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# Reaction of Difluoromethyl Pentafluorophenyl Sulfoxide with Nucleophiles

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**Abstract**—Reactions of 1-(difluoromethanesulfinyl)pentafluorobenzene with sodium methoxide, sodium phenoxide, potassium hydrosulfide, and methylamine resulted in substitution of fluorine atom in the 4-position (in the reaction with methylamine, also in the 2-position). Treatment of the title compound with sodium hydroxide afforded pentafluorobenzene due to cleavage of the $C_{arom}$–S bond.

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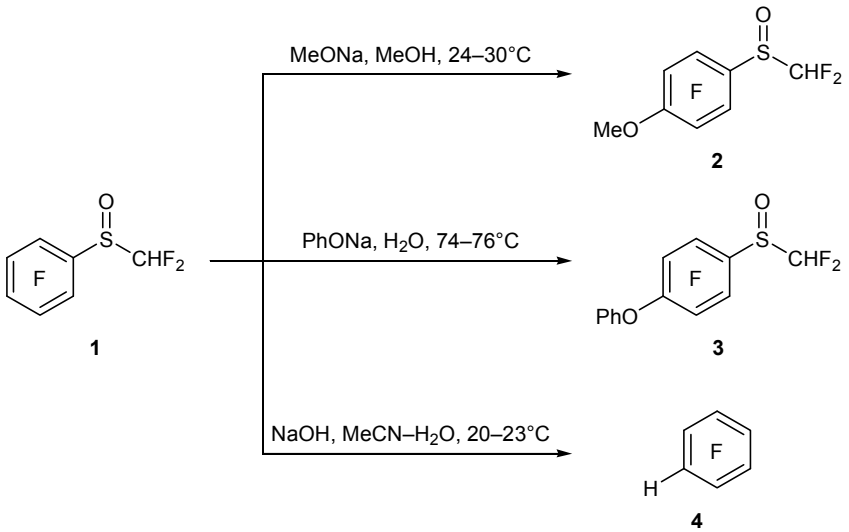
Aromatic polyfluorinated dendrimers are widely used as electrooptic chromophores; therefore, development of methods for their synthesis is an important problem [1, 2]. Polyfluoroaromatic blocks are commonly obtained from polyfluorobenzenes with various electron-donating substituents. Some fluorinated alkyl aryl sulfoxides in which the alkyl fragment contains a difluoromethyl group showed high insecticidal activity [3, 4], so that synthesis of new fluoroaryl difluoromethyl sulfoxides seems to be promising.

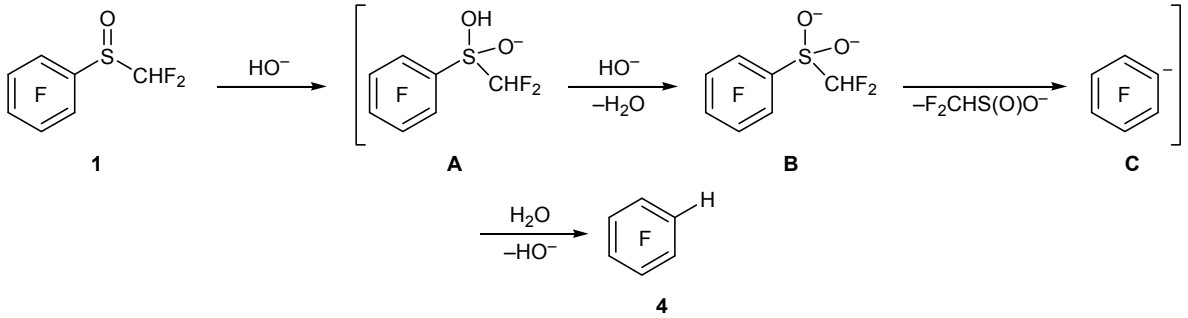
Polyfluoroaryl sulfoxides can be synthesized by reaction of polyfluoroarylmagnesium bromides with thionyl chloride [5] or by oxidation of polyfluoroaryl sulfides with nitric acid. Following the latter approach, we previously synthesized 1-(difluoromethanesulfinyl)pentafluorobenzene (**1**) [6] whose chemical properties were not studied previously.

