Synthesis and studies of nucleophilic reactions of vicinal bis(fluorosulfonyloxy)perfluoroisononanes

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The addition of peroxydisulfuryl difluoride $(FSO_3)_2$ to isomeric perfluoroisononenes (perfluoropropylene trimers) was studied. Isomers containing the trisubstituted C=C bond form adducts which were converted to the corresponding ketofluorosulfates under the action of weak nucleophiles (DMF and AcONa/AcOH) and underwent haloform-type decomposition under the action of CsF to form perfluoroalkenyl fluorosulfates and products of their subsequent conversions.

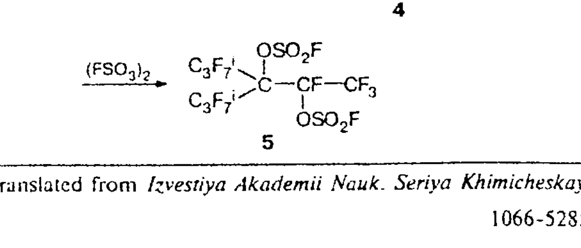
Key words: fluoroaliphatic compounds, fluorosulfates, stable radical.

It is known that the stability of free radicals is primarily determined by steric shielding of the radical center. Spectacular evidence of this statement is provided by the successful synthesis of perfluoroethyldiisopropylmethyl radicals by fluorination of perfluoropropylene trimers (**1** and **2**).[1]

One could expect that, like fluorination, radical fluorosulfation of higher polysubstituted fluoroolefins would afford α-fluorosulfonyloxyperfluoroalkyl radicals possessing enhanced stability owing to their branched structures. Therefore, we have studied the reactions of peroxydisulfuryl difluoride $(FSO_3)_2$ (PSF) with perfluoropropylene trimers (**1**—**3**).

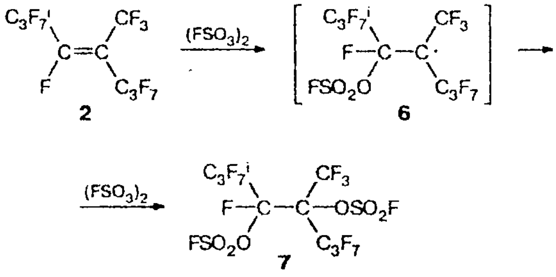
Previously,[2] we have demonstrated that the reaction of PSF with olefin **1** (molar ratio **1** : PSF = 1.5 : 1) at 20—25 °C afforded a stable radical (**4**) in high yield. This radical can readily be isolated from the reaction mixture in the individual form. The yield of the adduct (**5**) was no more than 12%. In this work, we demonstrated that radical **4** was also formed upon electrolysis of HSO_3F in the presence of **1** at 25—30 °C in a rather high yield (48.3%) with respect to the starting compound. When the reaction was carried out at higher temperature (40—45 °C), the percentage of bis(fluorosulfate) **5** increased substantially (Scheme 1).

Scheme 1



We failed to detect the fluorosulfonyloxyperfluoroalkyl radical (**6**) in the reaction of olefin **2** with PSF. The only product of both the chemical and electrochemical reactions (the latter proceeded at an appreciable rate only at 40—45 °C) was bis(fluorosulfate) **7** (Scheme 2) regardless of the reagent ratio.

Scheme 2



In our opinion, the results of this work (primarily, generation of stable radical **4** only in the case of olefin **1**) indicate that steric factors play a decisive role in the addition of PSF to polysubstituted fluoroolefins. Actually, structure **1** is most favorable for the formation of a stable radical species under conditions of free-radical addition of PSF. The fluorosulfonyloxy radical adds readily to **1** on the side of the CF_3 group to form radical **4** in which the α-fluorosulfonyloxyperfluoroethyl and two perfluoroisopropyl groups shield the radical center and create considerable steric hindrances to the addition of the second FSO_3 group. As a result, this addition proceeds at a substantially lower rate compared to the initiating stage of the reaction, which is a prerequisite for the successful synthesis of radical **4**.

