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AN EFFICIENT AND RECYCLABLE IONIC LIQUID MEDIATED SYNTHESIS OF 1-(3-METHYLBENZO[4,5]IMIDAZO[2,1-B]THIAZOL-2-YL)ETHAN-1-ONE AND ITS DERIVATIVES

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ABSTRACT: This approach uses a variety of aromatic and aliphatic aldehyde/ketone in the presence of recyclable ionic liquid to synthesize 1-(3-methylbenzo [4,5] imidazo [2,1-b] thiazol-2-yl) ethan-1-one and its derivatives (4a-h). The synthetic methodology makes use of inexpensive, safe, and non-toxic organic solvents as well as a straightforward process, mild reaction conditions, and high yields. The synthesized derivatives were obtained in 82-93% of yields. The synthesized compounds were confirmed by using spectroscopic data (^{1}H and ^{13}C NMR, IR, MS) and elemental analysis.

KEYWORDS: 2-Mercaptobenzimidazole, Aliphatic/Aromatic Ketone, Fused Heterocycles, Ionic Liquid.

INTRODUCTION:

Heterocyclic compounds are a class of organic compounds that contain a ring structure composed of at least one atom other than carbon, such as nitrogen (N), oxygen (O), or sulfur (S). These non-carbon atoms in the ring are called heteroatoms. The presence of heteroatoms in the ring imparts unique chemical and physical properties to heterocyclic compounds compared to carbocyclic compounds[i]. Organic heterocyclic chemistry is primarily concerned with organic unsaturated derivatives; unstrained organic 5- and 6-membered rings are the subject of the majority of research and applications in this field. Furan, pyrrole, thiophene, and pyridine are among them. The group of organic heterocycles joined to benzene rings is another significant subclass. For instance, quinoline, benzothiophene, indole, benzofuran and benzimidazole are the fused benzene derivatives of pyridine, thiophene, pyrrole, and furan, respectively. Two benzene rings coming together forms a third, sizable family of chemical molecules.

The heterocyclic molecule 2-Mercaptobenzimidazole (2-MBI) has a benzimidazole nucleus with a mercapto(-SH) group connected at the 2-position. A fused benzene ring and an imidazole ring, where nitrogen atoms constitute a component of the heterocycle, make up the

benzimidazole nucleus. The -SH group enhances the compound's ability to bind to various biological targets and increases its reactivity. Widely varying in bioactivity are fused heterocyclic compounds, like pyrrole [1,2-c] imidazoles, which are evaluated for their antioxidant and antibacterial properties, and derivatives of pyrrole fused with chromene, which are evaluated for their antibacterial properties[i-ii]. Functionalized fluroquinolones is potentially a novel class of compounds has dual anti-inflammatory and antiglycation properties[iii]. A series of new triazine, isoindole, and pyrimidine heterocycles fused to the selenopyrazole ring system was synthesized as an antibacterial and antifungal agent[iv]. The given nucleus i.e. mercaptobenzimidazole is also used in non-biological activity such as regulators for plant growth[v], it is also used as corrosion inhibitor for steel in sulphuric acid solution[vi], Steel and Zinc in solution of phosphoric acid[vii-viii], Stainless steel in aqueous solution of Na-Cl[ix], It is also widely used in rubber processing as accelerator[x] and anti-oxidant for plastic and rubber[xi]. Some fused heterocycles compounds like thieno[2,3-b]pyridine has anticancer properties[xii]. The nucleus of mercaptobenzimidazole display insecticidal properties[xiii], it is well known analytical reagent for mercury and also used for the determination for Fe (II), Cu(II), and Cd (II) metal ions in waste water, sewage water, and industrial water samplei[xiv-xvi].