The reaction of sulfoxide **1** with sodium methoxide gave 1-(difluoromethanesulfinyl)-4-methoxy-2,3,5,6-tetrafluorobenzene (**2**), and the reaction of **1** with sodium phenoxide afforded 1-(difluoromethanesulfinyl)-4-phenoxy-2,3,5,6-tetrafluorobenzene (**3**). Treatment of **1** with sodium hydroxide led to the formation of pentafluorobenzene (**4**) as a result of cleavage of the $C_{arom}$–S bond (Scheme 1). A probable mechanism of the latter reaction is shown in Scheme 2. The addition

**Scheme 1.**



**Scheme 2.**

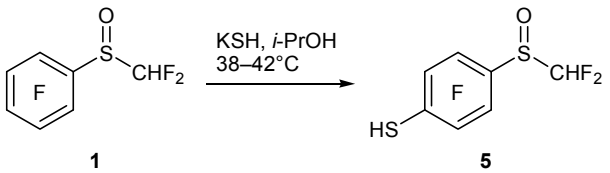


of hydroxide ion to **1** gives anion **A** which takes up the second hydroxide ion and loses water molecule, yielding dianion **B**. Elimination of difluoromethanesulfinate ion from intermediate **B**, followed by protonation, furnishes pentafluorobenzene (**4**).

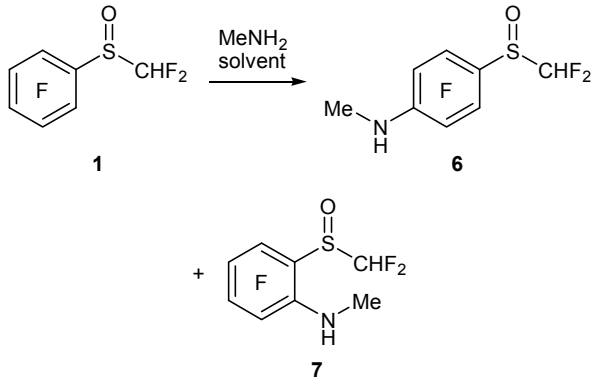
Dianion **B** was proposed as probable intermediate on the basis of comparison of the results of the reactions of **1** with sodium methoxide and sodium hydroxide. Attack of methoxide ion on the sulfur atom of **1** could give rise to the corresponding anion which, however, did not undergo haloform cleavage, so that substitution in the pentafluorophenyl ring occurred. Intermediate formation of dianion **B** is likely to be reasonable to rationalize haloform-type cleavage in the reaction with alkali. Nevertheless, further studies are required to get a deeper insight into the reaction mechanism. On the whole, the observed transformation is analogous to the haloform cleavage of pentafluorobenzaldehyde, octafluoroacetophenone, and methyl pentafluorophenyl ketone by the action of potassium hydroxide [7]. These reactions involve substitution of pentafluorophenyl group by hydroxide ion with formation of pentafluorophenyl anion whose protonation yields pentafluorobenzene (**4**) [8].

The reaction of **1** with potassium hydrogen sulfide gave 4-(difluoromethanesulfinyl)-2,3,5,6-tetrafluorobenzene-1-thiol (**5**) (Scheme 3). The major product in the reaction of **1** with methylamine in aqueous acetonitrile (ε = 36.2 [9]) was 4-(difluoromethanesulfinyl)-*N*-methyl-2,3,5,6-tetrafluoroaniline (**6**). The reaction mixture also contained small amount of isomeric 2-(difluoromethanesulfinyl)-*N*-methyl-3,4,5,6-tetrafluoroaniline (**7**) (Scheme 4). According to [10], the fraction of the *ortho* isomer in reactions of polyfluoroaromatic compounds with nucleophiles increased as the solvent polarity decreased. In fact, the reaction of **1** with methylamine in less polar methylene chloride (ε = 8.9 [9]) afforded a mixture of isomers **6** and **7**, the latter prevailing.