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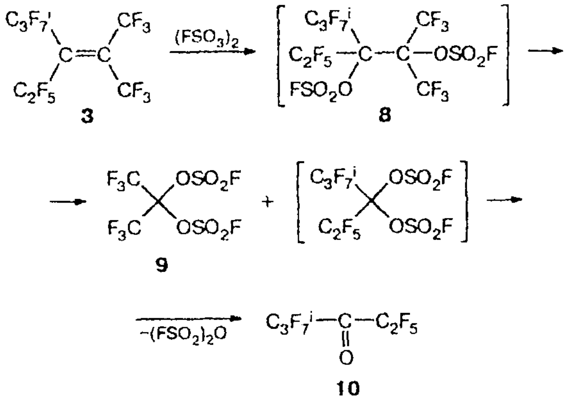
Olefin **2** also belongs to trisubstituted fluoroolefins. However, unlike compound **1**, olefin **2** contains the bulky perfluoroisopropyl group at the monosubstituted vinylic carbon atom, which precludes sterically the addition of the fluorosulfonyloxy radical. As a result, the rate of addition of $FSO_3^{\cdot}$ at the multiple bond of olefin **2** decreases sharply and the rate of formation of radical **6** is lower than the rate of its recombination with the radical $FSO_3^{\cdot}$.

Tetrasubstituted olefin **3** reacted with PSF (Scheme 3) only at 75—80 °C and was slowly (35—40 h) converted to 2,2-bis(fluorosulfonyloxy)perfluoropropane (**9**) and perfluoro(ethyl isopropyl ketone) (**10**). Apparently, compounds **9** and **10** resulted from homolysis of intermediate adduct **8** whose steric strain is, evidently, determined by the presence of two vicinal fluorosulfonyloxy groups at the tertiary carbon atoms.

Scheme 3

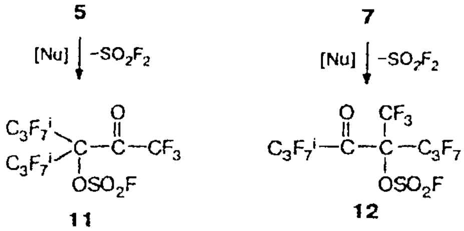


For comparison, it should be noted that, unlike the reaction of olefin **3** with PSF, its reaction with F_2 proceeded rather smoothly at 20 °C to form perfluoroisononane.[3] In our opinion, the difference in the reactivity of **3** with respect to PSF and F_2 is also determined by steric hindrances, but these are inherent in the addend rather than in the olefin molecule. The importance of the difference in the bulkiness of the FSO_3 group and the fluorine atom is evident from the products of fluorosulfatodimerization of perfluoropropylene[4] and fluorodimerization of perfluoroallyl fluorosulfate.[5] In the former case, only one product, *viz.*, 2,3-bis(trifluoromethyl)-1,4-bis(fluorosulfonyloxy)perfluorobutane, was formed, which is indicative of the regiospecific addition of the $FSO_3^{\cdot}$ radical to perfluoropropylene. In the latter case, three fluorodimerization products of the "head-to-head," "head-to-tail," and "tail-to-tail" types were obtained, which indicates that the fluorine atom adds not only to the terminal but also to the central carbon atom of perfluoroallyl fluorosulfate.

An attempt to perform electrochemical fluorosulfation of olefin **3** resulted in a mixture of 4-hydro-3-fluorosulfonyloxyperfluoro-2,4-dimethyl-3-ethylpentane and 3-hydro-2-fluorosulfonyloxyperfluoro-2,4-dimethyl-3-ethylpentane. These compounds are the anodic reduction products of intermediate radicals that were formed upon addition of the fluorosulfonyloxy radical to compound **3** at the vinylic carbon atom.[6]

In this work, we demonstrated that the synthesized vicinal bis(fluorosulfates) (**5** and **7**) very readily react with nucleophilic reagents. Even under the action of a weak nucleophile such as DMF, compounds **5** and **7** underwent selective decomposition with the participation of the fluorosulfate groups exclusively at the secondary carbon atoms to form the corresponding α-fluorosulfonyloxy ketones **11** and **12** (Scheme 4).

