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Iranian Chemical Communication
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Payame Noor University http://icc.journals.pnu.ac.ir

Isoquinoline promoted synthesis of alkyl 2-(1-alkyl-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden) acetate derivatives

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Received: 25 January 2016, Accepted: 26 April 2016, Published: 27 April 2016

Abstract

An efficient and one-pot method is described for the synthesis of alkyl 2-(1-alkyl-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden) acetate derivatives *via* simple reaction of dialkylacetylenedicarboxylate and benzyl phenylthiourea in the presence of isoquinoline, promoted component, under solvent-free conditions. The good yields of the products are synthetic advantage of this environmentally friendly method.

Keywords: Isoquinoline, solvent-free, benzyl phenylthiourea, imidazole, green chemistry.

Introduction

Derivatives of thiazolidinone ring systems are known to act as anti-HIV infections [1], analgesic, anti-bacterial, anti-convulsant, anti parasitic, potential anti-inflammatory, and herbicidal agents [2–7]. Due to the biological activities of thiazolidinones ring, several methods for their synthesis have been illustrated in the literature [8]. Imidazolidine-2-thiones were synthesized by the oxidative cyclization of 1-benzoyl-3-aryl-thioureas with bromine and enolizable carbonyl compounds in the presence of excess triethylamine [9,10]. A suitable method for the synthesis of fused thiazoles was described from the reaction of aroylphenyl thioureas with π-acceptor quinones [11]. 2-Acylimino-3-alkyl-3H-thiazolines were prepared from the condensation reaction of aroyl arylthiourea with halocarbonyl derivatives [12]. The reactions of N-aroyl-N'-arylthioureas with 2,3-diphenyl cyclopropenone give the E/Z mixtures of 3-(3'-aroylthioureido)-2,3-diphenyl-cinnamicacids [13]. The reactions between amidinothioureas, imidoylthioureas, and thioacylamidines with diethyl azodicarboxylate give the corresponding thiadiazoles *via* the oxidative cyclic S-N bond formation [14]. Thiazinones derivatives are produced from the reaction of N-aroyl-N'-arylthioureas with dimethylbut-2-ynedioate in the presence of acetic acid under reflux conditions [15]. 1-Acylthiosemicarbazides react with phenyl propiolate in acetic acid under reflux conditions to afford triazolothiazines [16]. The reaction of

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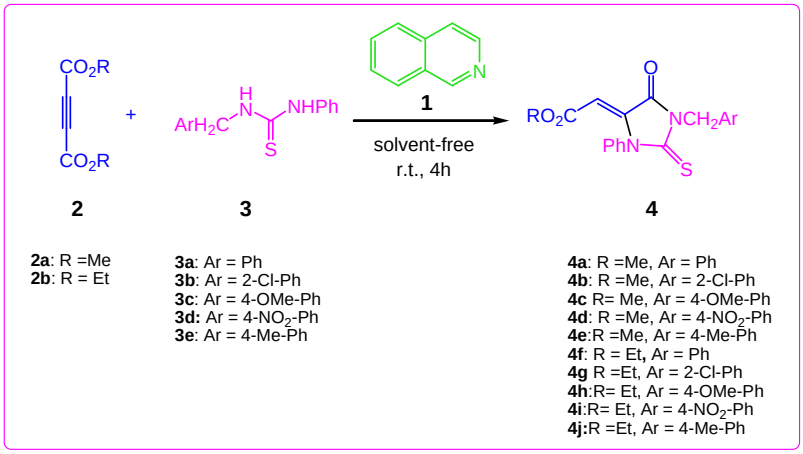
3-aroyl-1-arylthioureas with dimethylbut-2-ynedioate in the presence of triphenylphosphine, as catalyst, produced (Z)-methyl 2-[(Z)-2-(4-aroylimino)-4-oxo-3-aryl-1,3-thiazolidin-5-ylidene] acetates in good yields [17]. Synthesis of 2-amino-4*H*-1,3-thiazin-4-one and dimethyl 3,3'-thiodiacrylates were performed *via* the reaction of corresponding thiourea and dimethyl acetylenedicarboxylate in water at room temperature [18]. Herein, we report the synthesis of alkyl 2- (1-alkyl-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazole-4-yliden) acetate derivatives 4 *via* the reaction of dialkylacetylenedicarboxylates 2 and benzyl phenylthiourea 3, which proceeded smoothly in the presence of isoquinoline 1, as promoted component, under solvent-free conditions at room temperature (Scheme 1).

