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RESEARCH ARTICLE

Titanium trichloride-catalysed cyclocondensation: synthesis of 2-mercaptoquinoline substituted 1,2,3,4-tetrahydropyrimidinones

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2-Mercaptoquinoline substituted 1,2,3,4-tetrahydropyrimidinones have been efficiently synthesized in high yields in a short reaction time using $TiCl_3$ as catalyst, in THF solvent. The mechanism is proposed based on the presence of Lewis acid catalyst in Biginelli condensation.

Keywords: 2-Mercaptoquinoline; Multicomponent reaction; $TiCl_3$; Biginelli reaction; 3,4-Dihydropyrimidinone

1. Introduction

Multicomponent reactions (MCRs) are of increasing importance in organic and medicinal chemistry [1–3]. In times where a premium is put on speed, diversity, and efficiency in the drug discovery process [4, 5], MCR strategies offer significant advantages over conventional linear type synthesis [1–3]. In such reactions, three or more reactants come together in a single reaction vessel to form new products that contain portions of all the components [1–3]. The search and discovery for new MCRs, on one hand [6], and the full exploitation of already known multicomponent reactions, on the other hand, are therefore of considerable current interest.

One such MCR that belongs to the latter category is the Venerable Biginelli dihydropyrimidine synthesis. In 1893, Italian chemist Pietro Biginelli reported the acid catalysed cyclocondensation reaction. The product offered by Biginelli three-component protocol is particularly attractive, since the resulting dihydropyrimidinones (DHPMs) derivatives exhibit pharmacological activities as calcium channel blockers, antihypertensive agents, α-1a-antagonists, and neuropeptide Y(NPY) antagonists [7–10]. Several biologically active marine alkaloids also contain dihydropyrimidinones-5-carboxylate core [11]. Most notable among them are batzelladine alkaloids, which have been found to be potent HIV gp-120-CD4

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inhibitors [12–14]. Due to the great biological importance of DHPMs, Biginelli reaction has received renewed interest and strategies, for the synthesis of DHPM molecule have varied from one-pot to multistep approaches [15, 16]. Initially one-pot offered low yield – (20–50%) yields. Subsequent multistep syntheses produced somewhat higher yields but lack the simplicity of the one-pot – one-step synthesis [17, 18].

In recent years, several procedures have been reported, including the use of various Lewis acid such as BF_3OEt_2, $PhB(OH)_2$ [19, 20] lithium salts [21–23], transitional metal complexes [24–27], zinc chloride [28], cadmium chloride [29], bismuth [30, 31] and indium [32, 33] chloride, lanthanide compounds [34, 35] iodotrimethyl silane [36] and trimethylsilyl triflate [37], and polyphosphate ester (PPE) [38] or reusable polyaniline bismoclite complex [39] as catalyst. Recently, it was reported that the acidic clay montmorillonite KSF [40] could also catalyse Biginelli three-component one-pot cyclocondensation reaction.

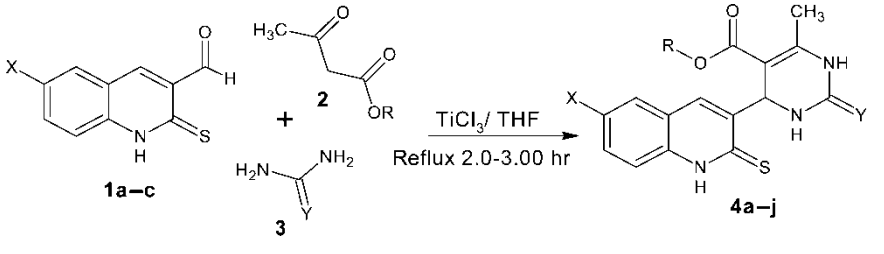
Further, titanium (III) chloride has emerged as an efficient Lewis acid catalyst in organic chemistry such as Pinacol coupling [41] pyrollidine syntheses [42], Claisen rearrangements [43] and asymmetric Aldol reaction [44] etc. In view of this and in continuation of our work on synthesis of fused heterocycles herein, we wish to present the synthesis 2-mercaptoquinoline substituted dihydropyrimidinones by Biginelli condensation in the presence of $TiCl_3$ catalyst.

