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A novel and efficient method for the synthesis of polyfluoroarenesulfonyl bromides from polyfluoroarenethiols

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Dedicated to Prof. Dr. H. C. Hermann-Josef Frohn on the occasion of his 65th birthday.

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ABSTRACT

The first general methodology has been developed for the synthesis of polyfluoroarenesulfonyl bromides from polyfluoroarenethiols. At heating of polyfluoroarenethiols with a mixture of Br_2 and fuming HNO_3 or Br_2, HNO_3 and H_2SO_4 polyfluoroarenesulfonyl bromides were obtained in good yields.

1. Introduction

Among the chemical properties of polyfluoroaromatic compounds, the reactions of polyfluoroarenes with electrophilic reagents, which consist in interaction of the latter with a functional group of polyfluoroarenes, are of interest. These reactions can transform simple readily installed substituents in perfluoroaromatic ring to more complicated and otherwise inaccessible functional group. Some examples of these reactions are considered in [1,2]. In this connection it seemed reasonable to study the transformation of polyfluoroarenethiols under the action of electrophilic reagents into practically inaccessible sulfonyl bromide derivatives of polyfluoroarenes.

Previously, pentafluorobenzenesulfonyl bromide (**1**) was described as the only representative of polyfluoroaromatic sulfonyl bromides. For the synthesis of compound **1**, the reaction of pentafluorophenylmagnesium chloride with sulfur dioxide and bromine was performed; this also produces a significant quantity of bis(pentafluorophenyl)sulfone. The chemistry of pentafluorobenzenesulfonyl bromide obtained was poor [3].

Polyfluoroarenesulfonyl bromides could be of considerable interest as sources of polyfluoroarenesulfonyl radicals (cf. [4]). In this connection polyfluoroarenesulfonyl bromides could be used for introduction of polyfluoroarenesulfonyl group in olefins.

It has been shown that N-substituted polyfluorobenzenesulfonamides inhibited the growth of cancer cells in vitro [5].

Therefore, the relative reactivities of polyfluoroarenesulfonyl chlorides and bromides with amines could be of interest for polyfluoroarenesulfonamide synthesis. It is known that polyfluoroarenesulfonyl chlorides are used for preparation of the corresponding sulfonamides [6].

In order to expand knowledge of the properties of polyfluoroarenesulfonyl halides it seems interesting to study the reactivities of Ar_fSO_2X (X = F, Cl, Br) to a more complete extent for a broader understanding of the synthetic aspects of these compounds.

In the present paper, the results of investigation of the reactions of polyfluoroarenethiols with Br_2 and fuming HNO_3 or other bromine-containing oxidative systems are considered, the objective being to determine the possibility of forming polyfluoroarenesulfonyl bromides.

2. Results and discussion

We have found that the action of mixture of bromine and fuming nitric acid on pentafluorobenzenethiol (**2**) affords pentafluorobenzenesulfonyl bromide **1** in good yield (Scheme 1).

The formation of compound **1** can also take place in the reactions of thiol **2** with Br_2, HBr, NaBr and acids (HNO_3, H_2SO_4,

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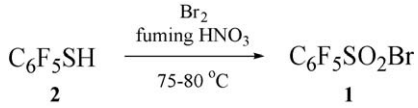
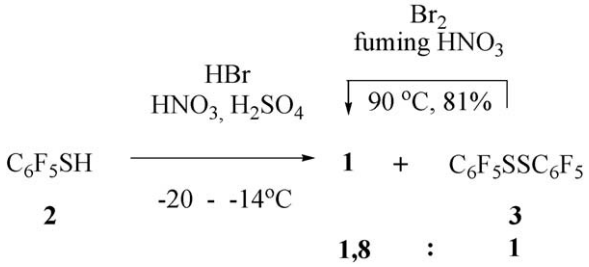
Table 1
Methods of synthesis of compound 1.

