

Reaction between enaminones and acetylenic esters in the presence of triphenylphosphine: a convenient synthesis of alkyl 2-(1-benzyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)acetates

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Abstract One-pot reaction between enaminocarbonyl compounds derived from six-membered 1,3-diketones and substituted benzylamines, and electron-deficient acetylenic esters in the presence of triphenylphosphine lead to alkyl 2-(1-benzyl-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)acetate derivatives in good yields.

Keywords β -Enaminones · Dialkyl acetylenedicarboxylates · Triphenylphosphine · One-pot reaction

Introduction

Enaminones are useful compounds which are used as intermediates in organic synthesis [1–8]. On the other hand, five-membered nitrogen-containing heterocycles, such as nonaromatic type of pyrrol-2-ones and indol-2-ones, exist in a wide range of biologically active organic compounds [9, 10]. Also these compounds are important building blocks for the synthesis of new biologically active heterocycles [11, 12]. In addition, the generation of 1,3-dipolar species and their trapping by suitable *H*-acids leading to heterocycles occupies an important position. Zwitterionic species

often result from the addition of various nucleophiles to electron-deficient acetylenic compounds [13–18]. Trivalent phosphorus compounds have been the most studied nucleophilic species [19–23] and there are many studies on the reaction between activated alkynes and *NH*-acids in the presence of triphenylphosphine to produce numerous heterocycles [24–31]. In continuation of our interest in generation and trapping of various zwitterionic species by *H*-acids [32–38], we wish to report the one-pot reaction between dialkyl acetylenedicarboxylates and enaminones in the presence of triphenylphosphine to produce alkyl 2-(1-benzyl-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)acetate derivatives in good yields.

Results and discussion

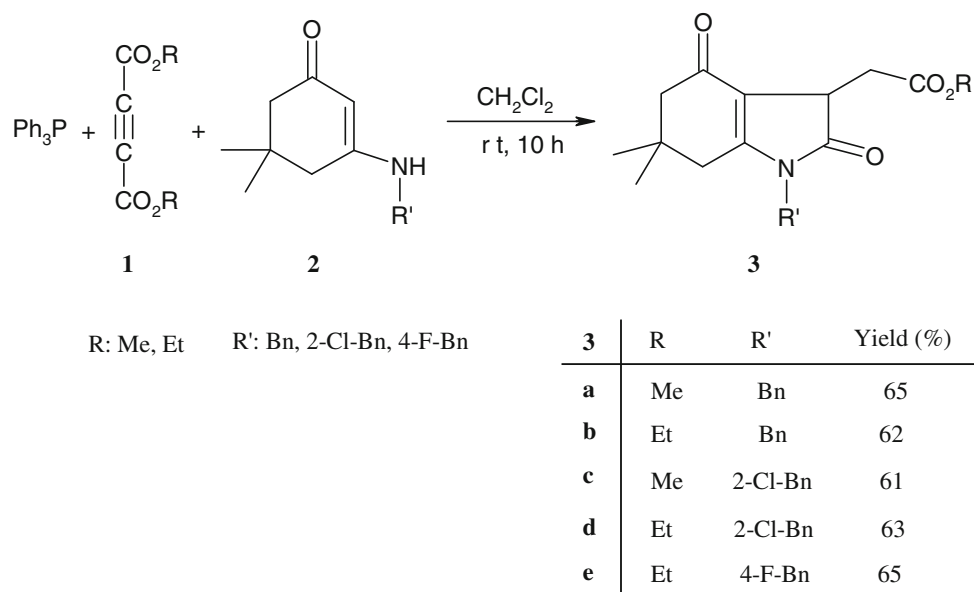
The reaction of dialkyl acetylenedicarboxylates **1** with enaminocarbonyl compound **2** in the presence of triphenylphosphine in dichloromethane was complete within a few hours at room temperature. The progress of the reaction was monitored by TLC. After 10 h, the TLC of the mixture of the reaction clearly showed the presence of compound **3** and triphenylphosphine oxide. IR, ¹H NMR, ¹³C NMR, mass spectra, and CHN data of the isolated products clearly support the formation of alkyl 2-(1-benzyl-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)acetate derivatives **3** (Scheme 1).

