gave colorless needles (560 mg), which were further recrystallized from benzene–isopropy alcohol for X-ray measurements: $[\alpha]^{20}{}_D$ +10.43° (*c* 0.3, $CHCl_3$); IR ($CHCl_3$) 2220, 1710, 1580, 1375 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.04, 1.12 (2 s, 3 H), 2.10 (m, 4 H), 2.46 (m, 2 H), 4.72 (q, 1 H), 7.78 (s, 4 H); MS, *m/z* (relative intensity) 365 (M^+, 0.4), 363 (0.4), 323 (0.9), 321 (0.9), 239 (3.7), 236 (3.6), 221 (5.4), 219 (5), 157 (11.5), 155 (12), 144 (39), 127 (50), 98 (34), 85 (100), 55 (25); mol wt calcd for $C_{14}H_{14}D_3O_4S^{79}Br$ 363.01416, found 363.02545; mol wt calcd for $C_{14}H_{14}D_3O_4S^{81}Br$ 365.01416, found 365.02052.

Acknowledgment. Financial assistance by the National Science Foundation (Grant No. CHE 80-25733) is gratefully asknowledged, as is support by NIH Grant No. RR-0711 and NSF Grant No. GP-23633 for the Stanford Magnetic Resonance Laboratory. The X-ray work was supported by NIH Grant No. RR-05716 and the Margaret L. Wendt Foundation of Buffalo, NY. We thank F. Elaine DeJarnette (Buffalo) and Ruth Records (Standord) for technical assistance.

Supplementary Material Available: Table II, nonhydrogen atomic coordinates and isotropic thermal parameters for 18 (1 page). Ordering information is given on any current masthead page.

Proximate Charge Effects. 5.[1] Enthalpies of Solvent Transfer of Reactants and Transition States in the Saponification of Betaine Ethyl Ester

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Received May 23, 1983

The rates and activation enthalpies of the alkaline hydrolysis of betaine ethyl ester [(carbethoxymethyl)-trimethylammonium chloride] were measured in water, in 60 mol % ethanol–water, and in 60 mol % dimethyl sulfoxide–water. Unlike the alkaline hydrolysis of ethyl acetate which exhibits a large increase in ΔH^* on going from water as the solvent to 60 mol % ethanol–water, no such solvent effect was observed in the present case. Calorimetric determination of the enthalpies of solvent transfer revealed that, whereas there was a large desolvation of the ethyl acetate saponification transition state on transfer into aqueous ethanol, no such desolvation of the betaine ethyl ester transition state took place, an observation which had been made earlier about the saponification of acetylcholine. In 60 mol % dimethyl sulfoxide–water, a polar, poor hydrogen bond donor solvent, the ΔH^* for betaine ethyl ester and ethyl acetate saponification were nearly the same as in water as the solvent. Calorimetric determination of the enthalpies of solvent transfer of the saponification transition states of betaine ethyl ester and ethyl acetate from aqueous dimethyl sulfoxide to hydrogen bond donor solvents revealed that the increase in solvation on going to the better solvent was about the same for both esters.

The enthalpy of transfer of the transition state in the saponification of ethyl acetate from water to a less polar solvent, 60 mol % aqueous ethanol, was found[2] to be highly endothermic as can be expected from the desolvation of its highly localized negative charge on transfer to the poorer solvent. By contrast, the enthalpy of transfer of the acetylcholine saponification transition state between the same two solvents was found[2] to be isothermic. This observation was taken as evidence that this latter transition state has a coiled conformation and is internally solvated.

The purpose of the present work was to determine whether this conclusion is justified, that is, whether a saponification transition state which has a proximate positive charge positioned close to the negatively charged carbonyl oxygen will indeed lack the characteristic[2,3] endothermic enthalpy of transfer into a solvent of inferior solvating power. The system chosen for study was betaine ethyl ester which serves this purpose because (1) the principal conformation of its saponification transition state in a poorly solvating solvent (I, Figure 1) has a distance between the two oppositely charged groups which is very close to that found in the coiled transition state in the acetylcholine saponification and (2) it is a "reversed" system compared to acetylcholine, its positive charge being on the acyl rather than on the alkyl side of the ester group, thus eliminating any possible effect which may be due to such positioning.

