Available online at www.sciencedirect.com
SCIENCE DIRECT®
Journal of Fluorine Chemistry 124 (2003) 55–59
JOURNAL OF FLUORINE CHEMISTRY
www.elsevier.com/locate/jfluchem

Perfluoroalkylation of 2-mercaptoethanol as a key step for a new synthesis of perfluoroalkyl vinyl sulfides, sulfoxides and sulfones

Emmanuel Magnier, Marc Tordeux, Régis Goumont, Karine Magder, Claude Wakselman*

SIRCOB-CNRS, Bâtiment Lavoisier, Université de Versailles-Saint-Quentin, 45 avenue des Etats-Unis, 78035 Versailles, France

Received 28 April 2003; received in revised form 28 April 2003; accepted 24 June 2003

Abstract

We have developed a new method of perfluoroalkylation of thiols, using a mixture of sodium formate/sodium sulfite to generate a sulfoxylate radical anion. This method is compatible with alcoholic functionality and was applied to mercaptoethanol. The obtained compounds were transformed into perfluoroalkyl vinyl sulfides, sulfoxides and sulfones.

Keywords: Perfluoroalkylation; Sodium formate; Sodium sulfite; Thiols

1. Introduction

Perfluoroalkyl vinyl sulfides, sulfoxides and sulfones are interesting building blocks for the elaboration of more complex molecules. However, the synthesis of these fluorinated sulfides and sulfoxides has been seldom described. This could be due to the use of the toxic trifluoromethanesulfenyl chloride in the first step of the trifluoromethyl vinyl sulfide [1] and sulfoxide [2] preparations. The corresponding sulfones have attracted more attention. As pointed out by Hendrickson et al., α,β-unsaturated triflones are less easily available than simple alkyl triflones [3]. Substituted examples can be prepared by various methods [3–5] but vinyl triflone itself has been made by a multistep route involving again the use of the toxic trifluoromethanesulfenyl chloride [2,3]. The homologous nonaflone has been prepared by condensation of nonafluorobutanesulfonylmethylmagnesium iodide with formaldehyde in 30% yield, followed by action of phosphorus pentachloride and triethylamine [6]. We report here a new route to perfluoroalkyl vinyl sulfides, sulfoxides and sulfones based on the perfluoroalkylation of 2-mercaptoethanol (Scheme 1).

Usually, condensation of perfluoroalkyl halides with alkyl thiolates produces a notable quantity of disulfides and the yield of perfluoroalkylsulfides is rather limited. However, we have shown recently that the presence of a sulfoxylate radical anion precursor in the medium improves the yield of the perfluoroalkylthioethers [7] because disulfides are perfluoroalkylated in these conditions [8,9] (Scheme 2).

For the generation of the sulfoxylate radical anion, we chose the mixture sodium formate/sodium sulfite that we employed previously [9]. Owing to the acidity of the thiol, sodium bisulfite is probably formed in the medium. Sodium formate is known to behave as a reductant [10] (see Scheme 3 for a tentative explanation of the sulfoxylate radical anion formation).

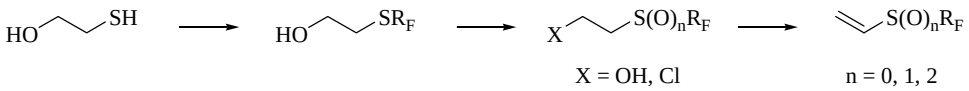
Combination of the reactions included in Schemes 2 and 3 can lead to the formation of perfluoroalkylsulfides in fair yields. Owing to the radical nature of the reaction [8,9], this methodology is compatible with the presence of non-protected hydroxy groups. Application of this method to 2-mercaptoethanol can produce a fluorinated sulfide bearing an hydroxy group on the adjacent carbon (Scheme 1; first step). This perfluoroalkylation reaction and the following transformations are detailed below.

2. Results

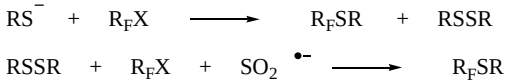
In order to avoid the use of low-boiling products, we have performed the alkylation of 2-mercaptoethanol with long chain perfluoroalkyl iodides. As depicted in Scheme 1, our synthetic plan was composed of three main parts.

