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Stereoselective synthesis of 3,4-dihydro-7-nitrocoumarins via isocyanide-based multicomponent reaction

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ARTICLE INFO

Article history:

Received 10 December 2011

Accepted after revision 23 January 2012

Available online 28 February 2012

Keywords:

Isocyanide

Multicomponent reaction

3,4-Dihydro-7-nitrocoumarin derivatives

ABSTRACT

A stereoselective method for the synthesis of 3,4-dihydro-7-nitrocoumarin derivatives via an isocyanide-based four-component reaction is described. The reaction can be carried out as a simple one-pot protocol in good yields without using any catalyst or activator at room temperature in short reaction time.

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1. Introduction

The coumarin nucleus is well represented in natural products (neoflavonoids), and in a variety of pharmacologically active compounds [1,2]. Some compounds containing the 3,4-dihydrocoumarin nucleus possess important biological activities [3], for example, inhibitors of aldose reductase [4] and protein kinases [5], and antiherpetic [6]. Therefore, many synthetic methods for 3,4-dihydrocoumarins have been reported up to the present [1–16]. But, the most of these methods suffer from disadvantages such as lack of substrate generality and the use of a large excess of expensive transition metal catalysts such as Pd(OAc)₂ [7], Y(OTf)₃ [8], Yb(OTf)₃ [9], Ru(III) [10] and Cr(CO)₅ [11] or corrosive organic acid such as CF₃CO₂H [12] and require harsh reaction conditions. Consequently, a mild and simple procedure is strongly desired.

Our literature survey showed that the most common method for the synthesis of coumarins involves the hydroarylation of cinnamic acids with phenols, Lewis acid-mediated reaction of highly activated phenols with

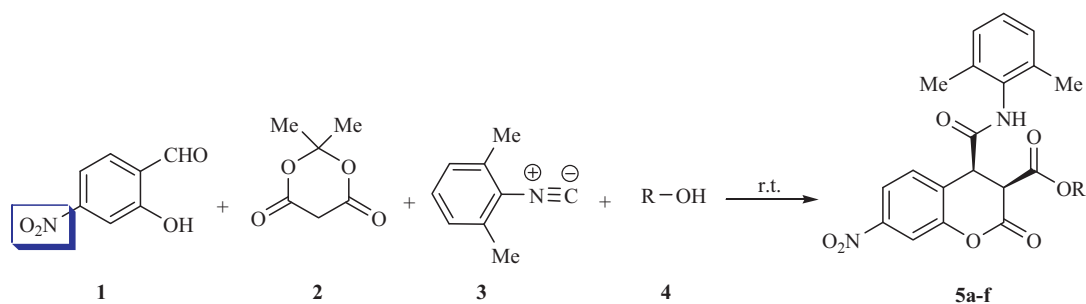
acrylonitrile (Pechmann method), and activated 2-hydroxy-benzaldehyde with CH-acid compounds (Knoevenagel method) limited to only electron-donating groups on aromatic ring of substrate. There are few examples in which the coumarin-bearing strong electron-withdrawing substituents such as NO₂—only one report in the literature [3]. It is interesting to note that in the reported method when the NO₂ group was positioned on acrylic acid, the reaction time in the presence of methanesulfonic acid as a catalyst (2 equiv.) is very long (37 days!). Therefore, our study was motivated to investigate the preparation of coumarin with NO₂ substituent via multicomponent reactions.

Due to atom economy, simplicity, and amenability to automated synthesis, multicomponent condensation reactions (MCRs) have an advantageous position among other reactions. MCRs are powerful tools in the modern drug discovery process and allow the fast, automated and high throughput generation of organic compounds. Therefore, the use of multicomponent reaction instead of multistep reaction is significantly increasing by academic research [17–20].

