

N-methyl imidazole or ammonium thiocyanate promoted synthesis of substituted pyrroles: Multicomponent reaction of alkyl propiolates in water

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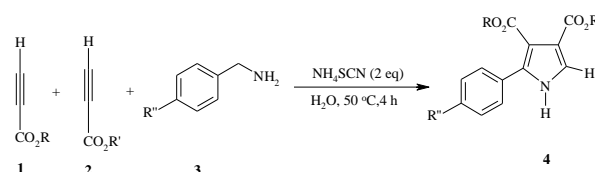
A novel, convenient, and efficient approach to the synthesis of substituted pyrroles is reported based on a three-component reaction. The reaction of primary amines with electron deficient acetylenic compounds in the presence of N-methylimidazole or ammonium thiocyanate in water lead to the formation of pyrroles in good yield.

Keywords: Water, Primary amine, N-methylimidazol, pyrroles, Three-component reaction, Green Chemistry.

INTRODUCTION

At the beginning of the new century, a move in importance in chemistry is obvious with the longing to extend environmentally gentle routes to a numerous of materials [1]. Green chemistry approaches hold out momentous potential not only for reduction of byproducts, a reduction in the waste produced, and lowering of energy costs but also in the development of new methodologies toward previously unobtainable materials, using existing technologies [2]. Of all of the existing areas of chemistry, medicinal and pharmaceutical chemistry, with their traditionally large volume of waste/product ratio, are maybe the most developed for greening [3]. Multicomponent reactions (MCRs) have been commonly employed by synthetic chemists as a too easy means to produce molecular diversity from bifunctional substrates that react successively in a intramolecular way [4]. Five membered, nitrogen-containing heterocycles are main building blocks in a broad number of biologically active compounds [5]. Among them, pyrroles are heterocycles of enormous importance because of their presence in several natural products like heme, chlorophyll, vitamin B₁₂, and various cytochrome enzymes [6]. Some of the recently isolated pyrrole-containing marine natural products have been set up to display considerable cytotoxicity and function as multidrug resistant reversal agents [7]. Many of these biologically active compounds have appeared as chemotherapeutic agents. In addition, substituted pyrroles are molecular skeleton having enormous importance in material science [8]. They have been also used as antioxidants, antibacterial, ionotropic,

antitumor, anti inflammatory and antifungal agents [9-14]. There are many methods for the synthesis of pyrroles [15-24]. As part of our current studies on the development of new routes in heterocyclic synthesis, we report an efficient synthesis of pyrrole derivatives **4** in good yield (Scheme 1).



1, 2, 3, 4	R	R'	R''	Yield (%) of 4
a	Me	Me	H	75
b	Me	Et	Me	87
c	Et	Me	MeO	70
d	Et	Et	Me	75

Scheme 1. Reaction of propiolate with benzyl amine in the presence of ammonium thiocyanate.

EXPERIMENTAL

Apparatus and analysis

All chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification. 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. Mass spectra were recorded on a FINNIGAN-MAT 8430 spectrometer operating at an ionization potential of 70 eV. IR spectra were measured on a Shimadzu IR-460 spectrometer. ¹H and ¹³C NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500.1 and 125.8 MHz, respectively. ¹H, ¹³C, spectra were obtained for solutions in CDCl₃ using TMS as the internal standard or 85% H₃PO₄ as the external standard.

General procedure for preparation of compounds

4: To a stirred mixture of amine **3** (2 mmol) and acetylenic ester **2** (2 mmol) in water (5 mL) was

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added mixture of NH₄SCN (2 mmol) and acetylenic ester **1** (2 mmol) in water at 50 °C. After completion of the reaction (4 h; TLC (AcOEt/hexane 1:5) monitoring), the residue was extracted by AcOEt and washed by cold diethyl ether to give pure product.

