

Reaction of *N*-Methylimidazole and Dimethyl Acetylenedicarboxylate in the Presence of *N*-Phenyl Carbamate under Solvent-free Conditions

Rahimeh Hajinasiri and Halimeh Khatoon Khajavi

Chemistry Department, Qaemshahr Branch, Islamic Azad University, Qaemshahr, I. R. Iran

Reprint requests to Dr. Rahimeh Hajinasiri. Fax: +981232240091. Tel: +981232145117.

E-mail: rhmhajinasiri@yahoo.com

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An efficient synthesis of 1,2,3-functionalized imidazoles and dimethyl-2-[(alkoxycarbonyl)anilino]-2-butenedioate derivatives *via* one-pot reactions between *N*-methylimidazole, dimethyl acetylenedicarboxylate and *N*-phenylcarbamates under solvent-free conditions is described. The mild reaction conditions and good yields exhibit the synthetic advantage of this method.

Key words: *N*-Methylimidazole, Solvent-free, *N*-Phenylcarbamate 1,2,3-Functionalized Imidazoles

Introduction

Functionalized imidazoles are an important class of heterocyclic compounds in organic chemistry because they are common structural units in a number of natural products and pharmaceuticals and useful building blocks for the construction of various biologically active molecules and functional materials [1–3].

Imidazole-based drugs such as Cimetidine, Etoposide and Ketoconazole are currently in clinical use [4]. Imidazole derivatives are used for the synthesis of imidazole-tailored ionic liquids and stable nucleophilic carbenes [5–9]. Because of the wide range of pharmacological and biological activities, the synthesis of functionalized imidazoles has become an important target in recent years.

Here we developed an efficient one-pot route for the synthesis of 1,2,3-functionalized imidazoles and dimethyl-2-[(alkoxycarbonyl)anilino]-2-butenedioate derivatives *via* the reaction between *N*-methylimidazole (**1**), dimethyl acetylenedicarboxylate (**2**) and *N*-phenylcarbamates (**3**) under solvent-free conditions at room temperature (Scheme 1).

Result and Discussion

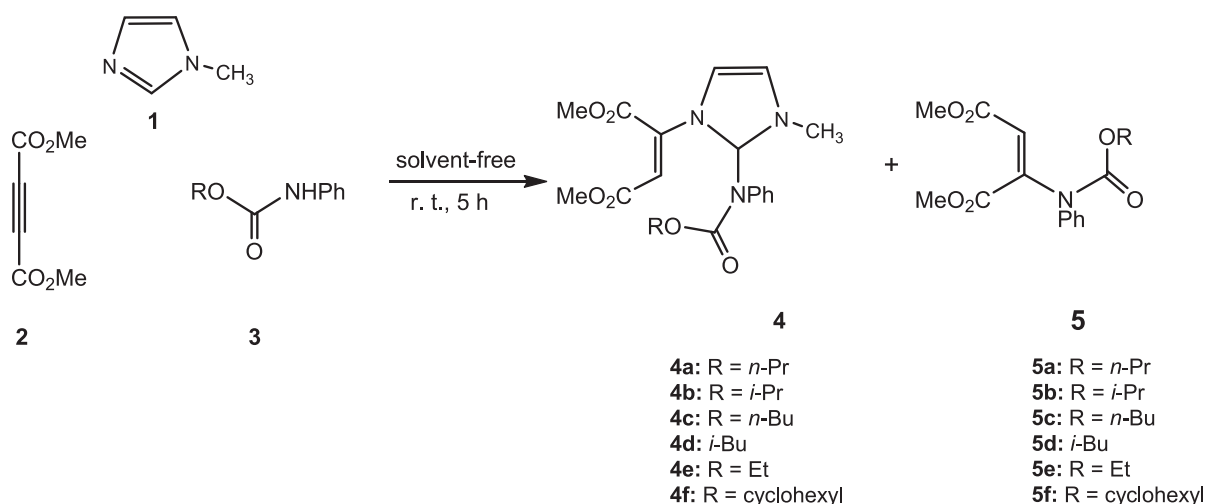
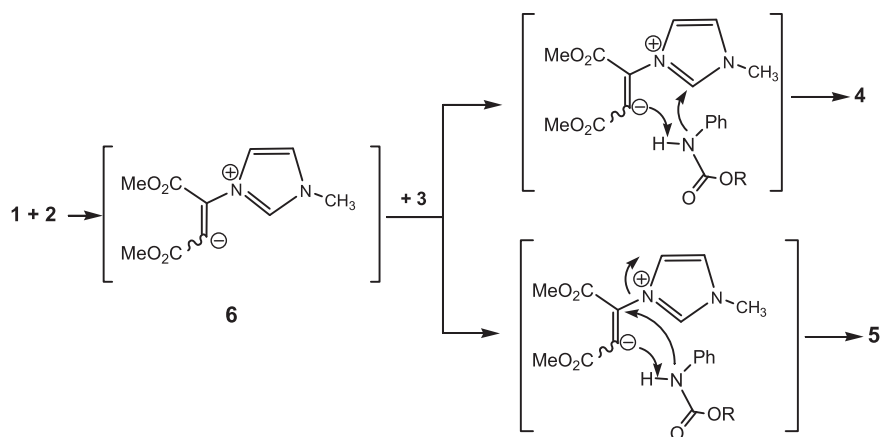
The reaction of *N*-methylimidazole (**1**), dialkyl acetylenedicarboxylates (**2**) and *N*-phenylcarbamates (**3**) proceeds smoothly under solvent-free conditions at

