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The investigation of fluorination reaction of *p*-substituted benzenesulfonimides with fluorine–nitrogen mixed gas to synthesize NFSI analogues

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Dedicated to Professor Weiyuan Huang on the occasion of his 90th birthday.

ABSTRACT

This paper studied the fluorination reaction of *p*-substituted benzenesulfonimides with diluted elemental fluorine to synthesize N-fluoro-benzenesulfonimide (NFSI) analogues. Several synthetic methods were compared and we found that, for many *p*-substituted benzenesulfonimides, the fluorination of their sodium salts with 10% F_2–N_2 mixed gas in acetonitrile at room temperature could afford NFSI analogues in moderate to good yields.

1. Introduction

Fluorine substituents have become widespread and important drug and material components [1–6], their introduction was facilitated by the development of safe and selective fluorinating agents [7–8]. Today, an increasing number of such agents are directly available to researchers from commercial suppliers [9–11].

Recently, NFSI, one of the commercial fluorinating reagents, has engendered many attentions for its applications in high enantioselective fluorination [12–17]. However, to become a good fluorinating reagent, there are still some drawbacks to overcome: (i) in the enantioselective fluorination of carbanions, only a few good examples appear in the literature, while for most compounds, the results are not satisfactory [18–19], (ii) in the fluorination of aromatic compounds, its reactivity is not high and usually the fluorinated products are afforded in low yields [20].

Structure modification of NFSI by changing the substituent in the benzene ring supplies one possible approach to improve its fluorinating reactivity and selectivity. The substituent modification to the benzene ring can change the molecular electronic state and N–F bond strength, therefore is able to adjust its reactivity for the fluorination reaction. Though several preparing methods for NFSI [21–24] appeared in the literature, there was no promising and effective approach for synthesizing NFSI analogues. Herein, the fluorination of *p*-substituted benzenesulfonimides with fluorine–nitrogen mixed gas was investigated in detail to develop an efficient method for the synthesis of NFSI analogues.

2. Results and discussion

According to Differding's method [21], the reaction of *p*-substituted benzenesulfonimides 1 with fluorine-nitrogen mixed gas in CH_3CN in the presence of sodium fluoride was firstly investigated. The results are shown in Table 1.

We found that the reaction was sensitive to many factors such the source of substrate, reaction temperature, reaction scale, reaction time, pH value, etc., and as a result, reproducibility could not be achieved at a satisfactory level. The source of substrates influenced our reaction results apparently due to their different solubility. For compound **1a** purchased from Aldrich, it was difficult to dissolve it in acetonitrile, thus compound **2a** was obtained in much low yield with the incomplete conversion of compound **1a** in this fluorination process (entries 1–5), while for compound **1a** prepared in our lab, which had a good solubility in acetonitrile, the fluorination reaction afforded compound **2a** in 82.2% yields (entry 6). Similarly, for compounds **1b** and **1c**

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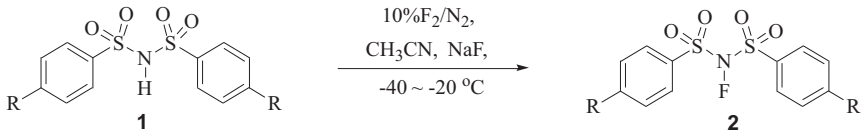
Table 1
The reaction of *p*-substitutent benzenesulfonimides with F_2–N_2 mixed gas.



