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Synthesis and New Synthetic Utility of some (Hetero)aryl Azido Compounds under Thermal and Ultrasonic Irradiation

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Some aryl and heteroaryl azido compounds were synthesized from the reaction of the corresponding fluoro and chloro compounds with sodium azide in DMF as solvent and under various reaction conditions especially ultrasonic irradiation. Reaction of aromatic and heteroaromatic azido compound with thioacetic acid in the presence of sodium hydrogen carbonate and in methanol as solvent led to the different products. Further intermolecular nucleophilic aromatic substitution reaction of *N*-(perfluoropyridin4-yl)acetamid allow the regioselective synthesis of 2,6,7-trifluoro-2-methyloxazolo[4,5-c]pyridine. The structures of all compounds were confirmed by IR, ^{1}H NMR and ^{13}C NMR spectroscopy as well as elemental analysis.

Keywords: (Hetero)aryl halides, (Hetero)aryl azide, Ultrasonic irradiation, Thioacetic acid, Sodium azide

INTRODUCTION

Alkyl and aryl azides are important and valuable intermediates in organic synthesis and have been frequently used for the synthesis of heterocycles such as triazoles and tetrazoles *via* cyclo addition reaction [1-3]. They are used in the synthesis of anilines, *N*-alkylated anilines and as precursors for nitrene intermediates [4]. These compounds are also used as blowing agents and as functional groups in pharmaceuticals. Thus, for example, azidonucleosides attract international interest in the treatment of AIDS [4,5]. An important application of the photochemistry of organic azides is the photo affinity labeling of biopolymers [6,7].

Aryl and heteroaryl azides may be prepared through several different methods. The oldest methods are the reaction of diazonium salts with hydrazine [8,9] or *O*-benzylhydroxylamine hydrochloride [10]; *i.e.* the synthesis of azidothalidomide by the decomposition of corresponding diazonium salts [11]. They can also be prepared by the reaction of aromatic amines by triflyl azide [12].

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Nucleophilic aromatic substitution (S_NAr Reactions) of aryl and heteroaryl halides with metal azides afforded the corresponding azido compounds [13].

Numerous methods for the preparation of aryl azides with organometallic reagents have been developed [14-16]. The reaction of nitrosoarenes with hydrogen azide leads to aryl azides in good yields [17]. In the recent years, we have concentrated on the reaction of various nucleophiles with chloro and fluoro compounds [18-20]. Continuing our research in this area, we would like to report the synthesis of some azido and tetrazolo compounds from the reaction of corresponding halo compounds with sodium azide under ultrasonic irradiation as well as the reaction of azido compounds with thioacetic acid.

EXPERIMENTAL

All the solvents and starting materials were obtained commercially (Merck). Solvents were dried using the literature recommended procedures and distilled before use. The ultrasonic device used was an UP 400 S instrument from Dr. Hielscher GmbH. An S3 immersion horn emitting

24 kHz ultrasound at intensity levels tunable to maximum sonic power density of 460 W cm^{-2} was used. Sonication was carried out at 100% (maximum amplitude 210 lm). A 3 mm long sonotrode (maximum immerse depth of 90 mm) was immersed directly into the reaction mixture. ^{1}H NMR spectra were recorded at 300 MHz. ^{13}C NMR spectra were recorded at 75 MHz. ^{19}F NMR spectra were recorded at 282 MHz. TLC analysis was performed on silica gel TLC plates (Merck).

General Procedure for Synthesis of Azido Compounds (2)

Method a. Halo compound 1 (1 mmol) was added to a solution of sodium azid (1 mmol) in DMF (2 ml). The reaction mixture was stirred at room temperature for 1-24 h. The progress of the reaction was monitored by TLC. After completion, the mixture was diluted with water (10 ml) and extracted with chloroform (3 × 10 ml). The combined organic layer was dried over anhydrous $MgSO_4$ and concentrated to afford the pure (hetero)aryl azides 2.

Method b. halo compound 1 (1 mmol) was added to a solution of sodium azid (1 mmol) in DMF (2 ml). The reaction mixture was irradiated with ultrasound for 10-240 min. The progress of the reaction was monitored by TLC. After completion, the mixture was diluted with water (10 ml) and extracted with chloroform (3 × 10 ml). The combined organic layer was dried over anhydrous $MgSO_4$ and concentrated to afford the pure (hetero)aryl azides 2.

