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SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL EVALUATION OF FLUORINE-CONTAINING IMIDAZOLE AND 1, 2, 4-TRIAZOLE DERIVATIVES

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ABSTRACT

Attempt to building block approach, and synthesized fluorine containing organic halo substituents into imidazole and 1, 2, 4-triazole heterocyclic ring via metathesis. Imidazole **(A)** and 1, 2, 4-Triazole **(B)** were reacted with fluorine containing halo (chloride/bromide) benzyl compounds **(1-10)** in the presence of triethylamine in benzene at reflux temperature for 24 hrs to form corresponding fluorine containing imidazole **(11-15)** and 1, 2, 4-triazole **(16-20)** derivatives in ~60% yields. The derivatives of Imidazole and 1, 2, 4-Triazole are stable and soluble in $CHCl_3$, CH_3CN, CH_2Cl_2, THF and DMSO. These compounds have been characterised by using IR, ^1H and ^{19}F NMR spectroscopy, MS and elemental analysis. These derivatives **(11-20)** of Imidazole and 1, 2, 4-Triazole have been found bio-active.

INTRODUCTION

Fluorine containing heterocyclic compounds are very fascinating targets for synthetic organic chemists because of their potentially high physiological activities[1]. Fluorine containing organic compounds are associated with antimicrobials[2], anti-bacterial[3] and anticancer[4-5] activities. The research dealing with the synthesis of fluorine containing five-membered unsaturated nitrogen containing heterocyclic compounds has intensified considerably during the last decade and has resulted in new synthetic approaches and new commercial applications. In particulars imidazole and 1, 2, 4-triazole substituted with fluorobenzyl or trifluoromethyl benzyl groups have become increasingly popular as building blocks towards bioactive compounds.

A number of fluorine substituted imidazoles, including clotrimazole, are selective inhibitors of nitric oxide synthase, which makes them interesting drug targets in inflammation, neurodegenerative diseases and tumors of the nervous system[6-7]. Imidazole derivatives have occupied a unique place in the field of medicinal chemistry[8-9]. The incorporation of the imidazole nucleus is an important synthetic strategy in drug discovery. The high therapeutic properties[10-14] of the imidazole related drugs have encouraged the medicinal chemists to synthesize a large number of novel chemotherapeutic agents. Triazoles are an important class of heterocyclic compounds which show various biological activities including antibacterial[15-17], antifungal[18-20], hypoglycaemic[21-22], antidepressant[23], analgesic[24], antitumor[25], anti-proliferative[26], antitubercular[27] and anticonvulsant[28]. We are prompted for building block approach, and synthesized fluorine containing organic halo substituents into imidazole and 1, 2, 4-triazole heterocyclic ring via metathesis.

RESULTS AND DISCUSSION

The heterocyclic compounds Imidazole **(A)** and 1, 2, 4-Triazole **(B)** were reacted with variety of fluorine containing compounds that contained labile halogen substituents in the presence of triethylamine in benzene at 80°C refluxing temperature for 24 hrs. Benzene was the solvent of choice for these reactions since the salts formed precipitated in essentially quantitative amounts indicating that the reactions occurred **(Scheme-1)**. The occurrence of reaction was observed as the precipitation of salt took place during the course of reaction. The progress of the reaction was also monitored by TLC (silica coated). It was also observed that the bromides reacted faster than the chlorides. No attempt was made to recover the unreacted materials. The stability of all the products was confirmed by ^{19}F NMR and it was found that all compounds are air stable and do not hydrolyse by moisture.

The reactions of chlorides **(1-5)** with imidazole **(A)** were slower than bromides **(6-10)** with imidazole **(A)**. The formation of N-C bond was confirmed by the ^1H NMR spectra of the compounds **(11-15)**. The singlet peak at δ 4.18-5.43 ppm in the spectrum of derivatives of imidazole **(11-15)** and 1, 2, 4-triazole **(16-20)**, shows the presence of $-CH_2$ in between