1 2

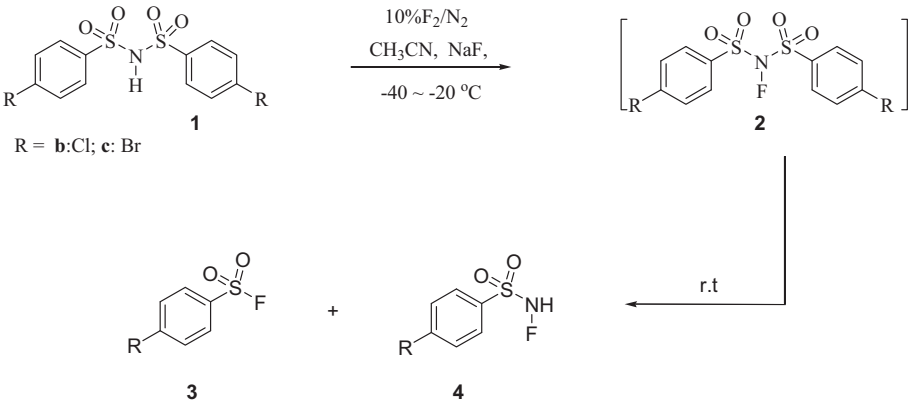
R = **a**:F; **b**:Cl; **c**: Br; **d**:CF_3; **e**:OCF_3; **f**:t-Bu; **g**:Me; **h**:OCH_3

Entry	Substrate **1** (R)	Quantity of **1** (g)[a]	Product/yield (%)[b]
1	**1a** (F)[c]	3.46	**2a** (10.7) (recovered **1a** 1.14 g)
2	**1a** (F)[c]	16.65	**2a** (34.7) (recovered **1a** 8.40 g)
3	**1a** (F)[c]	17.30	**2a** (38.4) (recovered **1a** 12.00 g)
4	**1a** (F)[c]	18.00	**2a** (25.8) (recovered **1a** 12.50 g)
5	**1a** (F)[c]	18.60	**2a** (19.4) (recovered **1a** 12.00 g)
6	**1a** (F)[d]	16.65	**2a** (82.2)
7	**1b** (Cl)[c]	0.92	**2b** (17.6)
8	**1b** (Cl)[c]	10.80	**2b** (35.3) (recovered **1b** 5.7 g)
9	**1b** (Cl)[c]	23.30	**2b** (37.2) (recovered **1b** 11.0 g)
10	**1b** (Cl)[d]	22.00	**2b** (7.9) [e]
11	**1c** (Br)[c]	1.10	**2c** (30.7)
12	**1c** (Br)[c]	28.00	**2c** (23.7) (recovered **1c** 21.0 g)
13	**1c** (Br)[c]	38.00	**2c** (7.6) (recovered **1c** 32.0 g)
14	**1c** (Br)[c]	43.75	**2c** (15.7) (recovered **1c** 40.0 g)
15	**1c** (Br)[d]	14.45	**2c** (7.3) [f]
16	**1d** (CF_3)[c]	4.30	**2d** (21.1)
17	**1e** (OCF_3)[c]	20.60	**2e** (19.6)
18	**1f** (t-Bu)[c]	5.00	**2f** (47.9)
19	**1f** (t-Bu)[c]	40.00	**2f** (51.5)
20	**1h** (OMe)[c]	5.00	**2h** (23.8)

[a] The reaction was carried out in a glass bottle in acetonitrile at −40 to −20 °C with 3–5 equivalents of 10% F_2–N_2 mixed gas in the presence of 8 equivalents of NaF.
[b] Separated yield.
[c] Purchased from Aldrich.
[d] Prepared in our lab.
[e] Byproduct **3b** was obtained except **2b**.
[f] Except **2c**, compound **3c** and **4c** were obtained in 38.3% and 19.2%, respectively.

