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Catalyst-free synthesis of functionalized dihydro-2-oxypyrroles by the reaction of enaminones and *N*,*N*′-bis(phenylmethylidene) phenylmethane

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ABSTRACT

A catalyst-free and convenient approach for the preparation of substituted dihydro-2-oxypyrrole is described. This three-component reaction between primary amines, dialkyl acetylenedicarboxylate, and *N*,*N*′-bis(phenylmethylidene)phenylmethane proceeds in MeOH under reflux conditions in good to excellent yields.

1. Introduction

Substituted pyrrole derivatives are very important heterocycles. Many biologically active compounds, potent pharmaceuticals, and natural products contain the pyrrole structural motif [1]. Among the pyrrole derivatives, dihydro-2-oxoypyrroles show such versatility and they are important substructures in a variety of pharmacy, including products active against viral infections (HIV [2,3], influenza [4] cytomegalovirus [5]), anticancer agents [6] and products active against microbiological diseases [7–9] (bacterial or fungal). Furthermore, dihydro-2-oxoy-pyrrole derivatives have been used as PI-091 [10], which is a novel platelet aggregation inhibitor, and EBPC, which is a highly specific aldose reductase inhibitor [11], as shown in Fig. 1. Besides, the well-known 5-alkyl-2-oxopyrroles [12], first described in 1890 by Emery [13], relatively little attention was given toward 5-aryl-2-oxopyrrole derivatives in the open literature.

As part of our continuing effort into design of new routes for the preparation of biologically active compounds using *N*,*N*′-bis(phenylmethylidene)phenylmethane and application of this reagent in the synthesis of numerous organic compounds, especially aza-cyclic compounds [14], herein, we describe a simple, one-pot, three-component synthesis of 5-phenyl-2-oxopyrrole derivatives **3** by the three-component reaction of *N*,*N*′-bis(phenylmethylidene)phenylmethane, primary amines **1** and dialkyl acetylenedicarboxylate **2** (Scheme 1).

2. Results and discussion

Firstly, an easily available starting material *N*,*N*′-bis (phenylmethylidene)phenylmethane was reacted with benzylamine **1a** and diethyl acetylenedicarboxylate **2a** in MeOH under refluxing temperature for 6 h. The 5-phenyl-2-oxopyrrole **3a** was successfully obtained in 85%

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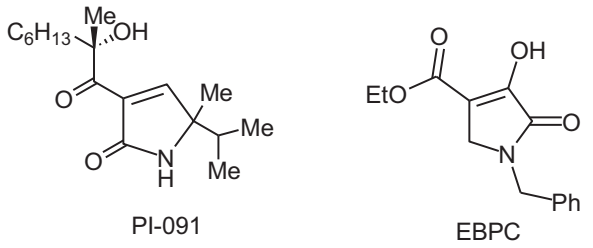
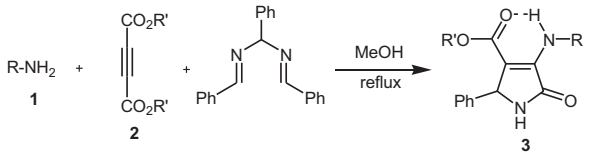


Fig. 1. Biologically active compounds having dihydro-2-oxypyrrole unit.



Scheme 1. Synthesis of 5-phenyl-2-oxopyrrole derivatives.

yield (Scheme 1). Different types of amines, such as benzyl and aliphatic amines were used to investigate the scope and limitation of the reaction.

A variety of benzylamines with substitutents Me, Cl at *para* position and aliphatic amines, such as propyl- and isobutylamine, were examined with DMAD and *N*,*N*′-bis(phenylmethylidene)phenylmethane under the same conditions and the corresponding dihydro-2-oxypyrrole derivatives **3a–i** were obtained in good yields as shown in Table 1.

Dialkyl acetylenedicarboxylate also showed very high reactivity in this reaction under the same conditions. All new compounds **3a–i** were fully characterized on the basis of elemental analysis, IR, ^{1}H NMR, ^{13}C NMR and mass spectra. The structure of product **3h** was further confirmed by X-ray crystallographic analysis (Fig. 2). The mass spectrum of **3a** displayed the molecular ion peak at the appropriate *m/z* value. The IR spectrum of compound **3a** showed two absorption bands due to the NH stretching

Table 1
5-Phenyl-2-oxopyrrole derivatives were prepared by the mentioned reaction.

