Isotope Effects in the Solvolysis of Sterically Hindered Arenesulfonyl Chlorides

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ABSTRACT: Solvent isotope effects in the ethanolysis of sterically hindered arenesulfonyl chlorides ruled out a proton transfer in the rate-determining step and agreed with a S_N2 mechanism involving at least a second solvent molecule in the transition state (TS). The lack of a secondary kinetic isotope effect in the *o*-alkyl groups allows us to disregard the possible contribution of σ–π hyperconjugation. The measured activation parameters are consistent with a S_N2 mechanism involving the participation of solvent molecules in the TS, possibly forming a cyclic TS through a chain of solvent molecules. © 2015 Wiley Periodicals, Inc. Int J Chem Kinet 47: 744–750, 2015

INTRODUCTION

Despite the existence of a large corpus of research on solvolytic processes near sulfonyl centers [1–23], details on the mechanism of this nucleophilic substitution remain unclear. A number of authors have proposed S_N2 [2–6,9–15] or borderline S_N1–S_N2 mechanisms for this process [16–19]. However, sterically hindered derivatives of aromatic sulfonic acids that contain *o*-alkyl groups show kinetic features that pose questions on the classical view of the bimolecular substitution mechanism [4–8,12].

o-Alkyl derivatives of benzenesulfonyl chlorides show increased reactivity in solvolysis processes [4–8,12]. In this respect, as shown in Scheme 1, the feasibility of uni- [6] and bimolecular mechanisms [20], structural modifications of the S_N2-type transition state (TS) involving a second molecule of nucleophile (S_N3-mechanism) [2,21,22], the stabilization of the bimolecular TS by intramolecular interactions, hyperconjugation effects [23], or stereochemical rearrangements during the nucleophilic attack [4,8] have been discussed.

In addition, the effect of substitution at sterically hindered sulfonyl center is usually considered a minor effect, which is not always the case.

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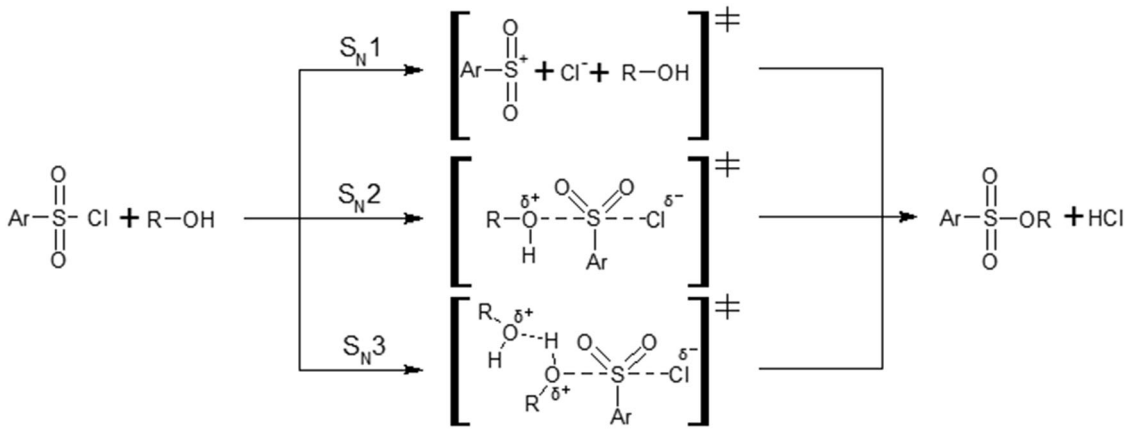
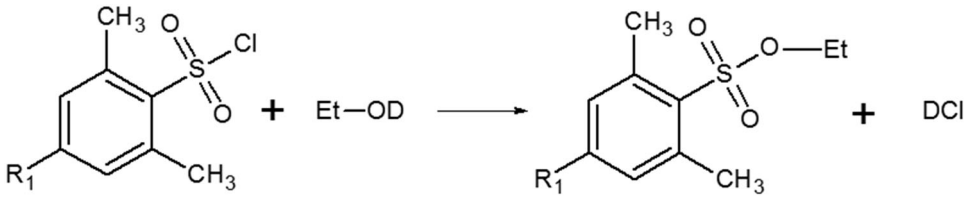
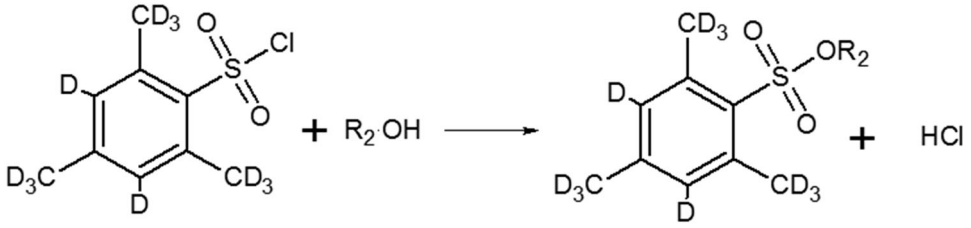


