Journal of Fluorine Chemistry, *67* (1994) 27–31

A new cyclic fluorinated trisulfone $(CF_2SO_2)_3$ and fluorinated derivatives: $SO_2(CF_2SO_2F)_2$, $CF_2(SO_2CF_3)_2$, $CF_3SO_2CF_2SO_2F$, $CF_2(SO_2F)_2$ and $CF_3SO_2CF_2SO_2CF_2SO_2F$

Alfred Waterfeld
Anorganische Chemie 2, Ruhr-Universität Bochum, P.O. Box 102148, D-4630 Bochum (Germany)

Ilse Weiss, Heinz Oberhammer
Institut für Anorganische und Theoretische Chemie, Universität Tübingen, D-7400 Tübingen (Germany)

Gary L. Gard*[†]
Department of Chemistry, Portland State University, Portland, OR 97207-0751 (USA)

and Rudiger Mews*
Institut für Anorganische und Physikalische Chemie, Universität Bremen, NW2, Leobenerstrasse, D-2800 Bremen 33 (Germany)

(Received November 24, 1992; accepted April 23, 1993)

Abstract

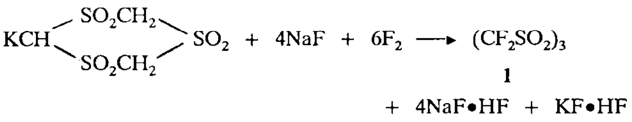
The preparation and characterization of the novel perfluorinated sulfone $(CF_2SO_2)_3$ (**1**) has been achieved via static fluorination of the potassium salt of $(CH_2SO_2)_3$. In addition to this product, the highly interesting by-products $SO_2(CF_2SO_2F)_2$ (**2**), $CF_2(SO_2CF_3)_2$ (**3**), $CF_3SO_2CF_2SO_2F$ (**4**), $CF_2(SO_2F)_2$ (**5**) and $CF_3SO_2CF_2SO_2CF_2SO_2F$ (**6**) were also prepared. Infrared, NMR and mass spectra are presented in order to support the assigned structures; only compound **5** is known. The gas-phase molecular structure of **1** has been studied by electron-diffraction methods. Assuming C_2 overall symmetry with equal axial and equatorial S=O and C−F bond lengths, nine geometric parameters were required to describe the structure of **1**. The six-membered ring possesses a chair conformation with C_3 symmetry and the following ring parameters: C−F = 1.338(4), O=S = 1.418(2), C−S = 1.866(4) Å and angles <CSC = 100.2(7), <FCF = 109.2(7), <OSO = 125.9(14)°.

Introduction

Static fluorination under controlled conditions has been found to be effective in producing a number of interesting fluorine-containing products such as $F_2C(SF_3)_2$ [1], $F_5SCF_2SO_2F$ [2], $F_2C(COF)_2$ [3] and $F_2C(OCF_2-CF_2-SO_2F)_2$ [4]. Application of this technique has been extended to the fluorination of the potassium salt of $(CH_2SO_2)_3$ [5]. It is interesting to note that the fluorinated dimer $(CF_2SO_2)_2$ has been prepared [6]. This material has failed to produce the trimer, $(CF_2SO_2)_3$, via decomposition/recombination reactions. Previously, the interesting trisulfone $[(CF_3)_2CSO_2]_3$ had been prepared and found to give various sulfones and sulfonyl fluoride derivatives [7]. The new sulfone $(CF_2SO_2)_3$ (**1**) gives rise to a number of interesting sulfonyl fluorides. The utilization of fluorocarbon sulfonyl fluorides as precursors to ion-exchange resins, surface-active agents and in the preparation of novel strong sulfonic acids continues to make them the subject of considerable study [8–11].

