Supporting Information

For

Oximes as reusable templates for the synthesis of ureas and carbamates by an in situ generation of carbamoyl oximes

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1. **General Information**  

\(^1\)H, \(^{13}\)C and DEPT NMR spectra were recorded on a 400 MHz Varian Unity Plus or Varian Mercury plus spectrometer 400MHz. The chemical shift (\(\delta\)) values are reported in parts per million (ppm), and the coupling constants (\(J\)) are given in Hz. The spectra were recorded using CDCl\(_3\) and DMSO-\(d_6\) as a solvent. \(^1\)H NMR chemical shifts are referenced to tetramethylsilane (TMS) (0 ppm). \(^{13}\)CNMR was referenced to CDCl\(_3\) (77.0 ppm) and DMSO-\(d_6\) (39.52 ppm). The abbreviations used are as follows: s, singlet; d, doublet; t, triplet; q, quartet; dd, doublet of doublet; ddd, doublet of doublet of doublet; dt, doublet of triplets; td, triplet of doublet; m, multiplet. Mass spectra and high-resolution mass spectra (HRMS) was measured using the LTQ Orbitrap XL (Thermo Fischer Scientific) at National Chung Hsing University, Taichung, Taiwan. Melting points were determined on an EZ-Melt (Automated melting point apparatus). All the synthesized products showed \(^1\)HNMR spectra in agreement with the assigned structures. Reaction progress and product mixtures were routinely monitored by TLC using Merck TLC aluminum sheets (silica gel 60 F254). Column chromatography was carried out with 230-400 mesh silica gel 60(Merck) using a mixture of hexane/ethyl acetate as the eluent.
Experimental Procedures

Scheme S1. General Schemes for the Synthesis of Oximes, Carbamoyl Oximes, Ureas and Carbamates

**General Procedure (A) for the Synthesis of Oximes (1a-1g)**

A suspension of hydroxylamine hydrochloride (1.6 equiv), and NaOAc (2.0 equiv) in 80% aqueous EtOH (20 mL) was stirred at room temperature for 30 min. To this solution, corresponding ketone/aldehyde (1.0 equiv) was added and the reaction mixture was heated gently to reflux for 1 h. After the completion of reaction by TLC, the reaction mass was cooled to rt followed by removal of excess ethanol under vacuum. The obtained residue was purified by column chromatography with hexane-ethyl acetate (9:1) as eluent to obtain crystalline oximes (1) in quantitative yield.

**General Procedure for (B) the Synthesis of Carbamoyl Oximes (3aa-3ah)**

To a stirred solution of oximes (1a-1g, 1.0 equiv) in 1,4-dioxane were added NIS (20 mol %), TBHP (70% in H₂O) (2.0 equiv), and isocyanide (1.2 equiv). The resultant mixture was heated at 90 °C for 4 h. After the completion of reaction by TLC, the reaction mixture was cooled to rt, diluted with water and extracted with ethyl acetate. The combined organic layer was dried over Na₂SO₄, filtered and concentrated to give crude material. The crude material was purified by column chromatography using hexane-ethyl acetate as a eluent to obtain pure carbamoyl oximes 3aa-3ah.
General Procedure (C) for the Synthesis of Ureas (5a-5q)

To a stirred solution of oxime (1a, 1.0 equiv) in 1,4-dioxane were added NIS (20 mol %), TBHP (70% in H₂O) (2.0 equiv), and isocyanide (1.2 equiv). The resultant mixture was heated at 90 °C for 4 h. After the completion of reaction by TLC, the reaction mixture was cooled to rt and amines (4, 1.2 equiv) was added and heated further at 90 °C for 16 h. After the completion of reaction by TLC, the reaction mixture diluted with water and extracted with ethyl acetate. The combined organic layer dried over Na₂SO₄, filtered and concentrated to give crude material. The crude material was purified by column chromatography using hexane and ethyl acetate as a eluent to obtain pure ureas 5a-5r.

General Procedure for (D) the Synthesis of Carbamates (7a-7g)

To a stirred solution of oxime (1a, 1.0 equiv) in 1,4-dioxane were added NIS (20 mol %), TBHP (70% in H₂O) (2.0 equiv), and isocyanide (1.2 equiv). The resultant mixture was heated at 90 °C for 4 h. After the completion of reaction by TLC, the reaction mixture was cooled to rt followed by the addition of NaH (1.5 equiv), and alcohol (6, 1.2 equiv). The resultant mixture was heated at 90 °C for 16 h. After the completion of reaction by TLC, the reaction mixture was diluted with water and extracted with ethyl acetate. The combined organic layer dried over Na₂SO₄, filtered and concentrated to give crude material. The crude material was purified by column chromatography using hexane and ethyl acetate as a eluent to obtain pure carbamates 7a-7g.