The structures of compounds **3a–e** were deduced from their elemental analyses and their IR, ¹H NMR, and ¹³C NMR spectra. The mass spectrum of these compounds displayed molecular ion peaks at appropriate *m/z* value. The ¹H NMR spectrum of **3a** exhibited three sharp singlets, readily recognizable as arising from the methyl ($\delta = 0.82, 0.99$ ppm) and methoxy ($\delta = 3.67$ ppm) protons. The four methylene

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Scheme 1 Synthesis of compounds **3**

protons in **3a** exhibit a complex pattern for the diastereotopic hydrogens. Two methylene protons of six-membered ring appear as a multiplets ($\delta = 2.25\text{--}2.46$ ppm). The methylene protons of $\text{CH}_2\text{CO}_2\text{Me}$ substituent have different chemical shifts and show an AMX system ($\delta_{\text{A}} = 2.72$ ppm, $\delta_{\text{M}} = 3.13$ ppm, $J_{\text{AM}} = 16.5$ Hz, $J_{\text{AX}} = 7.5$ Hz, $J_{\text{MX}} = 2.3$ Hz). The methylene protons of benzyl group show an AX system ($\delta_{\text{A}} = 4.63$ ppm, $\delta_{\text{X}} = 5.12$ ppm, $J_{\text{AX}} = 16.5$ Hz). The methine proton appears as broad doublet ($\delta = 4.08$ ppm, $J = 7.5$ Hz) and aromatic protons appear as multiplets ($\delta = 7.11\text{--}7.45$ ppm). The ^{13}C NMR spectrum of compound **3a** showed eighteen distinct resonances in agreement with the proposed structure. The structural assignments made on the basis of the NMR spectra of compound **3a** were supported by its IR spectrum. The carbonyl groups in **3a** exhibited strong absorption bands at 1726, 1690, and 1650 cm^{-1} .

Although the mechanistic details of the above reactions are unknown, a possible explanation for this transformation is proposed in Scheme 2. It is conceivable that the reaction involves the initial formation of a zwitterionic intermediate **4** from the reaction between Ph_3P and the acetylenic compound [20–32], which protonated with **2** to produce intermediates **5** and **6**. Then, the positively charged ion **6** is attacked by the conjugated base of enaminone **5** to produce ylide intermediate **7**. Subsequently, the intramolecular annulation of this intermediate produce the ylide **8**. This ylide is attacked by water, and subsequent elimination of triphenylphosphine oxide [39] produce final product **3**.

Conclusions

In summary, we have described a convenient route to alkyl 2-(1-benzyl-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1-

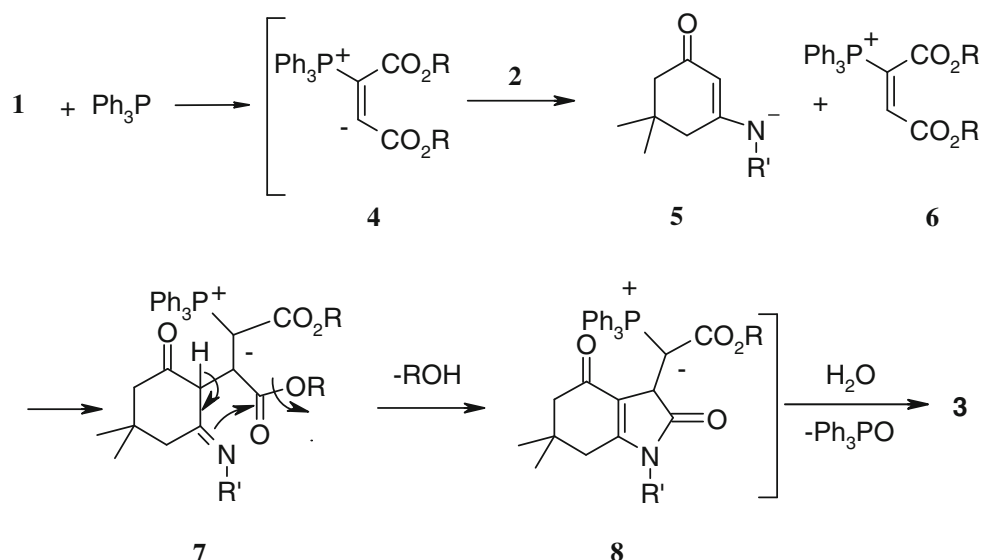
H-indol-3-yl)acetate derivatives from the one-pot reaction of enaminocarbonyl compounds and dialkyl acetylenedicarboxylates in the presence of triphenylphosphine.