Results and discussion

Static fluorination of the potassium salt of $(CH_2SO_2)_3$ results in the formation of the perfluorinated product $(CF_2SO_2)_3$:



$$KCH\begin{matrix} SO_2CH_2 \\ SO_2CH_2 \end{matrix}SO_2 + 4NaF + 6F_2 \longrightarrow \underset{\mathbf{1}}{(CF_2SO_2)_3} + 4NaF{\bullet}HF + KF{\bullet}HF \qquad (1)$$

with excess fluorine, ring-opening occurs via C–S bond cleavage forming SO_2F_2/CF_4 by-products. The following

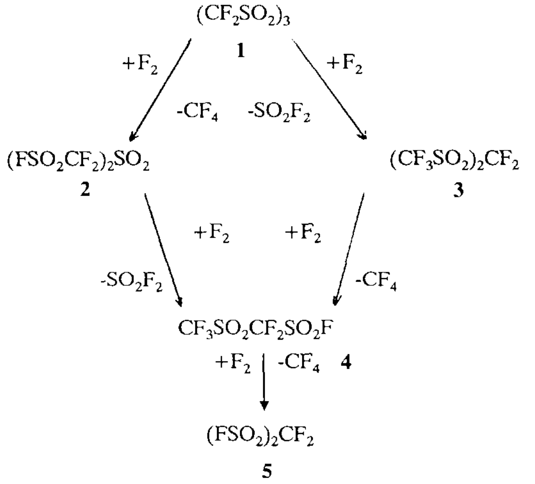
---

*Author to whom correspondence should be addressed.

[†]Senior Fulbright Fellow 1989/90 at the Institut für Anorganische und Physikalische Chemie der Universität, Bremen.

0022-1139/94/$07.00
SSDI 0022-1139(93)02925-5

scheme easily explains the formation of SO_2F_2 and CF_4, and also accounts for a number of interesting derivatives which were formed. These derivatives include $(CF_3SO_2)_2CF_2$, $(FSO_2CF_2)_2SO_2$, $CF_3SO_2CF_2SO_2F$, $(FSO_2)_2CF_2$ and $CF_3SO_2CF_2SO_2CF_2SO_2F$.



(2)

The structures of all the above products have been determined from their ^{19}F NMR spectra; in a separate run, GC–MS product analysis revealed the presence of a small amount of $CF_3(SO_2CF_2)_2SO_2F$ (**6**). The ^{19}F NMR spectrum of **1** shows only a singlet at -105.5 ppm. For the sultone $\overline{CF_2CF_2OSO_2}$ and $(CF_2SO_2)_2$, the chemical shifts of the CF_2 group bonded to the SO_2 group were found at -102.6 ppm and -95.9 ppm, respectively; the corresponding ^{13}C NMR chemical shifts for **1** and $\overline{CF_2CF_2OSO_2}$ were at 118.9 and 126 ppm [12]. In compounds **2** and **6**, the ^{19}F NMR resonances at -92.7, -93.5, -93.7 and 51.7–51.4 ppm were in the regions characteristic of CF_2 and SO_2F groupings [11]. In the compound $(CF_3CF_2SO_2CF_2CF_2CF_2)_2$, the CF_2 groups bonded to SO_2 had chemical shift values in the -106.6 to -111.3 ppm range [13]; for compounds **2** and **6**, the ^{19}F NMR resonances at -92.7 to -93.7 ppm are assigned to the CF_2 group. The chemical shift for the FSO_2 groups are in the region found for perfluoroalkyl sulfonyl fluorides [2, 14]. For compound **5**, the ^{19}F NMR resonances, splitting patterns and coupling constants for the CF_2 and SO_2F groupings are as expected [2, 14].

The infrared (IR) spectra of the new compounds have common features. The characteristic SO_2(asym.) and SO_2(sym.) stretching frequencies were found for compounds **1** and **2** in the 1446–1479 and 1223–1245 cm^{-1} spectral regions, respectively. The S–F absorption for **2** was found at 810 cm^{-1}. These assignments agree well with the results obtained for other fluorosulfonyl derivatives [2, 10, 14, 15]. In $(CF_2SO_2)_2$, the SO_2(asym.) and SO_2(sym.) stretching frequencies were found at 1401 and 1201 cm^{-1}, respectively [6]. For compounds containing isolated CF_2 groups, the absorption region allocated for C–F stretching modes [16] is 1050–1250 cm^{-1}.