room temperature to produce dimethyl-2-[(3-methyl-2-[(alkoxycarbonyl)anilino]-2,3-dihydro-1*H*-imidazol-1-yl)]-2-butenedioates (**4**) and dimethyl-2-[(alkoxycarbonyl)anilino]-2-butenedioate derivatives (**5**) in 80–85% yield (Scheme 1).

The structures of the compounds were apparent from the ¹H NMR, ¹³C NMR and IR spectra. The ¹H NMR spectrum of **4a** exhibited all expected signals at $\delta = 1.04$, 1.68 and 4.10 ppm for the propyl moiety, three singlet peaks at $\delta = 3.68$, 3.77 and 3.87 ppm for NMe and two methoxy groups, a singlet at $\delta = 6.12$ ppm for an olefinic proton, and a singlet at 7.63 ppm for a CH that is bonded to three nitrogens, along with signals for the phenyl and imidazole units at 6.96–7.26 ppm. The proton-decoupled ¹³C NMR spectrum of **4a** showed 17 distinct resonances in agreement with the proposed structure.

The stereochemistry of compound **4e** has been confirmed by nuclear Overhauser effect (NOE) measurements. Thus, when the olefinic proton signal was irradiated, the signal of the imidazole H-2 was enhanced by about 21%, while both ester-OMe signals showed no significant enhancement. The stereochemistry thus deduced was also assumed for the other derivatives of **4**.

The ¹H NMR spectrum of **5a** displayed five peaks at $\delta = 1.00$, 1.63, 4.14, 3.68, 3.77, and 6.14 ppm along with characteristic multiplet signals for the phenyl moiety. The proton-decoupled ¹³C NMR spectrum of

Scheme 1. Synthesis of compounds **4a–f** and **5a–f**.Scheme 2. Possible mechanism for the formation of compounds **4** and **5**.

5a showed signals in agreement with the proposed structure.

The stereochemistry of compound **5e** is confirmed by nuclear Overhauser effect (NOE) measurements. Thus, when the OCH₂ protons signal was irradiated, the olefinic protons were enhanced by about 10%, while the OMe proton signal showed no significant enhancement. The stereochemistry thus deduced was also assumed for the other derivatives of **5**.

A possible mechanism for this reaction is proposed in Scheme 2. The zwitterionic intermediate **6** produced from the reaction of *N*-methylimidazole and dialkyl acetylenedicarboxylate is subsequently protonated by an *N*-phenylcarbamate **3** and then attacked by the conjugate base of the carbamate to produce **4** and **5**.

Conclusion

In conclusion we have reported a convenient one-pot route for the synthesis of 1,2,3-functionalized imidazoles and dimethyl-2-[(alkoxycarbonyl)anilino]-2-butenedioates by reaction of *N*-methylimidazole, dialkyl acetylenedicarboxylate and *N*-phenylcarbamates at solvent-free conditions and at room temperature.

