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Interaction of 2,4,6-tris(fluorosulfonyl)chlorobenzene with O-, N-, S-, C-nucleophiles and F-anion

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ABSTRACT

Reactions of 2,4,6-tris(fluorosulfonyl)chlorobenzene **1** with O-, N-, S-, C-nucleophiles and F-anion showed high reactivity of **1** that was defined by three strong electron withdrawing SO_2F groups creating several electrophilic centers within the molecule. Conditions for selective chlorine atom substitution were defined that resulted in formation of corresponding ethers, amines and sulfides, while excess of nucleophile commonly led to SO_2F groups implication in the reaction. Two equivalents of fluoride-anion source gave rise not only to the chlorine-fluorine substitution but afforded in the anionic σ-complex formation with two fluorine atoms in the *hem*-position. Reduction of chlorobenzene **1** with zinc/AcOH was found to be a choice for 1,3,5-tris(fluorosulfonyl)benzene preparation.

1. Introduction

In the frame of our continuous interest to aromatic systems modified by fluorine containing electron withdrawing substituents we would like to attract attention to the accessible, stable and intriguing system of symmetric tris(fluorosulfonyl)benzenes [1,2] as a flat platform for three general pathway designs of various molecules (Picture 1):

(1) through nucleophilic attack of SO_2F function (potentially biologically active sulfamides, netlike polymers, etc.);
(2) by mild nucleophilic displacement of activated aromatic halogen (based on easy obtainable 2,4,6-tris(fluorosulfonyl)-benzene chloride **1**) with fluorosulfonyl fragments preservation;
(3) via combined processes including step by step procedures (heterocyclizations as well as polymerization).

Simple and effective preparative synthetic procedures for the syntheses of 2,4,6-tris(fluorosulfonyl)chlorobenzene (**1**), corresponding phenol **2** and aniline **3** have been elaborated and presented in our previous publications [1,2] that made these compounds available for thorough investigations.

Above mentioned molecules correspond to the structural analogs of 2,4,6-tris(trifluoromethylsulfonyl)benzene derivatives which were previously studied by our group in detail [3–5], but fluorosulfonyl derivatives are much more accessible. Noteworthy to mention that electronic properties of the SO_2F group (σ_p 1.01) are similar to SO_2CF_3 (σ_p 1.04–1.06 [6,7]). pK_a values of above mentioned 2,4,6-three substituted phenols in acetonitrile are close to each other: phenol **2** – 5.53 and 2,4,6-$(SO_2CF_3)_3$phenol – 4.79 that outdo the acidity of aromatic sulfonic acids [8,9]. ΔG_{acid} values of anilines in gaseous phase are: aniline **3** – 307.5 and 2,4,6-$(SO_2CF_3)_3$ aniline – 304.8 kcal/mol [10]. These facts predict high mobility of chlorine atom in chlorobenzene **1**.

On the other hand, SO_2F groups can also react with nucleophilic agents. The last circumstance make it possible to realize a consecutive introduction various fragments into the molecule of **1**.

As to reactivity of SO_2F group it is known that fluorosulfonylbenzenes react with bases specifically. Appreciable hydrolysis occurs when benzene ring is activated with nitro group or with halogen atom [11]. Reaction rates of *p*-substituted fluorosulfonylbenzenes with benzylamines were in 1000 times [12] and with aniline in five degree less [13] than the rate of chlorosulfonylbenzenes reactions.

Some comparison of fluorosulfonyl aromatic moiety and activated aromatic halogen atom reactivity towards various nucleophilic agents [2,14–16] did not give exact predictions about 2,4,6-tris(fluorosulfonyl)chlorobenzene **1** regioselective behavior in the basic conditions. The later has different electrophilic centers and its reactions with nucleophiles may cause to unexpected but important results. Previously investigated by us chemical reactivity of 1,3,5-tris(fluorosulfonyl)benzene gave no chance to generalize unambiguously outcome of nucleophilic attack towards this molecule that was strongly dependent on the nucleophile type, its

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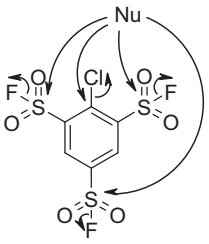


Picture 1. Various possible reaction centers of compound **1** towards nucleophile attack.

quantity and reaction conditions [17–19]. Thus, in order to perform consecutive introduction of various fragments into chlorobenzene **1** it is capitally crucial to ascertain the possibility of selective nucleophilic substitution in this compound.

Formulated synthetic potential of above mentioned tris(fluorosulfonyl)benzenes family stimulated us to perform the thorough investigation of 2,4,6-tris(fluorosulfonyl)chlorobenzene (**1**) chemical behavior towards the set of O-, N-, S-, C-nucleophiles in order to realize the scope and limitations of reaction pathways and products formed.

2. Results and discussion

General remark to investigation performed concerns the importance of ^{19}F NMR monitoring of reaction progress and products formation that showed the significant difference (~7 ppm) in the position between *ortho*- and *para*-SO_2F groups in the coarse of various 1-position substitution, or exact change of integral intensity and positioning of SO_2F groups in case of the attack towards fluorosulfonyl centers.

2.1. Reactions with O-nucleophiles

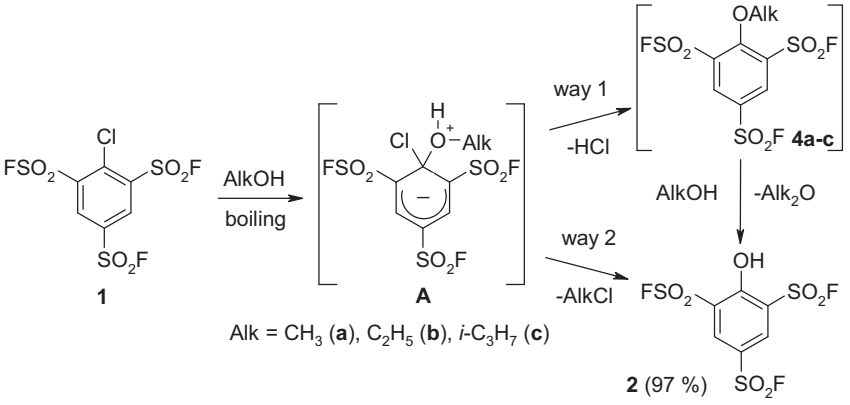
As we showed earlier [2], boiling of chlorobenzene **1** alcoholic solution gave phenol **2** (Scheme 1). The reaction presumably proceeded via σ-complex **A** formation followed by destruction that may occur in two different ways (Scheme 1) both leading to the formation of phenol **2**.