(E)-1-phenylethan-1-one O-cyclohexylcarbamoyl oxime (3aa):² The title compound was prepared according to the general procedure B to obtain as a yellow solid (225 mg, Yield = 86%); Mp. 58.2-60.8 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.66 (dd, J = 7.6, 1.2 Hz, 2H), 7.51-7.38 (m, 3H), 6.34 (d, J = 5.2 Hz, 1H), 3.67-3.62 (m, 1H), 2.02 (dd, J = 10.4, 1.2 Hz, 2H), 1.77-1.70 (dt, J = 13.6, 3.6 Hz, 2H), 1.62 (dt, J = 7.6, 3.6 Hz, 1H), 1.45-1.34 (m, 2H), 1.30-1.17 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.79, 154.57, 134.93, 130.43, 128.60, 126.65, 49.98, 33.11, 25.42, 24.75, 14.47; HRMS (ESI): Calc’d for [C₁₅H₂₀N₂O₂Na]⁺ [M+Na]⁺ 283.1417, found 283.1412.
(E)-1-(4-fluorophenyl)ethan-1-one O-cyclohexylcarbamoyl oxime (3ba): The title compound was prepared according to the general procedure B to obtain as a yellow solid (220 mg, yield = 79%); Mp. 66.3-68.5 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.70-7.64 (m, 2H), 7.15-7.09 (m, 2H), 6.25 (d, $J = 7.4$ Hz, 1H), 3.72-3.63 (m, 1H), 2.02 (dd, $J = 12.5, 3.6$ Hz, 2H), 1.74 (dt, $J = 8, 4$ Hz, 2H). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.70-7.64 (m, 2H), 7.15-7.09 (m, 2H), 6.25 (d, $J = 12.5, 3.6$ Hz, 2H), 1.74 (dt, $J = 8, 4$ Hz, 2H), 1.63 (dt, $J = 7.6, 3.6$ Hz, 1H), 1.45-1.34 (m, 2H), 1.30-1.20 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.10 (d, $J_F = 254.1$ Hz), 158.81, 154.45, 131.06 (d, $J_F = 3.6$ Hz), 128.70 (d, $J_F = 8.5$ Hz), 115.62 (d, $J_F = 21.6$ Hz), 50.04, 33.15, 25.44, 24.77, 14.48; HRMS (ESI): Calc’d for [C$_{15}$H$_{19}$FO$_2$N$_2$Na]$^+$ [M+Na]$^+$ 301.1323, found 301.1320.

(E)-1-(3,4-dichlorophenyl)ethan-1-one O-cyclohexylcarbamoyloxime (3ca): The title compound was prepared according to the general procedure B to obtain as a yellow solid (270 mg, yield = 82%); Mp. 91.2-93.1 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.76 (s, 1H), 7.51 (s, 2H), 6.15 (d, $J = 7.2$ Hz, 1H), 3.70-3.63 (m, 1H), 2.39 (s, 3H), 2.02-2.01 (m, 2H), 1.76-1.73 (m, 2H), 1.65-1.62 (m, 1H), 1.44-1.35 (m, 2H), 1.31-1.20 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 157.89, 154.06, 134.80, 133.07, 130.63, 128.57, 125.84, 50.18, 33.11, 25.41, 24.78, 14.32; HRMS (ESI): Calc’d for [C$_{15}$H$_{18}$Cl$_2$N$_2$O$_2$Na]$^+$ [M+Na]$^+$ 351.0638, found 351.0634.

Methyl(E)-4-(1-(((cyclohexylcarbamoyl)oxy)imino)ethyl)benzoate (3da): The title compound was prepared according to the general procedure B to obtain as a yellow solid (255 mg, yield = 80%); Mp. 111.6-118.2 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.09 (d, $J = 8.4$ Hz, 2H), 7.74 (d, $J = 8.4$ Hz, 2H), 6.25 (d, $J = 7.6$ Hz, 1H), 3.95 (s, 3H), 3.94-3.64 (m, 1H), 2.45 (s, 3H), 2.03 (dd, $J = 12.4, 3.2$ Hz, 2H), 1.74 (dt, $J = 8, 3.6$ Hz, 2H), 1.64 (dt, $J = 7.6, 4$ Hz, 1H), 1.44-1.35 (m, 2H), 1.31-1.19 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.31, 159.02, 154.22, 139.07, 131.74, 129.80, 126.68, 52.32, 50.10, 33.11, 25.42, 24.75, 14.47; HRMS (ESI): Calc’d for [C$_{17}$H$_{22}$N$_2$O$_4$Na]$^+$ [M+Na]$^+$ 341.1472, found 341.1468.

(E)-benzaldehyde O-cyclohexylcarbamoyloxime (3ea): The title compound was prepared according to the general procedure B to obtain as a yellow solid (185 mg, yield = 75%); Mp. 116.2-117.5 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.33 (bs, 1H), 7.67 (dd, $J = 8.0, 1.2$ Hz,
2H), 7.52-7.38 (m, 3H), 6.12 (d, J = 6.8 Hz, 1H), 3.73-3.61 (m, 1H), 2.03 (dd, J = 12.4, 3.2 Hz, 2H), 1.75 (dt, J = 8.0, 3.6 Hz, 2H), 1.64 (dt, J = 7.2, 3.6 Hz, 1H), 1.66-1.623 (m, 2H), 1.31-1.20 (m, 3H); 13C NMR (100 MHz, CDCl3) δ 154.27, 153.02, 131.57, 130.09, 128.95, 128.02, 50.15, 33.15, 25.46, 24.77; HRMS (ESI): Calc’d for [C14H19N2O2]+ [M+H]+ 247.14410, found 247.14398.

(E)-1-(p-tolyl)ethan-1-one O-(tert-butylcarbamoyl) oxime (3fb): The title compound was prepared according to the general procedure B to obtain as a brown oil (213 mg, yield = 86%); 1H NMR (400 MHz, CDCl3) δ 7.56 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 8.0 Hz, 2H), 6.37 (s, 1H), 2.39 (s, 3H), 2.38 (s, 3H), 1.41 (s, 9H); 13C NMR (100 MHz, CDCl3) δ 159.24, 153.50, 140.68, 132.12, 129.27, 126.51, 50.73, 28.71, 14.26; HRMS (ESI): Calc’d for [C14H20N2O2Na]+ [M+Na]+ 271.1417, found 271.1413.

