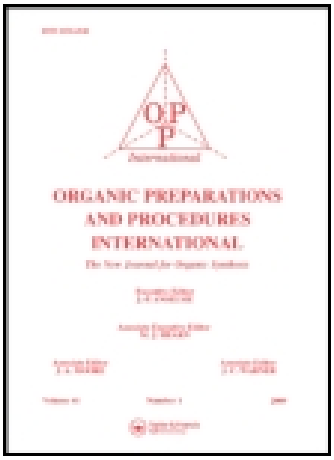

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One-pot Synthesis of Some New Indene Derivatives by Thermal Isomerization of Iminolactones

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The indene moiety found in many natural products,[1] has attracted interest due to its possible practical applications.[2,3] For example, some substituted indene derivatives exhibit biological activities such as fungicidal,[4] selective estrogen receptor modulation,[5,6] anti-proliferative[7] and D_2-like dopamine receptor agonists.[8] Various methods for the synthesis of indene derivatives have been reported.[9–14] We now describe a one-pot reaction of acetylenic esters and cyclohexyl isocyanide with aryl ketones to produce highly functionalized indenes **2a-f** in high yields (83–88%).

It had been previously established that one-pot and multi-component reactions of alkyl isocyanides, carbonyl compounds and acetylenic esters produce heterocyclic compounds.[15–22] However, in the present work the indene derivatives **2** are produced and it is postulated that the heterocyclic intermediates **1** that were formed initially in the reaction are converted to indenes **2**. Accordingly, to assess the validity of this postulate we decided to isolate the intermediates **1**. Thus, after cooling the reaction mixtures, the iminolactones **1** (the synthesis of **1a** had been reported previously[22]) were purified and their structure deduced by spectroscopic data. When these compounds **1** were refluxed in *p*-xylene for 35–48 h, indenes **2** were produced nearly quantitatively. The intramolecular N—H---O hydrogen bonding stabilized the products.[23] (*Scheme 1*)

Solvents have been shown to have an important effect on this reaction. Thus, in refluxing benzene (bp 80°C) only iminolactones **1** were obtained in less than 30% yields and no indenes **2** were obtained, Nair and coworkers had already reported the exclusive formation of iminolactone **1a** using benzene at 80°C in 41% yield.[21,22] In this study, we have found that in refluxing toluene (bp 110°C), iminolactones **1** were obtained in less than 50% yield and indenes **2** in less than 10% yield. In refluxing *p*-xylene (bp 138°C), only indenes **2** were obtained in 83–88% yield without any iminolactones **1.** This reaction did

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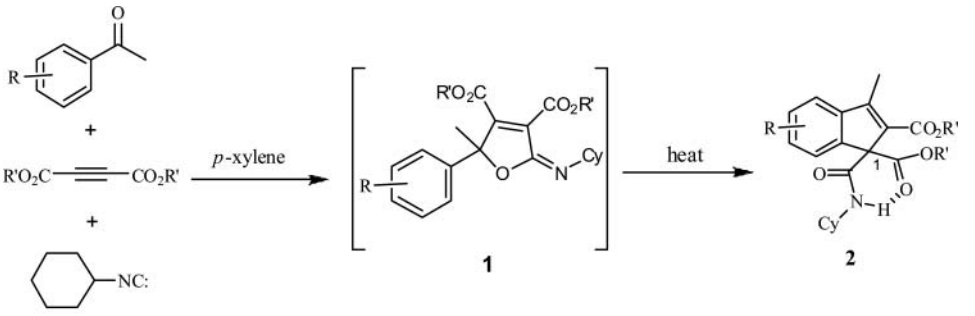


