Groenvik.[7] Both gave only a single peak when analyzed by high-pressure LC.

B. Preparation of Diacyl Derivatives. Compounds **1d**, **2d**, **5**, and **6** were prepared by the method described by Jacobs[6] and by Nelson.[5] A slurry of the acylamidophenol (0.007–0.03 mol) in a 5–15 molar excess of chloroacetyl chloride, acetyl chloride, or acetic anhydride was treated with a few drops of concentrated H_2SO_4, stirred for 15 min at 80 °C, and then poured into ice–water and stirred. The crude product was isolated by filtration, washed with water, dried, and recrystallized from benzene. All derivatives gave a single peak on high-pressure LC analysis.

Properties of the six compounds used in this study are listed in Table II. There is no correlation between UV absorbance and LC response because the latter depends on both retention time and UV absorbance. Thus, of two compounds with the same UV absorbance, the one with the shorter LC retention time will have the greater LC peak height per microgram.

Registry No. **1d**, 73048-40-5; **2d**, 73037-92-0; **3**, 614-80-2; **4**, 10147-68-9; **5**, 5467-64-1; **6**, 37161-45-8.

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[7] E. Groenvik, *Bull. Soc. Chim. Fr.*, **25**, 173 (1876).

Synthesis of a Sulfone α-Tosylate. Benzyl (Tosyloxy)methyl Sulfone

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In our studies of leaving-group activities in base-induced 1,3-elimination reactions of α-X sulfones, it was necessary to prepare related sulfones bearing an α′ H in which X = halogen,[1] NO_2,[2] and OTs. For OTs, benzyl (tosyloxy)methyl sulfone (**6**) was prepared. Its base-induced reactivity proved to be quite unexpected.[3] No sulfone α-sulfonate like **6** has previously been reported.[4] Because this new class of compound appears to have unusual properties warranting further investigation, and because the usual methods for preparing sulfonates seem to be ineffective in these cases, we are describing the synthesis of **6** (Scheme I).

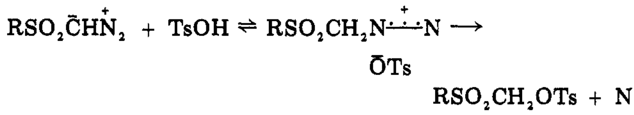
Failure of conventional approaches to provide tosylate **6** (vide infra) led us to consider the synthetic route of Engberts and Zwanenburg employing α-diazo sulfones.[4-6] As illustrated by reaction C in Scheme I, this approach was successful; **6** was formed in ca. 90% yield from the reaction of benzyl diazomethyl sulfone (**5**) with *p*-toluenesulfonic acid under very mild conditions. This reaction is interesting insofar as it involves a rare example of a rather facile intermolecular nucleophilic displacement of an α substituent of a sulfone, systems which usually resist such displacement reactions.[7] It was suggested that with α-diazo sulfones the reaction proceeds through the collapse of the intimately ion-paired diazonium salt intermediate; expulsion of stable N_2 from this highly energetic intermediate provides a very favorable transition energy.[4-6]

Scheme I

A. $$\underset{\textbf{1a}}{\mathrm{PhCH_2SO_2Na}} + \underset{\textbf{2}}{\mathrm{BrCH_2C(O)COOEt}} \xrightarrow[\text{10 h (N}_2\text{)}]{\text{EtOH, reflux}} \underset{\textbf{3}\ (53\%)}{\mathrm{PhCH_2SO_2CH_2C(O)COOEt}}$$

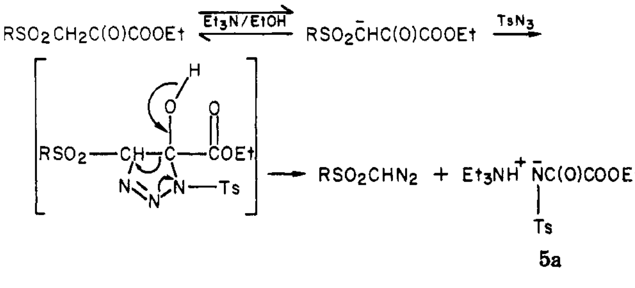
B. $$\mathbf{3} + \underset{\textbf{4}}{p\text{-}\mathrm{CH_3C_6H_4SO_2N_3}} \xrightarrow[\text{10–15 °C, 1 h (N}_2\text{)}]{\text{Et}_3\text{N, EtOH}} \underset{\textbf{5}\ (64\%)}{\mathrm{PhCH_2SO_2CHN_2}}$$