(E)-1-(4-chlorophenyl)ethan-1-one O-(tert-butylcarbamoyl) oxime (3gb): The title compound was prepared according to the general procedure B to obtain as a yellow solid (238 mg, yield = 87%); Mp. 82.7-83.8 °C; 1H NMR (400 MHz, CDCl3) δ 7.61 (d, J = 8.8 Hz, 2H), 7.40 (d, J = 8.8 Hz, 2H), 6.23 (bs, 1H), 2.39 (s, 3H), 1.42 (s, 9H); 13C NMR (100 MHz, CDCl3) δ 158.34, 153.14, 136.59, 133.45, 128.88, 127.93, 50.90, 28.74, 14.26; HRMS (ESI): Calc’d for [C13H17ClN2O2Na]+ [M+Na]+ 291.0871, found 291.0868.

(E)-1-phenylethan-1-one O-(tert-butylcarbamoyl)oxime (3ab): The title compound was prepared according to the general procedure B to obtain as a brown oil (202 mg, yield = 87%); 1H NMR (400 MHz, CDCl3) δ 7.69 (dd, J = 8.0, 1.6 Hz, 2H), 7.46-7.40 (m, 3H), 6.36 (bs, 1H), 2.41 (s, 3H), 1.42 (s, 9H); 13C NMR (100 MHz, CDCl3) δ 159.39, 153.43, 134.94, 130.37, 128.58, 126.58, 50.78, 28.69, 14.36; HRMS (ESI): Calc’d for [C13H18N2O2Na]+ [M+Na]+ 257.1260, found 257.1257.

(E)-1-phenylethan-1-one O-((4-methoxyphenyl)carbamoyl)oxime (3ac): The title compound was prepared according to the general procedure B to obtain as a yellow solid (235 mg, yield = 82%); Mp. 89.3-90.3 °C; 1H NMR (400 MHz, CDCl3) δ 8.27 (bs, 1H), 7.71 (dd, J = 8.0, 1.6 Hz, 2H), 7.50-7.40 (m, 5H), 6.89 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H), 2.49 (s, 3H); 13C NMR
(100 MHz, CDCl₃) δ 160.65, 156.58, 152.69, 134.72, 130.75, 129.99, 128.76, 126.80, 121.86, 114.30, 55.50, 14.77; HRMS (ESI): Calc’d for [C₁₆H₁₆N₂O₃Na⁺][M+Na⁺] 307.1053, found 307.1049.

(E)-1-phenylethan-1-one-O-((4-fluorophenyl)carbamoyl)oxime (3ad): The title compound was prepared according to the general procedure B to obtain as a yellow solid (200 mg, yield = 80%); Mp. 139.2-140.6 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.37 (bs, 1H), 7.71 (dd, J = 8, 1.6 Hz, 2H), 7.55-7.42 (m, 5H), 7.05 (t, J = 8.4 Hz, 2H), 2.49 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 160.96, 159.44 (d, J₇₁₁ = 244.9 Hz), 152.42, 134.54, 132.97 (d, J₇₁₁ = 2.7 Hz), 130.83, 128.76, 126.78, 121.61 (d, J₇₁₁ = 7.9 Hz), 115.72 (d, J₇₁₁ = 22.2 Hz), 14.79; HRMS (ESI): Calc’d for [C₁₅H₁₃FN₂O₂Na⁺][M+Na⁺] 295.0853, found 295.0850.

(E)-1-phenylethan-1-one O-((2,6-dimethylphenyl)carbamoyl)oxime (3ae): The title compound was prepared according to the general procedure B to obtain as a yellow solid (228 mg, yield = 80%); Mp. 112.8-113.6 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.90 (bs, 1H), 7.72 (dd, J = 8, 1.2 Hz, 2H), 7.51-7.42 (m, 3H), 7.16-7.09 (m, 3H), 2.52 (s, 3H), 2.33 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 160.45, 153.29, 135.68, 134.73, 133.21, 130.73, 128.75, 128.32, 127.52, 126.72, 18.34, 14.63; HRMS (ESI): Calc’d for [C₁₇H₁₈N₂O₂Na⁺][M+Na⁺] 305.1260, found 305.1254.

(E)-1-phenylethan-1-one O-benzylcarbamoyl oxime (3af): The title compound was prepared according to the general procedure B to obtain as a yellow solid (202 mg, yield = 75%); Mp. 84.7-85.3 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.64 (dd, J = 8.4, 2.0 Hz, 2H), 7.45-7.37 (m, 3H), 7.35-7.34 (m, 4H), 7.30-7.26 (m, 1H), 6.79 (bs, 1H), 4.54 (d, J = 4.0 Hz, 2H), 2.44 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 160.32, 155.58, 137.99, 134.72, 130.54, 128.66, 128.61, 127.53, 126.69, 45.04, 14.52; HRMS (ESI): Calc’d for [C₁₆H₁₆N₂O₂Na⁺][M+Na⁺] 291.1104, found 291.1098.

