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# CATALYST-FREE ONE-POT SYNTHESIS OF NOVEL KETENE IMINES

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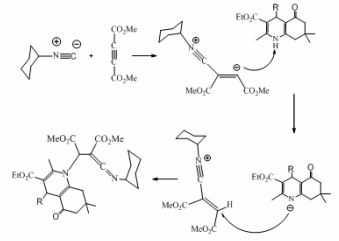
This report provides a description of an efficient and simple procedure for the synthesis of novel ketene imines *via* one-pot three-component reaction of cyclohexyl isocyanides, dimethyl acetylenedicarboxylates and hexahydroquinolines in $CH_2Cl_2$ under catalyst-free conditions at room temperature.



## INTRODUCTION

Isocyanide-based multicomponent reactions (IMCRs) play an important role in synthetic complex molecules.[1-4] Isocyanides are known to form zwitterions with activated acetylene compounds.[5-7] These zwitterions can be trapped by a variety of electrophiles and proton donors such as CH, NH and OH acids. Ketene imine derivatives are reactive synthetic intermediates, which react readily with a wide range of nucleophiles, electrophiles or radicals to afford the corresponding nitrogen-containing heterocycles.[8,9] They also undergo many pericyclic reactions such as electrocyclic ring closures, [2+2] and [4+2] cycloaddition reactions.[10-12] Ketene imine derivatives have been prepared *via* various procedures such as imidation of ketene precursors,[13] dehydrohalogenation of imidoyl halides under basic conditions,[14] treatment of nitriles with a Brønsted base followed by substitution reaction,[15] and the reaction of isocyanides, acetylenic esters, and various compounds as proton source.[16-24] However, these methods for preparing ketene imine derivatives have not been entirely satisfactory and involve some disadvantages such as low yields, prolonged reaction times and cumbersome procedures for product isolation. As part of our current studies for developing an efficient and more general protocol for the synthesis of ketene imines,[25] we now report the one-pot three-component synthesis of some new ketene imine derivatives by the reaction of cyclohexyl isocyanide, dimethyl acethylendicarboxylate and NH-acids such as hexahydroquinolines under catalyst-free conditions.

## RESULTS AND DISCUSSION

In our initial study, the reaction of cyclohexyl isocyanide (**1**), dimethyl acethylendicarboxylate (**2**), and **3a** was considered as a model one to optimize the conditions. To find the best solvent, several solvents such as MeCN $(Me)_2CO$, $CH_2Cl_2$, THF and EtOH were employed as media. It was noticed that the highest yield was achieved in $CH_2Cl_2$ under catalyst-free conditions (Table 1). Similar reactions were then attempted in $CH_2Cl_2$ at reflux and room temperature, which resulted in the isolation of the product in yields of 60% and 83% respectively. It was clear that the highest yield was produced when the reaction time was 4 h, although the yield did not to improve to any greater extent

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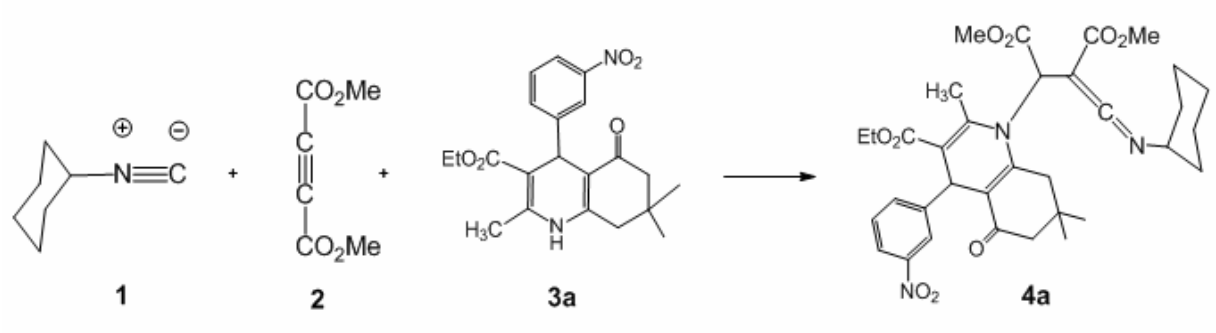
when the reaction time was increased from 4 h to 6 h. For the purpose of saving energy, we therefore chose 4 h as the reaction time. Hence, the best results were obtained in $CH_2Cl_2$ at room temperature for 4 h. The structures of the products were deduced by elemental analysis, IR, $^1$H- and $^{13}$C-NMR spectra. Comparison of the IR and $^1$H-NMR data with raw materials shows that **4a** displayed the absorption band for the ketene imine moiety at 2075 cm$^{-1}$ and a singlet for N-CH proton at 5.20 ppm respectively. The $^{13}$C-NMR spectrum of **4a** exhibited 32 sharp signals. The sp hybridized carbon atom appears at $\delta$ = 60.4 ppm.