4-Azido-2,3,5,6-tetrafluoropyridine (2a). Oily liquid, IR (KBr) (υ_{max}, cm^{-1}) = 2134 (N_3), 1525 (C=N), 1505 (C=C), 754 (C-F). ^{19}F NMR (282 MHz, $CDCl_3$): δF -89.34 (m, 2F, F-2,6), -152.44 (m, 2F, F-3,5) ppm.

2-Azido-7-chloroquinoline (2c). White solid, m.p.: 114-118 °C IR (KBr) (υ_{max}, cm^{-1}) = 2128 (N_3), 1609 (C=N), 1597 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.79 (d, J_{HH} = 4.8 Hz, 1H, Ar-H), 8.02 (d, J_{HH} = 1.6 Hz, 1H, Ar-H), 7.93 (d, J_{HH} = 8.9Hz, 1H, Ar-H), 7.43 (dd, $^3J_{HH}$ = 9.0 Hz, $^4J_{HH}$ = 1.8 Hz, 1H, Ar-H), 7.08 (d, J_{HH} = 4.8 Hz, 1H, Ar-H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 151.2, 149.5, 146.2, 136.5, 128.1, 127.5, 123.7, 119.8, 108.6 ppm.

Tetrazolo[1,5-*a*]pyrazine (2e). White solid, m.p.: 85-88 °C, IR (KBr) (υ_{max}, cm^{-1}) = 3093 (C-H), 2217 (N_3), 1518 (C=N), 1466 (C=C) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 9.63 (s, 1H, Ar-H), 8.85 (d, J_{HH} = 4.6 Hz, 1H, Ar-H), 4.6 (d, J_{HH} = 4.6 Hz, 1H, Ar-H).

Tetrazolo[1,5-*a*]pyridine (2f). White solid, m.p.: 128-132 °C, IR (KBr) (υ_{max}, cm^{-1}) = 3104 (C-H), 1632 (C=N). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 9.31 (d, J_{HH} = 6.8 Hz, 1H, Ar-H), 8.21 (d, J_{HH} = 8.1 Hz, 1H, Ar-H), 7.86 (d, J_{HH} = 6.8 Hz, 1H, Ar-H), 7.44 (d, J_{HH} = 6.8 Hz, 1H, Ar-H) ppm. ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 148.1, 133.1, 126.4, 117.3, 115.1 ppm.

4-Chlorotetrazolo[1,5-*a*]quinoxaline (2h). White solid, m.p.: 250, IR (KBr) (υ_{max}, cm^{-1}) = 1645 (C=N), 1557 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.75 (d, J_{HH} = 4.8 Hz, 1H, Ar-H), 8.05 (t, J_{HH} = 6.1 Hz, 2H, Ar-H), 7.93 (d, J_{HH} = 5.6 Hz, 1H, Ar-H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 144.6, 140.4, 140.0, 131.7, 131.0, 127.9, 122.7, 117.7 ppm.

General Procedure for Reaction of Azido Compounds 2 with Thioacetic Acid

Method a. Azido compound 2 (1 mmol) was added to a solution of thioacetic acid (1.7 mmol) and sodium hydrogencarbonat (1.6 mmol) in methanol (3 ml). The reaction mixture was stirred at room temperature for 8-18 h. The progress of the reaction was monitored by TLC. After completion, the mixture was diluted with water (10 ml) and extracted with ethylacetate (4 × 10 ml). The combined organic layer was dried over anhydrous $MgSO_4$ and concentrated to afford the crude products 3 that was purified by recrystallization from *n*-hexane.

Method b. Azido compound (1 mmol) was added to a solution of thioacetic acid (1.7 mmol) and sodium hydrogencarbonat (1.6 mmol) in methanol (3 ml). The reaction mixture was irradiated with ultrasound for 30-280 min. The progress of the reaction was monitored by TLC. After completion, the mixture was diluted with water (10 ml) and extracted with chloroform (3 × 10 ml). The combined organic layer was dried over anhydrous $MgSO_4$ and concentrated to afford the crude products 3 that was purified by recrystallization from *n*-hexane.