Experimental

General information

Melting points were taken on a Kofler hot stage apparatus and are uncorrected. ¹H NMR and ¹³C NMR spectra were obtained with a Bruker FT-500 spectrometer in

CDCl₃, and tetramethylsilane (TMS) was used as an internal standard. Mass spectra were recorded with a Finnigan Mat TSQ-70 spectrometer. Infrared (IR) spectra were acquired on a Nicolet 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 $\pm 0.4\%$ of the calculated values. The acetylenic ester and *N*-methylimidazole were obtained from Fluka and were used without further purification.

General procedure for the preparation of compounds 4a–f and 5a–f

To a magnetically stirred mixture of an *N*-phenylcarbamate **3** (2 mmol) and dimethyl acetylenedicarboxylate (2, 2 mmol) was slowly added *N*-methylimidazole (**1**, 2 mmol), and the reaction mixture was stirred for 5 h at r.t. After completion of the reaction as indicated by TLC, the residue was purified by chromatography over silica gel (Merck 230–400 mesh) using an *n*-hexane-AcOEt mixture (6:1) as eluant to afford the pure compounds **4** and **5**.

Dimethyl {3-methyl-2-[(propoxycarbonyl)anilino]-2,3-dihydro-1H-imidazol-1-yl}-2-butenedioate (4a)

Yellow oil; yield: 0.40 g (50%). – IR (KBr): $\nu = 1721$ (C=O), 1718 (C=O), 1706 (C=O), 2989 (CH) cm⁻¹. – ¹H NMR: $\delta = 1.04$ (t, 3 H, ³J = 6.9 Hz, CH₃), 1.68 (sextet, 2 H, ³J = 6.9 Hz, CH₂), 3.68 (s, 3 H, NCH₃), 3.77 (s, 3 H, OCH₃), 3.87 (s, 3 H, OCH₃), 4.10 (t, 2 H, ³J = 7.0 Hz, CH₂), 6.12 (s, 1 H, CH), 6.96–7.26 (m, 7 H, 7 CH), 7.63 (s, 1 H, CH) ppm. – ¹³C NMR: $\delta = 10.3$ (Me), 23.7 (CH₂), 38.7 (NMe), 51.7 (OMe), 52.0 (OMe), 61.1 (OCH₂), 68.2 (CH), 107.7 (CH), 120.6 (CH), 123.7 (2 CH), 124.5 (CH), 128.5 (CH), 129.0 (2 CH), 137.2 (C), 147.0 (C), 154.8 (C=O), 163.3 (C=O), 164.8 (C=O) ppm. – EI-MS: $m/z(\%) = 403$ (2) [M]⁺, 226 (5), 178 (40), 144 (54), 120 (42), 43 (100). – Anal. for C₂₀H₂₅N₃O₆ (403.17): calcd. C 59.54, H 6.25, N 10.42; found C 59.55, H 6.23, N 10.40%.

Dimethyl 2-[(propoxycarbonyl)anilino]-2-butenedioate (5a)

Yellow oil; yield: 0.23 g (35%). – IR (KBr): $\nu = 1720$ (C=O), 1716 (C=O), 1696 (C=O), 2985 (CH) cm⁻¹. – ¹H NMR: $\delta = 1.00$ (t, 3 H, ³J = 7.1 Hz, CH₃), 1.63 (sextet, 2 H, ³J = 6.9 Hz, CH₂), 3.67 (s, 3 H, OCH₃), 3.76 (s, 3 H, OCH₃), 4.14 (t, 2 H, ³J = 7.1 Hz, CH₂), 6.14 (s, 1 H, CH), 7.28–7.61 (m, 5 H, 5 CH) ppm. – ¹³C NMR: $\delta = 10.4$ (Me), 22.9 (CH₂), 52.9 (OMe), 53.1 (OMe), 68.3 (OCH₂), 107.3 (CH), 123.3 (2 CH), 124.0 (CH), 128.8 (2 CH), 128.9 (2 CH), 140.7 (C), 147.6 (C), 154.0 (C=O), 163.2 (C=O), 164.5 (C=O) ppm. – EI-MS: $m/z(\%) = 321$ (3) [M]⁺, 178 (12), 144 (40), 43 (100). – Anal. for C₁₆H₁₉NO₆ (321.12): calcd. C 59.80, H 5.96, N 4.36; found C 59.83, H 5.97, N 4.35%.