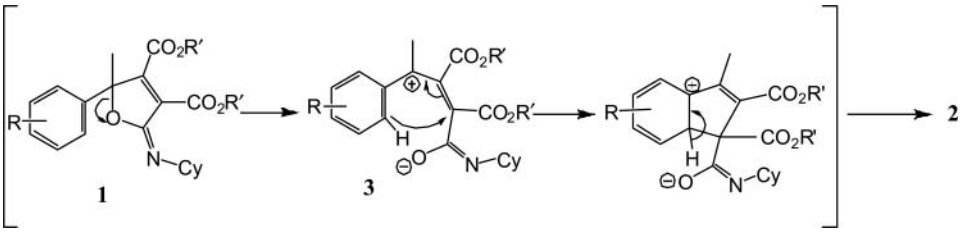
a) R = 4-NO_2, R' = Me; b) R = 4-NO_2, R' = Et; c) R = 3-NO_2 , R' = Me; d) R = 3-NO_2 , R' = Et;

e) R = 4-Cl, R' = Me ; f) R = 4-Cl , R' = Et

Scheme 1

not proceed in acidic solvents and indeed Schmiar and coworkers had previously reported that iminolactones are hydrolyzed to other products in acidic solvents.[24]

We suggest the following mechanism for the overall tranformation involving the conversion of intermediates **1** to indenes **2** *via* a ring opening-ring closure mechanism. Such a ring opening-ring closure mechanism has been reported previously for other heterocyclic frameworks[25–28] including a furan to indole process.[25] Several attempts to intercept the putative zwitterions **3** as carbocations (*Scheme 2*) with amines, thiols and phenols and the anion of acetylacetone, were unsuccessful; thus, if this mechanism is operative, the conversion of **1** to **2** must be rapid and quantitative.



Scheme 2

Structures **1** and **2** were assigned on the basis of their elemental analysis, IR, ^{1}H and ^{13}C NMR and mass spectral data. The mass spectra of these compounds displayed molecular ion peaks at appropriate m/z values. For iminolactones **1**, initial fragmentation involving the loss of $C_6H_{11}N$, CH_3OH for **1c**, **1e** and $C_6H_{11}N$, C_2H_5OH for **1b, 1d** and **1f**. For indenes **2**, fragments involves M^+- $C_6H_{11}NCO$ and M^+- $C_6H_{11}NCO$, R′OH were the most intense signals. The ^{1}H NMR spectra of the products **2b**, **2d** and **2f** clearly indicated the diasterotopic CH_2 protons of the ester groups on the C_1 of indenes (Table 3). The IR spectra of compounds **2a-f** showed N—H absorptions 3327–3353 cm^{-1}, which did not change upon dilution with CCl_4, evidence of intramolecular hydrogen bonding. The intramolecular N—H···O hydrogen bond for indene **2b** was detected and its details have been published.[23]

All compounds showed C=O absorptions (1715–1732, 1670–1679 cm^{-1}) and there were also two absorptions for the NO_2 groups in compounds **2a-d** (1339–1344, 1449–1525 cm^{-1}). The IR spectra of compounds **1b-f** showed C=O absorptions (1740–1755 cm^{-1}, 1719–1731 cm^{-1}), C=N absorption (1674–1682 cm^{-1}) and C—O absorptions (1253–1284, 1087–1129, 1020–1028 cm^{-1}). Additionally there were two absorptions for the NO_2 groups in compounds **1b-d** (1524–1533, 1339–1353 cm^{-1}).

Overall we have developed a one-pot synthesis of some new indene derivatives *via* an interesting isomerization reaction. The reaction described here represents a simple and efficient entry into the synthesis of highly functionalized indenes. High yields and simple conditions make it a useful addition to the modern synthetic methodologies. In this procedure, intermediates **1** can be isolated in high yields through modifications in the reaction conditions. These compounds **1** belong to the iminolactone family and some of their derivatives act as antibacterial agents, aldosterone inhibitors and as precursors for the preparation of a wide spectrum of natural compounds.[29,30] Further investigations of this method are currently in progress to establish its scope and utility.

Experimental Section

Chemicals and solvents were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. Column chromatography was performed on silica gel (0.015–0.04 mm, mesh-size) and TLC on precoated plastic sheets (25 DC_{UV-254}). Melting points were measured on a Barnstead Electrothermal melting point apparatus and are not corrected. Elemental analyses for C, H and N were performed using a Thermo Finnigan Flash EA1112 instrument. IR spectra were measured on a Shimadzu FT-IR-4300 spectrophotometer as KBr discs. ^1H NMR and ^{13}C NMR spectra were determined in $CDCl_3$ on a Brucker 500 spectrophotometer and chemical shifts were expressed in ppm downfield from tetramethylsilane. Mass spectra were recorded on a Finnigan-MAT 8430 spectrometer at an ionization potential of 70 ev.