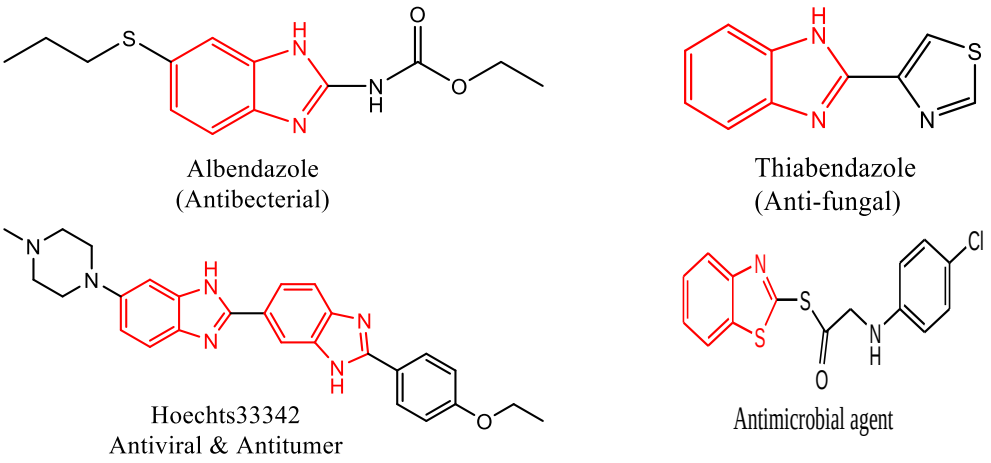


Fig.1

The reported method for synthesizing mercaptobenzimidazole derivatives, such as 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one, using methanesulphonic acid (MSA) as a catalyst, is an efficient methodology[xvii]. Nowadays, ionic liquids have become very important versatile and fruitful catalyst for organic synthesis. Ionic liquids (ILs) are a class of salts that exist in the liquid state at relatively low temperatures, often below 100°C. Unlike traditional salts such as sodium chloride (which are solid at room temperature), ionic liquids are made entirely of ions but remain in a liquid state. This property is due to the large size and irregular shape of their ions, which reduces the strength of the electrostatic forces holding them together, making it difficult for the ions to pack into a solid lattice[xviii]. In the present work, we synthesized 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one and its derivatives (4a-h) using N-methylpyridinium tosylate (NMPyTs) as an ionic liquid. The catalyst proved to be efficient for this conversion, demonstrating a high yield, a quick reaction time, and a more straightforward workup procedure.

EXPERIMENTAL:

MATERIALS AND METHODS

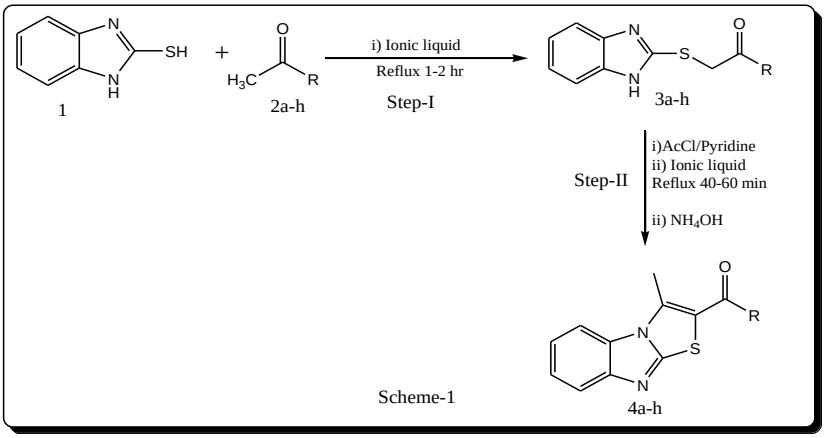
All the chemicals and synthetic grade reagent were obtained from Sigma Aldrich India and Merck chemicals. They were used without purification. Reaction was monitored by thin layer chromatography on silica plates visualizing with iodine chamber. Melting points were recorded in open capillaries using a Buchi Melting point B-450 apparatus. FT-IR measurements were done as KBr pellets on Bruker-FT-IR/4100, Japan. ^{1}H NMR spectra were recorded on a Bruker spectrometer (400 MHz) in DMSO using TMS as the internal standard. ^{13}C NMR were recorded on a Bruker DRX 100 MHz Spectrometer. Mass spectra were measured using high-resolution ESI–MS (DFS) Thermo spectrometers (70 eV).

Step-I: General Procedure for the Synthesis (1H-benzo[d]imidazol-2-yl) benzothioate (3a-h)

In a 100 mL round-bottom flask, a mixture of 2-mercaptobenzimidazole (0.1 mmol), substituted aliphatic or aromatic ketones (0.1 mmol), and a catalytic amount of N-methylpyridinium tosylate (NMPyTs) was heated at 80 °C until the reaction was complete. The reaction progress was monitored by thin layer chromatography. After the completion of the reaction, the mixture was poured into 75 mL of water and boiled for 10 minutes. The mixture was then filtered, and the solid crude product was collected, dried, and recrystallized from ethanol to obtain the purified product 3(a-h) (Scheme 1).

