RSC Advances

PAPER

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Cite this: *RSC Adv.*, 2026, **16**, 29603

Lanthanum triflate catalysed acyl transfer from β-diketones to amines: conversion of aryl methyl ketones to amides

Rajendra Prasad, Purnima Pradhan and Partha Ghosh *

Received 30th April 2026
Accepted 21st May 2026

DOI: 10.1039/d6ra03708a

rsc.li/rsc-advances

Lanthanum triflate was found to be an efficient catalyst for the amide formation through acyl transfer from β-diketones to amines overcoming the stable enamine formation pathway. The non-oxidative, C–C cleavage mediated catalytic amidation proceeds under neat or low-solvent conditions and was found to have a broad substrate scope with respect to both amine and β-diketone. Selective acyl transfer from unsymmetrical diketone 2-benzoyl-1-indanone to amine enabled the two-step conversion of aryl methyl ketones to amides.

Introduction

The amide bond, which is prevalent in biomolecules, is also widespread in synthetic polymers, pharmaceuticals, and agrochemicals due to its desirable properties such as stability, polarity, conformation, *etc.*[1,2] Consequently, the amidation reaction is a frequently performed transformation in organic synthesis and remains the most executed transformation in medicinal chemistry.[3,4] Conventionally, amides are synthesized by acylation of amines with carboxylic acids either with the help of stoichiometric coupling reagents or through activated derivatives such as acid chloride, anhydride, *etc.*[5] However, given the importance of the amidation reaction in synthesis, the development of alternative amidation methods that broaden the scope of acyl source remains an active area of research.[6–9] In the past few years', novel methods for the acylation of amines with unactivated esters and amides have been reported where acyl transfer proceeds through C–O and C–N bond cleavage, respectively.[10–17] Reactions involving C–C bond cleavage have also gained significant attention in recent years, which includes C(CO)–C cleavage induced amide synthesis from ketones, a functional group found frequently in organic molecules and feedstock chemicals.[18–22]

For the acyl transfer from ketone to amine to form amide, there are several reported methods which can broadly be categorised into two groups. Direct acyl transfer from ketones to amine through oxidative cleavage of C(CO)–C bond has been achieved, which requires a strong oxidant such as molecular oxygen.[23–27] The other strategy is to activate the α position of ketone with electron withdrawing group such as nitro, azide, halide, cyano, oxime, acyl, *etc.* to facilitate acyl transfer.[28–31]

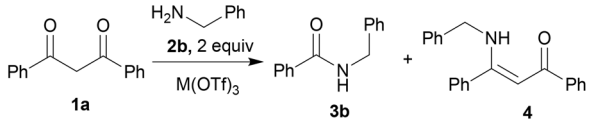
One of the common methods among the latter strategy for amidation is acyl transfer from readily available 1, 3-diketones (β-diketones).[32,33] Both oxidative and non-oxidative methods for the acyl transfer from 1,3-diketones to form amide have been reported.[34–39] However, due to the potential of amine oxidation, the oxidative methods reported are mainly limited to aromatic amines.[40] On the other hand, non-oxidative methods being acid catalysed are limited to aliphatic 1,3-diketones as aromatic β-diketones are prone to form stable enamine on reaction with amines (other than secondary amines).[41–46] In fact, aromatic acyl transfer from β-diketones to form amide is seldom reported albeit with poor yield, except when masked amines such as *N*-acyl urea or carboxamide were used.[47–49] Therefore, catalytic amidation methods with broad substrate scope with respect to both β-diketones and amines would be very useful.

For the past few decades metal triflates, for their high Lewis acidity, have found extensive use as catalysts in various organic transformations which otherwise require stoichiometric amounts of catalyst.[50] In particular, the lanthanide triflates [Ln(OTf)$_3$], owing to their stability, high catalytic activity, water compatibility and recoverability have emerged as favoured green catalysts for various transformations, including acylation.[51,52] We wondered whether metal triflates with their high catalytic ability would enable the acyl transfer from β-diketones to amines, overcoming the enamine formation. As shown in Scheme 2, while the reaction of readily available dibenzoyl methane **1a** with piperidine yields exclusively the secondary amide **3a**, with benzyl amine **2a** the reaction resulted mainly in the formation of enamine **4** with a low yield of amide **3b**. However, when the reaction of **1a** with amine **2b** was performed with scandium triflate as a catalyst (10 mol%), the desired amide **3b** and enamine **4** were isolated in almost equal amounts.

With the promising result, we embarked on optimizing the reaction condition while simultaneously screening several

Department of Chemistry, Central University of Jharkhand, Cheri-Manatu, Ranchi-835222, India. E-mail: partha.ghosh@cuj.ac.in

Table 1 Reaction optimization and screening of metal triflate catalyst



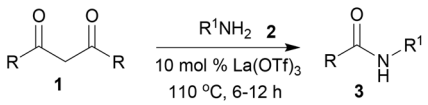
Sl no.	Catalyst (10 mol%)	Temperature, time	Yield[a] 3b	4
1[b]	$Sc(OTf)_3$	110 °C, 8 h	46%	50%
2[b]	$Y(OTf)_3$		50%	45%
3[b]	$La(OTf)_3$		70%	25%
4[b]	$Gd(OTf)_3$		45%	
5[b]	$In(OTf)_3$		38%	
6[b]	$Fe(OTf)_3$		42%	
7[b]	$Al(OTf)_3$		25%	
8[b]	TfOH		10%	85%
9[b]	$La(OTf)_3$ (20 mol%)	110 °C, 6 h	77%	20%
10[c]	$La(OTf)_3$	110 °C, 8 h	86%	10%
11[c]	$La(OTf)_3$	120 °C, 6 h	78%	
12[c]	$La(OTf)_3$	100 °C, 20 h	84%	
13[c,d]	$La(OTf)_3$	110 °C, 8 h	52%	