* Corresponding author. Tel.: +33-1-39-25-44-66; fax: +33-1-39-25-44-52.
E-mail addresses: magnier@chimie.uvsq.fr (E. Magnier), wakselma@chimie.uvsq.fr (C. Wakselman).

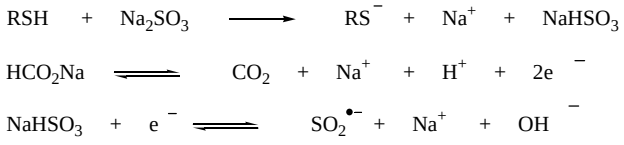
0022-1139/$ – see front matter © 2003 Elsevier B.V. All rights reserved.
doi:10.1016/S0022-1139(03)00170-2



Scheme 1. New route to perfluoroalkyl vinyl sulfides, sulfoxides and sulfones.



Scheme 2. Perfluoroalkylation reaction in the presence of the sulfoxylate radical anion.



Scheme 3. Tentative explanation of the sulfoxylate radical anion formation.

2.1. Perfluoroalkylation of 2-mercaptoethanol

Treatment of 2-mercaptoethanol by perfluoroalkyl iodides in the presence of sodium bisulfite and sodium formate gave rise to the corresponding fluorinated sulfides **1** in fair yields. The mode of introduction of the reagents is of importance for this reaction: they must be added as fast as possible under ''Barbier's conditions'' (Scheme 4).

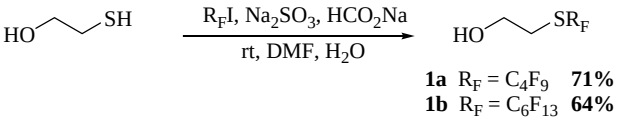
2.2. Chlorination and oxidation of perfluoroalkyl sulfides

The hydroxy derivatives **1** were easily converted into chlorides **2** under standard conditions. The compounds were then directly oxidized into sulfoxides **3** or sulfones **4** using trifluoroperacetic acid [11]. All compounds were isolated in good yield and fully characterized (Scheme 5).

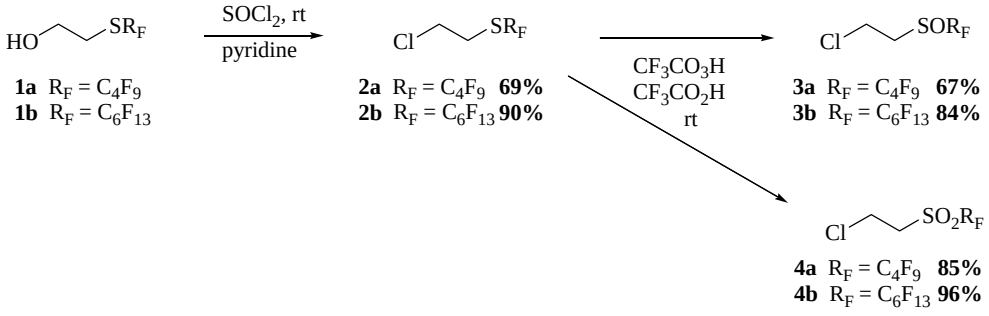
2.3. Synthesis of the vinyl derivatives

Vinyl perfluoroalkyl sulfide **5**, sulfoxide **6** and sulfone **7** were prepared by elimination of HCl from the corresponding precursors **2b**, **3b** and **4b**. Nevertheless, experimental conditions had to be adapted to each derivative (Scheme 6).

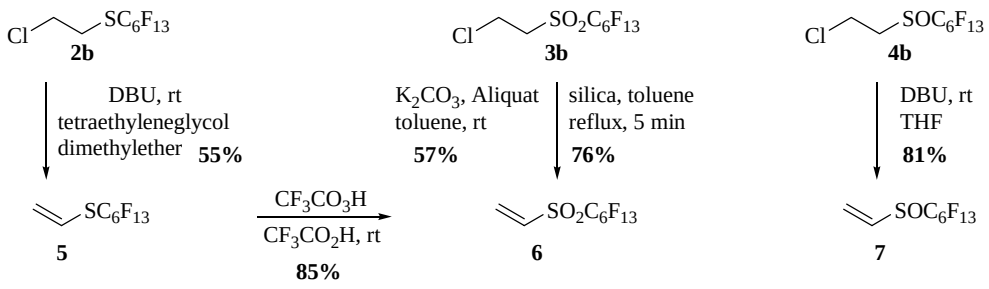
The perfluorohexyl sulfide **2b** was treated by 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) as a base. The rather



Scheme 4. Formation of perfluoroalkylsulfides in the presence of a mixture sodium sulfide/sodium formate.