Experimental

Enaminones **2** were prepared based on reported procedures [40]. Other starting materials and solvents were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. Melting points were measured with an Electrothermal 9100 apparatus. Elemental analyses C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. IR spectra were measured with a Shimadzu IR-460 spectrometer. NMR spectra were recorded with a Bruker DRX-250 AVANCE instrument (250.1 MHz for ^1H and 62.9 MHz for ^{13}C) with CDCl_3 as solvent. Mass spectra were recorded with an Agilent-5975 C inert XL MSD mass spectrometer operating at an ionization potential of 70 eV.

General procedure

To a magnetically stirred solution of triphenylphosphine (2 mmol) and enaminone **2** (2 mmol) in dichloromethane (5 mL) was added dropwise a mixture of dialkyl acetylenedicarboxylate (2 mmol) in dichloromethane (2 mL) at 0°C over 10 min. The reaction mixture was then allowed to warm up to room temperature and stirred for 10 h. The solvent was removed under reduced pressure and the residual was purified by silica gel (Merck 230–400 mesh) column chromatography.



Scheme 2 A proposed mechanism for the formation of compounds **3**

Methyl 2-(1-benzyl-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)acetate (**3a**)

Yellow oil, yield: 65%. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1726, 1690, 1650 (C=O). Anal. Calcd for $\text{C}_{20}\text{H}_{23}\text{NO}_4$ (341.4), C, 70.36; H, 6.79; N, 4.10. Found: C, 68.95; H, 6.68; N, 3.98. ^1H NMR (250.1 MHz, CDCl_3): δ_{H} 0.82 and 0.99 (6H, 2s, 2 CH_3), 2.25–2.46 (4H, m, 2 CH_2), 2.72 and 3.13 (2H, AMX system, $\delta_{\text{A}} = 2.72$ ppm, $\delta_{\text{M}} = 3.13$ ppm, $J_{\text{AM}} = 16.5$ Hz, $J_{\text{AX}} = 7.5$ Hz, $J_{\text{MX}} = 2.3$ Hz, $\text{CH}_2\text{CO}_2\text{CH}_3$), 3.67 (3H, s, OCH_3), 4.08 (1H, brd, $J = 7.5$ Hz, CH), 4.59 (2H, AX system, $\delta_{\text{A}} = 4.63$ ppm, $\delta_{\text{X}} = 5.12$ ppm, $J_{\text{AX}} = 16.5$ Hz, NCH_2), 7.11–7.45 (5H, m, CH–Ar) ppm. ^{13}C NMR (62.9 MHz, CDCl_3): $\delta_{\text{C}} = 27.3, 28.7, 32.7, 33.6, 33.8, 40.1, 44.8,$ and 49.4 (8C), 52.4 (OCH_3), 112.8 (N–C=C), 126.0 (2CH–Ar), 127.5 (CH–Ar), 128.8 (2CH–Ar), 136.8 (C), 154.3 (N–C=C), 169.2 (C=O, lactam), 172.3 (C=O, ester), 194.9 (C=O) ppm. MS: m/z (%) = 341 (M^+ , 22), 282 (48), 149 (50), 91 (100).