N-(perfluoropyridin-4-yl)acetamide (3a). White solid, m.p.: 120-124 °C (Found: C, 40.2; H, 1.5; N, 13.3. $C_7H_4F_4N_2O$ requires: C, 40.4; H, 1.9; N, 13.5%). IR (KBr) (υ_{max}, cm^{-1}) = 3247 (NH), 1692 (C=O), 1643 (C=N), 1537 (C=C). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H 6.49 (s, 1H, NH), 2.15 (s, 3H, CH_3) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ_c 167.8 (C=O), 143.2 (dm, $^1J_{CF}$ = 243.7 Hz, C-2,6),

138.4 (m, C-4), 136.6 (dm, $^1J_{CF}$ = 263 Hz, C-3,5), 22.6 (s, CH_3) ppm. ^{19}F NMR (282 MHz, $CDCl_3$): δ_F -93.29 (m, 2F, F-2,6), -145.92 (m, 2F, F-3,5) ppm.

4-Amino-2,3,5,6-tetrachloropyridine (3b). White solid, m.p.: 212-217 °C; IR (KBr) (υ_{max}, cm^{-1}) = 3493 (NH_2), 1588 (C=C), 1536 (C=C). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.34 (s, 2H, NH_2) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ_c 150.8, 144.1, 11.5 ppm.

7-Chloroquinoline-4(1H)-thione (3c). White solid, m.p.: 183-186 °C, IR (KBr) (υ_{max}, cm^{-1}) = 3446 (NH), 1077 (C=S). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.61 (d, J_{HH} = 8.9 Hz, 1H, Ar-H), 7.85 (d, J_{HH} = 6.7 Hz, 1H, CH), 7.66 (s, 1H, Ar-C), 7.44 (dd, $^3J_{HH}$ = 8.9 Hz, $^4J_{HH}$ = 1.3 Hz, 1H, Ar-H), 7.26 (d, J_{HH} = 6.7 Hz, 1H, CH) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 192.4 (C=S), 137.1 (Ar-C), 136.4 (Ar-C), 134.2 (Ar-C), 130.9 (Ar-C), 130.7 (Ar-C), 125.7 (Ar-C), 124.6 (CH), 118.5 (CH) ppm.

N-(2,4-dinitrophenyl)acetamide (3d). White solid, m.p.: 108-112 °C (Found: C, 42.6; H, 2.8; N, 18.8. $C_8H_7N_3O_5$ requires: C, 42.7; H, 3.1; N, 18.7%). IR (KBr) (υ_{max}, cm^{-1}) = 3334 (NH), 1710 (C=O). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 9.56 (s, NH), 8.78 (d, $^4J_{HH}$ = 2.7 Hz, Ar-H), 8.15 (dd, $^3J_{HH}$ = 6.7 Hz, $^4J_{HH}$ = 2.7 Hz, Ar-H), 7.13 (d, $^3J_{HH}$ = 6.7 Hz, Ar-H), 1.96 (s, 3H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ_c 168.2 (C=O), 133.5, 132.0, 129.6, 128.6, 127.9, 127.6, 21.0 (CH_3) ppm.

Synthesis of 4,6,7-Trifluoro-2-methyloxazolo[5,4-c]pyridine (4a)

To a solution of *N*-(perfluoropyridin-4-yl)acetamide **3a** (0.2 g, 1 mmol) in MDF (3 ml) was added potassium carbonate (0.28 g, 2 mmol). The reaction mixture was stirred and heated at 100 °C for 48 h. The progress of the reaction was monitored by TLC. After completion, the mixture was diluted with water (10 ml) and extracted with ethylacetate (4 × 10 ml). The combined organic layer was dried over anhydrous $MgSO_4$ and concentrated to afford the crude product. Column chromatography of the crude product on silica gel using hexane:ethylacetate ratio (3:1) as eluent, gave 4,6,7-trifluoro-2-methyloxazolo[5,4-c]pyridine 4a as white solid; m.p.: 195-200 °C. (Found: C, 44.4; H, 1.4; N, 14.5. $C_7H_3F_3N_2O$ requires C, 44.7; H, 1.6; N, 14.9%); IR (KBr) (υ_{max}, cm^{-1}) = 2924 (CH), 1638 (C=N). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 2.18 (s, 3H, CH_3) ppm. ^{13}C NMR (75 MHz, DMSO-d_6): δ_c 168.4 (O-C=N), 142.1 (dm, $^1J_{CF}$ = 210 Hz, C-2), 138.2 (dm, $^1J_{CF}$ = 190 Hz, C-7), 134.4 (m, C-8), 133.4 (dm, $^1J_{CF}$ = 233 Hz, C-6), 127.6 (m, C-9) 14.4 (s, CH_3) ppm. ^{19}F NMR (282 MHz, $CDCl_3$): δ_F -92.33 (m, 1F, F-2), -138.27 (m, 1F, F-7), -149.02 (m, 1F, F-6) ppm.

