CHAPTER 4

Synthesis of 6-arylimidazo[2,1-b][1,3]thiazole and 2-aryl-7-methoxy imidazo[2,1-b][1,3]benzothiazole derivatives
4.1. Introduction

Heterocyclic compounds comprise the major family of organic compounds. These are enormously essential with wide range of synthetic, pharmaceutical and industrial applications and are well known for their biological activities. The high therapeutic properties of these heterocycles have encouraged the medicinal chemists to synthesize a large number of novel chemotherapeutic agents. It is well known that the heterocycles are present in all kinds of organic compounds of interest in electronics, biology, optics, pharmacology, material sciences and so on. Mostly researchers have maintained their interest in sulfur and nitrogen-containing heterocyclic compounds through decades of historical development of organic synthesis. Imidazoles and thiazoles have been reported to show pharmacological activities. Imidazole drugs have broadened scope in remedying various dispositions in clinical medicines. A lot of work on the synthesis and biological activities of the condensed imidazo [2, 1-b]-thiazoles has been reported.

In the family of heterocyclic compounds, nitrogen and sulfur containing heterocycles are an important class of compounds in medicinal chemistry [1]. Thiazoles play a prominent role in nature. Large numbers of thiazole derivatives have emerged as active pharmaceutical ingredients in several drugs for their potential anti-inflammatory [2], anti-tumor [3], anti-hyperlipidemic [4], anti-hypertensive [5] and several other biological properties [6]. Thiazole, or 1, 3-thiazole (1), is a heterocyclic compound that contains both sulphur and nitrogen. The thiazole ring is notably a component of the vitamin thiamin. Thiazoles displayed broad range of biological activities and found in many potent biologically active molecules such as Sulfathiazole (2) (antimicrobial drug), Ritonavir (antiretroviral drug), Abafungin (3)
(antifungal drug) and Tiazofurin (4) (antineoplastic drug). So far, modifications of the thiazole ring have proven highly effective with improved potency and lesser toxicity.

In the field of five membered heterocyclic structures, imidazole nucleus shows various properties. The high therapeutic properties of the imidazole related drugs have encouraged the medicinal chemists to synthesize a large number of novel chemotherapeutic agents. Imidazole drugs have broadened scope in remedying various dispositions in clinical medicines.

Among nitrogen and sulphur containing heteroaromatic compounds, imidazole and imidazo [2, 1-b] thiazole derivatives are crucial core structures used to create drugs. Imidazo [2,1-b][1,3]thiazoles [7,8] occupy a prominent place in medicinal chemistry because of their significant properties as therapeutics. This has generated much interest in the synthesis of new classes of heterocyclic systems, thereby to explore their biological properties. During recent years there has been a large investigation on different classes of imidazothiazole compounds, many of which were found to possess an extensive spectrum of pharmacological activity.
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Many natural products and biologically active compounds containing imidazo[2,1-b][1,3]thiazole moieties have been synthesized and shown to exhibit potent biological activity. Tetramisole (5) is a strong anthelmintic [10] in the treatment of many nematodes. Tetramisole is a broad-spectrum anthelmintic that acts on both mature and immature stages of many important gastrointestinal nematodes and lungworms in cattle, sheep and goats.

![imidazo[2,1-b][1,3]thiazole structure](image)

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The imidazo[2,1-b][1,3]thiazole skeleton has been used as anthelmintic agents, anti-hypertensive, anti-inflammatory, immunosuppressive agents, fungicides, herbicides, antitumor agents, and cardio tonic agents [7-10]. Considering the potent bioactivities of compounds possessing an imidazothiazoles core, we synthesised imidazo[2,1-b][1,3]thiazole derivatives and evaluated them for their biological activities.

Generally, the imidazothiazoles are synthesized by the reaction of 2-aminothiazoles with acyl bromides in ethanol. Andrew Scribner et al [11] synthesized several novel 5,6-diaryl-2-substituted imidazo[2,1-b][1,3]thiazoles (6), which are potent against coccidiosis which is the major cause of death in poultry farms. Of these, 5-(2-aminoypyrimidin-4-yl)-2-methylpiperidin-4-yl)imidazo[2,1-b][1,3]thiazole showed significant activity versus apicomplexan protozoan parasites Eimeria tenella, E. acervulina, E. maxima, and E. mitis.
Aldo Andreani et al [12] synthesized guanylhydrazones (7) from aminothiazoles which are potent antitumor agents. The guanylhydrazones are prepared by reaction of amino guanidine with appropriate aldehydes which were obtained by Vilsmeier reaction on the corresponding imidazo[2,1-b]thiazoles.

