Journal of Fluorine Chemistry 131 (2010) 587–591

Contents lists available at ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

Regiochemistry of nucleophilic substitution of 4-phenylsulfonyl tetrafluoropyridine with unequal bidentate nucleophiles

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ARTICLE INFO

Article history:
Received 20 December 2009
Received in revised form 11 January 2010
Accepted 13 January 2010
Available online 22 January 2010

Keywords:
Pentafluoropyridine
Heterocycle
Multi-functional
Synthesis
Ring closure
Bidentate

ABSTRACT

The regiochemistry of nucleophilic substitution of 4-phenylsulfonyl tetrafluoropyridine with unequal bidentate nucleophiles was investigated. The first nucleophilic substitution occurs at the 2-position of the pyridine ring by nitrogen nucleophile site (secondary or primary amine) followed by intermolecular ring closure at the geometrically accessible 3-position of the pyridine ring (by S, O and N nucleophiles). From this investigation, difluorinated tetrahydropyrido[3,4-b][1,4]oxazine, thiazine and pyrazine scaffolds were synthesized very readily by a one-pot annelation reaction of 4-phenylsulfonyl tetrafluoropyridine with appropriate unequal bidentate nucleophiles.

1. Introduction

Synthesis of polyfunctional heterocyclic fused ring systems with low molecular weight are important in life science industries [1,2]. Pentafluoropyridine has attracted considerable interest due to its synthetic utility. Various multi-functional pyridine derivatives and construction of new heterocyclic and macrocyclic systems could be accessed from simple reaction conditions [3–10]. These include reaction of various bifunctional nucleophiles with pentafluoropyridine. All five fluorine atoms in pentafluoropyridine may be substituted by an appropriate nucleophile due to its highly electron efficient aromatic ring system. The site-reactivity order of pentafluoropyridine is well known [11–13] that, the order of activation toward nucleophilic attack follows the sequence 4-fluorine > 2-fluorine > 3-fluorine. Reactions of pentafluoropyridine with various nucleophiles are summarized and discussed in detail [14]. Representative examples of these reactions are given in Scheme 1.

Systematic exploitation of perfluoroheteroaromatic compounds with different susceptibilities due to different positioning can be used as a tool for combinatorial synthesis of other compounds. Chambers and co-workers recently demonstrated the feasibility of this concept. Further differentiation of its reactivity into hard and soft nucleophiles was achieved by partial replacement of fluorine by bromine in pentafluoropyridine [15] (Scheme 2).

There are several papers concerning the reactions involving equal bidentate nucleophiles with pentafluoropyridine derivatives [4]. Reaction of 4-phenylsulfonyl tetrafluoropyridine **2** with unequal bidentate nucleophiles has not been described previously in the literature. In this paper, we will describe our initial investigations on the regiochemistry nucleophilic substitution of **2** with a various unequal bidentate nucleophiles. We further develop our general annelation strategy to the synthesis of [6,6] and [6,6,6] ring fused bi- or tricyclic systems.

2. Results and discussion

Reaction of pentafluoropyridine **1** with sodium phenylsulfinate led to 4-phenylsulfonyl tetrafluoropyridine **2** following a literature procedure (Scheme 3) [16].

The phenylsulfonyl group is strong electron withdrawing group that helps to maintain the reactivity of pyridine ring toward further nucleophilic substitution processes. This allows annelation and further functionalization to proceed. Annelation processes involving the reaction between **2** and unequal binucleophiles in the presence of sodium bicarbonate and also in diluted acetonitrile solution to minimize intermolecular reaction were studied (Table 1). Unequal binucleophiles reacted efficiently with **2** to give tetrahydropyrido[2,3-b]oxazine, thiazine or pyrazine systems