Ethyl 2-(1-benzyl-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)acetate (**3b**)

Yellow oil, yield: 62%. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1722, 1697, 1618 (C=O). Anal. Calcd for $\text{C}_{21}\text{H}_{25}\text{NO}_4$ (355.4): C, 70.96; H, 7.09; N, 3.94. Found: C, 69.72; H, 6.92%; N, 3.81. ^1H NMR (250.1 MHz, CDCl_3): δ_{H} 0.82 and 1.00 (6H, 2s, 2 CH_3), 1.2 (3H, t, $^3J_{\text{HH}} = 7.5$ Hz, OCH_2CH_3), 2.17–2.40 (4H, m, 2 CH_2), 2.72 and 3.15 (2H, AMX system, $\delta_{\text{A}} = 2.72$ ppm, $\delta_{\text{M}} = 3.15$ ppm, $J_{\text{AM}} = 16.5$ Hz, $J_{\text{AX}} = 7.8$ Hz, $J_{\text{MX}} = 1.8$ Hz, $\text{CH}_2\text{CO}_2\text{C}_2\text{H}_5$), 4.10 (2H, m, OCH_2CH_3), 4.12 (1H, brd, $J = 7.8$ Hz, CH), 4.13 (2H, q, $^3J_{\text{HH}} = 7.5$ Hz, OCH_2CH_3), 5.03 (2H, AX system, $\delta_{\text{A}} = 4.60$ ppm, $\delta_{\text{X}} =$

5.45 ppm, $J_{\text{AX}} = 16.5$ Hz, NCH_2), 7.11–7.40 (5H, m, CH–Ar) ppm. ^{13}C NMR (62.9 MHz, CDCl_3): δ_{C} 14.1 (OCH_2CH_3), 27.0, 28.9, 32.7, 33.6, 33.9, 40.1, 44.8, and 49.4 (8C), 61.4 (OCH_2CH_3), 112.9 (N–C=C), 126.0 (2CH–Ar), 127.4 (CH–Ar), 128.9 (2CH–Ar), 136.8 (C), 154.1 (N–C=C), 169.3 (C=O, lactam), 171.8 (C=O, ester), 195.0 (C=O) ppm. MS: m/z (%) = 355 (M^+ , 46), 309 (15), 282 (85), 91 (100).

Methyl 2-(1-(2-chlorobenzyl)-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)acetate (**3c**)

Yellow oil, yield: 61%. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1730, 1700, 1650 (C=O). Anal. Calcd for $\text{C}_{20}\text{H}_{22}\text{ClNO}_4$ (375.8), C, 63.91; H, 5.90; N, 3.73. Found: C, 62.67; H, 5.78; N, 3.65. ^1H NMR (250.1 MHz, CDCl_3): δ_{H} 0.84 and 1.0 (6H, 2s, 2 CH_3), 2.29–2.34 (4H, m, 2 CH_2), 2.75, 3.13 (2H, AMX system, $\delta_{\text{A}} = 2.75$ ppm, $\delta_{\text{M}} = 3.13$ ppm, $J_{\text{AM}} = 16.5$ Hz, $J_{\text{AX}} = 8.0$ Hz, $J_{\text{MX}} = 0.0$ Hz, $\text{CH}_2\text{CO}_2\text{CH}_3$), 3.69 (3H, s, OCH_3), 4.09 (1H, brd, $J = 8.0$ Hz, CH), 4.88 (2H, AX system, $\delta_{\text{A}} = 4.90$ ppm, $\delta_{\text{X}} = 5.02$ ppm, $J_{\text{AX}} = 16.5$ Hz, NCH_2), 7.11–7.46 (4H, m, CH–Ar) ppm. ^{13}C NMR (62.9 MHz, CDCl_3): δ_{C} 27.2, 28.8, 32.8, 33.6, 33.9, 39.9, 42.7, and 49.4 (8C), 52.5 (OCH_3), 112.7 (N–C=C), 126.8, 127.3, 128.7, 129.6, 132.0, and 134.0 (6C–Ar), 154.0 (N–C=C), 169.1 (C=O, lactam), 172.4 (C=O, ester), 195.0 (C=O) ppm. MS: m/z (%) = 375 (M^+ , 12), 340 (25), 316 (50), 125 (100).

