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$TiCl_3(OTf)$/[bmim]Cl Assisted One-Pot Multicomponent Reaction for The Synthesis of Spiro-Thiazolidinone Derivatives

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Abstract: *Multicomponent reactions (MCRs) are efficient strategies for the development of biologically active heterocyclic compounds; however, the development of economical and ecofriendly catalytic systems remains a challenge. This study aims to explore an efficient catalytic system $TiCl_3(OTf)$/[bmim]Cl for the synthesis of spirothiazolidinones under mild reaction conditions. This protocol offers a sustainable and efficient way for the development of bioactive heterocycles, highlighting the potential use of ionic liquid- $TiCl_3(OTf$ catalytic systems.*

Keywords: Ionic liquid, $TiCl_3(OTf)$/[bmim]Cl, multicomponent reaction (MCR), spirothiazolidin-4-ones

I. INTRODUCTION

Multistep reactions always produce waste and require additional isolation procedures that often include traditionally used volatile organic compounds as well as hazardous and costly solvents after each step. Multicomponent reactions (MCRs) are very powerful synthetic strategies in chemistry where three or more reactants react in a single step to form a desired product. MCRs are having high atom economy, cost effective, can be carried out at mild reaction conditions and useful in rapid generation of structurally diverse biologically active molecules. [1]

In 2008, $TiCl_3$OTf-ionic liquid as an catalyst for the synthesis of thioamides was reported by J. Noei, and A.R. Khosropour.[2] Recently, Habib Firouzabadi and co-workers reported the use of $TiCl_3(OTf)$ solid trichloro titanium(IV) trifluoromethanesulfonate catalyzed acylation of *–OH* and *–SH* under neat conditions.[3] Benzoylation and transesterification of alcohols, phenols and thiols was also reported at higher temperature under solvent-free conditions. In 2015, Asadollah Farhadi and coworkers employed $TiCl_3(OTf)$ solid catalyst with ionic liquid in MCR to synthesize 3,4-dihydropyrimidin-2(1H)-ones/ thiones.[4]

Spiro compounds have been categorized with notable biological activities, such as antimycobacterial, anti-inflammatory, antiviral, and antitumor activities.[5-7] Heterocyclic moieties that contain a thiazolidin-4-one ring are also accompanying with pharmacological effects, such as antioxidant,[8] antitubercular,[9] anti-inflammatory,[10] antibacterial,[9] antifungal,[11] cytotoxic,[11] anticancer,[10] antiproliferative,[12] antihyperglycemic and antidyslipidemic agents.[13] Over the last three decades, many synthetic methodologies have been developed for the synthesis of spirothiazolidin-4-ones and 4-thiazolidinones using wide range of catalytic systems. Some methods use 1-butyl-3-methylimidazolium and 1-methylimidazolium with inorganic anions like PF_6, BF_4, and PTSA,[14] sulfonated mesoporous silica,[15] urea-choline chloride as a deep eutectic solvent with carbon-SO_3H as a solid acid catalyst,[16] and $CuSO_4.5H_2O$ (10 mol%) with PEG-400,[17] DCC,[18] HBTU,[19] deep eutectic solvent,[20] MCM-41supported Schiff base and $CuSO_4.5H_2O$,[21] nano-Fe_3SO_4@SiO_2,[22] montmorilloniteK-10,[23] Baker's yeast,[24] DBSA[25] and $[Et_3NH][HSO_4]$.[26] However, despite their usefulness, many of these protocols suffer from disadvantages such as