On the other hand, quinoline and their derivatives are important heterocyclic system due to their bactericidal, antitumour, anti-inflammatory and antifungicidal properties [45]. Likewise, dihydropyrimidinones flaked by substituted 2-mercaptoquinoline moieties may also be expected to possess a wide range of biological activities.

2. Results and discussion

A facile three-component Biginelli's one-pot cyclocondensation takes place between 3-formyl-2-mercaptoquinoline **1a**, β-ketoester **2** and (thio)urea **3** using 10 mol% of $TiCl_3$ in THF solvent at ambient temperature afforded 2-mercaptoquinoline substituted 3,4-tetrahydropyrimidine derivatives **4a–j** (scheme 1). In each experiments molar ratio 1:1:1.5 of the three components, **1, 2** and **3** were used as reactants [46]. The method is very simple and it can be used derivatives of 3-formyl-2-mercaptoquinoline **1**, different β-ketoester **2,** and (thio)urea **3** depending on X, Y, R groups to prepare various compounds (table 1). Solubility of impurities in THF solvent is another advantage of this method. Reactions were usually carried out for 2 to 3 h in high yields (table 1).

The results suggest a mechanism wherein the *in situ* acylimine intermediate [47] generated from the formylquinoline and urea is activated, either by protonation or coordination with titanium, wherein titanium halide act as a Lewis acid. This was followed by the



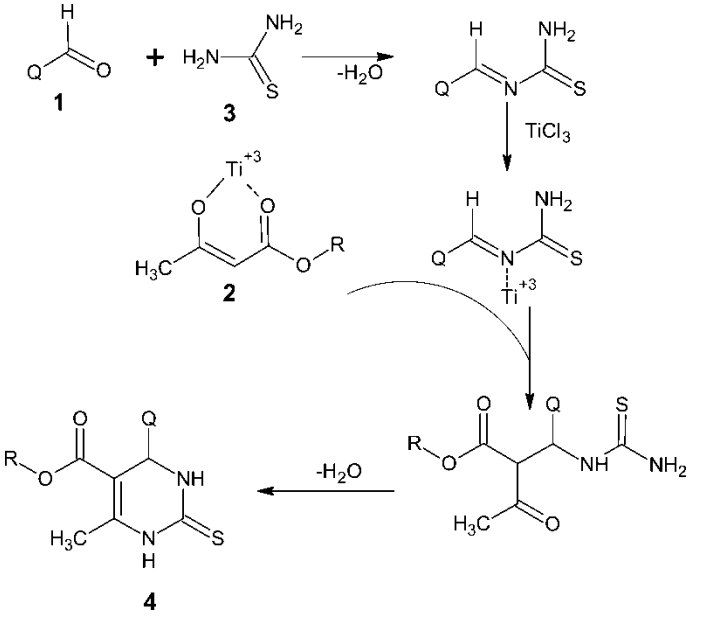
SCHEME 1. Synthesis of ethyl/methyl 6-methyl-4-(2-sulfanylquinolin-3-yl)-2-(thio)oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate derivatives.

Table 1. $TiCl_3$-catalysed efficient synthesis of ethyl/methyl 6-methyl-4-(2-sulfanylquinolin-3-yl)-2-(thio)oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate derivatives.

Compound[a]	X	R	Y	Time (h)	Yield (%)[b]	m.p. exp	C Calcd (Found)	H Calcd (Found)	N Calcd (Found)
4a	H	C_2H_5	S	2.0	94	239	56.80 (56.40)	4.77 (5.18)	11.69 (12.01)
4b	CH_3	C_2H_5	S	2.5	91	253	57.88 (57.49)	5.13 (5.52)	11.25 (11.63)
4c	OCH_3	C_2H_5	S	3.0	92	267	55.51 (55.11)	4.92 (5.31)	10.79 (11.18)
4d	H	CH_3	O	2.0	93	225	58.34 (57.93)	4.59 (4.98)	12.76 (13.15)
4e	CH_3	CH_3	O	2.0	92	233	59.40 (59.01)	4.99 (5.38)	12.24 (12.62)
4f	OCH_3	CH_3	O	2.5	94	251	56.81 (56.48)	4.77 (5.13)	11.69 (12.07)
4g	H	CH_3	S	2.5	93	198	55.63 (55.22)	4.38 (4.75)	12.16 (12.55)
4h	CH_3	CH_3	S	3.0	94	210	56.80 (56.47)	4.77 (5.13)	11.69 (12.11)
4i	OCH_3	CH_3	S	3.0	90	258	54.38 (53.13)	4.56 (4.91)	11.19 (11.53)
4j	H	C_2H_5	O	2.0	94	215	59.46 (59.01)	4.99 (5.33)	12.24 (12.61)