GLC investigation of the reaction solution (Alk = C_2H_5) showed that the second coproduct was diethylether that confirm the reaction pathway 1 and gave rise to the idea to investigate compounds **4** as alkylating agents due to the fact that they may be considered as alkyl esters of the strong acid **2**.

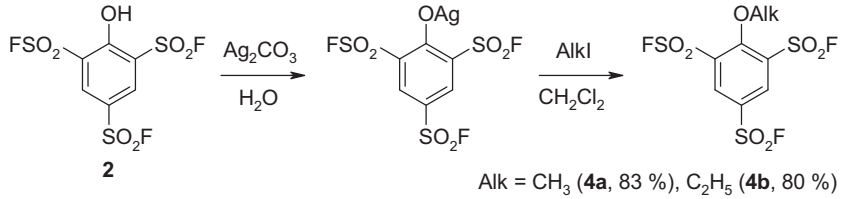
Alkyl ethers **4a** and **4b** were obtained in individual form by phenol **2** silver salt alkylation with alkyl iodides in dichloromethane (Scheme 2) in high yields.

It was found that the only dissolving of compound **4b** in ethanol resulted in diethyl ether (GLC data) and phenol **2** quantitative formation. Anisole **4a** reacts analogously.

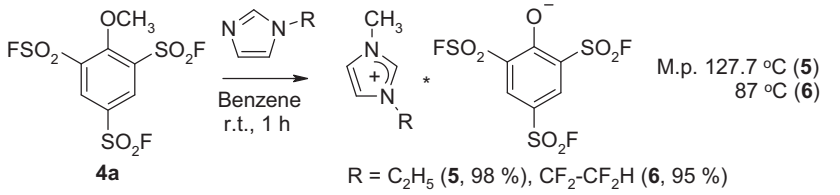
Compound **4a** easily alkylates nitrogen containing heterocycles. Thus, products of anisole **4a** reactions with *N*-alkylimidazoles in 1 h were corresponding salts – *N*-methyl-*N*′-alkylimidazolium 2,4,6-tris(fluorosulfonyl)phenolates **5** and **6** (Scheme 3).



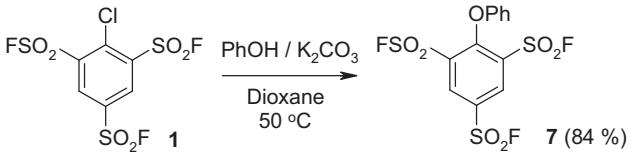
Scheme 1. Reactions of **1** with alcohols.



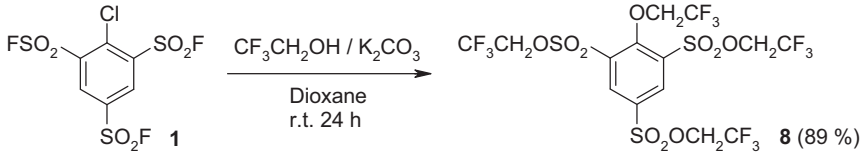
Scheme 2. Syntheses of anisole **4a** and phenetole **4b**.



Scheme 3. *N*-alkylation of imidazoles with anisole **4a**.



Scheme 4. Reaction of **1** with potassium phenolate.



Scheme 5. Reaction of **1** with trifluoroethanol excess.

Salts **5** and **6** have rather low melting points and show high stability on handling within half a year period in DMSO solution as well as in crystalline state.

Noteworthy to outline that as it was described earlier [20], *N*-(tetrafluoroethyl)imidasole was alkylated by methyl iodide within long-term boiling in acetonitrile. Thus, one can suppose anisole **4a** to be stronger methylating agent in comparison with CH_3I.

In contrast to reactions of chlorobenzene **1** with alcohols, its interaction with alkoxides and phenolate using alcohols or phenol in polar solvents (AlkOH, CH_3CN) in the presence of equimolar quantity of base even at negative temperature showed complete lost of selectivity and mixture of products, mainly due to the attack of SO_2F groups, were observed. That may be considered as a critical difference between chlorobenzene **1** and 2,4,6-tris(trifluoromethylsulfonyl)chlorobenzene – the later reacts mildly with alkoholates forming products of chlorine atom substitution in high yields [3,5].

Selective reaction of chlorobenzene **1** with phenol succeeded when the less polar solvent (dioxane) was used and K_2CO_3 was utilized as a base (Scheme 4).

Complete nucleophilic substitution on the all reactive centers was achieved with excess of trifluoroethylate that resulted in the product of chlorine and fluorine atoms substitution **8** (Scheme 5). Stability of compound **8** is due to low alkylating ability of fluoroalkyl esters of arenesulfonic acids [21]. Furthermore, 2,4,6-tris(trifluoroetoxysulfonyl)phenol [22] is somewhat weaker acid than phenol **2** (pK_a values 7.97 and 5.53, respectively [9]).

In general it must be noted that chlorine atom in 2,4,6-tris(fluorosulfonyl)chlorobenzene (**1**) easily hydrolyses in moist polar solvents with phenol **2** formation, while SO_2F groups remained unchanged.

2.2. Reactions with N-nucleophiles

Chlorobenzene **1** reacted with nitrogen containing nucleophiles smoothly but not as easy as in case of 2,4,6-tris(trifluoromethylsulfonyl)chlorobenzene. The later with excess of aqueous ammonia in acetonitrile gave 2,4,6-tris(trifluoromethylsulfonyl)aniline in quantitative yield [3]. In the same conditions chlorobenzene **1** formed the set of water-soluble products. ^{19}F NMR spectrum of reaction solution showed only the presence of fluoride-anion signal and no evidence of SO_2F groups, that may be rationalized as hydrolysis of SO_2F groups in aqueous ammonia resulting in the formation of fluorosulfonyl function ammonolysis products.