The mass spectra for compounds **1** and **2** are listed in the Experimental section. Molecular peaks for $(MH)^+$ were found for compounds **1**, **2** and **6**, while $(M-F)^-$ peaks were found for compounds **1** and **2**. Cracking patterns were obtained that support the assigned structures.

A study of the reaction conditions showed that at a temperature of 20 °C and with a rate of F_2 addition of 1 g d^{-1}, only a trace of **1** was formed. However, at 0 °C and with F_2 addition reduced to 0.7 g d^{-1}, the yield of **1** dramatically increased to 18%.

It was of interest to study the gas-phase structure of **1**. Analysis of the experimental radial distribution curve (Fig. 1) results in a chair conformation for the six-membered ring. In the least-squares refinement, the molecular intensities (Fig. 2) were modified with a

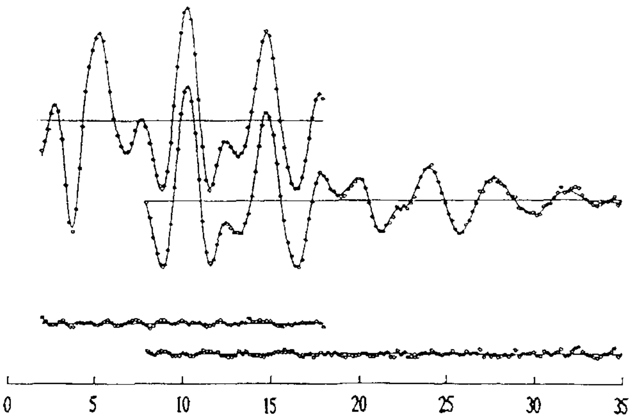


Fig. 1. Experimental radial distribution function and difference curve for $(SO_2CF_2)_3$ (**1**).

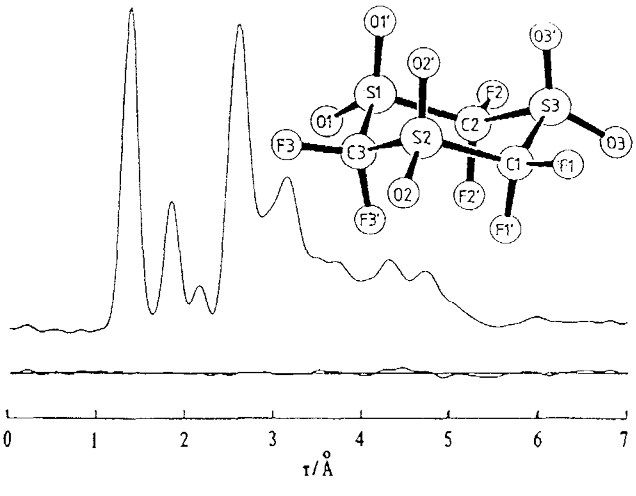


Fig. 2. Experimental (dots) and calculated (full line) molecular intensities and differences for $(SO_2CF_2)_3$ (**1**).

diagonal weight matrix and known scattering amplitudes and phases were used [17]. Assuming C_3 overall symmetry with equal axial and equatorial S=O and C−F bond lengths, nine geometric parameters are required to describe the geometric structure of **1**. The introduction of rocking angles for the SO_2 and CF_2 groups improves the fit of the experimental intensities considerably. The R factor for the long camera distance data decreased by *c.* 40%. Vibrational amplitudes for closely spaced distances were refined in groups and further constraints are evident from Table 1. With these assumptions, nine geometric parameters and nine vibrational amplitudes (a) were refined simultaneously. Only two correlation coefficients had values larger than $|0.6|$: $C-F/a_1=0.73$ and $a_4/a_6=0.68$. The results are summarized in Table 1.