The hexahydroquinolins **3a** and **3b-i**, as NH acids, were synthesized *via* four-component reaction of 5,5-dimethyl-1,3-cyclohexanedione, ethylacetoacetate, aldehyde and amounium acetate in one-pot at room temperature in EtOH (Scheme 1).[26]

Using the optimized reaction conditions, we extended our study to different hexahydroqinnolins to prepare a series of new substituted ketene imines (**4b-i**, Table 2).

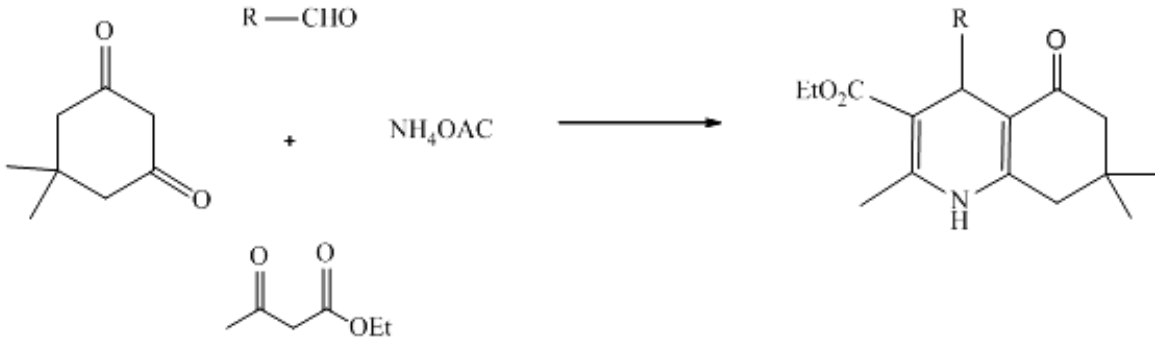
*Table 1*

The effect of solvent on the reaction of **1**, **2** and **3a**[a)]




| Entry | Solvent | Yield (%) |
|---|---|---|
| 1 | MeCN | 45 |
| 2 | Acetone | 75 |
| 3 | $CH_2Cl_2$ | 83 |
| 4 | THF | 55 |
| 5 | EtOH | 10 |

[a)] Reaction conditions: cyclohexyl isocyanide (1 mmol), dimethyl acethylendicarboxylate (1 mmol), **3a** (1 mmol), r.t.