General procedure for preparation of compounds 16: To a stirred mixture of alkyl propiolate **13** (2 mmol) and primary amine **14** (2 mmol) in water (5 mL) was added mixture of alkyl propiolate **15** and *N*-methylimidazole (5 mol%) in water (5 mL). The reaction mixture was then stirred for 1.5 h at 50 °C. After completion of the reaction [1.5 h; TLC (AcOEt/hexane 1:4) monitoring], the solid residue was filtered and washed by cold diethyl ether to give pure product **16**.

Dimethyl-2-phenyl-1H-pyrrole-3,4-dicarboxylate (4a): Yellow oil; yield: 0.39 g (75%). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1725, 1634, 1487. ¹H NMR (500.1 MHz, CDCl₃): δ = 3.75 (3 H, s, MeO), 3.82 (3 H, s, MeO), 6.87 (1 H, s, CH), 7.12 (2 H, d, ³*J* = 7.5 Hz, CH), 7.42 (3 H, m, CH), 9.32 (1 H, br s, NH) ppm. ¹³C NMR (125.7 MHz, CDCl₃): δ = 51.2 (MeO), 51.8 (MeO), 112.4 (C), 125.8 (CH), 127.5 (2 CH), 128.4 (C), 128.7 (2 CH), 129.2 (CH), 132.0 (C), 143.4 (C), 162.4 (C=O), 163.8 (C=O) ppm. MS: *m/z* (%) = 259 (M⁺, 10), 228 (46), 91 (58), 77 (87), 31 (100). Anal. Calc. for C₁₄H₁₃NO₄ (259.26): C, 64.86; H, 5.05; N, 5.40; Found: C, 64.92; H, 5.14; N, 5.52.

4-ethyl-3-methyl-2-(4-methylphenyl)-1H-pyrrole-3,4-dicarboxylate (4b): Pale yellow oil; yield: 0.49 g (87%). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1727, 1654, 1587, 1465. ¹H NMR (500.1 MHz, CDCl₃): δ = 1.24 (3 H, t, ³*J* = 7.4 Hz, CH₃), 2.42 (3 H, s, Me), 3.85 (3 H, s, MeO), 4.18 (2 H, q, ³*J* = 7.4 Hz, CH₂O), 7.12 (1 H, s, CH), 7.22 (2 H, d, ³*J* = 7.8 Hz, 2 CH), 7.38 (2 H, d, ³*J* = 7.8 Hz, 2 CH), 9.28 (1 H, br s, NH) ppm. ¹³C NMR (125.7 MHz, CDCl₃): δ = 13.8 (CH₃), 22.5 (Me), 52.7 (MeO), 61.4 (CH₂O), 115.2 (C), 125.4 (CH), 126.5 (2 C), 129.4 (C), 130.4 (2 CH), 131.2 (2 CH), 144.7 (C), 162.3 (C=O), 163.8 (C=O) ppm. MS: *m/z* (%) = 287 (M⁺, 15), 256 (45), 242 (58), 105 (100), 77 (86), 31 (100). Anal. Calc. for C₁₆H₁₇NO₄ (287.31): C, 66.89; H, 5.96; N, 4.88; Found: C, 66.93; H, 6.04; N, 4.92.

3-ethyl-4-methyl-2-(4-methoxyphenyl)-1H-pyrrole-3,4-dicarboxylate (4c): Yellow oil; yield: 0.42 g (70%). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1724, 1627, 1545, 1462, 1335, 1275. ¹H NMR (500.1 MHz, CDCl₃): δ = 1.35 (3 H, t, ³*J* = 7.4 Hz, CH₃), 3.75 (3 H, s, MeO), 3.94 (3 H, s, MeO), 4.32 (2 H, t, ³*J* = 7.5 Hz, CH₂O), 6.94 (1 H, s, CH), 7.12 (2 H, d, ³*J* = 7.8 Hz, CH), 7.46 (2 H, d, ³*J* = 7.8 Hz, CH), 9.24 (1