Scheme 5. Chlorination of the hydroxy group and oxidation of the sulfur atom.



Scheme 6. Transformation of the chlorinated intermediates to the vinyl derivatives.

volatile compound **5** was inseparable by distillation from usual extraction solvents. However, utilization of a higher boiling point solvent such as tetraethyleneglycol dimethylether allowed a direct distillation from the crude mixture and afforded pure vinyl derivative **5** in an acceptable 55% yield.

The sulfoxide **7** was also prepared by elimination reaction using DBU as base. After classical final treatment, distillation gave rise to the molecule **7** in 81% yield.

These conditions were not suitable to perform the elimination from the sulfone **3b**, which is poorly stable in basic media. Under phase transfer catalysis, sulfone **6** was isolated in only 57% yield, with many secondary products present. However, we had observed partial elimination of HCl during purification of the sulfone **3b** by flash chromatography on a silica gel column. We took advantage of this observation to develop an easy and quick method of formation of the vinylsulfone **6**. Heating a mixture of **3b**, silica and toluene for a short period gave rise to the desired sulfone **6**. Nevertheless, this method was not really suitable for large quantities. We then developed a new route to the compound **6** by direct oxidation of the sulfide **5** with an excess of trifluoroperacetic acid. In these conditions, sulfone **6** was isolated in good yield without any oxidation of the double bond. We assume that the same reaction is also possible starting from the sulfoxide **7**.

In summary, we have developed an easy access to vinyl perfluoroalkyl sulfides, sulfoxides and sulfones. Further synthetic use of these compounds are under current investigation in the laboratory and will be reported in due course.

3. Experimental

3.1. General

Melting points were determined on a Mettler FP61 apparatus. NMR spectra were recorded in $CDCl_3$ solutions, on a Bruker AC-300 spectrometer. Reported coupling constants and chemicals shifts were based on a first-order analysis. Internal reference was the residual peak of $CHCl_3$ (7.27 ppm) for ^1H (300 MHz) NMR spectra, central peak of $CDCl_3$ (77 ppm) for ^{13}C (75 MHz) NMR spectra, internal $CFCl_3$ (0 ppm) for ^{19}F (282 MHz) NMR spectra. Chemical shifts are reported in parts per million (ppm) and constants J in Hz. The peaks are characterized by s (singlet), d (doublet), t (triplet), m (multiplet). Mass spectra were carried out at the Ecole Normale Supérieure (Paris). IR spectra were recorded on a Nicolet 400SD spectrometer. Elemental analyses were determined by the Microanalytical Laboratory of the CNRS (Gif sur Yvette). Column chromatography was performed with Merck silica gel (70–230 mesh). Reagents were commercially available and used as received. All solvents were distilled prior use and reactions were usually carried out under argon except in the case of perfluoroalkylation reactions.

3.2. S-perfluoroalkylation of mercaptoethanol

3.2.1. General procedure

To a mixture of DMF (20 ml) and water (4 ml) were added mercaptoethanol (1.5 g, 20 mmol) and nonafluorobutyl or tridecafluorohexyl iodide (20 mmol). Then $Na_2SO_3{\cdot}7H_2O$ (4.8 g, 20 mmol) and HCO_2Na (2 g, 20 mmol) were rapidly added and the reaction mixture was stirred overnight. Water (100 ml) and ether (100 ml) were added and the reaction mixture was filtered through on Celite 545®. The organic layer was separated and the aqueous layer was extracted with ether (3×50 ml). The combined organic layers were washed with dilute HCl (100 ml), water (100 ml), dried over $MgSO_4$ and concentrated under vacuum. The sulfides were obtained as pure clear yellow liquids without further purification.