2,4,6-Tris(fluorosulfonyl)aniline **3** was obtained in high yield using ammonia solution in dry acetonitrile. In these conditions even an excess of ammonia gave no by-products (Scheme 6).

This conditions for aniline **3** synthesis are more convenient and productive than the method described by us earlier [2] starting with 2,4,6-tris(chlorosulfonyl)aniline.

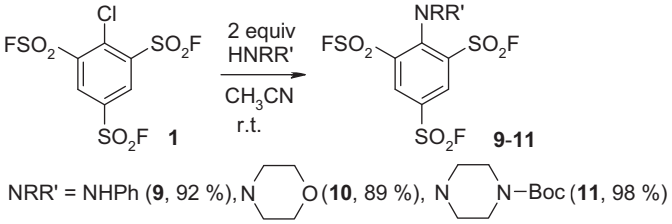
Interaction of chlorobenzene **1** with aniline or secondary amines passed with stoichiometrical ratio of reagents in acetonitrile at ambient temperature. The results were exclusively the products of chlorine atom substitution (Scheme 7).

Additional quantity of secondary amine leads to interaction with SO_2F group in step-by-step manner. Thus, using of 4 moles of morpholine gave compound **12** as a result of 4-SO_2F group attack (Scheme 8).

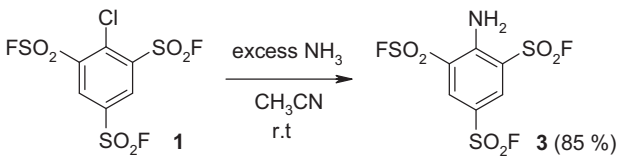
Difference in reactivity for *ortho*- and *para*-groups may be due to strong inductive deactivating influence of morpholyl moiety towards *ortho* positioned SO_2F reaction center accompanying with steric factors.

2.3. Reactions with S-nucleophiles

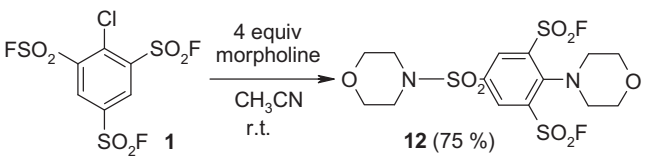
Selective reactions of chlorobenzene **1** with sulfurous nucleophiles such as sodium *p*-nitrothiophenolate, potassium ethylxantogenate and benzylmercaptan/Et_3N required milder conditions in comparison with N-nucleophiles. Interaction of equimolar quantity of reagents at −30 °C in acetonitrile (or DME in case of benzylmercaptane) and warming up to room temperature lead to



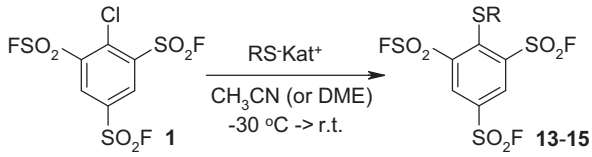
Scheme 7. Reactions of **1** with amines.



Scheme 6. Reaction of **1** with ammonia.

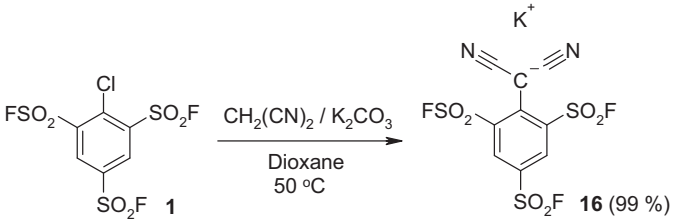


Scheme 8. Reaction of **1** with doubled quantity of morpholine.



RS$^-$Kat$^+$ = 4-NO_2-C_6H_4-SNa (**13**, 77 %), $C_2H_5OC(S)SK$ (**14**, 97 %), $PhCH_2SH$ / NEt_3 (**15**, 69 %)

Scheme 9. Reactions of **1** with thiolates.



Scheme 10. Reaction of **1** with malononitrile potassium salt.

the formation of corresponding sulfides **13**–**15** in good yields without participation of SO_2F groups (Scheme 9).

The excess of *p*-nitrothiophenolate used showed further attack of reaction centers (*para*-SO_2F, up to ^{19}F NMR data). However utilization of Na_2S gave unfortunately complex mixture and ambiguous results.

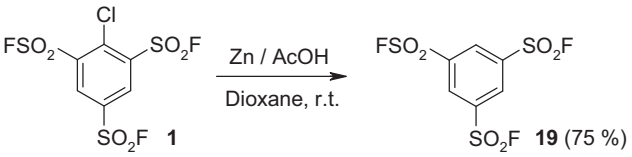
Saponification of compound **14** with K_2CO_3 in dioxane yielded not to expected potassium 2,4,6-tris(fluorosulfonyl)thiophenoxide but to potassium salt of phenol **2** that was isolated. The obtained result may be rationalized as attack of base to aromatic carbon atom bearing higher positive charge.

2.4. Reactions with C-nucleophiles

As an example for C-nucleophiles we choose malononitrile and it was found that chlorobenzene **1** reacted with $CH_2(CN)_2/K_2CO_3$ in dioxane at 50 °C to give the potassium salt **16** in quantitative yield (Scheme 10).

2.5. Reactions with Me_4NF

Chlorine atom in compound **1** may be easily substituted with fluorine by action with one equivalent of Me_4NF in DME yielding the fluorobenzene **17**. Reaction with two equivalents of Me_4NF gave the adduct of arene **17** with fluoride-anion – σ-complex **18** (Scheme 11). This adduct showed in NMR spectra the equivalence of two fluorine atoms in cyclohexadiene ring as well as analogous behavior of "aryl" protons and SO_2F groups chemical shifts to that of 1,3,5-tris(fluorosulfonyl)benzene anionic σ-complexes [18]. Analogous adduct of trinitrofluorobenzene with fluoride-anion was described by Terrier [23] on the basis of NMR data.



Scheme 12. Reduction of **1** with zinc/acetic acid.

2.6. Reduction with zinc

Chlorine atom in compound **1** may be easily replaced with hydrogen by zinc/acetic acid mixture in dioxane at 20–25 °C (Scheme 12), that is the convenient method to obtain 1,3,5-tris(fluorosulfonyl)benzene in substantial yield (75% isolated).