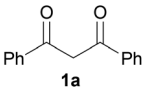
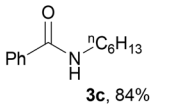
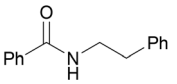
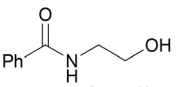
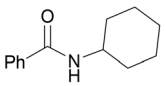
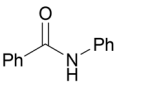
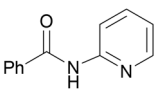
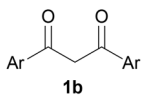
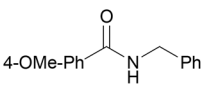
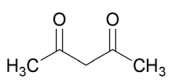
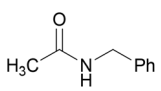
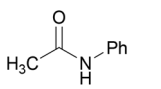
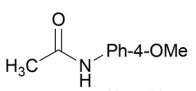
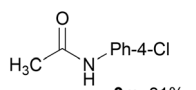
[a] Yields shown are isolated yields, which for the main byproduct **4** (all entries) shown only when isolated. [b] 1.0 mmol of **1a** reacted with 2.0 mmol of **2a** without solvent. [c] 1.0 mmol **1a** in 1.0 mL toluene was added slowly for 4 h to the mixture of **2b** and catalyst. [d] Only 1.2 equivalent amine was used.

metal triflates, results of which are summarized in Table 1. It was observed that while triflic acid as a catalyst promotes even more enamine **4** formation (entry 8), the metal triflates $Y(OTf)_3$, $La(OTf)_3$, $Gd(OTf)_3$, $In(OTf)_3$, $Fe(OTf)_3$, $Al(OTf)_3$ were also able to promote acyl transfer from the β-diketone **1a** to amine to form amide **3b** but with varying efficiency as significant amount of enamine **4** was also isolated (entry 1 to 7). However, among them, the $La(OTf)_3$ catalysed reaction stood out giving the best yield of amide **3b** (entry 3), which further increased with a higher 20 mol% catalyst load (entry 9). The later observation prompted us to perform the reaction with the slow addition of diketone **1a** to the mixture of amine and catalyst that effectively increases the catalyst concentration. To our pleasure the inverse addition method with only 10% catalyst gave the desired amide in good yield with minimal enamine formation (entry 10). Raising the reaction temperature resulted in a decreased yield of amide **3b** (entry 11), while lower temperatures only prolonged the reaction time without any significant change in the amide yield (entry 12). It was also observed that lesser than two equivalent of benzyl amine resulted in an incomplete reaction with a decrease in amide **3b** yield, probably as amine also forms enamine with the byproduct acetophenone (not isolated). Therefore, to get an optimum yield of amide, at least 2.0 equivalents of amine was found necessary.

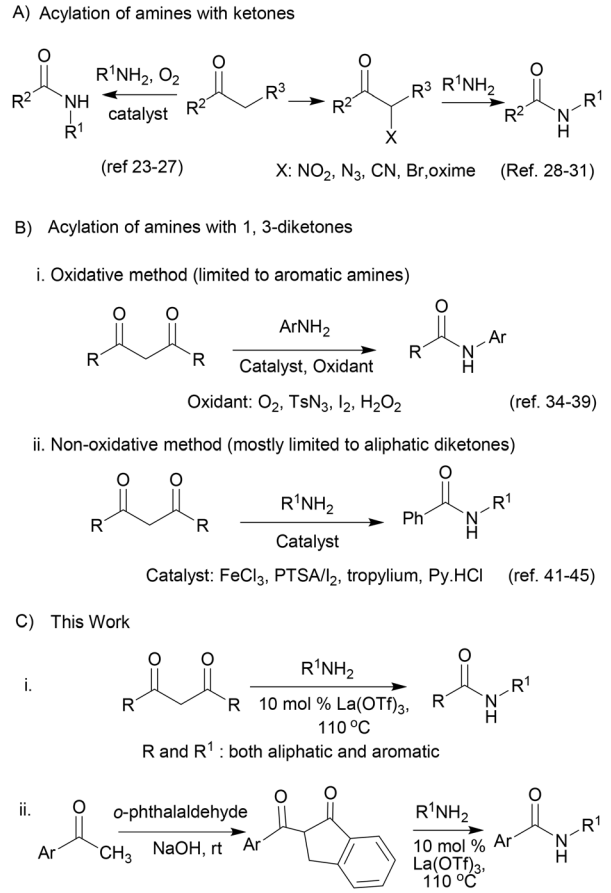
The scope of the catalytic acyl transfer from β-diketones was then examined with various primary amines (Table 2), as the amidation with secondary amines proceeds without a catalyst (Scheme 1). Amidation with other primary amines, including ethanolamine, proceeded efficiently with good yields of amides

Table 2 Substrate scope of $La(OTf)_3$ catalysed acyl transfer from β-diketones to amine