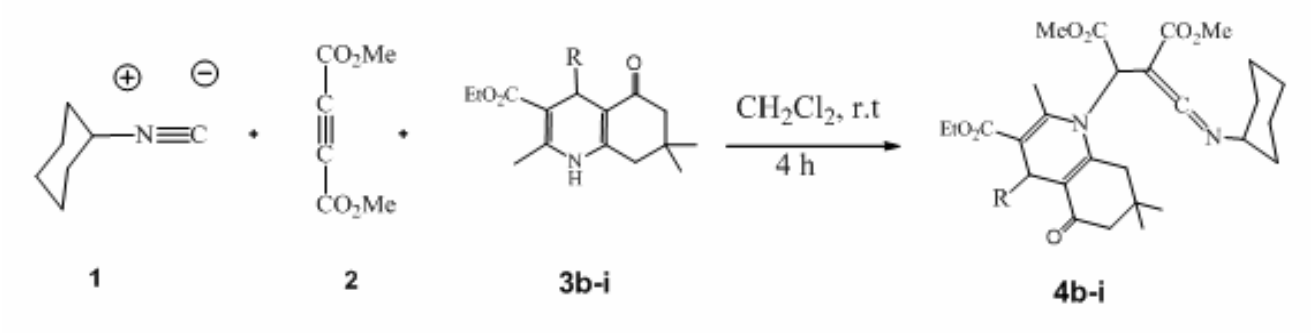


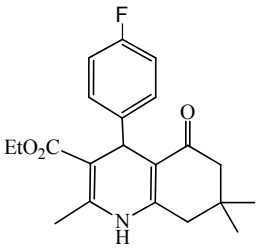
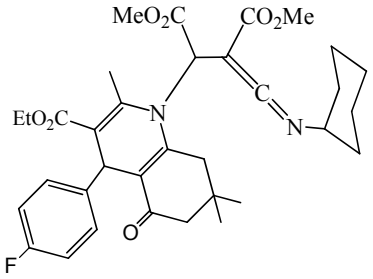
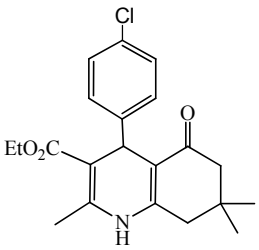
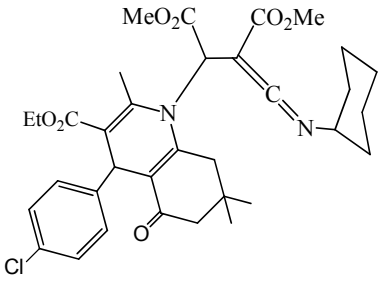
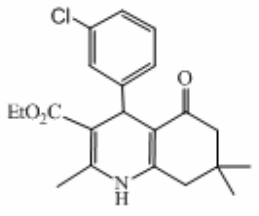
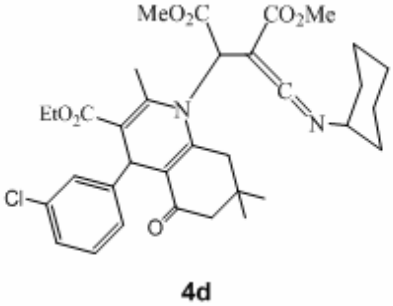

R= 3-$O_2NC_6H_4$, 4-$FC_6H_4$, 4-$ClC_6H_4$, 3-$ClC_6H_4$, 3-$BrC_6H_4$, 4-$MeOC_6H_4$, 4-$(Me)_2NC_6H_4$, 4-$MeC_6H_4$, 1-Naphthyl