3. Conclusions

2,4,6-Tris(fluorosulfonyl)chlorobenzene (**1**) have confirmed its ability to realize various nucleophilic substitution pathways in dependence of reaction conditions that may led to regioselective C–Ar center attack or further fluorosulfonyl groups involvement. The 1-R-2,4,6-tris(fluorosulfonyl)benzenes family generated included corresponding arene alkyl and aryl ethers, anilines, sulfides and an example of arenemethide. In the course of investigations 1-alkyloxy-2,4,6-tris(fluorosulfonyl)benzenes obtained were found to be rather strong alkylating agents forming salts with 2,4,6-tris(fluorosulfonyl)phenoxide, that applied to dialkyl imidazolium system showed rather low melting point compounds. In case of fluoride-anion the second equivalent of reactant adds to the 1-position of compound **17** to form the stable anionic σ-complex with two fluorine atoms at sp^3 C atom. Convenient method to obtain 1,3,5-tris(fluorosulfonyl)benzene was elaborated upon reduction of chlorine atom in 2,4,6-tris(fluorosulfonyl)chlorobenzene (**1**) by Zn/AcOH.

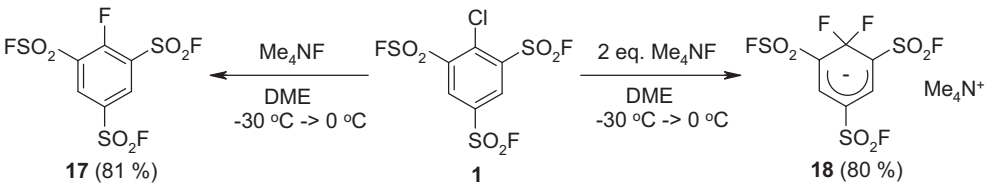
4. Experimental

4.1. General

All starting materials were obtained commercially. All solvents were dried using literature procedures.

^1H NMR spectra were recorded with a Varian-Gemini VXR 300 spectrometer at 299.9 MHz, ^{19}F NMR spectra – with a Bruker 200 spectrometer at 188.1 MHz and ^{13}C NMR spectra – with an Avance DRX 500 spectrometer in the solvents indicated.

Residual signals of the solvent protons with the chemical shifts $\delta = 7.25$ ppm ($CDCl_3$), $\delta = 2.07$ ppm ([D_6]acetone), $\delta = 2.50$ ppm ([D_6]DMSO) were used as an internal reference. For ^{19}F NMR spectra CCl_3F was used as an internal standard. Whenever possible the reactions were monitored by thin-layer chromatography (TLC). TLCs were run on Merck Kieselgel 60 F_{254} plates. Melting points were measured with an electro thermal Stuart Scientific melting point apparatus SMP3.



Scheme 11. Reactions of **1** with fluoride.

4.2. 2,4,6-Tris(fluorosulfonyl)phenol (2)

A solution of chlorobenzene (**1**, 0.5 g, 1.39 mmol) in abs. MeOH (25 ml) was boiled for 1 h. The solvent was removed under reduced pressure to afford **2** (0.46 g, 97%).

4.3. 2,4,6-Tris(fluorosulfonyl)aniline (3)

An access of gaseous ammonia was bubbled through a solution of chlorobenzene **1** (0.5 g, 1.39 mmol) in acetonitrile (15 mL) at 20 °C for about 1 min. Precipitate formed was filtered off. Filtrate was concentrated under reduced pressure to afford **3** (0.4 g, 85%) as a white powder; m.p. 203.5–205.5 °C (benzene). ^{1}H NMR ([D$_6$]DMSO): δ = 8.3 (s, 2 H, NH$_2$), 8.6 (s, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 61.4 (s, 2 F, *o*-SO$_2$F), 68.5 (s, 1 F, *p*-SO$_2$F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 116.5 (d, $^2J_{C,F}$ = 27.3 Hz, 1C, 4-C), 116.9 (d, $^2J_{C,F}$ = 26.3 Hz, 2C, 2-C, 6-C), 140.6 (s, 2C, 3-C, 5-C), 149.3 (s, 1C, 1-C) ppm.

4.4. 2,4,6-Tris(fluorosulfonyl)anisole (4a)

A solution of phenol **2** (3.0 g, 8.82 mmol) and silver carbonate (1.4 g, 5.08 mmol) in dist. H$_2$O (10 mL) was stirred at 60 °C for 2 h. Unreacted silver carbonate was filtered off, filtrate was concentrated under reduced pressure at 60 °C. Obtained silver phenoxide was dissolved in dry CH$_2$Cl$_2$ (50 mL) and CH$_3$I (10 mL, 160 mmol) was added, reaction mixture was stirred for 16 h at room temperature. Precipitate formed was filtered off. Filtrate was concentrated under reduced pressure to afford **4a** (2.6 g, 83%) as a white powder; m.p. 164–166 °C. ^{1}H NMR (CDCl$_3$): δ = 4.43 (s, 3 H, CH$_3$), 8.94 (s, 2 H, ArH) ppm. ^{19}F NMR (CDCl$_3$): δ = 63.7 (s, 2 F, *o*-SO$_2$F), 67.5 (s, 1 F, *p*-SO$_2$F) ppm. ^{13}C NMR (CDCl$_3$): δ = 67.8 (s, 3C, CH$_3$), 129.8 (d, $^2J_{C,F}$ = 30.9 Hz, 1C, 4-C), 132.5 (d, $^2J_{C,F}$ = 28.0 Hz, 2C, 2-C, 6-C), 138.1 (s, 2C, 3-C, 5-C), 163.4 (s, 1C, 1-C) ppm. Anal. calcd for C$_7$H$_5$F$_3$O$_7$S$_3$ (354.30): C 23.73, H 1.42, S 27.15. Found C 23.67, H 1.47, S 27.32.