Method	A^1	A^2	B	C	D	E	F	G	H
Reagents	Fuming $HNO_3 + Br_2$	$HNO_3 + Br_2$	$HNO_3 + H_2SO_4 + Br_2$	$HNO_3 + H_2SO_4 + HBr$	$Br_2 + H_2SO_4$	$Br_2 + CH_3COOH$	$Br_2 + H_2O$	NaBr + fuming HNO_3	NaBr + HNO_3 + H_2SO_4
Temp. (°C)	80	80	80	75–80	80–90	75–80	80–90	85–95	75–85
Yield of **1**	87	47	84	83	[a]	[a]	44[a]	76	67

[a] The reaction mixtures obtained by methods D, E and F contained **1** alongside bis(pentafluorophenyl)disulfide **3** in the ratio (**1**:**3**, according to ^{19}F NMR): ~10:1 (method D), ~8:1 (method E), 8.3:1 (method F).



Scheme 1.



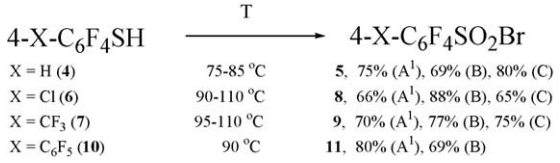
Scheme 2.

CH_3COOH) or with Br_2 and H_2O. The results are presented in Table 1.

These data permit us to conclude that the best result was obtained by the reaction of compound **2** with the Br_2 and fuming nitric acid (d 1.52) (method A^1). In the reactions of thiol **2** with mixtures of concentrated nitric (d 1.36) and sulfuric (d 1.83) acids and Br_2 (method B), or concentrated nitric, sulfuric and hydrobromic (d 1.49) acids (method C) or suspension of sodium bromide in fuming nitric acid (method G), the compound **1** was obtained in good yields. The other methods mentioned in Table 1 resulted in difficultly separable mixtures of sulfonyl bromide **1** and disulfide **3**.

When thiol **2** reacted with HBr, HNO_3 and H_2SO_4 at −20 °C, disulfide **3** was obtained alongside sulfonyl bromide **1**. Disulfide **3** was converted to sulfonyl bromide **1** (81% yield) when heated with a mixture of Br_2 and fuming HNO_3 (Scheme 2).

The methods A^1, B and C previously mentioned as affording good results were used for the synthesis of new polyfluoroarenesulfonyl bromides. Thus, 2,3,5,6-tetrafluorobenzenethiol (**4**) was converted to 2,3,5,6-tetrafluorobenzenesulfonyl bromide (**5**) in 80% yield (method C). The transformation of 4-chloro-2,3,5,6-tetrafluorobenzenethiol (**6**) and 4-trifluoromethyl-2,3,5,6-tetrafluorobenzenethiol (**7**) to the corresponding sulfonyl bromides (**8**) and (**9**) proceeded at higher temperatures. The best yields of (**8**) and (**9**) were obtained by method B (Scheme 3).

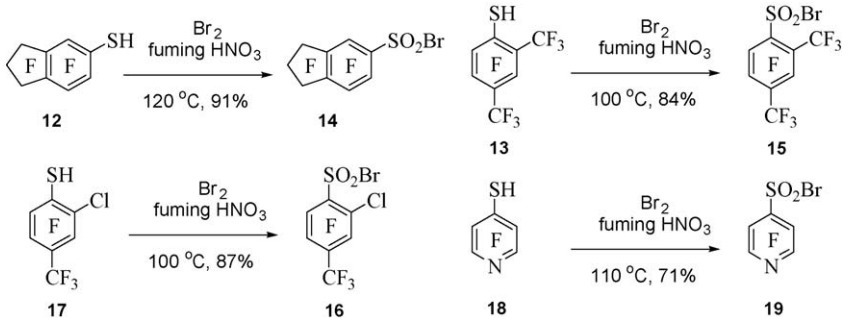


Scheme 3.