C. $$\mathbf{5} + p\text{-}\mathrm{CH_3C_6H_4SO_3H} \xrightarrow[\text{25 °C, 2 h (N}_2\text{)}]{\text{ClCH}_2\text{CH}_2\text{Cl}} \underset{\textbf{6}\ (88\%)}{\mathrm{PhCH_2SO_2CH_2OSO_2C_6H_4CH_3}\text{-}p}$$



$$\mathrm{RSO_2\bar{C}H\overset{+}{N}_2} + \mathrm{TsOH} \rightleftharpoons \underset{\bar{\mathrm{O}}\mathrm{Ts}}{\mathrm{RSO_2CH_2N\overset{+}{\equiv}N}} \longrightarrow \mathrm{RSO_2CH_2OTs} + \mathrm{N_2}$$

In preparing **5** we had a choice of several methods which have been used to make α-diazo sulfones: (1) cleavage of *N*-nitroso-*N*-(sulfonylmethyl)urethanes with alkali or neutral alumina;[8] (2) cleavage of α-diazo-β-oxo sulfones by treatment with triethylamine;[9] (3) base-catalyzed reaction of α-sulfonyl aldehydes with tosyl azide;[10] and (4) base-catalyzed reaction of sulfonylpyruvic esters with tosyl azide.[11] The last method seemed to be the most advantageous because sulfonylpyruvic esters appeared to be more easily obtained or, in some cases, more stable than the substrates required by the other methods, and because of the simple product isolation it afforded. As indicated by reaction B in Scheme I, we obtained **5** by this route in 64% yield from the brief treatment of ethyl (benzylsulfonyl)pyruvate (**3**) with *p*-toluenesulfonyl azide (**4**) in cold Et_3N/EtOH followed by the addition of water. Separation of **5** from the reaction mixture was also quite simple: it was readily extracted into ether, coformed triethylammonium imido salt **5a** remained in the aqueous solution,



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[1] The preparation and 1,3-elimination reactions of α-halo sulfones have recently been summarized: (a) Paquette, L. A. *Org. React.* **1977**, *25*, 1–71. (b) Meyers, C. Y.; Matthews, W. S.; Ho, L. L.; Kolb, V. M.; Parady, T. E. In "Catalysis in Organic Syntheses"; Smith, G. V., Ed.; Academic Press: New York, 1977; pp 197–278. (c) Meyers, C. Y. In "Topics in Organic Sulfur Chemistry"; Tišler, M., Ed.; University Press: Ljubljana, Yugoslavia, 1978; pp 207–60.

[2] For preliminary reports on the α-nitro sulfones see: (a) Meyers, C. Y.; Hua, D. H.; Peacock, N. J. "Abstracts of Papers", 13th Great Lakes Regional Meeting of the American Chemical Society, Rockford, IL, June 1979; American Chemical Society: Washington, DC, 1979; ORGN 198. (b) Hua, D. H.; Peacock, N. J.; Meyers, C. Y. *Ibid.*, ORGN 199.

[3] Meyers, C. Y.; Hua, D. H.; Peacock, N. J. *J. Org. Chem.*, see companion communication in this issue.

[4] Engberts and Zwanenburg (Engberts, J. B. F. N.; Zwanenburg, B. *Tetrahedron Lett.* **1967**, 831–6) mention the formation but not the characterization of the only other sulfone α-sulfonate reported, $p\text{-}CH_3C_6H_4SO_2CH_2OTs$; however, it contains no α′ H.

[5] Engberts, J. B. F. N.; Zwanenburg, B. *Tetrahedron* **1968**, 1737–54.

