9S, which was reduced directly. To a solution of **9S** (59 mg) in EtOH (2 mL) was added $NaBH_4$ (88 mg, 2.3 mmol). The reaction mixture was refluxed for 20 h and then neutralized by addition of Amberlite IR-120 (H^+). The resin was removed by filtration, and the filtrate was concentrated in vacuo. The residue was chromatogaphed on silica gel (20 g, EtOH/toluene, 1:20), and the fractions corresponding to R_f 0.40 (EtOH/toluene, 1:5) were concentrated to give **10S** (38 mg, 54%), a colorless oil; **3S** (462 mg) was also converted into **10S** (201 mg, 63% yield from **3S**) when **8S** was not purified by silica gel chromatography. **10S**: $[a]^{23}{}_D$ +34.0° (c 0.81); IR $\nu_{max}{}^{neat}$ 3440, 2990, 1640 cm^{-1}; ^1H NMR (90 MHz) δ 1.10 (d, 3 H, J = 7 Hz, $CHCH_3$ of the side chain at C-4), 1.34, 1.53 (each s, each 3 H, $C(CH_3)_2$), 1.65–1.98 (m, 1 H, $CHCH_3$ of the side chain at C-4), 2.10–2.93 (m, 2 H, 2 OH), 3.48–4.23 (m, 5 H, H-5, CH_2OH at C-5, CH_2OH of the side chain at C-4), 4.62 (d, 1 H, J = 4 Hz, H-3), 5.11 (dd, 1 H, J = 2 and 18 Hz, $CH{=}CH_2$), 5.30 (dd, 1 H, J = 2 and 12 Hz, $CH{=}CH_2$), 5.82 (d, 1 H, J = 4 Hz, H-2), 6.01 (dd, 1 H, J = 12 and 18 Hz; $CH{=}CH_2$); high-resolution mass spectrum calcd for $C_{12}H_{19}O_5$ m/z 243.1232, found, M − CH_3, 243.1235.

Compound **8R** (838 mg, 2.9 mmol) was converted into **10R** via **9R** as described in the preparation of **10S**; **10R** (659 mg, 88%) was obtained as a colorless oil: TLC R_f 0.19 (EtOH/toluene, 1:5); $[\alpha]^{30}{}_D$ +23.1° (c 1.22); IR $\nu_{max}{}^{neat}$ 3400, 2990, 1650 cm^{-1}; ^1H NMR (90 MHz) δ 1.02 (d, 3 H, J = 7 Hz, $CHCH_3$ of the side chain at C-4), 1.33, 1.53 (each s, each 3 H, $C(CH_3)_2$), 1.83–2.06 (m, 1 H, $CHCH_3$ of the side chain at C-4), 2.32–2.64 (m, 2 H, 2 OH), 3.37–4.08 (m, 4 H, CH_2OH at C-5, CH_2OH of the side chain at C-4), 4.27 (dd, 1 H, J = 4 and 7 Hz, H-5), 4.66 (d, 1 H, J = 4 Hz, H-3), 5.10 (dd, 1 H, J = 2 and 18 Hz, $CH{=}CH_2$), 5.29 (dd, 1 H, J = 2 and 11 Hz, $CH{=}CH_2$), 5.84 (d, 1 H, J = 4 Hz, H-2), 6.05 (dd, 1 H, J = 11 and 18 Hz, $CH{=}CH_2$); high-resolution mass spectrum calcd for $C_{13}H_{23}O_5$ m/z 259.1544, found, M + H, 259.1575.