STEP-II: General procedure for the synthesis 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one (4a-h)

A 100 mL round-bottom flask containing a mixture of 2-benzimidazolylthioacetophenone (0.1 mmol), acetyl chloride (0.1 mmol), and a catalytic amount of N-methylpyridinium tosylate (NMPyTs) was refluxed at 100 to 120°C for 45 to 60 minutes until the reaction was complete. The reaction progress was monitored by thin layer chromatography. After the completion of the reaction, mixture was poured over the crushed ice and add ammonium hydroxide (NH_4OH) for neutralization it. The precipitate was collected by filtration and evaporation of the mother liquor under reduced pressure led to the recovery of catalyst which could be reused for next run. The structures of the purified products 4(a-h) were characterized by physical constant and spectroscopic techniques and compared with the standard method.



Scheme-1

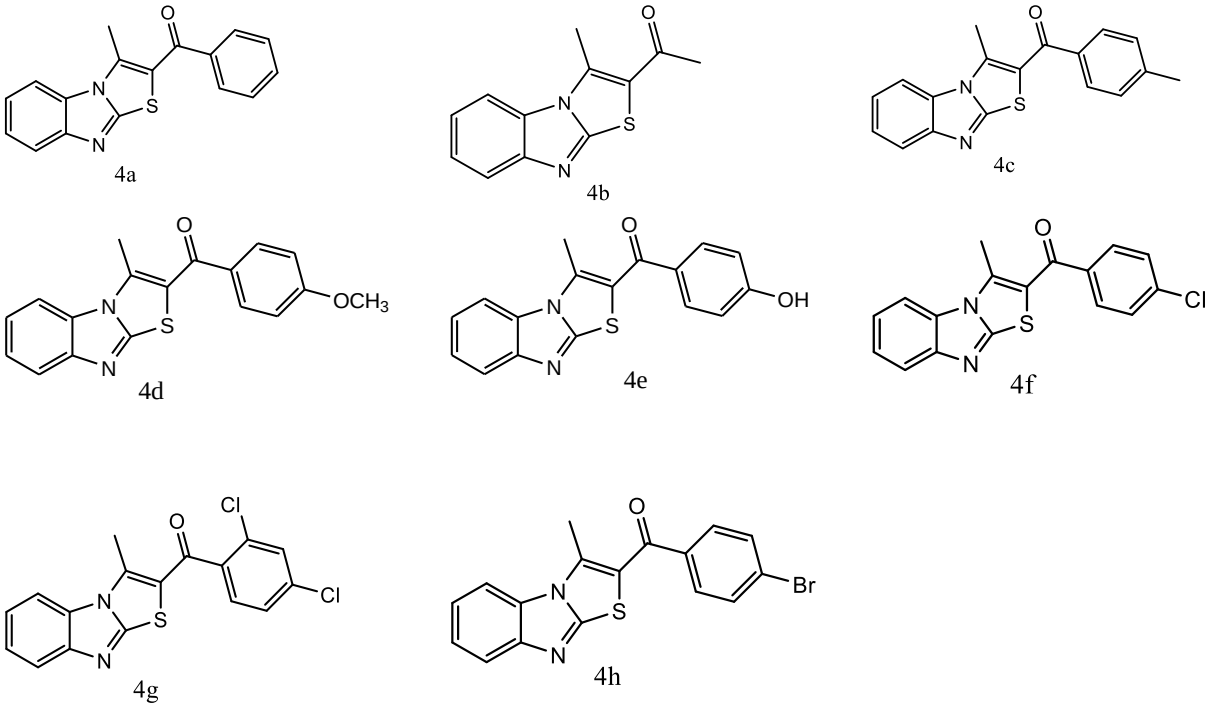


Fig.2 Fig. 2 The chemical structures of the products (4a-h).

RESULT AND DISCUSSION:

The first step of this well-reported method involves the reaction of 2-mercapto-benzimidazole (1) with substituted aliphatic or aromatic ketones (2) in the presence of a catalytic amount of N-methylpyridinium tosylate (NMPyTs) under heat, resulting in the formation of (1H-benzo[d]imidazol-2-yl) benzothioate (3a-h) derivatives. The final step of the synthesis for 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one is performed using 2-benzimidazolylthioacetophenone (0.1 mmol), acetyl chloride (0.1 mmol), and a catalytic amount of N-methylpyridinium tosylate (NMPyTs). In this step, the reaction undergoes cyclocondensation at 120°C in an ionic liquid, completing within 45-60 minutes to yield the target compound (Scheme 1). This reaction has been selected as a model to determine the optimal catalyst concentration for synthesizing 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one and its derivatives.