Entry	1,3-Diketone **1**	Amine **2**	Amide **3**, yield[a,b,c]
1	 **1a**	n-$C_6H_{13}NH_2$, **2c**	 **3c**, 84%
2		$PhCH_2CH_2NH_2$, **2d**	 **3d**, 84%
3		$HOCH_2CH_2NH_2$, **2e**	 **3e**, 82%
4		$CyNH_2$, **2f**	 **3f**, 78%
5		$PhNH_2$, **2g**	 **3g**, 35%
6		2-Aminopyridine, **2h**	 **3h**, 85%
7	 **1b** Ar: 4-OMe-Ph	**2b**	 **3i**, 84%
8	 **1c**	**2b**	 **3j**, 72%
9		$PhNH_2$, **2i**	 **3k**, 88%
10		4-OMe-$PhNH_2$, **2j**	 **3l**, 86%
11		4-Cl-$PhNH_2$, **2k**	 **3m**, 81%

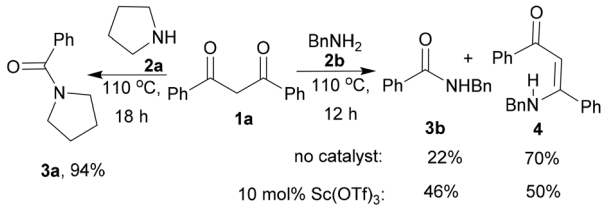
[a] Yields shown are isolated yields. [b] Reactions were performed with slow addition of 1.0 mmol of **1** (**1a**, **1b** as solution in 1.0 mL toluene while **1c** directly) on the mixture of amine and catalyst. [c] Reaction temperature 120 °C.



Scheme 1 Amidation through C–C cleavage induced acyl transfer from ketones to amines.

3c, **3d** and **3e** (entries 1 to 3). However, the reaction with bulky cyclohexylamine gave only a modest yield of amide **3f** (entry 4). Efficient acyl transfer to amine was also observed from bis(4-methoxybenzoyl) methane **1b** to the benzyl amine resulting in the formation of amide **3i** (entry 8). In case of aromatic amines while reaction with 2-amino pyridine gave amide **3h** in good yield 9 (entry 6), with aniline poor yield of amide **3g** was obtained (entry 5) probably due to the formation of enamine byproduct (not isolated). When aliphatic acetyl acetone was used as acyl source, the catalytic acyl transfer to benzylamine gave the modest yield of amide **3j**, whereas with aromatic amines (electron-rich and -poor), formation of the amides **3k**, **3l** and **3m** resulted in high yields (entries 9 to 12). Thus, amide synthesis through the lanthanum triflate catalysed acyl transfer reaction was found to have a broader substrate scope with respect to both β-diketones and amines. The other significant advantage of the non-oxidative acylation reaction is that it proceeds under solvent-free or in minimal-solvent conditions.[53–56]

The applicability of the catalytic amidation method can be further extended for the acyl transfer to amines from ketones by first converting them to β-diketones. Indeed, under mild conditions, aryl methyl ketones can be converted to beta diketones that we have previously employed for selective acyl

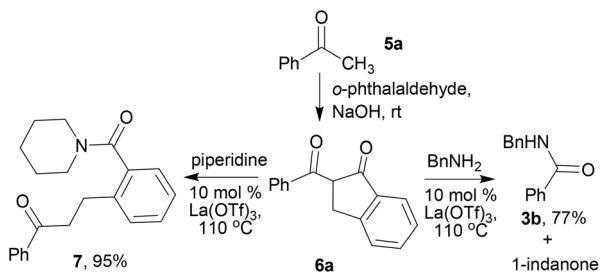


Scheme 2 Acyl transfer to amine *vs.* enamine formation.

transfer to alcohols for the synthesis of ester.[57,58] We wondered whether a similar strategy could be used for the conversion of aryl methyl ketones to amide (Scheme 3).

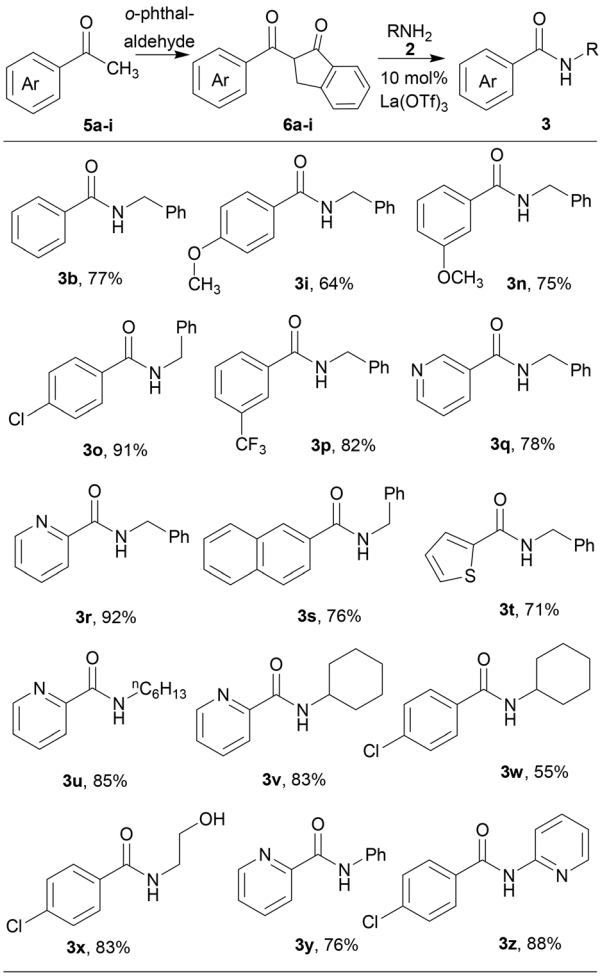
However, the regioselectivity of the acyl transfer from the unsymmetrical diketone such as **6a** derived from acetophenone **5a** is crucial for the desired outcome. When the catalytic amidation was performed with piperidine, the ring opened amide **7** was obtained as the sole product, an outcome previously reported under Brønsted acid catalysis.[59] On the contrary, and to our pleasure, reversal of selectivity was observed on reaction of **6a** with primary amine **2b** giving the desired amide **3b**.