(1*R*,3*S*,7*S*,8*S*,9*R*)-7,11,11-Trimethyl-8-vinyl-2,5,10,12-tetraoxatricyclo[7.3.0.0^{3,8}]dodecane (11S). A solution of **10S** (201 mg, 0.78 mmol) in pyridine (10 mL) containing TsCl (444 mg, 2.33 mmol) and DMAP (56 mg, 0.46 mmol) was heated at 60 °C for 16 h. The solution was then concentrated in vacuo, and the residue was partitioned between AcOEt (50 mL) and water (30 mL). The aqueous phase was extracted with AcOEt (50 mL × 2), and the combined extracts were dried (Na_2SO_4) and concentrated in vacuo. The residue was chromatographed on silica gel (AcOEt/hexane, 1:10), and the fractions corresponding to R_f 0.30 (AcOEt/hexane, 1:5) were concentrated to give **11S** (138 mg, 74%) as needles: mp 62–63 °C; $[\alpha]^{30}{}_D$ +1.6° (c 1.20); IR $\nu_{max}{}^{neat}$ 2970, 1640 cm^{-1}; ^1H NMR (400 MHz) δ 0.86 (d, 3 H, J = 7.3 Hz, CH_3-7), 1.34, 1.50 (each s, each 3 H, 2 CH_3-11), 1.73 (ddq, 1 H, $J_{CH_3,7}$ = 7.3 Hz, $J_{6_{ax},7}$ = 11.2 Hz, $J_{6_{eq},7}$ = 4.0 Hz, H-7), 3.11 (t, 1 H, $J_{6_{ax},7} = J_{6_{ax},6_{eq}}$ = 11.2 Hz, H-6_{ax}), 3.56 (dd, 1 H, $J_{6_{eq},7}$ = 4.0 Hz, $J_{6_{eq},6_{ax}}$ = 11.2 Hz, H-6_{eq}), 3.72 (d, 1 H, $J_{3,4_{ax}}$ = 0 Hz, $J_{4_{ax},4_{eq}}$ = 11.8 Hz, H-4_{ax}), 4.14 (s, 1 H, $J_{3,4_{ax}} = J_{3,4_{eq}}$ = 0 Hz, H-3), 4.15 (d, 1 H, $J_{3,4_{eq}}$ = 0 Hz, $J_{4_{ax},4_{eq}}$ = 11.8 Hz, H-4_{eq}), 4.47 (d, 1 H, $J_{1,9}$ = 3.4 Hz, H-9), 5.16 (dd, 1 H, J = 1.0 and 18.1 Hz, $CH{=}CH_2$), 5.42 (d, 1 H, J = 11.2 Hz, $CH{=}CH_2$), 5.88 (dd, J = 11.2 and 18.1 Hz, $CH{=}CH_2$), 5.91 (d, 1 H, $J_{1,9}$ = 3.4 Hz, H-1); high-resolution mass spectrum calcd for $C_{13}H_{21}O_4$ m/z 241.1437, found, M + H, 241.1425. Anal. Calcd for $C_{13}H_{20}O_4$: C, 64.98; H, 8.39. Found: C, 65.13; H, 8.22.

(1*R*,3*S*,7*R*,8*S*,9*R*)-7,11,11-Trimethyl-8-vinyl-2,5,10,12-tetraoxatricyclo[7.3.0.0^{3,8}]dodecane (11R). By the procedure analogous to that described for the preparation of **11S**, **10R** (81 mg) was converted into **11R** (52 mg, 69%) as needles: mp 59–60 °C; TLC R_f 0.80 (EtOH/toluene, 1:5); $[\alpha]^{29}{}_D$ +111.3° (c 0.95); IR $\nu_{max}{}^{neat}$ 2960, 1635 cm^{-1}; ^1H NMR (400 MHz) δ 1.22 (d, 3 H, J = 7.3 Hz, CH_3-7), 1.33, 1.51 (each s, each 3 H, 2 CH_3-11), 1.77 (dq, 1 H, $J_{CH_3,7}$ = 7.3 Hz, $J_{6_{ax},7}$ = 1.0 Hz, $J_{6_{eq},7}$ = 0 Hz, H-7), 3.52 (d, 1 H, $J_{6_{eq},7}$ = 0 Hz, $J_{6_{ax},6_{eq}}$ = 10.7 Hz, H-6_{eq}), 3.60 (dd, 1 H, $J_{6_{ax},7}$ = 1.0 Hz, $J_{6_{ax},6_{eq}}$ = 10.7 Hz, H-6_{ax}), 3.62 (dd, 1 H, $J_{3,4_{ax}}$ = 1.9 Hz, $J_{4_{ax},4_{eq}}$ = 13.2 Hz, H-4_{ax}), 4.03 (d, 1 H, $J_{3,4_{ax}}$ = 1.9 Hz, $J_{3,4_{eq}}$ = 0 Hz, H-3), 4.13 (d, 1 H, $J_{3,4_{eq}}$ = 0 Hz, $J_{4_{eq},4_{ax}}$ = 13.2 Hz, H-4_{eq}), 4.49 (d, 1 H, $J_{1,9}$ = 3.4 Hz, H-9), 5.11 (d, 1 H, J = 17.5 Hz, $CH{=}CH_2$), 5.38 (d, 1 H, J = 11.7 Hz, $CH{=}CH_2$), 5.81 (d, 1 H, $J_{1,9}$ = 3.4 Hz, H-1), 5.83 (dd, 1 H, J = 11.7 Hz and 17.5 Hz, $CH{=}CH_2$); high-resolution mass spectrum calcd for $C_{13}H_{21}O_4$ m/z 241.1438, found, M + H, 241.1428. Anal. Calcd for $C_{13}H_{20}O_4$: C, 64.98; H, 8.39. Found: C, 65.12; H, 8.16.