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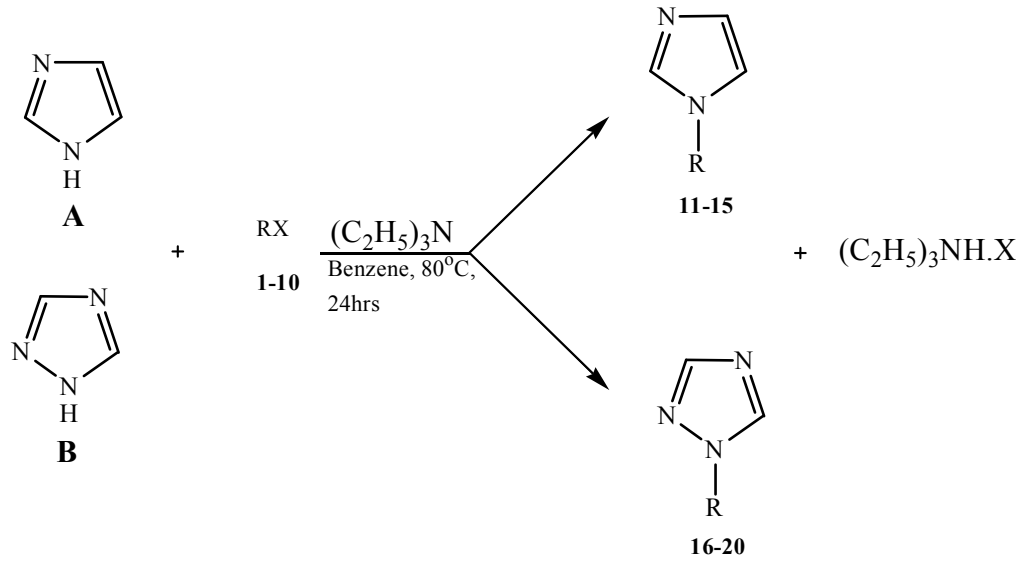
nitrogen of imidazole **(A)** and phenyl ring and similarly between nitrogen of 1, 2, 4-triazole **(B)** and phenyl ring. A multiplet observed δ 7.01-7.31, was accounted for four aromatic protons of phenyl ring. The two singlets for two and one protons δ 7.54 and δ 6.92 respectively of imidazole ring. Two singlets were observed δ 7.84 and δ 7.99 for two protons of 1, 2, 4-triazole ring. A multiplet observed δ 7.04-7.36, was accounted for four aromatic protons of phenyl ring.

checked by TLC on silica-gel coated Al plates (Merck). The IR spectra (4000-400cm$^{-1)}$ of synthesized compounds were recorded on Shimadzu 8400-s FT-IR spectrophotometer with KBr disc. ^{1}H NMR spectra were run in 5mm NMR tubes in $CDCl_3$ and the peak positions were measured relative to residual $CHCl_3$ and reported relative to Me_4Si. ^{19}F NMR spectra were obtained on a Bruker DRX 300MHz NMR spectrometer with $CFCl_3$ as internal reference.



A – Imidazole
B - 1, 2, 4-Triazole

R(X=Cl)
2-FC_6H_4-CH_2- **1**
3-FC_6H_4-CH_2- **2**
4-FC_6H_4-CH_2- **3**
3-CF_3-C_6H_4-CH_2- **4**
4-CF_3-C_6H_4-CH_2- **5**

R(X=Br)
2-FC_6H_4-CH_2- **6**
3-FC_6H_4-CH_2- **7**
4-FC_6H_4-CH_2- **8**
3-CF_3-C_6H_4- CH_2- **9**
4-CF_3-C_6H_4-CH_2- **10**

Scheme-1

Antibacterial activity

Synthesized compounds were screened for their antibacterial activities. The in vitro antibacterial activities of the synthesized compounds were assessed against bacteria. The bacteria were used gram positive (*Staphylococcus aureus* and *Bacillus subtilis*) and gram negative (*Escherichia coli* and *Pseudomonas aeruginosa*). Streptomycin is used as standard for comparison for antibacterial activities. The activities were determined by measuring the diameter of the inhibition zone in mm **(Table-1)**.

The mass spectra were recorded using Agilent 1100 series G1946 (EI mass).