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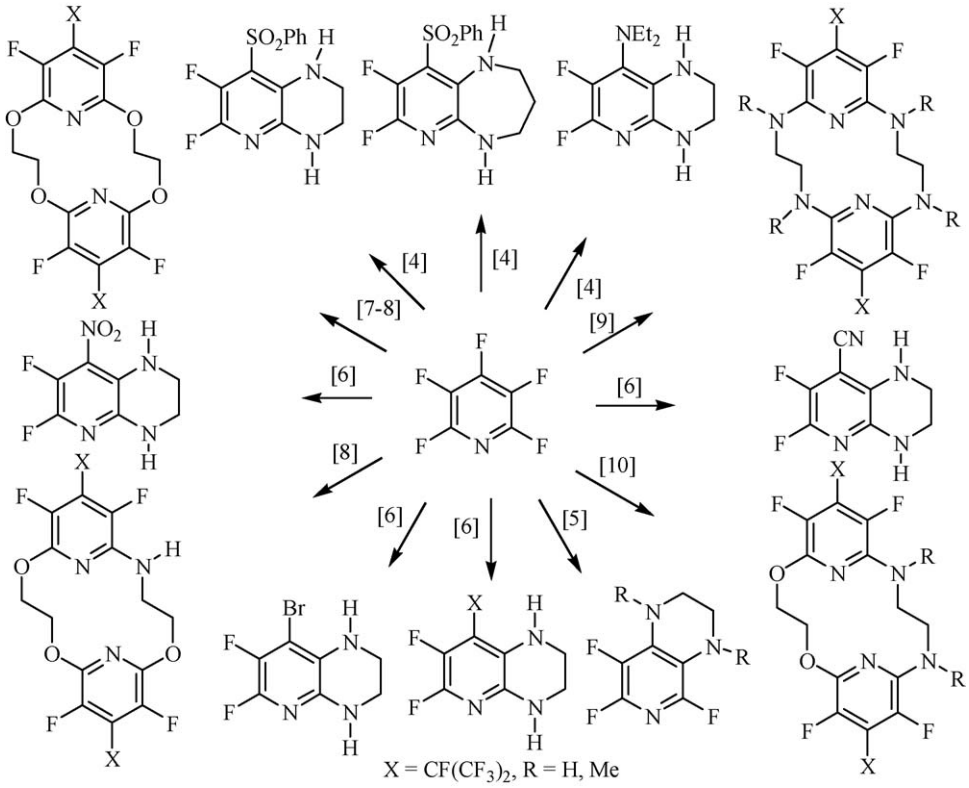
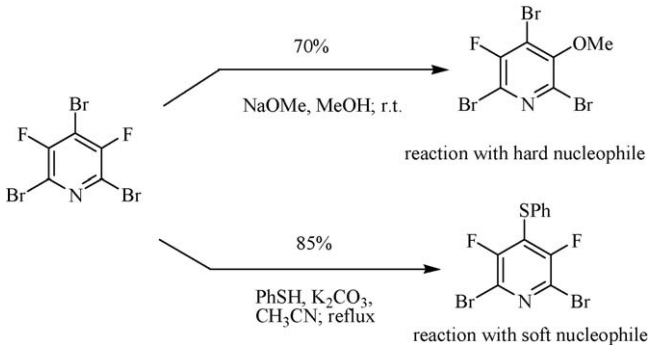
doi:10.1016/j.jfluchem.2010.01.006



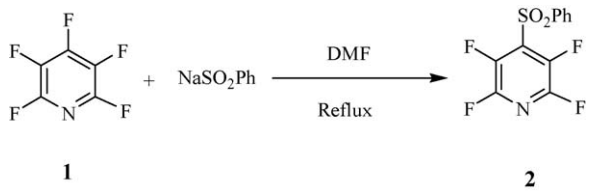
Scheme 1. Reactions on pentafluoropyridine.



Scheme 2. Reaction of pentahalopyridine with hard and soft nucleophiles.

by substitution at the 2-position of the pyridine ring followed by intermolecular ring closure at the geometrically accessible 3-position (Table 1). The regioselectivity of nucleophilic substitution of **2** with **3** may be explained by the high nucleophilicity of the secondary or primary amino groups and also the activating influences of the pyridine ring's nitrogen that significantly activates the *ortho* and *para* sites to itself.

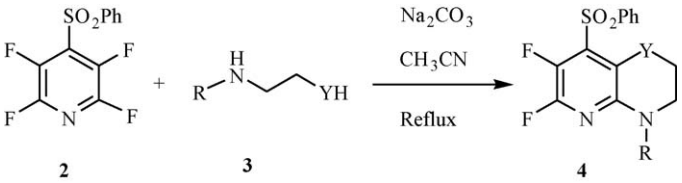
The reaction of **2** with unequal binucleophile **3a** bearing three nucleophilic sites, after refluxing in acetonitrile, gave a mixture of



Scheme 3. Reaction of pentafluoropyridine with sodium phenylsulfinate.