Scheme 4



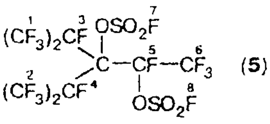
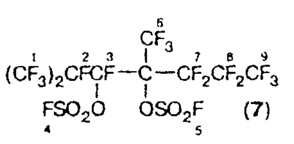
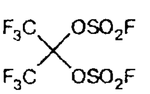
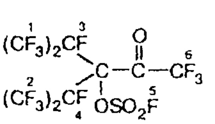
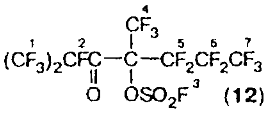
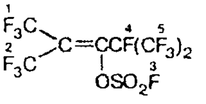
Nu = DMF, MeOH, diglyme, acetone, or AcONa

This reaction can be suggested as a preparative method for the synthesis of fluorosulfonyloxy ketone **12** but it cannot be used for the synthesis of compound **11**, which undergoes further decomposition under the action of DMF to give secondary products. The reaction of bis(fluorosulfate) **5** with AcONa in AcOH is a more convenient procedure for the synthesis of ketone **11**, which allows one to prepare fluorosulfonyloxy ketone **11** in a yield of higher than 70%.

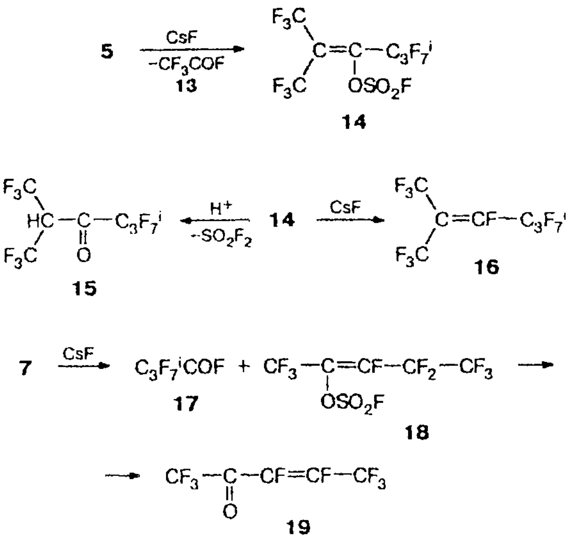
The formation of fluorosulfonyloxy ketones **11** and **12** from bis(fluorosulfates) **5** and **7** was also observed in the presence of other weak nucleophiles, such as diglyme, methanol, and acetone. However, these reactions proceeded at substantially lower rates than that in the presence of DMF (in the case of bis(fluorosulfate) **7**, this difference is particularly noticeable) and cannot be considered as preparative procedures for the synthesis of the above-mentioned fluorosulfonyloxy ketones.

The reactions of bis(fluorosulfates) **5** and **7** with alkali metal fluorides are more complicated processes. In these reactions, fluorosulfonyloxy ketones **11** and **12** were also formed initially (Table 1). The presence of these compounds in the reaction mixture was detected by GLC and ^{19}F NMR spectroscopy. However, in both cases we failed to terminate the reactions at this stage. The intermediate fluorosulfonyloxy ketones underwent haloform-type decomposition under the action of F^- to give the corresponding perfluoroacyl fluorides (**13** and **17**) and vinyl fluorosulfates (**14** and **18**). Their subsequent conversions depend substantially on their structures (Scheme 5).