Our initial work started with screening of various catalysts to demonstrate the advantages of our method for synthesizing 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one derivatives (Table 3). Results indicate that NMPyTs is an effective catalyst, significantly improving reaction times and product yields (4a-h, Scheme 1). Observations from Table 1 (entries 1 to 5) show that other catalysts were less efficient for this protocol. We report a straightforward synthesis using NMPyTs (Table 1, entry 6) for producing 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one and its derivatives. This is achieved by treating an equimolar mixture of 2-benzimidazolylthioacetophenone (0.1 mmol) and acetic anhydride (0.1 mmol) in Step II, as outlined in Scheme 1.

Table 1: Screening of catalyst for Synthesis of 4a-h derivatives

Entry	Catalyst	Time (min)	Yield (%)
1	No Catalyst	180 min	Trace
2	DBSA	130 min	48
3	TEBA	120 min	55
4	L-Proline	105 min	67
5	SAB-Pr-NH_2	70 min	80
6	**NMPyTS**	**45 min**	**93**

We also investigated and performed the model reaction without any catalyst and it was found that, in the absence of the catalyst, only traces amount of the desired product was detected in reaction mixture (Table 2, entry 1). As the reaction required a catalyst, we perform the reaction using 5% and 10% NMPyTs and the result declared that the reaction was possible with moderate yield (Table 2, entries 2 and 3). To improve the % of yield of the product, we continued our efforts by changing the mol % i.e. 10% to 15% and performed the model reaction. We observed that when 15% of catalyst was satisfactorily done the reaction, because of it got the 93% yield in 45 min (Table 2, entry 4). We observed also that increases the mol % of catalyst i.e. 20% to 25% and above, then % of yield of product decreases and time was increases (Table 2, Entry 5 and 6). From this investigation we observed that 15 % mol of NMPyTs catalyst is very remunerative and fruitful for the model reaction. By using this protocol, we have been synthesized 4a-h derivatives.

Table 2: Optimization of the reaction conditions for the model reaction[a]

Entry	Catalyst (% mol)	Time (min)	Yield[b] (%)
1	None	200	35
2	5	150	78
3	10	75	84
4	**15**	**45**	**93**
5	20	70	89
6	25	110	81

[a]Reaction conditions: 2-benzimidazolylthioacetophenone (3a-h) (0.1 mmol), acetic anhydride (0.1 mmol), and NMPyTs (15 Mol %). [b]Isolated yields.

Table 3: NMPyTs catalyzed synthesis of 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one derivatives.

Entry	R	Time (min)	Yield[b] (%)	M. P. (^oC)
4a	$-C_6H_5$	60	86	125
4b	$-CH_3$	55	82	115
4c	$-C_6H_4-CH_3$	58	90	100
4d	$-C_6H_4-OCH_3$	45	93	170
4e	$-C_6H_4-OH$	55	87	180
4f	$-C_6H_4-Cl$	48	91	205
4g	$-C_6H_3-Cl_2$	52	83	210
4h	- C6H4-Br	45	84	190

[a]Reaction conditions: 2-benzimidazolylthioacetophenone (3a-h) (0.1 mmol), acetic anhydride (0.1 mmol), and NMPyTs (15 Mol %). [b]Isolated yields.

Reusability of catalyst

Number of cycles	Fresh	1st	2nd	3rd
Model reaction (% Yield)	93	92	91	88

Recycling is a key aspect of the Green Chemistry principle, significantly enhancing the efficiency and cost-effectiveness of chemical reactions. In this study, the recyclability of NMPyTs was evaluated for synthesizing 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one derivatives. A catalyst was separated after the reaction and reused in a subsequent reaction. Results showed that the yield of the product remained consistent, and the catalyst could be effectively employed in three consecutive reactions without a noticeable loss in activity.

Spectroscopic analysis of the synthesized derivatives (4a-h).

4a (3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)(phenyl)methanone

IR (KBr, cm^{-1}): 3025 (C=C-H), 2875 (C-H), 1719 (C=O), 1313 (C-N), 1627 (C=N), 1615 (Ar C=C), 1675 (C=C). ^{1}H NMR ($CDCl_3$, 400 MHz) δppm: 7.25-7.95 (m, 7.8 Hz, 4H Ar-H), 7.72 (m, 7.8 Hz, 1H Ar H), 7.59 (dd, 2.2 Hz, Ar H), 7.87 (d, 2.2 Hz, 2H, Ar H), 2.25(s, 3H, CH_3). ^{13}C NMR ($CDCl_3$ 100 MHz) δppm:185.5 (C=O), 155.3 (N=C), 152.3 (N-C), 145.2 & 140.0 (Ar C-N), 99.5 (S-C), 19.5 (-CH_3).