All the solvents were AR. Imidazole **(A)**, 1, 2, 4-triazole **(B)**, 2-fluorobenzylchloride **(1)**, 3-fluorobenzylchloride **(2)**, 4-fluorobenzylchloride **(3),** 3-trifluoromethylbenzylchloride **(4)**, 4trifluoromethylbenzylchloride**(5),** 2-fluorobenzylbromide **(6),** 3-fluorobenzylbromide **(7),** 4-fluorobenzylbromide **(8)**, 3-trifluoromethylbenzylbromide **(9)** and 4-

Table-1 Antibacterial activity: Inhibition zone (mm) of fluorine containing derivatives of imidazole and 1, 2, 4-triazole (11-15 and 16-20) against different microbial strains

Compounds	Bacterial strain-Zone of inhibition (mm) and Minimum inhibition concentration of compound Test (10 µg/ml)			
	Gram positive		Gram negative	
	S.aureus	*B.subtillis*	*E. coli*	*P.aeruginosa*
11	20(14)	00(00)	00(00)	12(08)
12	22(18)	14(14)	10(06)	10(08)
13	14(10)	00(00)	00(00)	18(08)
14	12(08)	06(04)	08(04)	00(00)
15	10(06)	00(00)	06(02)	04(02)
16	00(00)	00(00)	00(00)	10(06)
17	00(00)	16(10)	10(06)	14(10)
18	18(08)	10(08)	14(12)	10(12)
19	16(08)	06(06)	16(08)	00(00)
20	20(10)	00(00)	00(00)	14(14)
Streptomycin	22(22)	22(20)	22(20)	22(20)

MATERIAL AND METHODS

Melting Points were determined in open capillary tubes and are uncorrected. The purity of newly synthesized compounds was

trifluoromethylbenzylbromide **(10)**, were obtained from ACROS and used as received. Triethylamine and benzene were

obtained from Aldrich and used after drying with 4 A° molecular sieves.

Table 2 Characterization data of compounds 11-15 and 16-20

Compounds	Molecular formula (Mol. Wt)	Yield %	M.P. (°C)	Elemental Analysis % Found (Calcd.)		
				C	H	N
1-(2-Fluorobenzyl)-1H-imidazole **(11)**	$C_{10}H_9N_2F$ (176.19)	78.0	Liquid	68.25 (68.16)	5.30 (5.14)	16.01 (15.89)
1-(3-Fluorobenzyl)- 1H-imidazole **(12)**	$C_{10}H_9N_2F$ (176.19)	70.0	Liquid	68.19 (68.16)	5.20 (5.14)	15.95 (15.89)
1-(4-Fluorobenzyl)- 1H-imidazole **(13)**	$C_{10}H_9N_2F$ (176.19)	68.0	Liquid	68.42 (68.16)	5.19 (5.14)	16.10 (15.89)
1-(3-Trifluoromethylbenzyl)- 1H-imidazole **(14)**	$C_{11}H_9N_2F_3$ (226.19)	60.0	Liquid	58.80 (58.40)	3.72 (4.01)	12.86 (12.38)
1-(4-Trifluoromethylbenzyl)- 1H-imidazole **(15)**	$C_{11}H_9N_2F_3$ (226.19)	62.0	Liquid	58.82 (58.40)	3.55 (4.01)	12.95 (12.38)
1-(2-Fluorobenzyl)-1H-[1,2,4]triazole **(16)**	$C_9H_8N_3F$ (177.17)	78.0	Liquid	61.32 (61.00)	4.60 (4.55)	23.24 (23.71)
1-(3-Fluorobenzyl)- 1H-[1,2,4]triazole **(17)**	$C_9H_8N_3F$ (177.17)	70.0	Liquid	61.38 (61.00)	4.57 (4.55)	23.89 (23.71)
1-(4-Fluorobenzyl)- 1H-[1,2,4]triazole **(18)**	$C_9H_8N_3F$ (177.17)	68.0	Liquid	62.03 (61.00)	5.00 (4.55)	24.15 (23.71)
1-(3-Trifluoromethyl-benzyl)- 1H-[1,2,4]triazole **(19)**	$C_{10}H_8N_3F$ (227.18)	60.0	Liquid	52.91 (52.86)	3.64 (3.54)	18.98 (18.49)
1-(4-Trifluoromethyl-benzyl)- 1H-[1,2,4]triazole **(20)**	$C_{10}H_8N_3F_3$ (227.18)	52.0	Liquid	53.76 (52.86)	4.01 (3.54)	18.75 (18.49)