H, br s, NH) ppm. ¹³C NMR (125.7 MHz, CDCl₃): δ = 14.2 (CH₃), 51.6 (MeO), 55.6 (MeO), 62.4 (CH₂O), 114.7 (2 CH), 115.2 (CH), 120.4 (C), 123.6 (C), 130.2 (C), 131.5 (2 CH), 141.7 (C), 160.2 (C), 161.7 (C=O), 164.5 (C=O) ppm. MS: *m/z* (%) = 303 (M⁺, 8), 272 (65), 182 (54), 121 (100), 77 (65), 31 (100). Anal. Calc. for C₁₆H₁₇NO₅ (303.31): C, 63.36; H, 5.65; N, 4.62; Found: C, 63.43; H, 5.74; N, 4.73.

Diethyl-2-(4-methylphenyl)-1H-pyrrole-3,4-tricarboxylate (4d): Yellow oil; yield: 0.45 g (75%). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1728, 1637, 1587, 1465, 1346, 1237. ¹H NMR (500.1 MHz, CDCl₃): δ = 1.27 (3 H, t, ³*J* = 7.2 Hz, CH₃), 1.35 (3 H, t, ³*J* = 7.5 Hz, CH₃), 2.38 (3 H, s, Me), 4.22 (2 H, q, ³*J* = 7.4 Hz, CH₂O), 4.37 (2 H, q, ³*J* = 7.5 Hz, CH₂O), 6.97 (1 H, s, CH), 7.26 (2 H, d, ³*J* = 7.5 Hz, 2 CH), 7.43 (2 H, d, ³*J* = 7.6 Hz, 2 CH), 9.23 (1 H, br s, NH) ppm. ¹³C NMR (125.7 MHz, CDCl₃): δ = 13.4 (Me), 13.8 (Me), 20.4 (Me), 61.4 (CH₂O), 61.8 (CH₂O), 116.4 (CH), 121.8 (C), 128.3 (C), 129.5 (2 CH), 130.2 (2 CH), 140.6 (C), 144.8 (C), 160.2 (C), 161.4 (C=O), 162.5 (C=O) ppm. MS: *m/z* (%) = 301 (M⁺, 10), 196 (85), 105 (100), 77 (44), 45 (87). Anal. Calc. for C₁₇H₁₉NO₄ (301.34): C, 67.76; H, 6.35; N, 4.65; Found: C, 67.83; H, 6.42; N, 4.74.

Dimethyl-1-methyl-1H-pyrrole-3,4-dicarboxylate (16a): Pale yellow powder, m.p. 173-175 °C, Yield: 0.36 g (92%) IR (KBr): 1735, 1729, 1587, 1435, 1295, 1126 cm⁻¹. ¹H NMR: 3.58 (3 H, s, NMe), 3.78 (6 H, s, 2 MeO), 6.92 (2 H, s, 2 CH) ppm. ¹³C NMR: 35.8 (NMe), 51.8 (2 MeO), 137.2 (2 C), 138.3 (2 CH), 165.4 (2 C=O) ppm. EI-MS: 197 (M⁺, 15), 135 (85), 79 (64), 31 (100). Anal. Calcd for C₉H₁₁NO₄ (197.19): C 54.82, H 5.62, N 7.10; Found: C 54.93, H 5.74, N 7.22.

Dimethyl-1-ethyl-1H-pyrrole-3,4-dicarboxylate (16b): Yellow powder, m.p. 167-169 °C, Yield: 0.37 g (87%). IR (KBr): 1730, 1727, 1562, 1454, 12876 cm⁻¹. ¹H NMR: 1.23 (3 H, t, ³*J* = 7.4 Hz, Me), 3.58 (2 H, q, ³*J* = 7.4 Hz, NCH₂), 3.82 (6 H, s, 2 MeO), 6.87 (2 H, s, 2 CH) ppm. ¹³C NMR: 14.2 (Me), 48.3 (NCH₂), 52.4 (2 MeO), 137.5 (2 C), 139.0 (2 CH), 166.2 (2 C=O) ppm. EI-MS: 211 (M⁺, 10), 121 (76), 79 (58), 45 (100). Anal. Calcd for C₁₀H₁₃NO₄ (211.22): C 56.86, H 6.20, N 6.63; Found: C 56.74, H 6.14, N 6.52.

