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## PREPARATION OF 2-SUBSTITUTED ETHYL PERFLUOROALKYLSULFONES

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Treatment of substituted ethylenes  $\text{CH}_2=\text{CH}-\text{X}$  (X: CN,  $\text{CH}_3\text{CO}$ ) or ethylene oxides  $\text{YCHCH}_2\text{O}$  (Y: H,  $\text{CH}_3$ ,  $\text{ClCH}_2$ ) with sodium perfluoroalkanesulfonates  $\text{R}_f\text{SO}_2\text{Na}$ , ( $\text{R}_f$ :  $\text{C}_4\text{F}_9$ ,  $\text{ClC}_4\text{F}_8$ ) which were obtained from diiodosulfonation reactions of  $\text{R}_f\text{I}$  with  $\text{Na}_2\text{S}_2\text{O}_4$ , gave 2-substituted ethyl perfluoroalkanesulfonates in moderated yields.

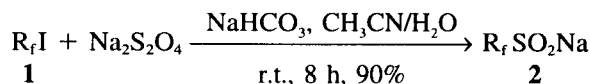
**Key words:** Nucleophilic addition, preparation, 2-substituted ethyl perfluoroalkylsulfones.

### INTRODUCTION

The last two decades have witnessed an impressive number of original publications devoted to the understanding and application of organosulfur chemistry. The sulfone functional group as a standard part of organosulfur chemistry has been well studied and reviewed.<sup>1–3</sup> The fluorine containing analogues have received more attention recently.<sup>4–6</sup> As a part of our continuous studies on the fluoroalkylsulfones and their derivatives<sup>7,8</sup> this paper describes a general and convenient method for preparation of the title compounds from the easy available starting materials perfluoroalkyl iodides.

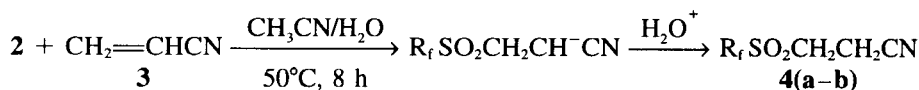
### RESULTS AND DISCUSSION

A straight forward route to perfluoroalkanesulfonates which proceeds via sulfonato deiodonation has been described, thus<sup>9,10</sup>:



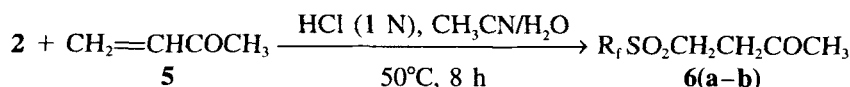
The availability of the starting material  $\text{R}_f\text{I}$  and the mild reaction condition required promoted us to further study the reaction of **2** and especially their synthetic applications.

As with many other nucleophilic additions to olefins, additions of **2** requires a group adjacent to the double bond capable of stabilizing the intermediate carbanion. Acrylonitrile readily reacts with **2**, thus:

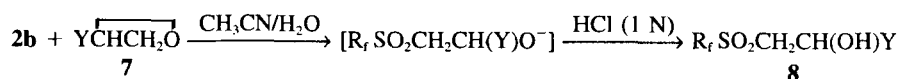


$\text{R}_f$ :  $\text{C}_4\text{F}_9$  **2a**;  $\text{ClC}_4\text{F}_8$  **2b**.

This reaction was easily carried out in aqueous  $\text{CH}_3\text{CN}$  solution; after acidifying with dilute  $\text{HCl}$  (1.0 N) the products **4** were obtained in 70% yield. Under the same reaction condition, treatment of **2** with methylvinyl ketone, however, only gave 10% of the expected product  $\text{R}_f\text{SO}_2\text{CH}_2\text{CH}_2\text{COCH}_3$ . Literature<sup>11</sup> has reported that *p*-toluene sulfinate does not react with  $\alpha,\beta$ -unsaturated ketones, but this reaction readily proceeds upon addition of an equivalent amount of acetic acid. When we added an equivalent amount of dilute hydrochloric acid the 2-acetyl ethyl perfluoroalkylsulfone was obtained in 65% yield:



Ethylene oxides reacted with **2** in a similar way. This reaction could be monitored by  $^{19}\text{F}$  NMR spectroscopy. The signal of  $\text{R}_f'\text{CF}_2\text{SO}_2\text{Na}$  is at 53.7 ppm (TFA as external standard and upfield as positive), but in the product  $\text{R}_f'\text{CF}_2\text{SO}_2\text{CH}_2\text{CH}(\text{Y})\text{O}^-$  it is at 40.0 ppm. After the signal at 53.7 ppm disappeared (about 24 h),  $\text{HCl}$  (1 N, 5 ml) was added. Similar work up as above gave 2-hydroxyethyl perfluoroalkylsulfone **8**.



Y: H (**a**),  $\text{CH}_3$  (**b**),  $\text{ClCH}_2$  (**c**).

Under acidic reaction condition, however, the main product is  $\text{YCH}(\text{OH})\text{CH}_2\text{Cl}$  and only a little amount of **8** was obtained (<10%).

It was found that under acidic reaction condition compounds **4** hydrolyzed slowly to the corresponding amide quantitatively.

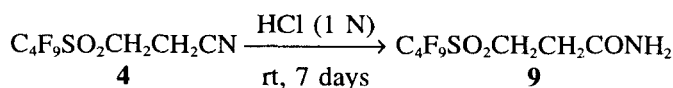


TABLE I  
Compounds **4**, **6**, and **8** prepared

Compounds <b>4</b> , <b>6</b> and <b>8</b>			Formula <sup>a</sup>	Yield (%) <sup>b</sup>	m.p. (°C) or b.p. (°C/Torr)
$\text{R}_f$	X (or Y)				
$\text{C}_4\text{F}_9$	CN	<b>4a</b>	$\text{C}_7\text{H}_4\text{F}_9\text{NO}_2\text{S}$	71	74–5
$\text{ClC}_4\text{F}_8$	CN	<b>4b</b>	$\text{C}_7\text{H}_4\text{F}_8\text{ClNO}_2\text{S}$	69	78–80
$\text{C}_4\text{F}_9$	$\text{CH}_3\text{CO}$	<b>6a</b>	$\text{C}_8\text{H}_7\text{F}_9\text{O}_3\text{S}$	68	72
$\text{ClC}_4\text{F}_8$	$\text{CH}_3\text{CO}$	<b>6b</b>	$\text{C}_8\text{H}_7\text{ClF}_8\text{O}_3\text{S}$	67	74–6
$\text{ClC}_4\text{F}_8$	H	<b>8a</b>	$\text{C}_6\text{H}_3\text{F}_8\text{ClO}_3\text{S}$	58	61–3/2
$\text{ClC}_4\text{F}_8$	$\text{CH}_3$	<b>8b</b>	$\text{C}_7\text{H}_7\text{ClF}_8\text{O}_3\text{S}$	64	71–4/2
$\text{C}_4\text{F}_9$	$\text{ClCH}_2$	<b>8c</b>	$\text{C}_7\text{H}_6\text{ClF}_9\text{O}_3\text{S}$	63	85–7/2

<sup>a</sup>Satisfactory elemental analysis obtained: C  $\leq +0.40\%$ , H  $\leq +0.38\%$ , F  $\leq +0.40\%$ , except **8a** (F =  $-0.47\%$ ) and **8b** (F =  $-0.56\%$ ).

<sup>b</sup>Based on the amount of  $\text{R}_f'\text{SO}_2\text{Na}$ .

## EXPERIMENTAL

Melting points were measured on a Thiele apparatus. The M.P. and B.P. are uncorrected. Solvents were purified before use.  $^1\text{H}$  NMR and  $^{19}\text{F}$  NMR spectra were recorded on a Varian 360L instrument with  $\text{Me}_4\text{Si}$  and TFA ( $\delta_{\text{CHCl}_3} = \delta_{\text{TFA}} + 77.6$  ppm) as internal and external standards, respectively. Elemental analyses were performed by this Institute. IR spectra were obtained with an IR-440 Shimadzu spectrophotometer. Low resolution mass spectra were obtained on a Finnigan GC-MS 4021 instrument.