3.2.2. 2-Hydroxyethyl nonafluorobutyl sulfide (*1a*)

Yellow liquid (71%) which had: Anal. Calcd for $C_6H_5F_9OS$: C, 24.33%, H, 1.70%; found C, 24.47%, H, 1.71%; ^1H NMR: 2.35 (1H, s, OH), 3.14 (2H, t, $J = 6.2$ Hz, SCH_2), 3.89 (2H, t, $J = 6.2$ Hz, OCH_2); ^{13}C NMR: 31.3 (CH_2), 61.3 (OCH_2); ^{19}F NMR: −126.0 (2F, m, CF_2), −121.1 (2F, m, CF_2), −87.4 (2F, m, SCF_2), −81.6 (3F, m, CF_3); EI (*m*/*z*): 279 $(M - OH)^+$ (100), 296 $(M)^+$ (70).

3.2.3. 2-Hydroxyethyl tridecafluorohexyl sulfide (*1b*)

Yellow oil (64%) which had: Anal. Calcd for $C_8H_5F_{13}OS$: C, 24.25%, H, 1.27%, found C, 24.34%, H, 1.25%; ^1H NMR: 2.35 (1H, s, OH), 3.14 (2H, t, $J = 6.1$ Hz, SCH_2), 3.88 (2H, t, $J = 5.9$ Hz, OCH_2); ^{13}C NMR: 31.5 (CH_2), 61.4 (OCH_2); ^{19}F NMR: −126.8 (2F, m), −123.4 (2F, m), −122.0 (2F, m), −120.4 (2F, m), −87.4 (2F, m, SCF_2), −81.6 (3F, m, CF_3); IR (Nujol, cm^{-1}): 1358, 1409, 2884, 2945, 3350; EI (*m*/*z*): 379 $(M - OH)^+$, 396 $(M)^+$.

3.3. Chlorination of hydroxysulfides

3.3.1. General procedure

To a solution of the perfluoroalkylated mercaptoethanol (1 eq.) in pyridine (1 eq.) was added dropwise thionyl chloride (2 eq.). The reaction mixture was stirred at this temperature for 1 h and then refluxed for an additional hour. After cooling, water was added. The reaction mixture was then extracted with ether. The organic layer was washed with a solution of NaOH (5%), with water and then dried over $MgSO_4$. The solvent was removed under vacuum and the oily residue was distilled under reduced pressure to give the chlorosulfide.

3.3.2. 2-Chloroethyl nonafluorobutyl sulfide (*2a*)

Colorless liquid (69%) which had: bp (15 mmHg): 46 °C; Anal. Calcd for $C_6H_4ClF_9S$: C, 22.91%, H, 1.28%, found C, 21.22%, H, 1.11%; HRMS calc for $C_6H_5F_9ClS$: 314.9657 $(M + H)^+$, found: 314.9778; ^1H NMR: 3.29 (2H, t, $J = 7.5$ Hz, SCH_2), 3.74 (2H, t, $J = 7.5$ Hz, $ClCH_2$); ^{13}C: 30.53

(CH_2), 42.45 (OCH_2);^{19}F NMR: −126.0 (2F, m), −121.2 (2F, m), −87.5 (2F, m, SCF_2), −81.5 (3F, m, CF_3); EI (*m/z*): 279 $(M - Cl)^+$, 219 $(C_4F_9)^+$, 69 $(CF_3)^+$.

*3.3.3. 2-Chloroethyl tridecafluorohexyl sulfide (**2b**)*

Colorless liquid (90%) which had: bp (15 mmHg): 80 °C; Anal. Calcd for $C_8H_4ClF_{13}S$: C, 23.18%, H, 0.97%, found C, 23.50%, H, 0.98%; ^{1}H NMR: 3.29 (2H, t, $J = 7.5$ Hz, SCH_2), 3.4 (2H, t, $J = 7.5$ Hz, $ClCH_2$); ^{13}C NMR: 30.6 (CH_2), 42.4 (OCH_2);^{19}F NMR: −127.1 (2F, m), −123.6 (2F, m), −122.2 (2F, m), −120.6 (2F, m), −87.7 (2F, m, SCF_2), −81.9 (3F, m, CF_3); EI (*m/z*): 379 $(M - Cl)^+$, 69 $(CF_3)^+$.