Ethyl(E)-(((1-phenylethylidene)amino)oxy)carbonyl)glycinate (3ag): The title compound was prepared according to the general procedure B to obtain as a brown oil (225 mg, yield = 85%); ¹H NMR (400 MHz, CDCl₃) δ 7.69 (dd, J = 8.0, 1.6 Hz, 2H), 7.50-7.39 (m, 3H), 6.97 (bs, 1H), 4.24 (q, J = 7.2 Hz, 2H), 4.12 (d, J = 5.6 Hz, 2H), 2.43 (s, 3H), 1.30 (t, J =
7.2 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.57, 160.52, 155.39, 134.55, 130.59, 128.61, 126.69, 61.54, 42.75, 14.39, 14.07; HRMS (ESI): Calc'd for [C$_{13}$H$_{16}$N$_2$O$_4$Na]$^+$ [M+Na]$^+$ 287.1002, found 287.0995.

(E)-1-phenylethan-1-one O-((tosylmethyl)carbamoyl) oxime (3ah): The title compound was prepared according to the general procedure B to obtain as a yellow solid (280 mg, yield = 80%); Mp. 125.4-126.5 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.82 (d, $J = 8.4$ Hz, 2H), 7.68 (dd, $J = 8.3$, 1.2 Hz, 2H), 7.53-7.43 (m, 3H), 7.35 (d, $J = 8.0$ Hz, 2H), 7.25-7.23 (m, 1H), 4.70 (d, $J = 6.8$ Hz, 2H), 2.45 (s, 3H), 2.41 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 161.51, 154.31, 145.60, 134.10, 133.62, 131.03, 130.05, 128.87, 128.80, 126.82, 62.05, 21.77, 14.58; HRMS (ESI): Calc'd for [C$_{17}$H$_{18}$N$_2$O$_4$SNa]$^+$ [M+Na]$^+$ 369.0879, found 369.0877.

1-cyclohexyl-3-phenylurea (5a):$^4$ The title compound was prepared according to the general procedure C to obtain as a white solid (105 mg, yield = 48%); Mp. 187.1-188.1 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.29-7.27 (m, 4H), 7.05 (ddd, $J = 8.8$, 5.6, 3.2 Hz, 1H), 6.86 (s, 1H), 5.06 (d, $J = 8$ Hz, 1H), 3.70-3.61 (m, 1H), 1.94 (dd, $J = 12.8$, 3.6 Hz, 2H), 1.67-1.64 (m, 1H), 1.58 (dt, $J = 7.6$, 3.6 Hz, 1H), 1.39-1.24 (m, 3H), 1.16-1.07 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 155.55, 138.89, 129.11, 123.29, 120.57, 48.90, 33.58, 25.49, 24.83; HRMS (ESI): Calc’d for [C$_{13}$H$_{19}$N$_2$O]$^+$ [M+H]$^+$ 219.1492, found 219.1500.

1-cyclohexyl-3-(p-tolyl)urea (5b):$^5$ The title compound was prepared according to the general procedure C to obtain as a white solid (170 mg, yield = 73%); Mp. 200-202 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.15-7.08 (m, 4H), 6.44 (s, 1H), 4.78 (d, $J = 7.6$ Hz, 1H), 3.69-3.60 (m, 1H), 2.30 (s, 3H), 1.93 (dd, $J = 12.4$, 4.0 Hz, 2H), 1.69-1.67 (m, 1H), 1.58 (dt, $J = 7.6$, 3.6 Hz, 1H), 1.38 -1.28 (m, 3H), 1.16-1.03 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 155.45 , 135.87 , 133.76 , 129.85, 121.91, 48.97, 33.66, 25.56, 24.87, 20.78; HRMS (ESI): Calc’d for [C$_{14}$H$_{21}$N$_2$O]$^+$ [M+H]$^+$ 233.1648, found 233.1646.
1-cyclohexyl-3-(2-methoxyphenyl)urea (5c): The title compound was prepared according to the general procedure C to obtain as a white solid (165 mg, yield = 66%); Mp. 160-162.5 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.00 (dd, \(J = 7.3, 2.0\) Hz, 1H), 6.99-6.92 (m, 2H), 6.85 (dd, \(J = 7.6, 3.6\) Hz, 2H), 1.99 (dd, \(J = 12.8, 3.6\) Hz, 2H), 1.70 (dt, \(J = 7.6, 3.6\) Hz, 2H), 1.59-1.58 (m, 1H), 1.48-1.33 (m, 2H), 1.19-1.10 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 154.54, 148.08, 128.69, 122.35, 121.20, 119.48, 110.11, 55.61, 49.15, 33.71, 25.58, 24.90; HRMS (ESI): Calc’d for [C\(_{14}\)H\(_{21}\)N\(_2\)O\(_2\)]\(^{+}\) [M+H]\(^{+}\) 249.1598, found 249.1595.

1-cyclohexyl-3-(3-methoxyphenyl)urea (5d): The title compound was prepared according to the general procedure C to obtain as a yellow solid (162 mg, yield = 65%); Mp. 148-150 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.20 (t, \(J = 8.4\) Hz, 1H), 7.07 (t, \(J = 2.0\) Hz, 1H), 6.86 (d, \(J = 7.6\) Hz, 1H), 6.65 (dd, \(J = 8.0, 2.0\) Hz, 1H), 4.11 (s, 1H), 3.79 (s, 3H), 3.50 (s, 1H), 1.93 (d, \(J = 9.2\) Hz, 1H), 1.70-1.66 (m, 1H), 1.60 (s, 4H), 1.36-1.25 (m, 1H), 1.18-1.05 (m, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 160.38, 153.04, 139.35, 129.89, 112.90, 109.78, 106.49, 77.31, 76.99, 76.67, 55.30, 49.32, 33.92, 25.53, 24.87, 1.01; HRMS (ESI): Calc’d for [C\(_{14}\)H\(_{20}\)N\(_2\)O\(_2\)Na\(^+\)] \([\text{M+Na}]^{+}\) 271.1417, found 271.1415.