**Scheme 3.**



**Scheme 4.**



Thus, difluoromethyl pentafluorophenyl sulfoxide (**1**) reacted with sodium methoxide and sodium phenoxide, as well as with potassium hydrogen sulfide, via substitution of the *para*-fluorine atom by nucleophile. In the reaction of **1** with methylamine, fluorine atoms in the *para* and *ortho* positions are replaced by methylamino group. The behavior of sulfoxide **1** toward methylamine is analogous to the reactions of pentafluorobenzoic acid, ethyl pentafluorobenzoate, and pentafluoronitrobenzene with methylamine and dimethylamine [11]. The reaction of **1** with sodium hydroxide yields pentafluorobenzene in a way similar to haloform-type cleavage observed previously for some polyfluoroaromatic carbonyl compounds.

## EXPERIMENTAL

The analytical and spectral studies were performed at the Joint Chemical Service Center, Siberian Branch, Russian Academy of Sciences.

The $^1$H and $^{19}$F NMR spectra were recorded on a Bruker AV-300 spectrometer at 300 and 282.4 MHz, respectively, using carbon tetrachloride containing some $CDCl_3$ as solvent; the chemical shifts were measured relative to hexafluorobenzene ($^{19}$F) or hexamethyldisiloxane ($^1$H, δ 0.04 ppm downfield from TMS). The $^{13}$C NMR spectra were recorded on the same instrument at 100.6 MHz using $CDCl_3$ as solvent and tetramethylsilane as internal standard. The IR spectra were measured on a Bruker Vector 22 IR instrument. The UV spectra were recorded on a Hewlett Packard 8453 UV spectrophotometer. The molecular weights and elemental compositions were determined from the mass spectra (electron impact, 70 eV) which were obtained on a Thermo Electron Corporation DFS instrument. Gas chromatographic–mass spectrometric analysis was performed using an HP 5890 gas chromatograph coupled with an HP 5971 mass-selective detector (electron impact, 70 eV; HP-5 column, 30 m×0.25 mm, film thickness 0.25 μm; carrier gas helium, flow rate 1 mL/min; oven temperature programming from 50 to 280°C; ion source temperature 173°C). Gas chromatographic analyses were obtained with an HP 5890 chromatograph equipped with a thermal conductivity detector (HP-5 column, 30 m× 0.52 mm, film thickness 2.6 μm; oven temperature programming from 40 to 280°C, detector temperature 280°C; carrier gas helium, flow rate 5 mL/min). The melting points were determined on a Kofler hot stage.