4a and **4b** in the ratio of 1.6:1 by ^{19}F NMR analysis of the reaction mixture, arising from the initial attack of the secondary or primary amine site at the 2-position of the pyridine ring and subsequent cyclization, respectively. Purification of **4a** and **4b** was achieved by column chromatography. Identification of **4a** was done by ^{19}F NMR analysis, in which the resonance attributed to fluorine located *ortho* to ring nitrogen have a chemical shift of −92.8 ppm similar to the shift observed for the **4c** and **4f**. The corresponding resonance for F-6 in **4b** occurs at −108.3 ppm similar to the analogous system **4d**, in which F-6 was adjacent to the NH group (−105.7 ppm). The major product **4a** was most likely formed from the initial attack of the secondary amine site, reflecting the higher nucleophilicity of the secondary amines over the primary systems. The [6,6] fused ring systems, **4c** and **4d**, were synthesized from the reaction **2** with diethanolamine **3b** and ethanolamine **3c**, respectively (Table 1). The reaction of **2** with diethylene triamine **3d** bearing three nucleophilic sites gave a mixture of **4e**, **4f** and **4g** in the ratio of 10:1.4:1 by ^{19}F NMR analysis of the reaction mixture, arising from initial attack of the secondary or primary amine sites at the 2-position of the pyridine ring and subsequent cyclization, respectively. Purification of **4e**, **4f** and **4g** was achieved by column chromatography. Identification of **4e** followed from ^{19}F NMR analysis in which the resonance attributed to fluorine located *ortho* to ring nitrogen (F-6) had a chemical shift of −108.4 ppm and the resonance attributed to fluorine located *meta* to ring nitrogen (F-7) had a chemical shift of −157.4 ppm, similar to shift observed for the **4d** in which F-6 is adjacent to the NH group (−105.7 ppm). The corresponding resonances for F-6 and F-7 in **4f** occur at −93.4 and −154.3 ppm, respectively, similar to the analogous system **4c** and **4a**. In **4g**, the resonance attributed to fluorine located *ortho* to ring nitrogen (F-6) had a chemical shift of −103.9 ppm similar to the shift observed for the **4d** and**4e** in which F-6 is adjacent to the NH group and the resonance attributed to fluorine located *meta* to ring nitrogen (F-7) had a chemical shift of −182.1 ppm. The major product **4e** is most likely formed from the initial attack of the

Table 1
Nucleophilic substitution of **2** with **3**



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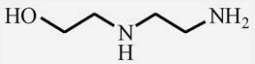
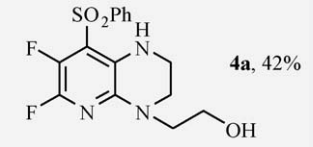
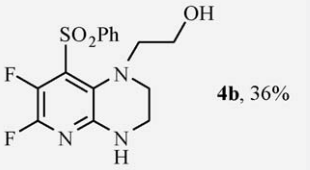
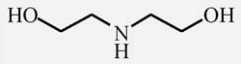
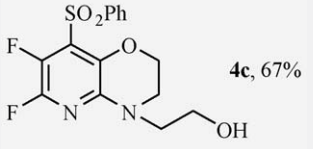
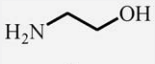
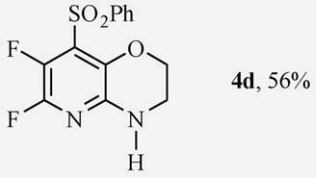
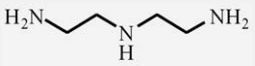
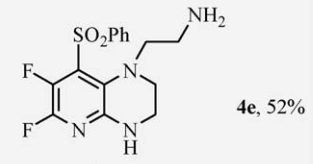
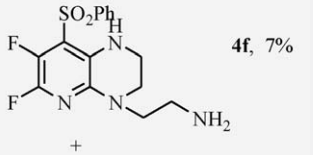
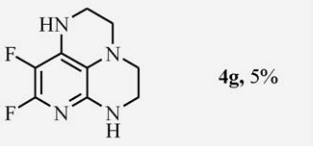
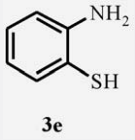
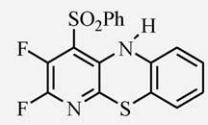
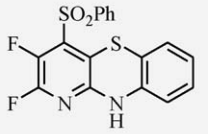
Entry	Binucleophile	Product(s)
1	 **3a**	 **4a**, 42% +  **4b**, 36%
2	 **3b**	 **4c**, 67%
3	 **3c**	 **4d**, 56%
4	 **3d**	 **4e**, 52% +  **4f**, 7% +  **4g**, 5%

Table 1 (*Continued*)

Entry	Binucleophile	Product(s)
5	 **3e**	 **4h**, 46% +  **4i**, 4%

primary amine site, reflecting the less steric hindrance around the primary amine over the secondary amine and then intramolecular cyclization. A small amount (5%) of **4g** formed from the intramolecular cyclization of **4e**.