Table I. Rate Constants[a] for the Alkaline Hydrolysis of Betaine Ethyl Ester

$$^{+}(CH_3)_3NCH_2CO_2C_2H_5 + OH^- \rightarrow {}^{+}(CH_3)_3NCH_2CO_2^- + C_2H_5OH$$

solvent[b]	temp, K	k_2, L mol^{-1} s^{-1}
H_2O	291.16	36.6 ± 4.5
H_2O	323.16	126.6 ± 21.1
0.60 aqueous ethanol	291.16	10.7 ± 1.5
0.60 aqueous ethanol	323.16	39.4 ± 4.2
0.60 aqueous Me_2SO	291.16	12.2 ± 3.1
0.60 aqueous Me_2SO	323.16	38.8 ± 1.1

[a] Extrapolated to zero ionic strength (log k_2 vs. $I^{1/2}$) by using six to ten kinetic runs at concentrations giving ionic strengths of 0.9×10^{-5}–1.5×10^{-4}. The uncertainties are given as the standard deviation of the extrapolated value. [b] Composition of mixed solvents indicated as the mole fraction of the organic component.

Saponification Rates

The rate of the alkaline hydrolysis of betaine ethyl ester (eq 1) was measured at two temperatures in three solvents:

$$^{+}(CH_3)_3NCH_2CO_2CH_2CH_3 + OH^- \rightarrow {}^{+}(CH_3)_3NCH_2CO_2^- + C_2H_5OH \quad (1)$$

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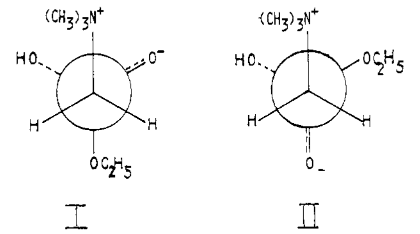

Figure 1. I is the transition state having the greatest degree of intramolecular electrostatic interaction and II the transition state having optimal solvation of the charges sites.

Table II. Activation Parameters for the Alkaline Hydrolysis of Betaine Ethyl Ester and Ethyl Acetate

ester	solvent[a]	$\Delta H^\ddagger$, kcal/mol	$\Delta S^\ddagger$, cal mol^{-1} K^{-1}
betaine ethyl ester	H_2O	6.65[b,f]	−29.1
betaine ethyl ester	0.60 ethanol (aq)	6.98[f]	−30.4
betaine ethyl ester	0.60 Me_2SO (aq)	6.19[f]	−32.3
ethyl acetate[c,d]	H_2O	11.1	−25.8
ethyl acetate[d]	0.60 ethanol (aq)	14.9	−20.0
ethyl acetate[e]	0.60 Me_2SO (aq)	10.9	−24

[a] Solvent composition is indicated as the mole fraction of the organic component. [b] This compares with a literature value at a much higher ionic strength (0.050 M) of 9.7 kcal/mol (Leach, B. E.; Angelici, Robert J. *J. Am. Chem. Soc.* **1968**, *90*, 2504). [c] Tommila, E.; Koivisto, A.; Lyyra, J. P.; Antell, K.; Heims, S. *Acad. Sci. Fennicae, Ser. A* **1952**, *47*, 3. [d] Roberts, D. D. *J. Org. Chem.* **1964**, *29*, 2714. [e] Roberts, D. D. *J. Org. Chem.* **1965**, *30*, 3516. [f] The maximum error in the $\Delta H^\ddagger$ values, propagated from the standard deviations of the k_2 values of Table I, ranged from 1.4 to 1.7 kcal/mol.

H_2O, 60 mol % aqueous ethanol, and 60 mol % aqueous dimethyl sulfoxide (Me_2SO). The results are shown in Table I. Activation enthalpies and entropies which were calculated from these data are listed in Table II. Also shown in Table II, for purposes of comparison, are literature values for the activation parameters of the alkaline hydrolysis of ethyl acetate. The faster rate of hydrolysis of betaine ethyl ester over its uncharged model in water is seen to be an enthalpic effect, as is also the case in the other two solvent systems. Although this is not the same pattern of activation energy changes as was observed in the comparison of acetylcholine with ethyl acetate,[2] there is an important similarity, namely, the increase in the gap between the ΔH^* values of the two esters on going from H_2O to 60 mol % aqueous ethanol (3.5 kcal/mol here and 3.8 kcal/mol in the case of acetylcholine). This could be caused by similar changes in the enthalpies of solvent transfer of the two transition states on addition of the proximate positive charge (vide supra) or by appropriate changes in the solvent transfer enthalpies of the reactants which might coincidentally lead to this similarity.