Dimethyl {3-methyl-2-[(isopropoxycarbonyl)anilino]-2,3-dihydro-1H-imidazol-1-yl}-2-butenedioate (4b)

Yellow oil; yield: 0.42 g (52%). – IR (KBr): $\nu = 1725$ (C=O), 1705 (C=O), 2983 (CH) cm⁻¹. – ¹H NMR: $\delta = 1.23$ (d, 3 H, ³J = 6.8 Hz, CH₃), 1.30 (d, 3 H, ³J = 6.9 Hz, CH₃), 3.68 (s, 3 H, NCH₃), 3.78 (s, 3 H, OCH₃), 3.88 (s, 3 H, OCH₃), 4.13–4.15 (heptet, 1 H, ³J = 6.8 Hz, CH), 6.12 (s, 1 H, CH), 7.00–7.30 (m, 7 H, 7 CH), 7.67 (s, 1 H, CH) ppm. – ¹³C NMR: $\delta = 21.5$ (2 Me), 38.5 (NMe), 51.6 (OMe), 52.1 (OMe), 61.5 (OCH₂), 68.6 (CH), 106.9 (CH), 118.1 (CH), 123.0 (2 CH), 123.5 (CH), 127.3 (2 CH), 128.8 (2 CH), 138.9 (C), 146.5 (C), 153.7 (C=O), 163.2 (C=O), 164.5 (C=O) ppm. – EI-MS: $m/z(\%) = 403$ (3) [M]⁺, 226 (6), 178 (35), 144 (49), 120 (41), 43 (100). – Anal. for C₂₀H₂₅N₃O₆ (403.17): calcd. C 59.54, H 6.25, N 10.42; found C 59.55, H 6.24, N 10.44%.

Dimethyl 2-[(isopropoxycarbonyl)anilino]-2-butenedioate (5b)

Yellow oil; yield: 0.18 g (28%). – IR (KBr): $\nu = 1724$ (C=O), 1699 (C=O), 2987 (CH) cm⁻¹. – ¹H NMR: $\delta = 1.21$ (d, 3 H, ³J = 6.2 Hz, CH₃), 1.30 (d, 3 H, ³J = 6.3 Hz, CH₃), 3.67 (s, 3 H, OCH₃), 3.77 (s, 3 H, OCH₃), 4.14–4.17 (heptet, 1 H, ³J = 7.0 Hz, CH), 6.12 (s, 1 H, CH), 7.24–7.60 (m, 5 H, 5 CH) ppm. – ¹³C NMR: $\delta = 21.0$ (2 Me), 52.9 (OMe), 53.1 (OMe), 66.7 (OCH₂), 108.1 (CH), 123.1 (2 CH), 123.8 (CH), 128.9 (2 CH), 140.0 (C), 147.6 (C), 153.5 (C=O), 163.8 (C=O), 165.0 (C=O) ppm. – EI-MS: $m/z(\%) = 321$ (5) [M]⁺, 178 (15), 144 (54), 43 (100). – Anal. for C₁₆H₁₉NO₆ (321.12): calcd. C 59.80, H 5.96, N 4.36; found C 59.83, H 5.97, N 4.32%.

Dimethyl {3-methyl-2-[(butoxycarbonyl)anilino]-2,3-dihydro-1H-imidazol-1-yl}-2-butene-dioate (4c)