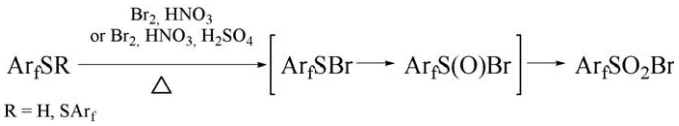
It has been shown that the use of method C resulted in the synthesis of polyfluoroarenesulfonyl bromides containing strong electron-acceptor group in the ring from the corresponding polyfluoroarenethiols in significantly low yields. For example, nonafluoro-5-indanethiol **12** and 2,4-bis(trifluoromethyl)-3,5,6-trifluorobenzenethiol **13** were converted to nonafluoro-5-indanesulfonyl bromide **14** and 2,4-bis(trifluoromethyl)-3,5,6-trifluorobenzenesulfonyl bromide **15** to a little extent. At the same time methods A^1 and B were used for the preparation of sulfonyl bromide **14** in 91% yield and 69% yield respectively. Method A^1 was the most suitable one for the synthesis of 2,4-bis(trifluoromethyl)-3,5,6-trifluorobenzenesulfonyl bromide (**15**) from thiol (**13**) and 2-chloro-4-trifluoromethyl-3,5,6-trifluorobenzenesulfonyl bromide (**16**) from thiol (**17**). An important application of the method A^1 is the transformation of 2,3,5,6-tetrafluoropyridinethiol (**18**) into the corresponding sulfonyl bromide (**19**) (Scheme 4).

Electrophilic brominating agents [7] and nitronium cation [8] take probably part in the formation of polyfluoroarenesulfonyl bromides. The intermediates Ar_fSBr [9] could be formed from polyfluoroarenethiols under action of electrophilic species. It is possible that formation of polyfluorinated diaryl disulfides proceeds with participation of Ar_fSBr (cf. [10]). Conversion of Ar_fSR (R = Br, SAr_f) under the action of, for example, nitronium cation or Br_2 and ONO_2^- to Ar_fSO_2Br could include intermediate formation of $Ar_fS(O)Br$. The proposed route of the formation of polyfluoroarenesulfonyl bromides from polyfluoroarenethiols and the corresponding disulfides could be illustrated in the following scheme (Scheme 5).

This way is in agreement with the data of oxidative chlorination of polyfluoroarenethiols into polyfluoroarenesulfonyl chlorides [6]



Scheme 4.



Scheme 5.

Table 2
Molar ratios of Ar_fSX (X = H, SAr_f, 1.0 mol) to electrophilic reagents.

Compound	Method	Br_2	HNO_3	H_2SO_4	Product	Yield (%)
3	A[1]	8.0	9.7		**1**	81
4	C	2.2[a]	4.2	9.8	**5**	80
6	B	2.7	5.2	11.2	**8**	88
7	B	2.5	4.6	8.5	**9**	77
10	A[1]	5.3	9.9		**11**	80
12	A[1]	2.7	7.9		**14**	91
13	A[1]	3.4	8.0		**15**	84
17	A[1]	2.4	5.8		**16**	87
18	A[1]	4.0	11.8		**19**	71

[a] Molar ratio of HBr.

and the conversion of sulfenyl chloride groups containing in polyfluorinated aliphatic polymers and alkane derivative to the sulfonyl chlorides by reaction in CFC-113 with Cl_2 in trifluoroacetic acid and water at about 100 °C or with aqueous sodium hypochlorite. Treatment of sulfenyl chloride derivatives of the polymers with HOF·acetonitrile reagent afforded mixtures of the corresponding sulfonyl chloride and sulfonyl fluoride derivatives [11].

However, many details of the mechanism of the formation of polyfluoroarenesulfonyl bromides including also the question of participation of Ar_fSO_2H in the formation of Ar_fSO_2Br still remain to be clarified.

3. Conclusion

Investigations of the reactions of polyfluoroarenethiols with mixture of Br_2 and fuming HNO_3, Br_2, HNO_3 and H_2SO_4 along with other electrophilic brominating agents expand the knowledge of transformation of substituent groups of polyfluoroarenes under the action of electrophilic reagents. And, although the questions of the mechanism of these reactions are quite complex, we can hope that a further investigation of processes of this type will promote a solution of these problems to a more complete extent. The appearance of available polyfluoroarenesulfonyl bromides as a result of the investigations carried out opens up the possibilities in the investigation of the chemistry of these compounds in different reactions.