As part of our research on the development of new synthetic methods in heterocyclic chemistry [21–26], here we report a stereoselective method to the synthesis of 3,4-dihydro-7-nitrocoumarin derivatives **5a–f** via a

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Scheme 1. Synthesis of 3,4-dihydrocoumarin derivatives.

four-component condensation reaction of 2-hydroxy-4-nitrobenzaldehyde, **1**, Meldrum's acid, **2**, and 2,6-Dimethylphenyl isocyanide, **3**, in the presence of aromatic or aliphatic alcohols, **4** at room temperature (Scheme 1).

2. Results and discussion

The reaction did not require any optimization; as indicated in Fig. 1, treatment of 2,6-dimethylphenyl isocyanide and 2-hydroxy-4-nitrobenzaldehyde with Meldrum's acid in the presence of ethanol or methanol (as a solvent and reagent) or in CH_2Cl_2 in the case of solid alcohol at room temperature led to the formation of the corresponding 3,4-dihydro-7-nitrocoumarin derivatives in good yields.

The structures of compounds **5a–f** were deduced from their IR, ^1H NMR, ^{13}C NMR and mass spectroscopic data.

For example, the ^1H NMR spectrum of **5a** exhibited three singlets at 2.07, 2.25 (2 CH_3) and 3.78 (O CH_3), two doublets at 4.20 ($^3J = 5.9$ Hz, CHCONH) and 4.57 ($^3J = 5.9$ Hz, CHCOO), two multiplets at 6.97–7.19 (4CH arom), 8.03–8.14 (2CH arom) and a broad singlet at 11.54 (NH). The ^1H decoupled ^{13}C NMR spectrum of **5a** showed 20 distinct resonances, and partial assignment of these resonances is given in experimental section. The mass spectra of these compounds displayed molecular ion peaks at the appropriate m/z values.

To explore the scope and limitation of this versatile reaction, we have examined various aromatic and aliphatic alcohols in the presence of 2-hydroxy-4-nitrobenzaldehyde, Meldrum's acid, and 2,6-dimethylphenyl isocyanide in dichloromethane at room temperature. As indicated Fig. 1, the reaction proceeded efficiently and led to 3,4-dihydro-7-nitrocoumarin derivatives **5a–f** in good yields.