Experimental

General

Melting points were taken on a Kofler hot stage apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were obtained with a Bruker FT-400 spectrometer in $CDCl_3$, and tetramethylsilane (TMS) as an internal standard. Mass spectra were recorded with a Finnigan Mat TSQ-70 spectrometer. Infrared (IR) spectra were acquired on a Nicollet Magna 550-FT spectrometer. Elemental analyses were carried out with a Perkin-Elmer model 240-C apparatus. The results of elemental analyses (C, H, N) were within ±0.4 % of the calculated values. All chemical compounds were obtained from Fluka and were used without further purification.



Scheme 1. Synthesis of compounds **4a-j**

General procedure for synthesis of 4a-4j

Dialkyl acetylenedicarboxylate **2** (2 mmol) was slowly added to a magnetically stirred mixture of an benzyl phenylthiourea **3** (2 mmol) and isoquinoline **1** (2 mmol), and the reaction mixture was stirred for 4 h at room temperature. After completion of the reaction as indicated by TLC, the residue was purified by column chromatography over silica gel (Merck 230-400 mesh) using an n-hexane-EtOAc mixture (6:1) as eluant to afford the pure compounds **4**.

Methyl 2-(1-benzyl-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden) acetate (4a)

Yellow powder, m.p. 146-148°C, 0.55 g (93%). IR (KBr): ν =1465 (C=S),

1721 (C=O), 1735 (C=O), 2985 (CH) cm^{-1}. EI-MS: 352 (3, M^+), 337 (24), 261 (68), 15 (100). Anal. Calc. for $C_{19}H_{16}N_2O_3S$ (352.41): C 64.76, H 4.58, N 7.95; found,%: C 65.85, H 5.61, N 8.23. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.73 (s, 3 H, OCH_3), 5.07 (s, 2 H, NCH_2), 5.34 (s, 1 H, CH), 7.18-7.48 (m, 10 H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 45.5 (NCH_2), 52.6 (OMe), 104.8 (CH), 128.2 (2 CH), 128.6 (CH), 128.7 (2 CH), 129.4 (2 CH), 130.0 (CH), 130.1 (2 CH), 134.0 (C), 135.2 (C), 137.1 (C), 159.7 (C=O), 164.1 (C=O), 178.4 (C=S).

Methyl 2-[1-(2-chlorobenzyl)-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden] acetate (4b)

Yellow powder, m.p. 154-156 °C; yield: 0.67 g (93 %). IR (KBr): ν = 1471 (C=S), 1722 (C=O), 1734 (C=O), 2982 (CH) cm^{-1}. EI-MS: 386 (5, M^+), 371 (56), 351 (21), 295 (32), 261 (54), 15 (100). Anal. Calc. for $C_{19}H_{15}ClN_2O_3S$ (386.82): C 58.99, H 3.91, N 7.24; found,%: C 58.81, H 4.21, N 8.13. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.72 (s, 3 H, OCH_3), 5.12 (s, 2 H, NCH_2), 5.32 (s, 1 H, CH), 7.14-7.46 (m, 9 H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 45.8 (NCH_2), 52.5 (OMe), 105.0 (CH), 128.5 (CH), 127.1(2 CH), 128.8 (2 CH), 128.9 (CH), 129.2 (CH), 130.1 (2 CH), 130.9 (CH), 133.9 (C), 134.1 (C), 135.3 (C), 137.1 (C), 160.1 (C=O), 163.9 (C=O), 178.6 (C=S).

Methyl 2-[1-(4-methoxybenzyl)-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden] acetate (4c)

Yellow powder, m.p. 148-150 °C; yield: 0.67 g (85 %). IR (KBr): ν = 1469 (C=S), 1725 (C=O), 1737 (C=O), 2981 (CH) cm^{-1}. EI-MS: 382 (3, M^+), 367 (53), 351 (21), 261 (75), 291 (36), 15 (100). Anal. Calc. for $C_{20}H_{18}N_2O_4S$ (382.43): C 62.81, H 4.74, N 7.33; found,%: C 61.11, H 5.36, N 6.24. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.64 (s, 3 H, OCH_3), 3.73 (s, 3 H, OCH_3), 5.06 (s, 2 H, NCH_2), 5.34 s (1 H, CH), 7.08 (d, 2 H, 2 CH, *J* = 7.6 Hz), 7.18-7.46 (m, 7 H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 46.2 (NCH_2), 52.6 (OMe), 53.2 (OMe), 104.5 (CH), 117.6 (2 CH), 128.5 (CH), 128.8 (2 CH), 129.9 (2 CH), 131.7 (2 CH), 133.8 (C), 135.2 (C), 135.4 (C), 136.9 (C), 154.6 (C), 160.2 (C=O), 164.0 (C=O), 178.8 (C=S).