Scheme 1 Mechanistic possibilities for solvolytic processes at a sulfonyl center.



Scheme 2 SIE on the ethanolysis of sterically hindered arenesulfonyl chlorides (R_1 = Me(**2**), *t*-Bu(**1**)).



Scheme 3 SKIE on the solvolysis of sterically hindered arenesulfonyl chlorides **2** and **2D** (R_2 = H-, Me-, Et-, Pr-, *i*-Pr-).

Here, we analyze the mechanism of solvolysis, hydrolysis, and alcoholysis of sterically hindered arenesulfonyl chlorides in the light of new results of solvent isotope effects (SIE; Scheme 2) and secondary kinetic isotope effects (SKIE; Scheme 3) in the temperature range 303–323 K.

EXPERIMENTAL

Kinetic runs were spectrophotometrically monitored under pseudo–first-order conditions with respect to the nucleophile on a Cary 1E UV–VIS spectrophotometer, in thermostated quartz cuvettes at temperatures between 303 and 323 K. Examples of UV–VIS spectra and time-resolved absorbance profiles are shown in the Supporting Information.

All solvents were thoroughly purified and tested for the presence of water impurities immediately before the kinetic experiments [24]. The acceptable water content was considered <0.002%.

Analytical-grade alcohols were purchased from Sigma-Aldrich (St. Louis, MO, USA). Molecular sieves (3 Å) were used for dehydration. All alcohols were redistilled immediately before the kinetic experiments at the temperatures specified in the literature (bp_{MeOH} = 64,4°C; bp_{EtOH} = 78.32°C; bp_{PrOH} = 97.15°C; $bp_{i\text{-}PrOH}$ = 82,6°C at 760 mmHg). Water obtained from the distiller Auga ELIX-Q was used for the hydrolysis.

Deuterated ethanol was obtained by the reaction of sodium ethoxide with pure heavy water. The initial ethoxide was prepared by dissolving of metal sodium in ethanol. Furthermore, after the alcohol distilling off,

Table I SIE Observed in the Ethanolysis of X-$ArSO_2Cl$

X-	T (K)	$k_{Et\text{-}OD} \times 10^4$ (s^{-1})	$k_{Et\text{-}OH} \times 10^4$ (s^{-1})	SIE = ($k_{Et\text{-}OH}/k_{Et\text{-}OD}$)
(**1**) 2,6-Me_2-4-*t*Bu-	303	1.78 ± 0.01	2.25 ± 0.01	1.26 ± 0.02
	313	4.00 ± 0.02	5.70 ± 0.04	1.42 ± 0.06
	323	8.04 ± 0.05	11.3 ± 0.1	1.41 ± 0.06
(**2**) 2,4,6-Me_3-	303	1.63 ± 0.01	2.24 ± 0.01	1.37 ± 0.02
	313	3.57 ± 0.01	4.99 ± 0.01	1.40 ± 0.02
	323	7.60 ± 0.04	10.2 ± 0.1	1.34 ± 0.02
(**3**) 4-Me-	303	0.26 ± 0.01	0.36 ± 0.01	1.39 ± 0.02
	313	0.66 ± 0.01	0.82 ± 0.01	1.24 ± 0.02
	323	1.42 ± 0.01	1.91 ± 0.01	1.35 ± 0.02

the residue was dried under vacuum. Heavy water in a fourfold excess was added to ethoxide; the reaction mixture was boiled under reflux (for 30 min) and distilled with a reflux condenser. Further purification corresponds to the methods mentioned previously.