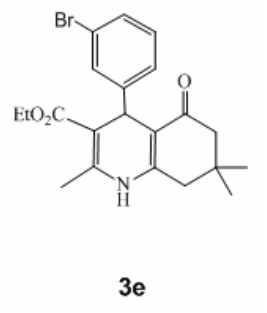
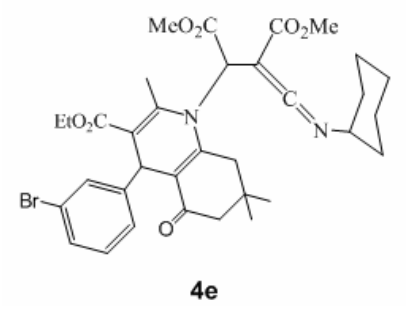
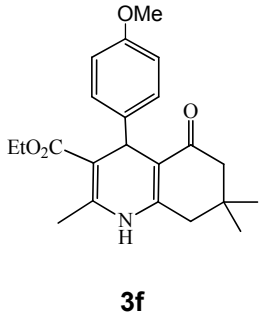
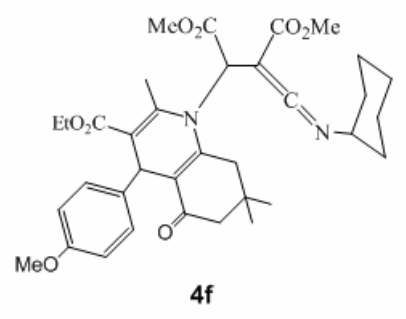
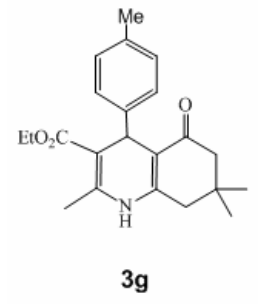
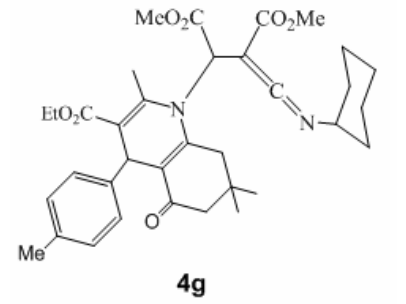
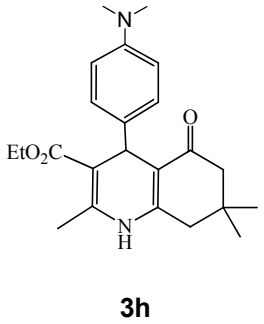
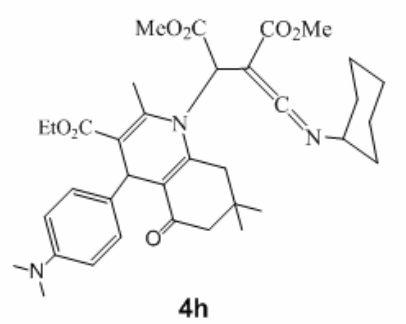
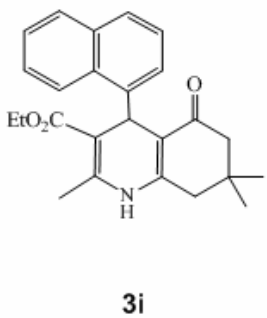
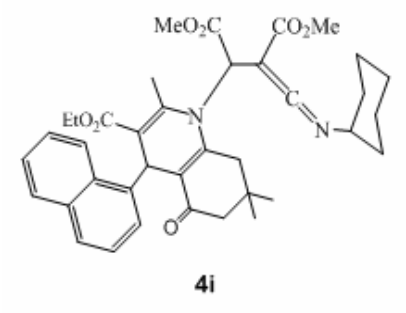
Scheme 1 – Synthesis of hexahydroquinolin derivatives.

*Table 2*

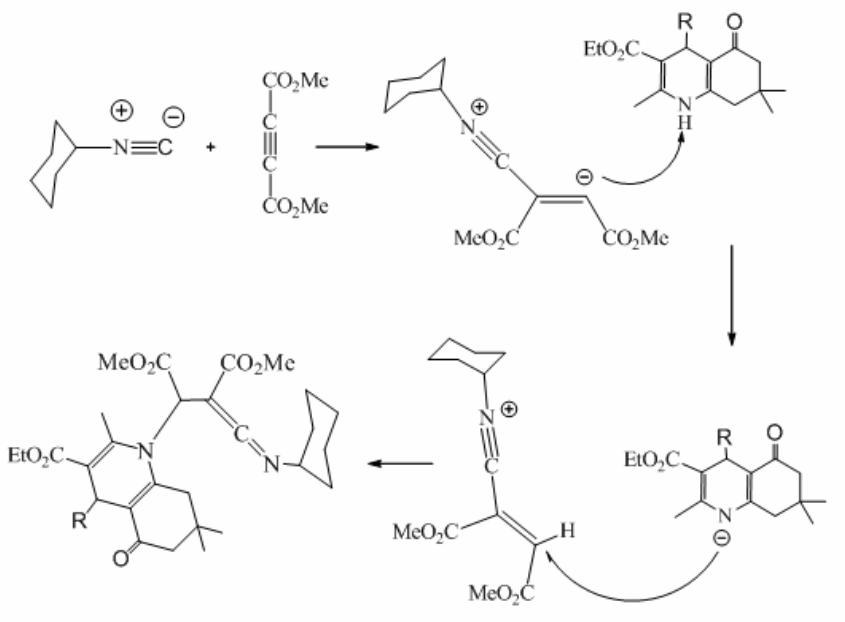
Synthesis of ketene imine derivatives under catalyst-free conditions [a)]




| Entry | Hexahydroqinnolin | Product | Yield(%) | Mp(°C) |
|---|---|---|---|---|
| 1 |  **3b** |  **4b** | 79 | 67-68 |
| 2 |  **3c** |  **4c** | 81 | 66-68 |
| 3 |  **3d** |  **4d** | 78 | 58-60 |