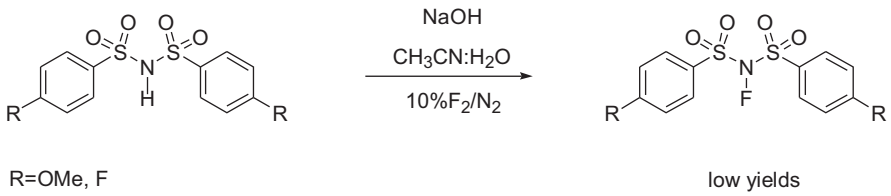
purchased from Aldrich, the conversion of substrate was incomplete, therefore **2b** and **2c** were obtained in low yields (entries 7–9, 11–14). While for **1b** and **1c** made in our lab, TLC monitoring indicated that compound **1b** and **1c** actually gave **2b** and **2c** respectively at −40 °C, but the solution color changed from colorless to red when solution temperature was gradually raised to 0 °C, it turned out that **2b** and **2c** were decomposed to give **3b**, **4b** and **3c**, **4c** respectively (entries 10 and 15, Scheme 1). Though the main reason for the decomposition of compounds **2b** and **2c** was unknown, we speculated it might result from the weakening of their stability with the increase of solution temperature at that pH circumstance. Different batches were tried, but the yields were fluctuated (entries 1–5 for **1a**, entries 7–9 for **1b**, entries 11–14 for **1c**).

A patent [17] described the fluorination reaction of the sodium salt of compound **1** in the acetonitrile–water cosolvent system. We tried the reaction of compound **1a** with 3–5 equivalents of 10% F_2–N_2 in 1:1 acetonitrile/water at 0 °C in the presence of 1.05 equivalents of sodium hydroxide, but no **2a** was obtained. Similarly, as the reaction of 1 h with 3–5 equivalents of 10% F_2–N_2 was tried in water at 25 °C in the presence of 1.05 equivalents sodium hydroxide, only a small amount of compound **2h** was obtained with a large amount of unconverted reactant 1 h (Scheme 2). In this process water reacted with F_2 accompanying the fluorination reaction of sodium salts, therefore the acidity of the solution was increased and the salt was changed back to compound **1**.



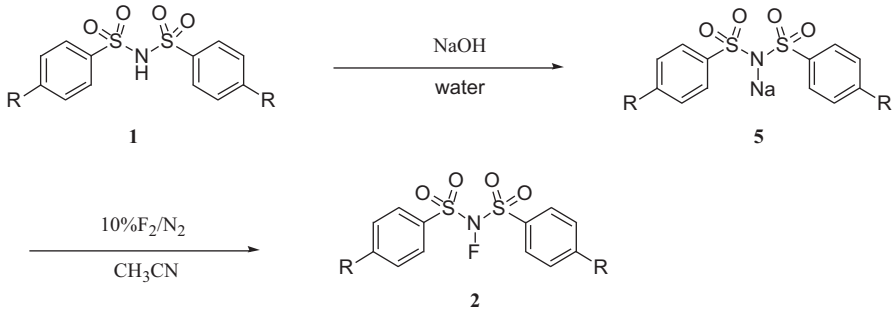
1 R = **b**:Cl; **c**: Br 2 3 4

Scheme 1. The decomposition of compounds **1b** and **1c**.



Scheme 2. The reaction of *p*-substitutent benzenesulfonimides with 10% F_2–N_2 mixed gas in the presence of sodium hydroxide in the aqueous acetonitrile or water.

Table 2
The reaction of sodium salts of *p*-substituted benzenesulfonimides with 10% F_2–N_2 mixed gas in acetonitrile.



R = **a**:F; **b**:Cl; **c**: Br; **d**:CF_3; **e**:OCF_3; **f**:t-Bu; **g**:Me; **h**:OCH_3

Entry	Substrate (R)	Conditions	Product	Yield (%)[a]
1	**1b** (Cl)	CH_3CN, −30 to −20 °C	**2b**	65.3
2	**1c** (Br)	CH_3CN, −30 to −20 °C	**2c**	69.6
3	**1f** (t-Bu)	CH_3CN, −30 to −20 °C	**2f**	77.6
4	**1d** (CF_3)	CH_3CN, 5–15 °C	**2d**	87.5
5	**1e** (OCF_3)	CH_3CN, 5–15 °C	**2e**	88.6
6	**1g** (Me)	CH_3CN, 5–15 °C	**2g**	30.0
7	**1g** (Me)	CH_3CN/H_2O = 200:1, 5–15 °C	**2g**	68.5
8	**1g** (Me)	CH_3CN/H_2O = 150:1, 5–15 °C	**2g**	89.4
9	**1h** (OMe)	CH_3CN, 5–15 °C	**2h**	20.9
10	**1h** (OMe)	CH_3CN/H_2O = 50:1, 5–15 °C	**2h**	28.0

[a] Separated yield.