Dimethyl-1-butyl-1H-pyrrole-3,4-dicarboxylate (16c): Pale yellow powder, m.p. 178-180 °C, Yield: 0.41 g (85%) IR (KBr): 1728, 1725, 1545, 1378, 1268, 1226 cm⁻¹. ¹H NMR: 0.92 (3 H, t, ³*J* = 7.3 Hz, Me), 1.27 (2 H, m, CH₂), 1.52 (2 H, m, CH₂), 3.62 (2 H, t, ³*J* = 7.3 Hz, NCH₂), 3.75 (6 H, s, 2 MeO), 6.86 (2 H, s, 2 CH) ppm. ¹³C NMR: 13.2 (Me), 18.6 (CH₂), 32.4 (CH₂), 52.2 (2 MeO), 53.3 (NCH₂), 135.4 (2 C), 137.6 (2 CH), 167.0 (2 C=O) ppm. EI-MS: 239 (M⁺,

15), 208 (88), 31(100). Anal. Calcd for $C_{12}H_{17}NO_4$ (239.27): C 60.24, H 7.16, N 5.85; Found: C 60.33, H 7.25, N 5.92.

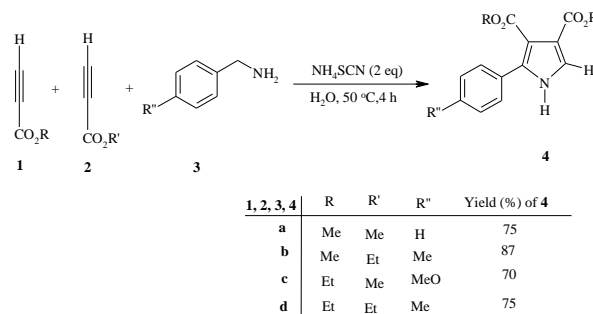
3-Ethyl-4-methyl-1-methyl-1*H*-pyrrole-3,4-dicarboxylate (16d): Yellow powder, m.p. 154-156°C, Yield: 0.34 g (80%) IR (KBr): 1735, 1730, 1587, 1345, 1347, 1252 cm^{-1} . 1H NMR: 1.22 (3 H, t, $^3J = 7.2$ Hz, Me), 3.64 (3 H, s, NMe), 3.78 (3 H, s, MeO), 4.28 (2 H, q, $^3J = 7.2$ Hz, CH_2O), 7.12 (1 H, s, CH), 7.22 (1 H, s, CH) ppm. ^{13}C NMR: 13.7 (Me), 37.3 (NMe), 61.6 (CH_2O), 134.5 (C), 135.8 (CH), 136.4 (CH), 137.2 (C), 164.5 (C=O), 167.4 (C=O) ppm. EI-MS: 211 (M^+ , 15), 180 (68), 45 (87), 31 (100). Anal. Calcd for $C_{10}H_{13}NO_4$ (211.22): C 56.86, H 6.20, N 6.63; Found: C 56.93, H 6.33, N 5.6.72.

Dimethyl- 1-(4-methylbenzyl)-1*H*-pyrrole-3,4-dicarboxylate (16e). Yellow powder, m.p. 185-187 °C, Yield: 0.45 g (78%) IR (KBr): 1737, 1734, 1687, 1597, 1465, 1254 cm^{-1} . 1H NMR: 2.38 (Me), 3.62 (3 H, s, NCH_2), 3.75 (3 H, s, MeO), 3.78 (3 H, s, MeO), 6.74 (1 H, s, CH), 6.85 (1 H, s, CH), 7.29 (2 H, d, $^3J = 7.6$ Hz, 2 CH), 7.36 (2 H, d, $^3J = 7.6$ Hz, CH) ppm. ^{13}C NMR: 21.4 (Me), 51.2 (MeO), 52.3 (MeO), 61.7 (NCH_2), 127.6 (2 CH), 128.5 (2 CH), 132.4 (C), 134.5 (C), 134.6 (CH), 135.8 (CH), 136.2 (C), 137.4 (C), 167.3 (C=O), 169.5 (C=O) ppm. EI-MS: 287 (M^+ , 10), 181 (87), 105 (100), 77 (46), 31(100). Anal. Calcd for $C_{16}H_{17}NO_4$ (287.31): C 66.89, H 5.96, N 4.88; Found: C 66.95, H 6.07, N 4.97.