4.5. 2,4,6-Tris(fluorosulfonyl)phenetole (4b)

Reaction was carried out similar to procedure for compound **4a**. C$_2$H$_5$I was used instead of CH$_3$I to give **4b** (2.46 g, 80%) as a white powder; m.p. 134–136 °C. ^{1}H NMR (CDCl$_3$): δ = 1.62 (t, $^2J_{H,F}$ = 7.2 Hz, 3 H, CH$_3$), 4.65 (q, $^2J_{H,F}$ = 7.2 Hz, 2 H, CH$_2$), 8.90 (s, 2 H, ArH) ppm. ^{19}F NMR (CDCl$_3$): δ = 63.1 (s, 2 F, *o*-SO$_2$F), 67.4 (s, 1 F, *p*-SO$_2$F) ppm. ^{13}C NMR (CDCl$_3$): δ = 15.3 (s, 3C, CH$_3$), 78.5 (s, 2C, CH$_2$) 129.4 (d, $^2J_{C,F}$ = 30.5 Hz, 1C, 4-C), 132.5 (d, $^2J_{C,F}$ = 27.8 Hz, 2C, 2-C, 6-C), 138.1 (s, 2C, 3-C, 5-C), 162.6 (s, 1C, 1-C) ppm. Anal. calcd for C$_8$H$_7$F$_3$O$_7$S$_3$ (368.33): C 26.09, H 1.97, S 26.12. Found C 26.19, H 2.01, S 26.33.

4.6. 1-Methyl-3-ethylimidazolium 2,4,6-tris(fluorosulfonyl)phenolate (5)

To a stirred solution of anisole **4** (0.25 g, 0.71 mmol) in benzene (2 mL) a solution of *N*-ethyl imidazole (0.07 g, 0.73 mmol) in benzene (2 mL) was added. The reaction mixture was stirred at room temperature for 1 h. The solvent was removed under reduced pressure to afford **5** (0.31 g, 98%) as a white solid; m.p. 125.5–127.7 °C (benzene). ^{1}H NMR ([D$_6$]DMSO): δ = 1.41 (t, $^2J_{H,H}$ 7.3 Hz, 3 H, CH$_3$CH$_2$), 3.84 (s, 3 H, CH$_3$N), 4.18 (q, $^2J_{H,H}$ 7.3 Hz, 2 H, CH$_2$CH$_3$), 7.68 (s, 1 H, C(5)H), 7.77 (s, 1 H, C(4)H), 8.24 (s, 2 H, ArH), 9.1 (s, 1 H, C(2)H) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 55.9 (s, 2 F, *o*-SO$_2$F), 69.6 (s, 1 F, *p*-SO$_2$F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 15.5 (s, 1C, CH$_2$*C*H$_3$), 36.2 (s, 3C, NCH$_3$), 44.6 (s, 1C, *C*H$_2$CH$_3$), 105.5 (d, $^2J_{C,F}$ = 26.8 Hz, 1C, 4-C), 122.4 (s, 1C, 5-C (Im)), 124.0 (s, 1C, 4-C (Im)), 124.3 (d, $^2J_{C,F}$ = 18.5 Hz, 2C, 2-C, 6-C), 136.7 (s, 1C, 2-C (Im)), 139.5 (s, 2C, 3-C, 5-C), 167.0 (s, 1C, 1-C) ppm. Anal. calcd for C$_{12}$H$_{13}$F$_3$N$_2$O$_7$S$_3$ (450.43): C 32.00, H 2.91, N 6.22, S 21.36. Found C 32.07, H 3.01, N 6.05, S 21.53.

4.7. 1-Methyl-3-(α,α,β,β-tetrafluoroethyl)imidazolium 2,4,6-tris(fluorosulfonyl)phenolate (6)

Reaction was carried out similar to procedure for compound **5** starting from anisole **4** (0.25 g, 0.71 mmol) and *N*-(α,α,β,β-tetrafluoroethyl)imidazole (0.12 g, 0.71 mmol). Reaction mixture was additionally stirred overnight to afford **6** (0.35 g, 95%) as a white solid; m.p. 85–87 °C (benzene). ^{1}H NMR ([D$_6$]DMSO): δ = 3.95 (s, 3 H, CH$_3$N), 7.17 (t, $^1J_{H,F}$ 51.4 Hz, 1 H, CHF$_2$), 8.06 (s, 1 H, C(5)H), 8.24 (s, 2 H, ArH), 8.29 (s, 1 H, C(4)H), 9.99 (s, 1 H, C(2)H) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = −137.4 (d, $^2J_{F,H}$ 51.4 Hz, 2 F, CF$_2$H), −99.1 (s, 2 F, CF$_2$N), 55.5 (s, 2 F, *o*-SO$_2$F), 69.2 (s, 1 F, *p*-SO$_2$F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 37.3 (s, 3C, CH$_3$), 105.6 (d, $^2J_{C,F}$ = 26.8 Hz, 1C, 4-C), 108.0 (tt, $^1J_{C,F}$ = 252,3 Hz, $^2J_{C,F}$ = 38.8 Hz, CF$_2$H), 112.2 (tt, $^1J_{C,F}$ = 269.2 Hz, $^2J_{C,F}$ = 29.9 Hz, 1C, NCF$_2$), 119.8 (s, 1C, 5-C (Im)), 124.3 (d, $^2J_{C,F}$ = 18.5 Hz, 2C, 2-C, 6-C), 126.3 (s, 1C, 2-C (Im)), 137.9 (s, 1C, 4-C (Im)), 139.4 (s, 2C, 3-C, 5-C), 167.0 (s, 1C, 1-C) ppm. Anal. calcd for C$_{12}$H$_9$F$_7$N$_2$O$_7$S$_3$ (522.40): C 27.59, H 1.74, N 5.36, S 18.41. Found C 27.51, H 1.80, N 5.21, S 18.65.

