



"ECOFRIENDLY ULTRASOUND ASSISTED SYNTHESIS OF SOME FLUORINATED AZOLES"

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ABSTRACT

Fluorine containing acid hydrazide when treated with aryl isothiocyanate to gave compound 2-[3,5-bis(trifluoromethyl)benzoyl]-N-phenylhydrazine carbothioamide 2 on treatment with NaOH and under Ultrasound irradiation gave 1,2,4-Triazoles 3.

KEYWORDS

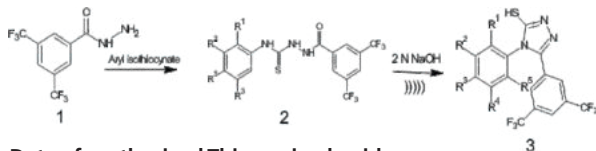
Ultrasound, Ecofriendly, Fluorine, Triazole.

Ultrasonication is one of the Eco-friendly tools for synthesizing the heterocyclic compounds. Besides conventional methods, the search for new methods of increased efficiency and selectivity constitutes a major goal in chemistry¹.

Trifluoromethyl group when introduce into a biologically active compounds can dramatically modify the potency, physicochemical properties and metabolic profile of the resulting compounds². Therefore there is considerable interest within the pharmaceutical industry to utilize the properties of the trifluoromethyl group in the target design.

Compounds having 1,2,4-triazole nucleus have been reported as antibacterial, sedative, anti-convulsant and anti-inflammatory agents³.

PresentWork: Equimolar amount (0.001 mol) of acid hydrazide (1) and phenyl isothiocyanate were dissolved in 100 mL RBF with 25 mL of ethanol. The reaction mixture was subjected for ultra sound irradiation for 20-30 minutes, the resultant compound (2) on treatment with 2N NaOH gives 1,2,4-triazoles (3).



Data of synthesized Thiosemicarbazides.

Sr. No.	Compds.	Aryl group	m.P (°C)	Time (min)	Yield (%) (U S)	Time (min)	Yield (%) Traditional
1	2a	Phenyl	158	30	85	45	76
2	2b	2- Methoxy	160	25	82	50	72
3	2c	2- Methyl	180	30	84	50	70
4	2d	3- Methyl	200	24	86	45	74

Spectral Study- Thiosemicarbazides.

IR:- IR spectrum of **2a** showed following characteristic absorption bands in its spectrum.

3257 cm^{-1} , 3224 cm^{-1} , 3188 cm^{-1} due to N-H groups, 1674 cm^{-1} due to C=O group.

H¹ NMR: The ¹H NMR spectrum of **2b** showed following characteristic signals. 3.63, 3H (s) due to -OCH₃- aliphatic protons, 6.91-7.21, 4H, (m) due to aromatic protons, 8.05-8.57 3H (m) due to aromatic protons, 9.42, 1H, (s), 9.88, 1H, (s) 11.12, 1H (s) due to three N-H protons.

• Data of synthesized Triazoles.

Sr. No.	Compds.	Aryl group	m.p. (°C)	Time (min)	Yield (%) (U S)	Time (min)	Yield (%) Traditional
1.	3a	Phenyl	258	30	82	50	76
2	3b	2- Methoxy	260	25	74	55	65
3	3c	2- Methyl	278	30	76	50	68
4	3d	3- Methyl	220	24	85	55	76

Triazoles:

IR : IR spectrum of **3b** showed following characteristic absorption bands in its spectrum.

3078 cm^{-1} Ar =C-H, 2910 cm^{-1} aliphatic C-H (stretch), 2762 cm^{-1} due to S-H, 1550-1602 cm^{-1} , -C=N ring stretch. 769 cm^{-1} due to -C-S group.

H¹NMR:

The ¹H NMR spectrum of **3a** showed following characteristic signals

7.42- 7.54 δ , 5H (m) Ar protons, 7.84- 8.27 δ 3H (m) Ar-protons, 14.42 δ , 1H (s), -SH proton.

Conclusion: The compounds synthesized by ultrasound irradiation requires shorter time as compared to traditional method and yields are also higher. Therefore in these environmentally cautious days this thought is worthwhile to find greener routes for the synthesis of biologically important heterocyclic compounds like Fluorinated Azoles by using Ultrasound technique for the synthesis 1,2,4-triazole derivatives.

References:

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- [2]. Jones L H & Mowbray C, Synlett, 9, (2006), 1404.
- [3]. Dong H, Quan B, Niu T & Xiao Z, Indian J Heterocycl Chem, 15, (2006), 415.