**1-(Difluoromethanesulfinyl)-2,3,5,6-tetrafluoro-4-methoxybenzene (2).** A ~0.36 M solution of sodium methoxide in methanol, 15.5 mL (5.58 mmol), was added with stirring over a period of 2 min at 24–30°C to a solution of 1.50 g (5.58 mmol) of sulfoxide **1** in 7 mL of methanol. The mixture was stirred for 3 h, the solvent was distilled off, and the residue was washed with methylene chloride (2×5 mL). The undissolved material was separated, and the solvent was distilled off. Yield 1.34 g (78%), purity 90% (GLC). The crude product was distilled under reduced pressure (2 mm), gradually raising the oil bath temperature to ~140°C. Three fractions were isolated: first fraction, 0.23 g, bp 122–131°C, purity 95%; second fraction, 0.53 g, bp 132–137°C, purity 98%; third fraction, 0.06 g, bp 137–139°C, purity 98% (GLC). Colorless liquid. UV spectrum (EtOH): $\lambda_{max}$ 226 nm (log ε 3.97). IR spectrum (film), ν, cm$^{-1}$: 2966, 1635, 1504, 1487, 1439, 1396, 1286, 1269, 1196, 1119, 1076, 993, 947, 791, 509, 449. $^1$H NMR spectrum, δ, ppm: 4.21 t ($OCH_3$, $^5J_{HF}$ = 2 Hz), 6.80 d.d ($CHF_2$, $^2J$ = 56, 55 Hz). $^{13}$C NMR spectrum, $\delta_C$, ppm: 62.0 t ($OCH_3$, $^4J_{CF} \approx$ 4 Hz), 108.8 m, 119.9 d.d.t ($CHF_2$, $^1J_{CF}$ = 292, 284, $^4J_{CF} \approx$ 4 Hz), 140.2 d.m (CF, $^1J_{CF}$ = 253 Hz), 143.4 t.t ($^2J_{CF} \approx$ 11, $^3J_{CF} \approx$ 4 Hz), 146.2 d.d.d.d (CF, $^1J_{CF}$ = 255, $^2J_{CF}$ = 13, $^3J_{CF} \approx$ 8, $^4J_{CF} \approx$ 4 Hz). $^{19}$F NMR spectrum, $\delta_F$, ppm: 7.1 m (2F, 3-F, 5-F), 23.3 m (2F, 2-F, 6-F), 43.4 d.t ($CHF_2$, $^2J_{FF}$ = 268, $^2J_{FH}$ = 56, $^5J_{FF} \approx$ 4 Hz), 46.5 d.t ($CHF_2$, $^2J_{FF}$ = 268, $^2J_{FH}$ = 55, $^5J_{FF}$ = 5 Hz). Found, %: C 34.25; H 1.56; F 41.21; S 11.25. *m*/*z* 277.9832 $[M]^+$. $C_8H_4F_6O_2S$. Calculated, %: C 34.54; H 1.45; F 40.98; S 11.53. *M* 277.9831.

**(1-Difluoromethanesulfinyl)-2,3,5,6-tetrafluoro-4-phenoxybenzene (3).** A ~0.6 M solution of sodium phenoxide in water, 12.7 mL (7.87 mmol), was added dropwise with stirring to a mixture of 2.01 g (7.48 mmol) of sulfoxide **1** and 40 mL of water, heated to 42°C. Water, 10 mL, was then added, and the mixture was stirred for 24 h at 42–50°C (oil bath). The mixture was cooled to room temperature, and the precipitate was filtered off, washed with water, and dried in air. Yield 2.41 g (94%), white solid, mp 74–76°C. UV spectrum ($CHCl_3$), $\lambda_{max}$, nm (log ε): 267 (3.96), 272 (3.96). IR spectrum (KBr), ν, cm$^{-1}$: 2985, 1637, 1591, 1493, 1483, 1458, 1392, 1296, 1211, 1165, 1130, 1095, 1076, 1024, 982, 760, 729, 711, 688, 660, 507, 478, 440. $^1$H NMR spectrum, δ, ppm: 6.83 d.d ($CHF_2$, $^2J_{HF}$ = 56, 55 Hz), 7.16 m (5H, $H_{arom}$). $^{13}$C NMR spectrum, $\delta_C$, ppm: 112.2 m, 115.9 s (CH), 119.9 d.d.t ($CHF_2$, $^1J_{CF}$ = 293, 285, $^4J_{CF} \approx$ 4 Hz), 124.6 s (CH), 129.9 s (CH), 139.1 t.t ($^2J_{CF} \approx$ 12, $^3J_{CF} \approx$ 4 Hz), 141.5 d.d.d (CF, $^1J_{CF}$ = 258, $^2J_{CF} \approx$ 12, $^3J_{CF} \approx$ 5 Hz), 146.1 d.d.d.d (CF, $^1J_{CF}$ = 257, $^2J_{CF}$ = 12, $^3J_{CF} \approx$ 7, $^4J_{CF} \approx$ 4 Hz), 156.3 s. $^{19}$F NMR spectrum, $\delta_F$, ppm: 11.8 m (2F, 3-F, 5-F), 24.2 m (2F, 2-F, 6-F), 43.4 d.t ($CHF_2$, $^2J_{FF}$ = 269, $^2J_{FH}$ = 56, $^5J_{FF}$ = 3 Hz), 47.4 d.t ($CHF_2$, $^2J_{FF}$ = 269, $^2J_{FH}$ = 55, $^5J_{FF}$ = 4 Hz). Found, %: C 45.82; H 1.83; F 33.63; S 9.14. *m*/*z* 339.9988 $[M]^+$. $C_{13}H_6F_6O_2S$. Calculated, %: C 45.89; H 1.78; F 33.50; S 9.42. *M* 339.9987.