Enthalpies of Solvent Transfer

A choice between the above two alternatives can be made by measuring the enthalpies of transfer of the reactants in the saponification of betaine ethyl ester from water to aqeous ethanol. The enthalpy of transfer of a substance from one solvent to another, $\delta\Delta H_{S_1\rightarrow S_2}$, is the difference between the heats of solution, ΔH_S, of the substance in the two solvents (eq 2).

$$\delta\Delta H_{S_1\rightarrow S_2} = \Delta H_{S_2} - \Delta H_{S_1} \qquad (2)$$

The enthalpies of transfer of betaine ethyl ester chloride and of ethyl acetate were obtained by this method and are listed in Table III. The enthalpies of solvent transfer of Cl^- and OH^- were taken from our earlier work[4] and are based on the $(n\text{-}C_4H_9)_4N^+(n\text{-}C_4H_9)_4B^-$ extrathermodynamic assumption.[3] The enthalpies of solvent transfer of the betaine ethyl ester cation are obtained by subtracting the Cl^- transfer enthalpies from the corresponding transfer enthalpies of betaine ethyl ester chloride. All of the transfer enthalpies of these compounds and ions are shown in Table III.

Table III. Enthalpies of Solvent Transfer ($\delta\Delta H_{S_1\rightarrow S_2}$) of Compounds and Ions at 25.0 °C

	solvent transfer[a]	$\delta\Delta H_{S_1\rightarrow S_2}$, kcal/mol
betaine ethyl ester chloride	$H_2O \rightarrow$ 0.60 ethanol (aq)	2.47
betaine ethyl ester chloride	$H_2O \rightarrow$ 0.60 Me_2SO (aq)	2.70
Cl^-	$H_2O \rightarrow$ 0.60 ethanol (aq)	1.6[b]
Cl^-	$H_2O \rightarrow$ 0.60 Me_2SO (aq)	1.9[b]
$^+(CH_3)_3NCH_2CO_2C_2H_5$	$H_2O \rightarrow$ 0.60 ethanol (aq)	0.9
$^+(CH_3)_3NCH_2CO_2C_2H_5$	$H_2O \rightarrow$ 0.60 Me_2SO (aq)	0.8
OH^-	$H_2O \rightarrow$ 0.60 ethanol (aq)	−0.5[b]
OH^-	$H_2O \rightarrow$ 0.60 Me_2SO (aq)	13.7[b]
ethyl acetate	$H_2O \rightarrow$ 0.60 ethanol (aq)	3.33[c]
ethyl acetate	$H_2O \rightarrow$ 0.60 Me_2SO (aq)	3.04[d]

[a] Composition of mixed solvent is indicated as the mole fraction of the organic component. [b] From ref 4. [c] From ref 2. [d] From the above transfer enthalpy plus the transfer enthalpy of ethyl acetate from 0.60 aqueous ethanol to 0.60 Me_2SO taken from ref 3.

Table IV. Enthalpies of Solvent Transfer of Reactants and Transition States in the Reaction $RCO_2C_2H_5 + OH^- \rightarrow RCO_2^- + C_2H_5OH$ at 25 °C

	enthalpy, kcal/mol	
solvent transfer[a]	reactants	transition state
$CH_3CO_2C_2H_5 + OH^-$		
$H_2O \rightarrow$ 0.60 ethanol (aq)	2.8	6.6
$H_2O \rightarrow$ 0.60 Me_2SO (aq)	16.7	16.5
$^+(CH_3)_3NCH_2CO_2C_2H_5 + OH^-$		
$H_2O \rightarrow$ 0.60 ethanol (aq)	0.4	0.7
$H_2O \rightarrow$ 0.60 Me_2SO (aq)	14.5	14.0

[a] Composition of mixed solvent is indicated as the mole fraction of the organic component.