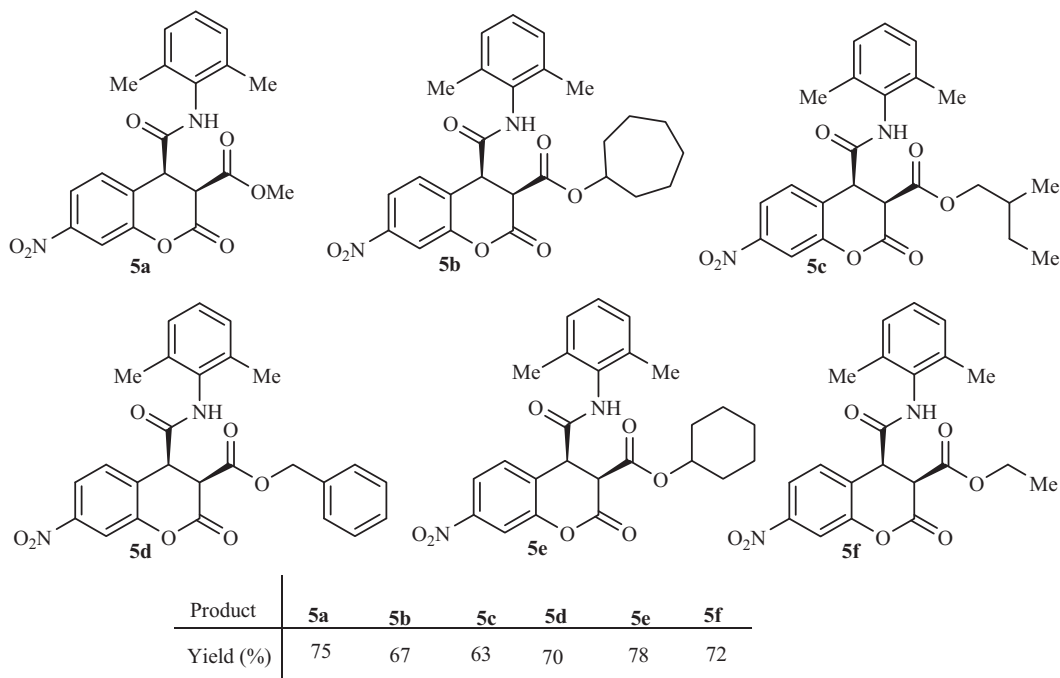
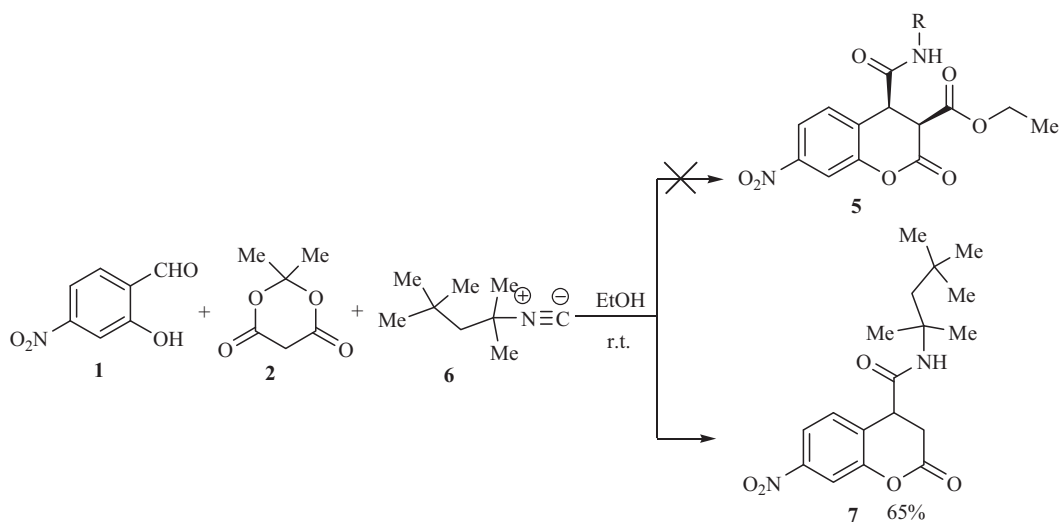


Fig. 1. Synthesis of 3,4-dihydrocoumarins from 2-hydroxy-4-nitrobenzaldehyde, Meldrum's acid, and 2,6-dimethylphenyl isocyanide in the presence of different alcohols.



Scheme 2. Use of 1,1,3,3-tetramethylbutyl isocyanide instead of 2,6-dimethylphenyl isocyanide.

It is important to note that compounds **5a–f** have two stereogenic centers, and therefore, two pairs of diastereoisomers are expected. The relative configurations of product can be determined. The ^1H NMR and ^{13}C NMR spectra of the crude reaction mixture showed that the reaction is stereoselective and only *cis* diastereoisomer is produced. For example, in the ^1H NMR of **5a**, the coupling constant (3J) of two vicinal hydrogens at δ_{H} (ppm) = 4.20 (CH-CONH) and 4.57 (CH-COO) is 5.9 Hz. However, why only the *cis* diastereoisomer was produced, is not clear for us but it may be because of the electron-withdrawing inductive effect of NO_2 group on salicylaldehyde that caused the production of the stable product. Product stability is probably due to hydrogen bonding between NH and C=O groups.

The versatility of this multicomponent reaction with respect to the 2,6-dimethylphenyl isocyanide **3** was also studied (Scheme 2). As indicated in Scheme 2, when 1,1,3,3-tetramethylbutyl isocyanide **6**, was used instead of 2,6-dimethylphenyl isocyanide **3**, the desired four component product **5** was not obtained, but the reaction progressed via a three component reaction and afforded the 3,4-dihydrocoumarin derivatives **7** (Scheme 2). It should be mentioned that only 1,1,3,3-tetramethylbutyl isocyanide could participate in this reaction.