Entry	Product	R	R′	Time (h)	Yield (%)
1	**3a**	Bn	Et	6	85
2	**3b**	4-Cl-Bn	Me	6	81
3	**3c**	4-Cl-Bn	Et	7	74
4	**3d**	4-Me-Bn	Me	5	77
5	**3e**	4-Me-Bn	Et	6	82
6	**3f**	Propyl	Me	6	74
7	**3g**	Propyl	Et	5	75
8	**3h**	Isobutyl	Me	5	80
9	**3i**	Isobutyl	Et	5	79

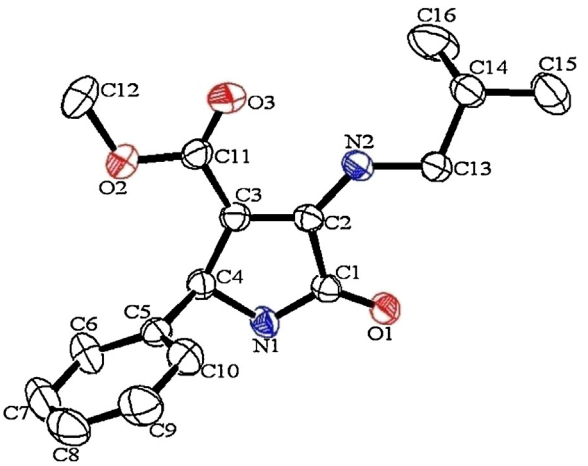
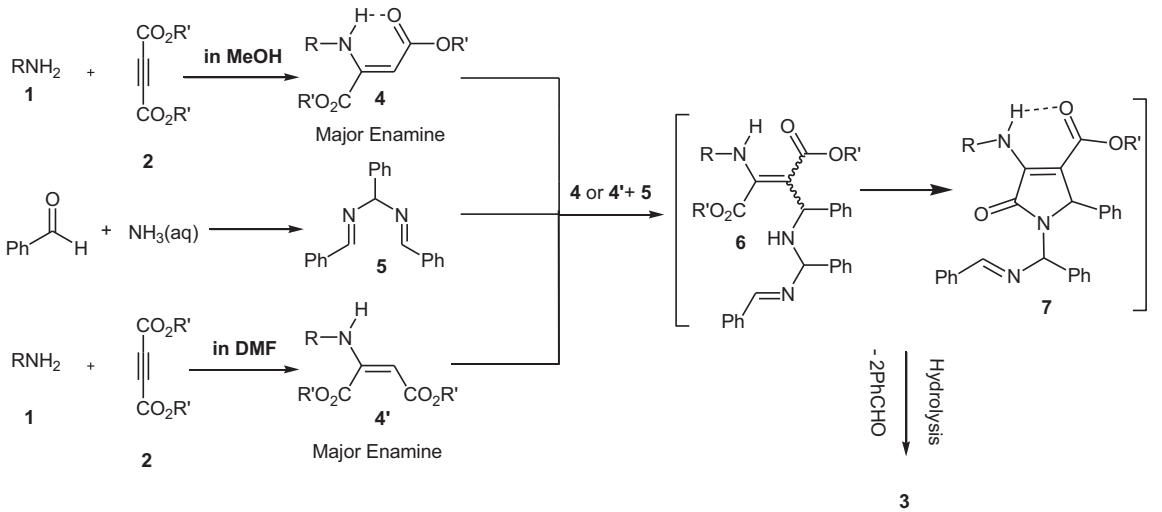


Fig. 2. X-ray crystal structure of compound **3h**.

frequency at 3338 and 3192 cm^{-1}, respectively. Absorption bands at 1705 and 1666 cm^{-1} are due to the COOEt and CONH groups, respectively. The ^{1}H NMR spectrum of **3a** showed a triplet for the CH_3 group (δ = 1.02 ppm, $^{3}J_{HH}$ = 7.1 Hz), three singlets for the two NH and CH groups