*Preparation of 2-cynoethylperfluoroalkylsulfone 4*

A mixture of  $\text{C}_4\text{F}_9\text{I}$  (3.46 g, 10 mmol), (88%, 2.24 g, 12 mmol),  $\text{NaHCO}_3$  (0.93 g, 15 mmol),  $\text{CH}_3\text{CN}$  (7 ml) and water (7 ml) was heated at  $70^\circ\text{C}$ . After stirring for 8 h, the  $^{19}\text{F}$  NMR spectrum showed that the signal of  $\text{ICF}_2-$  (at  $-8.30$  ppm) had disappeared and a new peak at  $53.7$  ppm had appeared ( $-\text{CF}_2\text{SO}_2\text{Na}$ ). Acrylonitrile (1.1 g, 21 mmol) was added dropwise and the mixture continuously stirred for 8 h at  $70^\circ\text{C}$ .  $\text{HCl}$  (1 N, 5 ml) was added, the oily layer was separated and the aqueous layer was extracted with  $\text{Et}_2\text{O}$  (10 ml  $\times$  2). The organic layers were combined and dried over  $\text{Na}_2\text{SO}_4$ . After removing the solvent, the residue was sublimed under vacuum giving the crude product  $\text{C}_4\text{F}_9\text{SO}_2\text{CH}_2\text{CH}_2\text{CN}$  **4a** (2.1 g). Recrystallization from  $\text{CH}_3\text{CN}/\text{CHCl}_3$  gave the pure product. IR (KBr,  $\nu_{\text{max}}/\text{cm}^{-1}$ ): 2956, 2900 (s,  $\text{CH}_2$ ), 2250 (s, CN), 1368, 1330 (s,  $\text{SO}_2$ ), 1110, 1060 (s, C—F).  $\delta_{\text{H}}$  (ppm) ( $\text{CDCl}_3$ ): 3.70 (t,  $^3J_{\text{H-H}} = 7$  Hz,  $\text{SCH}_2$ ), 2.63 (t,  $\text{CH}_2$ ).  $\delta_{\text{F}}$  (ppm): 4.5 (s,  $\text{CF}_3$ ), 36.9 (s,  $\text{CF}_2\text{S}$ ), 44.4 (m,  $\text{CF}_2$ ), 49.0 (m,  $\text{CF}_2$ ). MS ( $m/e$ , %): 338 ( $\text{M}^+$ , 0.46), 283 ( $\text{M}^+ - \text{CH}_2\text{CH}_2\text{CN}$ , 11.34), 119 ( $\text{C}_2\text{F}_5^+$  or  $\text{M}^+ - \text{C}_4\text{F}_9$ , 63.71), 69 ( $\text{CF}_3^+$ , 76.54), 54 ( $\text{M}^+ - \text{C}_4\text{F}_9\text{SO}_2$ , 100.00).

Compound  $\text{ClC}_4\text{F}_8\text{SO}_2\text{CH}_2\text{CH}_2\text{CN}$  **4b** was prepared similarly.

IR (KBr,  $\nu_{\text{max}}/\text{cm}^{-1}$ ): 2992, 2900 (s,  $\text{CH}_2$ ), 2250 (s, CN), 1370, 1338 (m,  $\text{SO}_2$ ), 1112, 1065 (s, C—F).  $\delta_{\text{H}}$  (ppm): 3.75 (t,  $^3J_{\text{H-H}} = 7$  Hz,  $\text{SCH}_2$ ), 2.70 (t,  $\text{CH}_2$ ).  $\delta_{\text{F}}$  (ppm):  $-9.6$  (s,  $\text{ClCF}_2$ ), 41.0 (s,  $\text{CF}_2\text{S}$ ), 43.1 (m,  $\text{CF}_2$ ), 44.7 (m,  $\text{CF}_2$ ).