[6] More O'Ferrall, R. A. *Adv. Phys. Org. Chem.* **1967**, *5*, 331–99 (especially pp 347–8).

[7] See note 16 in ref 3.

[8] Leusen, A. M. Van; Strating, J. *Recl. Trav. Chim. Pays Bas* **1965**, *84*, 151–64.

[9] Hodson, D.; Holt, G.; Wall, D. *J. Chem. Soc. C* **1968**, 2201–5.

[10] Leusen, A. M. Van; Strating, J.; Leusen, D. Van *Tetrahedron Lett.* **1973**, 5207–8.

[11] Fridman, A. L.; Andreichikov, Yu. S.; Gein, V. L.; Gein, L. F. *J. Org. Chem. USSR (Engl. Transl.)* **1976**, *12*, 457; *Zh. Org. Khim.* **1976**, *12*, 463.

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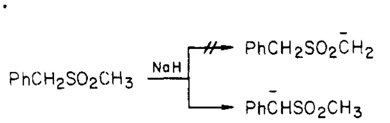
and residual **3** was removed with dilute NaOH washes of the extract. Since 21% of **4** was recovered after the 1-h reaction at 10–15 °C, the yield of **5** no doubt could be enhanced by increasing the reaction temperature or time. It was not necessary to purify **5** before it was used in reaction C; the presence of **4** seemed to have little if any adverse effect.

The published account of the synthesis of α-diazo sulfones from sulfonylpyruvic esters dealt with the preparation only of aromatic α-diazo sulfones ($ArSO_2CHN_2$) from (arylsulfonyl)pyruvic esters.[11] The latter were obtained from the reaction of arylsulfonyl carbanions with diethyl oxalate:

$$\mathrm{ArSO_2CH_3 \xrightarrow{NaH} ArSO_2CH_2^- \xrightarrow{EtOC(O)COOEt} ArSO_2CH_2C(O)COOEt}$$

Our required (benzylsulfonyl)pyruvic ester, **3**, cannot be made this way; treatment of benzyl methyl sulfone with strong base would provide the wrong sulfonyl carbanion regioisomer:[12]



However, as shown by reaction A in Scheme I, **3** was easily prepared by a modification of this procedure: sodium α-toluenesulfinate (**1a**) was the nucleophile and ethyl bromopyruvate (**2**) was the electrophile. This method for the production of sulfonylpyruvic esters should be a general one, as sulfinic acids are prepared by a variety of routes[14] and **2** is commercially available or is easily prepared.

Other approaches to the synthesis of **6** were either known to be futile or found to be unsatisfactory: (1) Tosylation of hydroxymethyl sulfones with TsCl in pyridine fails because sulfonylcarbinols are unstable and are decomposed rapidly in the presence of base.[15] (2) The reaction of benzyl hydroxymethyl sulfide with TsCl in pyridine provided a mixture in which the desired tosylate could not be detected. (3) The reaction of benzyl chloromethyl sulfide with AgOTs in acetonitrile[16] likewise produced a mixture in which the desired tosyl ester was not evident. (4) Treatment of $TsOCH_2OTs$[16b] with sodium sulfinates in refluxing methanol gave rise to methanolysis (TsOH was isolated) and in DMF at 60 °C produced NaOTs and some of the sulfinic acid on aqueous workup; the desired sulfonylmethyl tosylates were not detected.

Experimental Section

^1H NMR spectra were recorded on a Perkin-Elmer R32 90-MHz spectrometer; IR spectra were recorded on a Beckman IR10 spectrophotometer. Melting points are corrected. Elemental analysis was carried out by Galbraith Laboratories.