A comparison of the ring geometries of 1,3,5-trithiane, $(CH_2S)_3$, with those of **1** demonstrates that the C−S bonds in the former ring [1.812 (4) Å] [18] are considerably shorter than those in the latter [1.866 (4) Å]. The same trend in the C−S bond lengths is observed between the open-chain compounds $(CH_3)_2S$ [C−S = 1.807 (2) Å] [19] and $(CF_3)_2SO_2$ [C−S = 1.858 (5) Å] [20]. This lengthening can be rationalized as a superposition of two opposing effects: (i) shortening due to an increase in the oxidation state from S^{II} to S^{VI} [e.g. C−S = 1.807 (2) Å in $(CH_3)_2S$ versus 1.771 (4) Å in $(CH_3)_2SO_2$] [21] and (ii) lengthening due to fluorination at the carbon atoms [22] [e.g. C−S = 1.771 (4) Å in $(CH_3)_2SO_2$ versus 1.858 (5) Å in $(CF_3)_2SO_2$]. This latter effect (ii) overrides the former (i). The CSC bond angles in **1** and 1,3,5-trithiane are equal within their experimental uncertainties [100.2 (7)° versus 99.1 (4)°] and the SCS angles increase slightly in the S^{VI} ring [117.4 (6)° versus 115.8 (1)°]. This leads to a somewhat less puckered ring structure in **1** [torsional angle around C−S bonds $\Phi=62.1$ (14)°] compared to ($\Phi=65.4°$) in 1,3,5-trithiane. Both C_3S_3 rings, however,

TABLE 1. Results of an electron diffraction study of **1**[a]

Geometric parameters

C–F	1.338 (4)	∠OSO	125.9 (14)
S=O	1.418 (2)	$\rho(CF_2)$[b]	4.1 (12)
C–S	1.866 (4)	$\rho(SO_2)$[b]	−0.9 (7)
∠CSC	100.2 (7)	Φ(S–C)[c]	62.1 (14)[f]
∠SCS	117.4 (6)	Θ(S)[d]	51.9 (23)[f]
∠FCF	109.2 (7)	Θ(C)[e]	60.8 (23)[f]

Interatomic distances and vibrational amplitudes

C−F	1.34	0.040[g]	O(1′)−F(2′)	3.73	0.103 (18) (a_7)
S=O	1.42	0.042 (2) (a_1)	S(1)−F(1′)	3.81	0.115[g]
C−S	1.87	0.051 (3) (a_2)	C(1)−O(1′)	3.96	
F(1)−F(1′)	2.18	0.060[g]	S(1)−C(1)	3.55	0.090[g]
O(1)−O(1′)	2.53		C(1)−F(2)	4.07	0.098 (10) (a_g)
S(1)−F(2′)	2.57	0.083 (4) (a_3)	S(1)−O(2)	4.34	
S(1)−F(2)	2.63		F(1)−F(2′)	4.18	0.160[g]
C(1)−O(2)	2.64		O(1)−O(2′)	4.72	
C(1)−O(2′)	2.66		(O1′)−F(1′)	4.62	0.130[g]
F(1)−F(2′)	2.47	0.260[g]	O(1)−F(1′)	4.63	
O(1)−O(2′)	3.02		O(1′)−F(1)	5.06	
C(1)−C(2)	2.86	0.083 (8) (a_4)	C(1)−O(1)	4.70	0.130 (33) (a_9)
S(1)−S(2)	3.19		S(1)−F(1)	4.83	
O(1)−F(2′)	2.90	0.143 (2) (a_5)	F(1)−F(2)	5.15	0.240[g]
O(1)−F(2′)	3.03		O(1)−O(2)	5.28	
O(1′)−F(2)	3.17		O(1)−F(1)	6.03	0.140[g]
C(1)−F(2′)	2.98	0.135 (15) (a_6)			
S(1)−O(2′)	3.41				

[a] r_a distances in Å and $\angle_\alpha$ angles in degree. Error limits are 3σ values and include a possible scale error of 0.1% for bonded distances. For atom numbering see Fig. 2.