Table 1. ^{19}F NMR spectra of the synthesized compounds

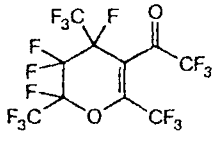
Compound	δ
 (5)	−130.0 (2 F-7, F-8); −12.6 (6 F-1); −9.5 (6 F-2); −3.2 (3 F-6); group of signals from 30 to 45 (1 F-5); group of signals from 80 to 98 (2 F-3, F-4)
 (7)	−127.5 and −130.0 (1 F-4, F-5); −14.5 (3 F-6); −4.5 (6 F-1); 5.8 (3 F-9); 30.0 (2 F-7); 40.0 (2 F-8); 48.1 (1 F-3); 98.5 (1 F-2)
 (9)	−1.5 (6 F); −128.4 (2 F)
 (11)	−129.2 (1 F-5); −8.2 (12 F-1, F-2); −5.9 (3 F-6); 87.6 (2 F-3, F-4)
 (12)	−130.0 (1 F-3); −15.5 (3 F-4); −4.4 (6 F-1); 4.6 (3 F-7); 33.3 (2 F-5); 40.6 (2 F-6); 110.0 (1 F-2)
 (14)	−125.0 (1 F-3); −20.1 and −16.0 (3 F-1 + 3 F-2); −4.3 (6 F-5); 96.6 (1 F-4)

Scheme 5



Perfluoro-2,4-dimethylpent-2-en-3-yl fluorosulfate (**14**), which was formed upon decomposition of bisfluorosulfate **5**, is sufficiently stable and can be isolated in 70—75% yield with 90—92% purity. In the subsequent reaction of vinyl fluorosulfate **14** with CsF, the cleavage of the S—O bond to form ketone **15**, which is typical of fluoroaliphatic fluorosulfates, as well as the replacement of the FSO_3 group by the fluorine atom to give olefin **16**, take place. Apparently, the fact that vinyl fluorosulfate **14** behaves not only as a fluorosulfonating agent but also as a vinylating agent with respect to the fluoride ion is due largely to the presence of three perfluoralkyl substituents at the multiple bond, whose combined electron-withdrawing effect is responsible for the enhanced electrophilic character of the olefin. This allows the vinylic carbon atom to successfully compete with the fluorosulfonyloxy group in the reaction with a strong nucleophile such as F^-.

Decomposition of bis(fluorosulfate) **7** under the action of CsF at 50—90 °C (50 Torr) afforded perfluoroisobutyryl fluoride (**17**) and vinyl fluorosulfate **18**. Under the reaction conditions, the latter underwent further decomposition to form perfluoropent-3-en-2-one (**19**). The formation of compounds **18** and **19** was detected by removing the products from the reaction mixture in the course of the reaction. This reaction under atmospheric pressure at 120 °C afforded a compound for which the acyloxene structure (**20**) was suggested based on the data of elemental analysis and mass spectrometry (the complex character of the ^{19}F NMR spectrum precluded unambiguous establishment of its structure). The formation of this product can be represented as a result of cyclodimerization of ketone **19** in the presence of F^-, which makes it possible to draw the analogy of this reaction with cyclodimerization of perfluorocrotonyl fluoride.[7]



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Experimental

The ^{19}F NMR spectra were recorded on a Bruker-WP200 SY instrument (188.3 MHz, CF_3COOH as the external standard). The IR spectra were obtained on a UR-20 spectrometer. The ESR spectra were measured on a Varian-E12A spectrometer. The mass spectra were obtained on a VGMS-70E spectrometer. The energy of ionizing electrons was 70 eV.

Electrochemical fluorosulfation of olefins 1—3 (general procedure). *A.* A solution of $NaSO_3F$ (3 g) in HSO_3F (100 mL) and a mixture of trimers **1**—**3** (100 g; **1** : **2** : **3** = 35 : 3 : 62 (GLC)) were placed in a diaphragmless glass cell (an SU-2000 glassy-carbon anode and a 2Kh13 steel cathode) cooled with water. Electrolysis was carried out with stirring at 20—25 °C for 3 h with a current of 2 A. The reaction mixture was poured onto ice. The organic layer was separated and dried with $MgSO_4$. Distillation gave the initial trimers in a yield of 68 g in a ratio **1** : **2** : **3** = 2 : 1 : 18, perfluoro-3-isopropyl-4-methyl-2-fluorosulfonyloxypentan-3-yl radical **4** in a yield of 16.5 g (48.3% with respect to the consumed compound **1**), b.p. 53—56 °C (11 Torr) (literature data:[2] b.p. 54—56 °C (12 Torr); the ESR spectrum of radical **4** is identical to that reported in the literature[2]), and 2,3-bis(fluorosulfonyloxy)perfluoro-3-isopropyl-4-methylpentane (**5**) in a yield of 1 g (2.5% with respect to the consumed compound **1**), b.p. 82—83 °C (5 Torr), m.p. 73—75 °C. Found (%): C, 19.49; F, 65.34; S, 6.2. $C_9F_{20}S_2O_6$. Calculated (%): C, 19.67; F, 65.75; S, 5.83. IR, ν/cm^{-1}: 1490, 1510 (SO_3F).