Methyl 2-[1-(4-nitrobenzyl)-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden] acetate (4d)

Yellow powder, m.p. 157-158 °C; yield: 0.67 g (85 %). IR (KBr): ν = 1471 (C=S), 1722 (C=O), 1734 (C=O), 2982 (CH) cm^{-1}. EI-MS: 397 (4, M^+), 382 (48), 351 (27), 306 (31), 261 (36), 15 (100). Anal. Calc. for $C_{19}H_{15}N_3O_5S$ (397.41): C 57.42, H 3.80, N 10.57; found,% C 58.21, H 3.96, N 9.14. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.73 (s, 3 H, OCH_3), 5.09 (s, 2-H, NCH_2), 5.35 (s,1H, CH), 7.12-7.42 (m,5 H, Ar), 7.67 (d, 2 H, Ar, *J* = 7.6 Hz), 7.84 (d, 2 H, Ar, *J* =7.5 Hz). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 45.6 (NCH_2), 52.6 (OMe), 104.9 (CH), 125.4 (2 CH), 128.3(2 CH), 128.6 (CH), 128.9 (2 CH), 130.3 (2 CH), 134.0 (C), 135.1 (C), 137.4 (C), 139.2 (C), 147.7 (C), 160.2 (C=O), 164.1 (C=O), 178.3 (C=S).

Methyl 2-[1-(4-methylbenzyl)-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden] acetate (4e)

Yellow powder, m.p. 132-134 °C; yield: 0.63 g (87 %). IR (KBr): ν = 1471 (C=S), 1719 (C=O), 1733 (C=O), 2984 (CH) cm^{-1}. EI-MS: 366 (7, M^+), 351 (59), 261 (32), 275 (56), 15 (100). Anal. Calc. for $C_{20}H_{18}N_2O_3S$ (366.45): C 65.55, H 4.95, N 7.64;

found,% C 65.41, H 5.81, N 7.12. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 2.31 (s, 3 H, CH_3), 3.73 (s, 3 H, OCH_3), 5.12 (s, 2 H, NCH_2), 5.38 (s, 1H, CH), 6.94-7.11 (m, 9-H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 21.1 (CH_3), 45.6 (NCH_2), 52.5 (OMe), 104.8 (CH), 128.2 (2 CH), 128.6 (2 CH), 128.8 (CH), 130.1 (2 CH), 130.3 (CH), 131.4 (C), 134.2 (C), 135.5 (C), 137.5 (C), 160.1 (C=O), 164.2 (C=O), 178.1 (C=S).

Ethyl 2-(1-benzyl-5-oxo-3-phenyl-2-thioxotetrahydro- 4*H*-imidazol-4-yliden] acetate (4f)

Yellow powder, m.p. 143-145 °C; yield: 0.55 g (93 %). IR (KBr): ν =1465 (C=S), 1721 (C=O), 1735 (C=O), 2985 (CH) cm^{-1}. EI-MS: 366 (5, M^+), 337 (42), 275 (59), 29 (100). Anal. Calc. for $C_{20}H_{18}N_2O_3S$ (366.43): C 65.55, H 4.95, N 7.64; found,%: C 64.58, H 5.12, N 8.03. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 1.21 (t, 3 H, *J* = 6.6, CH_3), 4.10 (q, 2H, OCH_2, *J* = 6.9 Hz), 5.07 (s, 2-H, NCH_2), 5.34 (s, 1 H, CH), 7.17-7.46 (m, 10 H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 14.5 (CH_3), 45.5 (NCH_2), 52.5 (OMe), 62.1 (OCH_2), 104.8 (CH), 128.2 (2 CH), 128.6 (CH), 128.7 (2 CH), 129.5 (2 CH), 129.9 (CH), 130.1 (2 CH), 134.0 (C), 135.2 (C), 137.2 (C), 159.6 (C=O), 164.0 (C=O), 178.4 (C=S).