*Preparation of  $\text{C}_4\text{F}_9\text{SO}_2\text{CH}_2\text{CH}_2\text{COOCH}_3$  6a*

To a solution of  $\text{CH}_3\text{CN}/\text{H}_2\text{O}$  and  $\text{C}_4\text{F}_9\text{SO}_2\text{Na}$  (2.7 g, 9 mmol) prepared as above in a 50 ml flask equipped with a Teflon stop cock, methylvinylketone (1.4 g, 20 mmol) was added. After addition the stop cock was sealed. The reaction mixture was stirred at  $50-60^\circ\text{C}$  for 8 h. Similar work up as above gave **6a** (2.1 g). IR (KBr,  $\nu_{\text{max}}/\text{cm}^{-1}$ ): 2998, 3005 (s,  $\text{CH}_3$ ,  $\text{CH}_2$ ), 1720 (s, C=O), 1400, 1380 (s,  $\text{SO}_2$ ), 1200, 1160, 1130 (s, C—F).  $\delta_{\text{H}}$  (ppm) ( $(\text{CD}_3)_2\text{CO}$ ): 3.19 (t,  $^3J_{\text{H-H}} = 7$  Hz,  $\text{CH}_2$ ), 2.63 (t,  $\text{CH}_2$ ), 1.93 (s,  $\text{CH}_3$ ).  $\delta_{\text{F}}$  (ppm): 4.6 (s,  $\text{CF}_3$ ), 37.0 (m,  $\text{CF}_2\text{S}$ ), 45.3 (m,  $\text{CF}_2$ ), 49.3 (m,  $\text{CF}_2$ ). MS ( $m/e$ , %): 355 ( $\text{M}^+$ , 0.65), 311 ( $\text{M}^+ - \text{CH}_3\text{CO}$ , 0.30), 289 ( $\text{M}^+ - \text{SO}_2 - \text{H}$ , 0.41), 219 ( $\text{C}_4\text{F}_9^+$ , 1.33), 135 ( $\text{M}^+ - \text{C}_4\text{F}_9$ , 6.24), 107 ( $\text{CH}_3\text{COSO}^+$ , 8.87), 69 ( $\text{CF}_3^+$ , 5.62), 43 ( $\text{CH}_3\text{CO}^+$ , 100.00).

 *$\text{ClC}_4\text{F}_8\text{SO}_2\text{CH}_2\text{CH}_2\text{COCH}_3$  6b*

IR (KBr,  $\nu_{\text{max}}/\text{cm}^{-1}$ ): 2990, 3000 (s,  $\text{CH}_3$ ,  $\text{CH}_2$ ), 1740 (s, C=O), 1360, 1380 (s,  $\text{SO}_2$ ), 1100–1200 (s, C—F).  $\delta_{\text{H}}$  (ppm) ( $(\text{CD}_3)_2\text{CO}$ ): 3.20 (t,  $^3J_{\text{H-H}} = 7$  Hz,  $\text{CH}_2$ ), 2.73 (t,  $\text{CH}_2$ ), 1.97 (s,  $\text{CH}_3$ ).  $\delta_{\text{F}}$  (ppm):  $-8.3$  (s,  $\text{CF}_3$ ), 37.3 (m,  $\text{CF}_2\text{S}$ ), 44.7 (m,  $\text{CF}_2$ ), 47.3 (m,  $\text{CF}_2$ ).