B. Electrolysis was performed under conditions of experiment *A* at 40—45 °C for 8 h. Radical **4** and bis(fluorosulfate) **5** were isolated in yields of 3.2 g (7.5%) and 41.4 g (80%), respectively. Compound **5** contained 0.5% of 3,4-bis(fluorosulfonyloxy)perfluoro-2,4-dimethylheptane (**7**) as an admixture. The trimers were also isolated in a ratio **1** : **2** : **3** = 1 : 1 : 60 in a yield of 60 g.

C. Electrolysis of olefin **2** (45 g) was carried out under conditions of experiment *A* at 40—45 °C for 14 h. Bis(flurosulfate) **7** was obtained in a yield of 53.1 g (81%), b.p. 90—91 °C (20 Torr). Found (%): C, 16.84; F, 58.75; S, 9.90. $C_9F_{20}S_2O_6$. Calculated (%): C, 16.66; F, 58.64; S, 9.70.

Reaction of radical 4 with $(FSO_3)_2$. $(FSO_3)_2$ (1.5 g, 7.5 mmol) was added to radical **4** (5.5 g, 10 mmol) at 0 °C. The reaction mixture was kept at 25 °C for 5 days and then distilled. Bis(fluorosulfate) **5** was obtained in a yield of 6.0 g (92%), b.p. 82—83 °C (5 Torr).

Reactions of olefins 2 and 3 with $(FSO_3)_2$. *A.* $(FSO_3)_2$ (4.5 g, 22 mmol) was added to olefin **2** (9.0 g, 20 mmol) at 0 °C. The reaction mixture was kept at −20 °C for 6 days and distilled. Bis(fluorosulfate) **7** was isolated in a yield of 9.5 g (73%).

B. A mixture of olefin **3** (9.0 g, 20 mmol) and $(FSO_3)_2$ (6.0 g, 30 mmol) was heated in a sealed glass tube at 80 °C for 8 days. Perfluoro(ethyl isopropyl ketone) (**10**) was obtained in a yield of 3.8 g (61%) (identified by GLC by comparing with a specimen prepared according to a known procedure[8]). 2,2-Bis(fluorosulfonyloxy)perfluoropropane (**9**) was obtained in a yield of 3.4 g (49%), b.p. 105—106 °C (decomposed upon storage). MS of **9**, *m*/*z* ($I_{\rm rel}$ (%)): 279 $[M - CF_3]^+$ (1); 249 $[M - SO_3F]^+$ (21); 47 $[C_3F_5O]^+$ (9); 119 $[C_2F_5]^+$ (3); 97 $[CF_3CO]^+$ 48; 83 $[SO_2F]^+$ 84; 69 $[CF_3]^+$ (100). IR, ν/cm^{-1}: 1500 (SO_3F).