Experimental section

Synthesis of fluorine containing imidazole and 1, 2, 4-triazole derivatives

A mixture of imidazole **A** (1.0 mmol) or 1, 2, 4-triazole **B** (1.0 mmol) and fluoro benzylchloride/bromide (1.0mmol) **(1-10)** were dissolved in benzene (30ml) and added triethylamine (1.0mmol) and refluxed for 24 hrs at 80°C. After completion of reaction as followed by t.l.c. examination, the reaction mixture was filtered; the solvent was removed from the filtrate by rotoevoporator. The resulted reaction product mixture was purified by a column chromatography on silica gel by using hexane-ethylacetate (70:30) as eluent.

1-(2-Fluorobenzyl)-1*H*-imidazole (11) - Yield: 78%, oil; IR (KBr disc, cm^{-1}): 3048(b), 2828(s), 1350(s), 1425(w), 1620(s), 1228(w); ^{1}HNMR ($CDCl_3$): δ 7.01-7.31 (m, 4H), 5.10 (s, 2H), 7.54 (s, 2H), 6.92 (s, 1H); ^{19}F NMR (282MHz, $CDCl_3$) δ (ppm): -116.61(s, 1F); MS: *m/z* 176 (M^++1); Elemental analysis for $C_{10}H_9N_2F$, Found (Calcd)%: C, 68.25(68.16); H, 5.30(5.14), N, 16.01(15.89).

1-(3-Fluorobenzyl)-1*H*-imidazole (12) - Yield: 70%, oil; IR (KBr disc, cm^{-1}): 3029(b), 2848(w), 1365(s), 1470(s), 1530(w), 1230(s); ^{1}HNMR ($CDCl_3$): δ 6.77-7.12 (m, 4H), 4.99 (s, 2H), 7.24 (s, 2H), 6.74 (s, 1H); ^{19}F NMR (282MHz, $CDCl_3$) δ (ppm): -118.23(s, 1F); MS: *m/z* 176 (M^++1); Elemental analysis for $C_{10}H_9N_2F$, Found (Calcd)%: C, 68.19(68.16); H, 5.20(5.14), N, 15.95(15.89).

1-(4-Fluorobenzyl)-1*H*-imidazole (13) - Yield: 68%, oil; IR (KBr disc, cm^{-1}): 3036(b), 2835(s), 1345(w), 1460(w), 1610(s), 1235(s); ^{1}HNMR ($CDCl_3$): δ 6.85-7.04 (m, 4H), 5.21 (s, 2H), 7.18 (s, 2H), 6.78 (s, 1H); ^{19}F NMR (282MHz, $CDCl_3$) δ (ppm): -117.58(s, 1F); MS: *m/z* 176 (M^++1); Elemental analysis for $C_{10}H_9N_2F$, Found (Calcd)%: C, 68.42(68.16); H, 5.19(5.14), N, 16.10(15.89).

1-(3-Trifluoromethylbenzyl)-1*H*-imidazole (14) - Yield: 60%, oil; IR (KBr disc, cm^{-1}): 3065(b), 2842(w), 1340(s), 1420(s), 1605(w), 1248(s); ^{1}HNMR ($CDCl_3$): δ 7.06-7.36 (m, 4H), 5.15 (s, 2H), 7.61 (s, 2H), 6.99 (s, 1H); ^{19}F NMR (282MHz, $CDCl_3$) δ (ppm): -64.63(s, 1F); MS: *m/z* 176 (M^++1); Elemental analysis for $C_{11}H_9N_2F_3$, Found (Calcd)%: C, 58.80(58.40); H, 3.72(4.01), N, 12.86(12.38).