3-Ethyl-4-methyl-1-(4-methoxybenzyl)-1*H*-pyrrole-3,4-dicarboxylate (16f). Yellow powder, m.p. 182-184 °C, Yield: 0.53 g (83%) IR (KBr): 1733, 1728, 1694, 1587, 1486, 1375 cm^{-1} . 1H NMR: 1.27 (3 H, t, $^3J = 7.4$ Hz, Me), 3.68 (3 H, s, NCH_2), 3.75 (3 H, s, MeO), 3.82 (3 H, s, MeO), 4.32 (2 H, q, $^3J = 7.4$ Hz, CH_2O), 6.65 (1 H, s, CH), 6.74 (1 H, s, CH), 7.15 (2 H, d, $^3J = 7.5$ Hz, 2 CH), 7.23 (2 H, d, $^3J = 7.5$ Hz, CH) ppm. ^{13}C NMR: 14.2 (Me), 52.3 (MeO), 55.4 (MeO), 61.4 (CH_2O), 62.4 (NCH_2), 114.2 (2 CH), 132.4 (2 CH), 132.8 (C), 134.3 (CH), 134.8 (CH), 135.2 (C), 136.5 (C), 158.7 (C), 165.3 (C=O), 167.2 (C=O) ppm. EI-MS: 317 (M^+ , 10), 286 (88), 272 (82), 196 (68), 121 (100). Anal. Calcd for $C_{17}H_{19}NO_5$ (317.34): C 64.34, H 6.03, N 4.41; Found: C 64.42, H 6.15, N 4.52.