| | | | | |
|---|---|---|---|---|
| 4 |  **3e** |  **4e** | 82 | 71-73 |
| 5 |  **3f** |  **4f** | 80 | 84-85 |
| 6 |  **3g** |  **4g** | 78 | 73-75 |
| 7 |  **3h** |  **4h** | 76 | 119-121 |
| 8 |  **3i** |  **4i** | 78 | 72-74 |

a) Reaction conditions: cyclohexyl isocyanide (1 mmol), dimethyl acethylendicarboxylate (1 mmol), hexahydroquinolines (1 mmol).



Scheme 2 – A possible mechanism for the synthesis of ketene imines.

Although the mechanism of the reaction has not been established experimentally, the formation of product involves the addition of isocyanide to diethyl acetylenedicarboxylate and the subsequent protonation of adduct by the NH-acid. Then, the positively charged ion might be attacked by the anion of NH-acid to form ketene imine (Scheme 2).

## EXPERIMENTAL

### General

The products were characterized by elemental analysis, IR, $^1$H- and $^{13}$C-NMR spectra. $^1$H- and $^{13}$C-NMR spectra were obtained in DMSO-$d_6$ on a Bruker Avance 400 MHz spectrometers. The elemental analysis was done by a Costech ECS 4010 CHN analyser. The melting points were determined by a Buchi melting point B-540 B.V.CHI apparatus. Column chromatography was performed on silica gel (230–400) mesh. Analytical TLC was performed on pre-coated plastic sheets of silica gel G/UV-254 of 0.2 mm thickness.

### Typical procedure for the synthesis of ketene imine derivatives

To a magnetically stirred solution of ethyl 2,7,7-trimethyl-4-(3-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (1 mmol) and diethyl acetylenedicarboxylate (1 mmol) in dry $CH_2Cl_2$ (3 mL) was added a solution of cyclohexyl isocyanide (1 mmol) in dry $CH_2Cl_2$ (2 mL) dropwise at room temperature over 10 minutes and the mixture was stirred at room temperature for 4 h. After completion of the reaction as indicated by TLC, the solvent was removed under reduced pressure, and dimethyl-2-((cyclohexylimino)methylene)-3-(3-(ethoxycarbonyl)-2,7,7-trimethyl-4-(3-nitrophenyl)-5-oxo-5,6,7,8-tetrahydroquinoline-1(4H)-yl)succinate (**4a**) was separated by silica gel column chromatography using a hexane/ethyl acetate (70:30) as eluent.

### Spectroscopic data of new compounds 4a-i

*Dimethyl-2-((cyclohexylimino)methylene)-3-(3-(ethoxycarbonyl)-2,7,7-trimethyl-4-(3-nitrophenyl)-5-oxo-5,6,7,8-tetrahydroquinolin-1(4H)-yl)succinate* (**4a**)

Yellow crystal, mp 60-62 °C. IR: 2075 (C=C=N), 1744 (CO), 1698 (CO). $^1$H-NMR: δ = 0.88 (s, 3H), 0.97 (s, 3H), 1.14-1.34 (m, 5H), 1.58 (t, 3H), 1.64-1.72 (m, 6H), 1.92 (d, 1H), 2.07 (d, 1H), 2.12 (d, 1H), 2.27 (d, 1H), 2.29 (s, 3H), 3.68 (s, 1H), 3.74 (s, 3H), 3.87 (s, 3H), 4.06 (q, 2H), 5.20 (s, 1H), 7.34 (t, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.87 (d, 1H), 8.08 (s, 1H . $^{13}$C-NMR: δ = 14.1, 17.3, 23.8, 26.5, 29.7, 30.8, 32.1, 33.2, 36.8, 39.7, 40.1, 49.9, 50.4, 52.0, 53.5, 59.1, 60.4, 107.7, 115.2, 121.5, 122.9, 128.5, 130.9, 134.9, 143.8, 147.6, 152.3, 153.8, 164.3, 167.3, 169.9, 195.7. Anal. calc. for $C_{34}H_{41}N_3O_9$ (635.70): C 64.24, H 6.50, N 6.61; found: C 64.5, H 6.2, N 6.6.