1-cyclohexyl-3-(4-methoxyphenyl)urea (5e):\(^6\) The title compound was prepared according to the general procedure C to obtain as a white solid (158 mg, yield = 64%); Mp. 162-164 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.17 (d, \(J = 8.8\) Hz, 2H), 6.87 (d, \(J = 9.2\) Hz, 2H), 6.12 (s, 1H), 4.53 (d, \(J = 8.0\) Hz, 1H), 3.80 (s, 3H), 3.79-3.61 (m, 1H), 1.93 (dd, \(J = 12.8, 3.6\) Hz, 2H), 1.70-1.64 (m, 3H), 1.39-1.32 (m, 2H), 1.15-1.05 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 157.03, 155.84, 130.92, 125.02, 114.64, 55.49, 48.99, 33.93, 33.68, 25.58, 25.55, 24.87; HRMS (ESI): Calc’d for [C\(_{14}\)H\(_{21}\)N\(_2\)O\(_2\)]\(^{+}\) [M+H]\(^{+}\) 249.1598, found 249.1606.

1-cyclohexyl-3-(3,4,5-trimethoxyphenyl)urea (5f): The title compound was prepared according to the general procedure C to obtain as a dark green (154 mg, yield = 50%); Mp. 215-217 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.58 (s, 2H), 6.38 (s, 1H), 4.74 (d, \(J = 7.6\) Hz,
1H, 3.82 (s, 6H), 3.81 (s, 3H), 3.69-3.62 (m, 1H), 1.97-1.94 (m, 2H), 1.69 (dt, J = 9.6, 3.6 Hz, 2H), 1.42-1.32 (m, 3H), 1.17-1.07 (m, 3H); 13C NMR (100 MHz, CDCl3) δ 155.04, 153.49, 134.74, 98.69, 60.95, 56.05, 49.03, 33.68, 25.52, 24.88; HRMS (ESI): Calc’d for [C16H25N2O4]+ [M+H]+ 309.1809, found 309.1815.

1-(3-chlorophenyl)-3-cyclohexylurea (5g): The title compound was prepared according to the general procedure C to obtain as a white solid (175 mg, yield = 69%); Mp. 161–163 °C; 1H NMR (400 MHz, CDCl3) δ 7.40 (t, J = 2.0 Hz, 1H), 7.22-7.18 (m, 2H), 7.02 (dt, J = 7.2, 2.0 Hz, 1H), 6.37 (s, 1H), 4.63 (d, J = 7.6 Hz, 1H), 3.67-3.61 (m, 1H), 1.98 (dd, J = 12.4, 3.6 Hz, 2H), 1.72 (dt, J = 8.0, 4.0 Hz, 2H), 1.64 (s, 1H), 1.43-1.32 (m, 2H), 1.18-1.12 (m, 3H); 13C NMR (100 MHz, CDCl3) δ 154.24, 140.07, 134.74, 130.07, 123.25, 120.07, 117.97, 49.22, 33.63, 25.50, 24.85; HRMS (ESI): Calc’d for [C13H18ClN2O]+ [M+H]+ 253.1102, found 253.1099.

1-(4-chlorophenyl)-3-cyclohexylurea (5h): The title compound was prepared according to the general procedure C to obtain as a white crystal solid (150 mg, yield = 59%); Mp. 185.1-187 °C; 1H NMR (400 MHz, CDCl3) δ 7.25 (s, 4H), 6.39 (s, 1H), 4.58 (d, J = 7.8 Hz, 1H), 3.65-3.62 (m, 1H), 1.97 (dd, J = 12.4, 3.6 Hz, 2H), 1.71 (dt, J = 7.6, 4.0 Hz, 2H), 1.38-1.35 (m, 3H), 1.18-1.11 (m, 3H); 13C NMR (100 MHz, CDCl3) δ 137.30, 129.18, 121.76, 49.20, 33.66, 29.69, 25.50, 24.85; HRMS (ESI): Calc’d for [C13H18ClN2O]+ [M+H]+ 253.1102, found 253.1108.

1-(4-bromophenyl)-3-cyclohexylurea (5i): The title compound was prepared according to the general procedure C to obtain as a white solid (185 mg, yield = 62%); Mp. 241-242 °C; 1H NMR (400 MHz, CDCl3) δ 8.03 (s, 1H), 7.31-7.28 (m, 4H), 5.69 (d, J = 7.6 Hz, 1H), 3.64-3.57 (m, 1H), 1.92 (dd, J = 12.4, 3.2 Hz, 2H), 1.70 (dt, J = 8.0, 4.0 Hz, 2H), 1.61-1.58 (m, 1H), 1.42-1.31 (m, 2H), 1.20-1.10 (m, 3H); 13C NMR (100 MHz, CDCl3) δ 154.56, 139.21, 131.00, 119.19, 112.78, 47.76, 33.18, 25.18, 24.33; HRMS (ESI): Calc’d for [C13H18BrN2O]+ [M+H]+ 297.0597, found 297.0600.
1-cyclohexyl-3-ethylurea (5j): The title compound was prepared according to the general procedure C to obtain as a yellow solid (110 mg, yield = 64%); Mp. 112–114 °C; \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 4.24 (s, 1H), 3.49 (s, 1H), 3.19 (q, \(J = 7.2\) Hz, 2H), 1.93 (dd, \(J = 12.4, 3.2\) Hz, 2H), 1.70 (dt, \(J = 7.6, 3.6\) Hz, 2H), 1.60 (dt, \(J = 7.6, 3.6\) Hz, 1H), 1.45-1.29 (m, 3H), 1.17-1.09 (m, 5H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 157.44, 49.16, 35.38, 33.93, 25.56, 24.93, 15.42; HRMS (ESI): Calc’d for [C\(_9\)H\(_{19}\)N\(_2\)O\(^+\)] [M+H\(^+\)] 171.14919, found 171.14925.