RESULTS AND DISCUSSION

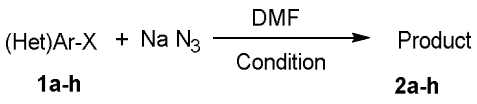
Reaction of pentafluoropyridine 1a with sodium azid in DMF at room temperature and under ultrasonic irradiation gave a single product, 4-azido-2,3,5,6-tetrafluoropyridine 2 in high yield and in short reaction time (Table 1, entry 1). Two resonances by ^{19}F NMR (-89.3 and -152.4), indicate displacement of fluorine atom attached to 4-position of the pyridine ring. With this encouraging result in hands we performed reaction of some chloro and fluoro aromatic and heteromatic compounds with sodium azide under ultrasonic irradiation (Table 1).

Perchloropyridine 1b, 4,7-dichloroquinoline 1c and 1-chloro-2,4-dinitrobenzene 1d in reaction with sodium azide under ultrasonic irradiation gives corresponding azido compound in short reaction time compared to stirring at room temperature. 2-Chloropyrazine 1e, 2-chloropyridine 1f, 2,6-difluoropyridine 1g and 2,3-dichloroquinoxaline 1h undergo a facile reaction with sodium azide to give corresponding tetrazolo compounds 2d-h which exist in dynamic equilibrium with (hetero)aryl azides.

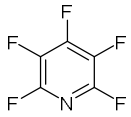
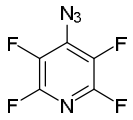
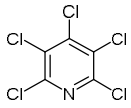
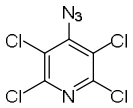
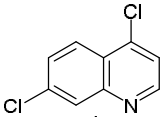
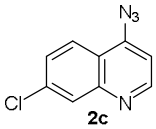
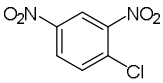
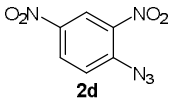
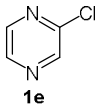
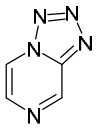
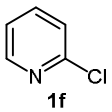
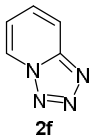
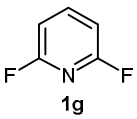
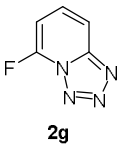
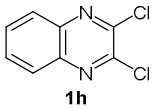
The (hetero)aryl azides derivatives are relatively reactive compounds and reactions of these substrates with thioacetic acid could, in principle, lead to acetamide derivatives 3. Reaction of azido compounds 2 with thioacetic acid were carried out, and the results are collected in Scheme 1.

Reaction of thioacetic acid with 4-azido-2,3,5,6-tetrafluoropyridine 2a in the presence of sodium bicarbonate and in methanol as solvent gave the desired *N*-(perfluoropyridin-4-yl)acetamide 3a in good yield after simple recrystallization of the crude product from *n*-hexane. This process could also be affected by ultrasonic irradiation, and in a much shorter reaction time, a similar yield of 3a was obtained from 2a and thioacetic acid. Identification of 3a was done by IR and NMR. The IR spectrum of 3a showed a broad absorption band at 3247 cm^{-1} for NH