Jin-Hun Park et al [13] synthesized 6-(4-fluorophenyl)-5-(2-substituted pyrimidin-4-yl)imidazo[2,1-b]thiazole derivatives (8). Among them the cyclic sulphamide derivatives exhibited the most potent antiproliferative activity against A375P human melanoma cell line.
Mohammad mahdavi et al [14] synthesized N-cyclohexyl-3-methyl-6-phenylimidazo [2,1-b]thiazol-5-amine (9) through one pot synthesis. The synthesis involves four component reaction of chloroacetone, aromatic aldehydes, thiourea, and isocyanides.

\[
\begin{align*}
\text{\text{CH}_2\text{Cl}=\text{C}=\text{O}} & + \text{\text{C}_6\text{H}_5\text{C}=\text{O}} + \text{\text{NH}_2\text{S}} + \text{\text{C}_6\text{H}_{11}\text{N}} & \xrightarrow{\text{NH}_4\text{Cl} \text{, Toluene, reflux}} \text{9}
\end{align*}
\]


\[
\begin{align*}
\text{\text{C}_6\text{H}_5\text{N}=\text{S}} & + \text{\text{C}_6\text{H}_5\text{C}=\text{O}} & \xrightarrow{\text{RNC, Ethanol}} \text{10}
\end{align*}
\]

Rajurkar V. G. et al [16] synthesized novel 3,6-disubstituted imidazo[2,1-b][1,3]thiazole derivatives (11) by the cyclocondensation of α-bromo ketones with thiourea afforded 4-substituted 1,3-thiazole-2-amino followed by further reaction with α-bromo ketones. Among these some of derivatives were shown to possess highest antimicrobial activity against S. aureus, K. pneumonia, and C. albicans.

\[
\begin{align*}
\text{\text{CH}_2\text{Br}=\text{C}=\text{O}} & + \text{\text{NH}_2\text{S}} & \xrightarrow{\text{Ethanol, reflux}} \text{\text{NH}_{2} \text{S}=\text{NH}} & \text{\xrightarrow{\text{Br} \text{, Ethanol}}} \text{11}
\end{align*}
\]
Mohammad Bakherad et al. reported the synthesis of 6-(substituted benzyl)imidazo[2,1-b][1,3]thiazoles (12) using the polystyrene-supported palladium(II) ethylenediamine complex, in the Sonogashira coupling reaction.

\[
\begin{align*}
\text{HN-S-NH}_2 + \text{Br} & \xrightarrow{\text{Ar}[\text{PS-en-Pd(II)}]} \text{ArS-NH}_2 \\
& \xrightarrow{\text{CuI,Et}_3\text{N,DMF}} \text{Sonogashira coupling} \\
& \xrightarrow{\text{HN-S-NH}_2 \text{Br}} 12
\end{align*}
\]

Huaiwei Ding et al. synthesized and evaluated cytotoxic activity of some novel N-pyridinyl-2-(6-phenylimidazo [2, 1-b] thiazol-3-yl) acetamide (13) derivatives. Ethyl 2-(2-aminothiazol-4-yl)acetate reacted with 2-bromoacetophenones to give the crude esters which were hydrolyzed to acid using aqueous sodium hydroxide followed by coupling with amine to form amides.

Chang's et al. proposed a multicomponent reaction for the synthesis of imidazo thiazoles using Ogi reaction. They synthesized 3-amino-benzo[d]imidazo [2, 1-b] thiazole derivatives (14) by reacting a mixture of 2-aminobenzothiazole and an aldehyde with ammonium chloride and isocyanide in toluene under reflux condition.
Gundurao Kolavi et al. [20] synthesized imidazo[2,1-b][1,3]thiazole derivatives (15) by reacting benzopyran-2-one in ethanol. Further the carbaldehydes which are prepared by Vilsmeier–Haack reaction treated with hydrazine hydrate to form imidazo[2,1-b][1,3]thiazole fused diazipinones via lactone ring opening.