*Preparation of 2-hydroxyethylperfluoroalkanesulfones 8*

Ethylene epoxide (1 g, 23 mmol) was added into a flask equipped with a Teflon stop cock and containing a solution of  $\text{ClC}_4\text{F}_8\text{SO}_2\text{Na}$  in  $\text{CH}_3\text{CN}$  which was prepared from  $\text{ClC}_4\text{F}_8\text{I}$  (4 g, 11 mmol),  $\text{Na}_2\text{S}_2\text{O}_4$  and  $\text{NaHCO}_3$  as above. After addition the stop cock was sealed and the mixture was heated at  $60^\circ\text{C}$  for 24 h. Similar work up as above gave  $\text{ClC}_4\text{F}_8\text{SO}_2(\text{CH}_2)_2\text{OH}$  (2.0 g) **8a**. IR ( $\nu_{\text{max}}/\text{cm}^{-1}$ ): 3410 (vs, OH), 2950, 2980, 1430 (s,  $\text{CH}_2$ ), 1352, 1335 (s,  $\text{SO}_2$ ), 1138–1110 (s, C—F), 1038 (C—O).  $\delta_{\text{H}}$  (ppm) ( $\text{CD}_3\text{Cl}$ ): 4.10 (d,  $\text{CH}_2\text{S}$ ), 3.96 (s, OH), 3.78 (d,  $\text{CH}_2\text{O}$ ).  $\delta_{\text{F}}$  (ppm):  $-8.6$  (s,  $\text{ClCF}_2$ ), 40.8 (m,  $\text{CF}_2\text{S}$ ), 43.0 (m,  $\text{CF}_2$ ), 45.0 (m,  $\text{CF}_2$ ).

Compounds **8b**, **8c** and **8d** were prepared similarly.

 *$\text{ClC}_4\text{F}_8\text{SO}_2\text{CH}_2\text{CH}(\text{OH})\text{CH}_3$  8b*

IR ( $\nu_{\text{max}}/\text{cm}^{-1}$ ): 3400 (s, OH), 2995, 2985, 1450 ( $\text{CH}_3$ ,  $\text{CH}_2$ ), 1370, 1350 (m,  $\text{SO}_2$ ), 1180–1110 (vs, C—F), 1030 (C—O).  $\delta_{\text{H}}$  (ppm): 4.03 (d,  $\text{CH}_2$ ), 3.93 (s, OH), 3.80 (m, CH), 1.23 (d,  $\text{CH}_3$ ).  $\delta_{\text{F}}$  (ppm):  $-9.0$  (s,  $\text{ClCF}_2$ ), 41.0 (m,  $\text{CF}_2\text{S}$ ), 43.3 (m,  $\text{CF}_2$ ), 44.3 (m,  $\text{CF}_2$ ).

 *$\text{C}_4\text{F}_9\text{SO}_2\text{CH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$  8c*

IR ( $\nu_{\text{max}}/\text{cm}^{-1}$ ): 3400 (s, OH), 2900, 2980 (m,  $\text{CH}_2$ ), 1430 (s,  $\text{CH}_2$ ), 1370, 1330 (m,  $\text{SO}_2$ ), 1110–1140 (s, C—F), 1040 (C—O).  $\delta_{\text{H}}$  (ppm) ( $\text{CD}_3\text{Cl}$ ): 4.13 (d,  $^3J_{\text{H-H}} = 7$  Hz,  $\text{CH}_2$ ), 4.03 (s, OH), 3.83 (m, CH), 3.50 (d,  $\text{CH}_2\text{Cl}$ ).  $\delta_{\text{F}}$  (ppm): 3.3 (s,  $\text{CF}_3$ ), 42.0 (m,  $\text{CF}_2\text{S}$ ), 44.5 (m,  $\text{CF}_2$ ), 48.3 (m,  $\text{CF}_2$ ). MS ( $m/e$ , %): 377/379 ( $\text{M}^+$ , 3.73/1.33), 359/361 ( $\text{M}^+ - \text{OH}$ , 2.40/1.02), 341 ( $\text{M}^+ - \text{Cl}$ , 1.04), 327 ( $\text{M}^+ - \text{ClCH}_2$ , 45.91), 219 ( $\text{C}_4\text{F}_9^+$ , 16.48), 93/95 ( $\text{M}^+ - \text{C}_4\text{F}_9\text{SO}_2$ , 36.49/12.57), 79/81 ( $\text{M}^+ - \text{C}_4\text{F}_9\text{SO}_2\text{CH}_2$ , 36.42/12.86), 57 ( $\text{C}_3\text{H}_5\text{O}^+$ , 48.08), 43 ( $\text{C}_2\text{H}_3\text{O}^+$ , 100.00).