Considering that water existing in this system resulted in a decrease in the yield of fluorinated products, we investigated the solubility of the sodium salts in acetonitrile and water. The sodium salts **5** were prepared by the reaction of compound **1** with 2% NaOH solution and subsequent precipitation, while for **1g** and **1h**, NaCl was additionally added to deposit their sodium salts **5g** and **5h** from the aqueous solution. Surprisingly, we found the solubility of the salt 5**b**, **5c**, **5d**, **5e** and **5f** were poor in water but good in acetonitrile, and only the salts **5g** and **5h** had a better solubility in water than acetonitrile. On the basis of the above experiments, we tried the fluorination reaction of sodium salts according to their solubility. To our delight, the reaction of the sodium salts **5b**, **5c** and **5f** with elemental fluorine was finished quickly at −30 to −20 °C and compounds **2** were given in moderate to good yields (Scheme 2, entries 1–3). For the sodium salts **5d** and **5e**, the fluorination reaction occurred at room temperature, similarly compounds **2d** and **2e** were given in good yields (entries 4, 5). For the sodium salt **5g**, when some water was added to acetonitrile, its solubility was enhanced greatly, and the reaction was carried out completely with the generation of compound **2g** in high yield (entries 6–8). However, for the sodium salt **5h**, the fluorination reaction solution turned red and low yield was given both in acetonitrile and in 50:1 acetonitrile/water cosolvent (entries 9 and 10), which might be contributed to the electron-donating effect of methoxyl group on the N–F bond weakening (Table 2).

3. Conclusion

In conclusion, in this paper we investigated the fluorination reaction of benzenesulfonimides with 10% F_2–N_2 mixed gas. Different reaction methods were tried and we found that, for many *p*-substituted benzenesulfonimides, the reaction of their sodium salts with 10% F_2–N_2 mixed gas was finished quickly and gave compounds **2** in moderate to good yields, which supplied one efficient method for the synthesis of NFSI analogues.

4. Experimental

4.1. General

Melting points were measured on WRS-1B digital instrument. ^1H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker 400 MHz instrument with TMS and $CFCl_3$ as external references, respectively. Infrared spectra were measured on a Shimadzu IR-440. Mass spectra were recorded on a Finnigan GC-MS-4021.

4.2. General experimental procedure for the fluorination reaction of p-substituted benzenesulfonimides with 10% F_2–N_2 mixed gas

Method A: A mixture of compound **1** (50 mmol), NaF (400 mmol) and acetonitrile (380 ml) was stirred and cooled to

−40 °C, a 13–18 L gaseous mixture of 10% F_2 in nitrogen (volume percent) was added at a rate of 150 ml/min to the solution. Then the insoluble solid was removed by filtration. The filtrate was evaporated under vacuum, residues were washed with water, and a yellow solid was obtained. Recrystallization in ethanol afforded a white crystal **2**.

Method B: The mixture of **1** (19.6 mmol) and 2% sodium hydroxide aqueous solution (350 ml, 175 mmol) was stirred for 20 min, the precipitate was filtered and the solid (compound **5**) was air-dried. The mixture of compound **5** and acetonitrile (150 ml) was stirred and cooled to −30 °C. A 5–8 L gaseous mixture of 10% F_2 in nitrogen (volume percent) was introduced at a rate of 150 ml/min to the solution. The insoluble solid was removed by filtration. The filtrate was evaporated under vacuum and washed with water, a yellow solid was obtained. After recrystallization in 8:1 ethanol/acetonitrile a white crystal **2** was afforded.