Assuming, as before,[2] that the enthalpy of activation for the saponification reaction is approximately equal to the enthalpy of activation for the formation of the tetrahedral intermediate, we then calculate the enthalpy of transfer of the transition state leading to the tetrahedral intermediate from water to the other two solvents. For a single step for which the activation enthalpies in two solvents are known, one can calculate the enthalpy of solvent transfer for the transition state in this step from eq 3, where

$$\delta H_{S_1\rightarrow S_2}{}^{t} = \delta\Delta H_{S_1\rightarrow S_2} + \delta\Delta H^* \qquad (3)$$

$\delta H_{S_1\rightarrow S_2}{}^{t}$ is the enthalpy of transfer of the transition state from solvent 1 to solvent 2, $\delta\Delta H_{S_1\rightarrow S_2}$ is the enthalpy of transfer of the reactants from solvent 1 to solvent 2, and $\delta\Delta H^*$ is the difference between the activation enthalpies

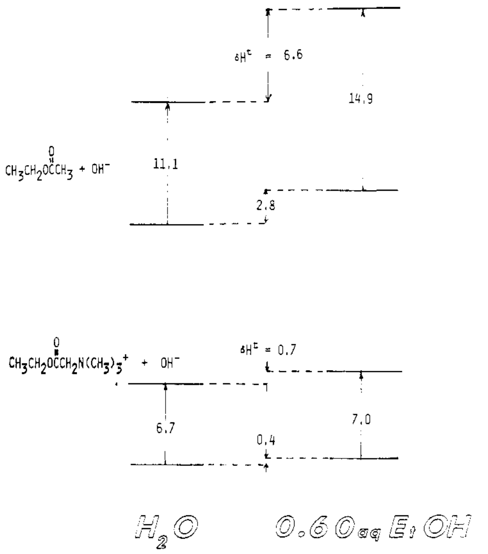

Figure 2. Relative enthalpies (kilocalories/mole) of reactants and transition states in the saponification of ethyl acetate and betaine ethyl ester in water and in aqueous ethanol.

for the reaction in the two solvents. These transition-state solvent-transfer enthalpies, along with the solvent-transfer enthalpies of the reactants, are shown in Table IV.

Solvent transfer enthalpies have two components: (1) the difference between the solute–solvent interactions in the two solvents and (2) the difference between the cavity formation energies in the two solvents. As a measure of the differences in the cavity formation energies in these solvents, we use the enthalpy of transfer of benzene. The enthalpy of transfer of benzene from water to 60 mol % aqueous ethanol was found to be 0.49 kcal/mol, and the enthalpy of transfer of benzene from 60 mol % aqueous enthalpy to 60 mol % aqueous Me_2SO was found to be 0.41 kcal/mol.[4] The enthalpies of transfer of the ethyl acetate transition state as well as that of the betaine ethyl ester transition state from aqueous ethanol to aqueous Me_2SO are seen (Table IV) to be an order of magnitude larger than these differences in the cavity formation energies, and hence these transfer enthalpies can reasonably be discussed in terms of solute–solvent interactions.

The enthalpy of transfer of the betaine ethyl ester transition state from aqueous ethanol to water is essentially nil, unlike that of the uncharged model (see Figure 2). We attribute this to internal solvation of the negatively charged carbonyl oxygen by the neighboring quaternary ammonium group[5,6] (Figure 1), as had been observed in the case of acetylcholine.

The enthalpies of transfer between aqueous Me_2SO and aqueous ethanol, however, are about equally exothermic for transfer into the better H-bonding solvent for both the betaine ethyl ester transition state and its uncharged model (Figure 3). If the large exothermic enthalpy of transfer of the ethyl acetate transition state into the better H-bond donor solvent is due to H bonding to the highly localized charge on the carbonyl oxygen, as seems reasonable, then we can offer no explanation why this effect is not attenuated in the case of the transition state having the neighboring positively charged group.[7]