4.8. 2,4,6-Tris(fluorosulfonyl)diphenyl ether (7)

A mixture of chlorobenzene **1** (0.87 g, 2.43 mmol), phenol (0.24 g, 2.55 mmol) and dry grinded potash (0.5 g, 3.62 mmol) in dioxane (20 mL) was stirred at 50 °C for 8 h. Precipitate was filtered off. Filtrate was concentrated under reduced pressure to afford **8** (0.85 g, 84.2%) as a white powder; m.p. 155–156.7 °C. ^{1}H NMR ([D$_6$]DMSO): δ = 7.08 (d, $^2J_{H,H}$ 8.3 Hz, 2 H, 2,6-C$_6$H$_5$), 7.20 (t, $^2J_{H,H}$ 7.5 Hz, 1 H, 4-C$_6$H$_5$), 7.39 (t, $^2J_{H,H}$ 7.9 Hz, 2 H, 3,5-C$_6$H$_5$), 9.16 (s, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 64.6 (s, 2 F, *o*-SO$_2$F), 66.4 (s, 1 F, *p*-SO$_2$F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 105.5 (d, $^2J_{C,F}$ = 26.4 Hz, 1C, 4-C), 115.7 (s, 2C, 2,6-C$_6$H$_5$), 119.2 (s, 1C, 4-C$_6$H$_5$), 124.3 (d, $^2J_{C,F}$ = 19.1 Hz, 2C, 2-C, 6-C), 129.8 (s, 2C, 3,5-C$_6$H$_5$) 139.5 (s, 2C, 3-C, 5-C), 157.8 (s, 1C, 1-C$_6$H$_5$), 167.0 (s, 1C, 1-C) ppm. Anal. calcd for C$_{12}$H$_7$F$_3$O$_7$S$_3$ (416.37): C 34.62, H 1.69, S 23.10. Found C 34.55, H 1.74, S 22.89.

4.9. 2,4,6-Tris(β,β,β-trifluoroethoxysulfonyl)-β,β,β-trifluorophenetole (8)

A mixture of chlorobenzene **1** (0.5 g, 1.39 mmol), 2,2,2-trifluoroethanol (2 g, 20 mmol) and dry grinded potash (2 g, 14.47 mmol) in dioxane (30 mL) was stirred at room temperature for 24 h. Reaction mixture was diluted with water, formed precipitate was filtered off to afford **7** (0.82 g, 89%) as a white powder; m.p. 133–134.5 °C. ^{1}H NMR ([D$_6$]DMSO): δ = 4.90 (q, $^2J_{H,F}$ 8.4 Hz, 2 H, Ar-OCH$_2$CF$_3$), 5.08 (q, $^2J_{H,F}$ 8.4 Hz, 4 H, *o*-SO$_2$OCH$_2$CF$_3$), 5.13 (q, $^2J_{H,F}$ 8.4 Hz, 2 H, *p*-SO$_2$OCH$_2$CF$_3$), 8.73 (s, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = -72.1 (br s, 9 F, SO$_2$OCH$_2$CF$_3$), −72.0 (br s, 3 F, Ar-OCH$_2$CF$_3$) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 66.4 (q, $^2J_{C,F}$ = 37.4 Hz, 1C, SOCH$_2$), 66.7 (q, $^2J_{C,F}$ = 37.4 Hz, 2C, SOCH$_2$), 72.2 (q, $^2J_{C,F}$ = 36.8 Hz, 1C, ArOCH$_2$), 122.6 (q, $^1J_{C,F}$ = 277.3 Hz, 2C, SOCH$_2$*C*F$_3$), 122.8 (q, $^1J_{C,F}$ = 277.4 Hz, 1C, SOCH$_2$*C*F$_3$), 122.9 (q, $^1J_{C,F}$ = 277.6 Hz, 1C, ArOCH$_2$*C*F$_3$) 132.7 (s, 1C, 4-C), 133.4 (s, 2C, 2-C, 6-C), 137.9 (s, 2C, 3-C, 5-C), 157.4 (s, 1C, 1-C) ppm. Anal. calcd for C$_{14}$H$_{10}$F$_{12}$O$_{10}$S$_3$ (662.40): C 25.39, H 1.52, S 14.52. Found C 25.30, H 1.58, S 14.33.

4.10. 2,4,6-Tris(fluorosulfonyl)diphenylamine (9)

To a solution of chlorobenzene **1** (0.5 g, 1.39 mmol) in CH$_3$CN (20 mL) a solution of aniline (0.27 g, 2.9 mmol) in CH$_3$CN (2 mL) was added at room temperature. Reaction mixture was stirred for

1 h and poured in water. Precipitate formed was filtered off to afford **9** (0.53 g, 91.5%) as a yellow powder. 167–169 °C (benzene + hexane). ^{1}H NMR ([D$_6$]DMSO): δ = 7.35 (m, 5 H, C_6H_5), 8.96 (s, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 63.2 (s, 2 F, *o*-SO_2F), 67.7 (s, 1 F, *p*-SO_2F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 123.0 (s, 2C), 123.7 (d, $^2J_{C,F}$ = 43.6 Hz, 1C, 4-C), 125.7 (s, 1C), 127.9 (d, $^2J_{C,F}$ = 25.9 Hz, 2C, 2-C, 6-C), 129.4 (s, 2C), 140.7 (s, 2C, 3-C, 5-C), 143.1 (s, 1C), 148.9 (s, 1C, 1-C) ppm. Anal. calcd for $C_{12}H_8F_3NO_6S_3$ (415.39): C 34.70, H 1.94, N 3.37. Found C 34.46, H 1.89, N 3.48.

4.11. N-[2,4,6-Tris(fluorosulfonyl)phenyl]morpholine (10)

Reaction was carried out similar to procedure for compound **9**, starting from chlorobenzene **1** and morpholine to afford **10** (0.51 g, 89.4%) as a yellow powder; m.p. (decomp.) 198 °C, (benzene). ^{1}H NMR ([D$_6$]Aceton): δ = 3.48 (t, $^2J_{H,F}$ = 4.8 Hz, 4 H, CH_2N), 3.87 (t, $^2J_{H,F}$ = 4.8 Hz, 4 H, CH_2O), 9.12 (s, 2 H, ArH) ppm. ^{19}F NMR (Aceton): δ = 61.5 (s, 2 F, *o*-SO_2F), 66.5 (s, 1 F, *p*-SO_2F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 51.3 (s, 2C, CH_2N), 66.2 (s, 2C, CH_2O), 130.1 (d, $^2J_{C,F}$ = 29.2 Hz, 1C, 4-C), 137.8 (d, $^2J_{C,F}$ = 25 Hz, 2C, 2-C, 6-C), 139.1 (s, 2C, 3-C, 5-C), 155. 8 (s, 1C, 1-C) ppm. Anal. calcd for $C_{10}H_{10}F_3NO_7S_3$ (409.38): C 29.34, H 2.46, N 3.42. Found C 29.39, H 2.50, N 3.53.