Scheme 2. Probing the mechanism for the formation of title compounds.

at δ = 1.6, 5.18 and 6.28, respectively, one quartet for the O–CH_2 group (δ = 3.99 ppm, $^3J_{HH}$ = 6.8 Hz), doublet of doublet for the CH_2 group (δ = 5.06 ppm, $^3J_{HH}$ = 6.6 Hz, and 5.15 ppm, $^3J_{HH}$ = 6.6 Hz), and the phenyl moiety gave rise to characteristic signals in the aromatic region of the spectrum. The ^{1}H-decoupled ^{13}C NMR spectrum of **3a** showed 16 distinct resonances in agreement with the suggested structure.

To explain the mechanism of this reaction, we propose that the compound **3** could result from the initial addition of the primary amines **1** to dialkyl acetylenedicarboxylate **2** and subsequent attack of the resulting enaminone **4** on the diamine to yield intermediate **6**. Cyclization of the intermediate **6** gives intermediate **7** and subsequent hydrolysis of intermediate **7** leads to compound **3**. To confirm the mechanism hypothesis initially, we mentioned Zhu et al.'s paper [15]. In this report, solvent has an important role in the stereochemistry of compound **4**. In MeOH, *Z/E* ratio is 95:5 and in DMF, this ratio is 2:98. After considering this issue, we carried out the reaction in MeOH as a protic solvent and DMF as an aprotic solvent in the same time and the progress of the reactions was followed by thin layer chromatography. After completion, the solvent was removed under reduced pressure and the residue was purified by column chromatography (*n*-hexane/EtOAc, 3:1). Then, by the spectral analysis of two product of reactions, we observed that this process was independent of the stereochemistry of the intermediate **4** (Scheme 2).

3. Conclusions

In summary, we have developed a simple, one-pot, three-component synthesis of 5-phenyl-2-oxopyrrole derivatives, which is of potential synthetic interest. High yields of the products, relatively short reaction times, using simple and cheap starting materials are the main advantages of this method. Also, the product could have high diversity via various functional groups instead of amine and ester groups. The simplicity of the present procedure makes it an interesting alternative to the complex multi-step approaches.

4. Experimental

4.1. Materials and techniques

Melting points were measured on an Electrothermal 9100 apparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. IR spectra were recorded on a Shimadzu IR-460 spectrometer. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. ^{1}H and ^{13}C NMR spectra were recorded at 500.1 and 125.7 MHz, respectively, on a BRUKER DRX 500-AVANCE FT-NMR instrument with $CDCl_3$ as solvent. The reagents and solvents used in this work were obtained from Fluka (Buchs, Switzerland) and were used without further purification.

4.2. General procedure for the preparation of compounds 3a–i, exemplified on 3a

To a magnetically stirred 5 mL flat bottom flask containing benzylamine (0.107 g, 1 mmol) was added diethyl acetylenedicarboxylate (0.170 g, 1 mmol). After 30 min, *N,N′*-bis (phenylmethylidene)phenylmethane (0.298 g, 1 mmol) was added to the reaction mixture which was allowed to stir for 6 h. Purification of the crude product by column chromatography [silica gel (Merck 230–240 mesh), *n*-hexane/EtOAc (6:1)] gave the title compound **3a**.