**Pentafluorobenzene (4).** A solution of 1.47 g (36.75 mmol) of sodium hydroxide in 5 mL of water was added at 20–23°C to a solution of 1.91 g (7.00 mmol) of sulfoxide **1** in 18 mL of acetonitrile. The mixture was stirred for 3 h and poured into 200 mL of water (two layers were formed). The bottom layer was separated and dried over $CaCl_2$. Yield 0.70 g (57%). $^1$H NMR spectrum: δ 6.86 ppm, m (6-H) [12]. $^{13}$C NMR spectrum, $\delta_C$, ppm: 100.6 t.d (CH, $^2J_{CF}$ = 23, $J_{CF}$ = 3 Hz), 137.6 d.d.d.d.d ($C^1$, $C^5$ or $C^2$, $C^4$, $^1J_{CF}$ = 251, $J_{CF}$ = 18, 13, 5, 1 Hz), 141.8 d.t.t ($C^3$, $^1J_{CF}$ = 254, $^2J_{CF}$ = 13, $^3J_{CF}$ = 5 Hz), 146.2 d.t.t ($C^2$, $C^4$ or $C^1$, $C^5$, $^1J_{CF}$ = 249, $J_{CF} \approx$ 12, $J_{CF} \approx$ 4 Hz). $^{19}$F NMR spectrum,

$\delta_F$, ppm: 0.1 m (2F, 2-F, 4-F), 8.1 t.t (1F, 3-F, $^3J_{FF}$ = 20, $^4J_{FF}$ = 1 Hz), 23.8 m (2F, 1-F, 5-F) [12]. Found: *m*/*z* 168 $[M]^+$. $C_6HF_5$. Calculated: *M* 168.06.

**4-(Difluoromethanesulfinyl)-2,3,5,6-tetrafluorobenzenethiol (5).** A ~4 M solution of potassium hydrogen sulfide in ethylene glycol, 3.37 mL (13.48 mmol), was added dropwise with vigorous stirring at 33–37°C to a solution of 2.00 g (7.74 mmol) of sulfoxide **1** in 35 mL of isopropyl alcohol. The mixture was heated for 5 h at 38–42°C, poured into a mixture of ~50 g of ice and 100 mL of ~10% aqueous HCl, and extracted with methylene chloride (4×25 mL). The solvent was distilled off from the combined extracts to obtain 1.63 g of a product which contained (according to the $^{19}$F and $^1$H NMR data), ~90% of thiol **5** (yield 69%). A 0.99-g portion of the crude product was sublimed at 90–95°C over a period of 10 min. We thus isolated 0.12 g of solid thiol **5** with a purity of 95% ($^{19}$F and $^1$H NMR) and 0.06 g of a liquid containing 53% of thiol **5**. Overall yield 31% [13]. Recrystallization of the solid product from a mixture of 0.5 mL of methylene chloride and 2 mL of hexane gave 22 mg of thiol **5** as greenish solid, mp 58–60°C (from $CH_2Cl_2$–hexane). UV spectrum ($CHCl_3$): $\lambda_{max}$ 271 nm (log$\varepsilon$ 4.27). IR spectrum (KBr), $\nu$, cm$^{-1}$: 1627, 1477, 1252, 1115, 1072, 943. $^1$H NMR spectrum, $\delta$, ppm: 4.03 s (SH), 6.78 d.d ($CHF_2$, $^2J_{HF}$ = 56, 55 Hz). $^{13}$C NMR spectrum, $\delta_C$, ppm: 112.7 m, 119.9 d.d.t ($CHF_2$, $^1J_{CF}$ = 293, 285, $^4J_{CF} \approx$ 4 Hz), 121.4 t ($^2J_{CF}$ = 21 Hz), 142.8 d.d.t (CF, $^1J_{CF}$ = 250, $^2J_{CF}$ = 15, $J_{CF} \approx$ 5 Hz), 145.1 d.d.t (CF, $^1J_{CF}$ = 258, $^2J_{CF} \approx$ 16, $J_{CF} \approx$ 5 Hz). $^{19}$F NMR spectrum, $\delta_F$, ppm: 24.0 m, 27.4 m, 43.4 d.t (1F, $CHF_2$, $^2J_{FF}$ = 269, $^2J_{FH}$ = 56, $^5J_{FF}$ = 3 Hz), 47.1 d.t (1F, $CHF_2$, $^2J_{FF}$ = 269, $^2J_{FH}$ = 55, $^5J_{FF}$ = 4 Hz). Found: *m*/*z* 279.9445 $[M]^+$. $C_7H_2F_6OS_2$. Calculated: *M* 279.9446.