1-(4-Trifluoromethylbenzyl)-1*H*-imidazole (15) - Yield: 62%, oil; IR (KBr disc, cm^{-1}): 3055(b), 2875(s), 1330(w), 1445(s), 1615(w), 1250(s); ^{1}H NMR ($CDCl_3$): δ 7.06-7.39 (m, 4H), 5.18 (s, 2H), 7.65 (s, 2H), 6.97 (s, 1H); ^{19}F NMR (282MHz, $CDCl_3$) δ (ppm): -63.18 (s, 1F); MS: *m/z* 176 (M^++1); Elemental analysis for $C_{11}H_9N_2F_3$, Found (Calcd)%: C, 58.82(58.40); H, 3.55.30(4.01), N, 12.95(12.38).

1-(2-Fluorobenzyl)-1*H*-[1, 2, 4] triazole (16) - Yield: 76%, oil; IR (KBr disc, cm^{-1}): 3035(b), 2845(s), 1315(s), 1475(w), 1610(s), 1235(s); ^{1}H NMR ($CDCl_3$): δ 7.05-7.37 (m, 4H), 5.39 (s, 2H), 7.94-8.15 (s, 2H); ^{19}F NMR: δ -117.92 (s, 1F) ; ^{19}F NMR (282MHz, $CDCl_3$) δ (ppm): -117.92 (s, 1F); MS: *m/z* 176 (M^++1); Elemental analysis for $C_9H_8N_3F$, Found (Calcd)%: C, 61.32(61.00); H, 4.60(4.55), N, 23.24(23.71).

1-(3-Fluorobenzyl)-1*H*-[1, 2, 4] triazole (17) - Yield: 64%, oil; IR (KBr disc, cm^{-1}): 3030(b), 2853(s), 1328(w), 1490(s), 1625(s), 1230(s); ^{1}H NMR ($CDCl_3$): δ 6.92-7.25 (m, 4H), 5.12 (s, 2H), 7.82-8.01 (s, 2H); ^{19}F NMR: δ -116.89 (s, 1F) ; ^{19}F NMR (282MHz, $CDCl_3$) δ (ppm): -116.61(s, 1F); MS: *m/z* 176 (M^++1); Elemental analysis for $C_9H_8N_3F$, Found (Calcd)%: C, 61.38(61.00); H, 4.57(4.55), N, 23.89(23.71).

1-(4-Fluorobenzyl)-1*H*-[1, 2, 4] triazole (18) - Yield: 78%, oil; IR (KBr disc, cm^{-1}): 3045(b), 2860(s), 1310(w), 1460(s), 1620(s), 1226(w); ^{1}H NMR ($CDCl_3$): δ 7.04-7.36 (m, 4H), 5.19 (s, 2H), 7.84-7.99 (s, 2H); ^{19}F NMR: δ -117.69 (s, 1F); MS: *m/z*

176 (M^++1); Elemental analysis for $C_9H_8N_3F$, Found (Calcd)%: C, 61.03(61.00); H, 5.00(4.55), N, 24.15(23.71).

1-(3-Trifluoromethylbenzyl)-1*H*-[1, 2, 4] triazole (19) - Yield: 60%, oil; IR (KBr disc, cm^{-1}): 3065(b), 2840(s), 1325(w), 1470(s), 1642(w), 1238(s); ^1H NMR ($CDCl_3$): δ 7.08-7.40 (m, 4H), 5.47 (s, 2H), 7.82-7.99 (s, 2H); ^{19}F NMR: δ -59.19 (s, 3F); MS: *m/z* 176 (M^++1); Elemental analysis for $C_{10}H_8N_3F_3$, Found (Calcd)%: C, 52.91(52.86); H, 3.64(3.54), N, 18.98(18.49).

1-(4-Trifluoromethylbenzyl)-1*H*-[1, 2, 4] triazole (20) - Yield: 59%, oil; IR (KBr disc, cm^{-1}): 3060(b), 2838(s), 1330(w), 1410(s), 1610(w), 1290(s); ^1H NMR ($CDCl_3$): δ 7.02-7.39 (m, 4H), 5.43 (s, 2H), 7.84-8.02 (s, 2H); ^{19}F NMR: δ -67.63 (s, 3F) ; MS: *m/z* 176 (M^++1); Elemental analysis for $C_{10}H_8N_3F_3$, Found (Calcd)%: C, 53.76(52.86); H, 4.01(3.54), N, 18.75(18.49).

CONCLUSION

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