[b] Rocking angle, axial fluorines are pushed together and axial oxygens are pushed apart.

[c] Torsional angle around S−C bonds.

[d] Puckering angle C(1)−S(3)−C(2)/C(2)−S(1)−S(2)−C(1).

[e] Puckering angle S(1)−C(3)−S(2)/S(2)−C(1)−C(2)−S(1).

[f] Dependent parameter.

[g] Not refined.

are more strongly puckered than in cyclohexane [$\Phi = 54.9(9)°$] [23].

Experimental

Materials

The following compounds used in this work were obtained from commercial sources: F_2 (Solvay Deutschland GmbH); NaF (Riedel-de Haën). $\mathrm{K\overline{CHSO_2CH_2SO_2CH_2S}O_2}$ was prepared using the literature method [5].

General procedures

Gases were manipulated in a conventional Pyrex glass vacuum apparatus equipped with a Leybold–Heraeus thermocouple gauge. Infrared spectra were obtained by using a 10-cm Pyrex gas cell with KBr windows on a Nicolet 55DX-FT spectrometer. NMR spectra were recorded with an AC 80 Bruker spectrometer operating at 80.0 MHz for proton and 75.4 MHz for fluorine resonances or on a WH 360 Bruker spectrometer operating at 90.5 MHz for carbon-13 resonances; TMS, $CFCl_3$ and $CDCl_3$ were used as internal standards. Mass spectra were recorded with either a Varian MAT CH5 or a Finnigan MAT 8230 system operating at 70 eV.

Elemental analyses were determined by Beller Microanalytical Laboratory, Göttingen, Germany.

Electron diffraction

The scattering intensities of **1** were recorded using a Gasdiffractograph KD-G2 [24] at two camera (nozzle-to-plate) distances. The electron wavelength (accelerating voltage *c.* 60 kV) was determined with ZnO powder patterns. The sample reservoir was heated to 45 °C and the inlet system and nozzle to 55 °C. The camera pressure during the experiment was $< 2 \times 10^{-5}$ mbar. Two plates for each camera distance were analyzed by the usual procedures [25]. Averaged molecular intensities in the s ranges 2–18 $Å^{-1}$ and 8–35 $Å^{-1}$ in steps of $\Delta s = 0.2$ $Å^{-1}$ are presented in Fig. 2.

Fluorination of $\mathrm{K\overline{CHSO_2CH_2SO_2CH_2S}O_2}$

Run 1

To 10.1 g of $\mathrm{K\overline{CHSO_2CH_2SO_2CH_2S}O_2}$ (37.2 mmol) and 30.0 g of NaF (715 mmol), which were well mixed and placed in a 1-l stainless-steel cylinder, was added 11.4 g of F_2 (300 mmol) at 20 °C keeping the total pressure of F_2 and volatile products between 200 and 500 Torr. The rate of F_2 addition was *c.* 1 g d^{-1}. The volatile products were removed from the cylinder yielding: $(F_2CSO_2)_3$ (**1**) (trace), $(FSO_2CF_2)_2SO_2$ (**2**) (2.0 g, 8.8 mmol), $FSO_2CF_2SO_2CF_3$ (**4**) (0.4 g, 1.5 mmol), $FSO_2CF_2SO_2F$ (**5**) (0.2 g, 0.9 mmol), and COF_2, CF_4, CHF_3 and SO_2F_2 (all identified via their characteristic IR spectra).