Reaction of bis(fluorosulfate) 5 with CH_3COONa/CH_3COOH. A mixture of bis(fluorosulfate) **5** (50 g, 76 mmol), CH_3COONa (2.5 g, 30 mmol), and CH_3COOH (20 mL) was stirred at 25 °C for 4 h. The reaction mixture was poured into water. The organic layer was separated, washed with water, and dried with $MgSO_4$. Distillation gave 3-fluorosulfonyloxyperfluoro-3-isopropyl-4-methylpentan-2-one (**11**) in a yield of 40 g (94%), b.p. 91—95 °C (13 Torr). Found (%): C, 19.56; S, 6.23. $C_9F_{18}SO_4$. Calculated (%): C, 19.72; S, 5.86. IR, ν/cm^{-1}: 1490 (SO_3F); 1770 (C=O). MS, *m*/*z* ($I_{\rm rel}$ (%)): 527 $[M - F]^+$ (0.5); 49 $[M - CF_3CO]^+$ (1); 397 $[C_8F_{15}O]^+$ (1.2); 359 $[C_8F_{13}O]^+$ (1); 309 $[C_7F_{11}O]^+$ (6); 281 $[C_6F_{11}]^+$ (1); 259 $[C_6F_9O]^+$ (3); 243 $[C_6F_9]^+$ (1); 197 $[C_4F_7O]^+$ (10); 169 $[C_3F_7]^+$ (9); 119 $[C_2F_5]^+$ (2); 97 $[C_2F_3O]^+$ (27); 83 $[SO_3F]^+$ (11); 69 $[CF_3]^+$ (100); 64 $[SO_2]^+$ (3).

Reaction of bis(fluorosulfate) 7 with DMF. DMF (1 mL) was added with cooling to bis(fluorosulfate) **7** (2 g, 3 mmol). After 2 days, the mixture was poured into water. The organic layer was separated and dried with $MgSO_4$. Distillation gave 4-fluorosulfonyloxyperfluoro-2,4-dimethylheptan-3-one (**12**) in a yield of 1.2 g (74%), b.p. 143 °C. Found (%): C, 19.89; S, 5.88. $C_9F_{18}SO_4$. Calculated (%): C, 19.72; S, 5.86. IR, ν/cm^{-1}: 1490 (SO_3F); 1775 (C=O). MS, *m*/*z* ($I_{\rm rel}$ (%)): 425 $[C_9F_{12}O_2]^+$ (2); 349 $[C_5F_{11}O_3S]^+$ (7); 247 $[C_5F_9O]^+$ (8); 197 $[C_4F_7]^+$ (100); 169 $[C_3F_7]^+$ (60); 97 $[C_2F_3O]^+$ (12); 83 $[SO_2F]^+$ (15); 69 $[CF_3]^+$ (80).

Reaction of bis(fluorosulfate) 5 with CsF. A mixture of bis(fluorosulfate) **5** (12.5 g, 18.8 mmol) and freshly calcined CsF (1 g, 6.6 mmol) was stirred and heated to 100 °C *in vacuo* (50 Torr). Volatile reaction products were collected in a trap (−78 °C). After 4 h, vacuum was reduced to 3 Torr. Fractionation of the distillate obtained (9.7 g) gave a mixture of products in a yield of 0.5 g, b.p. 50—54 °C (70 Torr), which contained perfluoro-2,4-dimethylpent-2-ene (**16**) (identified by comparing with a specimen prepared according to a known procedure[9]) and 3-fluorosulfonyloxyperfluoro-2,4-dimethylpent-2-ene (**14**) in a ratio of 3 : 1 (GLC). The second fraction (3.2 g) with a b.p. of 55—56 °C (70 Torr) contained fluorosulfate **14** (90—92%, GLC), and the third fraction (6.0 g) with a b.p. of 90—95 °C (3 Torr) contained fluorosulfonyloxy ketone (**12**) and bis(fluorosulfate) **5** in a ratio of 1 : 4 (GLC). The Raman spectrum of compound **14**, ν/cm^{-1}: 1483 (SO_3F); 1646 (C=C). MS of compound **14**, *m*/*z* ($I_{\rm rel}$ (%)): 411 $[M - F]^+$ (11); 309 $[C_6F_9O]^+$ (22); 197 $[C_4F_7O]^+$ (18); 178 $[C_3F_6CO]^+$ 32; 169 $[C_3F_7]^+$ (18); 159 $[C_3F_5CO]^+$ (97); 150 $[C_3F_6]^+$ (36); 83 $[SO_2F]^+$ (99); 69 $[CF_3]^+$ (100); 67 $[SOF]^+$ (55). The yield of compound **14** with respect to the consumed bis(fluorosulfate) **5** was 74.4%.