Medium Effects as a Criterion for Reaction Mechanism. Application of the SWAG Procedures to the Mechanism of the Neutral Hydrolysis of [(*p*-Nitrophenyl)sulfonyl]methyl Perchlorate

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Introduction

According to the Savage–Wood additivity of group interactions (SWAG) procedure,[1,2] the thermodynamic properties of dilute aqueous solutions can be described in terms of pairwise solute–solute interaction parameters and, at the next level of sophistication, in terms of pairwise group interaction parameters. Recently we used these descriptions to quantify the effects of added cosolvents on the pseudo-first-order rate constants for the water-catalyzed hydrolysis of activated esters[3] and amides[4] in highly aqueous binary mixtures. In terms of the SWAG analysis, the effect of a cosolvent on the pseudo-first-order rate constant is expressed as:

$$\ln (k_{obsd}/k^0{}_{obsd}) = (2/RT)(1/m^0)^2(g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS})m_A - n\phi m_A M_1 \quad (1)$$

where k_{obsd} is the rate constant in the water-rich aqueous solution (molality of cosolvent, m_A), $k^0{}_{obsd}$ is the rate constant in water ($m_A = 0$), $m^0 = 1$ mol kg^{-1}, n is the order of the reaction with respect to water, ϕ is the practical osmotic coefficient and M_1 is the molar mass of water. The terms $g_{A\leftrightarrow IS}$ and $g_{A\leftrightarrow TS}$ are the pairwise cosolvent–initial state and cosolvent–transition state Gibbs energies of interaction. For chemical reactions, self-consistent sets of Gibbs energy interaction parameters were developed[3,4] describing interactions between functional groups (e.g. CH_2 and OH) in cosolvents and in key groups within both initial and transition states. The success of the analysis suggested the possibility of using the SWAG approach in studies of reaction mechanism. The present study is the first endeavor along these lines, using as a model reaction the water-catalyzed hydrolysis of [(*p*-nitrophenyl)sulfonyl]methyl perchlorate (1).

Results and Discussion

Since the first synthesis of a covalent (arylsulfonyl)methyl perchlorate,[5,6] considerable attention has been paid to the mechanism of the hydrolysis[5] and to medium effects on the hydrolytic process.[7–10] Hydrolysis does not proceed via nucleophilic displacement of the perchlorate moiety but involves general base catalysis via rate-determining deprotonation at the α-sulfonyl carbon atom. The transition state is symmetric as indicated by large, primary kinetic deuterium isotope effects ($k_H/k_D \approx 6$) and the Brønsted β value of 0.51. One mechanism for the water-catalyzed reaction is outlined in Scheme I. Medium effects on the hydrolysis induced by alcohols, 1,3- and 1,4-dioxane,[7–9] and electrolytes[10] have been interpreted pre-