3. Experimental

3.1. Materials and techniques

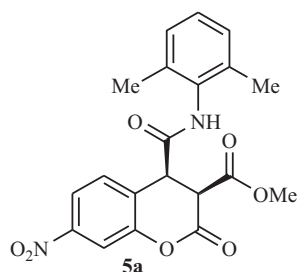
Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Mass spectra were recorded on a Finnigan-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^1H and ^{13}C NMR spectra were recorded on a Bruker DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz. NMR spectra were obtained on solution in dimethyl sulfoxide (DMSO) using tetramethylsilane (TMS) as internal standard. The chemicals used in this work were purchased from Merck and Fluka Chemical Companies.

3.2. Typical procedure for the preparation of 3,4-dihydrocoumarin **5a**

To a magnetically stirred solution of Meldrum's acid (0.14 g, 1 mmol), 2-hydroxy-4-nitrobenzaldehyde (0.16 g, 1 mmol) in methanol (10 mL) was added 2,6-dimethylphenyl (0.13 g, 1 mmol) and the reaction mixture stirred for 24 h at room temperature. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 2:1), the precipitate was washed with ethanol and the product **5a** was obtained as a white powder.

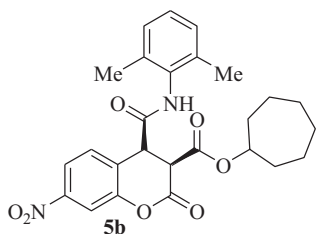
3.2.1. Methyl 4-(2,6-dimethylphenylcarbamoyl)-3,4-dihydro-7-nitro-2-oxo-2H-chromene-3-carboxylate (**5a**)

White powder (0.3 g, yield 75%), mp 163–166 °C. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3450, 3085, 2921, 1786, 1709, 1624, 1596. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} (ppm) 2.07 and 2.25 (6H, s, 2CH₃), 3.78 (3H, s, OCH₃), 4.20 (1H, d, 3J = 5.9 Hz, CHCONH), 4.57 (1H, d, 3J = 5.9 Hz, CHCOO), 6.97–7.19 (4H, m, CH arom), 8.03–8.14 (2H, m, CH arom), 11.54 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO- d_6): δ_{C} (ppm) 17.6, 17.8, 48.2, 52.7, 53.4 (OCH₃), 115.9, 122.6, 126.2, 127.9, 128.5, 128.6, 129.6, 130.2, 135.9, 136.5, 140.1 (11C-Ar), 161.4 (=C=O), 168.1 (CONH), 170.2, 174.3 (2C=O). MS, *m/z* (%): 398 (*M*⁺, 2), 364 (40), 336 (30), 291 (10), 192 (45), 147 (80), 118 (75), 91 (85), 77 (100), 39 (70). Anal. Calcd for C₂₀H₁₈N₂O₇: C, 60.30; H, 4.55; N, 7.03. Found: C, 60.87; H, 4.34; N, 6.78.



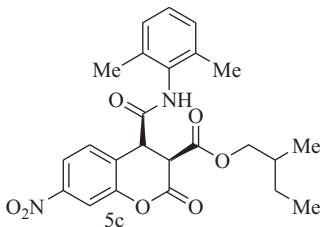
3.2.2. Cycloheptyl 4-(2,6-dimethylphenylcarbamoyl)-3,4-dihydro-7-nitro-2-oxo-2H-chromene-3-carboxylate (5b)