4. Experimental

^{19}F and ^{1}H NMR spectra were recorded on a Bruker AV-300 instrument at 282 and 300 MHz respectively for solutions in CCl_4. Chemical shifts are given in δ (ppm); the internal standards were C_6F_6 (−162.9 ppm from CCl_3F) and HMS (0.04 ppm from TMS). The ^{19}F and ^{1}H chemical shifts are reported vs. CCl_3F and TMS. Coupling constants (J) are given in Hz. IR spectra were measured on a Bruker Vector 22 IR spectrophotometer. UV spectra were recorded on a Hewlett Packard 8453 UV spectrophotometer for solutions in hexane. High resolution mass spectra were recorded on the Finnigan MAT 8200 (EI mode, 70 eV). Polyfluoroarenethiols and disulfide **3** were synthesized according to [12,13].

General synthetic procedure: polyfluoroarenethiol (5–10 mmol) was added dropwise to a stirred mixture of Br_2 and fuming HNO_3 (d 1.52) (method A[1]), Br_2, HNO_3 (d 1.36) and H_2SO_4 (d 1.83) (method B) or HBr (d 1.49), HNO_3 and H_2SO_4 (method C) (Table 2). The temperature rose to ~56 °C (methods A[1] and B), 30–35 °C (method C) as a result of the exothermic reactions which ensued. Vigorous evaluation of gas of brown color (probably nitrogen dioxide) ensued with changing of color of mixture. After complete addition of polyfluoroarenethiol, the resulting reaction mixture was heated for several hours (2–5 h) and then allowed to cool to room temperature. The mixture was poured into CH_2Cl_2 (25–50 mL), the organic layer was washed with 10% aqueous solution of Na_2CO_3 (2× 25 mL) and dried over $CaCl_2$. The solvent was distilled off to give pure polyfluoroarenesulfonyl bromide.

4.1. Pentafluorobenzenesulfonyl bromide (1)

Compound **1** was obtained following the general synthetic procedure and using also other methods. Molar ratio (temperature, time of reaction): **2**:Br_2:HNO_3 (d 1.52) = 1.0:2.7:4.8 (80 °C, 2 h, 87% yield, method A[1]); **2**:Br_2:HNO_3 (d 1.36) = 1.0:2.8:4.6 (80 °C, 2 h, 47% yield, method A[2]); **2**:Br_2:HNO_3:H_2SO_4 = 1.0:3.2:5.3:10.4 (80 °C, 2 h, 84% yield, method B); **2**:HBr:HNO_3:H_2SO_4 = 1.0:2.0:4.1:8.1 (80 °C, 2 h, 83% yield, method C); **2**:Br_2:H_2SO_4 (d 1.83) = 1.0:3.7:8.0 (80–90 °C, 6 h, method D); **2**:Br_2:CH_3COOH = 1.0:4.1:16.4 (75–80 °C, 2 h, method E); **2**:Br_2:H_2O = 1.0:4.6:8.4 (80–90 °C, 2 h, method F); **2**:NaBr:HNO_3 = 1.0:2.9:8.5 (85–95 °C, 2 h, 76% yield, method G); **2**:NaBr:HNO_3:H_2SO_4 = 1.0:2.1:4.2:8.3 (75–85 °C, 2 h, 67% yield, method H). Following the general synthetic procedure the methods D, E and F afforded reaction mixtures which contained **1** alongside disulfide **3** in the ratio (**1**:**3** according to ^{19}F NMR): ~10:1 (method D), ~8:1 (method E), 8.3:1 (44% yield of 1, method F). When NaBr is used (methods G and H) the reaction mixture is poured into CH_2Cl_2 and the solid residue is washed with CH_2Cl_2 (2× 5 mL). The CH_2Cl_2 solutions are combined and treated as described above.