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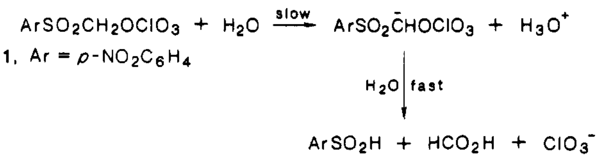
Table I. Pseudo-First-Order Rate Constants for Hydrolysis of 1 in H_2O–D_2O Mixtures at 25 °C

n^a	$k_{obsd} \times 10^3$, s^{-1}	$k_{calcd} \times 10^{3,b}$ s^{-1}
0	3.33	
0.18	2.92	3.00
0.38	2.60	2.68
0.57	2.41	2.37
0.78	2.05	2.07
1.00	1.79	

[a] Atom fraction of D in the solvent. [b] Calculated using eq 2.

Scheme I



viously in terms of changes in the kinetic basicity of water. Before applying the SWAG procedure in the analysis of the medium effects of 1,4-dioxane, *s*-trioxane, and THF, a proton inventory study[11] confirmed that the hydrolysis is first order in water.

Proton Inventory Study. By use of the standard approach,[11,12] pseudo-first-order rate constants for the hydrolysis of 1 were measured in H_2O–D_2O mixtures as a function of the atom fraction of D (Table I) and analyzed by the Gross–Butler equation (eq 2).

$$k_n = k^0 \prod_{i}^{TS}(1 - n + n\phi^*_i) / \prod_{j}^{IS}(1 - n + n\phi_j) \qquad (2)$$

In eq 2 k_n and k^0 are the pseudo-first-order rate constants for hydrolysis in the H_2O–D_2O mixture and in H_2O, respectively. The contribution of TS is dependent on the exchangeable TS protons i with a fractionation factor ϕ^*_i. Similarly for the initial state protons j, the fractionation factor is ϕ_j.

In the case of hydrolysis of 1, there is no proton exchange in the initial state. The water protons have unit fractionation factors, and so the denominator of eq 2 is unity. Since water is on the Brønsted plot,[5] ϕ^*_i can be calculated[11] by using β. Observed and calculated rate constants are given in Table I. The linear correlation between k_n and n indicates that only one proton contributes to the observed primary kinetic deuterium isotope effect. This conclusion is consistent[5,10] with a mechanism in which a single water molecule in the transition state acts as a general base.

Application of the SWAG Analysis. On the basis of the proton inventory study and previous mechanistic evidence, the activation process can be represented by the following equation:

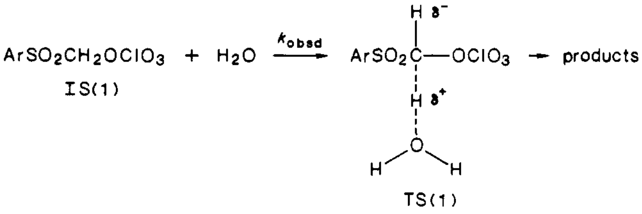
Table II. Pairwise Group Gibbs Function Interaction Parameters

i	j	$G_{i \leftrightarrow j}$,^{a} J kg^{-1}	$G^*_{i \leftrightarrow j}$,^{b} J kg^{-1}
CH_2	CH_2	−34	
CH_2	OH	+29	+47
OH	OH	−23	−81
O	OH	−22	−31
O	CH_2	+37	

[a] References 13 and 14. [b] Reference 4.