Yuji Matsuya et al. [21] described the syntheses and in vitro evaluation of new SIRT1 activator candidates possessing the imidazo[1,2-b]thiazole core (16, 17), as a lead compound.
Shashikant M. Patil et al [22] have developed a practical and metal free method for methylthiolation of imidazo[1,2-a]pyridines, imidazo[2,1-b]thiazoles (18) and other imidazo-fused heterocycles using DMSO and POCl₃ as a reagent.


Ahmed R. Ali et al [24] synthesized series of 2-imino-4-arylthiazoles bearing acetamidomorpholine or acetamidopiperazine moieties (21) and evaluated them for their antitumor activities.

Jitender K. Malik et al [25] synthesized substituted diaryl imidazo[2,1,b]-benzothiazole derivatives (22) among which some of the compounds shows promising antifungal activities.
Sheelavath et al synthesized imidazothiazole derivatives (23, 24) of benzofuran and evaluated their antimicrobial activity.

Ravindra M. Kumbhare and coworkers [26] synthesized a new class of Mannich bases of 2-arylimidazo[2,1-b]benzothiazoles (25) and evaluated for their antiproliferative activities.
Elif Gursoy et al. [27] synthesized imidazo[2,1-b]thiazole derivatives (26) and evaluated for primary cytotoxicity. Some of these compounds showed promising anticancer activities.

![Chemical structure of 26]

David Alagille [28] synthesized 2-arylimidazo[2,1-b]benzothiazoles (27) which are potent amyloid binding agents. The load of amyloid deposit in the brain correlates with the cognitive deficit in Alzheimer’s disease. The synthesized benzothiazoles help in its early diagnosis and better qualification for monitoring the amyloid burden in the brain.

![Chemical structure of 27]

4.2. Present work

Considering the potent bioactivities of compounds possessing an imidazo thiazole moiety, in this paper we reported the synthesis of some novel fused imidazo [2,1-b] [1,3] thiazole derivatives that have pharmacological activities. In general the imidazo thiazoles are synthesised by the reaction of amino thiazoles with \( \alpha \)-bromoketones in alcohols. Various ring systems containing the \( \text{C(NH}_2\text{)=N} \) moiety as a part of the ring have been found to condense with \( \alpha \)-bromoketones to yield condensed...
imidazo-heterocyclic systems. The ring nitrogen attacks the CH₂Br unit rather than the primary exocyclic amino group during the formation of imidazole ring.

The synthetic strategy involves the following steps

1. Synthesis of substituted phenyl imidazo[2,1-b][1,3]thiazole derivatives 30(a-e)

   ![Scheme-4](image)

2. Synthesis of substituted phenyl-7-methoxy imidazo[2,1-b][1,3]benzothiazole derivatives 32(a-h) scheme-5

   The schematic representation of the synthesized molecules is given in Scheme-4 and 5.

![Scheme-4](image)

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Substituted phenyl imidazo[2,1-b][1,3]thiazole derivatives 30(a-e) were obtained by the reaction of 2-bromo-1(3,4-disubstituted phenyl) ethanones 28(a-e) with aminothiazole in ethanol under reflux for 6-8 hrs. The completion of the reaction was monitored by TLC. The solvent was evaporated under vacuum and the product was isolated by recrystalisation in ethanol.

The structure of substituted phenyl imidazo[2,1-b][1,3]thiazole derivatives 30(a-e) were characterized by \(^1\)HNMR, \(^13\)CNMR and Mass spectral studies. The \(^1\)HNMR 6-(4-chlorophenyl) imidazo[2,1-b][1,3]thiazole (30e) shows singlet at \( \delta \)
8.45ppm due to imidazole ring proton. Multiplet observed between δ 8.12-7.49 ppm was due to 6 aromatic protons. Further, the mass spectrum of compound (30e) displayed a molecular ion peak M⁺ at m/z 235.0 corresponding to the molecular mass of the compound and isotopic peak at m/ 237.1 [M⁺2].