**$\text{ClC}_4\text{F}_8\text{SO}_2\text{CH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$  **8d****

IR ( $\nu_{\text{max}}/\text{cm}^{-1}$ ): 3350 (s, OH), 2950 (m,  $\text{CH}_2$ ), 1430 (s,  $\text{CH}_2$ ), 1360, 1330 (m,  $\text{SO}_2$ ), 1110–1070 (s, C—F), 1040 (C—O).  $\delta_{\text{H}}$  (ppm) ( $\text{CD}_3\text{Cl}$ ): 4.23 (d,  $^3J_{\text{H-H}} = 7$  Hz,  $\text{CH}_2$ ), 4.07 (s, OH), 3.90 (m, CH), 3.65 (d,  $\text{CH}_2\text{Cl}$ ).  $\delta_{\text{F}}$  (ppm): -9.7 (s,  $\text{CF}_3$ ), 40.0 (m,  $\text{CF}_2\text{S}$ ), 42.3 (m,  $\text{CF}_2$ ), 44.3 (m,  $\text{CF}_2$ ). MS (m/e, %): 393/395/397 ( $\text{M}^+\text{H}$ , 6.65/4.75/0.72), 375/377 ( $\text{M}^+ - \text{OH}$ , 6.50/0.82), 343/345 ( $\text{M}^+ - \text{ClCH}_2$ , 14.41/5.58), 235/237 ( $\text{ClC}_4\text{F}_8^+$ , 3.21/1.14), 93/95 ( $\text{CH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}^+$ , 100/35.14), 85/87 ( $\text{ClCF}_2^+$ , 28.1/9.95), 79/81 ( $\text{CH}(\text{OH})\text{CH}_2\text{Cl}^+$ , 66.06/24.13), 43 ( $\text{C}_2\text{H}_3\text{O}^+$ , 100.00).

 **$\text{C}_4\text{F}_9\text{SO}_2\text{CH}_2\text{CH}_2\text{CONH}_2$  **9****

M.P. 74°C. IR (KBr,  $\nu_{\text{max}}/\text{cm}^{-1}$ ): 3350 (s,  $\text{NH}_2$ ), 2950, 2800 (m,  $\text{CH}_2$ ), 1710 (s, CO), 1405, 1360 (s,  $\text{SO}_2$ ), 1240–1120 (s, C—F).  $\delta_{\text{H}}$  (ppm) ( $(\text{CD}_3)_2\text{CO}$ ): 4.05 (s,  $\text{NH}_2$ ), 2.92 (t,  $^3J_{\text{H-H}} = 7$  Hz,  $\text{CH}_2$ ), 2.95 (t,  $\text{CH}_2$ ).  $\delta_{\text{F}}$  (ppm): 4.5 (s,  $\text{CF}_3$ ), 37.0 (m,  $\text{CF}_2\text{S}$ ), 44.3 (m,  $\text{CF}_2$ ), 49.3 (m,  $\text{CF}_2$ ). MS (m/e, %): 339 ( $\text{M}^+ - \text{NH}_2$ , 5.27), 286 ( $\text{M}^+ - \text{CF}_3$ , 1.00), 219 ( $\text{C}_4\text{F}_9^+$ , 2.46), 169 ( $\text{C}_3\text{F}_7^+$ , 2.64), 73 ( $\text{M}^+\text{H} - \text{C}_4\text{F}_9\text{SO}_2$ , 28.2), 69 ( $\text{CF}_3^+$ , 47.71), 55 ( $\text{CH}_2=\text{CHCO}^+$ , 100.00), 45 ( $\text{M}^+\text{H} - \text{C}_4\text{F}_9\text{SO}_2\text{CH}_2\text{CH}_2$ , 50.19).

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