 pages) of rate data; ordering information is given on any current masthead page.

General Base Catalyzed Hydrogen Exchange of 1-Octyne, 4-Nitrophenylacetylene, and 3-(Phenyldimethylammonio)-1-propyne. Brønsted Relations and Normal Acid Behavior

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Rates of loss of tritium from the title compounds labeled at the acetylenic hydrogen position were measured in aqueous primary amine buffer solutions at 25 °C. These data give Brønsted relations with essentially unit exponents: $\beta = 0.97 \pm 0.06$ for 1-octyne, $\beta = 0.97 \pm 0.05$ for 4-nitrophenylacetylene, and $\beta = 0.98 \pm 0.05$ for 3-(phenyldimethylammonio)-1-propyne, which indicates that these acetylenes are "normal" rather than pseudo carbon acids.

Proton transfers to and from carbon are generally intrinsically slow processes, in contrast to the very rapid proton-transfer reactions of "normal", i.e., oxygen and nitrogen, acids and bases.[1] One of the factors believed to be responsible for this difference is the presence of localized electron pairs at the proton-accepting sites of normal bases; the proton-accepting electron pairs of carbon bases which protonate slowly, on the other hand, are strongly delocalized.

It follows from this idea that carbon bases with localized electron pairs might be protonated rapidly and that such bases and their conjugate acids might, therefore, show normal acid–base behavior. We have recently found support for this hypothesis in the nature of the Brønsted relations for detritiation of phenylacetylene and chloroform catalyzed by a series of amine bases.[2] These hydrogen-exchange processes occur via thermodynamically uphill acid–base reactions, eq 1, which generate carbanions, 1 and

$$RT + B \rightarrow R^- + BT^+ \qquad (1)$$

2, whose basic electron pairs are formally localized on single

$PhC{\equiv}C:^-$ (1) $\qquad Cl_3C:^-$ (2)

atoms. The Brønsted relations for these reactions have unit exponents ($\beta = 1$), just like those for uphill proton transfers from normal oxygen and nitrogen acids to normal oxygen and nitrogen bases. Brønsted exponents for carbon-acid/normal-base reactions which generate delocalized carbanions, on the other hand, are invariably less than unity.

In order to determine just how general this phenomenon is, we have examined the detritiation of other acetylenes. In this paper we report our results for 4-nitrophenylacetylene, **3**, 1-octyne, **4**, and 3-(phenyldimethylammonio)-1-propyne, **5**.

$4\text{-}NO_2C_6H_4C{\equiv}CH$ (3) $\qquad C_6H_{13}C{\equiv}CH$ (4) $\qquad PhN^+(CH_3)_2CH_2C{\equiv}CH$ (5)

Experimental Section

Materials. 3-(Phenyldimethylammonio)-1-propyne was prepared as its bromide salt by the reaction of propargyl bromide with *N,N*-dimethylaniline.[3] 1-Octyne was purchased commercially (ICN Pharmaceuticals) and 4-nitrophenylacetylene was a gift from Professor K. Yates. These acetylenes were tritiated by exchange with tritiated water in dioxane solution by using sodium hydroxide as a basic catalyst. The site of isotopic labeling was verified by doing parallel experiments with D_2O and examining the exchanged material by proton NMR. Tritiated substrates for kinetic determinations were purified by distillation and recrystallization, and their purity was checked by gas chromatography.

Kinetics. Detritiation reactions of 4-nitrophenylacetylene and 3-(phenyldimethylammonio)-1-propyne were conducted in stoppered flasks immersed in a constant-temperature bath operating at 25.0 ± 0.02 °C. 1-Octyne, however, was too volatile to be handled in this way; it partitioned between the vapor and solution phases and, when the flask was opened for sampling, sufficient octyne escaped to give poor kinetic results. Individual portions of reaction mixtures of this acetylene were, therefore, contained in the constant-temperature bath in screw-cap vials; when these vials were filled, care was taken to leave as little vapor space as possible in order to minimize substrate partitioning.

Samples of reaction mixtures were taken by removing 5-mL aliquots from the reaction flasks or individual vials by pipet and then immediately quenching these by addition to an excess of aqueous acid. The quenched solutions were subsequently extracted with accurately pipetted 15-mL portions of toluene, the toluene extracts were dried with anhydrous calcium chloride, and 10-mL aliquots of the dried solutions were added to 10-mL portions of toluene-based counting solution (8 g of 2,5-diphenyloxazole plus 0.1 g of 1,4-bis[2-(5-phenyloxazoyl)]benzene per liter). These solutions were then assayed for radioactivity by using a Packard 314 EX liquid scintillation counter; data were collected for a sufficiently long time to ensure good counting statistics (at least 10^5 counts).

The ionic amine 3-(phenyldimethylammonio)-1-propyne could not be extracted from quenched aqueous solution by toluene in

† Present address: Institute of Pharmaceutical Sciences, Syntex Research, Palo Alto, CA 94304.

(1) Eigen, M. *Angew. Chem., Int. Ed. Engl.* **1964**, *3*, 1–19.
(2) Lin, A. C.; Chiang, Y.; Dahlberg, D. B.; Kresge, A. J. *J. Am. Chem. Soc.* **1983**, *105*, 5380–5386.
(3) Stanko, V. I.; Gol'tyapin, Yu. V.; Klimova, T. P. *Zh. Obsch. Khim.* **1969**, *39*, 1290–1292.

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