[a]Products were characterized by IR, ^{1}H NMR and mass spectral data.
[b]Isolated and unoptimized yields.

addition of titanium enolate derived from ethyl acetoacetate cyclization and dehydration (scheme 2). Titanium (III) chlorides can trap nitrogen atoms of imines or tertiary amines that decompose readily in the presence of water, as well as highly effective for the activation of imines.



SCHEME 2. The suggested pathway to the Biginelli reaction.

Structural elucidation of the newly synthesised compounds was established on the basis of their IR, ^{1}H NMR and mass spectral data. The formation of **4a–j** was evident from the complete disappearance of absorption bands in the region 1635–1641 cm^{-1} and singlet at 10.31 ppm due to –CHO group from **(1a–c)**. It also showed an intense peak around 3246–3250 cm^{-1} due to NH group and the tautomeric form of C=S appears at 760 cm^{-1}. ^{1}H NMR spectra exhibited peaks for the presence of NH group δ9.19 (s, 1H, NH) and 7.42 (s, 1H, NH). In addition, the aromatic quinoline protons appeared around 7.19–8.68 ppm and SH group appears at $\delta = 11.21$ ppm. The molecular ion peak at 360 [M + H$^+$] confirms the formation of compound **4a**.

3. Conclusion

In conclusion, we have developed a simple and efficient method for a one pot three component one pot procedure for the synthesis of 2-mercaptoquinoline substituted 1,2,3,4 tetrahydropyrimidine-5 carboxylate using $TiCl_3$ as catalyst in THF solvent.

4. Experimental section

Melting points were determined in on open capillary and uncorrected. Infrared spectra were recorded using KBr disc on Shimadzu FTIR-8400S spectrophotometer and expressed in cm^{-1}. The ^{1}H NMR spectra (300 MHz) were recorded on a Bruker supercon FT NMR instrument using TMS as internal standard and mass spectra on a Jeol JMS-D 300 mass spectrometer operating at 70 eV. Standard and peak multiplicities are designated as follows: s, singlet; d, doublet; dd, doublet of doublets; t, triplet; br, broad signal; m, multiplet. Elemental microanalyses were obtained in a CHN analyzer and gave results for the elemental stated with ±0.4% of the theoretical values. Purity of the compounds was checked by TLC on silica gel.

4.1 *General procedure for the synthesis of ethyl 6-methyl-4-phenyl-2-thioxo-1, 2,3,4-tetrahydropyrimidine-5-carboxylate*

Solution of aromatic aldehyde (2 mmol), ethyl acetoacetate (2 mmol) and urea (2.5 mmol) in THF (10 mL) were taken in a 100 mL round bottom flask. To this 10 mol% $TiCl_3$ (10 mmol%) was added with constant stirring and the reaction mixtures was refluxed for 2–3 h at 70–75 °C. The reaction mixture was cooled, poured into crushed ice. The crude product thus obtained was filtered, washed with water and 40% of ethanol. The product obtained was dried and recrystalized from suitable solvent. All these compounds were characterized by different physico-chemical techniques such as elemental analysis, mp, IR, ^{1}H NMR and mass spectral data.

4.1.1 Data for compounds of ethyl 6-methyl-4-phenyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate. IR (cm^{-1}): 3246, 3111, 2985, 2956, 2931, 1730, 1703, 1514, MS $m/z(\%) = 276$ [M + H$^+$]. ^{1}H NMR (DMSO-d6): δ 9.14 (s, 1H, NH), 7.67 (s, 1H, NH), 7.15 (d, $J = 8.6$ Hz, 2H); 6.88 (d, $J = 8.7$ Hz, 2H), 5.10 (d, $J = 3.2$ Hz, 1H), 3.98 (q, $J = 7.1$ Hz, 2H, OCH_2), 1.10 (t, $J = 7.1$ Hz, 3H, CH_3).