Dimethyl 1-(Cyclohexylcarbamoyl)-3-methyl-6-nitro-1H-indene-1,2-dicarboxylate (2a). General Procedure

To a stirred solution of 4-nitroacetophenone (0.33 g., 2 mmol) and dimethyl acetylenedicarboxylate (0.28 g., 2 mmol) in *p*-xylene (15 mL) was added a mixture of cyclohexyl isocyanide (0.22 g., 2 mmol) in *p*-xylene (10 mL) dropwise at -10°C over 2 h. The reaction mixture was stirred for 2 h at room temperature and then refluxed for 36 h. The reaction was monitored by TLC, when the intermediate spots ($R_f \approx 0.2$ on silica gel, *n*-hexane:ethyl acetate 4:1) disappeared, the reaction was complete (indene spot: $R_f = 0.6$ on silica gel, *n*-hexane: ethyl acetate 4:1). The solvent was removed under reduced pressure and the residue was washed with *n*-hexane and recrystallized from acetone-ethyl acetate. The product (0.724 g., 87%) was obtained as colorless crystals, mp. 134–135°C. The same procedure was used to prepare **2b-f** as colorless crystals in of 83–88% yields.

Dimethyl 1-(Cyclohexylcarbamoyl)-3-methyl-6-nitro-1H-indene-1, 2-dicarboxylate (2a) from 1. General Procedure

A magnetically stirred solution of the imino-lactone **1** (0.83 g., 2 mmol) in *p*-xylene (50 mL) was refluxed for 32–45 h. The reaction was monitored by TLC. When the