Reaction of vinyl fluorosulfate 14 with CsF. A mixture of compound **14** (0.9 g, 2 mmol) and freshly calcined CsF (0.3 g, 2 mmol) was stirred at 100 °C with simultaneous distillation of the products obtained. A mixture was obtained in a yield of 0.3 g, which contained (according to the data of GLC-mass spectrometry) olefin **16** and 2-hydro-2,4-bis(trifluoromethyl)heptafluoropentan-3-one (**15**) in a ratio of 1 : 2. MS of compound **15**, *m*/*z* ($I_{\rm rel}$ (%)): 197 $[C_4F_7O]^+$ (20); 179 $[C_4F_6HO]^+$ (48); 169 $[C_3F_7]^+$ (9); 113 $[C_3F_4H]^+$ (5); 91 $[C_3F_2HO]^+$ (27); 69 $[CF_3]^+$ (100). IR, ν/cm^{-1}: 1770 (C=O).

Reaction of bis(fluorosulfate) 7 with CsF. *A.* A mixture of bis(fluorosulfate) **7** (10 g, 15 mmol) and freshly calcined CsF (1 g, 6.6 mmol) was heated with stirring from 40 to 90 °C *in vacuo* (50 Torr). After 1.5 h, a mixture (8.3 g) was collected in a trap. Distillation of the mixture gave a fraction (3.7 g) with a b.p. of 52—100 °C, which contained perfluoropenten-3-en-2-one (**19**) (identified by ^{19}F NMR spectroscopy and by GLC by comparing with a specimen prepared according to a procedure reported previously[10]) and 2-fluorosulfonyloxyperfluoropent-2-ene (**18**) in a ratio of 1 : 2.5. Distillation also afforded flurosulfonyloxy ketone **12** (3.5 g), b.p. 130—145 °C, and perfluoroisobutyryl fluoride (**17**) (1.2 g). Compound **17** was converted to ethyl perfluorisobutyrate under the action of ethanol and identified by comparing with the known sample (^{19}F NMR and GLC).[11] MS of compound **18**, *m*/*z* ($I_{\rm rel}$ (%)): 330 $[M]^+$ (15); 311 $[M - F]^+$ (2); 261 $[M - CF_3]^+$ (10); 211 $[M - C_2F_5]^+$ (32); 169 $[C_3F_7]^+$ (100); 97 $[CF_3CO]^+$ (33); 83 $[SO_2F]^+$ (10); 69 $[CF_3]^+$ (90).

B. A mixture of bis(fluorsulfate) **7** (10.5 g, 16 mmol) and freshly calcined CsF (2.3 g, 15 mmol) was stirred for 1 h. The temperature was increased from 40 to 120 °C and the reaction products that distilled off were collected in a trap, while the gaseous reaction products that evolved were absorbed with ethanol. Distillation gave a compound in a yield of 3.6 g (49%), b.p. 110 °C, for which the structure of perfluoro-2,4,6-trimethyl-3-trifluoroacetyl-2-oxene (**20**) was suggested based on the data of elemental analysis and mass spectrometry. Found (%): C, 26.36; F, 66.82. $C_{10}F_{16}O_2$. Calculated (%): C, 26.31; F, 66.66. MS, *m*/*z* ($I_{\rm rel}$ (%)): 437 $[M - F]^+$ (3); 387 $[M - CF_3]^+$ (2); 359 $[M - CF_3CO]^+$ (38); 299 $[C_8F_9O_2]^+$ (10); 271 $[C_7F_9O]^+$ (12); 97 $[CF_3CO]^+$ (28); 69 $[CF_3]^+$ (100). The ethanolic solution was poured into water. Ethyl perfluoroisobutyrate was isolated in a yield of 3.7 g (92%) (identified by comparing (^{19}F NMR and GLC) with a sample prepared according to a procedure reported previously[11]).

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