Run 2

To 12.3 g of $\mathrm{K\overline{CHSO_2CH_2SO_2CH_2S}O_2}$ (45.2 mmol) and 24.9 g of NaF (593 mmol), was added 12.9 g of F_2 (340 mmol) at 0 °C keeping the total pressure between 150 and 450 Torr. The rate of F_2 addition was *c.* 0.7 g d^{-1}. The distribution of volatile products was $(F_2CSO_2)_3$ (**1**) (2.8 g, 8.2 mmol), $SO_2(CF_2SO_2F)_2$ (**2**) (3.0 g, 9.1 mmol), $CF_3SO_2CF_2SO_2F$ (**4**) (0.3 g, 1.1 mmol), $CF_2(SO_2F)_2$ (**5**) (trace) and $CF_2(SO_2CF_3)_2$ (**3**) (trace).

Run 3

Under conditions similar to Run 2, another derivative, $CF_3SO_2CF_2SO_2CF_2SO_2F$ (**6**) (0.19 g, 0.50 mmol) was found. In Run 2, separation was obtained via distillation. In Run 3, separation was achieved using a HP5 890 series II GC system (methyl silicone stationary phase).

$(CF_2SO_2)_3$ (**1**): M.p. 135 °C. IR (cm^{-1}): 1446 (st); 1425 (m); 1223 (m); 1206 (sst); 1182 (s); 1150 (s); 1124 (s); 907 (s); 889 (s); 708 (st); 567 (st); 529 (m). ^{19}F NMR: $\phi_{CF_2} = -105.5$ ppm (s) ($^1J_{13C\text{-}F} = 346.0$ Hz). ^{13}C NMR: $\delta_{CF_2} = +118.9$ ppm (t, quint) ($^1J_{13C\text{-}F} = 345.8$ Hz, $^3J_{13C\text{-}F} = 4.15$ Hz). MS (Cl^-, NH_3) *m/z*: 361 $(MF)^-$; 323 $(M-F)^-$; 292 $(M-CF_2)^-$; 228 $(M-CF_2SO_2)^-$; 211 $(C_3F_5S_2O)^-$; 195 $(C_3F_5S_2)^-$; 164 $(CF_2SO_2CF_2)^-$; 128 $(MH-C_2F_5SO_4)^-$; 114 $(CF_2SO_2)^-$; 98 $(CF_2SO)^-$; 64 $(SO_2)^-$. MS (CI, isobutane) *m/z*: 361 $(MF)^+$; 343 $(MH)^+$; 285 $(M-3F)^+$; 279 $(MH-SO_2)^+$; 267 $(MH-4F)^+$; 250 $(M-OF_4)^+$; 239 $(MH-OCF_4)^+$; 231 $(M-OF_5)^+$; 223 $(MH-O_2CF_4)^+$; 199 $(MF-CF_2SO_5)^+$; 185 $(MH-O_2CF_6)^+$. Analysis: Calc. for $C_3F_6S_3O_6$: C, 10.53; S, 28.11%. Found: C, 10.53; S, 26.77%.

$SO_2(CF_2SO_2F)_2$ (**2**): B.p. 87.5 °C/142 mbar.