4b 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one

IR (KBr, cm^{-1}): 3029 (C=C-H), 2873 (C-H), 1722 (C=O), 1317 (C-N), 1628 (C=N), 1614 (Ar C=C), 1679 (C=C). ^{1}H NMR ($CDCl_3$, 400 MHz) δppm: 7.25-7.96 (m, 7.8 Hz, 4H Ar H), 7.75 (m, 7.8 Hz, 1H Ar H), 7.60 (dd, 2.2 Hz, Ar H), 2.44 (s, 3H, CH_3), 2.24 (s, 3H, CH_3). ^{13}C NMR ($CDCl_3$ 100 MHz) δppm: 195.5 (C=O, Carbonyl Carbon), 151.3 (N=C), 142.3 (N-C), 135.2 & 128.0 (Ar C-N), 119.5 (S-C), 9.5 (-CH_3).

4c (3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)(p-tolyl)methanone

IR (KBr, cm^{-1}): 3032 (C=C-H), 2874 (C-H),1725 (C=O), 1322 (C-N), 1631 (C=N), 1639 (Ar C=C), 1677 (C=C). ^{1}H NMR ($CDCl_3$, 400 MHz) δppm: 7.26-7.98 (m, 7.8 Hz, 4H Ar H), 7.42 (dd, 2.2 Hz, Ar H), 8.2 (d, 2.2Hz, 2H, Ar H), 2.20 (s, 3H, CH3), 2.43 (s, 3H, CH3). ^{13}C NMR ($CDCl_3$ 100 MHz) δppm:191 (C=O, Carbonyl Carbon), 141 (C=N), 148 (N-C), 134 & 128 (Ar C-N), 98.9 (S-C), 23.6 (CH3), 10.5 (CH_3).

4d (4-methoxyphenyl)(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)methanone

IR (KBr, cm^{-1}): 3033 (C=C-H), 2873 (C-H), 1723 (C=O), 1322 (C-N), 1633 (C=N), 1639 (Ar C=C), 1673 (C=C). ^{1}H NMR ($CDCl_3$, 400 MHz) δppm: 7.25-7.96 (m, 7.8 Hz, 4H Ar H), 7.07 (dd, 2.2 Hz, Ar H), 7.42 (d, 2.2Hz, 2H, Ar H), 2.22 (s, 3H, CH3), 3.81 (s, 3H, CH3). ^{13}C NMR ($CDCl_3$ 100 MHz) δppm:190 (C=O, Carbonyl Carbon), 141.5 (C=N), 150 (N-C), 135 & 128 (Ar C-N), 130.1 (S-C), 55.6 (CH3), 10.5 (CH_3).

4e (4-hydroxyphenyl)(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)methanone

IR (KBr, cm^{-1}): 3039 (C=C-H), 2878 (C-H), 1726 (C=O), 1324 (C-N), 1634 (C=N), 1636 (Ar C=C), 1674 (C=C). ^{1}H NMR ($CDCl_3$, 400 MHz) δppm: 7.25-7.96 (m, 7.8 Hz, 4H Ar H), 7.67 (dd, 2.2 Hz, Ar H), 7.89 (d, 2.2Hz, 2H, Ar H), 2.22(s, 3H, CH3), 9.71 (s, 1H, OH). ^{13}C NMR ($CDCl_3$ 100 MHz) δppm:191 (C=O, Carbonyl Carbon), 141.5 (C=N), 150.9 (N-C), 134 & 129 (Ar C-N), 128.7 (S-C), 9.6 (CH_3).

4f (4-chlorophenyl)(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)methanone

IR (KBr, cm^{-1}): 3040 (C=C-H), 2879 (C-H), 1728 (C=O), 1327 (C-N), 1639 (C=N), 1633 (Ar C=C), 1675 (C=C). ^{1}H NMR ($CDCl_3$, 400 MHz) δppm: 7.25-7.96 (m, 7.8 Hz, 4H Ar -H),

7.64 (dd, 2.2 Hz, Ar -H), 7.96 (d, 2.2Hz, 2H, Ar -H), 2.24 (s, 3H,-CH_3). ^{13}C NMR ($CDCl_3$ 100 MHz) δppm: 190.3 (C=O, Carbonyl Carbon), 141.5 (C=N), 150.8 (N-C), 135.5 & 128.4 (Ar -C-N), 130.7 (S-C), 9.8 (-CH_3).