4.3. Spectral data

4.3.1. Ethyl 4-(benzylamino)-2,5-dihydro-5-oxo-2-phenyl-1H-pyrrole-3-carboxylate (3a)

White crystals, mp: 120–122 °C; IR (KBr) (ν_{max}, cm^{-1}): 3338 (NH), 3192 (NH), 2924 (CH), 1705 and 1666(C=O), 1457 (Ar). ^{1}H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm) 1.02 (2H, t, $^3J_{HH}$ = 7.1 Hz, CH_3), 1.60 (1H, s, NH), 3.99 (3H, q, $^3J_{HH}$ = 6.8 Hz, CH_3), 5.06 (1H, dd, $^3J_{HH}$ = 6.6 Hz, $^2J_{HH}$ = 15 Hz, CH), 5.15 (1H, dd, $^3J_{HH}$ = 6.6 Hz, $^2J_{HH}$ = 15 Hz, CH), 5.18 (1H, s, CH), 6.28 (1H, s, NH), 7.23-7.42 (10H, m, CH of Ar). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 14.03 (CH_3), 46.39 (CH_2–N), 57.80 (CH), 59.53 (CH_2–O), 107.45 (C–CH), 127.31 (2 CH of Ar), 127.38 (CH_{para} of Ph), 127.55 (2 CH of Ar), 128.09 (CH_{para} of Ph), 128.37 (2 CH of Ar), 128.70 (2 CH of Ar), 134.18 (C_{ipso}), 138.67 (2 C_{ipso}), 139.41 (C–NH), 165.90 (C=O), 167.53 (C=O). MS (EI, 70 eV): *m/z* (%) = 336 (M^+, 32), 307 (28), 263 (92), 91 (100). Anal. calcd for $C_{20}H_{20}N_2O_3$ (336.38): C, 71.41; H, 5.99; N, 8.33%. Found: C, 71.37; H, 5.89; N, 8.26%.

4.3.2. Methyl 4-(4-chlorobenzylamino)-2,5-dihydro-5-oxo-2-phenyl-1H-pyrrole-3-carboxylate (3b)

White crystals, mp: 164–166 °C; IR (KBr) (ν_{max}, cm^{-1}): 3324 (NH), 3196 (NH), 2942 (CH), 1689 (C=O), 1619 (Ar). ^{1}H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm)1.26 (1H, s, NH), 3.55 (3H, s, CH_3), 5.01 (1H, dd, $^3J_{HH}$ = 6.8 Hz, $^3J_{HH}$ = 14 Hz, CH), 5.14 (1H, dd, $^3J_{HH}$ = 6.7 Hz, $^3J_{HH}$ = 14 Hz, CH), 5.16 (1H, s, CH), 6.48 (1H, s, NH), 7.20–7.32 (9H, m, CH of Ar). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 45.55 (CH_2–N), 50.75 (CH), 57.77 (OCH_3), 106.00 (C–CH), 127.14 (2 CH of Ar), 128.29 (CH_{para} of Ph), 128.51 (2 CH of Ar), 128.83 (2 CH of Ar), 128.88 (2 CH of Ar), 132.54 (C–Cl), 133.18 (C_{ipso}), 138.03(C_{ipso}), 138.45 (C–NH), 165.95 (C=O), 167.34 (C=O). MS (EI, 70 eV): *m/z* (%) = 356 (M^+, 26), 323(9), 297 (88), 125 (100). Anal. calcd for $C_{19}H_{17}ClNO_2$ (356.09): C, 63.96; H, 4.80; N, 7.85%. Found: C, 63.90; H, 4.74; N, 7.75%.

4.3.3. Ethyl 4-(4-chlorobenzylamino)-2,5-dihydro-5-oxo-2-phenyl-1H-pyrrole-3-carboxylate (3c)

White crystals, mp: 128–130 °C; IR (KBr) (ν_{max}, cm^{-1}): 3330 (NH), 3231 (NH), 2923 (CH), 1691 and 1661 (C=O), 1604 (Ar). ^{1}H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm) 1.03 (2H, t, $^3J_{HH}$ = 7.3 Hz, CH_2), 4.00 (3H, q, $^3J_{HH}$ = 7.1 Hz, CH_3), 5.01 (1H, dd, $^3J_{HH}$ = 6.9 Hz, $^3J_{HH}$ = 15 Hz, CH), 5.10 (1H, dd, $^3J_{HH}$ = 6.8 Hz, $^3J_{HH}$ = 15 Hz, CH), 5.17 (1H, s, CH), 6.34 (1H, s, NH), 7.04 (1H, s, NH), 7.21–7.32 (9H, m, 9CH of Ar). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 14.01 (CH_3), 45.59

(CH_2–N), 57.83 (CH), 59.63 (CH_2–O), 106.45 (C–CH), 127.27 (2 CH of Ar), 128.14 (CH_{para} of Ph), 128.39 (2 CH of Ar), 128.82 (2 CH of Ar), 128.89 (2 CH of Ar), 133.16 (C–Cl), 138.08 (2 C_{ipso}), 138.49 (C–NH), 165.41 (C=O), 167.45 (C=O). MS (EI, 70 eV): *m/z* (%) = 370 (M^+, 28), 341 (29), 297 (95), 125 (100). Anal. calcd for $C_{20}H_{19}ClN_2O_3$ (370.83): C, 64.78; H, 5.16; N, 7.55%. Found: C, 64.71; H, 5.09; N, 7.48%.