To assess the scope of the above ketone to amide transformation, several aryl and heteroaryl methyl ketones were first converted to beta diketones before catalytic acyl transfer to various primary amines, the results of which are summarized in Table 3. The acyl transfers from diketone **6a** to benzylamine and hexylamine were found to be modestly selective resulting in amides **3b** and **3i** respectively. The selectivity of acyl transfer improved with electron-deficient aromatic methyl ketones resulting in amides **3n**, **3o** and **3p** in good yields whereas in the case of electron-donating group in the aryl ring, only modest yield of amide **3i** obtained. Efficient and selective acyl transfer was also observed with heteroaromatic picolinoyl methyl ketone to benzyl, *n*-hexyl, cyclohexyl amine, as well as to aromatic aniline resulting in the formation of amides **5r**, **5u**, **5v** and **5y** respectively, in good to excellent yield. However, transfer of the nicotinoyl group was found to be less selective, which was also the case with the napthoyl and 2-thiophene carbonyl groups, giving the amides **3q**, **3s** and **3t** respectively, in modest yield. The efficient transfer of the picolinoyl group from ketones to the bulky cyclohexyl amine to give amide **3v** in good yields is noteworthy in comparison to the modest yield of cyclohexyl amide **3w**. The amidation was also proceeded well with



Scheme 3 Chemoselectivity of acyl transfer in unsymmetrical 1, 3-diketone.

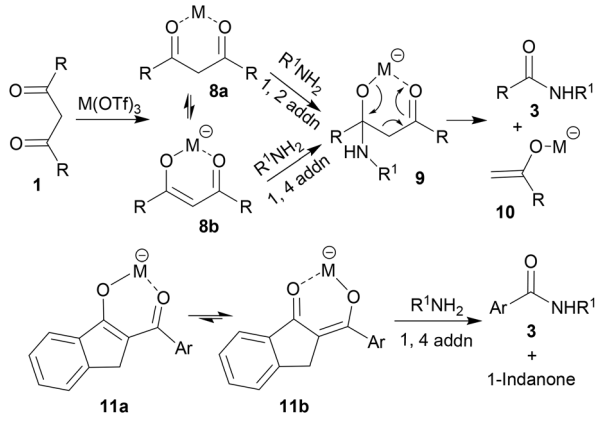
Table 3 Aryl methyl ketones to amide[a]



[a] # Reaction condition: diketones 6 (1.0 mmol in 1 mL toluene) added slowly to the mix of amines (2.0 mmol) and catalyst (0.1 mmol) for 4 hours at 110 °C, then heated additional 2 to 8 hours. ## Yields shown are for the conversion of diketone 6 to amide 3. Yields for **5a–i** to **6a–i** are > 90% (see Experimental).

ethanolamine resulting in amide **3x**. However, the acyl transfer from ketones other than picolinoyl ketones to aromatic amines was found to be less efficient; instead, mostly enamine (not isolated) formation was observed, except in the case of 2-aminopyridine in which an excellent yield of amide **3z** was obtained.

The two key observations in the present study, *i.e.* the promotion of C–C bond cleavage induced acyl transfer by metal triflates and it's selectivity in unsymmetrical diketones might be explained through a plausible mechanism shown in Scheme 4. Thus, the diketone **1** initially forms chelate **8a** and its enolate form **8b** both of are activated for the addition of amine to form hemiaminal intermediate **9** through 1, 2 and 1,4 addition respectively. The Lewis acidic metal further facilitates the cleavage of beta ketone (C–C cleavage) as enolate **10** from the hemiaminal intermediate **9** yielding the desired amide **3**. The unsymmetrical diketones are known to exists predominantly in exocyclic enol form (X-ray)[57] and in presence of metal triflate, chelate **11b** (preferentially over **11a**), to which 1, 4 addition of primary amine followed by cleavage of 1-indanone gives the desired amide **3**. On the other hand, with more nucleophilic piperidine (secondary amine reacts without catalyst, Scheme 2),



Scheme 4 Plausible mechanism.

the kinetically controlled 1, 2 addition to the **11b** followed by C–C cleavage gives the ring opened amide **7** as observed in Scheme 3.

Conclusions

Amide synthesis through non-oxidative acyl transfer to amine from β-diketones was found to be efficiently catalysed by lanthanum triflate under solvent-free or minimal-solvent conditions. The substrate scope of the catalytic acylation of amine was found to be broader than existing methods that are either oxidative and thus restricted to aromatic amines or limited to aliphatic diketones. The method also enables a two-step conversion of aromatic methyl ketones to amides through chemoselective acyl transfer from unsymmetrical β-diketone intermediate.