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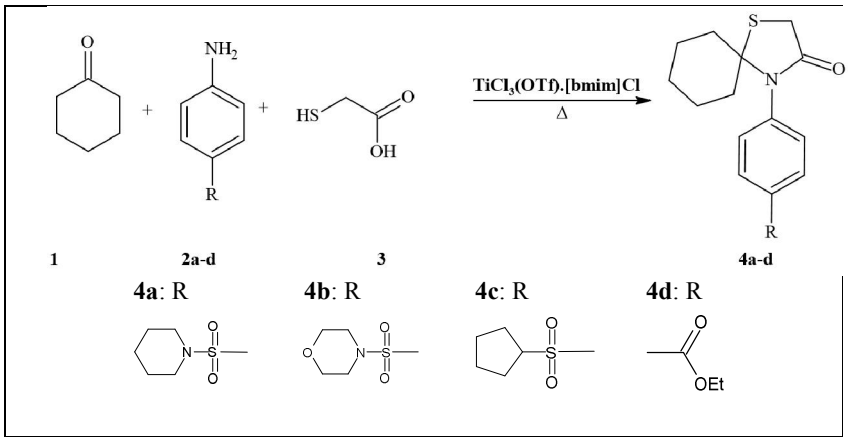
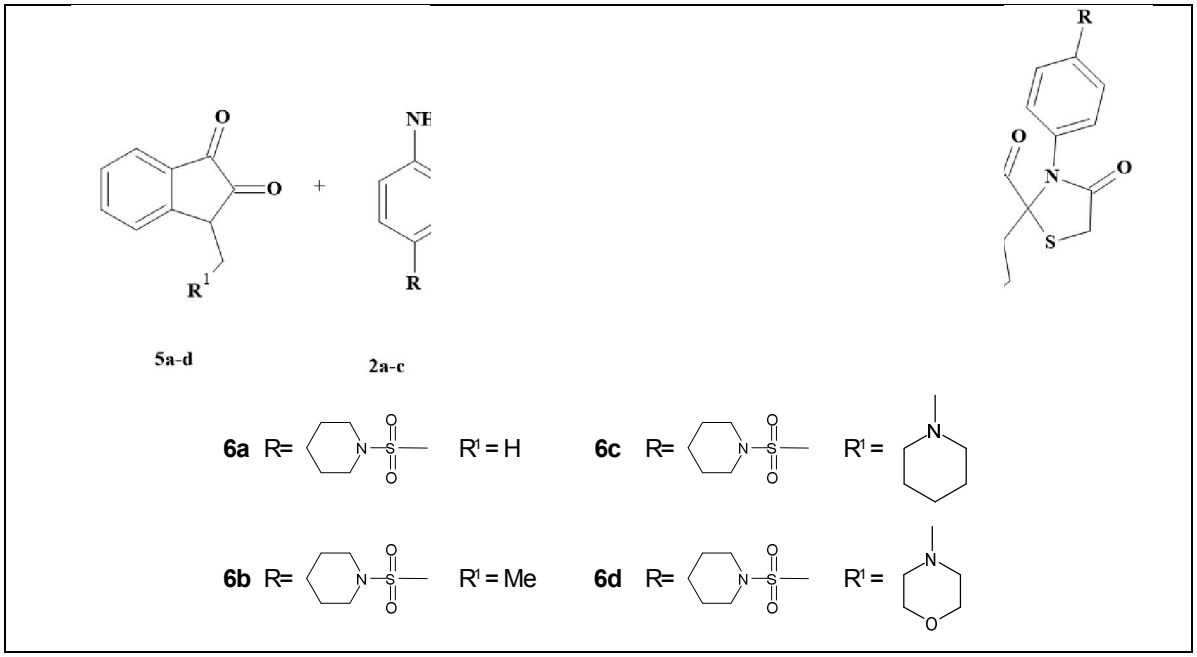
hazardous and highly flammable solvents, use of expensive catalysts, high reaction temperatures, low yield of products, and the use of costly equipment. To overcome these disadvantages and to develop an efficient and environmentally benign protocol, we report the MCR to synthesize polyfunctionalized spirothiazolidin-4-one derivatives by employing $TiCl_3(OTf)$/[bmim]Cl catalytic system.



Scheme 1: Synthesis of 1-thia-4-azaspiro[4.5]decan-3-one derivatives **4a–d** catalyzed by $TiCl_3(OTf)$/[bmim]Cl.