4.3.4. Methyl 4-(4-methylbenzylamino)-2,5-dihydro-5-oxo-2-phenyl-1H-pyrrole-3-carboxylate (3d)

White crystals, mp: 136–138 °C; IR (KBr) (ν_{max}, cm^{-1}): 3323 (NH), 3194 (NH), 2930 (CH), 1694 and 1667 (C=O), 1617 (Ar). ^1H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm) 2.35 (3H, s, CH_3), 3.54 (3H, s, OCH_3), 5.00 (1H, dd, $^3J_{HH}$ = 6.8 Hz, $^3J_{HH}$ = 15 Hz, CH), 5.08 (1H, dd, $^3J_{HH}$ = 6.7 Hz, $^3J_{HH}$ = 15 Hz, CH), 5.16 (1H, s, CH), 6.50 (1H, s, NH), 7.16 (2H, d, $^3J_{HH}$ = 7.8 Hz, 2CH of Ar), 7.23–7.32 (7H, m, 7 CH of Ar), 7.36 (1H, s, NH). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 21.10 (CH_3), 46.16 (CH_2–N), 50.72 (CH), 57.80 (OCH_3), 106.05 (C–CH), 127.19 (2 CH of Ar), 127.56 (2 CH of Ar), 128.09 (CH_{para} of Ph), 128.47 (2 CH of Ar), 129.39 (2 CH of Ar), 132.47 (C–Me), 136.36 (C_{ipso}), 137.04 (C_{ipso}), 138.73 (C–NH), 165.92 (C=O), 167.50 (C=O). MS (EI, 70 eV): *m/z* (%) = 336 (M^+, 89), 277 (92), 232 (31), 105 (100), 77(53). Anal. calcd for $C_{20}H_{20}N_2O_3$ (336.38): C, 71.41; H, 5.99; N, 8.33%. Found: C, 71.35; H, 5.91; N, 8.25%.

4.3.5. Ethyl 4-(4-methylbenzylamino)-2,5-dihydro-5-oxo-2-phenyl-1H-pyrrole-3-carboxylate (3e)

White crystals, mp: 102–104 °C; IR (KBr) (ν_{max}, cm^{-1}): 3347 (NH), 3209 (NH), 2924 (CH), 1712 and 1666 (C=O), 1621 (Ar). ^1H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm) 1.02 (2H, t, $^3J_{HH}$ = 7.1 Hz, CH_2), 1.61 (1H, s, NH), 2.34 (3H, s, CH_3), 3.98 (2H, q, $^3J_{HH}$ = 7.1 Hz, CH_2), 5.00 (1H, dd, $^3J_{HH}$ = 6.8 Hz, $^3J_{HH}$ = 15 Hz, CH), 5.09 (1H, dd, $^3J_{HH}$ = 6.8 Hz, $^3J_{HH}$ = 15 Hz, CH), 5.17 (1H, s, CH), 6.28 (1H, s, NH), 7.16 (2H, d, $^3J_{HH}$ = 7.9 Hz, 2CH of Ar), 7.21–7.31 (7H, m, 7 CH of Ar). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 14.04 (CH_3), 21.10 (CH_3), 46.19 (CH_2–N), 57.82 (CH), 59.51 (CH_2–O), 105.78 (C–CH), 127.31 (2 CH of Ar), 127.56 (2 CH of Ar), 128.06 (CH_{para} of Ph), 128.35 (2 CH of Ar), 129.37 (2 CH of Ar), 136.39 (C–Me), 137.02 (2 C_{ipso}), 138.74 (C–NH), 165.42 (C=O), 167.56 (C=O). MS (EI, 70 eV): *m/z* (%) = 350 (M^+, 10), 321 (18), 277 (16), 105 (100), 77 (38). Anal. calcd for $C_{21}H_{22}N_2O_3$ (350.41): C, 71.98; H, 6.33; N, 7.99%. Found: C, 71.93; H, 6.29; N, 7.91%.