*Dimethyl-2-((cyclohexylimino)methylene)-3-(3-(ethoxycarbonyl)-4-(4-fluorophenyl)-2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinolin-1(4H)-yl)succinate* (**4b**)

Yellow crystal, mp 67-68 °C. IR: 2070 (C=C=N), 1743 (CO), 1684 (CO). $^1$H-NMR: δ = 1.09 (s, 3H), 1.11 (s, 3H), 1.13-1.26 (m, 5H), 1.49 (t, 3H), 1.65-1.75 (m, 6H), 1.94 (d, 1H), 2.00 (d, 1H), 2.09 (d, 1H), 2.16 (d, 1H), 2.27 (s, 3H), 3.52 (s, 1H), 3.74 (s, 3H), 3.76 (s, 3H), 3.97 (q, 2H), 5.10 (s, 1H), 6.84 (m, 2H), 7.21 (m, 2H). $^{13}$C-NMR: δ = 14.2, 17.9, 23.6, 23.9, 25.2, 26.3, 29.4, 29.7, 31.5, 32.9, 35.7, 39.5, 50.4, 52.6, 52.8, 60.1, 60.4, 107.9, 114.9, 123.7, 129.5, 141.9, 142.8, 146.4, 147.8, 160.3, 164.8, 165.1, 167.5, 195.8. Anal. calc. for $C_{34}H_{41}FN_2O_7$ (608.70): C 67.09, H 6.79, N 4.60; found: C 67.5, H 6.5, N 4.4.

*Dimethyl-2-(4-(4-chlorophenyl)-3-(ethoxycarbonyl)-2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinolin-1(4H)-yl)-3-((cyclohexylimino)methylene)succinate* (**4c**)

Yellow crystal, mp 66-68 °C. IR: 2067 (C=C=N), 1731 (CO), 1696 (CO), $^1$H-NMR: δ = 0.75 (s, 3H), 0.96 (s, 3H), 1.12-125 (m, 5H), 1.49 (t, 3H), 1.75-180 (m, 6H), 1.97-2.17

(2d, 2H), 2.20-2.32 (2d, 2H), 2.37 (s, 3H), 3.51 (s, 1H), 3.74 (s, 3H), 3.86 (s, 3H), 3.94 (q, 2H), 5.09 (s, 1H), 7.12 (d, 2H), 7.19 (d, 2H). $^{13}$C-NMR: δ = 14.2, 17.1, 23.6, 25.2, 26.3, 29.4, 29.7, 31.7, 32.1, 32.9, 35.9, 39.5, 50.5, 52.3, 52.8, 59.8, 60.2, 107.9, 113.8, 123.5, 128.8, 129.5, 143.0, 144.6, 146.1, 147.8, 164.2, 165.9, 167.5, 195.7. Anal. calc. for $C_{34}H_{41}ClN_2O_7$ (625.15): C 65.32, H 6.61, N 4.48; found: C 65.0, H 6.2, N 4.2.

*Dimethyl-2-(4-(3-chlorophenyl)-3-(ethoxycarbonyl)-2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinolin-1(4H)-yl)-3-((cyclohexylimino)methylene)succinate* (**4d**)

Yellow crystal, mp 58-60 °C. IR: 2074 (C=C=N), 1744 (CO), 1697 (CO). $^1$H-NMR: δ = 0.88 (s, 3H), 1.05 (s, 3H), 1.19-1.27 (m, 3H), 1.61 (t, 3H), 1.75-1.85 (m, 6H), 2.10-2.27 (4d, 4H), 2.36 (s, 3H), 3.66 (s, 1H), 3.83 (s, 3H), 3.95 (s, 3H), 4.07-4.13 (q, 2H), 5.19 (s, 1H), 7.13 (d, 1H), 7.18 (t, 1H), 7.26 (d, 1H), 7.30 (brs, 1H). $^{13}$C-NMR: δ = 14.1, 17.1, 23.3, 23.7, 25.3, 26.8, 29.70, 31.6, 32.7, 36.4, 39.7, 50.4, 50.52, 52.6, 52.8, 60.1, 60.4, 107.5, 113.0, 123.6, 126.6, 129.2, 132.3, 137.4, 143.1, 146.7, 148.0, 151.8, 164.2, 165.0, 167.0, 195.7. Anal. calc. for $C_{34}H_{41}ClN_2O_7$ (625.15): C 65.32, H 6.61, N 4.48; found: C 65.5, H 6.3, N 4.6.