2,4,6-Me_3-benzenesulfonyl chloride (**2**) and 4-Me-benzenesulfonyl chloride (**3**) were obtained from Sigma-Aldrich and recrystallized from hexane prior to its use.

Deuterated mesitylene sulfonyl chloride (**2D**) was synthesized from deuterated mesitylene [mesitylene-d_{12} (98 atom%D); Sigma-Aldrich] as follows [25]: The reaction was carried out under constant stirring at 273 K, 1 mol of NaCl was added to 1 mol of the mesitylene in 450 mL of an inert solvent ($CHCl_3$, CCl_4, hexane); then 5 mol of chlorosulfonic acid were slowly added dropwise for half an hour. After 3–4 h, the reaction mixture was poured onto ice, treated with chloroform; the extract was dried over Na_2SO_4 and filtered. The filtrate was evaporated under vacuum. The resulting deuterated sulfonyl chloride (**2D**), the middle fraction, was collected, and the product recrystallized from hexane with a yield of 80%, mp = 66.5–67°C. As there is no isotopic exchange, the percentage of deuteration in **2D** must be the same as in the parent mesitylene-d_{12}.

2,6-Me_2-4-*t*Bu-benzenesulfonyl chloride (**1**) was synthesized analogously from the 1-*tert*-butyl-3,5-dimethylbenzene with a yield of 85%; mp = 66–67°C.

The structure and purity of the obtained sulfonyl compounds were confirmed by NMR spectroscopy and monocrystal X-ray diffraction (see the Supporting Information).

RESULTS AND DISCUSSION

The SIE was studied to check the catalytic assistance of the solvent as a nucleophile in the ethanolysis of X-$ArSO_2Cl$ in the range 303–323 K [2]; the observed rate constants and the corresponding SIE are collected in Table I.

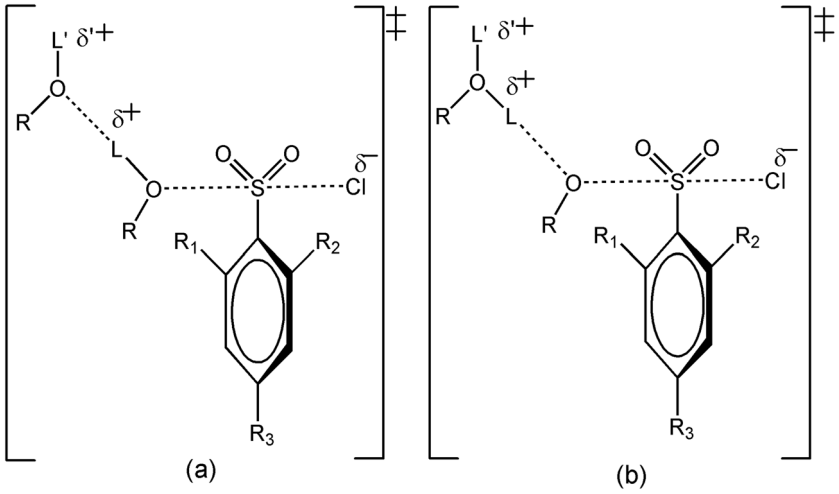
Ortho-methylated substrates show increased reactivity toward solvolysis in all cases (Table I). Change from protonated to deuterated solvent causes a slight decrease in the observed solvolytic rate constants. For compound **3**, adopted as a model compound, SIE ranges between 1.24 and 1.39 (Table I). SIE for hindered substrates **1** and **2** shows a similar range of SIE, 1.26–1.42, in agreement with previous observations for the methanolysis of sulfonyl chlorides [9,26]. The observed SIEs are comparable when considering different compounds and temperatures. For the methanolysis of **2** and **3** SIE ($k_{Me\text{-}OH}/k_{Me\text{-}OD}$) are, respectively, 1.68 and 1.72, in good agreement with those observed here [9,26]. Similar SIEs, ranging from 1.2 to 1.6, are typical for S_N2 processes at sulfonyl centers [9,26–29]. These results point to a SKIE of the alcoholic proton, the proton transfer between the oxygen of the attacking alcohol and that of a solvent molecule, not taking place in the rate-determining step.