Materials. **α-Toluenesulfinic acid (1)** was prepared as described[17] from benzylmagnesium chloride and sulfur dioxide: waxy solid (61%); IR (melt) 3600–2700 (broad, OH), 1070 (strong, SO_2H) cm^{-1}; ^1H NMR ($CDCl_3$) δ 8.80 (s, 1, OH), 7.5–7.2 (m, 5, Ph), 4.00 (s, 1, CH_2). **Ethyl bromopyruvate (2)** was prepared as described[18] from ethyl pyruvate and Br_2: colorless oil (62%); bp 85–90 °C (5 mm) [lit.[18] bp 98–105 °C (14 mm)]; IR and ^1H NMR were identical with those reported for the commercial product (Aldrich IR, 2nd ed., 354B; Aldrich NMR, Vol. 3, 62A). ***p*-Toluenesulfonyl azide (4)** was prepared as described[19] from TsCl and NaN_3: oil (94%); IR (neat) 2130 (strong, N=N=N), 1380 and 1180 (strong, SO_2) cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.84 (d, J = 8 Hz, 2, o-SO_2Ar H), 7.40 (d, J = 8 Hz, 2, m-SO_2Ar H), 2.47 (s, 3, CH_3).

Ethyl (Benzylsulfonyl)pyruvate (3). In a flask maintained under N_2, 50 mL of absolute EtOH was allowed to react with 0.92 g (0.040 mol) of sodium. When this reaction was completed 7.80 g (0.050 mol) of α-toluenesulfonic acid (**1**) was added, followed by 9.75 g (0.050 mol) of ethyl bromopyruvate (**2**). After the solution was refluxed for 10 h the solvent was removed by evaporation and 100 mL of water was added. This solution was acidified with dilute HNO_3 and extracted twice with Et_2O. The combined extracts were washed thoroughly with water[20] and saturated aqueous NaCl, dried ($MgSO_4$), and concentrated in vacuo, leaving 14.6 g of a pale yellow oil which was chromatographed (silica gel, petroleum ether/EtOAc) to provide **3**, a viscous pale yellow oil which would not crystallize: 5.67 g (0.021 mol, 53%); IR (neat) 3700–3300 (strong, broad, enolic OH), 1758 (strong, ester C=O), 1740 (strong, ketone C=O), 1630 (medium, enolic C=C), 1330 and 1130 (strong, SO_2) cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.58–7.30 (m, 5, Ph), 4.44 (s, 2, CH_2Ph), 4.38 (quartet, J = 7 Hz, 2, CH_2CH_3), 4.35 (s, 2, CH_2C=O), 1.38 (t, J = 7 Hz, 3, CH_3).[21]

Benzyl Diazomethyl Sulfone (5). A solution of 1.08 g (0.0040 mol) of ethyl (benzylsulfonyl)pyruvate (**3**), 0.788 g (0.0040 mol) of TsN_3 (**4**), and 0.405 g (0.0040 mol) of freshly distilled Et_3N in 10 mL of EtOH was stirred under N_2 in a flask immersed in an ice–water bath. The solution became pale orange and gradually dark orange-red. At the end of 1 h 100 mL of water was added and the mixture was extracted three times with Et_2O. The combined extracts were washed with 3 N NaOH, water, and saturated aqueous NaCl,[22] dried ($MgSO_4$), and evaporated. The residue was dried in vacuo (dark, 25 °C) for 3 h: oily yellow crystals; 0.670 g. ^1H NMR indicated the composition as 75 mol % **5** and 25 mol % **4**. Thus, 0.50 g (0.0026 mol, 64%) of **5** was formed and 0.17 g (0.00086 mol, 21%) of **4** was recovered. For **5**: IR (neat) 2120 (strong, N≡N), 1340 and 1130 (strong, SO_2) cm^{-1} [lit.[8] IR (CCl_4) 2110 (very strong, N≡N), 1345, and a triplet at 1150, 1135, 1119 (strong, medium, SO_2) cm^{-1}]; ^1H NMR ($CDCl_3$) δ 7.5–7.3 (m, 5, Ph), 4.98 (s, 1, CH), 4.37 (s, 2, CH_2) [lit.[8] ^1H NMR ($CDCl_3$) δ 5.02 (CH)].[23]

Benzyl (Tosyloxy)methyl Sulfone (6). To a stirred mixture of 0.88 g (0.0046 mol) of p-toluenesulfonic acid monohydrate (mp 102–103 °C; recrystallized from $CHCl_3$)[24] in 20 mL of 1,2-dichloroethane maintained under N_2 was added 0.50 g (0.0026 mol)