**Reaction of 1-(difluoromethanesulfinyl)pentafluorobenzene (1) with methylamine.** A solution of methylamine in water was added to a solution of sulfoxide **1** in acetonitrile or methylene chloride. The mixture was stirred at nearly room temperature and dried over $MgSO_4$, and the drying agent was filtered off and washed with the corresponding solvent (acetonitrile or methylene chloride, 4×5 mL). The filtrate was evaporated, the residue was treated with 6 mL of hexane at room temperature, and the precipitate was filtered off and washed with hexane (2×6 mL) to isolate pure isomer **6**. Isomer **7** was isolated from the combined mother liquor and hexane washings.

*a*. The reaction mixture obtained from 1.16 g (4.27 mmol) of **1** in 14 mL of acetonitrile and 0.72 mL (6.54 mmol) of aqueous methylamine (9.08 M) at 25–32°C (12 h) contained compounds **6** and **7** at a ratio of 1 : 0.15 ($^{19}$F NMR). After purification, we isolated 0.89 g of **6** (75%).

*b*. The reaction mixture obtained from 1.40 g (5.16 mmol) of **1** in 14 mL of methylene chloride and 1.24 mL (11.26 mmol) of aqueous methylamine (9.08 M) at 23–27°C (24 h) contained isomers **6** and **7** at a ratio of 0.42 : 1 ($^{19}$F NMR). After appropriate treatment, we isolated 0.36 g (25%) of **6**. The solvent (hexane) was distilled off to a volume of 4 mL, the residue was cooled to –15°C, and the precipitate (compound **7**) was filtered off and recrystallized from hexane at –15°C. Yield 0.94 g (64%).

**4-(Difluoromethanesulfinyl)-2,3,5,6-tetrafluoro-*N*-methylaniline (6).** White solid, mp 97–98°C (from hexane). UV spectrum (EtOH), $\lambda_{max}$, nm (log$\varepsilon$): 224 (3.96), 298 (4.40). IR spectrum (KBr), $\nu$, cm$^{-1}$: 3404, 1645, 1547, 1502, 1458, 1433, 1390, 1325, 1286, 1178, 1113, 1086, 1057, 1003, 960, 507, 457. $^1$H NMR spectrum, $\delta$, ppm: 3.21 d.t ($CH_3$, $^3J_{HH}$ = 5, $^5J_{HF}$ = 3 Hz), 4.52 br.s (NH), 6.77 d.d ($CHF_2$, $^2J_{HF}$ = 56, 55 Hz). $^{13}$C NMR spectrum, $\delta_C$, ppm: 32.2 t ($CH_3$, $^4J_{CF} \approx$ 5 Hz), 100.6 m, 120.0 d.d.t ($CHF_2$, $^1J_{CF}$ = 291, 282, $^4J_{CF} \approx$ 5 Hz), 134.6 t.t ($^2J_{CF} \approx$ 10, $^3J_{CF} \approx$ 4 Hz), 135.9 d.d.t (CF, $^1J_{CF}$ = 244, $^2J_{CF} \approx$ 16, $J_{CF} \approx$ 4 Hz), 146.4 d.m (CF, $^1J_{CF}$ = 250 Hz). $^{19}$F NMR spectrum, $\delta_F$, ppm: 1.4 m (2F, 2-F, 6-F), 21.7 m (2F, 3-F, 5-F), 43.2 d.t ($CHF_2$, $^2J_{FF}$ = 267, $^2J_{FH}$ = 56, $^5J_{FF}$ = 5 Hz), 47.7 d.t ($CHF_2$, $^2J_{FF}$ = 267, $^2J_{FH}$ = 55, $^5J_{FF}$ = 4 Hz). Found, %: C 34.78; H 1.75; F 40.89; N 4.93; S 11.32. *m*/*z* 276.9990 $[M]^+$. $C_8H_5F_6NOS$. Calculated, %: C 34.66; H 1.82; F 41.12; N 5.05; S 11.57. *M* 276.9991.