White powder (0.32 g, yield 67%), mp 161–163 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3320, 2930, 2865, 1785, 1743, 1630, 1560. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} (ppm) 1.40–1.72 (12H, m, 6CH₂ of cycloheptan), 2.10 and 2.17 (6H, s, 2CH₃), 4.19 (1H, d, $^3J = 6.0$ Hz, CHCONH), 4.92 (1H, d, $^3J = 6.0$ Hz, CHCOO), 4.99 (1H, m, OCH), 7.04–7.32 (4H, m, CH arom), 8.15–8.38 (2H, m, CH arom), 12.04 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO- d_6): δ_{C} (ppm) 17.6, 17.8, 22.6, 22.6, 28.2, 28.3, 33.4, 33.4, 47.7, 53.1, 77.1 (OCH), 116.1, 123.8, 126.6, 128.4, 128.5, 128.73, 128.9, 129.8, 130.8, 136.0, 136.9, 139.9 (11C-Ar), 162.1 (=C=O), 167.2 (CONH), 170.9, 174.9 (2C=O). MS, m/z (%): 385 ($\text{M}^+ - 2$, 97, 2), 364 (25), 323 (10), 192 (55), 165 (20), 147 (100), 118 (65), 91 (80), 77 (85), 57 (95). Anal. Calcd for C₂₆H₂₈N₂O₇: C, 64.99; H, 5.87; N, 5.83. Found: C, 65.21; H, 5.66; N, 5.79.



3.2.3. 2-Methylbutyl 4-(2,6-dimethylphenylcarbamoyl)-3,4-dihydro-7-nitro-2-oxo-2H-chromene-3-carboxylate (5c)

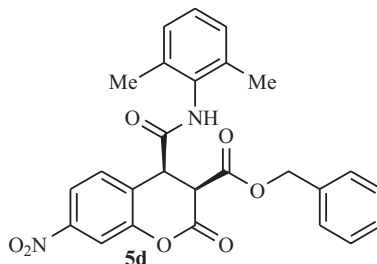
White powder (0.285 g, yield 63%), mp 213–215 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3214, 2962, 2926, 1743, 1693, 1601. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} (ppm) 1.33–1.41 (6H, m, 2CH₃ Aliphatics), 1.56–1.74 (1H, m, CH₂CH₃), 1.86–2.00 (1H, m, CH₂CH₃), 2.17–2.28 (1H, m, CH), 2.64 (3H, s, CH₃ of Ar), 2.68 (3H, s, CH₃ of Ar), 4.48–4.65 (2H, m, CH₂-O), 4.84 (1H, d, $^3J = 6.4$ Hz, CHCONH), 5.37 (1H, d, $^3J = 6.3$ Hz, CHCOO), 7.62–7.73 (4H, m, 4H-Ar), 8.65 (1H, dd, $^3J = 6.2$, 2.7 Hz, 1H-Ar), 8.82 (1H, d, $^3J = 2.7$ Hz, 1H-Ar), 11.27 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO- d_6): δ_{C} (ppm) 11.0, 16.0, 17.4, 17.6, 26.0, 48.6, 53.5, 70.7, 116.25, 124.0, 126.4, 128.2, 128.3, 128.7, 128.8, 129.8, 131.3, 136.4, 137.2, 141.3 (12C-Ar), 161.2 (=C=O), 168.2 (CONH), 170.6, 174.4 (2C=O). MS, m/z (%): 454 ($\text{M}^+ + 1$, 100), 300 (5), 217 (50), 173 (20), 135 (30), 108 (50), 83 (95), 55 (80). Anal. Calcd for C₂₄H₂₆N₂O₇: C, 63.43; H, 5.77; N, 6.16. Found: C, 63.22; H, 5.72; N, 6.12.



3.2.4. Benzyl 4-(2,6-dimethylphenylcarbamoyl)-7-nitro-2-oxochroman-3-carboxylate (5d)

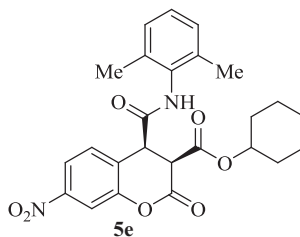
White powder (0.33 g, yield 70%), mp 167–169 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3373, 2916, 1783, 1758, 1654, 1530. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} (ppm) 1.99 (3H, s, CH₃ of Ar), 2.17 (3H, s, CH₃ of Ar), 4.36 (1H, d, $^3J = 6.1$ Hz, CH-OCO),