Light-yellow oil. UV λ_{max}, nm (lg ε): 219 (3.95), 278 (3.36). IR (neat): ν 1644, 1503, 1389, 1306, 1269, 1175, 1103, 1025, 997, 728, 645, 592, 558, 538, 484 cm^{-1}. ^{19}F NMR: δ −135.6 (m, $2F^{2,6}$), −140.5 (tt, $1F^4$, $J_{F^4-F^{3.5}}$ = 21, $J_{F^4-F^{2.6}}$ = 9), −157.7 (m, $2F^{3,5}$) [3]. Calcd for $C_6BrF_5O_2S$: 309.8723, found 309.8713. Anal. Calcd for $C_6BrF_5O_2S$: C 23.2; F 30.5; Br 25.7; S 10.3. Found: C 23.1; F 30.4; Br 25.5; S 10.3.

4.2. 2,3,5,6-Tetrafluorobenzenesulfonyl bromide (5)

Mp 51–52 °C, white crystals. UV λ_{max}, nm (lg ε): 215 (4.04), 291 (3.43). IR (KBr): ν 3075, 1504, 1386, 1374, 1249, 1189, 1159, 939, 877, 715, 589, 555, 501, 488 cm^{-1}. ^{1}H NMR: δ 7.4 (tt, $J_{H-F^{3.5}}$ = 9.5, $J_{H-F^{2.6}}$ = 7.0). ^{19}F NMR: δ −134.7 (m, $2F^{3,5}$), −136.6 (m, $2F^{2,6}$). Calcd for $C_6HBrF_4O_2S$: 291.8817, found 291.8824. Anal. Calcd for $C_6HBrF_4O_2S$: C 24.6; H 0.3; Br 27.3; F 25.9; S 10.9. Found: C 24.6; H 0.4; Br 27.2; F 25.7; S 10.8.

4.3. 4-Chloro-2,3,5,6-tetrafluorobenzenesulfonyl bromide (8)

Mp 41–42 °C, white crystals. UV λ_{max}, nm (lg ε): 229 (4.12), 287 (3.44). IR (KBr): ν 1631, 1493, 1459, 1395, 1373, 1270, 1170, 983, 963, 627, 567, 537 cm^{-1}. ^{19}F NMR: δ −136.2 (m, 2F), −136.7 (m, 2F). Calcd for $C_6BrClF_4O_2S$: 325.8428. Found 325.8427. Anal. Calcd for $C_6BrClF_4O_2S$: C 22.0; Cl 10.8; F 23.2; S 9.8. Found: C 22.3; Cl 10.9; F 23.6; S 9.6.

4.4. 4-Trifluoromethyl-2,3,5,6-tetrafluorobenzenesulfonyl bromide (9)

Mp 51–52 °C, white crystals. UV λ_{max}, nm (lg ε): 218 (4.05), 298 (3.45). IR (KBr): ν 1504, 1385, 1326, 1169, 996, 945, 717, 670, 578, 556, 525 cm^{-1}. ^{19}F NMR: δ −57.5 (t, $3F^{CF3}$, $J_{CF3-F^{3.5}}$ = 22), −134.1 (m, $2F^{2,6}$), −135.6 (m, $2F^{3,5}$). Calcd for $C_7BrF_7O_2S$: 359.8691. Found 359.8698. Anal. Calcd for $C_7BrF_7O_2S$: C 23.3; Br 22.1; F 36.8; S 8.9. Found: C 23.3; Br 22.0; F 36.6; S 9.0.

4.5. Nonafluorobiphenyl-4-sulfonyl bromide (11)

Mp 61–62 °C. UV λ_{max}, nm (lg ε): 203 (4.36), 252 (4.15). IR (KBr): ν 1530, 1510, 1477, 1391, 1272, 1172, 1133, 1003, 975, 726, 618, 563, 539 cm^{-1}. ^{19}F NMR: δ −133.6 (m, $2F^{3,5}$), −135.6 (m, $2F^{2,6}$), −137.1 (m, $2F^{2',6'}$), −148.6 (tt, $1F^{4'}$, $J_{F^{4'}-F^{3',5'}}$ = 21, $J_{F^{4'}-F^{2',6'}}$ = 4), −160.4 (m, $2F^{3',5'}$). Calcd for $C_{12}BrF_9O_2S$: 457.8659, found 457.8669. Anal. Calcd for $C_{12}BrF_9O_2S$: C 31.4; Br 17.4; F 37.2; S 7.0. Found: C 31.5; Br 17.5; F 37.2; S 6.7.