4.2 *General procedure for the synthesis of compound 4a–k*

Solution of 3-formyl-2-mercaptoquinoline **1a–c** (2 mmol), ethyl acetoacetate **2** (2 mmol) and urea **3** (2.5 mmol) in THF (10 mL) were taken in a 100 mL round bottom flask. To this 10 mol% $TiCl_3$ (10 mmol%) was added with constant stirring and the reaction mixtures was refluxed for 2–3 h at 70–75 °C. The reaction mixture was cooled, poured into crushed ice. The crude product thus obtained was filtered, washed with water and 40% of ethanol. The product obtained was dried and recrystalized from a suitable solvent. All these compounds were characterized by different physico-chemical techniques such as elemental analysis, mp, IR, ^{1}H NMR and mass spectral data.

4.3 *Data for compounds 4a–j*

4.3.1 Ethyl 6-methyl-4-(2-sulfanylquinolin-3-yl)-2-thioxo-1,2,3,4-tetrahydro-pyrimidine-5-carboxylate (4a). Yellow solid, $C_{17}H_{17}N_3O_2S_2$; IR (cm^{-1}): 3246, 2985, 1730, 1703, 1651, 760, MS $m/z(\%) = 360$ [M + H$^+$]. ^{1}H NMR (DMSO-d$_6$): δ 1.12 (t, $J = 7.5$ Hz, 3H, CH_3). 2.28 (s, 3H, CH_3), 3.98 (q, $J = 7.5$ Hz, 2H, OCH_2), 7.14–7.75 (m, 5Har), 7.67 (br, s, 1H, NH); 9.22 (br, s, 1H, NH), 11.21 (s, 1H, SH).

4.3.2 Ethyl 6-methyl-4-(6-methyl-2-sulfanylquinolin-3-yl)-2-thioxo-1,2,3,4-tetrahydro-pyrimidine-5-carboxylate (4b). Yellow solid, $C_{18}H_{19}N_3O_2S_2$; IR (cm^{-1}): 3248 , 2981, 1731, 1703, 1649, 761 MS $m/z(\%) = 373.50$ [M + H$^+$]. ^{1}H NMR (DMSO-d$_6$): δ 1.31 (t, $J = 7.5$ Hz, 3H, CH_3). 2.28 (s, 3H, CH_3), 2.35 (t, $J = 7.5$ Hz, 3H, CH_3), 4.03 (q, $J = 7.5$ Hz, 2H, OCH_2), 7.14–7.75 (m, 4Har), 7.67 (br, s, 1H, NH); 9.22 (br, s, 1H, NH), 11.21 (s, 1H, SH),

4.3.3 Ethyl 4-(6-methoxy-2-sulfanylquinolin-3-yl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (4c). Yellow solid. $C_{18}H_{19}N_3O_3S_2$; IR (cm^{-1}): 3248, 2981, 1731, 1703, 1649, 760, MS $m/z(\%) = 390.10$ [M + H$^+$]. ^{1}H NMR (DMSO-d$_6$): δ 1.11 (t, $J = 7.5$ Hz, 3H, CH_3). 2.28 (s, 3H, CH_3), 2.55 (s, 3H, OCH_3), 4.03 (q, $J = 7.5$ Hz, 2H, OCH_2), 7.74–8.12 (m, 4.H), 7.65 (br, s, 1H, NH); 9.19 (br, s, 1H, NH), 11.01 (s, 1H, SH),

4.3.4 Methyl 6-methyl-2-oxo-4-(2-sulfanylquinolin-3-yl)-1,2,3,4-tetrahydropyrimidine-5-carboxylate (4d). Pale yellow solid. $C_{16}H_{15}N_3O_3S$; IR (cm^{-1}): 3253, 2983, 1735, 1710, 1650, 761, MS $m/z(\%) = 329.57$ [M + H$^+$]. ^{1}H NMR (DMSO-d$_6$): δ 1.12 (t, $J = 7.5$ Hz, 3H, CH_3). 2.28 (s, 3H, CH_3), 7.16–8.01 (m, 5Har), 7.77 (br, s, 1H, NH); 9.13 (br, s, 1H, NH), 11.21 (s, 1H, SH).