5.01 (1H, d, $^3J = 6.1$ Hz, CH-CONH), 5.22 (1H, d, $^2J_{\text{AB-q}} = 12.4$ Hz, OCH₂), 5.28 (1H, d, $^2J_{\text{AB-q}} = 12.4$ Hz, OCH₂), 7.04–7.40 (9H, m, 4H-Ar), 8.17 (1H, dd, $^3J = 6.2$, 2.8 Hz, 1H-Ar), 8.39 (1H, dd, $^3J = 6.2$, 2.8 Hz, 1H-Ar), 12.06 (1H, br s, NH). ^{13}C NMR (75 MHz, DMSO- d_6): δ_{C} (ppm) 17.5, 17.8, 47.7, 52.7, 68.8, 116.1, 123.6, 126.6, 128.5, 128.7, 128.8, 129.0, 129.8, 130.7, 135.7, 136.1, 136.9, 139.9, 162.1 (=C=O), 167.8 (CONH), 170.8, 174.9 (2C=O). MS, m/z (%): 474 ($\text{M}^+ + 1$, 100), 300 (5), 217 (50), 173 (20), 135 (30), 108 (50), 83 (95), 55 (80). Anal. Calcd for C₂₆H₂₂N₂O₇: C, 65.82; H, 4.67; N, 5.90. Found: C, 65.67; H, 4.50; N, 5.93.



3.2.5. Cyclohexyl 4-(2,6-dimethylphenylcarbamoyl)-3,4-dihydro-7-nitro-2-oxo-2H-chromene-3-carboxylate (5e)

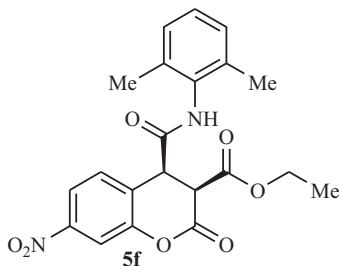
White powder (0.36 g, yield 78%), mp 217–218 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3419, 2933, 2865, 1738, 1709, 1596. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} (ppm) 1.10–1.80 (10H, m, 5CH₂), 2.10 (3H, s, CH₃ of Ar), 2.17 (3H, s, CH₃ of Ar), 4.21 (1H, d, $^3J = 6.0$ Hz, CH-OCO), 4.82 (1H, br s, CH-O), 4.90 (1H, d, $^3J = 6.0$ Hz, CHCONH), 7.02–7.31 (4H, m, 4H-Ar), 8.14–8.42 (2H, m, 2H-Ar) 8.73 (1H, br s, 2NH). ^{13}C NMR (75 MHz, DMSO- d_6): δ_{C} (ppm) 17.6, 17.8, 23.2, 25.2, 31.1, 35.8, 47.8, 53.1, 68.7, 74.5, (CH₃, CH₂, CH and C) 116.1, 123.8, 126.6, 128.4, 128.7, 128.9, 129.2, 129.8, 130.8, 136.0, 136.9, 139.6 (7C-Ar), 162.5 (=C=O), 167.3 (CONH), 170.9, 175.0 (2C=O). MS, m/z (%): 367 ($\text{M}^+ - 99$, 2), 192 (30), 147 (35), 118 (35), 91 (35), 77 (45), 57 (100). Anal. Calcd for C₂₅H₂₆N₂O₇: C, 64.37; H, 5.62; N, 6.01. Found: C, 64.44; H, 5.54; N, 5.95.



3.2.6. Ethyl 4-(2,6-dimethylphenylcarbamoyl)-3,4-dihydro-7-nitro-2-oxo-2H-chromene-3-carboxylate (5f)

White powder (0.29 g, yield 72%), mp 162–165 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3542, 3460, 3075, 2983, 1737, 1709, 1591. ^1H NMR (300 MHz, D₃COD): δ_{H} (ppm) 1.28 (3H, t, $^3J = 7.1$ Hz, OCH₂CH₃), 2.15 and 2.22 (6H, s, 2CH₃), 4.28 (2H, q, $^3J = 7.1$ Hz, OCH₂CH₃), 4.74 (1H, br s, CHCONH), 4.90 (1H, br s, CHCOO), 6.96–7.25 (4H, m, CH arom), 8.13–8.17 (2H, m, CH arom), 8.26 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO- d_6): δ_{C} (ppm) 13.0, 16.4, 16.7, 46.8, 48.5, 62.2 (OCH₂), 114.9, 123.0, 125.8, 127.6, 128.0, 128.2, 128.8, 129.3, 130.2, 135.9, 136.7, 140.4 (12C-Ar), 160.9 (=C=O), 167.8 (CONH), 170.9, 175.2