Ethyl 2-(1-(2-chlorobenzyl)-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)acetate (**3d**)

Yellow oil, yield: 63%. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1722, 1680, 1657 (C=O). Anal. Calcd for $\text{C}_{21}\text{H}_{24}\text{ClNO}_4$ (389.9), C, 64.70;

H, 6.20; N, 3.59. Found: C, 63.68; H, 6.07; N, 3.50. ^1H NMR (250.1 MHz, CDCl_3): δ_{H} 0.84 and 1.01 (6H, 2s, 2 CH_3), 1.22 (3H, t, $^3J_{\text{HH}} = 7.0$ Hz, OCH_2CH_3), 2.21–2.42 (4H, m, 2 CH_2), 2.75 and 3.13 (2H, AMX system, $\delta_{\text{A}} = 2.75$ ppm, $\delta_{\text{M}} = 3.13$ ppm, $J_{\text{AM}} = 16.5$ Hz, $J_{\text{AX}} = 7.8$ Hz, $J_{\text{MX}} = 1.8$ Hz, $\text{C}_2\text{H}_5\text{CO}_2\text{C}_2\text{H}_5$), 4.12 (1H, brd, $J = 7.8$ Hz, CH), 4.10 (2H, m, OCH_2CH_3), 5.04 (2H, AX system, $\delta_{\text{A}} = 4.78$, $\delta_{\text{X}} = 5.37$ ppm, $J_{\text{AX}} = 17.5$ Hz, NCH_2), 7.11–7.45 (4H, m, CH–Ar) ppm. ^{13}C NMR (62.9 MHz, CDCl_3): δ_{C} 14.1 (OCH_2CH_3), 26.9, 29.0, 32.7, 33.6, 34.1, 39.9, 42.7, and 49.5 (8C), 61.4 (OCH_2CH_3), 112.8 (N=C=C), 126.7, 127.3, 128.7, 129.6, 131.9, 134.1 (6C–Ar), 153.8 (N=C=C), 169.2 (C=O, lactam), 171.9 (C=O, ester), 194.9 (C=O) ppm. MS: m/z (%) = 390 (M^+ , 30), 354 (20), 316 (65), 125 (100).

Ethyl 2-(1-(4-fluorobenzyl)-6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1H-indol-3-yl)acetate (**3e**)

Yellow oil, yield: 65%. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 1731, 1695, 1649 (C=O). Anal. Calcd for $\text{C}_{21}\text{H}_{24}\text{FNO}_4$ (373.4), C, 67.55; H, 6.48; N, 3.75. Found: C, 66.41; H, 6.23; N, 3.68. ^1H NMR (250.1 MHz, CDCl_3): δ_{H} 0.84 and 1.01 (6H, 2s, 2 CH_3), 1.22 (3H, t, $^3J_{\text{HH}} = 7.5$ Hz, OCH_2CH_3), 2.25–2.38 (4H, m, 2 CH_2), 2.68 and 3.12 (2H, AMX system, $\delta_{\text{A}} = 2.68$ ppm, $\delta_{\text{M}} = 3.12$ ppm, $J_{\text{AM}} = 16.3$ Hz, $J_{\text{AX}} = 7.7$ Hz, $J_{\text{MX}} = 0.0$ Hz, $\text{C}_2\text{H}_5\text{CO}_2\text{C}_2\text{H}_5$), 4.10 (2H, m, OCH_2CH_3), 4.15 (1H, brd, $J = 7.7$ Hz, CH), 4.52 (2H, AX system, $\delta_{\text{A}} = 4.55$, $\delta_{\text{X}} = 5.40$, $J_{\text{AX}} = 16.5$ Hz, NCH_2), 7.11–7.45 (4H, m, CH–Ar) ppm. ^{13}C NMR (62.9 MHz, CDCl_3): δ_{C} 14.1 (OCH_2CH_3), 27.1, 28.9, 32.7, 33.6, 34.0, 40.2, 44.2, and 49.5 (8C), 61.4 (OCH_2CH_3), 113.1 (N=C=C), 115.8 (d, $^2J_{\text{CF}} = 22.0$ Hz, 2 CH–Ar), 127.8 (d, $^3J_{\text{CF}} = 7.0$ Hz, 2 CH–Ar), 132.6 (C), 153.7 (N=C=C), 169.2 (C=O, lactam), 171.8 (C=O, ester), 194.7 (C=O) ppm. MS: m/z (%) = 373 (M^+ , 10), 300 (20), 109 (100).

Supplementary Material

Supplementary data (^1H NMR and ^{13}C NMR spectra for compounds **3a–3e**) associated with this article can be found in the online version.

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