Table 1
Spectroscopic Data of **1b-f**

Cmpd	^{1}H NMR (δ:ppm)	^{13}C NMR (δ:ppm)	MS (m/z)
1b	1.24–2.12 (10H, 3m, CH_2 of cyclohexyl); 1.27, 1.41 (6H, 2t, $2CH_3$ of CO_2Et); 2.12 (3H, s, CH_3); 3.68–3.69 (1H, m, CH of cyclohexyl); 4.18-4.21, 4.40–4.45 (4H, 2q, J = 13.81, 6.91 Hz, 2 CH_2 of CO_2Et); 7.63- 7.66, 8.25–8.27 (4H, 2m, Ar protons)	14.20, 14.45 (2 CH_3 of CO_2Et); 24.17, 25.14, 26.14 (CH_2 of cyclohexyl); 33.79 (CH_3); 57.01 (CH of cyclohexyl); 62.44, 62.73 (CH_2 of CO_2Et); 89.32 (C−CH_3); 124.06, 127.48 (CH of C_6H_4); 136.38, 148.23 ($C_{ipso\ (C=C)}$ of C_6H_4); 145.74, 147.13 (2 C=CO_2Et); 154.70 (C=N-cyclohexyl); 160.85, 162.29 (2 C=O of CO_2Et)	444 (M^+, 13) 415 (M^+-C_2H_5, 30) 398 (m^+-C_2H_5OH, 75) 315 (M^+- $C_6H_{11}N$,100)
1c	1.24–1.82 (10H, 4m, CH_2 of cyclohexyl); 2.13 (3H, s, CH_3); 3.67–3.70 (1H, m, CH of cyclohexyl); 3.77, 3.96 (6H, 2s, CH_3 of CO_2Me); 7.58–7.61 (1H, t, Ar proton); 7.76–7.78 (1H, d, J = 8.01Hz, Ar proton); 8.23–8.24 (1H, d, J = 8.01 Hz, Ar proton); 8.36 (1H, s, Ar proton)	24.77, 25.20, 26.11 (CH_2 of cyclohexyl); 33.73 (CH_3); 53.27, 53.54 ($2CH_3$ of CO_2Me); 57.19 (CH of cyclohexyl); 89.24 (C−CH_3); 121.56, 123.93, 130.03, 132.38 (CH of C_6H_4); 127.85, 134.19 ($C_{ipso(c=c)}$ of C_6H_4); 146.00, 148.74 (2C=CO_2Me); 154.55(C=N-cyclohexyl); 161.23, 162.70 (2 C=O of CO_2Me)	416 (M^+, 7) 304 (M^+-CH_3, 15) 384 (M^+-CH_3OH, 73) 287 (M^+-$C_6H_{11}N$, 100)
1d	1.23–1.81(10H, m, CH_2 of cyclohexyl); 1.26, 1.38 (6H, 2t, 2 CH_3 of CO_2Et); 2.12 (3H, s, CH_3); 3.68 (1H, m, CH of cyclohexyl); 4.16-4.21,4.40–4.44 (4H,2q, J = 14.02, 6.99 Hz, $2CH_2$ of CO_2Et); 7.58- 7.61, 7.61–7.73, 7.73–7.78, 8.23–8.36 (4H, 4m, Ar protons)	14.23, 14.71 (2 CH_3 of CO_2Et);24.66,25.01,26.11 (CH_2 of cyclohexyl); 33.79 (CH_3); 56.98 (CH of cyclohexyl); 62.30, 62.79 (CH_2 of CO_2Et); 89.24 (C−CH_3);121.49, 123.91, 127.85, 130.03, 132.38, 134.19(Ar carbons); 146.85, 148.56 (2 C=CO_2Et); 154.59 (C=N-cyclohexyl); 161.24, 162.78 (2 C=O of CO_2Et)	444 ($M^{+\cdot}$ 11) 415 (M^+-C_2H_5, 28) 398 (M^+-C_2H_5OH, 74) 315 (M^+-$C_6H_{11}N$, 100)
1e	1.22–1.36, 1.42–1.46, 1.64–1.81 (10H, 3m, CH_2 of cyclohexyl); 2.11 (3H, s, CH_3); 3.64–3.68 (1H, m, CH of cyclohexyl); 3.75, 3.94 (6H, 2s, CH_3 of CO_2Me); 7.06–7.07, 7.15–7.16 (4H, 2m, Ar protons)	24.63, 25.21, 26.10 (cyclohexyl carbons); 33.76 (CH_3); 53.35, 53.61 (2 CH_3 of CO_2Me); 57.21 (CH of cyclohexyl); 89.47 (C−CH_3); 124.67, 127.45 (CH of C_6H_4); 130.37, 138.25 ($C_{ipso\ (C=C)}$ of C_6H_4); 136.112, 136.85 (2 C=CO_2Me); 154.63 (C=N-cyclohexyl);161.52, 162.73 (2 C=O of CO_2Me)	405, 407 (M^+, M+2, 15, 5) 390, 392 (M^+, M^++2-CH_3, 28, 9) 373, 375 (M^+, M^++ 2-CH_3OH, 78, 28) 276,278 (M^+, M^++ 2-$C_6H_{11}N$, 100, 37)

(Continued on next page)

Table 1
Spectroscopic Data of **1b-f** *(Continued)*

Cmpd	^{1}H NMR (δ:ppm)	^{13}C NMR (δ:ppm)	MS (m/z)
1f	1.23–1.81 (10 H, 4m, CH_2 of cyclohexyl); 1.27, 1.36 (6H, 2t, 2 CH_3 of CO_2Et); 2.136 (3H, s, CH_3); 3.68 (1H, m, CH of cyclohexyl); 4.186–4.23, 4.40–4.45 (4H, 2q, $J = 14.36$, 7.11 Hz, 2 CH_2 of CO_2Et); 7.05 – 7.07, 7.13–7.15 (4H, 2m, Ar protons)	14.20, 14.67 (2 CH_3 of CO_2Et); 24.73, 25.40, 26.28 (CH_2 of cyclohexyl); 32.69 (CH_3); 57.35 (CH of cyclohexyl); 62.64, 63.30 (CH_2 of CO_2Et); 88.98 (C−CH_3); 124.84, 127.25 (CH of C_6H_4); 130.47, 135.69 ($C_{ipso(C=C)}$ of C_6H_4); 136.14, 138.65 (2 C=CO_2Et); 154.63 (C=N-cyclohexyl); 161.26, 162.62 (2 C=O of CO_2Et)	433, 435 (M^+, M^++2, 16, 6) 404, 402 (M^+, M^++2-C_2H_5, 29, 9) 387, 389 (M^+, M^++2 -C_2H_5OH, 8, 30) 304, 306 (M^+, M^++2 -$C_6H_{11}N$, 100, 38)