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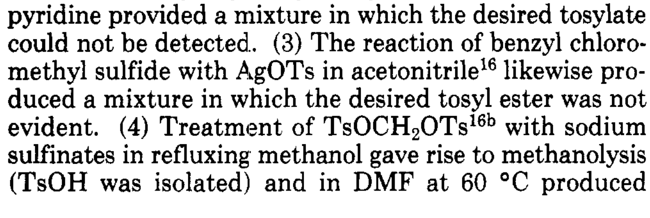
[12] (a) For $PhCH_2SO_2CH_3$ (in Me_2SO) the K_a of CH_2 is ca. 10^5 times as large as the K_a of CH_3: Bordwell, F. G.; Bares, J. E.; Bartmess, J. E.; McCollum, G. J.; Van Der Puy, M.; Vanier, N. R.; Matthews, W. S. *J. Org. Chem.* **1977**, *42*, 321–5. (b) An excess of strong base would form the α,α' dianion[13] whose initial reaction with diethyl oxalate would be from the desired position, but subsequent cyclization would probably occur:



[13] Kaiser, E. M.; Hauser, C. R. *Tetrahedron Lett.* **1967**, 3341–4. Hauser, C. R.; Harris, T. M. *J. Am. Chem. Soc.* **1959**, *81*, 1154–9. Kaiser, E. M.; Slocum, D. W. In "Organic Reactive Intermediates"; McManus, S. P., Ed.; Academic Press: New York, 1973; Chapter 5, pp 365–80.

[14] Oae, S.; Kuneida, N. In "Organic Chemistry of Sulfur"; Oae, S., Ed.; Plenum Press: New York, 1977; Chapter 11. See also: Filby, W. G.; Günther, K.; Penzhorn, R. D. *J. Org. Chem.* **1973**, *38*, 4070–1.

[15] Truce, W. E. In ref 14, Chapter 10. Suter, C. M. "Organic Chemistry of Sulfur"; Wiley: New York, 1944; Chapter VII.

[16] In many cases the reaction of silver sulfonates with alkyl and benzyl halides to produce sulfonic esters under these conditions is quite successful. E.g.: (a) Kornblum, N.; Jones, W. J.; Anderson, G. J. *J. Am. Chem. Soc.* **1959**, *81*, 4113–4. (b) Emmons, W. D.; Ferris, A. F. *J. Am. Chem. Soc.* **1953**, *75*, 2257.

[17] Rothstein, E. *J. Chem. Soc.* **1934**, 684–7.

[18] Borrows, E. T.; Holland, D. O. *J. Chem. Soc.* **1947**, 672–4.

[19] Doering, W. E.; DePuy, C. H. *J. Am. Chem. Soc.* **1953**, *75*, 5955–7. Regitz, M.; Hocker, J.; Liedhegener, A. "Organic Syntheses"; Wiley: New York, 1973; Collect Vol. V, pp 179–83.

[20] The water wash removed any residual sulfinic acid; washing with even a weakly basic solution would also remove **3**, which is quite acidic.

[21] The enolic moiety exhibited in the IR spectrum was not evident in the ^1H NMR spectrum.

[22] These washes removed any residual **3** and coproduct **5a** (or TsNHC(O)COOEt).

[23] This product was used immediately without further purification; α-diazo sulfones are prone to decomposition especially in the presence of light (cf. ref 8).

of benzyl diazomethyl sulfone (**5**) [as a mixture with 0.17 g (0.00086 mol) of TsN_3 (**4**); see above], and stirring was continued for 2 h. Et_2O (150 mL) was added and the solution was washed thoroughly with water, dried ($MgSO_4$), and evaporated, leaving 0.97 g of yellow crystals shown by ^1H NMR to be composed of **6** (87 mol %) and **4** (13 mol %). Trituration with hexane removed **4**, 0.17 g (0.00086 mol, 100% recovery), leaving **6**: white crystals; mp 147–150 °C; 0.77 g (0.0023 mol, 88%); mp 151–152 °C (hexane/benzene; EtOH); IR (Nujol) 1380 and 1180 (strong, SO_3), 1345 and 1130 (strong, SO_2) cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.84 (d, J = 8 Hz, 2, o-SO_3Ar H), 7.5–7.3 (m, 7, m-SO_3Ar H, Ph), 4.66 (s, 2, SCH_2O), 4.30 (s, 2, CH_2Ph), 2.47 (s, 3, CH_3). Anal. Calcd for $C_{15}H_{16}O_5S_2$: C, 52.93; H, 4.74; S, 18.84. Found: C, 52.91; H, 4.85; S, 18.78.