The [6,6,6] fused ring system **4h** was synthesized from the reaction of **2** with 2-aminothiophenol **3e** (Table 1). Cyclization processes could also be affected by microwave heating, and in a much shorter reaction time, a similar yield of **4h** was obtained from **2** and **3e**. The acceleration of reactions by microwave results from material-wave interactions leading to thermal effects connected to the intervention of "hot spots" (localized microscopic high temperatures) and specific (non-thermal) effects [17]. A small amount (4%) of **4i** product was identified by ^{19}F NMR and GC–MS analysis but could not be isolated. Purification of **4h** was achieved by recrystallization of the crude product mixture from n-hexane/ethyl acetate. Identification of **4h** and **4i** followed from ^{19}F NMR analysis in which the resonance attributed to fluorine located at *ortho* to ring nitrogen had a chemical shift of −91.19 and −106.13 ppm for **4h** and **4i**, respectively, similar to the shifts observed for the analogous systems.

The structures of all [6,6] and [6,6,6]-fused ring systems were confirmed by ^{13}C NMR analysis. For example, comparisons of the published ^{13}C NMR data of 6,7-difluoro-1,4-dimethyl-1,2,3,4-tetrahydropyrido[2,3-b]pyrazine-8-carbonitrile of which structure was proved unambiguously by X-ray crystallography [6] (C-2*b* 142.0; C-3b 133.5 ppm) and other similar compounds [4,5] with **4a** (C-2*b* 146.1; C-3*b* 133.1 ppm) confirm the structure of **4a**.

3. Conclusion

In conclusion, we showed that 4-phenylsulfonyl tetrafluoropyridine can successfully react with a variety of unequal bidentate nucleophiles. The regioselectivity of nucleophilic substitution in this process may be explained by the high nucleophilicity of the secondary or primary amino groups and by the activating influence of pyridine ring nitrogen that significantly activates the *ortho* and *para* sites to itself. In contrast, the aromatic unequal bidentate nucleophiles such as 2-aminothiophenol, the major product is most likely formed from the initial attack of the S-nucleophile and subsequent cyclization. From this investigation, it was revealed that difluorinated tetrahydropyrido[3,4-b][1,4]oxazine, thiazine and pyrazine scaffolds were synthesized very readily by a one-pot annelation reaction.

4. Experimental

All solvents were dried using the literature procedures and distilled before use. The reactions were carried out under an atmosphere of argon unless otherwise specified. The elemental analyses for C, H, and N were performed using Heraeus CHN-O-Rapid analyzer. The ^{13}C NMR spectra were recorded at 75 or 125 MHz. The ^{19}F NMR spectra were recorded at 470 MHz. In the ^{19}F NMR spectra, upfield shifts were quoted as negative and referenced to $CFCl_3$. Mass spectra were taken by a Micromass Platform II: EI mode (70 eV). Medium pressure ('flash') column chromatography was performed using silica (Merck #60). Silica plates (Merck) were used for TLC analysis.

4.1. General procedure for preparation of 6,7-difluoro-2,3-dihydro-8-(phenylsulfonyl)pyrido[3,2-b][1,4]oxazin or pyrazine system

Sodium carbonate (15 mmol) was added to the mixture of **3** (5 mmol) in acetonitrile (150 mL) under argon. Then **2** (2.5 mmol) was added and the resulting solution was refluxed at 95 °C for 2 d. The reaction mixture was cooled to room temperature and the solvent was evaporated. The reaction mixture was poured onto 0.2 M hydrochloric acid (50 mL) and then extracted with dichloromethane. The solvent was evaporated to yield the crude product, which was then purified by recrystallization or column chromatography on silica gel.