Experimental section

General information

All reagents and chemicals were purchased from commercial sources and were used as such without further purification. All reactions were performed in flame dried glassware. Reactions were monitored by thin layer chromatography (TLC) performed on 0.25 mm silica gel coated aluminium sheets which was visualized under UV (254 nm) light. To purify the compounds, silica gel (100–200 mesh) column chromatography was performed using hexane and ethyl acetate as eluent. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ on 600 MHz Bruker-AVANCE spectrometers with TMS as internal standard. High-resolution electrospray ionization mass spectra (HRMS-ESI) were obtained with an Agilent 6520 QTOF instrument.

General procedure I

Acyl transfer from aromatic β-diketone (1a and 1b) to amine to form amide (3b–i). In a flame dried round bottom flask with magnetic bar were taken amine **2** (2.00 mmol) and $La(OTf)_3$ (0.1 mmol). The reaction flask was then degassed with argon, sealed with septa and placed in an oil bath heated to 110 °C. Then the β-diketone (1.00 mmol) dissolved in 1.0 mL toluene was added slowly (0.1 mL at once) through syringe to the stirring reaction mixture over 4 hours. The reaction was then additionally heated for 2 to 8 hours and monitored by tlc. Upon completion, the reaction mixture was cooled and directly loaded in silica gel column. Elution with 20 to 50% EtOAc in hexane gave pure amide (**3b–i**), which were analysed by spectroscopic method.

General procedure II

Acyl transfer from acetylacetone 1c to amine to form amide (3j–m). In a flame dried round bottom flask with magnetic bar were taken amine **2** (2.00 mmol) and $La(OTf)_3$ (0.1 mmol). The reaction flask was then degassed with argon, sealed with septa and placed in an oil bath heated to 120 °C. Then the acetyl acetone (0.11 mL, 1.05 mmol) was added dropwise through syringe to the stirring reaction mixture over 4 hours. The reaction was then additionally heated for 4 hours and monitored by tlc. Upon completion, the reaction mixture was cooled and directly loaded in silica gel column. Elution with 20 to 50% EtOAc in hexane gave pure amide (**3j–m**), which were analysed by spectroscopic method.

General procedure III

Conversion of aryl methyl ketones (5a–e) to β-diketones (6a–e). The methyl ketones acetophenone **5a**, 4-methoxy acetophenone **5b**, 3-methoxyacetopenone **5c**, 3-trifluoromethyl acetophenone **5d**, 4-chloroacetophenone **5e**, 3-acyl pyridine **5f**, 2-acyl pyridine **5g**, 2-acyl thiophene **5h**, and 2-acyl naphthalene **5i** were converted to β-diketones **6a**, **6b**, **6c**, **6d**, **6e**, **6f**, **6g**, **6h** and **6i** respectively by following general procedure which is similar to reported procedure.[57,58]

In a solution of sodium hydroxide (90 mg, 2.25 mmol) in ethanol (10 mL) cooled to 0 °C were added *o*-phthalaldehyde (350 mg, 2.61 mmol) and aryl methyl ketone **5** (2.00 mmol) respectively. The reaction was stirred at 0 °C for 1 hour and then slowly warmed to room temperature over 1 hour and monitored by TLC. The reaction mixture was then neutralized with 5% HCl (5.0 mL) and extracted with ethyl acetate ($50 \times 2 = 100$ mL). The combined organic layer was then dried over anhydrous sodium sulphate before evaporated to get crude product which was loaded in silica gel column. Elution with 5 to 20% EtOAc in hexane gave pure β-diketone (**6a–e**).

Conversion of β-diketones 6a–e to amide (3n–z). In a flame dried round bottom flask with magnetic bar were taken amine **2** (2.00 mmol) and $La(OTf)_3$ (0.1 mmol). The reaction flask was then degassed with argon, sealed with septa and placed in an oil bath heated to 110 °C. Then β-diketone **6** (1.00 mmol) dissolved in 1.0 mL toluene (heated when required) was added slowly (0.1 mL at once) through syringe to the stirring reaction mixture over 4 hours. The reaction was then additionally heated for 2 to 8 hours and monitored by tlc. Upon completion, the reaction mixture was cooled and directly loaded in silica gel column. Elution with 20 to 50% EtOAc in hexane gave pure amide (**3n–z**), which were analysed by spectroscopic method.

Spectroscopic data for the isolated products. Phenyl(pyrrolidin-1-yl)methanone (**3a**).[61] White solid. Yield. 165 mg (94%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (2H, d, $J = 6.6$ Hz), 7.36–7.42 (3H, m), 3.65 (2H, t, $J = 6.6$ Hz), 3.42 (2H, t, $J = 6.6$ Hz), 1.92–2.00 (2H, m), 1.84–1.90 (2H, m). ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.7, 137.1, 129.7, 128.2, 127.0, 49.6, 46.1, 26.3, 24.4.

3-(Benzylamino)-1,3-diphenylprop-2-en-1-one **4**.[46] White solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 11.70 (1H, bs),7.90 (2H, d, $J =$ 7.8 Hz), 7.37–7.45 (8H, m), 7.31 (2H, t, $J = 7.8$ Hz), 7.21–7.27 (3H, m), 5.84 (1H, s), 4.42 (2H, d, $J = 6.6$ Hz).