3.4. Oxidation of chlorosulfides

3.4.1. Oxidation to sulfoxide

To a solution of chlorosulfide (12 mmol) in trifluoroacetic acid (6.5 ml) at room temperature was added freshly prepared trifluoroperacetic acid (3 ml, 12 mmol) The reaction mixture was stirred overnight then water (100 ml) and ether (100 ml) were added. The organic layer was separated and washed with a saturated solution of $NaHCO_3$ (100 ml), with water (100 ml) and dried over $MgSO_4$. The solvent was removed under vacuum and the residue was purified by column chromatography using a pentane/ether (9/1) mixture as eluent.

*3.4.1.1. 2-Chloroethyl nonafluorobutyl sulfoxide (**3a**).* Colorless liquid (67%) which had: Calcd for $C_6H_4ClF_9OS$: C, 21.80%, H, 1.22%, found C, 21.85%, H, 1.19%; ^{1}H NMR: 3.30 (1H, m, $SOCH_2$), 3.50 (1H, m, $^2J = 13.1$ Hz, $^3J = 4.0$ Hz, $SOCH_2$), 4.00 (2H, m, $ClCH_2$); ^{13}C NMR: 35,5 (Cl–CH_2), 50,7 (S–CH_2); ^{19}F NMR: −126.5 (2F, m), −121.8 (2F, m), −121.2 (1F, dt, $^1J_F = 249.2$ Hz,$^3J_F = 12.7$ Hz, SO–CF_2), −115.4 (1F, dt, $^1J_F = 249.2$ Hz, $^3J_F = 12.7$ Hz, $SOCF_2$), −81.3 (3F, m, CF_3).

*3.4.1.2. 2-Chloroethyl tridecafluorohexyl sulfoxide (**3b**).* White solid (84%) which had: mp 52.1 °C; Anal. Calcd for $C_8H_4ClF_{13}OS$: C, 22.31%, H, 0.94%, found C, 22.42%, H, 0.98%; ^{1}H NMR: 3.27 (1H, m, $^2J = 13.1$ Hz, $^3J = 4.0$ Hz, $SOCH_2$), 3.58 (1H, m, $^2J = 13.1$ Hz, $^3J = 4.0$ Hz, $SOCH_2$), 4.04 (2H, m, $ClCH_2$); ^{13}C NMR: 35.5 (Cl–CH_2), 50.8 (S–CH_2); ^{19}F NMR: −126.6 (2F, m), −123.2 (2F, m), −122.4 (2F, m), −121.0 (2F, m), −120.9 (1F, m, $SOCF_2$), −120.4 (1F, m, $SOCF_2$), −81.2 (3F, m, CF_3); IR (Nujol, cm^{-1}): 1445, 1634, 2848, 2925, 2950, 3432; CI NH_3 (*m/z*): 448 $(M + NH_4)^+$, 431 $(M + H)^+$.

3.4.2. Oxidation to sulfone

To a solution of chlorosulfide (12 mmol) in trifluoroacetic acid (10 ml) at room temperature was added freshly prepared trifluoroperacetic acid (9 ml, 12 mmol) and the reaction mixture was stirred overnight. The addition of the peracid solution (9 ml) was regularly repeated every 12 h. After completion of the reaction water (50 ml) was added, then the reaction mixture was extracted by CH_2Cl_2 (3×100 ml). Combined organic layers were washed with a saturated solution of $NaHCO_3$ (50 ml), with water (50 ml), dried over $MgSO_4$ and concentrated under vacuum to give the chlorosulfone as a white solid.

*3.4.2.1. 2-Chloroethyl nonafluorobutyl sulfone (**4a**).* White solid (85%) which had: mp 50 °C; Anal. Calcd for $C_6H_4ClF_9SO_2$: C, 20.79%, H, 1.16%, found C, 20.06%, H, 1.04%; ^{1}H NMR: 3.72 (2H, t, $J = 7.7$ Hz, SO_2CH_2), 3.95 (2H, t, $J = 7.7$ Hz, $ClCH_2$); ^{13}C NMR: 33.6 (CH_2), 53.4 (SO_2CH_2); ^{19}F NMR: −126.4 (2F, m), −121.6 (2F, m), −113.2 (2F, m, SCF_2), −81.2 (3F, m, CF_3).