**2-(Difluoromethanesulfinyl)-3,4,5,6-tetrafluoro-*N*-methylaniline (7).** White solid, mp 31–33°C (from hexane). UV spectrum (EtOH), $\lambda_{max}$, nm (log$\varepsilon$): 211 (4.26), 240 (3.82), 333 (3.55). IR spectrum (KBr), $\nu$, cm$^{-1}$: 3327, 1649, 1622, 1593, 1537, 1502, 1464, 1435, 1265, 1196, 1163, 1117, 1095, 1047, 939, 784, 519, 444. $^1$H NMR spectrum, $\delta$, ppm: 3.10 d.d ($CH_3$, $^5J_{HF}$ = 7, $^3J_{HH}$ = 5 Hz), 6.64 d.d ($CHF_2$, $^2J_{HF}$ = 56, 54 Hz), 6.77 br.s (NH). $^{13}$C NMR spectrum, $\delta_C$, ppm: 32.5 d ($CH_3$, $^4J_{CF}$ = 11 Hz), 100.4 d ($^2J_{CF}$ = 16 Hz), 117.8 t ($CHF_2$, $^1J_{CF}$ = 286 Hz), 131.2 d.t (CF, $^1J_{CF}$ = 247, $^2J_{CF} \approx$ 15 Hz), 137.5 d.d.d (CF, $^1J_{CF}$ = 249, $^2J_{CF} \approx$ 13, $^3J_{CF} \approx$ 4 Hz), 138.6, 145.5 d.d.d.d (CF, $^1J_{CF}$ = 248, $^2J_{CF} \approx$ 15, 13, $^3J_{CF}$ = 6 Hz), 145.9 d.t.d (CF, $^1J_{CF}$ = 248, $J_{CF} \approx$ 11, 5 Hz). $^{19}$F NMR spectrum, $\delta_F$, ppm: –9.7 d.d.d (4-F, $J_{4,3}$ = 24, $J_{4,5}$ = 22, $J_{4,6}$ = 6 Hz), 3.7 m

(6-F), 15.4 d.d.d (5-F, $J_{5,4}$ = 22, $J_{5,6}$ = 18, $J_{5,3}$ = 6 Hz), 22.9 m (3-F), 42.1 d.d ($CHF_2$, $^2J_{FF}$ = 265, $^2J_{FH}$ = 56, $^5J_{FF}$ = 3 Hz), 45.4 d.d ($CHF_2$, $^2J_{FF}$ = 265, $^2J_{FH}$ = 54 Hz, $^5J_{FF}$ = 5 Hz). Found, %: C 34.81; H 1.78; F 41.18; N 4.86; S 11.62. *m*/*z* 276.9993 $[M]^+$. $C_8H_5F_6NOS$. Calculated, %: C 34.66; H 1.82; F 41.12; N 5.05; S 11.57. *M* 276.9991.

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