Yellow oil; yield: 0.45 g (56%). – IR (KBr): $\nu = 1722$ (C=O), 1717 (C=O), 1700 (C=O), 2982 (CH) cm⁻¹. – ¹H NMR: $\delta = 0.93$ (t, 3 H, ³J = 7.1 Hz, CH₃), 1.52–1.54 (m, 2 H, CH₂), 1.68–1.71 (m, 2 H, CH₂), 3.69 (s, 3 H, NCH₃), 3.78 (s, 3 H, OCH₃), 3.88 (s, 3 H, OCH₃), 4.15 (t, 2 H, ³J = 7.0 Hz, CH₂), 6.12 (s, 1 H, CH), 7.04–7.34 (m, 7 H, 7 CH), 7.61 (s, 1 H, CH), ppm. – ¹³C NMR: $\delta = 14.1$ (Me), 22.7 (CH₂), 31.5 (CH₂), 38.5 (NMe), 51.6 (OMe), 52.1 (OMe), 61.3 (OCH₂), 68.3 (CH), 106.7 (CH), 118.0 (CH), 123.0 (2 CH), 123.5 (CH), 127.1 (CH), 128.8 (2 CH), 139.1 (C), 146.0 (C), 153.6 (C=O), 162.8 (C=O), 164.5 (C=O) ppm. – EI-MS: $m/z(\%) = 417$ (2) [M]⁺, 226 (7), 192 (39), 144 (53), 120 (41), 57 (100). – Anal. for C₂₁H₂₇N₃O₆ (417.19): calcd. C 60.42, H 6.52, N 10.07; found C 60.40, H 6.54, N 10.05%.

Dimethyl 2-[(butoxycarbonyl)anilino]-2-butenedioate (5c)

Yellow oil; yield: 0.19 g (29%). – IR (KBr): $\nu = 1728$ (C=O), 2981 (CH) cm^{-1} . – ^1H NMR: $\delta = 0.83$ (t, 3 H, $^3J = 6.9$ Hz, CH_3), 1.48–1.51 (m, 2 H, CH_2), 1.58–1.65 (m, 2 H, CH_2), 3.68 (s, 3 H, OCH_3), 3.77 (s, 3 H, OCH_3), 4.16 (t, 2H, $^3J = 6.8$ Hz, CH_2), 6.14 (s, 1 H, CH), 7.38–7.59 (m, 5 H, 5 CH) ppm. – ^{13}C NMR: $\delta = 14.0$ (Me), 23.0 (CH_2), 31.4 (CH_2), 53.0 (OMe), 53.1 (OMe), 66.7 (OCH_2), 108.0 (CH), 123.0 (2 CH), 123.4 (CH), 128.7 (2 CH), 128.9 (2 CH), 138.8 (C), 147.9 (C), 153.8 (C=O), 163.5 (C=O), 165.0 (C=O) ppm. – EI-MS: $m/z(\%) = 307$ (5) $[\text{M}]^+$, 192 (15), 144 (45), 57 (100). – Anal. for $\text{C}_{17}\text{H}_{21}\text{NO}_6$ (335.13): calcd. C 60.89, H 6.31, N 4.18; found C 60.87, H 6.34, N 4.16%.

Dimethyl {3-methyl-2-[(isobutoxycarbonyl)anilino]-2,3-dihydro-1H-imidazol-1-yl}-2-butene-dioate (4d)

Yellow oil; yield: 0.48 g (58%). – IR (KBr): $\nu = 1726$ (C=O), 1718 (C=O), 1706 (C=O), 2984 (CH), cm^{-1} . – ^1H NMR: $\delta = 1.01$ (d, 6 H, $^3J = 6.8$ Hz, 2 CH_3), 2.35–2.39 (m, 1 H, CH), 3.69 (s, 3 H, NCH_3), 3.78 (s, 3 H, OCH_3), 3.88 (s, 3 H, OCH_3), 4.11 (d, 1 H, $^3J = 6.9$ Hz, CH), 6.12 (s, 1 H, CH), 7.04–7.34 (m, 7 H, 7 CH), 7.65 (s, 1 H, CH) ppm. – ^{13}C NMR: $\delta = 17.7$ (Me), 17.9 (Me), 21.7 (CH), 37.1 (NMe), 51.9 (OMe), 52.0 (OMe), 58.5 (OCH_2), 67.1 (CH), 106.7 (CH), 118.0 (CH), 123.0 (2 CH), 123.5 (CH), 127.1 (CH), 128.8 (2 CH), 139.1 (C), 146.0 (C), 154.5 (C=O), 162.5 (C=O), 163.2 (C=O) ppm. – EI-MS: $m/z(\%) = 417$ (5) $[\text{M}]^+$, 226 (9), 192 (36), 144 (48), 120 (42), 57 (100). – Anal. for $\text{C}_{21}\text{H}_{27}\text{N}_3\text{O}_6$ (417.19): calcd. C 60.42, H 6.52, N 10.07; found C 60.41, H 6.53, N 10.09%.