4.2.1. N-fluoro-p-fluorobenzenesulfonimide (2a)

White crystal, m.p. 114.8–116.0 °C. IR (cm^{-1}, KBr): ν 3112, 3071, 1589, 1492, 1191, 839, 784; ^1H NMR ($CDCl_3$, 400 MHz) δ: 8.08–8.05 (m, 4H), 7.33–7.29 (m, 4H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −36.42 (s, 1F), −98.36 (s, 2F); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 168.53 (d, J = 259 Hz), 133.12 (d, J = 10.3 Hz), 130.46, 117.24 (d, J = 23.1 Hz); HRMS (EI): calculated for $C_{12}H_8F_3NO_4S_2$: 350.9847; found: 350.9850.

4.2.2. N-fluoro-p-fluorobenzenesulfonimide (2b)

White crystal, m.p. 142.8–143.9 °C. IR (cm^{-1}, KBr): ν 3103, 1574, 1474, 1200, 833, 761; ^1H NMR ($CDCl_3$, 400 MHz) δ: 7.97–7.94 (d, J = 8.8 Hz, 4H), 7.61–7.59 (d, J = 8.8 Hz, 4H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −36.43 (s, 1F); ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 143.25, 132.88, 131.24, 129.97; HRMS (EI): calculated for $C_{12}H_8Cl_2FNO_4S_2$: 382.9256; found: 382.9260.

4.2.3. p-Chlorobenzene-1-sulfonyl fluoride (3b)

White crystal, m.p. 38.4–39.8 °C, yield: 55.8%. IR (cm^{-1}, KBr): ν 3099, 1574, 1492, 1214, 832, 787; ^1H NMR ($CDCl_3$, 400 MHz) δ: 7.97–7.95 (d, J = 8.4 Hz, 2H), 7.63–7.61 (d, J = 8.4 Hz, 2H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: 66.45 (s, 1F); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 142.68, 133.25, 130.12, 129.87; HRMS (EI): calculated for $C_6H_4ClFO_2S$: 193.9605; found: 193.9606.

4.2.4. N-fluoro-p-bromobenzenesulfonimide (2c)

White crystal, m.p. 181.8–182.9 °C. IR (cm^{-1}, KBr): ν 3112, 3071, 1589, 1492, 1191, 839, 784; ^1H NMR ($CDCl_3$, 400 MHz) δ: 7.87∼7.85 (d, J = 8.8 Hz, 4H), 7.78–7.75 (d, J = 8.8 Hz, 4H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −36.46 (s, 1F); ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 133.40, 132.98, 132.03, 131.17; HRMS (EI): calculated for $C_{12}H_8Br_2FNO_4S_2$: 470.8246; found: 470.8252.

4.2.5. 4-Bromobenzene-1-sulfonyl fluoride (3c)

White crystal, m.p. 58.1–59.8 °C. IR (cm^{-1}, KBr): ν 3096, 1574, 1492, 1212, 827, 783; ^1H NMR ($CDCl_3$, 400 MHz) δ: 7.89–7.87 (d, J = 8.4 Hz, 2H), 7.80–7.78 (d, J = 8.4 Hz, 2H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: 66.36 (s, 1F); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 133.37, 133.12, 131.31, 129.83; HRMS (EI): calculated for $C_6H_4BrFO_2S$: 239.9079; found: 239.9079.

4.2.6. N-fluoro-p-bromo-benzenesulfonimide (4c)

White crystal, m.p. 88.6–89.7 °C. IR (cm^{-1}, KBr): ν 3096, 1574, 1492, 1212, 827, 783; ^1H NMR ($CDCl_3$, 400 MHz) δ: 8.63–8.49 (d, J = 52.8 Hz, 1H), 7.87–7.85 (d, J = 8.8 Hz, 2H), 7.78–7.76 (d, J = 8.8 Hz, 2H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −89.69 (s, 1F); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 133.34, 132.98, 130.89, 130.51; HRMS (EI): calculated for $C_6H_5BrFNO_2S$: 254.9188; found: 254.9188.