Table III. Pseudo-First-Order Rate Constants for Hydrolysis of 1 in Aqueous Binary Mixtures at 25 °C

cosolvent	m_A, mol kg^{-1}	$k_{obsd} \times 10^3$, s^{-1}
–	0	3.33
1,4-dioxane	0.23	3.46
	0.45	3.80
	0.70	4.30
	0.94	4.45
	1.17	4.55
	1.40	4.90
	1.63	5.03
	1.87	5.72
	2.04	5.84
	2.64	6.63
s-trioxane	0.15	3.49
	0.46	3.59
	0.62	3.63
	0.75	3.74
	0.97	3.82
	1.06	4.03
THF	0.39	3.74
	0.50	3.81
	0.62	3.94
	0.95	4.26
	1.07	4.37
	1.15	4.46
	1.18	4.52
	1.35	4.71
	1.43	5.03



In this mechanism the transition state is labeled TS(1). In TS(1) one C–H bond is partially broken and two polarized OH groups are exposed to the reaction medium. To a good and first approximation, the SWAG analysis considers those chemical bonds or groups for which the solvation is changed substantially during the activation process. Some relevant estimates[4,13,14] for $G_{i \leftrightarrow j}$ are given in Table II. For hydrolysis of 1, $g_{A \leftrightarrow IS} - g_{A \leftrightarrow TS}$ is equivalent to $^1/_2 G_{A \leftrightarrow CH_2} - 2G_{A \leftrightarrow OH}$. Addition of an apolar cosolvent should stabilize the IS and destabilize the TS; consequently, the rate constants should decrease. Rate constants for hydrolysis of 1 in water-rich mixtures of 1,4-dioxane, *s*-trioxane, and THF are listed in Table III. Plots of ln (k_{obsd}/k^0_{obsd}) vs m_A are linear (Figure 1) and the slopes of these plots (SL) are given in Table IV. In all cases the rate constants *increase* upon addition of the cosolvent, the effect increasing in the series *s*-trioxane < THF ≲ 1,4-

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Table IV. Experimental and Calculated Values of ($g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS}$) for Hydrolysis of 1 in Some Aqueous Binary Mixtures at 25 °C

cosolvent	SL	*r*	$g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS}$, J kg^{-1} (exp)	$g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS}$,[a] J kg^{-1} (calcd for TS(1))	$g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS}$,[a] J kg^{-1} (calcd for TS(2))
1,4-dioxane	0.26	0.9907	+322	−175	+72 (+129)
THF	0.25	0.9978	+309	−237	+94 (+160)
s-trioxane	0.15	0.9688	+186	−38	+21 (+51)

[a] Calculated using pairwise group Gibbs function interaction parameters taken from ref 13 and 14. Values in parentheses obtained by using interaction parameters taken from ref 4.

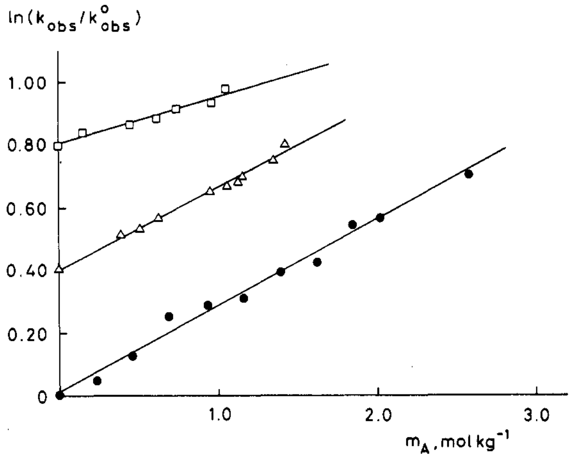
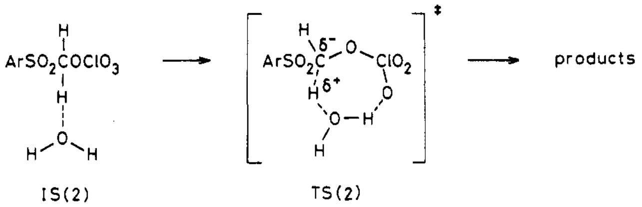


Figure 1. Plots of ln (k_{obsd}/k^0_{obsd}) vs molality of cosolvent for hydrolysis of 1 in aqueous solutions of 1,4-dioxane (●), THF (△), and *s*-trioxane (□) at 25 °C. The plots of THF and *s*-trioxane are shifted upward along the vertical axis by 0.4 and 0.8 units, respectively.