Dimethyl 2-[(isobutoxycarbonyl)anilino]-2-butenedioate (5d)

Yellow oil; yield: 0.18 g (27%). – IR (KBr): $\nu = 1730$ (C=O), 2985 (CH) cm^{-1} . – ^1H NMR: $\delta = 0.99$ (d, 6 H, $^3J = 7.2$ Hz, 2 CH_3), 2.31–1.34 (m, 1 H, CH), 3.68 (s, 3 H, OCH_3), 3.77 (s, 3 H, OCH_3), 4.14 (d, 1 H, $^3J = 6.8$ Hz, CH), 6.14 (s, 1 H, CH), 7.36–7.64 (m, 5 H, 5 CH) ppm. – ^{13}C NMR: $\delta = 18.0$ (2 Me), 21.7 (CH), 50.7 (OMe), 51.8 (OMe), 58.6 (OCH_2), 68.80 (CH), 108.0 (CH), 123.0 (2 CH), 123.4 (CH), 128.0 (2CH), 138.8 (C), 147.9 (C), 156.5 (C=O), 163.2 (C=O), 165.0 (C=O) ppm. – EI-MS: $m/z(\%) = 335$ (2) $[\text{M}]^+$, 192 (157), 144 (47), 57 (100). – Anal. for $\text{C}_{17}\text{H}_{21}\text{NO}_6$ (335.13): calcd. C 60.89, H 6.31, N 4.18; found C 60.91, H 6.30, N 4.20%.

Dimethyl {3-methyl-2-[(ethoxycarbonyl)anilino]-2,3-dihydro-1H-imidazol-1-yl}-2-butene-dioate (4e)

Yellow oil; yield: 0.44 g (57%). – IR (KBr): $\nu = 1721$ (C=O), 1716 (C=O), 1696 (C=O), 2986 (CH) cm^{-1} . – ^1H

NMR: $\delta = 1.25$ (t, 3 H, $^3J = 6.8$ Hz, CH_3), 3.68 (s, 3 H, NCH_3), 3.77 (s, 3 H, OCH_3), 3.87 (s, 3 H, OCH_3), 4.13 (q, 2 H, $^3J = 7.0$ Hz, CH_2), 6.12 (s, 1 H, CH), 6.98–7.24 (m, 3 H, 7 CH), 7.63 (s, 1 H, CH) ppm. – ^{13}C NMR: $\delta = 13.5$ (Me), 37.7 (NMe), 50.6 (OMe), 50.7 (OMe), 60.1 (OCH_2), 67.1 (CH), 106.7 (CH), 118.0 (CH), 122.3 (2 CH), 123.5 (CH), 127.2 (CH), 128.0 (2 CH), 137.5 (C), 146.0 (C), 153.8 (C=O), 162.2 (C=O), 163.7 (C=O) ppm. – EI-MS: $m/z(\%) = 389$ (2) $[\text{M}]^+$, 226 (8), 164 (41), 144 (52), 120 (40), 29 (100). – Anal. for $\text{C}_{19}\text{H}_{23}\text{N}_3\text{O}_6$ (389.16): calcd. C 58.60, H 5.95, N 10.79; found C 58.61, H 5.94, N 10.81%.