4.3.6. Methyl 2,5-dihydro-5-oxo-2-phenyl-4-(propylamino)-1H-pyrrole-3-carboxylate (3f)

White crystals, mp: 143–145 °C; IR (KBr) (ν_{max}, cm^{-1}): 3325 (NH), 3196 (NH), 2946 (CH), 1692 (C=O), 1633 (Ar). ^1H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm) 0.96 (3H, t, $^3J_{HH}$ = 7.4 Hz, CH_3), 1.58–1.65 (2H, m, CH_2), 3.55 (3H, s, CH_3), 3.78–3.84 (2H, m, CH_2), 5.14 (1H, s, CH), 6.46 (1H, s, NH), 6.98 (1H, s, NH), 7.19–7.30 (5H, m, 5 CH of Ar). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 11.09 (CH_3), 24.55 (CH_2), 44.26 (CH_2–NH), 50.61 (CH), 57.75 (OCH_3), 104.16 (C–CH), 127.15 (2 CH of Ar), 128.02 (CH_{para} of Ph), 128.42 (2 CH of Ar), 137.12 (C_{ipso}), 138.97 (C–NH), 165.99 (C=O), 167.47 (C=O). MS (EI, 70 eV): *m/z* (%) = 274 (M^+, 51), 245 (11), 215 (100), 173 (32), 77 (28). Anal. calcd for $C_{15}H_{18}N_2O_3$ (274.32): C, 65.68; H, 6.61; N, 10.21%. Found: C, 65.62; H, 6.55; N, 10.16%.

4.3.7. Ethyl 2,5-dihydro-5-oxo-2-phenyl-4-(propylamino)-1H-pyrrole-3-carboxylate (3g)

White crystals, mp: 169–171 °C; IR (KBr) (ν_{max}, cm^{-1}): 3284 (NH), 2951 (CH), 1710 and 1675 (C=O), 1611(Ar). ^1H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm) 0.99 (3H, t, $^3J_{HH}$ = 7.5 Hz,CH_3), 1.05 (3H, t, $^3J_{HH}$ = 7.4 Hz, CH_3), 1.59–1.67 (2H, m, CH_2), 3.79–3.86 (2H, m, CH_2), 4.01 (2H, q, $^3J_{HH}$ = 7.4 Hz, CH_2), 5.16 (1H, s, CH), 6.22 (1H, s, NH), 6.97 (1H, s, NH), 7.23-7.37 (5H, m, 5 CH of Ar). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 11.11 (CH_3), 14.08 (CH_3), 24.54 (CH_2), 44.27 (CH_2-NH), 50.71 (CH), 59.39 (CH_2-O), 104.20 (C-CH), 127.28 (2 CH of Ar), 128.00 (CH_{para} of Ph), 128.32 (2 CH of Ar), 137.99 (C_{ipso}), 138.98 (*C*-NH), 165.97 (C = O), 167.53 (C = O). MS (EI, 70 eV): *m/z* (%) = 288 (M^+, 6), 232 (14), 125 (95), 105 (100), 77 (28). Anal. calcd for $C_{16}H_{20}N_2O_3$ (288.34): C, 66.65; H, 6.99; N, 9.72%. Found: C, 66.60; H, 6.92; N, 9.67%.