*Dimethyl-2-(4-(3-bromophenyl)-3-(ethoxycarbonyl)-2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinolin-1(4H)-yl)-3-((cyclohexylimino)methylene)succinate* (**4e**)

Yellow crystal, mp 71-73 °C. IR: 2073 (C=C=N), 1737 (CO), 1695 (CO). $^1$H-NMR: δ = 0.79 (s, 3H), 0.96 (s, 3H), 1.22-129 (m, 5H), 1.48 (t, 3H), 1.62-170 (m, 6H), 2.01-2.18 (4d, 4H), 2.27 (s, 3H), 3.38 (s, 3H), 3.58 (s, 1H), 3.83 (s, 3H), 3.97 (q, 2H), 5.08 (s, 1H), 7.03 (t, 1H), 7.18-7.22 (m, 2H), 7.37 (brs, 1H). $^{13}$C-NMR: δ = 14.1, 17.1, 23.2, 23.5, 25.3, 26.5, 29.3, 29.7, 31.7, 32.9, 36.8, 39.7, 50.4, 52.6, 52.8, 60.3, 60.5, 107.5, 113.0, 123.6, 127.7, 129.5, 131.2, 143.1, 146.0, 146.7, 147.8, 148.9, 164.7, 165.9, 167.0, 195.6. Anal. calc. for $C_{34}H_{41}BrN_2O_7$ (669.60): C 60.99, H 6.17, N 4.18; found: C 60.7, H 6.5, N 4.0.

*Dimethyl-2-((cyclohexylimino)methylene)-3-(3-(ethoxycarbonyl)-4-(4-methoxyphenyl)-2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinolin-1(4H)-yl)succinate* (**4f**)

Yellow crystal, mp 84-85 °C. IR: 2073 (C=C=N), 1745 (CO), 1698 (CO). $^1$H-NMR: δ = 0.76 (s, 3H), 0.95 (s, 3H), 1.37-1.45 (m, 5H), 1.54 (t, 3H), 1.74-1.85 (m, 6H), 1.98-2.06 (2d, 2H), 2.09 (d, 1H), 2.15 (d, 1H), 2.25 (s, 3H), 3.57 (s, 1H), 3.69 (s, 3H), 3.73 (s, 3H), 3.86 (s, 3H), 4.10 (q, 2H), 5.06 (s, 1H), 6.69 (d, 2H), 7.16 (d, 2H). $^{13}$C-NMR: δ = 14.6, 17.0, 23.6, 23.9, 25.3, 26.4, 29.9, 31.6, 32.1, 35.7, 39.4, 47.8, 50.5, 52.5, 52.8, 55.2, 59.2, 60.5, 108.3, 113.4, 123.7, 129.1, 130.4, 138.5, 142.7, 147.8, 157.9, 164.4, 165.8, 167.9, 195.8**.** Anal. calc. for $C_{35}H_{44}N_2O_8$ (620.73): C 67.72, H 7.14, N 4.51; found: C 67.5, H 7.2, N 4.8.

*Dimethyl-2-((cyclohexylimino)methylene)-3-(3-(ethoxycarbonyl)-2,7,7-trimethyl-5-oxo-4-(p-tolyl)-5,6,7,8-tetrahydroquinolin-1(4H)-yl)succinate* (**4g**)

Yellow crystal mp 73-75 °C. IR: 2070 (C=C=N), 1734 (CO), 1694 (CO). $^1$H-NMR: δ = 0.78 (s, 3H), 0.92 (s, 3H), 0.99-1.20 (m, 5H), 1.50 (t, 3H), 1.70-180 (m, 6H), 1.93-2.03 (2d, , 2H), 2.10-2.15 (2d, 2H), 2.20 (s, 3H), 2.25 (s, 3H), 3.56 (s, 1H), 3.73 (s, 3H), 3.86 (s, 3H), 4.00 (q, 2H), 5.07 (s, 1H), 5.96 (s, 1H), 6.95 (d, 2H), 7.13 (d, , 2H). $^{13}$C-NMR: δ = 14.5, 17.7, 21.4, 23.6, 23.8, 25.3, 26.5, 29.8, 31.6, 32.4, 32.9, 35.9, 39.7, 50.2, 52.5, 52.8, 60.3, 60.5, 108.3, 113.7, 123.6, 128.0, 128.7, 135.7, 142.4, 143.1, 146.2, 164.3, 167.3, 169.5, 195.8. Anal. calc. for $C_{35}H_{44}N_2O_7$ (604.73): C 69.51, H 7.33, N 4.63; found: C 69.5, H 7.1, N 4.3.