Scheme 2: Synthesis of spiro[indoline-3,2'-thiazolidine]-2,4'-dione derivatives

II. Results and Discussion

With the aforementioned aspects in mind, we have developed a $TiCl_3OTf$/[bmim]Cl catalyzed approach for the synthesis of 1-thia-4-azaspiro[4.5]decan-3-ones (4a–d) (Scheme 1) and spiro[indoline-3,2'-thiazolidine]-2,4'-diones (6a–d) (Scheme 2). The reaction of cyclohexanone **1** (1.0 mmol), 4-(piperidin-1-yl sulfonyl) aniline **2a** (1.0 mmol), thioglycolic acid **3** (1.0 mmol) selected as a model reaction to optimize the reaction parameters (Scheme 1). The optimization of the reaction conditions for the synthesis of 1-thia-4-azaspiro[4.5]decan-3-ones (**4a**) was investigated by using 15 mol % $TiCl_3OTf$ as a catalyst with different solvents and temperature conditions (Table 1). Initially, the model

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reaction was carried out in various traditionally used organic solvents. These solvents offered lower yield of the desired product **4a** (Entry 1-6). To further improve the efficiency of the reaction, ILs were explored as reaction media. The use of [bmim]OTf with provided a moderate yield of 58% (Entry 7), while [bmim]Cl improved to 63% (Entry 10). Further, optimization of temperature in [bmim]Cl observed that increasing the temperature led to an increase in the yield and a decrease in reaction time. Notably, [bmim]Cl showed better performance with $TiCl_3OTf$, producing the desired product in 95 % yield within 35 min at 90 °C (Entry 15). Thus, the optimal reaction conditions for this methodology were identified as $TiCl_3OTf$ (15 mol%) in [bmim]Cl (2 mmol) at 90 °C, providing the better yield (98 %) in the 35 minutes time.

TABLE 1: The effects of catalyst, solvent, and temperature on the yield of 4a*

Entry	Catalyst (15 mol%)	Solvent (5 cm^3)/ IL (2 mmol)	Temperature (°C)	Time (min)	Yield (%)[a]
1.	-	C_2H_5OH	Reflux	90	20
2.	$TiCl_3OTf$	C_2H_5OH	Reflux	90	45
3.	$TiCl_3OTf$	2-PrOH	Reflux	90	27
4.	$TiCl_3OTf$	CH_3OH	Reflux	90	30
5.	$TiCl_3OTf$	CH_3CN	Reflux	90	35
6.	$TiCl_3OTf$	CH_2Cl_2	Reflux	90	28
7.	$TiCl_3OTf$	[bmim]OTf	60	90	58
8.	$TiCl_3OTf$	[bmim]Br	60	80	55
9.	$TiCl_3OTf$	[bmim]PF_6	60	70	55
10.	$TiCl_3OTf$	[bmim]Cl	60	70	63
11.	$TiCl_3OTf$	[bmim]Cl	70	65	67
12.	$TiCl_3OTf$	[bmim]Cl	75	55	88
13.	$TiCl_3OTf$	[bmim]Cl	80	50	88
14.	$TiCl_3OTf$	[bmim]Cl	85	45	93
15.	$TiCl_3OTf$	[bmim]Cl	90	35	95
16.	$TiCl_3OTf$	[bmim]Cl	95	35	95
17.	$TiCl_3OTf$	[bmim]Cl	100	35	95

[a]Isolated yield; *Reaction conditions: cyclohexanone 1 (1.0 mmol), 4-(piperidin-1-yl sulfonyl) aniline 2a (1.0 mmol), thioglycolic acid 3 (1.0 mmol), 15 mol % catalyst/2 mmol IL

Under the above optimized reaction conditions, a series of structurally diverse 1-thia-4-azaspiro[4.5]decan-3-ones (4a–d) and spiro[indoline-3,2'-thiazolidine]-2,4'-diones (6a–d) were successfully synthesized to evaluate the efficiency of the catalytic system (Table 2). All the reactions progressed smoothly, producing the desired products in good to excellent yields within 35-65 min. Overall, the successful synthesis of structurally diverse derivatives with varying substituents demonstrates the versatility, and efficiency of the $TiCl_3OTf$.[bmim]Cl catalytic system for the synthesis of spiro heterocyclic moieties.