*3.4.2.2. 2-Chloroethyl tridecafluorohexyl sulfone (**4b**).* White solid (96%) which had: mp 76.0 °C; Anal. Calcd for $C_8H_4ClF_{13}O_2S$: C, 21.51%, H, 0.90%, found C, 21.80%, H, 0.69%; ^{1}H NMR: 3.75 (2H, t, $J = 7.7$ Hz, SO_2CH_2), 3.96 (2H, t, $J = 7.5$ Hz, $ClCH_2$); ^{13}C NMR: 33.6 (CH_2), 53.6 (SO_2CH_2); ^{19}F: −126.6 (2F, m), −123.1 (2F, m), −122.1 (2F, m), −120.7 (2F, m), −113.0 (2F, m, SO_2CF_2), −81.2 (3F, m, CF_3); IR (Nujol, cm^{-1}): 1378, 1450, 2837, 2960, 3411.

3.5. Synthesis of the vinylic compounds

*3.5.1. Vinyl tridecafluorohexyl sulfide (**5**)*

DBU (3.35 g, 22.1 mmol) was added dropwise to a solution of chlorosulfide **2b** (7.6 g, 18.4 mmol) in tetraethyleneglycol dimethylether (76 ml). The reaction mixture was stirred overnight and was distilled under reduced pressure to give the vinylsulfide **5** as a colorless liquid in a 55% yield which had: bp (15 mmHg): 28 °C; HRMS calc for $C_8H_3F_{13}S$: 378.9826 $(M + H)^+$, found: 378.9825; ^{1}H NMR: 5.74 (1H, d, $J_{cis} = 9.2$ Hz, $SCHCH_2$), 5.79 (1H, d, $J_{trans} = 16.7$ Hz, $SCHCH_2$), 6.49 (1H, dd, $J_{cis} = 9.2$ Hz, $J_{trans} = 16.7$ Hz, SCH); ^{13}C NMR: 120,7 (SCH), 125.0 (CH_2); ^{19}F NMR: −126.8 (2F, m), −123.5 (2F, m), −122.2 (2F, m), −120.4 (2F, m), −89.5 (2F, m, SCF_2), −81.5 (3F, m, CF_3); IR (Nujol, cm^{-1}): 1363, 1588, 2346, 2960, 3022, 3058; CI NH_3 (*m/z*): 335 $(C_6F_{13} + CH_4)^+$, 359 $(M - F)^+$, 379 $(M + H)^+$.

*3.5.2. Vinyl tridecafluorohexyl sulfoxide (**7**)*

DBU (1.95 g, 12.8 mmol) was added dropwise to a solution of chlorosulfoxide **3b** (5 g, 11.6 mmol) in tetrahydrofuran (50 ml) under argon. At the completion of the addition, water (100 ml) was immediately added to the reaction mixture. The organic layer was separated, washed with water (50 ml), dried over $MgSO_4$ and concentrated under vacuum at room temperature to give an oily product which was distilled under reduced pressure to give 3.7 g (81%) of the vinylsulfoxide **7** as a colorless liquid which had: bp (15 mmHg): 78 °C; HRMS calc for $C_8H_3F_{13}OS$: 394.9826 $(M + H)^+$, found: 394.9770; ^{1}H NMR: 6.37 (1H, d, $J_{cis} = 9.5$ Hz, $SOCHCH_2$), 6.47 (1H, d, $J_{trans} = 16.4$ Hz,

$SOCHCH_2$), 6.74 (1H, ddd, $J_{cis} = 9.8$ Hz, $J_{trans} = 16.7$ Hz, $J_F = 2.0$ Hz, $SOCHCH_2$); ^{13}C NMR: 129.1 (SCH), 133.0 (CH_2); ^{19}F NMR: −126.7 (2F, m), −123.3 (2F, m), −122.5 (2F, m), −120.9 (2F, m), −120.9 (1F, m, $SOCF_2$), −113.9 (1F, m, $SOCF_2$), −81.3 (3F, m, CF_3); IR (Nujol, cm^{-1}): 1368, 3012, 3048, 3109; CI NH_3 (*m/z*): 395 $(M + H)^+$.