4.1.1. 2-(6,7-Difluoro-2,3-dihydro-8-(phenylsulfonyl)pyrido-[2,3-b]pyrazin-4(1H)-yl)ethanol 4a

Sodium carbonate (1.26 g, 15 mmol), 2-(2-aminoethylamino)ethanol **3a** (0.52 g, 5 mmol), 4-phenylsulfonyl tetrafluoropyridine **2** (0.72 g, 2.5 mmol) and acetonitrile (150 mL) gave an oily product that was purified by column chromatography on silica gel (ethyl acetate/hexane, 1:3) gave 2-(6,7-difluoro-2,3-dihydro-8-(phenylsulfonyl)pyrido-[2,3-*b*]pyrazin-4(1H)-yl)ethanol **4a** and 2-(6,7-difluoro-3,4-dihydro-8-(phenylsulfonyl)pyrido[2,3-*b*]pyrazin-1(2H)-yl)ethanol **4b**. **4a**: 0.36 g (42%), yellow solid; mp: 152–154 °C. ^{13}C NMR ($CDCl_3$): δ (ppm) 35.48 (s, CH_2NH_2), 41.68 (s, CH_2N), 58.62 (s, CH_2N), 59.31 (s, CH_2O), 124.96 (m, C-8), 127.85 (s, Ar-C), 129.01 (s, Ar-C), 133.12 (d, $^3J_{CF}$ = 7.3 Hz, C-3*b*), 132.77 (dd, $^1J_{CF}$ = 284.2 Hz, $^2J_{CF}$ = 30.7 Hz, C-7), 134.21 (s, Ar-C), 141.13 (s, Ar-C), 146.17 (d, $^3J_{CF}$ = 16.2 Hz, C-2*b*), 147.30 (dd, $^1J_{CF}$ = 251.6 Hz, $^2J_{CF}$ = 16.5 Hz, C-6). ^{19}F NMR ($CDCl_3$): δ (ppm) −154.08 (d, $^3J_{FF}$ = 27.9 Hz, 1F, F-7), −92.87 (d, $^3J_{FF}$ = 27.9 Hz, 1F, F-6). MS (EI), *m*/*z* (%) = 355 (M^+, 100), 310 (25), 214 (30). Anal. Calcd for $C_{15}H_{15}N_3F_2O_3S$: C, 50.7; H, 4.2; N, 11.8. Found: C, 50.8; H, 4.3; N, 11.8. **4b**: 0.31 g (36%), yellow solid; mp 113–116 °C. ^{13}C NMR ($CDCl_3$): δ (ppm) 38.93 (s, CH_2OH), 46.70 (s, CH_2N), 52.31 (s, CH_2N), 60.70 (s, CH_2N), 127.17 (s, Ar-C), 127.18 (s, Ar-C), 129.30 (s, Ar-C), 115.75 (d, $^3J_{CF}$ = 15.5 Hz, C-3*b*), 131.20 (dd, $^2J_{CF}$ = 18.2 Hz, $^3J_{CF}$ = 2.4 Hz, C-8), 134.21 (s, Ar-C), 140.69 (dd, $^1J_{CF}$ = 298.4 Hz, $^2J_{CF}$ = 23.0 Hz, C-7), 141.50 (s, Ar-C), 128.67 (d, $^3J_{CF}$ = 2.9 Hz, C-2*b*), 140.67 (dd, $^1J_{CF}$ = 240.1 Hz, $^2J_{CF}$ = 16.6 Hz, C-6). ^{19}F NMR ($CDCl_3$): δ (ppm) −159.83 (d, $^3J_{FF}$ = 25.5 Hz, 1F, F-7), −108.33 (d,

$^3J_{FF}$ = 25.5 Hz, 1F, F-6). MS (EI), *m*/*z* (%) = 355 (M^+, 100), 310 (20), 214 (40). Anal. Calcd for $C_{15}H_{15}N_3F_2O_3S$: C, 50.7; H, 4.2; N, 11.8. Found: C, 50.8; H, 4.3; N, 11.8.