The analysis of the SIE could be done in terms of fractionation factors:

$$\mathrm{SIE} = \frac{k_{\mathrm{EtoH}}}{k_{\mathrm{EtOD}}} = \frac{\phi_{\mathrm{EtOL}}}{\Pi_i \phi_i^{\ddagger}}$$

where ϕ_{EtOL} is the fractionation factor of the alcoholic hydrogen/deuterium of ethanol, whereas $\phi^{\ddagger}$ is/are the fractionation factor(s) of the alcoholic hydrogen/deuterium atom(s) involved in the TS.

The fractionation factor of any hydrogen/deuterium, which behaves like a hydrogen/deuterium of the solvent, is equal to one, whereas that of the corresponding lyonium ion is lower than unity, for example, $\phi(L_3O^+) = 0.69$ [30] or $\phi(MeOL_2{}^+) = 0.60$ [31], where L designates either H or D. From there, it follows that the more charge on the hydrogen/deuterium, the lower is the value of its fractionation factor, i.e. the value of the

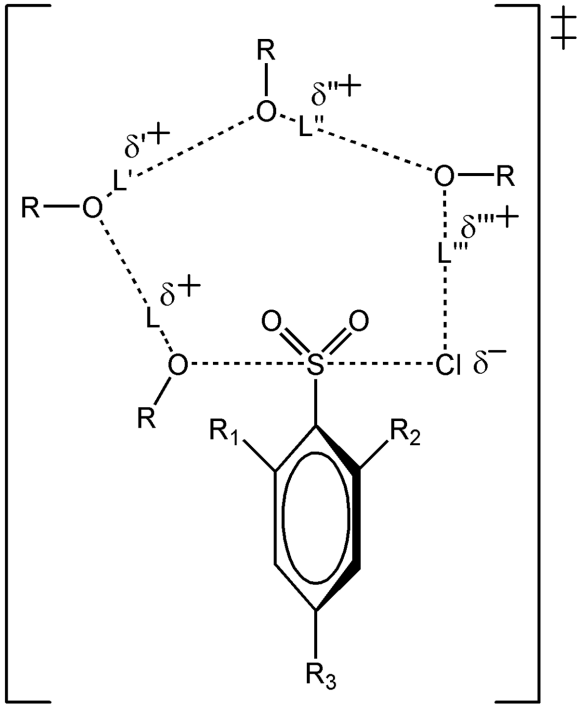


Scheme 4 "Early" (a) and "late" (b) TSs for the S_N2 mechanism of arenesulfonyl chlorides solvolysis with general base catalysis by a second solvent molecule.

fractionation factor gives information on the amount of charge on the hydrogen/deuterium relative to the solvent.

The value of the SIE could be consistent, regardless of the position of the hydrogen/deuterium, with both "early" (Scheme 4a) or "late" (Scheme 4b) S_N2 TSs. When the extreme "early" TS is considered, the hydrogen/deuterium is only starting to be transferred to another solvent molecule, and assuming the fractionation factor of the alcoholic hydrogen/deuterium should be similar to that of $MeOL_2^+$ ($\phi_L{}^+$ca. 0.60 relative to MeOL) [31], a SIE = (k_{EtOH}/k_{EtOD} = 1/0.60) ca. 1.6 would be expected; in this case, the hydrogen/deuterium should resemble that of protonated alcohol. Under the same assumption, SIE = (k_{EtOH}/k_{EtOD} = $1/0.60^2$) ca. 2.8 would be expected for the extreme "late" TS, where the hydrogen/deuterium has been almost fully transferred to another solvent molecule, so two hydrogens/deuteriums contribute to the SIE.