TABLE 2: Synthesis of 1-thia-4-azaspiro[4.5]decan-3-one (4a-d) and spiro[indoline-3,2'-thiazolidine]-2,4'-diones (6a–d)derivatives under optimized conditions.

Compound	Time (min)	Yield (%)[a]	Melting Points (°C) [15]
4a	35	95	123-125
4b	40	95	132-134
4c	35	94	126-128
4d	45	94	110-112
6a	30	96	135-137
6b	30	95	109-111

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6c	65	96	140-142
6d	65	93	149-151

*Reaction conditions: cyclohexanone **1** or **5a–d** (1.0 mmol), 4-(piperidin-1-yl sulfonyl) aniline **2a** (1.0 mmol), thioglycolic acid **3** (1.0 mmol), 15 mol % catalyst/2 mmol IL; [a] isolated yield.

Synergistic Effects of [bmim]Cl and $TiCl_3$(OTf)

This reaction proceeds at 90° C temperature. The ionic liquid [bmim]Cl is liquid at room-temperature, which is composed of an imidazolium cation and chloride anion. It has low vapor pressure, makes it ecofriendly and suitable for green chemistry synthesis. It has high thermal stability up to 250-300 °C, enabling synthesis at elevated temperature. High polarity and hydrogen bonding ability of this ionic liquid makes it better choice for organic synthesis. $TiCl_3$(OTf)/[bmim]Cl is a strong Lewis acid catalyst which activates carbonyl carbon and enhances the rate of reaction. It plays a key role to carry out the multicomponent reaction successfully. It exhibits dual anion effect due to the presence of both chloride and triflate (OTf^-) which enhances its catalytic activity through improved coordination flexibility. Compared to the $TiCl_4$ and other titanium chloride-based systems it has high moisture tolerance. [bmim]Cl and $TiCl_3$(OTf) has better synergic effect. Ionic liquid stabilizes the metal center and activate intermediates, which improves rate of reaction and the yield of desired products. It creates a homogeneous catalytic system due to this both organic and inorganic catalyst dissolve effectively in it. This ionic liquid-catalyst system employed in this methodology is recyclable multiple times without loss in its activity. It eliminates the use of traditionally used organic solvents. This methodology offers mild reaction conditions, economical and environmentally friendly. It promotes one pot multicomponent synthesis of biologically active heterocycles. This catalytic system activates functional groups simultaneously.

III. EXPERIMENTAL SECTION

All the Chemicals were purchased from Sigma Aldrich and Merck. Melting points were recorded using an Electrothermal 9100 apparatus and are uncorrected. ^{1}HNMR spectra were recorded in DMSO-d6 on a Bruker Avance DRX 300 MHz instrument using TMS as an internal standard. All the IR spectra were taken on a Bruker 870 FTIR spectrometer (ATR, Alpha). The purity of compounds and progress of all the reactions were monitored by TLC.

Synthesis of 1-thia-4-azaspiro[4.5]decan-3-ones (4a–d) and spiro[indoline-3,2'-thiazolidine]-2,4'-diones (6a–d)

A mixture of cyclic ketone **1** or **5a–d** (1.0 mmol), aromatic amine **2a–d** (1.0 mmol), thioglycolic acid **3** (1.0 mmol), and $TiCl_3$(OTf)/[Bmim]Cl (15 mol % catalyst and 2 mmol IL) was heated at 90 **°C** with constant stirring for 35-65 minutes. After the completion of the reaction, the reaction mixture was cooled at room temperature, and the product was extracted by ethyl acetate. The catalytic system stays in non-volatile ionic liquid phase and is recyclable up to four runs efficiently.