4.3.8. Methyl 2,5-dihydro-4-(isobutylamino)-5-oxo-2-phenyl-1H-pyrrole-3-carboxylate (3h)

White crystals, mp: 130–132 °C; IR (KBr) (ν_{max}, cm^{-1}): 3327 (NH), 3192 (NH), 2945 (CH), 1689 and 1645 (C=O), 1614 (Ar). ^1H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm) 0.96 (3H, d, $^3J_{HH}$ = 1.6 Hz, CH_3), 0.97 (3H, d, $^3J_{HH}$ = 1.6 Hz, CH_3), 1.80–1.84 (1H, m, CH), 3.55 (3H, s, CH_3), 3.64–3.73 (2H, m, CH_2), 5.14 (1H, s, CH), 6.55 (1H, s, NH), 7.03 (1H, s, NH), 7.21–7.32 (5H, m, 5 CH of Ar). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 19.80 (CH_3), 30.03 (CH), 49.56 (CH_2–NH), 50.61 (CH), 56.67 (OCH_3), 104.18 (C–CH), 127.14 (2 CH of Ar), 128.00 (CH_{para} of Ph), 128.41(2 CH of Ar), 138.13 (C_{ipso}), 139.00 (C–NH), 165.85 (C=O), 167.49 (C=O). MS (EI, 70 eV): *m/z* (%) = 288 (M^+, 11), 245 (16), 229 (65), 104 (32), 77 (100). Anal. calcd for $C_{16}H_{20}N_2O_3$ (288.34): C, 66.65; H, 6.99; N, 9.72%. Found: C, 66.59; H, 6.94; N, 9.66%. Crystal data for **3h** $C_{16}H_{20}N_2O_3$ (CCDC 876237): M_W = 285.32, monoclinic, space group *P*21/*c*, *a* = 5.359(5) Å, *b* = 11.357(5) Å, *c* = 13.755(5) Å, α = 68.388(5) β = 87.730(5), γ = 88.346(5), *V* = 777.6(9) $Å^3$, *Z* = 2, D_c = 1.219 mg/m^3, F (000) = 302, crystal dimensions 0.19 × 0.12 × 0.11 mm, radiation, Mo Kα (λ = 0.71073 Å), $2.9 \leq 2\theta \leq 25.1$, intensity data were collected at 295(2) K with a Bruker APEX area-detector diffractometer, and employing $\omega/2\theta$ scanning technique, in the range of $-6 \leq h \leq 6$, $-12 \leq k \leq 13$, $-15 \leq l \leq 16$; the structure was solved by a direct method, all non-hydrogen atoms were positioned and anisotropic thermal parameters refined from 2035 observed reflections with *R* (into) = 0.0880 by a full-matrix least-squares technique converged to *R* = 0.0666 and Raw = 0.1928 [I > 2sigma(I)].

4.3.9. Ethyl 2,5-dihydro-4-(isobutylamino)-5-oxo-2-phenyl-1H-pyrrole-3-carboxylate (3i)

White crystals, mp: 154–156 °C; IR (KBr) (ν_{max}, cm^{-1}): 3352 (NH), 3189 (NH), 2957 (CH), 1691 (C = O), 1621 (Ar). ^1H NMR (500.13 MHz, $CDCl_3$): δ_H (ppm) 0.97 (6H, d, $^3J_{HH}$ = 6.6 Hz,CH_3), 1.05 (3H, t, $^3J_{HH}$ = 7.5 Hz,CH_3), 1.81–1.86 (1H, m, CH), 3.65-3.75 (2H, m, CH_2), 4.01 (2H, q, $^3J_{HH}$ = 7.5 Hz, CH_2), 5.15 (1H, s, CH), 6.34 (1H, s, NH),

7.02 (1H, s, NH), 7.22-7.34 (5H, m, 5 CH of Ar). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C (ppm) 13.53 (CH_3), 19.27 (CH_3), 29.48 (CH), 49.17 (CH), 57.14 (CH_2–NH), 58.83 (O-CH_2), 104.18 (C–CH), 126.58 (2 CH of Ar), 127.42 (CH_{para} of Ph), 127.76 (2 CH of Ar), 138.10 (C_{ipso}), 139.45 (C–NH), 165.88 (C=O), 167.03 (C=O). MS (EI, 70 eV): *m/z* (%) = 302 (M^+, 25), 259 (27), 229 (100), 213(65), 173 (18), 77 (15). Anal. calcd for $C_{17}H_{22}N_2O_3$ (302.37): C, 67.53; H, 7.33; N, 9.26%. Found: C, 67.48; H, 7.29; N, 9.18%.

Acknowledgements

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