N-Benzylbenzamide (**3b**).[60] White solid. Yield. 182 mg (86%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 (2H, d, $J = 7.2$ Hz), 7.49 (1H, t, $J = 7.2$ Hz), 7.41 (2H, t, $J = 7.2$ Hz), 7.34 (4H, d, $J = 4.2$ Hz), 7.26–7.31 (1H, m), 6.54 (1H, bs), 4.63 (2H, d, $J = 5.4$ Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.3, 138.2, 134.3, 131.5, 128.7, 128.5, 127.9, 127.6, 126.9, 44.1.

N-Hexylbenzamide (Table 2, **3c**).[60] White solid. Yield. 172 mg (84%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (2H, d, $J = 7.8$ Hz), 7.48 (1H, t, $J = 7.2$ Hz), 7.42 (2H, t, $J = 7.2$ Hz), 6.23 (1H, bs), 3.44 (2H,

dd, J = 7.2 Hz, 6.6 Hz), 1.55–1.65(2H, m), 1.25–1.42 (6H, m), 0.89 (3H, t, J = 6.6 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.5, 134.8, 131.2, 128.5, 126.8, 40.1, 31.5, 29.6, 26.6, 22.5, 14.0.

N-Phenethylbenzamide (Table 2, **3d**).[61] White solid. Yield. 190 mg (84%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (2H, d, J = 7.2 Hz), 7.47 (1H, t, J = 7.2 Hz), 7.39 (2H, t, J = 7.2 Hz), 7.32 (2H, t, J = 7.2 Hz), 7.25 (1H, d, J = 7.2 Hz), 7.23 (2H, d, J = 7.8 Hz), 6.27 (1H, bs), 3.71 (2H, q, J = 6.6 Hz), 2.93 (2H, t, J = 6.6 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.4, 138.9, 134.6, 131.4, 128.8, 128.7, 128.5, 126.8, 126.6, 41.1, 35.7.

N-(2-Hydroxyethyl)benzamide (Table 2, **3e**).[61] White solid. Yield. 135 mg (82%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (2H, d, J = 7.2 Hz), 7.46 (1H, t, J = 7.2 Hz), 7.37 (2H, t, J = 7.2 Hz), 7.13 (1H, bs), 4.31 (1H, bs), 3.76 (2H, t, J = 5.4 Hz), 3.56 (2H, dd, J = 5.4 Hz, 6.6 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.7, 134.1, 131.6, 128.6, 127.0, 62.1, 42.8.

N-Cyclohexylbenzamide (Table 2, **3f**).[60] White solid. Yield. 158 mg (78%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (2H, d, J = 7.8 Hz), 7.48 (1H, t, J = 7.2 Hz), 7.41 (2H, t, J = 7.2 Hz), 6.02 (1H, bs), 3.95–4.00 (1H, m), 2.03(2H, d, J = 10.2 Hz), 1.62–1.80 (3H, m), 1.36–1.46 (2H, m), 1.16–1.26 (3H, m). ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.6, 135.0, 131.2, 128.4, 126.8, 48.6, 33.2, 25.5, 24.9.

N-Phenylbenzamide (Table 2, **3g**).[60] White solid. Yield. 70 mg (35%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (1H, bs), 7.86 (2H, d, J = 7.8 Hz), 7.64 (2H, d, J = 7.8 Hz), 7.54 (1H, t, J = 7.2 Hz), 7.47 (2H, t, J = 7.2 Hz), 7.36 (2H, t, J = 7.2 Hz), 7.15 (1H, t, J = 7.8 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.7, 137.9, 135.0, 131.8, 129.1, 128.8, 127.0, 124.6, 120.2.

N-(Pyridin-2-yl)benzamide (Table 2, **3h**).[47] White solid. Yield. 168 mg (85%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.10 (1H, bs), 8.41 (1H, d, J = 8.4 Hz), 8.21 (1H, bs), 7.94 (2H, d, J = 7.2 Hz), 7.59 (1H, t, J = 7.2 Hz), 7.56 (1H, t, J = 7.2 Hz), 7.49 (2H, t, J = 7.2 Hz), 7.05 (1H, t, J = 5.4 Hz), ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.8, 151.6, 147.8, 138.5, 134.3, 132.2, 128.8, 127.2, 119.9, 114.2.

N-Benzyl-4-methoxybenzamide (Table 2, **3i**).[60] White solid. Yield. 202 mg (84%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (2H, d, J = 8.4 Hz), 7.22–7.35 (5H, m), 6.90 (2H, d, J = 8.4 Hz), 6.41 (1H, bs), 4.62 (2H, d, J = 4.8 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.8, 162.2, 138.4, 128.8, 128.7, 127.9, 127.5, 126.6, 113.7, 55.4, 44.0.

N-Benzylacetamide (Table 2, **3j**).[45] White solid. Yield. 107 mg (72%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24–2.36 (5H, m), 5.92 (1H, bs), 4.41 (2H, d, J = 5.4 Hz), 2.00 (3H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.9, 138.2, 128.7, 127.8, 127.5, 43.7, 23.2.

N-Phenylacetamide (Table 2, **3k**).[45] White solid. Yield. 120 mg (88%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (1H, bs), 7.50 (2H, d, J = 7.8 Hz), 7.29 (2H, t, J = 7.8 Hz), 7.09 (1H, t, J = 7.8 Hz), 2.14 (3H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.7, 137.9, 128.9, 124.2, 120.0, 24.4.