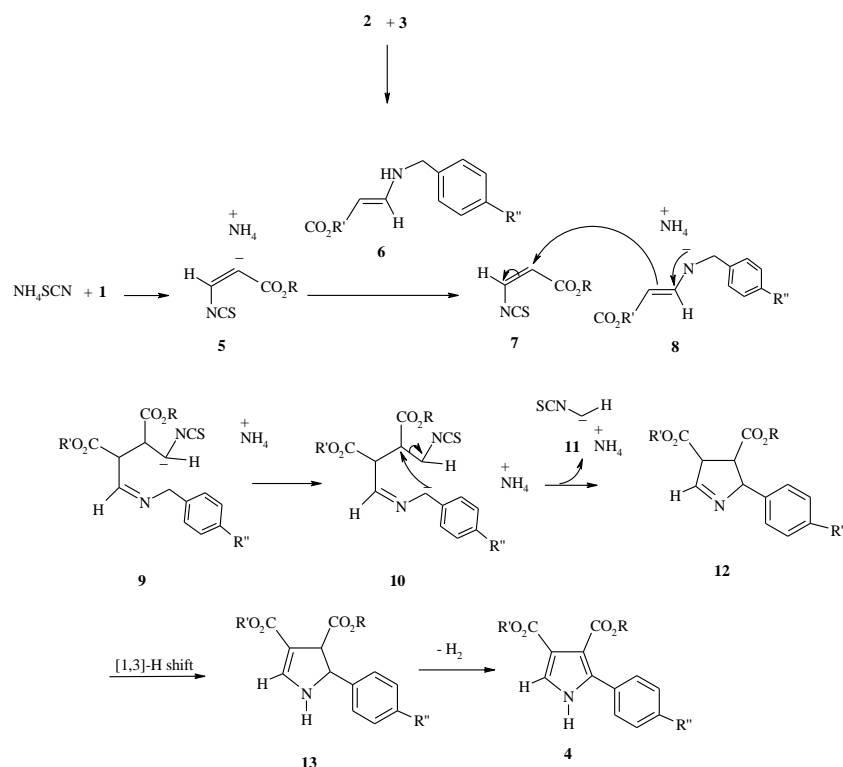
RESULTS AND DISCUSSION

The reaction of propiolates **1**, **2** with benzyl amines **3** in the presence of ammonium thiocyanate produce pyrrole derivatives **4** in excellent yield (Scheme 1). In these reactions the order of adding reagent is important. In compound **4b**, ammonium thiocyanate was mixed with methyl propiolate **1** for 1 h at 50 °C and added to mixture of amine and ethyl propiolate **2** that mixed for 1 h at 50 °C, but in compound **4c** is reverse, ammonium thiocyanate was mixed with ethyl propiolate **1** for 1 h at 50 °C and added to mixture of amine and methyl propiolate **2** that mixed for 1 h at 50 °C. The two component reaction of amine and propiolates is in the mixture of reaction but very low yield (10%). These reactions were not performed with aliphatic primary amines. Although there are many articles for the synthesis of pyrroles [15-20], the number of methods for synthesis of substituted pyrroles caused by benzylic oxidative cyclization, is restricted [25].



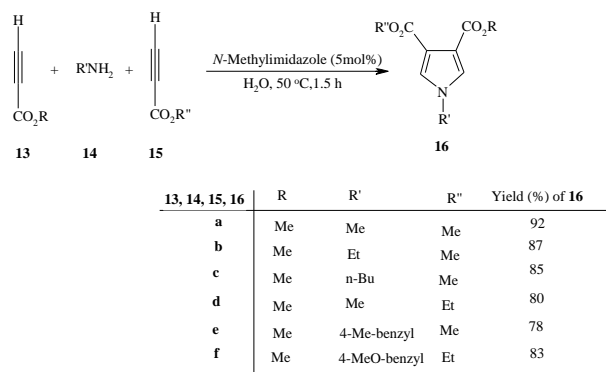
Scheme 1. Reaction of propiolate and benzyl amine in the presence of ammonium thiocyanate.

The structures of compounds **4** were assigned by IR, 1H NMR, ^{13}C NMR and mass spectral data. For example, the 1H NMR spectrum of **4a** exhibited two singlets for two methoxy protons at (δ 3.75 and 3.82 ppm) and one singlet for methin protons at (δ 6.87 ppm). The ^{13}C NMR spectrum of **4a** exhibited carbonyl resonance at 162.4 and 163.8 ppm which further confirmed the proposed structure. Probably, the first event includes protonation of the zwitterionic intermediate **5** formed from NH_4SCN and **1**, by the enaminoester intermediate **6** generated *in situ* from the primary amine **3** and acetylenic ester **2** to produce intermediates **7** and **8**. Then, nucleophilic attack of the conjugate base **8** on intermediate **7** leads to adduct **9**, which undergoes intramolecular protontransfer reactions to afford **10**. Intermediate **10** undergoes intramolecular cyclization by elimination of salt **11** to generate the dihydropyrrol derivative **12**, which is converted to desired product **4** by [1,3]-H shift and air oxidation (Scheme 2).



Scheme 2. Proposed mechanism for the synthesis of compound **4**.

Under similar conditions, the reaction of propiolates **13**, **15** and primary amines **14** in the presence of *N*-methyl imidazole produced pyrrole derivatives **16** in excellent yield (Scheme 3).