Ethyl 2-[1-(2-chlorobenzyl)-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden] acetate (4g)

Yellow powder, m.p. 153-155 °C; yield: 0.72 g (90 %). IR (KBr): ν = 1471 (C=S), 1726 (C=O), 1738 (C=O), 2980 (CH) cm^{-1}. EI-MS: 400 (3, M^+), 371 (48), 365 (24), 275 (32), 309 (51), 29 (100). Anal. Calc. for $C_{20}H_{17}ClN_2O_3S$ (400.87): C 59.92, H 4.27, N 6.99; found,%: C 61.01, H 3.31, N 7.11. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 1.23 (t, 3 H, CH_3, *J* = 6.8 Hz), 4.12 (q, 2H, OCH_2, *J* = 6.6 Hz), 5.06 (s, 2 H, N CH_2), 5.32 (s, 1 H, CH), 7.16-7.49 (m, 9 H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 13.9 (CH_3), 45.8 (NCH_2), 52.5 (OMe), 61.5 (OCH_2), 105.0 (CH), 128.4 (CH), 127.1(2 CH), 128.6 (2 CH), 128.9 (CH), 129.2 (CH), 130.1 (2 CH), 130.9 (CH), 133.9 (C), 134.1 (C), 135.3 (C), 137.1 (C), 159.9 (C=O), 163.9 (C=O), 178.5 (C=S).

Ethyl 2-[1-(4-methoxybenzyl)-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden] acetate (4h)

Yellow powder, m.p. 150-152 °C; yield: 0.67 g (85 %). IR (KBr): ν = 1470 (C=S), 1726 (C=O), 1735 (C=O), 2981 (CH) cm^{-1}. EI-MS: 396 (3, M^+), 367 (53), 275 (75), 291 (36), 29 (100). Anal. Calc. for $C_{20}H_{18}N_2O_4S$ (396.46): C 63.62, H 5.08, N 7.07; found,%: C 62.11, H 4.36, N 7.86. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 1.21 (t, 3H, CH_3, *J* = 6.7 Hz), 4.12 (q, 2 H, OCH_2, *J* = 6.6 Hz), 5.06 (s, 2 H, NCH_2), 5.34 (s, 1 H, CH), 7.06 (d, 2 H, *J* = 7.6, Ar), 7.16-7.48 (m,7 H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 13.9 (CH_3), 46.2 (NCH_2), 52.6 (OMe), 62.3 (OCH_2), 53.2 (OMe), 104.6 (CH), 117.5 (2 CH), 128.5 (CH), 128.8 (2 CH), 129.9 (2 CH), 131.6 (2 CH), 133.6 (C), 135.2 (C), 135.5 (C), 136.9 (C), 154.4 (C), 160.0 (C=O), 164.0 (C=O), 178.4 (C=S).

Ethyl 2-[1-(4-nitrobenzyl)-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden] acetate (4i)

Yellow powder, m.p. 155-157 °C; yield: 0.72 g (88 %). IR (KBr): ν = 1470 (C=S), 1724 (C=O), 1736 (C=O), 2983 (CH) cm^{-1}. EI-MS: 411 (4, M^+), 365 (25), 275 (27), 320 (31), 136 (37), 29 (100). Anal. Calc. for $C_{20}H_{17}N_3O_5S$ (411.43): C 58.38, H 4.16, N 10.21; found,% C 58.33, H 5.10, N 9.53. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 1.23 (t, 3H,CH_3, *J* = 6.6 Hz), 4.09 (q, 2H,OCH_2, *J* = 6.6 Hz-

), 5.10 (s, 2-H, NCH_2), 5.36 (s, 1H, CH), 7.10-7.41 (m, 5 H, Ar), 7.64 (d, 2 H, Ar, *J* = 7.6 Hz), 7.84 (d, 2 H, Ar, *J* =7.5 Hz), ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 13.8 (CH_3), 45.6 (NCH_2), 62.5 (OCH_2), 104.7 (CH), 125.4 (2 CH), 128.1(2 CH), 128.4 (CH), 128.8 (2 CH), 130.3 (2 CH), 134.1 (C), 134.9 (C), 137.5 (C), 139.1 (C), 147.5 (C), 160.5 (C=O), 164.1 (C=O), 178.2 (C=S).