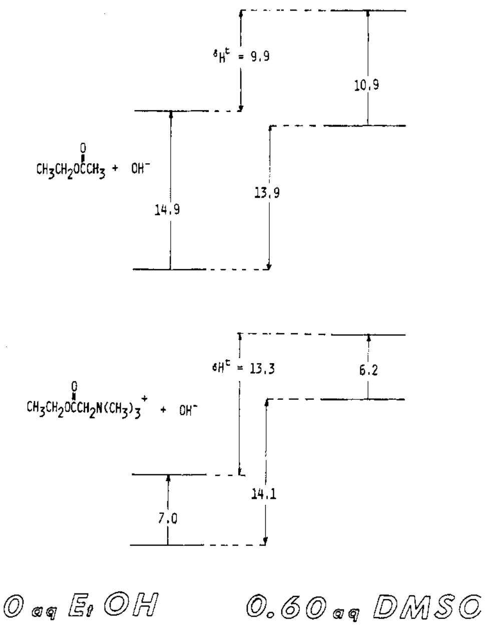

Figure 3. Relative enthalpies (kilocalories/mole) of reactants and transition states in the saponification reaction of ethyl acetate and betaine ethyl ester in aqueous ethanol and in aqueous dimethyl sulfoxide.

Experimental Section

Materials. Dimethyl sulfoxide (Aldrich Chemical Co.) was decanted from calcium hydride and fractionally distilled through a three-foot column at 20 mm pressure. Ethanol (Commercial Solvents Corp.) was used without further purification. Betaine ethyl ester chloride (Eastman) was recrystallized, dried, and titrated with $AgNO_3$ solution.

Solution Preparation. The mixed solvents were prepared from weighed amounts of distilled water and ethanol or Me_2SO. Dissolved carbon dioxide was removed from the solvents by passing purified nitrogen gas through a sintered-glass bubbler immersed in the solvent. All subsequent operations were carried out under a nitrogen atmosphere. Betaine ethyl ester chloride and NaOH solutions were standardized by titration with $AgNO_3$ and HCl, respectively, followed by dilution where necessary.

Rate Measurements. To a sodium hydroxide solution in an all-glass conductance cell immersed in a constant temperature bath was added an equal volume of a betaine ethyl ester chloride solution which had been kept at the same temperature in the same bath. This operation was carried out under an atmosphere of nitrogen. The cell was shaken or stirred to effect complete mixing, and conductance measurements were taken at intervals on a Wayne Kerr bridge (Model B224) at a frequency of 1592 Hz.

The basic hydrolysis of betaine ethyl ester, being a reaction between ions, is subject to a substantial variation in rate as a function of the ionic strength of the solution.[8,9] The stoichiometry of the process (eq 1) involves a continuous and, in the absence of added salts, substantial decrease in the ionic strength as the reaction proceeds. To overcome this difficulty, we measured initial rates of this reaction, the ionic strength being takes as the sum of the initial concentrations of the reactants. The second-order rate constant, k_2, was calculated from eq 4, where $[OH^-]_i$ = the

$$k_2 = \frac{dx/dt}{[OH^-]_i[\text{ester}]_i} \tag{4}$$

initial OH^- concentration and $[\text{ester}]_i$ = the initial betaine ethyl ester concentration. The initial rate, dx/dt, was obtained from eq 5, where L_i = the initial conductance, L_∞ = the final conductance, and L_t = the conductance at time t.

$$x = (L_i - L_t)/(L_i - L_\infty) \tag{5}$$

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[7] It is possible that the highly reactive OH^- present in the 60 mol % Me_2SO-H_2O causes a shift to a much earlier transition state in this solvent (see: Kovach, I. M.; Elrod, J. P.; Schowen, R. L. *J. Am. Chem. Soc.* **1980**, *102*, 7530).

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Table V. Heats of Solution[a] of Betaine Ethyl Ester Chloride

solvent[b]	ΔH_S, kcal/mol
H_2O	1.56 ± 0.15
0.60 ethanol (aq)	4.03 ± 0.04
0.60 Me_2SO (aq)	4.26 ± 0.04

[a] Integral heats of solution were measured at concentrations of 0.001-0.004 M; the error is given as the standard deviation of the mean of three measurements; temperature 25.0 °C. [b] Composition of mixed solvents is indicated as the mole fraction of the organic component.

In each solvent, initial rates were measured at initial concentrations which had ionic strengths ranging from 0.9×10^{-5} to 1.5×10^{-3}. To obtain a rate constant for the pure solvent, that is, at zero ionic strength, we extrapolated the rate constants obtained from the above initial rates to zero ionic strength by plotting ln k_2 vs. $I^{1/2}$. This gave a linear plot, the intercept being the k_2 at zero ionic strength, and the standard deviation of this intercept being taken as the error of this k_2 value (both tabulated in Table I).