3-cyclohexyl-1-methyl-1-phenylurea (5k): The title compound was prepared according to the general procedure C to obtain as a yellow solid (155 mg, yield = 66%); Mp. 72.2–72.7 °C; \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.43–7.39 (m, 2H), 7.31–7.22 (m, 3H), 4.20 (d, \(J = 7.2\) Hz, 1H), 3.64–3.61 (m, 1H), 3.26 (s, 3H), 1.85 (dd, \(J = 12.8, 4.0\) Hz, 2H), 1.61–1.58 (m, 2H), 1.37–1.30 (m, 2H), 1.05-0.92 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 156.56, 143.61, 129.88, 127.15, 126.98, 49.27, 37.00, 33.60, 25.53, 24.82; HRMS (ESI): Calc’d for [C\(_{14}\)H\(_{21}\)N\(_2\)O\(^+\)] [M+H\(^+\)] 233.1648, found 233.1653.

1-(tert-butyl)-3-phenylurea (5l): The title compound was prepared according to the general procedure C to obtain as a yellow solid (80 mg, yield = 41%); Mp. 150-152 °C; \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.30–7.24 (m, 4H), 7.07–7.02 (m, 1H), 6.39 (bs, 1H), 4.79 (bs, 1H), 1.36 (s, 9H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 154.85, 138.88, 129.20, 123.48, 120.77, 50.75, 29.31; HRMS (ESI): Calc’d for [C\(_{11}\)H\(_{17}\)N\(_2\)O\(^+\)] [M+H\(^+\)] 193.13351, found 193.13354.

1,3-diphenylurea (5l’): The title compound was prepared according to the general procedure C to obtain as a yellow solid (46 mg, yield = 20%); Mp. 236–237 °C; \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.21 (s, 2H), 7.46-7.43 (m, 4H), 7.28-7.24 (m, 4H), 6.98 (t, \(J = 7.6\) Hz, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 152.66, 139.09, 128.33, 121.69, 118.16; HRMS (ESI): Calc’d for [C\(_{13}\)H\(_{13}\)N\(_2\)O\(^+\)] [M+H\(^+\)] 213.10624, found 213.10630.

1-phenyl-3-(p-tolyl)urea (5m): The title compound was prepared according to the general procedure C to obtain as a yellow solid (155 mg, yield = 66%); Mp. 218-220 °C; \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.04 (d, \(J = 7.6\) Hz, 2H), 7.44 (dd, \(J = 8.4, 2.8\) Hz, 2H), 7.32 (dd, \(J = 8.5, 2.7\) Hz, 2H), 7.28-7.23 (m, 2H), 7.07 (dd, \(J = 8.8, 2.8\) Hz, 2H), 7.00-6.95 (m, 1H), 2.28 (s, 3H); \(^{13}\)C
NMR (100 MHz, CDCl$_3$) $\delta$ 153.15, 139.33, 136.62, 131.35, 129.04, 128.53, 121.86, 118.69, 118.43, 20.43; HRMS (ESI): Calc’d for [C$_{14}$H$_{14}$N$_2$O$\text{Na}$]$^+$ [M+Na]$^+$ 249.0998, found 249.0995.

1-(4-methoxyphenyl)-3-phenylurea (5n): The title compound was prepared according to the general procedure C to obtain as a lite grey solid (150 mg, yield = 61%); Mp. 185-187 ºC; $^1$H NMR (400 MHz, CDCl$_3$+DMSO-d$_6$) $\delta$ 8.07 (s, 1H), 7.97 (s, 1H), 7.42 (d, $J$ = 7.6 Hz, 2H), 7.34-7.30 (m, 2H), 7.25 (t, $J$ = 7.6 Hz, 2H), 6.97 (t, $J$ = 7.4 Hz, 1H), 6.84-6.80 (m, 2H), 3.77 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$+DMSO-d$_6$) $\delta$ 154.88, 153.23, 139.27, 132.10, 128.45, 121.77, 120.62, 118.38, 113.74, 55.12; HRMS (ESI): Calc’d for [C$_{14}$H$_{15}$N$_2$O$\text{Na}$]$^+$ [M+Na]$^+$ 249.0995, found 249.0995.