![Scheme-5](image)

Substituted phenyl imidazo[2,1-b][1,3]thiazole derivatives 32(a-h) were obtained by the reaction of 2-bromo-1(3,4-disubstituted phenyl) ethanones 28(a-h) with methoxy aminothiazole in ethanol under reflux for 6-8 hrs. The mixture of 2-bromo-1(3,4-disubstituted phenyl ethanone) and methoxy aminothiazole in dry ethanol was refluxed for 6-8 hrs. The solvent was evaporated and the products were isolated by recrystallisation in ethanol. The structures of phenyl-7-methoxyimidazo[2,1-b][1,3] benzothiazole 32(a-g) were characterized ¹HNMR, ¹³CNMR and Mass spectral studies. The ¹HNMR of compound (32c) showed singlet at δ8.84 due to imidazo proton. Multiplet was observed between δ7.93–7.16 is due to seven aromatic protons. The singlet at 3.83 is due to the methoxy protons on the aromatic ring. Further, the formation of compound (32c) was confirmed by ¹³CNMR spectrum. The compound (32c) showed a signal at δ157.7 ppm assigned to bridge carbon of imidazole ring. Signal appeared at δ 56.36 ppm corresponds to the CH₃ carbon. Further the mass of compound (32c) displayed a molecular ion peak [M⁺ 2] at m/z 361.1.
$^{1}$HNMR spectrum of compound 30e
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$^{13}$CNMR spectrum of compound 30e
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LCMS spectrum of compound 30e
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1H NMR spectrum of compound 32c

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$^{13}$CNMR spectrum of 32c
Sample Report.

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Peak ID 1, Time: 2.39

LCMS spectrum of compound of 32c
4.3. Experimental protocol

4.3.1. Synthesis of 6-(3,4-disubstituted)imidazo[2,1-b][1,3]thiazole derivatives 30(a-e)

**General procedure**

The mixture of 1,3-thiazol-2-amine (29) (0.01 mol) and 2-bromo-1-(3,4-disubstituted-phenyl) ethanone (28a-e) (0.01 mol) in dry ethanol was refluxed at 85-90 °C for about 6-8 hours. The reaction mass was allowed to cool to room temperature. The solvent was evaporated under vacuum. The product was diluted with sodium carbonate and extracted with ethyl acetate. The organic layer was evaporated and the products were isolated by recrystallisation in ethanol.

6-(3,4-Dichlorophenyl) imidazo [2,1-b] [1,3] thiazole (30a)

Off white solid, Yield 62%, m.p.: 296-298 °C; $^1$H NMR (400MHz, DMSO-$d_6$, δ ppm): 9.52 (s, 1H), 8.48 (s, 1H), 8.10-7.42 (m, 4H, Ar-H), $^{13}$C NMR (400MHz, DMSO-$d_6$, δ ppm): 149.8, 142.4, 133.8, 132.1, 131.5, 130.2, 126.9, 125.4, 120.9, 115.5, 111.6; MS (LCMS): m/z = 270 (M$^+$ + 1): Mol. Formula:C$_{11}$H$_6$Cl$_2$N$_2$S.

4-(Imidazo[2,1-b][1,3]thiazol-6-yl) benzonitrile (30b)

Off solid, Yield 71%; m.p 339-341 °C; $^1$H NMR (400MHz, DMSO-$d_6$, δ ppm): 8.56 (s, 1H), 8.10-7.46 (m, 6H, Ar-H); $^{13}$CNMR (400MHz, DMSO-$d_6$, δ ppm): 150.1, 142.7, 137.3, 133.3, 127, 125.9, 121.0, 119.3, 119.1, 115.9; MS (LCMS); m/z = 227[M+2]: Mol. Formula; C$_{12}$H$_7$N$_3$S.

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6-(4-Chloro-3-fluorophenyl) imidazo [2, 1-b][1,3]thiazole (30c)

Off white solid, Yield-57%; mp 152-155 °C; \(^1\)H NMR (400MHz, DMSO-\(d_6\), \(\delta\) ppm): 8.45 (s, 1H), 8.08-8.07 (s, 1H), 7.88-7.85 (m, 4H, Ar-H); MS (LCMS): m/z = 254.1 (M+1); Mol. Formula: C\(_{11}\)H\(_6\)ClFN\(_2\)S.