IR (cm^{-1}): 1479 (st); 1434 (m); 1245 (st); 1213 (m); 1153 (m); 1121 (m); 908 (w); 829 (m); 810 (m); 664 (w); 589 (m); 567 (m); 537 (m). ^{19}F NMR: $\phi_{CF_2} = -93.5$ ppm (m); $\phi_{SO_2F} = +51.7$ (m) ($^4J_{^{13}CF_2\text{-}CF_2} = 11.1$ Hz, $^3J_{^{13}CF_2\text{-}SF} = 6.6$ Hz). ^{13}C NMR: $\delta_{CF_2} = 119.7$ ppm ($^1J_{FA} = 347.0$ Hz, $^1J_{FB} = = 349.0$ Hz, $^2J_{^{13}C\text{-}SF} = 38.3$ Hz, $^3J_{^{13}C\text{-}F_2} = 2.1$ Hz). MS (CI^-, NH_3) *m/z*: 349 $(MF)^-$; 235 $(M-5F)^-$; 197 $(M-CF_2SO_2F)^-$; 183 $(CF_2SCF_2SF)^-$; 164 $(M-2SO_2F)^-$; 147 $(MH-CF_2SO_4F_2)^-$; 133 $(CF_2SO_2F)^-$; 127 $(FSO_2CS)^-$; 101 $(CF_2SF)^-$; 83 $(SO_2F)^-$; 79 $(FSOC)^-$; 67 $(SOF)^-$; 64 $(SO_2)^-$. MS $(EI)^+$ *m/z*: 31 $(CF)^+$; 32 $(S)^+$; 48 $(SO)^+$; 50 $(CF_2)^+$; 63 $(FSC)^+$; 64 $(SO_2)^+$; 67 $(SOF)^+$; 69 $(CF_3)^+$; 82 $(CF_2S)^+$; 83 $(FSO_2)^+$; 98 $(CF_2SO)^+$; 101 $(CF_2SF)^+$; 117 $(CF_2SOF)^+$; 133 $(CF_2SO_2F)^+$. Analysis: Calc. for $C_2F_6S_3O_6$: C, 7.27; F, 34.5; S, 29.13%. Found: C, 7.53; F, 36.2; S, 28.72%.

$F_2C(SO_2CF_3)_2$ (**3**): ^{19}F NMR: $\phi_{CF_2} = -95.5$ ppm (sept); $\phi_{CF_3} = -70.2$ ppm (t) ($^4J_{FF} = 8.13$ Hz). $F_3CSO_2CF_2SO_2F$ (**4**): ^{19}F NMR: $\phi_{CF_3} = -69.8$ ppm (d, t); $\phi_{CF_2} = -96.4$ ppm (q,d); $\phi_{SO_2F} = 50.8$ ppm (t,q) ($J_{CF_3\text{–}CF_2} = 7.95$ Hz, $J_{CF_3\text{–}SO_2F} = 1.57$ Hz, $J_{CF_2\text{–}SO_2F} = 6.11$ Hz).

$CF_2(SO_2F)_2$ (**5**): M.p. -86 °C, b.p. 57.2 °C [26]. ^{19}F NMR: $\phi_{CF_2} = -97.2$ ppm (t); $\phi_{SO_2F} = +47.8$ ppm (t) ($^3J_{FF} = 6.45$ Hz, $^1J_{CF} = 334.4$ Hz). ^{13}C NMR: $\delta = 116.5$ ppm (t,t) ($^1J = 334.2$ Hz, $^2J = 40.6$ Hz). MS (Cl^-, NH_3) *m/z*: 133 $(CF_2SO_2F)^+$; 83 $(SO_2F)^+$: 67 $(SOF)^+$; 64 $(SO_2)^+$; 51 $(SF)^+$; 50 $(CF_2)^+$; 48 $(SO)^+$.

$^1CF_3SO_2{}^2CF_2SO_2{}^3CF_2SO_2F$ (**6**): ^{19}F NMR: $\phi_{CF_3} = -69.9$ ppm (t); $\phi_{^2CF_2} = -92.7$ ppm (mult); $\phi_{^3CF_2} = -93.7$ ppm (mult); $\phi_{SO_2F} = 51.4$ ppm ($^4J_{CF_3\text{–}CF_2} = 7.8$ Hz, $^3J_{FSO_2\text{–}CF_2} = 6.5$ Hz, $^5J_{FSO_2CF_2} = 1.2$ Hz). ^{13}C NMR: $\delta_{1C} = 119.1$ ppm (q,t); $\delta_{2C} = 121.4$ ppm (t,q,d); $\delta_{3C} = 119.6$ ppm (t,d) ($^1J_{1C} = 332.1$ Hz, $^1J_{2C} = 354.2$ Hz, $^1J_{3C} = 346.0$ Hz, $^3J_{1C} = 0.6$ Hz, $^3J_{2CF_3} = 3.15$ Hz, $^2J_{3C} = 37.75$ Hz, $^4J_{2C} = 2.2$ Hz). MS (CI) *m/z*: 381 $(MH)^+$; 297 $(M - SO_2F)^+$; 183 $(CF_3SO_2CF_2)^+$; 133 $(CF_2SO_2F, CF_3SO_2)^+$; 69 $(CF_3)^+$, 64 $(SO_2)^+$.