1-(2,6-dimethylphenyl)-3-phenylurea (5o): The title compound was prepared according to the general procedure C to obtain as a white solid in (140 mg, yield = 56%); Mp. 231.1-232.4 ºC; $^1$H NMR (400 MHz, CDCl$_3$+DMSO-d$_6$) $\delta$ 7.40 (d, $J$ = 7.6 Hz, 2H), 7.26 (t, $J$ = 7.6 Hz, 2H), 7.10 (s, 3H), 6.99 (t, $J$ = 7.2 Hz, 1H), 2.31 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$+DMSO-d$_6$) $\delta$ 153.93, 136.36, 128.69, 128.18, 126.90, 122.36, 118.97, 18.32; HRMS (ESI): Calc’d for [C$_{15}$H$_{17}$N$_2$O$\text{Na}$]$^+$ [M+Na]$^+$ 243.1133, found 243.1133.

1-allyl-3-(2,6-dimethylphenyl)urea (5p): The title compound was prepared according to the general procedure C to obtain as a lite yellow solid (135 mg, yield = 66%); Mp. 189-190.5 ºC; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.16-7.10 (m, 3H), 5.85-5.77 (m, 2H), 5.07 (ddd, $J$= 16.8, 15.6, 1.6 Hz, 2H), 4.35 (s, 1H), 3.83 (t, $J$ = 5.6 Hz, 2H), 2.30 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 156.58, 137.37, 135.24, 133.75, 128.77, 127.98, 115.50, 42.59, 18.27; HRMS (ESI): Calc’d for [C$_{12}$H$_{17}$N$_2$O$\text{Na}$]$^+$ [M+Na]$^+$ 205.1335, found 205.1328.

1-(2,6-dimethylphenyl)-3-(prop-2-yn-1-yl)urea (5q): The title compound was prepared according to the general procedure C to obtain as a lite yellow solid (135 mg, yield = 67%); Mp. 204-205 ºC; $^1$H NMR (400 MHz, CDCl$_3$+DMSO-d$_6$) $\delta$ 7.12 (s, 1H), 7.04 (s, 3H), 5.93 (s, 1H), 3.98 (dd, $J$ = 5.6, 2.8 Hz, 2H), 2.30 (t, $J$ = 2.4 Hz, 1H), 2.25 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$+DMSO-d$_6$) $\delta$ 155.61, 135.64, 127.47, 125.92, 113.32, 80.82, 70.30, 29.06, 17.86; HRMS (ESI): Calc’d for [C$_{12}$H$_{15}$N$_2$O$\text{Na}$]$^+$ [M+Na]$^+$ 203.1179, found 203.1185.
Benzyl cyclohexylcarbamate (7a): The title compound was prepared according to the general procedure D to obtain as a white solid (170 mg, yield = 72%); Mp. 84.3-85.8 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.46-7.28 (m, 5H), 5.09 (s, 2H), 4.64 (bs, 1H), 3.51 (bs, 1H), 1.58 (s, 1H), 1.94 (dt, \(J\) = 12.4, 2.8 Hz, 2H), 1.71 (dt, \(J\) = 7.6, 3.6 Hz, 2H), 1.40-1.30 (m, 2H), 1.21-1.09 (m, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 155.50, 136.67, 128.48, 128.11, 128.03, 66.45, 49.86, 33.39, 25.46, 24.74; HRMS (ESI): Calc’d for [C\(_{14}\)H\(_{19}\)NO\(_2\)Na]+ [M+Na]+ 256.13080, found 256.13089.

4-bromophenethyl cyclohexylcarbamate (7b): The title compound was prepared according to the general procedure D to obtain as a white solid (265 mg, yield = 80%); Mp. 145.5-147.5 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.42 (d, \(J\) = 8.4 Hz, 2H), 7.10 (d, \(J\) = 8.4 Hz, 2H), 4.53 (s, 1H), 4.24 (t, \(J\) = 6.8 Hz, 2H), 3.49-3.45 (s, 1H), 2.88 (t, \(J\) = 6.6 Hz, 2H), 1.92-1.20 (m, 2H), 1.69 (dt, \(J\) = 7.6, 3.6 Hz, 3H), 1.69 (dt, \(J\) = 7.6, 3.6 Hz, 1H), 1.35-1.32(m, 1H), 1.17-1.09 (m, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 155.49, 137.12, 131.46, 130.62, 120.27, 34.95, 33.38, 29.67, 25.44, 24.76; HRMS (ESI): Calc’d for [C\(_{15}\)H\(_{21}\)BrNO\(_2\)]+ [M+H]+ 326.0750, found 326.0755.

Butyl cyclohexylcarbamate (7c): The title compound was prepared according to the general procedure D to obtain as a yellow solid (80 mg, yield = 40%); Mp. 105.6-107.7 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 4.44 (s, 1H), 3.97 (s, 2H), 3.40 (s, 1H), 1.87-1.84 (m, 2H), 1.62 (dt, \(J\) = 7.6, 3.6 Hz, 3H), 1.58-1.46 (m, 4H), 1.34-1.25 (m, 4H), 1.11-1.03 (m, 3H), 0.86 (t, \(J\) = 7.4 Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 64.45, 33.48, 25.51, 24.79, 19.11, 13.76; HRMS (ESI): Calc’d for [C\(_{11}\)H\(_{22}\)NO\(_2\)]+ [M+H]+ 200.1645, found 200.1644.