4.1.2. 2-(6,7-Difluoro-2,3-dihydro-8-(phenylsulfonyl)pyrido-[3,2-b][1,4]oxazin-4-yl)ethanol **4c**

Sodium carbonate (1.26 g, 15 mmol), diethanolamine **3b** (0.52 g, 5 mmol), 4-phenylsulfonyl tetrafluoropyridine **2** (0.72 g, 2.5 mmol) and acetonitrile (150 mL) gave an oily product that was purified by column chromatography on silica gel (ethyl acetate/hexane, 1:3) gave 2-(6,7-difluoro-2,3-dihydro-8-(phenylsulfonyl)-pyrido-[3,2-b][1,4]oxazin4-yl)ethanol **4c**, 0.59 g (67%), yellow solid; mp 123–124 °C. ^{13}C NMR ($CDCl_3$): δ (ppm) 46.43 (s, CH_2N), 51.53 (s, CH_2N), 60.64 (s, CH_2OH), 64.27 (s, CH_2O), 128.11 (s, Ar-C), 129.03 (s, Ar-C), 129.19 (s, Ar-C), 129.52 (d, $^3J_{CF}$ = 22.9 Hz, C-3*b*), 131.37 (dd, $^1J_{CF}$ = 256.0 Hz, $^2J_{CF}$ = 32.0 Hz, C-7), 133.98 (s, Ar-C), 134.09 (d, $^3J_{CF}$ = 23.0 Hz, C-2*b*), 141.00 (s, Ar-C), 141.14 (dd, $^2J_{CF}$ = 7.4 Hz, $^3J_{CF}$ = 3.5 Hz, C-8), 144.35 (dd, $^1J_{CF}$ = 234.1 Hz, $^2J_{CF}$ = 16.2 Hz, C-6). ^{19}F NMR ($CDCl_3$): δ (ppm) −160.08 (d, $^3J_{FF}$ = 25.4 Hz, 1F, F-7), −97.13 (d, $^3J_{FF}$ = 25.4 Hz, 1F, F-6). MS (EI): *m*/*z* (%) = 356 (M^+, 15), 311 (30), 215 (50). Anal. Calcd for $C_{15}H_{14}N_2F_2O_4S$: C, 50.6; H, 4.0; N, 7.7. Found: C, 50.5; H, 3.9; N, 7.8.

4.1.3. 6,7-Difluoro-3,4-dihydro-8-(phenylsulfonyl)-2H-pyrido-[3,2-b][1,4]oxazine **4d**

Sodium carbonate (1.26 g, 15 mmol), 2-aminoethanol **3c** (0.30 g, 5 mmol), 4-phenylsulfonyl tetrafluoropyridine **2** (0.72 g, 2.5 mmol) and acetonitrile (150 mL) gave an oily product that was purified by column chromatography on silica gel (ethyl acetate/hexane, 1:3) gave 6,7-difluoro-3,4-dihydro-8-(phenylsulfonyl)-2H-pyrido[3,2-b][1,4]oxazine **4d**, 0.43 g (56%), brown oil; ^{13}C NMR ($CDCl_3$): δ (ppm) 38.80 (s, CH_2NH), 64.76 (s, CH_2O), 119.90 (d, $^3J_{CF}$ = 15.2 Hz, C-3*b*), 127.43 (s, Ar-C), 127.44 (s, Ar-C), 127.95 (d, $^3J_{CF}$ = 6.5 Hz, C-8), 129.41 (s, Ar-C), 134.60 (s, Ar-C), 136.28 (dd, $^1J_{CF}$ = 257.4 Hz, $^2J_{CF}$ = 29.2 Hz, C-7), 139.54 (dd, $^1J_{CF}$ = 229.3 Hz, $^2J_{CF}$ = 19.1 Hz, C-6), 140.92 (s, Ar-C), 142.80 (d, $^3J_{CF}$ = 13.0 Hz, C-2*b*). ^{19}F NMR ($CDCl_3$): δ (ppm) −147.60 (d, $^3J_{FF}$ = 24.6 Hz, 1F, F-7), −105.68 (d, $^3J_{FF}$ = 24.6 Hz, 1F, F-6). MS (EI): *m*/*z* (%) = 312 (M^+, 43), 311 (100), 171 (35). Anal. Calcd for $C_{13}H_{10}N_2F_2O_3S$: C, 50.0; H, 3.2; N, 9.0. Found: C, 50.1; H, 3.2; N, 8.9.