iminolactone spot disappeared, the thermal interconversion of **1** into **2** was complete. At this stage, the solvent was removed under reduced pressure. The same procedure for the purification of **2a-f** was performed as for the one-pot procedure. Although the purified **2a-f** were obtained in 96–99% yields, the yields of indenes **2** reported in *Table 4* are those obtained in the one-pot reaction which is simpler and more convenient.

Compounds **1b-f** which were not previously described in *references* 21 and 22, can be obtained in 85–90% yields by the following procedure.

Synthesis of Compounds 1b-f. General Procedure

To a stirred solution of the acetophenone derivative (2 mmol) and dialkyl acetylenedicarboxylate (2 mmol) in *p*-xylene (15 mL) was added a mixture of alkyl isocyanide (2 mmol) in *p*-xylene (10 mL) dropwise at -10°C over 2 h. The reaction mixture was warmed to 75°C and stirred at 75°C for 10 h then allowed standing in a refrigerator for 24 h. The solvent was removed under reduced pressure and the residue was purified by column chromatography (silica gel, *n*-hexane: ethyl acetate 3:1) and recrystallized in acetone to afford the pure products **1** as colorless crystals. The results are shown in *Tables 1*, *2*.

Table 2
Yields, mps and Elemental Analysis of **1b-f**

Cmpd	Yields (%)	mp (°C)	Elemental Analysis (Found) C	H	N	Formula (M.W.)
1b	86	125	62.15(62.12)	6.35(6.33)	6.30(6.32)	$C_{23}H_{28}N_2O_7$ (444.48)
1c	90	108	60.57(60.56)	5.81(5.82)	6.73(6.73)	$C_{21}H_{24}N_2O_7$ (416.42)
1d	87	112	62.15(62.13)	6.35(6.33)	6.30(6.31)	$C_{23}H_{28}N_2O_7$ (444.48)
1e	87	97	62.14(62.11)	5.96(5.93)	3.45(3.49)	$C_{21}H_{24}ClNO_5$ (405.87)
1f	85	105	63.66(63.63)	6.50(6.47)	3.23(3.25)	$C_{23}H_{28}ClNO_5$ (433.93)