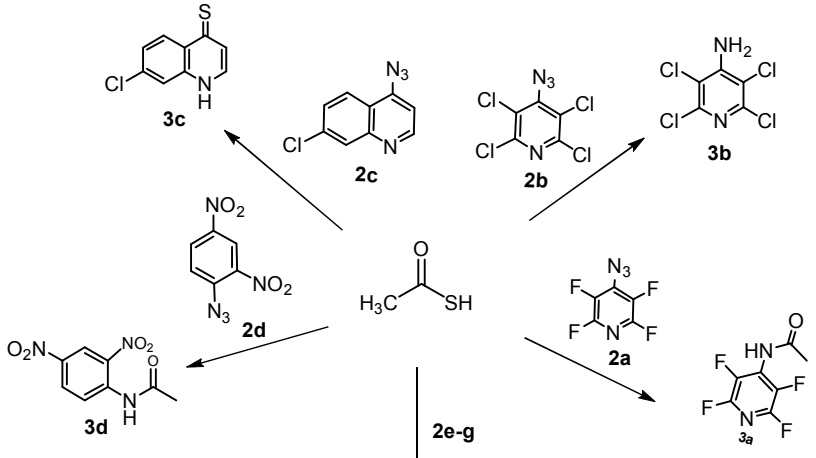
Table 1. Synthesis of Azido Compounds



1a-h **2a-h**

Entry	(Het)Ar-X	Product[a]	Yield (%) (Time (h))[b]	Yield (%) (Time (min))[c]
1	 **1a**	 **2a**	75 (6)	85 (15)
2	 **1b**	 **2b**	92 (1)	90 (10)
3	 **1c**	 **2c**	92 (24)	92 (240)
4	 **1d**	 **2d**	60 (24)	70(240)
5	 **1e**	 **2e**	65 (24)	75 (240)
6	 **1f**	 **2f**	45 (24)	65 (230)
7	 **1g**	 **2g**	50 (24)	65 (230)
8	 **1h**	 **2h**	60 (5)	70 (60)

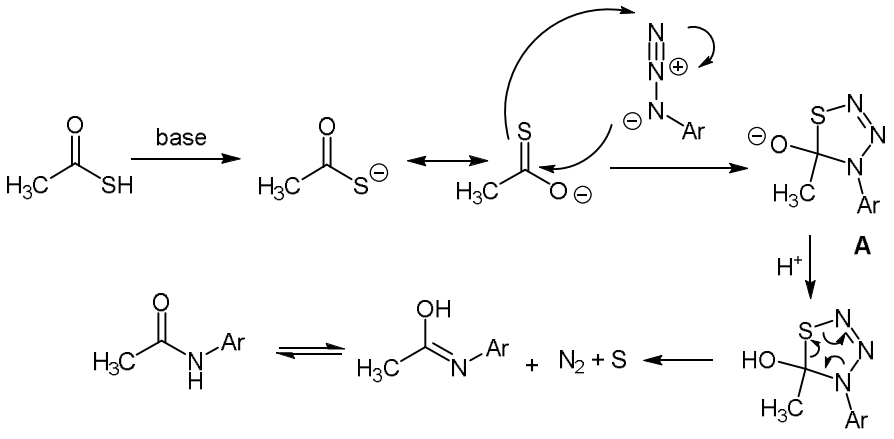
[a]All compounds were identified by comparison of their physical and spectral data with those of authentic samples [21-26]. [b]Stirring at room temperature. [c]Under ultrasonic irradiation.



Compound	%yield[a] (time/h)	%yield[b] (time/min)
3a	85 (8)	85 (60)
3b	65 (10)	70 (90)
3c	90 (10)	90 (80)
3d	85 (12)	90 (100)

[a] stirring at room temperature
[b] under ultrasonic irradiation stirring.