4.1.4. 2-(6,7-Difluoro-3,4-dihydro-8-(phenylsulfonyl)pyrido[2,3-b]pyrazin-1(2H)-yl)ethanamine **4e**

Sodium carbonate (1.26 g, 15 mmol), diethylene triamine **3d** (0.51 g, 5 mmol), 4-phenylsulfonyl tetrafluoropyridine **2** (0.72 g, 2.5 mmol) and acetonitrile (150 mL) gave an oily product that was purified by column chromatography on silica gel (ethyl acetate/hexane, 1:4) gave 2-(6,7-difluoro-2,3-dihydro-8-(phenylsulfonyl)-pyrido[2,3-b]pyrazin-4(1H)-yl)ethanamine **4e**, 0.72 g (52%), yellow solid; mp 143–145 °C, δ (ppm) 40.46 (s, CH_2NH_2), 40.83 (s, CH_2N), 47.15 (s, CH_2N), 47.84 (s, CH_2N), 127.31 (s, Ar-C), 127.41 (s, Ar-C), 128.71 (s, Ar-C), 130.37 (d, $^3J_{CF}$ = 20.2 Hz, C-3*b*), 126.81 (dd, $^1J_{CF}$ = 232.1 Hz, $^2J_{CF}$ = 31.5 Hz, C-7), 134.82, (s, Ar-C), 135.75 (s, Ar-C), 136.31 (d, $^3J_{CF}$ = 18.0 Hz, C-2*b*), 141.21 (s, Ar-C), 142.01 (dd, $^2J_{CF}$ = 8.1 Hz, $^3J_{CF}$ = 3.1 Hz, C-8). (dd, $^1J_{CF}$ = 220.8 Hz, $^2J_{CF}$ = 11.6 Hz, C-6), 159.85. ^{19}F NMR ($CDCl_3$): δ (ppm) −157.40 (d, $^3J_{FF}$ = 25.9 Hz, 1F, F-7), −108.42 (d, $^3J_{FF}$ = 25.9 Hz, 1F, F-6). MS (EI): *m*/*z* (%) = 354 (M^+, 40), 311 (100), 214 (30), 171 (25). Anal. Calcd for $C_{15}H_{16}N_4F_2O_2S$: C, 50.8; H, 4.5; N, 15.8. Found: C, 50.9; H, 4.6; N, 15.9.

4.1.5. 4-Benzenesulfonyl-2,3-difluoro-5H-benzo[b]pyrido[3,2-e][1,4]thiazine **4h**

Sodium carbonate (1.26 g, 15 mmol), 2-aminobenzenethiol **3e** (0.62 g, 5 mmol), 4-phenylsulfonyl tetrafluoropyridine **2** (0.72 g, 2.5 mmol) and acetonitrile (150 mL) gave an oily product that was purified by column chromatography on silica gel (ethyl acetate/hexane, 1:5) gave 4-benzenesulfonyl-2,3-difluoro-5H-benzo[b]-pyrido[3,2-e][1,4]thiazine **4h**, 0.63 g (46%), brown solid; mp 137–139 °C, ^{13}C NMR ($CDCl_3$): δ (ppm) 110.10 (s, Ar-C), 115.69 (s, Ar-C), 119.03 (s, Ar-C), 127.44 (s, Ar-C), 130.55 (m, C-3*b*), 132.32 (s, Ar-C), 132.56 (m, C-8), 137.25 (s, Ar-C), 138.41 (s, Ar-C), 139.72 (s, Ar-C), 139.9 (dd, $^1J_{CF}$ = 272.8 Hz, $^2J_{CF}$ = 26.4 Hz, C-7), 140.92 (s, Ar-C), 142.26 (d, $^3J_{CF}$ = 12.7 Hz, C-2*b*), 144.37 (dd, $^1J_{CF}$ = 262.1 Hz, $^2J_{CF}$ = 17.5 Hz, C-6), 144.56 (s, Ar-C). ^{19}F NMR ($CDCl_3$): δ (ppm) −138.62 (d, $^3J_{FF}$ = 24.9 Hz, 1F, F-7), −91.19 (d, $^3J_{FF}$ = 24.9 Hz, 1F, F-6). MS (EI): *m*/*z* (%) = 376 (M^+, 51), 375 (60), 236 (35), 300 (41), 255 (20), 156 (35). Anal. Calcd for $C_{17}H_{10}N_2F_2O_2S_2$: C, 54.2; H, 2.7; N, 7.4. Found: C, 54.3; H, 2.6; N, 7.5.

Acknowledgement

The authors wish to thank Rafsanjan Vali-e-Asr University (Rafsanjan, Iran) for the partial support of this work.

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