Registry No. **1**, 4403-73-0; **2**, 70-23-5; **3**, 73178-38-8; **4**, 941-55-9; **5**, 1588-80-3; **6**, 73178-37-7; p-toluenesulfonic acid, 104-15-4.

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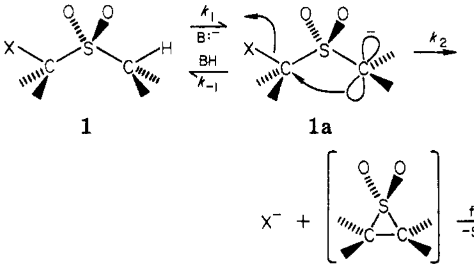
[24] This recrystallized material is the monohydrate: ^1H NMR (CD_3COCD_3) δ 8.57 (s, 3, OH), 7.78 (d, J = 8 Hz, 2, o-SO_3Ar H), 7.28 (d, J = 8 Hz, 2, m-SO_3Ar H), 2.40 (s, 3, CH_3). We did not find it necessary to use anhydrous TsOH (cf. ref 4).

Communications

Unexpected Behavior of an α-Tosyloxy Sulfone Compared with an α-Chloro Sulfone in Base-Induced Reactions

Summary: Unexpectedly, a rate ratio for k_{OTs}/k_{Cl} of 0.0011 was observed in 1,3 eliminations of (tosyloxy)methyl benzyl sulfone and chloromethyl benzyl sulfone in t-BuOK/t-BuOH (25 °C), while in MeONa/MeOH (25 °C) neither 1,3 elimination occurred; instead, the α-tosyloxy sulfone underwent facile S–O cleavage to provide methyl p-toluenesulfonate and α-toluenesulfinic acid.

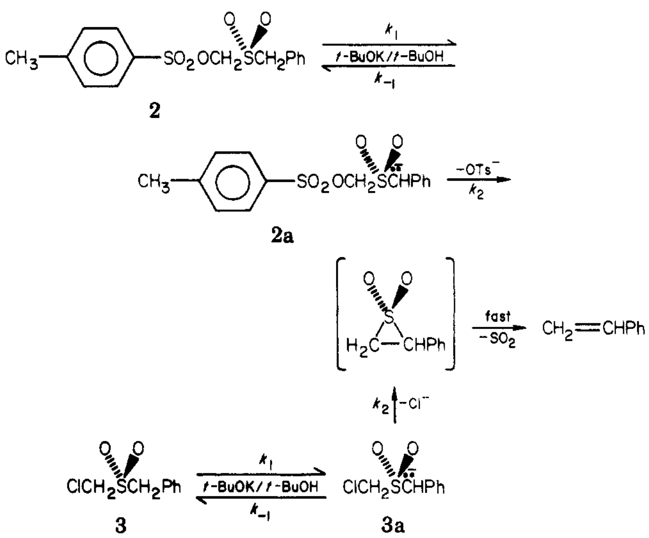
Sir: It is generally believed that base-induced 1,3-elimination reactions of α-halo sulfones (**1**) (Ramberg–Bäcklund reactions) proceed via intramolecular nucleophilic displacement from the semi-W conformation of the α′ carbanion (**1a**) and that the elimination step is rate limiting insofar as halide ion and alkene are formed from **1a** at the same rate, k_2.[1] Although the conventional order of leaving-group reactivity for ionic displacements and eliminations is exhibited in these reactions ($k_I > k_{Br} > k_{Cl}$), the values of k_I/k_{Cl} and k_{Br}/k_{Cl} are as much as 10 times the expected values, even compared to those of other intramolecular nucleophilic displacements.[2]