Table 3
Spectroscopic Data of **2a-f**

Cmpd	^{1}H NMR (δ:ppm)	^{13}C NMR (δ:ppm)	MS (m/z)
2a	1.33–1.94 (10H, 4m, CH_2 of cyclohexyl); 2.67 (3H, s, CH_3); 3.68, 3.88 (6H, 2s, CH_3 of CO_2Me); 3.78–3.80 (1H, m, CH of cyclohexyl); 7.61–7.62, 8.32–8.37 (1H, d, J = 8.35 Hz; 2H, m, Ar protons); 8.48–8.50 (1H, d, J = 0.65 Hz, NH).	23.04, 23.72, 24.60, CH_2 of cyclohexyl); 32.94 (CH_3); 49.17 (C_{ipso} of cyclopentene); 61.56, 63.08 (2 CH_3 of CO_2Me); 68.92(C of cyclohexyl); 117.33, 123.00, 125.77, 137.22, 144.15, 148.41, 151.23, 152.49 (Alkene and Ar carbons); 163.71, 164.09 (C=O of CO_2Me); 171.03(C=O of amide).	416 (M^+, 15) 384 (M^+-MeOH,16) 291 (M^+-$C_6H_{11}NCO$, 95) 252 (M^+-$C_6H_{11}NCO$, CH_3OH, 100)
2b	1.15–1.18 (3H, t, CH_3 of, CO_2Et); 1.34–1.44, 1.75–1.93(13 H, 4m, CH_3 of CO_2Et, CH_2 of cyclohexyl); 2.66 (3H, s, CH_3); 3.7–3.78 (1H,m,CH of cyclohexyl); 4.00–4.06, 4.26, 4.28(2H, 2m, CH_2 of CO_2Et); 4.28–4.34 (2H, q,CH_2 of CO_2Et); 7.30–7.61, 8.32–8.36 (3H, 2m, Ar protons); 8.56–8.57 (1H, d, J = 7.6 Hz, NH)	13.04, 14.15 (CH_3 of CO_2Et); 24.94, 25.97, 26.60 (CH_2 of cyclohexyl); 33.02 (CH_3); 49.17 (C_{ipso} of cyclohexyl); 61.28, 63.08 (CH_2 of CO_2Et); 68.92 (CH of cyclohexyl); 118.06, 122.63, 125.32, 137.43, 143.74, 148.41, 151.50, 152.49 (Alkene and Ar carbons); 163.73, 164.09 (2 C=O of CO_2Et); 170.75 (C=O of amide)	444 (M^+, 12) 398 (M^+-C_2H_5OH, 25) 319 (M^+-$C_6H_{11}NCO$, 83) 273(M^+-$C_6H_{11}NCO$, C_2H_5OH, 100)
2c	1.23–1.82 (10H, 4m, CH_2 of cyclohexyl); 2.70 (3H, s, CH_3); 3.40–3.96 (1H, m, CH of cyclohexyl); 4.04–4.16 (6H, 2s, CH_3 of CO_2Me); 7.68–7.71, 8.33–8.58 (3H, 2m, Ar protons); 8.60 (1H, d, J = 7.1 Hz, NH)	23.23, 24.15, 24.71 (CH2 of cyclohexyl); 32.39 (CH_3); 50.56 (C_{ipso} of cyclopentene ring); 62.04, 63.15 (2 CH_3 of CO_2Me); 69.12 (CH ofcyclohexyl); 120.56,123.93, 125.85, 135.19, 141.45, 146.30, 149.00, 149.74 (Alkene and Ar cabons); 161.23, 162.70 (2 C=O of CO_2Me); 171.03 (C=O of amide)	416 (M^+, 13) 384 (M^+-CH_3OH, 23) 291 (M^+-$C_6H_{11}NCO$, 80) 259 (M^+-$C_6H_{11}NCO$, CH_3OH, 100)

(Continued on next page)