Ethyl 2-[1-(4-methylbenzyl)-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazol-4-yliden] acetate (4j)

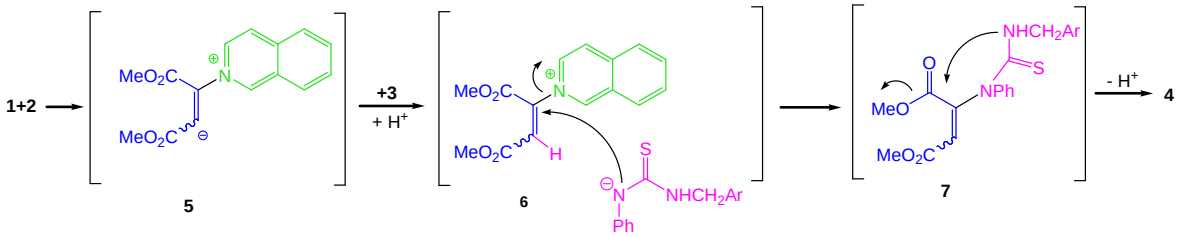
Yellow powder, m.p. 138-141 °C; yield: 0.65 g (85 %). IR (KBr): ν = 1474 (C=S), 1723 (C=O), 1736 (C=O), 2983 (CH) cm^{-1}. EI-MS: 380 (7, M^+), 365 (49), 351 (57), 275 (27), 29 (100). Anal. Calc. for $C_{21}H_{18}N_2O_3S$ (380.46): C 66.29, H 5.30, N 7.36; found,% C 66.84, H 4.91, N 6.58. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 1.22 (t, 3 H, *J* = 6.7, CH_3), 4.11 (q, 2-H,OCH_2, *J* = 6.6 Hz), 5.07 (s, 2 H, NCH_2), 5.35 (s, 1H, CH), 6.95-7.13 (m, 9-H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$), δ, ppm: 14.1 (CH_3), 45.6 (NCH_2), 62.3 (OCH_2), 104.8 (CH), 128.2 (2 CH), 128.6 (2 CH), 128.8 (CH), 130.1 (2 CH), 130.3 (CH), 131.4 (C), 134.2 (C), 135.5 (C), 137.5 (C), 160.1 (C=O), 164.2 (C=O), 178.1 (C=S).

Results and discussion

In this method, isoquinoline **1** promoted reaction of dialkylacetylenedicarboxylates **2** and benzyl phenylthiourea **3**, as promoted component for the synthesis of alkyl 2-(1-alkyl-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazole-4-yliden) acetates drivatives. This precedure is performed under solvent free conditions and as green and enviromentally route. Also, the performance of this reaction at room temprature is another advantage.

The structures of **4a-j** compounds were apparent from the ^{1}H NMR, ^{13}C NMR and IR spectra. The mass spectra of compounds **4a–j** displayed molecular ion peaks at appropriate *m*/*z* values. The ^{1}H NMR spectrum of **4a** displayed three peeks at *δ* 3.73, 5.07 and 5.34 ppm for the methoxy group, benzylic and olefinic protons, respectively, along with characteristic multiplet signals for the aromatics moiety. The proton-decoupled ^{13}C NMR spectrum of **4a** showed 15 signals in agreement with the proposed structure.

A possible mechanism for the synthesis of compounds **4** is proposed in Scheme 2. The zwitterionic intermediate **5** produced from the reaction of isoquinoline and dialkyl acetylenedicarboxylate is subsequently protonated by a phenylthiourea **3** and formed intermediate **6**. The conjugate base of phenylthiourea attacked to intermediate **6** and removed isoquinoline ring as leavining group and compound **7** was produced. Then, nucleophilic attack of NH on carbonyl synthesized compound **4**.



Scheme 2. Possible mechanism for the formation of compounds **4**

Conclusion

In conclusion, we have reported a convenient one-pot route for the synthesis of alkyl 2- (1-alkyl-5-oxo-3-phenyl-2-thioxotetrahydro-4*H*-imidazole-4-yliden) acetates *via* the reaction of dialkylacetylenedicarboxylate and benzyl phenylthiourea in the presence of isoquinoline, at room temperature under solvent-free conditions.

Acknowledgments

We gratefully acknowledge financial and spiritual support from Islamic Azad University of Qaemshahr.

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