3.5.3. vinyl tridecafluorohexyl sulfone **6**

3.5.3.1. Method A. To a solution of chlorosulfone **3b** (0.3 g, 0.67 mmol) in toluene (0.5 ml) was added K_2CO_3 (3 eq., 279 mg) and three drops of Aliquat 336. The reaction mixture was stirred for 2 h. The crude mixture was applied to a silica column. Elution with CH_2Cl_2 and evaporation of the solvent afforded 254 mg (57%) of the sulfone **6** which had mp: 66.4 °C; HRMS calc for $C_8H_3F_{13}O_2S$: 410.9724 $(M + H)^+$, found: 410.9720; ^1H NMR: 6.61 (3H, m); ^{13}C NMR: 131.4 (SCH); 142.1 (CH_2); ^{19}F NMR: −126.6 (2F, m), −123.2 (2F, m), −122.3 (2F, m), −120.6 (2F, m), −113.5 (2F, m, SO_2CF_2), −81.3 (3F, m, CF_3); IR (Nujol, cm^{-1}): 2843, 2945, 3073, 3421; CI NH_3 (*m/z*): 411 $(M + H)^+$, 429 $(M + CH_4)^+$.

3.5.3.2. Method B. To a mixture of chlorosulfone **3b** (1 g, 2.24 mmol) and silica gel (100 g) was added the minimum amount of toluene to obtain a jelly-like reaction mixture which was heated at 80 °C for 5 min under stirring. The silica gel was then filtered off and washed with dichloromethane. The solvent was removed under vacuum and the residue was purified by column chromatography using a pentane/ether (9/1) mixture as eluent to give 0.7 g (76%) of the sulfone **6**.

3.5.3.3. Method C. To a solution of the vinyl tridecafluorohexylsulfide (**5**) (2 g, 5 mmol) at room temperature in trifluoroacetic acid (30 ml) was added freshly prepared trifluoroperacetic acid (10 mmol, 10 ml) The reaction mixture was kept under stirring overnight then water (100 ml) and ether (100 ml) were added. The organic layer was separated and washed with a saturated solution of $NaHCO_3$ (100 ml), with water (100 ml), dried over $MgSO_4$ and concentrated to afford 1.9 g (85%) of pure sulfone **6**.

Acknowledgements

This work was supported by the Centre National de la Recherche Scientifique. We thank Dr. André Loupy for helpful discussions, Joëlle Moïse for performing experiments and Atofina for the generous gift of the perfluoroalkyl halides.

References

[1] J.F. Harris, J. Org. Chem. 32 (1967) 2063–2074.
[2] I.L. Knunyants, I.N. Rozhov, A.M. Aleksandrov, L.M. Yagupolskii, J. Gen. Chem. USSR (Engl. Transl.) 37 (1967) 1210–1213.
[3] J.B. Hendrickson, D.D. Sternbach, K.W. Bair, Acc. Chem. Res. 10 (1977) 306–312.
[4] R. Goumont, K. Magder, M. Tordeux, J. Marrot, F. Terrier, C. Wakselman, Eur. J. Org. Chem. (1999) 2969–2976.
[5] T. Billard, B.R. Langlois, Tetrahedron 55 (1999) 8065–8074.
[6] K. Laping, M. Hanack, Tetrahedron Lett. (1979) 1309–1310.
[7] E. Anselmi, J-C. Blazejewski, M. Tordeux, C. Wakselman, J. Fluorine Chem. 105 (2000) 41–44.
[8] C. Wakselman, M. Tordeux, J.-L. Clavel, B. Langlois, J. Chem. Soc., Chem. Commun. (1991) 993–994.
[9] J-L. Clavel, B. Langlois, R. Nantermet, M. Tordeux, C. Wakselman, J. Chem. Soc., Perkin Trans. 1 (1992) 3371–3375.
[10] H.W. Gibson, Chem. Rev. 69 (1969) 673–692.
[11] C.G. Vernier, T.G. Squires, Y.Y. Chen, G.P. Hussman, J.C. Shei, J. Org. Chem. 47 (1982) 2969–2976.