The observed SIE is ca. 1.35 (Table I), which implies the TS lies in between the "early" and "late" TSs for the S_N2 mechanism described above (Scheme 4), the product of the fractionation factors of the hydrogens/deuteriums involved in the TS being ≈0.74. It could be the value for only one hydrogen/deuterium (i.e., $\phi^{\ddagger}_L = 0.74$) that of the alcohol molecule bonded to the sulfur atom at the TS, or the product of several of them somehow participating in the TS; such figure is compatible even with a cyclic one involving several solvent molecules as shown in Scheme 5, i.e., $0.74 = (\phi_L \cdot \phi_{L'} \cdot \phi_{L''} \cdot \phi_{L'''})^{\ddagger}$ with ($\phi_L < \phi_{L'} \approx \phi_{L''} \approx \phi_{L'''} \leq 1$). These results support the participation of, at least, a second solvent molecule in the TS, through a general-base catalysis mechanism [2]. There is strong evidence in the literature for this kind of mechanism, with participation of solvent chains [32–35]. It has been found that there are an optimal number of solvent molecules that facilitates the mechanism, for example, via linear proton transfer in the TS [34].



Scheme 5 TS for the general-base catalyzed S_N2 mechanism for solvolysis of arenesulfonyl chlorides involving a chain of solvent. molecules.

Table II Activation Parameters for X-$ArSO_2Cl$ Ethanolysis in EtOH and EtOD

X-	Solvent	$\Delta H^{\ddagger}$ (kJ·mol^{-1})[a]	$\Delta S^{\ddagger}$ (J·mol^{-1}·K^{-1})[a]	$\Delta G^{\ddagger}$ (kJ·mol^{-1})[a,b]
(**1**) 2,6-Me_2–4-*t*Bu-	Et-OD	59 ± 2	–146 ± 5	105 ± 1
	Et-OH	64 ± 4	–126 ± 14	104 ± 9
(**2**) 2,4,6-Me_3-	Et-OD	60 ± 1	–143 ± 2	105 ± 3
	Et-OH	57 ± 3	–150 ± 8	104 ± 5
(**3**) 4-Me-	Et-OD	66 ± 3	–137 ± 9	109 ± 6
	Et-OH	66 ± 2	–137 ± 6	109 ± 3

[a]Estimated considering the second-order constant, i.e., $k_2 = k_{obs}$/[solvent].
[b]Value calculated at 313 K.

Activation parameters ($\Delta H^{\ddagger}$, $\Delta S^{\ddagger}$, and $\Delta G^{\ddagger}$) are within statistical error when the solvent is isotopically modified, i.e. EtOD instead of EtOH (Table II). Sterically hindered substrates **1**, **2** show slightly lower $\Delta G^{\ddagger}$ ~ (104–105 kJ·mol^{-1}) in comparison with the less sterically hindered **3** [31,36,37], which provides additional evidence against proton transfer in the rate-determining step.

On the other hand, the large negative $\Delta S^{\ddagger}$ values are consistent with highly ordered TSs, which support the participation of solvent molecules in the TS, as is shown in Scheme 4. The low and similar $\Delta H^{\ddagger}$ values

Table III Effective Rate Constants and Activation Parameters for Solvolysis of **2** and Deuterated-**2 (2D)** using Different Solvents as Nucleophiles

Compound	Nucleophile	T (K)	$k_{obs} \times 10^4$ (s^{-1})	$\Delta H^{\ddagger}$ (kJ·mol^{-1})[a]	$\Delta S^{\ddagger}$ (J·mol^{-1}·K^{-1})[a]	$\Delta G^{\ddagger}$ (kJ·mol^{-1})[a,b]
2	H_2O	293	505 ± 8	49 ± 2	–134 ± 7	91 ± 5
		298	746 ± 25			
		303	1090 ± 42			
		308	1640 ± 57			
		313	2000 ± 31			
		318	2650 ± 116			
	EtOH	303	2.24 ± 0.01	57 ± 3	–150 ± 8	104 ± 5
		313	4.99 ± 0.02			
		318	7.06 ± 0.01			
		323	10.2 ± 0.1			
		328	14.2 ± 0.01			
	MeOH	303	13.2 ± 0.1	54 ± 4	–148 ± 13	100 ± 8
	PrOH	323	6.13 ± 0.02	59 ± 1	–147 ± 2	105 ± 1
	i-PrOH	323	0.84 ± 0.01	55 ± 2	–173 ± 5	110 ± 3
2D	H_2O	293	493 ± 2	51 ± 2	–128 ± 5	91 ± 3
		298	764 ± 10			
		303	1120 ± 35			
		308	1550 ± 27			
		313	2140 ± 37			
		318	2790 ± 81			
	EtOH	303	2.23 ± 0.01	59 ± 2	–143 ± 7	104 ± 4
		313	4.95 ± 0.01			
		318	7.59 ± 0.01			
		323	10.2 ± 0.01			
		328	13.8 ± 0.01			
	MeOH	303	12.5 ± 0.1	–	–	–
	PrOH	323	5.83 ± 0.01	–	–	–
	i-PrOH	323	0.87 ± 0.01	–	–	–