N-(4-Methoxyphenyl)acetamide (Table 2, **3l**).[45] White solid. Yield. 207 mg (86%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (1H, bs), 7.38 (2H, d, J = 8.4 Hz), 6.83 (2H, d, J = 8.4 Hz), 3.77 (3H, s), 2.12 (3H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.5, 156.3, 131.0, 122.0, 114.0, 55.4, 24.1.

N-(4-Chlorophenyl)acetamide (Table 2, **3m**).[45] White solid. Yield. 200 mg (81%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.45 (2H, d, J = 8.4 Hz), 7.27 (2H, d, J = 8.4 Hz), 2.17 (3H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.3, 136.4, 129.3, 129.0, 121.0, 24.5.

The spectroscopic data of β-diketones **6a**, **6b**, **6e–h** matched with the reported data of compounds synthesized following similar procedure.[57,58]

2-(3-Methoxybenzoyl)-indan-1-one **6c**. White solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 15.1 (1H, bs), 7.88 (1H, d, J = 7.8 Hz), 7.30–7.60 (6H, m), 7.05 (1H, d, J = 7.8 Hz), 3.91 (2H, s), 3.88 (3H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 195.7, 170.7, 159.7, 148.5, 137.8, 136.1, 133.3, 129.5, 127.4, 125.5, 123.4, 120.6, 117.1, 113.2, 109.5, 55.4, 32.2. HRMS (ESI) m/z calcd for $C_{17}H_{15}O_3$ ($[M + H]^+$), 267.1021, found 267.1013.

2-(3-(Trifluoromethyl)benzoyl)-indan-1-one **6d**. White solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 14.97 (1H, bs), 8.20 (1H, s), 8.12 (1H, d, J = 7.8 Hz), 7.90 (1H, d, J = 7.8 Hz), 7.77 (1H, d, J = 7.8 Hz), 7.50–7.67 (3H, m), 7.46 (1H, d, J = 7.8 Hz), 3.94 (2H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 196.2, 168.6, 148.5, 137.6, 135.7, 133.8, 131.2, 131.1, 129.2, 127.7, 127.6, 125.7, 124.9, 123.6, 109.9, 31.9. HRMS (ESI) m/z calcd for $C_{17}H_{12}F_3O_2$ ($[M + H]^+$), 305.0789, found 305.0792.

3-(Benzylamino)-1,3-diphenylprop-2-en-1-one **4**.[46] White solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 11.70 (1H, bs).

1-Phenyl-3-(2-(piperidine-1-carbonyl)phenyl) propan-1-one **7**.[59] Pale yellow oil. ^{1}H (300 MHz, $CDCl_3$) δ 7.97 (2H, dt, J = 7.2 Hz, 1.5 Hz), 7.36–7.55 (3H, m), 7.15–7.33 (4H, m), 3.60–3.90 (2H, m), 3.45–3.55 (1H, m), 2.85–3.30 (5H, m), 1.58–1.78 (4H, m), 1.42–1.53 (2H, m).

N-Benzyl-3-methoxybenzamide (Table 3, **3n**).[62] White solid. Yield. 180 mg (75%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 (1H, s), 7.27–7.37 (7H, m), 7.03 (1H, d, J = 7.8 Hz), 6.43 (1H, bs), 4.64 (2H, d, J = 5.4 Hz), 3.84 (3H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.2, 159.8, 138.1, 135.8, 129.5, 128.7, 127.8, 127.6, 118.6, 117.7, 112.3, 55.4, 44.1.

N-Benzyl-4-chlorobenzamide (Table 3, **3o**).[60] White solid. Yield. 224 mg (91%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 (2H, d, J = 8.4 Hz), 7.38 (2H, d, J = 7.8 Hz), 7.28–7.36 (5H, m), 6.48 (1H, bs), 4.61 (2H, d, J = 6.0 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.3, 137.9, 137.7,132.7, 128.8, 128.4, 127.9, 44.2.

N-Benzyl-3-(trifluoromethyl)benzamide (Table 3, **3p**).[63] White solid. Yield. 230 mg (82%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 (1H, s), 7.97 (1H, d, J = 7.8 Hz), 7.75 (1H, d, J = 7.8 Hz), 7.56 (1H, t, J = 7.8 Hz), 7.48–7.58 (5H, m), 6.55 (1H, bs), 4.65 (2H, d, J = 6.0 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.9.

N-Benzylnicotinamide (Table 3, **3q**).[60] White solid. Yield. 165 mg (78%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.94 (1H, d, J = 2.4 Hz), 8.65 (1H, dd, J = 4.8 Hz, 1.2 Hz), 8.13 (1H, dt, J = 8.4 Hz, 1.2 Hz), 7.26–7.38 (5H, m), 6.95 (1H, bs), 4.63 (2H, d, J = 5.4 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.5, 152.1, 147.8, 137.7, 135.2, 130.1, 128.8, 127.9, 127.7, 123.5, 44.1.

N-Benzylpicolinamide (Table 3, **3r**).[60] White solid. Yield. 195 mg (92%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.52 (1H, d, J = 4.8 Hz), 8.39 (1H, s), 8.24 (1H, d, J = 7.8 Hz), 7.85 (1H, t, J = 7.8 Hz), 7.42 (1H, t, J = 6.0 Hz), 7.28–7.40 (5H, m), 4.67 (2H, d, J = 10 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.2, 149.8, 148.0, 138.2, 137.3, 128.7, 127.8, 127.4, 126.2, 122.3, 43.5.