dioxane. The SWAG analysis, based on the transition state model TS(1), clearly does not predict the rate-enhancing effects of these cosolvents. Table IV lists experimental values of $g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS}$ calculated from the kinetic data. The differences are large and positive, contrasting with the negative values calculated using pairwise group interaction parameters taken from Table II. The differences are so large that they cannot be attributed either to the approximations inherent in the SWAG analysis or to uncertainties in the $G_{i\leftrightarrow j}$ parameters. This leads us to propose a more refined scheme for the activation process (Scheme II), which allows an alternative interpretation of the medium effects compared with one previously couched in terms of water polarization by the cosolvents.[7-9] We assume that the IS is preactivated[15] by hydrogen bonding of the acidic α-sulfonyl hydrogen atoms to water[19,20] (IS(2)) and that, via a fluctuation in the hydration shell, a cyclic transition state (TS(2)) is formed in which an oxygen atom of the perchlorate function assists in the stabilization of the partially formed hydronium ion.[21] In the activation process one OH group becomes shielded from interaction with the reaction medium and, therefore, OH polar interactions become weaker when the IS(2) forms TS(2). The values calculated for ($g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS}$) are now positive (Table IV), in accord with the experimental values, and reflect the decrease in hydrophilicity upon transferring IS(2) into TS(2). The observation that the cosolvent-induced increase of the rate follows the sequence of cosolvent hydrophobicity can now be rationalized. The new scheme can be criticized on the basis of the numerical differences between the experimental and calculated values for ($g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS}$) in the SWAG analysis. The following factors probably account for these differences: (i) the strong polarization of the OH bonds exposed to the solvent in IS(2) and TS(2), (ii) a second C–H⋯OH$_2$ hydrogen bond in IS(2) is most likely lost in TS(2), and (iii) one of the oxygens of the perchlorate moiety is shielded from interaction with the solvent in TS(2). These factors, if incorporated properly in the analysis, would all tend to increase the calculated ($g_{A\leftrightarrow IS} - g_{A\leftrightarrow TS}$). It appears clear, however, that application of the SWAG procedure can afford quantitative data, which allows a choice between two (or more) mechanistic possibilities for an organic reaction in water-rich reaction media.

Scheme II



Experimental Section

Materials. The covalent perchlorate 1 was prepared according to the literature procedure.[5] The water used in all experiments was demineralized and distilled twice in an all-quartz distillation unit. Deuterium oxide (Merck AG, uvasol quality, 99.75% D$_2$O) was used as received. 1,4-Dioxane and THF (Merck AG, uvasol) were filtered through active neutral alumina and stored at 0 °C under nitrogen. *s*-Trioxane (Merck AG) was used as such. All solutions were made up by weight. Aqueous solutions of *s*-trioxane were prepared using an ultrasonic bath.

Kinetic Measurements. These were performed at 235 nm by the method described previously[10] (Varian Cary 210 spectrophotometer, connected to an Apple computer). Substrate concentrations were 10^{-4}–10^{-5} M. In all cases the pH of the reaction media was 3 in order to suppress catalysis by hydroxide ions.

Registry No. 1, 26452-84-6; D$_2$, 7782-39-0.

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[15] In the past, much evidence has been obtained for preassociation in proton transfer reactions[16,17] and in nucleophilic substitutions[18] in water.

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[19] The propensity for hydrogen bonding is supported by (i) the large solvent dependence of the ^{1}H NMR methylene resonance, (ii) strong intermolecular C–H⋯sulfonyl hydrogen bonding in the crystalline state, and (iii) high values (>3000 cm^{-1}) for the (asymmetrical) C–H stretching vibrations. See: (a) Reference 5. (b) Bruggink, A. Ph.D. Thesis, University of Groningen, 1971. (c) Reference 6.

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[21] Alternatively, one could assume hydrogen bonding of water to sulfonyl oxygen instead of perchlorate oxygen. However, this is less likely for steric reasons and because negative charge on the α-sulfonyl carbon may be partly delocalized into the chlorate leaving group.