(2C=O). MS, m/z (%): 412 (M^+ , 2), 367 (40), 339 (30), 291 (10), 192 (45), 147 (80), 118 (75), 91 (85), 77 (100), 39 (70). Anal. Calcd for $C_{21}H_{20}N_2O_7$: C, 61.16; H, 4.89; N, 6.79. Found: C, 60.93; H, 4.84; N, 6.88.

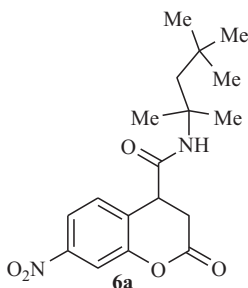


3.3. Typical procedure for the preparation of 3,4-dihydrocoumarin 6a

To a magnetically stirred solution of Meldrum's acid (0.14 g, 1 mmol), 2-hydroxy-4-nitrobenzaldehyde (0.16 g, 1 mmol) in dichloromethane (10 mL) was added 1,1,3,3-tetramethylbutyl isocyanide (0.14 g, 1 mmol). The reaction mixture was stirred for 24 h at room temperature. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 2:1), the solvent was removed under vacuum and the solid residue was washed with ether and the product **6a** was obtained as a white powder.

3.3.1. 3,4-Dihydro-*N*-(2,4,4-trimethylpentan-2-yl)-7-nitro-2-oxo-2H-chromene-4-carboxamide (6a)

White powder (0.22 g, yield 65%); mp 209–213 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3419 (NH), 2952, 2875, 1745, 1602, 1534. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} (ppm) 1.21 (9H, s, $\text{C}(\text{CH}_3)_3$), 1.35 (2H, s, CH_2), 1.44 (6H, s, $2\text{C}(\text{CH}_3)_2$), 2.75 (1H, dd, $^2J_{\text{HH}} = 15.1$ Hz, $^3J_{\text{HH}} = 2.4$ Hz, $\text{CH}_2\text{-CH}$), 2.93 (1H, dd, $^2J_{\text{HH}} = 15.1$ Hz, $^3J_{\text{HH}} = 6.0$ Hz, $\text{CH}_2\text{-CH}$), 3.95 (1H, dd, $^2J_{\text{HH}} = 6.0$ Hz, $^3J_{\text{HH}} = 2.4$ Hz, $\text{CH}_2\text{-CH}$), 7.97 (1H, s, NH), 8.35–8.49 (2H, m, H-Ar), 8.82 (1H, d, $^4J_{\text{HH}} = 2.2$ Hz). ^{13}C NMR (75 MHz, DMSO- d_6): δ_{C} (ppm) 29.4, 29.8, 31.1, 31.3, 31.6, 49.8, 54.8 (C-aliphatic), 118.3, 123.8, 124.9, 125.0, 148.4, 157.8 (C-Ar), 167.3, 170.5 (2C=O). MS, m/z (%): 348 (M^+ , 5), 192 (30), 156 (40), 147 (100), 131 (25), 120 (25), 91 (50), 57 (90), 41 (65).



4. Conclusions

In conclusion, we succeed to introduce a stereoselective method to NO_2 substituent 3,4-dihydrocoumarin derivatives via four-component condensation reaction from commercial available substrate under neutral conditions without using any activator or catalyst in short reaction time. The isolation of product is very straightforward. We hope that this approach may be of value to others seeking novel synthetic fragments with unique properties for medicinal chemistry programs.

Acknowledgments

We gratefully acknowledge the financial support from the Research Council of Azad University of Tabriz Branch.

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