4.2.7. N-fluoro-p-trifluoromethylbenzenesulfonimide (2d)

White crystal, m.p. 127.9–128.6 °C. IR (cm^{-1}, KBr): ν 3113, 3062, 2925, 1413, 1395, 1133, 851, 787; ^1H NMR ($CDCl_3$, 400 MHz) δ: 8.18–8.16 (d, J = 8.4 Hz, 4H), 7.91–7.89 (d, J = 8.4 Hz, 4H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −35.99 (s, 1F), −63.50 (s, 6F); ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 137.96 (dd, J_1 = 30.4 Hz, J_2 = 63.9 Hz), 130.45, 126.78 (d, J = 3.7 Hz), 124.09, 121.37; HRMS (EI) calculated for $C_{14}H_8F_7NO_4S_2$: 450.9783; found: 450.9790.

4.2.8. N-fluoro-p-trifluoromethoxylbenzenesulfonimide (2e)

White crystal, m.p. 81.5–82.4 °C. IR (cm^{-1}, KBr): ν 3110, 3065, 1590, 1491, 1199, 857, 829; ^1H NMR ($CDCl_3$, 400 MHz) δ: 8.10–8.08 (d, J = 8.8 Hz, 4H), 7.44–7.42 (d, J = 8.8 Hz, 4H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −36.23 (s, 1F), −57.65 (s, 6F); ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 154.71, 132.35, 121.41, 120.82, 118.82; HRMS (EI): calculated for $C_{14}H_8F_7NO_6S_2$: 482.9681; found: 482.9685.

4.2.9. N-fluoro-p-t-butylbenzenesulfonimide (2f)

White crystal, m.p. 149.5–150.3 °C. IR (cm^{-1}, KBr): ν 3102, 2967, 1591, 1374, 1185, 840, 800; ^1H NMR ($CDCl_3$, 400 MHz) δ: 7.96–7.94 (d, J = 8.8 Hz, 4H), 7.62–7.60 (d, J = 7.2, 4H), 1.36 (s, 18H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −37.6 (s, 1F); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 160.24, 131.74, 129.83, 126.51, 35.58, 30.97; HRMS (EI): calculated for $C_{20}H_{26}FNO_4S_2$: 427.1287; found: 427.1283.

4.2.10. N-fluoro-p-methylsulfonimide (2g)

White crystal, m.p. 113.1–113.8 °C. IR (cm^{-1}, KBr): ν 3096, 3069, 2925, 1593, 1489, 1192, 814, 780; ^1H NMR ($CDCl_3$, 400 MHz) δ: 7.90–7.88 (d, J = 8.4 Hz, 4H), 7.40–7.38 (d, J = 8.4 Hz, 4H), 2.48 (s, 6H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −37.629 (s, 1F); ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 147.44, 131.70, 130.08, 129.92, 21.93; HRMS (EI): calculated for $C_{14}H_{14}FNO_4S_2$: 343.0348; found: 343.0349.

4.2.11. N-fluoro-p-methoxyphenylsulfonimide (2h)

White crystal, m.p. 138.2–138.8 °C. IR (cm^{-1}, KBr): ν 3121, 2976, 2948, 1592, 1498, 1199, 809, 785; ^1HNMR ($CDCl_3$, 400 MHz) δ: 7.94–7.92 (d, J = 9.2 Hz, 4H), 7.04–7.02 (d, J = 8.8 Hz, 4H), 3.91 (s, 6H); ^{19}F NMR ($CDCl_3$, 376 MHz) δ: −37.59 (s, 1F); ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 165.51, 132.42, 125.65, 114.67, 55.91; HRMS (EI): calculated for $C_{14}H_{14}FNO_6S_2$: 375.0247; found: 375.0245.

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