Dimethyl 2-[(ethoxycarbonyl)anilino]-2-butenedioate (5e)

Yellow oil; yield: 0.17 g (28%). – IR (KBr): $\nu = 1732$ (C=O), 1710 (C=O), 2990 (CH) cm^{-1} . – ^1H NMR: $\delta = 1.22$ (t, 3 H, $^3J = 6.8$ Hz, CH_3), 3.67 (s, 3 H, OCH_3), 3.76 (s, 3 H, OCH_3), 4.22 (q, 2 H, $^3J = 6.9$ Hz, CH_2), 6.14 (s, 1 H, CH), 7.27–7.61 (m, 5 H, 5 CH) ppm. – ^{13}C NMR: $\delta = 13.1$ (Me), 51.8 (OMe), 51.9 (OMe), 60.0 (OCH_2), 108.0 (CH), 123.0 (2 CH), 127.8 (CH), 127.9 (2 CH), 137.4 (C), 146.6 (C), 153.1 (C=O), 162.5 (C=O), 164.0 (C=O) ppm. – EI-MS: $m/z(\%) = 307$ (2) $[\text{M}]^+$, 164 (17), 144 (43), 29 (100). – Anal. for $\text{C}_{15}\text{H}_{17}\text{NO}_6$ (307.16): calcd. C 58.63, H 5.58, N 4.56; found C 58.62, H 5.59, N 4.54%.

Dimethyl {3-methyl-2-[(cyclohexyloxycarbonyl)anilino]-2,3-dihydro-1H-imidazol-1-yl}-2-butenedioate (4f)

Yellow oil; yield: 0.49 g (55%). – IR (KBr): $\nu = 1722$ (C=O), 1715 (C=O), 1696 (C=O), 2983 (CH) cm^{-1} . – ^1H NMR: $\delta = 1.58$ –1.63 (m, 2 H, CH_2), 1.72–1.76 (m, 4 H, 2 CH_2), 1.92–1.96 (m, 4 H, 2 CH_2), 3.69 (s, 3 H, NCH_3), 3.78 (s, 3 H, OCH_3), 3.88 (s, 3 H, OCH_3), 4.12–4.16 (m, 1 H, CH), 6.11 (s, 1 H, CH), 7.00–7.26 (m, 7 H, 7 CH), 7.64 (s, 1 H, CH) ppm. – ^{13}C NMR: $\delta = 23.4$ (2 CH_2), 25.3 (CH_2), 31.2 (2 CH_2), 37.3 (NMe), 50.8 (OMe), 51.9 (OMe), 60.1 (CH), 67.5 (CH), 107.0 (CH), 118.3 (CH), 122.5 (2 CH), 123.7 (CH), 128.7 (2 CH), 137.8 (C), 146.8 (C), 154.2 (C=O), 162.5 (C=O), 163.7 (C=O) ppm. – EI-MS: $m/z(\%) = 443$ (3) $[\text{M}]^+$, 226 (7), 164 (45), 218 (48), 120 (36), 83 (100). – Anal. for $\text{C}_{23}\text{H}_{29}\text{N}_3\text{O}_6$ (443.21): calcd. C 62.29, H 6.59, N 9.47; found C 62.31, H 6.56, N 9.45%.

Dimethyl 2-[(cyclohexyloxycarbonyl)anilino]-2-butenedioate (5f)

Yellow oil; yield: 0.20 g (28%). – IR (KBr): $\nu = 1726$ (C=O), 1712 (C=O), 2984 (CH) cm^{-1} . – ^1H NMR: $\delta = 1.52$ –1.56 (m, 2 H, CH_2), 1.65–1.70 (m, 4 H, 2 CH_2), 1.86–1.91 (m, 4 H, 2 CH_2), 3.68 (s, 3 H, OCH_3), 3.77 (s, 3 H, OCH_3), 4.11–4.14 (m, 1 H, CH), 6.14 (s, 1 H, CH), 7.33–7.61 (m, 5 H, 5 CH) ppm. – ^{13}C NMR: $\delta = 23.2$ (2 CH_2), 25.1 (CH_2), 31.0 (2 CH_2), 50.7 (OMe), 51.8 (OMe),

59.6 (CH), 68.7 (CH), 108.0 (CH), 123.3 (2 CH), 127.7 (CH), 128.2 (2 CH), 140.0 (C), 147.2 (C), 154.5 (C=O), 163.0 (C=O), 164.7 (C=O) ppm. – EI-MS: $m/z(\%) = 361 (3)$ [M]⁺, 218(19), 144 (36), 83 (100). – Anal. for C₁₉H₂₃NO₆ (361.15): calcd. C 63.15, H 6.41, N 3.88; found C 63.13, H 6.43, N 3.86%.

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