4.3.5 Methyl 6-methyl-2-oxo-4-(6-methyl-2-sulfanylquinolin-3-yl)-1,2,3,4-tetrahydro-pyrimidine-5-carboxylate (4e). Pale yellow solid. $C_{16}H_{15}N_3O_3S$; IR (cm^{-1}): 3251, 2990, 1731, 1703, 1655, 768, MS $m/z(\%) = 343.40$ [M + H$^+$]. ^{1}H NMR (DMSO-d$_6$): δ 1.15 (t, $J = 7.5$ Hz, 3H, CH_3). 2.21 (s, 3H, CH_3), 2.35 (t, $J = 7.5$ Hz, 3H), 7.69–8.12 (m, 4Har), 7.67 (br, s, 1H, NH); 9.13 (br, s, 1H, NH), 11.01 (s, 1H, SH).

4.3.6 Methyl 6-methyl-2-oxo-4-(6-methoxy-2-sulfanylquinolin-3-yl)-1,2,3,4-tetrahydro-pyrimidine-5-carboxylate (4f). Pale yellow solid. $C_{17}H_{17}N_3O_3S$; IR (cm^{-1}): 3255, 2989, 1730, 1705, 1645, 761, MS $m/z(\%) = 359.40$ [M + H$^+$]. ^{1}H NMR (DMSO-d$_6$): δ 1.12 (t,

$J = 7.5$ Hz, 3H, CH_3). 2.28 (s, 3H, CH_3), 2.6 (s, 3H), 7.8–8.11 (m, 4H), 7.77 (br, s, 1H, NH); 9.13 (br, s, 1H, NH), 11.11 (s, 1H, SH).

4.3.7 Methyl 6-methyl-4-(2-sulfanylquinolin-3-yl)-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (4g). Pale yellow solid. $C_{16}H_{15}N_3O_2S_2$; IR (cm^{-1}): 3258, 2990, 1745, 1700, 1653, 764, MS $m/z(\%) = 344.50$ $[M + H^+]$.^{1}H NMR (DMSO-d_6): δ 1.06 (t, $J = 7.5$ Hz, 3H, CH_3). 2.31 (s, 3H, CH_3), 7.51–7.5 (m, 5Har), 7.77 (br, s, 1H, NH); 9.22 (br, s, 1H, NH), 10.79 (s, 1H, SH).

4.3.8 Methyl 6-methyl-4-(6-methyl-2-sulfanylquinolin-3-yl)-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (4h). Pale yellow solid. $C_{17}H_{17}N_3O_2S_2$; IR (cm^{-1}): 3258, 2990, 1745, 1700, 1653, 762, MS $m/z(\%) = 359.46$ $[M + H^+]$. ^{1}H NMR (DMSO-d_6): δ 1.12 (t, $J = 7.5$ Hz, 3H, CH_3). 2.151 (s, 3H, CH_3), 2.35 (t, $J = 7.5$ Hz, 3H), 7.69–8.12 (m, 4H), 7.67 (br, s, 1H, NH); 9.13 (br, s, 1H, NH), 11.01 (s, 1H, SH).

4.3.9 Methyl 6-methyl-4-(6-methoxy-2-sulfanylquinolin-3-yl)-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (4i). Pale yellow solid. $C_{17}H_{17}N_3O_3S_2$; IR (cm^{-1}): 3255, 2989, 1730, 1705, 1645, 761, MS $m/z(\%) = 360.11$ $[M + H^+]$. ^{1}H NMR (DMSO-d_6): δ 1.01 (t, $J = 7.5$ Hz, 3H, CH_3). 2.22 (s, 3H, CH_3), 2.51 (s, 3H, CH_3), 7.8–8.11 (m, 4Har), 7.75 (br, s, 1H, NH); 9.22 (br, s, 1H, NH), 11.12 (s, 1H, SH).

4.3.10 Ethyl 6-methyl-2-oxo-4-(2-sulfanylquinolin-3-yl)-1,2,3,4-tetrahydropyrimidine-5-carboxylate (4j). Pale yellow solid. $C_{17}H_{17}N_3O_3S$; IR (cm^{-1}): 3246 , 2985, 1730, 1703, 1651, 760, MS $m/z(\%) = 343.51$[$M + H^+$]. δ 1.12 (t, $J = 7.5$ Hz, 3H, CH_3). 2.28 (s, 3H), 3.98 (q, $J = 7.5$ Hz, 2H, OCH_2), 7.14–7.75 (m, 5Har), 7.67 (br, s, 1H, NH); 9.22 (br, s, 1H, NH), 11.21 (s, 1H, SH),

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