4.12. N-(2,4,6-Tris(fluorosulfonyl)phenyl)piperazine-N′-Boc (11)

To a stirred solution of chlorobenzene **1** (0.91 g, 2.54 mmol) in CH_3CN (20 mL) at −30 °C a solution of *N*-Boc-piperazine (0.50 g, 2.68 mmol) and *N*-ethyldiisipropylamine (0.36 g, 2.78 mmol) in CH_3CN (5 mL) was slowly added. Reaction mixture was stirred for 1 h at ambient temperature and then poured in water. Formed precipitate was filtered off to afford **11** (1.26 g, 97.7%) as a yellow powder; m.p. (decomp.) 173 °C. ^{1}H NMR ($CDCl_3$): δ = 1.48 (s, 9 H, CH_3), 3.36 (m, 4 H, CH_2N), 3.64 (m, 4 H, CH_2N), 8.92 (s, 2 H, ArH) ppm. ^{19}F NMR ($CDCl_3$): δ = 61.1 (s, 2 F, *o*-SO_2F), 67.4 (s, 1 F, *p*-SO_2F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 28.5 (s, 9C, CH_3), 43.0–44.3 (m, 2C, NCH_2), 50.8 (s, 2C, NCH_2), 79.8 (s, 1C, $C(CH_3)_3$), 130.3 (d, $^2J_{C,F}$ = 28.7 Hz, 1C, 4-C), 137.7 (d, $^2J_{C,F}$ = 24.5 Hz, 2C, 2-C, 6-C), 139.1 (s, 2C, 3-C, 5-C), 154.4 (s, 1C, C(O)O), 156.1 (s, 1C, 1-C) ppm. Anal. calcd for $C_{15}H_{19}F_3N_2O_8S_3$ (508.51): C 35.43, H 3.77, N 5.51, S 18.92. Found C 35.45, H 3.70, N 5.59, S 19.02.

4.13. 1-Morpholino-4-morpholinosulfonyl-2,6-bis(fuorosulfonyl)benzene (12)

Reaction was carried out similar to procedure for compound **10**, starting from chlorobenzene **1** and doubled quantity of morpholine to afford **12** (0.5 g, 75%); m.p. (decomp.) ~215 °C (toluene + ethylacetate). ^{1}H NMR ([D$_6$]DMSO): δ = 3.09 (s, 4 H, CH_2N), 3.28 (s, 4 H, CH_2N), 3.68 (s, 4 H, CH_2O), 3.75 (s, 4 H, CH_2O), 8.54 (s, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 61.0 (s, 2 F, SO_2F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 45.9 (s, 2C, CH_2NSO_2), 50.8 (s, 2C, CH_2N), 65.8 (s, 2C, CH_2O), 66.4 (s, 2C, CH_2O), 134.9 (s, 1C, 4-C), 137.2 (s, 2C, 3-C, 5-C),138.1 (d, $^2J_{C,F}$ = 23 Hz, 2C, 2-C, 6-C), 153. 4 (s, 1C, 1-C) ppm. Anal. calcd for $C_{14}H_{18}F_2N_2O_8S_3$ (476.50): C 35.29, H 3.81, N 5.88, S 20.19. Found C 35.35, H 3.79, N 5.66, S 19.53.

4.14. 2,4,6-Tris(fluorosulfonyl)-4′-nitrodiphenylsulfide (13)

To a stirred solution of chlorobenzene **1** (0.5 g, 1.39 mmol) in dioxane (20 mL) sodium p-nitrothiophenolate (0.05 g, 0.28 mmol) was added. Reaction mixture was stirred for 8 h at ambient temperature. Formed precipitate was filtered off. Filtrate was concentrated under reduced pressure to afford **13** (0.51 g, 77%); m.p. 177–178.5 °C (benzene). ^{1}H NMR ([D$_6$]DMSO): δ = 7.41 (d, $^2J_{H,F}$ = 9.2 Hz, 2 H, *p*-NO_2-C_6H_4-S), 8.12 (d, $^2J_{H,F}$ = 9.2 Hz, 2 H, *p*-NO_2-C_6H_4-S), 9.21 (s, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 62.2 (s, 2 F, *o*-SO_2F), 66.7 (s, 1 F, *p*-SO_2F) ppm. Anal. calcd for $C_{12}H_6F_3NO_8S_4$ (477.43): C 30.19, H 1.27, N 2.93, S 26.86. Found C 30.22, H 1.30, N 2.85, S 27.14.

4.15. S-[2,4,6-Tris(fluorosulfonyl)phenyl] O-ethylxanthate (14)

Reaction was carried out similar to procedure for compound **13**, starting from chlorobenzene **1** and potassium *O*-ethylxanthate to afford **14** (1.2 g, 97%); m.p. 83–84 °C (benzene). ^{1}H NMR ([D$_6$]DMSO): δ = 1.14 (t, $^2J_{H,H}$ 7.0 Hz, 3 H, CH_3), 4.55 (q, $^2J_{H,H}$ 7.0 Hz, 2 H, CH_2), 9.20 (s, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 62.4 (s, 2 F, *o*-SO_2F), 66.7 (s, 1 F, *p*-SO_2F) ppm. ^{13}C NMR ($CDCl_3$): δ = 13.3 (s, 3C, CH_3), 72.3 (s, 2C, CH_2), 135.8 (s, 2C, 3-C, 5-C), 137.0 (d, $^2J_{C,F}$ = 31.3 Hz, 2C, 2-C, 6-C), 138.0 (s, 1C, 1-C), 143.5 (d, $^2J_{C,F}$ = 26.9 Hz, 1C, 4-C), 202.1 (s, 1C, C=S) ppm. Anal. calcd for $C_9H_7F_3O_7S_5$ (444.47): C 24.32, H 1.59, S 36.07. Found C 24.32, H 1.59, S 36.23.