[a]Estimated considering the second-order constant, i.e., $k_2 = k_{obs}$/[solvent].
[b]Value calculated at 313 K.

Table IV SKIE Observed for the Solvolysis of **2** and **2D** with Different Solvents as Nucleophiles between 303 and 323 K

Nucleophile	T (K)	SKIE (k_2/k_{2D})
H_2O	293	1.03 ± 0.01
	298	0.98 ± 0.01
	303	0.97 ± 0.01
	308	1.06 ± 0.02
	313	0.93 ± 0.02
	318	0.95 ± 0.02
EtOH	303	1.00 ± 0.01
	313	1.01 ± 0.01
	318	0.93 ± 0.02
	323	1.00 ± 0.01
	328	1.03 ± 0.01
MeOH	303	1.06 ± 0.01
PrOH	323	1.05 ± 0.01
i-PrOH	323	0.96 ± 0.02

point to a highly concerted TS, which would support the hypothesis of formation of solvent chains as in Scheme 5.

To check for nonbonding intramolecular interactions between the hydrogens of the *ortho*-methyl groups and the oxygens of the sulfonyl groups, activation parameters and SKIEs were investigated for the solvolysis of mesitylene sulfonyl chloride, 2,4,6-$(CH_3)_3$-$C_6H_2SO_2Cl$ (**2**) and its deuterated analog 2,4,6-$(CD_3)_3$-$C_6D_2SO_2Cl$ (**2D**) (SKIE $= k_2/k_{2D}$), using different solvents as nucleophiles. Comparable results were obtained in both cases (Tables III and IV).

The reactivities of **2** and **2D** are similar, within statistical error, for all solvolytic processes. Similarly activation parameters are statistically indistinguishable (Table III). Values of SKIE are in all cases very close to unity, within statistical error, for all studied nucleophiles (Table IV). Thus, it follows that noncovalent intramolecular interactions between the *ortho*-methyl hydrogens and oxygen atoms of the sulfonyl group are not a significant factor in the stabilization of the S_N2-transition state. This conclusion also poses serious doubts on the applicability of the idea of $\sigma-\pi$ hyperconjugation to this particular case [8].

CONCLUSIONS

SIE in the ethanolysis of sterically hindered arenesulfonyl chlorides slows the reaction rate by ca. 35%. Such a result rules out a proton transfer in the rate-determining step, and the analysis of the SIE in terms of fractionation factors agrees with a S_N2 mechanism involving at least a second solvent molecule in the TS. No SIKE is observed when hydrogens of the *o*-alkyl groups are replaced by deuteriums, which discard $\sigma-\pi$ hyperconjugation. Activation parameters are similar regardless of the steric hindrance of the substrates, which points to a similar reaction mechanism for all of them. Large negative $\Delta S^{\ddagger}$ and low comparable $\Delta H^{\ddagger}$ are consistent with a S_N2 mechanism with participation of, at least, a second solvent molecule in the TS, and most possibly with a cyclic TS in which a reduced number of solvent molecules form chains, in a general-base catalysis mechanism. The reasons for the "positive steric effect" of *o*-alkyl (methyl) groups should be attributed to structural features of the S_N2 transition state. Further experimental and computational studies are in progress to clarify this point.

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