Spectral Data for Selected Compounds

4a: 4-[4-(Piperidin-1-ylsulfonyl)phenyl]-1-thia-4-azaspiro[4.5]decan-3-one: colour: colorless, melting point: 123–125 °C.[15] IR spectrum (ν, cm^{-1}): 3085, 2995, 2677, 1715 (C=O stretch), 1680, 1312, 1161. ^{1}H NMR (δ, ppm, J, Hz): 1.41–1.46 (4H, m, $2CH_2$); 1.59–1.63 (8H, m, $4CH_2$); 1.91 (4H, t, J = 5.0, $2CH_2$); 2.95 (4H, t, J = 5.0, $2CH_2$); 3.61 (2H, s, CH_2CO); 7.02 (2H, d, J = 8.5); 7.49 (2H, d, J = 8.5). ^{13}C NMR (δ, ppm: 21.78 (CH_2); 23.58 ($2CH_2$); 25.31 (CH_2); 25.50 ($2CH_2$); 32.03 ($2CH_2$); 38.55 ($2CH_2$); 46.91 (CH_2); 62.31 (spiro C); 117.12 (2CH); 129.93 (2CH); 146.33 (C); 176.37 (C=O).

4b: 4-[4-(Morpholin-4-ylsulfonyl)phenyl]-1-thia-4-azaspiro[4.5]decan-3-one: colour: colour: colorless; melting point: 132–134 °C.[15] IR spectrum (ν, cm^{-1}): 3133, 2942, 2633, 1710, 1680, 1320, 1189; ^{1}H NMR (δ, ppm, J, Hz):1.51–1.53 (2H, m, CH_2); 1.69–1.71 (4H, m, $2CH_2$); 1.81 (4H, t, J = 5.5, $2CH_2$); 3.01 (4H, t, J = 4.5, $2CH_2N$); 3.73 (2H, s, CH_2CO); 3.83 (4H, t, J = 4.5, $2CH_2O$); 6.76 (2H, d, J = 8.5); 7.53 (2H, d, J = 8.5); ^{13}C NMR (δ, ppm: 23.10

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(CH_2); 25.47 ($2CH_2$); 32.12 ($2CH_2$); 37.82 ($2CH_2$); 46.89 (CH_2); 61.19 (spiro C); 66.13 ($2CH_2$); 114.49 (2CH); 128.03 (C); 130.27 (2CH); 150.48 (C); 176.94 (C=O).

6a: 1-Methyl-3'-[4-(piperidin-1-ylsulfonyl)phenyl]spiro [indoline-3,2'-thiazolidine]-2,4'-dione: Colour: pale-yellow, melting point 135–137 °C.[15] IR spectrum (ν, cm^{-1}): 3066, 2978, 2631, 1773, 1728, 1668, 1340, 1169. ^{1}H NMR (δ, ppm, J, Hz):1.21–2.48 (6H, m, $3CH_2$); 3.09 (4H, t, J = 7.0, $2CH_2N$); 3.62 (3H, s, CH_3); 4.43 (1H, d, J = 15.5) and 4.42 (1H, d, J = 15.5, 5'-CH_2); 6.88 (1H, d, J = 7.5, Ar-H); 6.88–7.10 (1H, m, Ar-H); 7.31–7.66 (2H, m, Ar-H); 7.79 (2H, d, J = 8.5, Ar-H); 7.83 (2H, d, J = 8.5, Ar-H); ^{13}C NMR (δ, ppm: 22.88 (CH_2); 25.22 (CH_2); 33.43 (CH_3); 44.3 (CH_2); 47.21 (CH_2); 68.92 (spiro C); 109.37 (CH); 119.11 (CH); 123.93 (CH); 126.09 (CH); 128.33 (CH); 129.30 (CH); 131.77 (C); 132.93 (C); 139.33 (C); 143.28 (C); 167.89 (C=O); 175.71 (C=O).

IV. CONCLUSION

We have introduced a new methodology to synthesize spiro-thiazolidinones by using $TiCl_3$(OTf) catalyst with IL. The $TiCl_3$(OTf)/[bmim]Cl catalytic system shows a synergistic combination of IL-mediated stabilization of intermediates and activation of functional groups, providing an ecofriendly, recyclable, and efficient catalytic medium for multicomponent reactions to synthesize bioactive heterocyclic synthesis.

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