Acknowledgement

We express our appreciation to the Fulbright Commission and thank Solvay Deutschland GmbH (Hannover, Germany) for the gift of fluorine. We are grateful for financial support by the Fonds der Chemischen Industrie.

References

1 A. Waterfeld and R. Mews, *J. Fluorine Chem., 23* (1984) 475.

2 G.L. Gard, A. Waterfeld, R. Mews, J. Mohtasham and R. Winter, *Inorg. Chem., 29* (1990) 4588.

3 A. Jin, H.G. Mark, A. Waterfeld and H. Oberhammer, *J. Am. Chem. Soc., 113* (1991) 7847.

4 G.L. Gard, A. Waterfeld, N.N. Hamel, R. Mews and J. Mohtasham, *J. Fluorine Chem., 55* (1991) 313.

5 R. Camps, *Ber. Dtsch. Chem. Ges., 25* (1892) 223.

6 R. Seelinger and W. Sundermeyer, *Angew. Chem., Int. Ed. Engl., 19* (1980) 203.

7 A.F. Eleev, G.A. Soko'skii and I.L. Knunyants, *Izv. Akad. Nauk SSSR, Ser. Khim.,* (1977) 1573.

8 G.A. Olah, P.S. Iyer and P. Surya, *Synthesis,* (1982) 512.

9 C. Bunyagdj, H. Plotrowka and M.H. Aldridge, *J. Chem. Eng. Data, 26* (1981) 344.

10 R.J. Willenbring, R. Winter, J. Mohtasham and G.L. Gard, *Can. J. Chem., 67* (1989) 2037.

11 L.F. Chen, J. Mohtasham and G.L. Gard, *J. Fluorine Chem., 49* (1990) 331.

12 V. Castro, J.L. Boyer, J.P. Canselier, R.J. Terjeson, J. Mohtasham, D.H. Peyton and G.L. Gard, *Magn. Reson. Chem., 28* (1990) 998.

13 S. Temple, *J. Org. Chem., 33* (1986) 344.

14 L.F. Chen, J. Mohtasham and G.L. Gard, *J. Fluorine Chem., 48* (1990) 107.

15 Y. Katsuhara, R.M. Hammaker and D.D. DesMarteau, *Inorg. Chem., 19* (1980) 607.

16 J.K. Brown and K.J. Morgan, *Adv. Fluorine Chem., 4* (1965) 256.

17 J. Haase, *Z. Naturforsch., 25* (1970) 936.

18 K. Bencze, A. Kucsman, G. Schultz and I. Hargittai, *Acta Chem. Scand., 43* (1989) 953.

19 T. Iijima, S. Tsuchiya and M. Kimura, *Bull. Chem. Soc. Jpn., 50* (1977) 2564.

20 H. Oberhammer, G.D. Knerr and J.M. Shreeve, *J. Mol. Struct., 82* (1982) 143.

21 M. Hargittai and J. Hargittai, *J. Mol. Struct., 20* (1974) 283.

22 H. Oberhammer, *J. Fluorine Chem., 23* (1983) 147.

23 O. Bastianson, L. Fernholt, H.M. Seip, H. Kambera and K. Kuchitsu, *J. Mol. Struct., 18* (1973) 163.

24 H. Oberhammer, *Molecular Structure by Diffraction Methods,* The Chemical Society, London, 1976, Vol. 4, p. 24.

25 H. Oberhammer, H. Willner and W. Gombler, *J. Mol. Struct., 70* (1981) 273.

26 E.J.M. O'Sullivan, F.W. Klink, C.C. Liu and E.B. Yeager, *J. Electrochem. Soc., 132* (1985) 2424.