N-Benzyl-2-naphthamide (Table 3, **3s**).[62] White solid. Yield. 198 mg (76%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.30 (1H, s), 7.84–7.90 (4H, m), 7.50–7.56 (2H, m), 7.27–7.40 (5H, m), 6.64 (1H, bs), 4.70 (2H, d, J = 6.0 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.4,

138.2, 134.7, 132.6, 131.5, 128.9, 128.8, 128.4, 127.9, 127.7, 127.6, 127.6, 127.4, 126.7, 123.6, 44.2.

N-Benzylthiophene-2-carboxamide (Table 3, **3t**).[61] White solid. Yield. 155 mg (71%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (1H, d, J = 2.4 Hz), 7.47 (1H, d, J = 4.8 Hz), 7.28–7.38 (5H, m), 7.07 (1H, t, J = 3.6 Hz), 6.29 (1H, bs), 4.62 (2H, d, J = 7.8 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.8, 138.7, 138.0, 130.0, 128.7, 128.1, 127.9, 127.6, 127.6, 44.0.

N-Hexylpicolinamide (Table 3, **3u**).[64] White solid. Yield. 175 mg (85%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.55 (1H, d, J = 5.6 Hz), 8.20 (1H, d, J = 7.8 Hz), 8.06 (1H, bs), 7.85 (1H, dt, J = 1.2 Hz, 4.8 Hz), 3.47 (2H, q, J = 7.2 Hz), 1.58–1.68 (4H, m), 1.28–1.44 (7H, m). ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.2, 150.1, 148.0, 137.3, 126.0, 122.1, 39.4, 31.5, 29.6, 26.6, 22.5, 14.0.

N-Cyclohexylpicolinamide (Table 3, **3v**).[64] White solid. Yield. 170 mg (83%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.54 (1H, d, J = 4.2 Hz), 8.20 (1H, d, J = 7.8 Hz), 7.97 (1H, bs), 7.84 (1H, t, J = 7.8 Hz), 7.41 (1H, dd, J = 7.2 Hz, 5.4 Hz), 3.93–4.01 (1H, m), 2.01 (2H, d, J = 9.6 Hz), 1.62–1.80 (3H, m), 1.18–1.50 (5H, m).

4-Chloro-*N*-cyclohexylbenzamide (Table 3, **3w**).[65] White solid. Yield. 130 mg (55%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (2H, d, J = 7.8 Hz), 7.39 (2H, d, J = 7.8 Hz), 5.97 (1H, bs), 3.92–4.00 (1H, m), 2.02 (2H, d, J = 10.2 Hz), 1.60–1.80 (3H, m), 1.14–1.46 (5H, m). ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.5, 137.4, 133.4, 128.7, 128.3, 48.8, 33.1, 25.5, 24.9.

4-Chloro-*N*-(2-hydroxyethyl)benzamide (Table 3, **3x**).[66] White solid. Yield. 165 mg (83%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 (2H, d, J = 7.8 Hz), 7.36 (2H, d, J = 7.8 Hz), 6.94 (1H, bs), 3.79 (2H, t, J = 4.8 Hz), 3.58 (2H, dt, J = 5.4 Hz, 4.8 Hz), 3.30 (1H, bs). ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.5, 137.9, 132.5, 128.8, 128.4, 62.0, 42.7.

N-Phenylpicolinamide (Table 3, **3y**).[12] White solid. Yield. 50 mg (76%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.03 (1H, bs), 8.61 (1H, d, J = 4.2 Hz), 8.30 (1H, d, J = 7.8 Hz), 7.90 (1H, t, J = 6.6 Hz), 7.39 (2H, t, J = 7.8 Hz), 7.15 (1H, t, J = 7.8 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.0, 149.8, 147.9, 137.7, 137.6, 129.0, 126.4, 124.3,122.4, 119.6.

4-Chloro-*N*-(pyridin-2-yl)benzamide (Table 3, **3z**).[47] White solid. Yield. 205 mg (88%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.77 (1H, bs), 8.37 (1H, d, J = 8.4 Hz), 8.26 (1H, dd, J = 1.2 Hz, 4.8 Hz), 7.88 (2H, d, J = 8.4 Hz), 7.77 (1H, t, J = 8.4 Hz), 7.47 (2H, d, J = 8.4 Hz), 7.08 (1H, dt, J = 1.2 Hz, 6.0 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.6, 151.4, 147.9, 138.6, 138.5, 132.6, 129.1, 128.7, 120.1, 114.2.

Author contributions

Experimental work was carried out by R. Prasad and P. Pradhan under the supervision of P. Ghosh while the manuscript was written by R. Prasad and P. Ghosh. All authors have given permission to the final version of the manuscript.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: copies of ^{1}H, ^{13}C NMR spectra of the synthesized compounds are available as SI. See DOI: https://doi.org/10.1039/d6ra03708a.

Acknowledgements

The authors thank sophisticated analytical & technical help institute SATHI, BHU, Varanasi, India for NMR and HRMS analysis. The financial grant received from FIST-DST (SR/FST/CS-I/2022/259) is thankfully acknowledged. R. P. thanks the University Grants Commission (UGC), India for SRF fellowship.

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