4.16. 2,4,6-Tris(fluorosulfonyl)phenylbenzylsulfide (15)

Reaction was carried out similar to procedure for compound **13**, starting from chlorobenzene **1** (1 g, 2.8 mmol) and benzylmercaptane (0.35 g, 2.8 mmol) and triethylamine (0.31 g, 3.07 mmol) in DME (30 mL) to afford **15** (0.86 g, 69%); m.p. 191–192 °C (benzene + hexane). ^{1}H NMR ($CDCl_3$): δ = 4.50 (s, 2 H, CH_2), 7.46 (m, 5 H, C_6H_5), 9.10 (s, 2 H, ArH) ppm. ^{19}F NMR ($CDCl_3$): δ = 58.2 (s, 2 F, *o*-SO_2F), 66.1 (s, 1 F, *p*-SO_2F) ppm. Anal. calcd for $C_{13}H_9F_3O_6S_4$ (446.46): C 34.97, H 2.03, S 28.73. Found C 35.15, H 2.10, S 28.28.

4.17. Potassium 2,4,6-tris(fluorosulfonyl)phenyldicyanomethide (16)

A mixture of chlorobenzene **1** (3 g, 8.36 mmol), malononitrile (0.6 g, 9.08 mmol) and dry grinded potash (2.5 g, 18.1 mmol) in dioxane (80 mL) was stirred at 60 °C for 10 h. Orange precipitate formed was filtered off and washed with acetone. Filtrate was concentrated under reduced pressure to afford **16** (3.54 g, 99%). ^{1}H NMR ([D$_6$]DMSO): δ = 8.44 (s, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 71.4 (s, 2 F, *o*-SO_2F), 68.5 (s, 1 F, *p*-SO_2F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 53.0 (s, 2C, C^-), 114.7 (d, $^2J_{C,F}$ = 28.3 Hz, 1C, 4-C), 117.9 (s, 2C, CN), 120.7 (d, $^2J_{C,F}$ = 27.1 Hz, 2C, 2-C, 6-C), 140.2 (s, 2C, 3-C, 5-C), 143.7 (s, 1C, 4-C) ppm. Anal. calcd for $C_9H_2F_3N_2O_6S_3$ (426.42): C 25.35, H 0.47, N 6.57, S 22.56. Found C 24.97, H 0.66, N 6.49, S 22.85.

4.18. 2,4,6-Tris(fluorosulfonyl)fluorobenzene (17)

To a stirred solution of chlorobenzene **1** (1.2 g, 3.34 mmol) in dry DME (25 mL) at −30 °C in dry argon atmosphere Me_4NF [24] (0.44 g, 4.72 mmol) was added in one portion. Reaction mixture was slowly heated to 0 °C. Precipitate was filtered off. Filtrate was concentrated under reduced pressure. Extraction with dry benzene afforded **17** (0.93 g, 81%) as a white powder (insoluble in benzene residue was identified as σ-complex **18**); m.p. 132.5–134.0 °C. ^{1}H NMR ($CDCl_3$): δ = 8.94 (d, $^4J_{H,F}$ = 5.3 Hz, 2 H, ArH) ppm. ^{19}F NMR ($CDCl_3$): δ = −89.0 (tt, $^4J_{F,F}$ = 11.9 Hz, $^4J_{F,H}$ = 5.3 Hz, 1 F, ArF), 65.5 (d, $^4J_{F,F}$ = 11.9 Hz, 2 F, *o*-SO_2F), 67.3 (s, 1 F, *p*-SO_2F). ^{13}C NMR ($CDCl_3$): δ = 124.7 (dd, $^2J_{C,F}$ = 31.6 Hz, $^2J_{C,F}$ = 15.1 Hz, 2C, 2-C, 6-C), 129.7 (dd, $^2J_{C,F}$ = 31.5 Hz, $^4J_{C,F}$ = 4.6 Hz, 1C, 4-C), 137.4 (s, 2C, 3-C, 5-C), 159.4 (d, $^1J_{C,F}$ = 283.6 Hz, 1C, 1-C) ppm. Anal. calcd for $C_6H_2F_4O_6S_3$ (342.26): C 21.06, H 0.59, S 28.10. Found C 21.11, H 0.62, S 27.81.

4.19. Tetramethylammonium 1,1-difluoro-2,4,6-tris(fluorosulfonyl)cyclohexadienate (18)

Reaction was carried out similar to procedure for compound **17** but with a halved quantity of chlorobenzene **1** to afford **18** as a

greenish powder (0.58 g, 80%). ^{1}H NMR ([D$_6$]DMSO): δ = 3.10 (s, 14 H, CH_3), 7.91 (t, $^4J_{H,F}$ = 3.8 Hz, 2 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO, 282.2 MHz): δ = −36.6 to −36.8 (m, 2 F, CF_2), 69.2 (t, $^6J_{F,F}$ = 10 Hz, 1 F, *p*-SO_2F), 71.4 (t, $^4J_{F,F}$ = 13.5 Hz, 2 F, *o*-SO_2F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 54 9 (s, 12C, CH_3), 56.2 (br t, $^1J_{C,F}$ = 154.7 Hz, 1C, CF_2), 96.5 (d, $^2J_{C,F}$ 25.3 Hz, 1C, 4-C), 105.6 (dt, $^2J_{C,F}$ = 22.4 Hz, $^4J_{C,F}$ = 33.4 Hz, 2C, 2-C, 6-C), 138.5 (s, 2C, 3-C, 5-C) ppm.

*4.20. 1,3,5-Tris(fluorosulfonyl)benzene (**19**)*

A mixture of chlorobenzene **1** (10 g, 27.88 mmol), zinc dust (3.7 g, 56.59 mmol), glacial acetic acid (30 mL) and dioxan (30 mL) was stirred at room temperature for 16 h. Precipitate was filtered off and washed with dioxane. Filtrate was concentrated under reduced pressure and then poured in water. The formed precipitate was filtered off to afford **19** (6.78 g, 75%) as a white powder; m.p. 166–167 °C. ^{1}H NMR ([D$_6$]DMSO): δ = 9.26 (s, 3 H, ArH) ppm. ^{19}F NMR ([D$_6$]DMSO): δ = 67.6 (s, 3 F, SO_2F) ppm. ^{13}C NMR ([D$_6$]DMSO): δ = 135.7 (d, $^2J_{C,F}$ = 28.9 Hz, 3C, CS), 136.1 (s, 3C, CH) ppm.

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