Product Verification. Reaction solutions of betaine ethyl ester chloride and NaOH (10^{-2} M) in H_2O and in 60 mol % aqueous dimethyl sulfoxide as the solvent were subjected to VPC analysis (6-ft Porapak Q column, 150 °C, followed by 220 °C to elute the Me_2SO). In each case, the volatile product was found to be ethanol. No methanol was detected.

Heats of Solution. The integral molar heat of solution of betaine ethyl ester chloride was measured in the three solvents of interest with a LKB-8700 calorimeter (see Table V).

Acknowledgment. This work was supported in part by a grant from the City University of New York Faculty Research Award Program.

Registry No. Betaine ethyl ester chloride, 3032-11-9.

Photosensitized (Electron-Transfer) Rearrangements of Cinnamyl Alcohol Derivatives

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Received March 8, 1983

Specific electron-donor compounds are capable of photosensitizing the rearrangement and solvolysis of several cinnamyl alcohol derivatives. Under suitable conditions, the formation of rearranged α-phenylallyl derivatives occurs in synthetically useful yields. The intermediates in this process have ionic character and have been trapped by methanol as methyl ether derivatives. The ability of the photosensitizers to effect these electron-transfer-initiated reactions correlates well with the enthalpy of reaction calculated by the Weller equation.

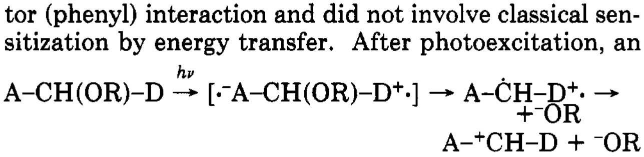
Introduction

The reactions of organic molecules with photoexcited sensitizers leading to electron-transfer processes or to exciplex formation have received considerable attention in recent years. In many of these instances, electronic energy transfer is endothermic and the processes leading to new product formation are dependent upon the electron-donating and -accepting properties of the quencher and the excited state. Solvent polarity, excitation energies, and redox potentials of the reaction partners play a critical role in determining if such processes may occur.[1-4] The free energy (ΔG) associated with these processes can be calculated by using the expression developed by Weller[5] (eq 1). In this expression E_D^{oxidn} and E_A^{redn} are the electro-

$$\Delta G = (E_D^{\text{oxidn}} - E_A^{\text{redn}}) - \Delta E(S^*) \qquad (1)$$

chemically determined potentials for the one-electron oxidation of the donor and reduction of the acceptor, respectively. $\Delta E(S^*)$ is the 0–0 transition energy of the excited state of the light-absorbing sensitizer for this process.

A number of reports have appeared recently in which photoexcited donor molecules affect ionic-like reactions with suitable acceptor substrates. Arnold[6] has shown that electron-donating photosensitizers will catalyze the addition of alcohols and of HCN to olefins when the requirements of the Weller equation (eq 1) are met. Similarly, Yonemitsu[7] reported the photochemical removal of an *N*-tosyl protecting group from *p*-toluenesulfonamides by using electron-donating sensitizer compounds. In 1976, Ullman and co-workers described the photosolvolysis of certain benzyl alcohol derivatives.[8] These reactions appeared to require an intramolecular electron donor (dimethylamino or *p*-(dimethylamino)phenyl) and an acceptor (phenyl) interaction and did not involve classical sensitization by energy transfer. After photoexcitation, an



$$\text{A-CH(OR)-D} \xrightarrow{h\nu} [\cdot^{-}\text{A-CH(OR)-D}^{+}\cdot] \rightarrow \underset{+\,^{-}\text{OR}}{\text{A-}\dot{\text{C}}\text{H-D}^{+}\cdot} \rightarrow \text{A-}^{+}\text{CH-D} + {}^{-}\text{OR}$$

intermediate phenyl radical anion ($A^{-}\cdot$) is generated that expels acetate or hydroxide ion to give a benzyl radical. This new intermediate is then reoxidized by the electron-deficient donor (radical cation, $D^{+}\cdot$) to generate a ground-state carbenium ion that produces the observed products. Cristol et al.[9] have reported several examples of photochemically induced Wagner–Meerwein rearrangements and attendant photosolvolyses in allylic substrates capable of intramolecular electron transfer.

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