4g (2,4-dichlorophenyl)(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)methanone

IR (KBr, cm^{-1}): 3031 (C=C-H), 2880 (C-H), 1729 (C=O), 1324 (C-N), 1637 (C=N), 1631 (Ar C=C), 1671 (C=C). ^{1}H NMR ($CDCl_3$, 400 MHz) δppm: 7.25-7.96 (m, 7.8 Hz, 4H Ar H), 7.72 (s, Ar H), 7.56-7.73 (d, 2.2 Hz, Ar H), 2.21 (s, 3H,-CH_3). ^{13}C NMR ($CDCl_3$ 100 MHz) δppm:191.3 (C=O, Carbonyl Carbon), 140.5 (C=N), 150.7 (N-C), 135.1 & 128.2 (Ar C-N), 130.3 (S-C), 9.6 (CH_3).

4h (4-bromophenyl)(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)methanone

IR (KBr, cm^{-1}): 3023 (C=C-H), 2872 (C-H),1722 (C=O), 1314 (C-N), 1632 (C=N), 1627 (Ar C=C), 1672 (C=C) 686 (C-Cl). ^{1}H NMR ($CDCl_3$, 400 MHz) δppm: 7.25-7.99 (m, 7.8 Hz, 4H Ar H), 7.62 (dd, 2.2 Hz, Ar H), 7.76 (d, 2.2Hz, 2H, Ar H), 2.27(s, 3H, CH_3). ^{13}C NMR ($CDCl_3$ 100 MHz) δppm:198 (C=O, Carbonyl Carbon), 141 (C=N), 148.3 (N-C), 137 & 125 (Ar C-N), 99.1 (S-C), 12.1 (CH_3).

Antibacterial Activity

All compounds prepared in this study were screened for their potential antibacterial activities. The bacterial strains tested included E. coli [MTCC 443], P. aeruginosa [MTCC 1688], S. aureus [MTCC 96], and S. pyogenes [MTCC 442] as Gram-positive bacteria, as well as C. albicans [MTCC 227], A. niger [MTCC 282], and A. clavatus [MTCC 1323] as Gram-negative bacteria. The antibacterial activities of the compounds were tested using the tube dilution technique. Each test compound, along with the standard antibiotics Ampicillin and Ciprofloxacin, was dissolved in DMSO. The minimum inhibitory concentrations (MIC) were determined as the lowest concentrations of the compounds that inhibited visible growth.

Table 4. Anti-bacterial activity of synthesized compounds 4a–4h in MIC (mg/mL).

Antibacterial Activity							
Minimal Inhibition Concentration [mg/mL**]**							
Tested Compound Code	**EC**	**PA**	**SA**	**SP**	**CA**	**AN**	**AC**
4a	200	200	125	100	500	500	1000
4b	250	200	100	200	500	1000	1000
4c	125	250	200	250	500	1000	1000
4d	100	100	200	250	250	500	500
4e	125	100	125	125	500	1000	1000
4f	62.5	125	100	200	1000	1000	1000
4g	200	250	62.5	125	500	500	1000
4h	100	200	125	100	500	1000	1000
Ampicillin	30	--	40	25	100	100	100
Ciprofloxacin	25	25	50	50	500	100	100

The Minimal Inhibition Concentration (MIC) values for the antibacterial studies of compounds 4a to 4h are presented in Table 1. The anti-bacterial activity of all the compounds against EC, PA, SA, SP, CA, AN and AC showed good potencies. Among the synthesized compounds 4e, 4f, 4g and 4h showed very good to moderate activity with MIC values against all the bacterial strains. The electron-withdrawing nature of substituents such as OH, Cl, and Br on the aromatic ring significantly influenced their activity. In this study, ampicillin and ciprofloxacin were used as standard antibacterial agents.

CONCLUSION:

The present work represent a simple, efficient and environmentally benign protocol for the synthesis of 1-(3-methylbenzo[4,5]imidazo[2,1-b]thiazol-2-yl)ethan-1-one derivatives in presence of N-methyl pyridinium tosylate (NMPyTs) ionic liquid under reflux. The catalyst offers excellent reusability, is non-corrosive, and is easy to handle and separate. The given strategy can be concluded that easy handling, high product of yield due to using proper amount of green catalyst in very shorter reaction time and simply handling the reaction condition compared other methods.

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