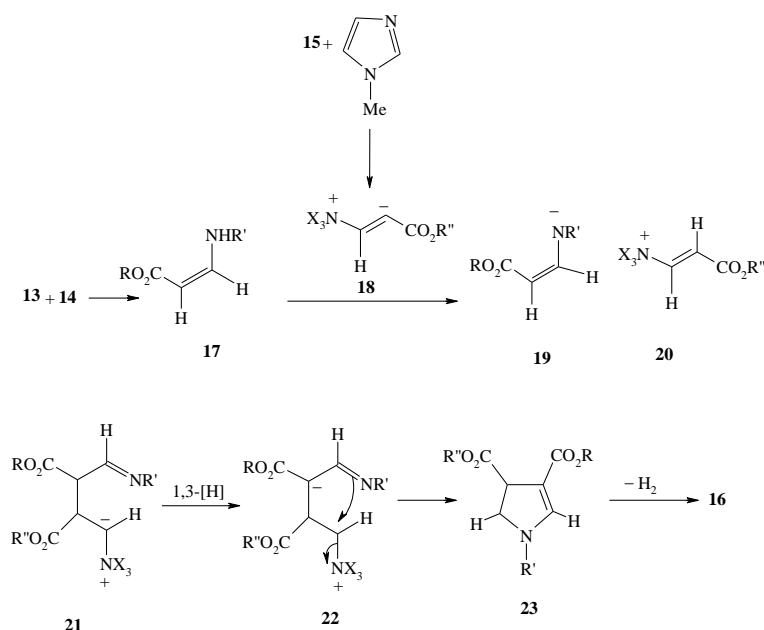
Scheme 3. Reaction of propiolate and primary amines in the presence of *N*-methyl imidazole.

The structures of compounds **16** were assigned by IR, ^1H NMR, ^{13}C NMR and mass spectral data. For example, the ^1H NMR spectrum of **16a** exhibited one singlet for two methoxy protons at (δ 3.78 ppm), one singlet for NMe protons at (δ 3.58 ppm) and one singlet for two methin groups at (δ 6.92 ppm). The ^{13}C NMR spectrum of **16a** exhibited 9 distinct resonances which further confirmed the proposed structure. The IR spectrum of **16a** displayed characteristic C=O bands. The mass spectra of **16a** displayed the molecular ion peak at the appropriate m/z . Presumably, the

zwitterionic intermediate **18**, formed from *N*-methylimidazole (X_3N) and alkyl propiolate **15**, is protonated by the enaminoester **17**, generated *in situ* from primary amine **14** and alkylpropiolate **13**, to produce intermediates **19** and **20** (Scheme 4). Nucleophilic attack of the conjugate base **19** to intermediate **20** leads to adduct **21**, which undergoes two proton shifts to afford new zwitterionic intermediate **22**. Finally, intramolecular cyclization of intermediate **22** affords compound **23** by elimination of *N*-methyl imidazole, which is converted into **16** by elimination of hydrogen molecule (Scheme 4).

CONCLUSION

In summary, we report a reaction involving alkyl propiolates and primary amines in the presence of catalytic amount of *N*-methylimidazole at 50 °C in water which affords a new route to the synthesis of functionalized pyrroles. Also, these reactions were performed with alkyl propiolates and benzylamine in the presence of ammonium thiocyanate which afforded pyrrole derivatives. The present procedure has the advantage that not only is the reaction performed under neutral conditions, but the reactants can be mixed without any prior activation or modification.



Scheme 4. Proposed mechanism for the synthesis of compound 16.

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**N-МЕТИЛИМИДАЗОЛ ИЛИ АМОНИЕВ ТИОЦИАНАТ ПРОМОТИРАНА СИНТЕЗА НА
ЗАМЕСТЕНИ ПИРОЛИ: МНОГОКОМПОНЕНТНА РЕАКЦИЯ НА АЛКИЛ ПРОПИОЛАТИ
ВЪВ ВОДА**

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(Резюме)

Докладва се нов, удобен и ефикасен подход към синтеза на заместени пироли, на базата на трикомпонентна реакция. Реакцията на първични амини с електрон дефицитни ацетиленови съединения в присъствието на N-метилимидазол или амониев тиоцианат във вода води до образуването на пироли с добър добив.