*Dimethyl-2-((cyclohexylimino)methylene)-3-(4-(4-(dimethylamino)phenyl)-3-(ethoxycarbonyl)-2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinolin-1(4H)-yl)succinate* (**4h**)

Yellow crystal, mp 119-121 °C. 2082 (C=C=N), 1745 (CO), 1692 (CO). $^1$H-NMR: δ = 0.88 (s, 3H), 1.04 (s, 3H), 1.24-132 (m, 5H), 1.45 (t, 3H), 1.78-1.85 (m, 6H), 2.12-2.23 (4d, 4H), 2.33 (s, 3H), 2.90 (s, 6H), 3.67 (s, 1H), 3.82 (s, 3H), 3.95 (s, 3H), 4.13 (q, 2H), 5.11 (s, 1H), 6.62 (d, 2H), 7.18 (d, 2H). $^{13}$C-NMR: δ = 14.3, 17.9, 23.7, 24.3, 25.4, 26.6, 29.1, 29.7, 31.1, 31.6, 32.9, 35.1, 39.6, 40.7, 50.8, 52.6, 52.9, 60.1, 60.4, 108.9, 112.8, 123.6, 128.7, 134.6, 139.6, 145.8, 146.7, 149.5, 164.40, 165.5, 167.6, 195.9. Anal. calc. for $C_{36}H_{47}N_3O_7$ (633.77): C 68.22, H 7.47, N 6.63; found: C 68.5, H 7.2, N 6.5.

*Dimethyl-2-((cyclohexylimino)methylene)-3-(3-(ethoxycarbonyl)-2,7,7-trimethyl-4-(naphthalen-1-yl)-5-oxo-5,6,7,8-tetrahydroquinolin-1(4H)-yl)succinate* (**4i**)

Yellow crystal, mp 72-74 °C. IR: 2074 (C=C=N), 1741 (CO), 1699 (CO). $^1$H-NMR: δ = 0.79 (s, 3H), 0.95 (s, 3H), 1.18-125 (m, 5H), 1.55 (m, 3H), 1.74-1.83 (m, 6H), 1.98-2.12 (m, 4H), 2.27 (s, 3H), 3.68 (m, 1H), 3.75 (s, 3H), 3.88 (s, 3H), 4.02-4.07 (q, 2H), 5.72 (s, 1H), 7.26 (t, 1H), 7.34-7.39 (m, 2H), 7.46 (t, 1H), 7.51 (t, 1H), 7.58 (d, 1H), 7.68 (d, 1H). $^{13}$C-NMR: δ = 13.8, 17.1, 23.9, 24.1, 25.4, 26.5, 29.7, 31.6, 32.3, 32.8, 39.6, 50.6, 52.6, 52.8, 53.4, 60.2, 60.9, 105.9, 114.8, 123.9, 125.0, 125.5, 125.9, 125.9, 126.6, 127.3, 127.8, 133.4, 141.5, 145.1, 146.6, 147.9, 164.3, 165.9, 167.4, 195.8. Anal. calc. for $C_{38}H_{44}N_2O_7$ (640.70): C 71.23, H 6.92, N 4.37; found: C 70.9, H 6.8, N 4.5.

## CONCLUSIONS

In summary, we have developed a straightforward and efficient method for the preparation of novel ketene imine derivatives through the three-component coupling of cyclohexyl isocyanide, dimethyl acetylenedicarboxylates and hexahydroquinoline derivatives under catalyst-free conditions. This method provides several advantages such as operational simplicity, catalyst-free, high yields, and short reaction time under mild reaction conditions.

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