6-[3, 4-bis (trifluoromethyl) phenyl]imidazo[2,1-b][1,3]thiazole (30d)

Off white solid, Yield-63%; m.p.: 151-155 °C; \(^1\)H NMR (400MHz, DMSO-\(d_6\), \(\delta\) ppm): 8.68 (1H, s), 7.97 (s, 1H), 8.49 (2H, s), 8.06-7.37 (m, 4H, Ar-H); \(^{13}\)C NMR (400MHz, DMSO-\(d_6\), \(\delta\) ppm): 150.4, 143.3, 137.1, 131.4, 131.1, 128.4, 125.33, 121.6, 120.7, 115.0, 112.5; MS (LCMS): m/z = 337 [M\(^+\) 1]; Mol. Formula: C\(_{13}\)H\(_6\)F\(_6\)N\(_2\)S.

6-(4-Chlorophenyl) imidazo [2, 1-b] [1, 3] thiazole (30e)

Off white solid; Yield-65%; m.p. 287-290 °C; \(^1\)H NMR (400MHz, DMSO-\(d_6\), \(\delta\) ppm): 8.45 (1H, s), 8.12-7.49 (m, 6H, Ar-H); \(^{13}\)C NMR (400MHz, DMSO-\(d_6\), \(\delta\) ppm): 149.3, 142.1, 133.0, 131.5, 132.1, 130.7, 129.4, 127.2, 121.2; MS (LCMS); m/z = 235(M\(^+\) 1); Mol. Formula:C\(_{11}\)H\(_7\)ClN\(_2\)S.

4.3.2. Synthesis of 2-substituted phenyl 7-methoxyimidazo[2,1-b][1,3] benzothiazole

General procedure:

The mixture of 7-methoxyimidazo[2,1-b][1,3]benzothiazole (31) (0.01mol) and 2-bromo-1-(3,4-disubstituted-phenyl)ethanone (28a-e) (0.01 mol) in dry ethanol was refluxed at 80-85 °C for about 7-8 hours. The solvent was evaporated under vacuum and the products were isolated by recrystallization in ethanol.
2-(3,4-dichlorophenyl)-7-methoxyimidazo[2,1-b][1,3]benzothiazole (32a)

Off white solid; Yield -73%; m.p. 261-264 °C; \( ^1H \) NMR (400MHz DMSO-d_6, \( \delta \) ppm): 8.85 (1H, s), 8.03 (1H, s), 7.85 (s, 1H), 7.80-7.13 (m, 4H, Ar-H), 3.83 (1H, s); \( ^13C \) NMR (400MHz, DMSO-d_6, \( \delta \) ppm): 157.6, 147.2, 143.2, 134.7, 132, 131.5, 131.0, 129.6, 126.5, 126.1, 124.9, 117.8, 114.5, 110.8, 109.9, 56.3; MS (LCMS): m/z = 351 [M+1]; Mol. Formula: C_{16}H_{10}Cl_{2}N_{2}O_{5}S.

2-(4-Chlorophenyl)-7-methoxyimidazo[2,1-b][1,3]benzothiazole (32b)

Off White solid; Yield -58%; m.p. 273-275 °C; \( ^1H \) NMR (400MHz, DMSO-d_6, \( \delta \) ppm): 8.83 (1H, s), 7.94-7.92 (m, 3H, Ar-H), 7.72 (s, 1H), 7.52-7.17 (m, 3H, Ar-H), 3.84 (s, 3H); \( ^13C \) NMR (400MHz, DMSO-d_6, \( \delta \) ppm): 157.7, 147.0, 143.8, 132.3, 132.2, 131.0, 129.3, 126.7, 126.2, 114.6, 114.5, 110.2, 109.9, 56.3; MS (LCMS); m/z = 316 [M+2]; Mol formula; C_{16}H_{11}Cl_{2}N_{2}OS.

2-(4-Bromophenyl)-7-methoxyimidazo[2,1-b][1,3]benzothiazole (32c)

Off white solid; Yield -64%, m.p. 295-298 °C; \( ^1H \) NMR (400MHz, DMSO-d_6, \( \delta \) ppm): 8.84 (1H, s), 7.93-7.16 (m, 7H, Ar-H), 3.83 (s, 3H); \( ^13C \) NMR (400MHz, DMSO-d_6, \( \delta \) ppm): 157.7, 142.0, 143.0, 132.4, 132.2, 131.9, 131.0, 127.0, 126.1, 120.8, 114.7, 114.6, 110.2, 109.9, 56.3; LCMS; m/z= 362.1[M+2]; Mol. formula; C_{16}H_{11}Br_{2}N_{2}OS.