4.6. Nonafluoroindane-5-sulfonyl bromide (14)

Mp 40.5–42.5 °C, white crystals. UV λ_{max}, nm (lg ε): 211 (4.17), 286 (3.49). IR (KBr): ν 1501, 1395, 1328, 1311, 1250, 1207, 1177, 1091, 957, 914, 675, 586, 535 cm^{-1}. ^{19}F NMR: δ −107.6 (m, $2F^1$ or $2F^3$), −109.0 (m, $2F^3$ or $2F^1$), −110.0 (dt, $1F^4$, $J_{F^4-F^7}$ = 21, $J_{F^4-F^3}$ = 8), −116.3 (d, $1F^6$, $J_{F^6-F^7}$ = 21), −130.5 (quintet, $2F^2$, $J_{F^2-F^1}$ = $J_{F^2-F^3}$ = 3), −135.9 (tt, $1F^7$, $J_{F^7-F^1}$ = 8). Calcd for $C_9BrF_9O_2S$: 421.8656. Found 421.8659. Anal. Calcd for $C_9BrF_9O_2S$: C 25.6; Br 18.9; F 40.4; S 7.6. Found: C 25.6; Br 18.9; F 40.8; S 7.5.

4.7. 2,4-Bis(trifluoromethyl)-3,5,6-trifluorobenzenesulfonyl bromide (15)

Yellow oil. UV λ_{max}, nm (lg ε): 307 (3.91). IR (neat): ν 1604, 1489, 1441, 1395, 1363, 1329, 1229, 1172, 952, 878, 738, 678, 655, 581, 561, 526 cm^{-1}. ^{19}F NMR: δ −50.9 (d, 3F, 2-CF_3, J_{CF3-F^3} = 38), −57.8 (dd, 3F, 4-CF_3, J_{CF3-F^5} = 25, J_{CF3-F^3} = 22), −108.6 (m, $1F^3$), −119.3 (m, $1F^5$), −125.6 (dd, $1F^6$, $J_{F^5-F^6}$ = 22, $J_{F^3-F^6}$ = 12.5). Calcd for $C_8BrF_9O_2S$: 409.8655. Found 409.8653.

4.8. 2-Chloro-4-trifluoromethyl-3,5,6-trifluorobenzenesulfonyl bromide (16)

Yellow oil. UV λ_{max}, nm (lg ε): 221 (3.96), 307 (3.61). IR (neat): ν 1471, 1390, 1336, 1312, 1244, 1165, 1090, 954, 883, 689, 670, 574, 557, 521 cm^{-1}. ^{19}F NMR: δ −57.8 (t, 3F, CF_3, J_{CF3-F^3} = J_{CF3-F^5} = 22), −109.9 (qd, $1F^3$, $J_{F^3-F^6}$ = 13), −130.3 (quintet, $1F^5$, $J_{F^5-F^6}$ = 21), −131.3 (dd, $1F^6$). Calcd for $C_7BrClF_6O_2S$: 375.8390. Found 375.8393. Anal. Calcd for $C_7BrClF_6O_2S$: C 22.3; Br 21.2; F 30.2; S 8.5. Found: C 22.5; Br 21.4; F 30.4; S 8.2.

4.9. 2,3,5,6-Tetrafluoropyridine-4-sulfonyl bromide (19)

Light-yellow oil. UV λ_{max}, nm (lg ε): 203 (4.04), 298 (3.37). IR (neat): ν 1483, 1392, 1265, 1246, 1169, 968, 611, 564, 503, 434 cm^{-1}. ^{19}F NMR: δ −84.3 (m, $2F^{2,6}$), −137.0 (m, $2F^{3,5}$). Calcd for $C_5BrF_4NO_2S$: 292.8770. Found 292.8762. Anal. Calcd for $C_5BrF_4NO_2S$: C 20.4; Br 27.2; F 25.9; N 4.8; S 10.9. Found: C 20.7; Br 27.2; F 25.7; N 5.0; S 10.9.

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