Hex-5-yn-1-yl cyclohexylcarbamate (7d): The title compound was prepared according to the general procedure D to obtain as a white solid (150 mg, yield = 67%); Mp. 59.5-61.5 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 4.53 (s, 1H), 4.06 (t, \(J\) = 6 Hz, 2H), 3.46 (s, 1H), 2.23 (td, \(J\) = 7.2, 2.8 Hz, 2H), 2.00-1.85 (m, 3H), 1.78-1.66 (m, 4H), 1.64-1.56 (m, 3H), 1.42-1.29 (m, 2H), 1.22-1.07 (m, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 155.77, 84.00, 68.57, 63.99, 49.72, 33.45, 28.10, 25.48, 24.93, 24.77, 18.09; HRMS (ESI): Calc’d for [C\(_{13}\)H\(_{22}\)NO\(_2\)]+ [M+H]+ 224.1645, found 224.1647.

S13
Benzyl tert-butylcarbamate (7e): The title compound was prepared according to the general procedure D to obtain as a brown oil (130 mg, yield = 62%); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.37-7.28 (m, 5H), 5.05 (s, 2H), 4.73 (bs, 1H), 1.33 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 136.70, 130.03, 128.46, 128.01, 127.96, 65.95, 50.37, 29.65, 28.90; HRMS (ESI): Calc’d for [C$_{12}$H$_{18}$NO$_2$]+ [M+H]$^+$ 208.1332, found 208.1336.

4-methoxybenzyl tert-butylcarbamate (7f): The title compound was prepared according to the general procedure D to obtain as a colorless oil (143 mg, yield = 60%); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.29 (d, $J = 8.8$ Hz, 2H), 6.88 (d, $J = 8.8$ Hz, 2H), 4.97 (s, 2H), 4.67 (bs, 1H), 3.80 (s, 3H), 1.31 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 159.44, 129.87, 128.82, 113.88, 65.77, 55.25, 50.33, 29.68, 28.91; HRMS (ESI): Calc’d for [C$_{13}$H$_{19}$NO$_3$Na]$^+$ [M+Na]$^+$ 260.1257, found 260.1260.

4-methoxyphenethyl tert-butylcarbamate (7g): The title compound was prepared according to the general procedure D to obtain as a colorless oil (147 mg, yield = 58%); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.15 (d, $J = 8.4$ Hz, 2H), 6.85 (d, $J = 8.8$ Hz, 2H), 4.61 (bs, 1H), 4.19 (s, 2H), 3.79 (s, 3H), 2.86 (t, $J = 7.0$ Hz, 2H), 1.30 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 158.17, 130.14, 129.81, 113.83, 64.75, 55.22, 50.24, 34.66, 29.68, 28.93; HRMS (ESI): Calc’d for [C$_{14}$H$_{21}$NO$_3$Na]$^+$ [M+Na]$^+$ 274.14136, found 274.14140.

Cyclohexylcarbamic acid (10): The title compound obtain white solid (50 mg, yield = 69%); MP. 182-184 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.09 (d, $J = 7.2$ Hz, 1H), 3.51 – 3.44 (m, 1H), 1.93 (dd, $J = 12.4$, 3.6 Hz, 2H), 1.69 (dt, $J = 7.6$, 3.6 Hz, 2H), 1.60-1.57 (m, 1H), 1.40 – 1.29 (m, 2H), 1.19 – 1.07 (m, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 156.71, 49.15, 33.94, 25.61, 24.93; HRMS (ESI): Calc’d for [C$_7$H$_{13}$NO$_2$]+ [M]+ 143.0941, found 143.1177.

N-phenylacetamide (A): The title compound obtain white solid (25 mg, yield = 25%); MP. 114-116 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.50 (d, $J = 8.0$ Hz, 2H), 7.37 (s, 1H), 7.31 (t, $J = 6.8$ Hz, 2H), 7.10 (t, $J = 7.2$ Hz, 1H), 2.17 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 168.33, 137.85, 128.96, 124.28, 119.86, 24.57; HRMS (ESI): Calc’d for [C$_8$H$_{10}$NO]+ [M+H]$^+$ 136.0757, found 136.0759
## Results and Discussions

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Optimization studies

Table 1 Screening of the reactions conditions.a

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\[ ^a \text{Reaction conditions: 1a (1.0 mmol), 2a (1.2 mmol), [I] source (20 mol %), oxidant (2.0 mmol) in solvent (0.25 M) at 90 °C for 4 h unless otherwise noted. } ^b \text{Isolated yields. } ^c \text{5.5M TBHP in decane was used. } ^d \text{H}_2\text{O}_2\; 30\% \text{ wt in H}_2\text{O. } ^e \text{Isocyanide was added in two portions with an time interval of 1 h; the first portion (0.6 mmol) was added at rt and second portion (0.6 mmol) was added at 90 °C. TBHP, tertiary butyl hydroperoxide. DTBP, ditertbutyl hydroperoxide. CHP, cumene hydroperoxide. H}_2\text{O}_2\text{, dihydrogen peroxide. K}_2\text{S}_2\text{O}_8\text{, potassium persulfate. Aq. TBHP (70% in H}_2\text{O).} \]
References

Copies of $^1$H, $^{13}$C and DEPT NMR Spectra
Solvent: CDCl₃
Spectrometer Frequency: 100.69 MHz

**Diagram 1:**

**Diagram 2:**

**Diagram 3:**

MR B1-13
Pulse Sequence: SOFT
Solvent: CDCl₃
Spectrometer Frequency: 400.13 MHz

**NMR Spectra**

**Top Spectra**
- Resonance peaks at various ppm values
- Assignments for different chemical shifts

**Bottom Spectra**
- Resonance peaks at various ppm values
- Assignments for different chemical shifts

*Diagram 7g*