2-(4-Nitrophenyl)-7-methoxyimidazo[2,1-b][1,3]benzothiazole (32d)

Yellow solid, Yield 72%; m.p. 306-309 °C; \( ^1H \) NMR (400MHz, DMSO-d_6, \( \delta \) ppm): 8.97 (1H, s), 8.26-7.14 (m, 6H, Ar-H), 7.67 (s, 1H), 3.83 (s, 1H); \( ^13C \) NMR (400MHz, DMSO-d_6, \( \delta \) ppm): 157.2, 147.9, 146.3, 143.6, 140.5, 131.1, 128.3, 125.9, 125.5, 124.7, 114.7, 114.5, 112.4, 109.9, 56.3; MS (LCMS) m/z = 325 [M+1]; Mol. formula; C_{16}H_{11}N_{3}O_{5}S.
4-(7-Methoxyimidazo[2,1-b][1,3]benzothiazol-2-yl)benzonitrile (32e)

Off white solid, Yield- 75%, m.p. 198-201°C; $^1$H NMR (400MHz, DMSO-$d_6$, δ ppm):
8.93 (s, 1H), 7.99-7.85 (m, 6H, Ar-H), 7.68 (s, 1H), 3.83 (s, 3H); $^{13}$C NMR (400MHz, DMSO-$d_6$, δ ppm): 156.2, 147.6, 143.8, 138.4, 137.5, 133.2, 132.9, 131.6, 131.1 127.0, 126.0, 125.4, 119.4, 114.5, 109.6, 56.3; MS(LCMS) m/z = 305 [M$^+$]: Mol. Formula; C$_{17}$H$_{11}$N$_3$OS.

2-(4-MethyIphenyl)-7-methoxyimidazo[2,1-b][1,3] benzothiazole (32f)

White solid, Yield-67%, m.p. 273-275°C; $^1$HNMR (400MHz, DMSO-$d_6$, δ ppm): 8.72 (s, 1H), 7.94 (s, 1H), 7.74-7.17 (m, 6H, Ar-H), 3.84, (s, 3H), 2.34 (s, 3H); $^{13}$C NMR (400MHz, DMSO-$d_6$, δ ppm): 157.8, 146.5, 143.6, 137.8, 131.0, 129.9, 129.4, 126.1, 125.1, 114.8, 109.9, 109.5, 56.3, 21.3; MS (LCMS) m/z = 295[M$^+$]: Mol formula; C$_{17}$H$_{14}$N$_2$OS.

2-(4-Chloro-3-fluorophenyl)-7-methoxyimidazo[2,1-b][1,3]benzothiazole (32g)

Off white solid, Yield-61%, m.p 285-288 °C; $^1$H NMR (400MHz, DMSO-$d_6$, δ ppm): 8.83 (1H, s), 7.87 (s, 1H), 7.81 (s, 1H), 7.69-7.15 (m, 4H, Ar-H), 3.84, (s, 3H); $^{13}$C NMR (400MHz, DMSO-$d_6$, δ ppm): 157.6, 147.2, 143.7, 135.5, 131.5, 131.0, 126.1, 121.9, 118.1, 114.4, 113.0, 112.7, 110.8, 109.9, 56.35; MS (LCMS) m/z=334 [M$^+$2]: Mol. Formula; C$_{16}$H$_{10}$ClFN$_2$OS.

2-[3,4-Bis (trifluoromethyl)phenyl]-7-methoxyimidazo[2,1-b][1,3]benzothiazole (32h)

Off white solid, Yield- 63%, m.p 280-283 °C; $^1$H NMR (400MHz, DMSO-$d_6$, δ ppm):
9.09 (1H, s), 8.42 (s, 1H), 7.95 (s, 1H), 7.83-7.15 (m, 4H, Ar-H), 3.84 (s, 3H); $^{13}$C NMR (400MHz, DMSO-$d_6$, δ ppm): 157.6, 147.2, 143.7, 135.5, 131.5, 131.0, 126.1, 121.9, 118.1, 114.4, 113.0, 112.7, 110.8, 109.9, 56.35; MS (LCMS) m/z = 418 [M$^+$2]: Mol. formula; C$_{18}$H$_{10}$F$_6$N$_2$OS.
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