Table 3
Spectroscopic Data of **2a-f** *(Continued)*

Cmpd	^{1}H NMR (δ:ppm)	^{13}C NMR (δ:ppm)	MS (m/z)
2d	1.15–1.78 (3H, t, CH_3 of CO_2Et); 1.34–1.42, 1.75–1.93 (13H, 4m, CH_3 of CO_2Et, CH_2 of cyclohexyl); 2.68(3H, s, CH_3); 3.75 (1H, m, CH of cyclohexyl); 4.26–4.35 (4H, 2q, CH_2 of CO_2Et); 7.30–7.38, 7.60- 7.62, 8.33–8.38 (3H, 3m, Ar protons); 8.57–8.58 (1H, d, J = 7.55Hz, NH)	14.94, 15.98 (2 CH_3 of CO_2Et); 23.04, 24.15, 24.60 (CH_2 of cyclohexyl); 33.69 (CH_3); 49.12 (C_{ipso} of cyclopentene); 61.43, 63.63 (CH2 of CO_2Et); 68.32 (CH of cyclohexyl);117.28, 123.08, 125.92, 141.06, 142.02, 149.41,152.13, 153.60 (Alkene and Ar carbons); 163.23, 164.05 (2 C=O of CO_2Et); 171.08 (C=O of amide)	444 (M^+, 11) 398 (M^+-C_2H_5OH, 30) 319 (M^+- $C_6H_{11}NCO$, 78) 273 (M^+-$C_6H_{11}NCO$, C_2H_5OH, 100)
2e	1.25–1.82 (10H, 4m, CH_2 of cyclohexyl); 2.63 (3H, s, CH_3); 3.96–3.99 (1 H, m, CH of cyclohexyl); 4.04, 4.15 (6H, 2s, CH_3 of CO_2Me); 7.06–7.08, 7.13–7.17 (3H, 2m, Ar protons); 8.56–8.57(1H, d, J = 4.41Hz, NH)	23.84, 24.49, 24.92 (CH_2 of cyclohexyl); 33.24 (CH_3); 50.06 (C_{ipso} of cyclopentene ring); 62.13–63.49 (2 CH_3 of CO_2Me); 69.08 (^{13}CH of cyclohexyl); 123.45, 126.02, 127.44, 128.44, 129.17, 132.43, 146.24, 148.12 (Alkene and Ar carbons); 163.12, 164.39 (2 C=O of CO_2Me); 170.43 (C=O of amide)	405, 407 (M^+, M^++2, 40, 12) 373, 375 (M^+, M^++2-CH_3OH, 25, 6), 280, 282 (M^+, M^++2-$C_6H_{11}NCO$, 85, 35) 248, 250 (M^+, M^++2-$C_6H_{11}NCO$, CH_3OH, 100, 40)
2f	1.15–1.18 (3H, t, CH_3 of CO_2Et); 1.34–1.42, 1.75–1.76, 1.77–1.93 (13H, 4m, CH_3 of CO_2Et, CH_2 of cyclohexyl); 2.66 (3H, s, CH_3); 4.03 (1H, m, CH of cyclohexyl); 4.24–4.26, 4.26–4.29 (2H, 2m, CH_2 of CO_2Et); 4.31–4.34 (2H, q, CH_2 of CO_2Et); 7.06–7.07 (1H, d, J = 4.59Hz, Ar protons); 7.14–7.17 (2H, m, Ar protons), 8.56–8.58 (1H, d, J = 9.15Hz, NH)	14.32, 14.88 (2 CH_3 of CO_2Et); 24.62, 25.91, 26.17 (CH_2 of cyclohexyl); 33.13 (CH_3); 49.08 (C_{ipso} of cyclopentene); 61.13, 63.48 (CH_2 of CO_2Et); 68.92 (CH of cyclohexyl); 122.63, 125.32, 127.47, 128.42, 129.47, 132.43, 146.74, 148.41 (Alkene and Ar carbons); 163.19, 164.28 (2 C=O of CO_2Et); 171.15 (C=O of amide)	432, 434 (M^+, M^++2, 25,8) 396, 398 (M^+, M+ 2-C_2H_5OH, 35, 9) 307, 309 (M^+, M+2-$C_6H_{11}NCO$, 85, 30) 261, 263 (M^+, M+2-$C_6H_{11}NCO$, C_2H_3OH, 100, 39)

Table 4
Yields, mps and Elemental Analysis of **2a-f**

Cmpd	*Yields (%)	mp (°C)	Elemental Analysis (Found) C	H	N	Formula (M.W.)
2a	87	134–135	60.57(60.55)	5.81(5.80)	6.73(6.74)	$C_{21}H_{24}N_2O_7$ (416.42)
2b	85	142–143	62.15(62.13)	6.35(6.32)	6.30(6.33)	$C_{23}H_{28}N_2O_7$ (444.48)
2c	88	131–132	60.57(60.58)	5.81(5.80)	6.73(6.75)	$C_{21}H_{24}N_2O_7$ (416.42)
2d	86	139–140	62.15(62.13)	6.35(6.35)	6.30(6.34)	$C_{23}H_{28}N_2O_7$ (444.48)
2e	85	121–122	62.14(62.12)	5.96(5.94)	3.45(3.48)	$C_{21}H_{24}ClNO_5$ (405.87)
2f	83	128–129	63.66(63.65)	6.50(6.47)	3.23(3.24)	$C_{23}H_{28}ClNO_5$ (433.93)

*Yields from one-pot procedure.

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