X = Cl, Br, I

Unfortunately, tosylate, one of the most commonly used leaving groups, was not included in these comparative rate studies; indeed, the Ramberg–Bäcklund reaction of α-tosyloxy sulfones has not been previously investigated, primarily because of the difficulty posed by their preparation. In nucleophilic displacement reactions, OTs is a much better leaving group than Cl; e.g., values of k_{OTs}/k_{Cl} in the range of 70–3500 were recently reported by Harris and co-workers,[3] while Lowry and Richardson[4] tabulated a range of 60–1500. The successful preparation of (tosyloxy)methyl benzyl sulfone (**2**)[5a] finally afforded the opportunity to investigate the base-induced reactivity of this type of sulfone and to determine the value of k_{OTs}/k_{Cl} in these 1,3 eliminations, the results of which might help to evaluate the significance of the surprisingly large values of k_I/k_{Cl} and k_{Br}/k_{Cl} previously reported.

In t-BuOK/t-BuOH. We were intrigued to find that in t-BuOK/t-BuOH (6.7 × 10^{-2} N, 25 °C) 1,3 elimination of OTs⁻ from **2** was much *slower* than 1,3 elimination of Cl⁻ from the counterpart chloromethyl benzyl sulfone (**3**),[5b] the value of $k_{obsd(OTs)}/k_{obsd(Cl)}$ being 0.0011. In each run of the series required for this kinetic study, styrene was



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[1] These reactions have been reviewed: (a) Bordwell, F. G. In "Organosulfur Chemistry"; Janssen, M. J., Ed.; Interscience: New York, 1967; Chapter 16. (b) Paquette, L. A. In "Mechanisms of Molecular Migrations"; Thyagarajan, B. S., Ed.; Interscience: New York, 1968; Vol. 1, pp 121–56. (c) *Acc. Chem. Res.* **1968**, *1*, 209–16. (d) *Org. React.* **1977**, *25*, 1–71. (e) Meyers, C. Y.; Matthews, W. S.; Ho, L. L.; Kolb, V. M.; Parady, T. E. In "Catalysis in Organic Syntheses"; Smith, G. V., Ed.; Academic Press: New York, 1977; pp 197–278. (f) Meyers, C. Y. In "Topics in Organic Sulfur Chemistry"; Tišler, M., Ed.; University Press: Ljubljana, Yugoslavia, 1978; pp 207–60.

[2] (a) Bordwell, F. G.; Wolfinger, M. D. *J. Org. Chem.* **1974**, *39*, 2521–5. (b) Bordwell, F. G.; Williams, J. M. *J. Am. Chem. Soc.* **1968**, *90*, 435–9.

[3] Harris, J. M.; Shafer, S. G.; Moffatt, J. R.; Becker, A. R. *J. Am. Chem. Soc.* **1979**, *101*, 3295–3300. [Rate ratios are estimated and are corrected to reflect reactions at 25 °C: solvolysis in 70% (v/v) aqueous ethanol of p-Y-$C_6H_4CH_2$(OTs or Cl), Y = NO_2, Cl, H, CH_3.]

[4] Lowry, T. H.; Richardson, K. S. "Mechanism and Theory in Organic Chemistry"; Harper and Row: New York, 1976; pp 192–3. [Reactions at 25 °C in ethanol: n-C_3H_7(OTs or Cl) + p-$CH_3C_6H_4S^-$ and C_2H_5(OTs or Cl) + $C_2H_5O^-$.]

[5] (a) Mp 151.5–152.5 °C. Synthetic details are described by: Hua, D. H.; Peacock, N. J.; Meyers, C. Y. *J. Org. Chem.*, see companion note in this issue. (b) Mp 102–103 °C [lit. mp 103 °C (Böhme, H.; Fischer, H.; Frank, R. *Justus Liebigs Ann. Chem.* **1949**, *563*, 54–72); lit. mp 103–104 °C (Bordwell, F. G.; Cooper, G. D. *J. Am. Chem. Soc.* **1951**, *73*, 5184–6)].

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