Scheme 2. Reaction of thioacetic acid with azido compounds 2

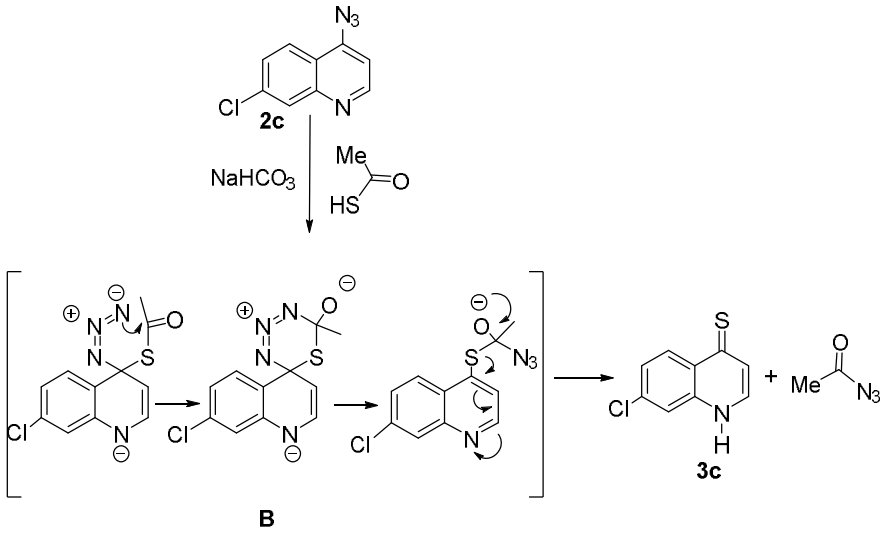


Scheme 2. Postulated mechanism for the synthesis of aryl acetamide 3

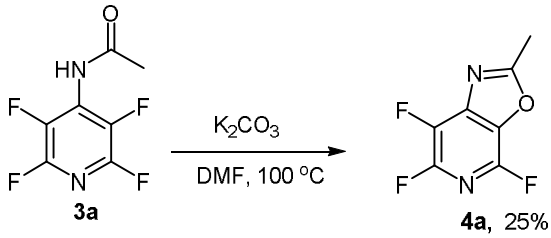
stretching and a sharp absorption band at 1692 cm^{-1} for C=O group. ^{1}H NMR spectrum of compound 3a showed a broad singlet in 6.48 ppm for NH and one singlet at 2.15 ppm for methyl hydrogenes. In ^{19}F NMR spectrum, 3a showed a signal at -93.29 ppm for fluorines located *ortho* to ring nitrogen and a signal at -145.92 ppm for fluorines located *meta* to ring nitrogen similar to those was previously reported [27].

Similarly, the reaction of thioacetic acid with 1-azido-2,4-dinitrobenzene 2d gave the desired *N*-(2,4-dinitrophenyl)acetamide 3d in a good yield.

According to the postulated mechanism by Shangguan



Scheme 4. Postulated mechanism for the synthesis of 3c



Scheme 4. synthesis of 4,6,7-trifluoro-2-methyloxazolo[5,4-c]pyridine 4a

and co-worker, formation of a thiatriazoline intermediate A (*via* either a 2+3 cycloaddition or a stepwise diazo transfer-like mechanism) accounts for the above observations [28]. Decomposition of thiatriazoline intermediate A would ultimately lead to acetamide 3 (Scheme 2).

4-Azido-7-chloroquinoline 2c reacts also with thioacetic acid in the presence of sodium bicarbonate and in methanol to give the 7-chloroquinoline-4(1H)-thione 3c. This reaction is assumed to take place via the formation of the intermediate derivative B (Scheme 3). IR spectrum of compound 3c revealed absorption bands ν_{max} = 3446 and 1077 cm^{-1} assignable to the NH and the thiocarbonyl groups. The ^1H NMR spectrum of 3c revealed signals as expected.

Reaction of thioacetic acid with 4-azido-2,3,5,6-tetrachloropyridine 2b in the presence of sodium bicarbonate and in methanol as solvent gave only 2,3,5,6-tetrachloropyridin-4-amine 3b. IR spectrum of compound 3b revealed absorption bands ν_{max} = 3493 cm^{-1} assignable to the NH_2 group. The ^1H NMR spectrum of compound 3b exhibited a singlet at δ = 7.34 ppm for the NH_2 group.

The reaction of tetrazolo[1,5-a]pyrazine 2e, 4-chlorotetrazolo[1,5-a]quinoxaline 2h, tetrazolo[1,5-a] pyridine 2f and 5-fluorotetrazolo[1,5-a]pyridine 2g with thioacetic acid under above conditions did not yield the acetamide or thione product.

In the course of our research we tried to further intermolecular nucleophilic aromatic substitution reaction of *N*-(perfluoropyridin4-yl)acetamid 3a. Attempt to intrermolecular cyclization of 3a, in the presence of potassium carbonate and in the DMF as solvent led to 4,6,7-trifluoro-2-methyloxazolo[5,4-*c*]pyridine 4a. Three

resonances by ^{19}F NMR (-92, -138 and -149 ppm), indicate displacement of fluorine atom attached to the 3-position of the pyridine ring by the oxygen nucleophile (Scheme 4).

CONCLUSIONS

In conclusion, we have shown that azido and tetrazolo aromatic and heteroaromatic compounds can be synthesized from reaction of the corresponding fluoro and chloro compounds with sodium azide under ultrasonic irradiation in short reaction time. Reaction of azido aromatic and heteroaromatic compounds with thioacetic acid mostly led to the different products. The strategy outlined in schemes 4 allowed synthesis of target 4,6,7-trifluoro-2-methyloxazolo [5,4-c]pyridine 4a by intermolecular cyclization of *N*-(perfluoropyridin4-yl)acetamid 3a**.**

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