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Iron(III) chloride-catalyzed convenient one-pot synthesis of β-acetamido carbonyl compounds

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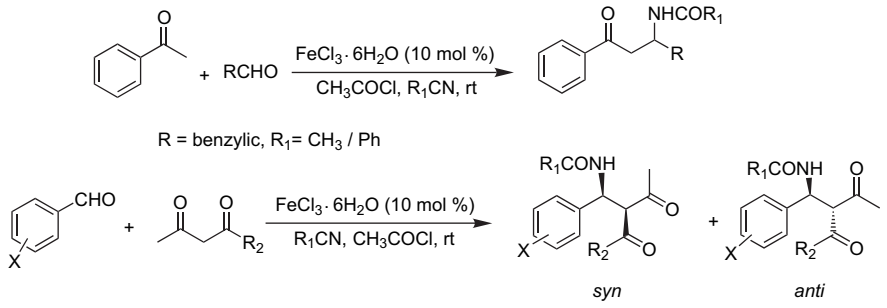
Abstract—A one-pot multi-component reaction of aldehydes, enolizable ketones or 1,3-dicarbonyls, acetonitrile/benzonitrile, and acetyl chloride is described for the preparation of β-acetamido carbonyl compounds using $FeCl_3 \cdot 6H_2O$ as a mild, inexpensive, and highly efficient catalyst. The effect of substrate as well as substituent for multi-component reaction versus Knoevenagel condensation is also illustrated. The key features of this methodology are operational simplicity, mild reaction conditions, and good yields.

1. Introduction

Multi-component reactions (MCRs) are a promising and vital field of chemistry because the synthesis of complicated molecules can be achieved in a very fast, efficient, and time-saving manner without the isolation of any intermediate. As a result, it requires minimum effort, which minimizes the environmental loading and is acceptable from a 'Green Chemistry' point of view. In recent years, the discovery of novel MCRs has become an increasingly active area of research, yielding novel chemical scaffolds for drug discovery. Thus, the development of new multi-component reactions is a popular area of research in current organic chemistry.[1]

The synthesis of β-acetamido carbonyl compounds has gained considerable attention in organic synthesis, owing to their importance as valuable building blocks for the preparation of 1,3-amino alcohols[2a,b] or β-amino acids,[2c] as well as for the synthesis of various bioactive molecules such as antibiotic nikkomycins or neopolyoxines.[3] The conventional way for the preparation of these compounds is the Dankin–West reaction using α-amino acids and acetic anhydride.[4] Later on, Iqbal et al. introduced both $CoCl_2$[5] and Montmorillonite K-10 clay[6] catalyzed multi-component reactions involving an aldehyde, enolizable ketone or keto ester in acetonitrile, and acetyl chloride for the one-pot synthesis of β-acetamido carbonyl compounds. Subsequently, $Cu(OTf)_2$/$Sc(OTf)_3$,[7] silica supported sulfuric acid,[8] BiOCl,[9] $ZrOCl_2 \cdot 8H_2O$,[10] $H_6P_2W_{18}O_{62}$,[11a] $H_3PW_{12}O_{40}$,[11b] I_2,[12] and Amberlyst-15[13] are reported as effective catalysts for the synthesis of β-acetamido carbonyl compounds. Recently, we have introduced $CeCl_3 \cdot 7H_2O$ as an efficient catalyst for the preparation of β-acetamido carbonyl compounds by MCRs.[14] Though the above methodologies are quite useful, most of the methods encounter some limitations, such as requirement of expensive catalysts, longer reaction time, and



Scheme 1.

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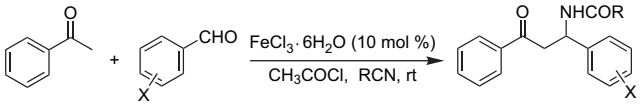
harsh reaction conditions. It is thus evident that there remains a wide scope for the development of clean and efficient methodologies for the preparation of β-acetamido carbonyl compounds using a cheap and readily available catalyst.

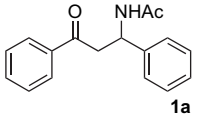
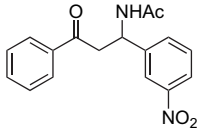
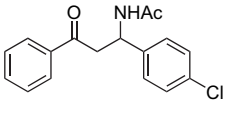
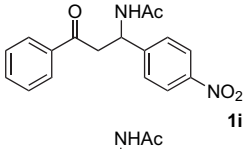
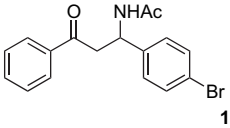
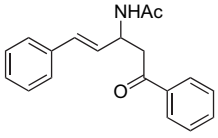
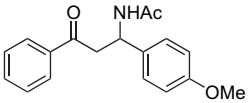
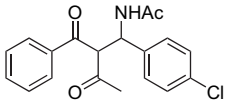
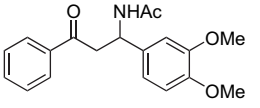
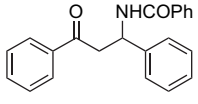
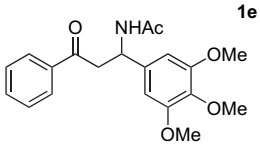
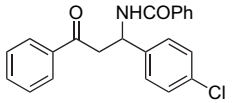
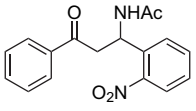
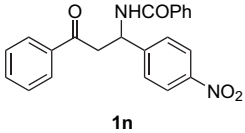
$FeCl_3 \cdot 6H_2O$ has emerged as a potentially useful Lewis acid and has been extensively used in various organic transformations.[15] Due to its unique catalytic properties, it has been used for a plethora of organic transformations such as the construction of 10*H*-indeno[1,2-*b*]triphenylene skeletons by oxidative cyclization,[16] cleavage of silyl protecting groups,[17] amino halogenation of arylmethylenecyclopropanes and arylvinylidenecyclopropanes,[18] hydroarylation of styrenes,[19] one-pot synthesis of 1,2-dihydro-2-oxo-3-pyridinecarboxylate derivatives,[20] Friedlander synthesis of quinolines,[21] one-pot synthesis of homoallyl benzyl ethers from aldehydes,[22] and synthesis of nicotinic acid derivatives,[23] etc (Scheme 1).

2. Results and discussion

For the preliminary study, 4-chlorobenzaldehyde (2 mmol) and acetophenone (2 mmol) in acetonitrile (5 mL) were stirred in the presence of a catalytic amount of $FeCl_3 \cdot 6H_2O$ (10 mol %) and acetyl chloride (3 mmol) at room temperature. The progress of the reaction was monitored by TLC. After completion of the reaction, it was poured into a beaker containing crushed ice to solidify the product. On solidification of the product, it was filtered off and dried to obtain the corresponding β-acetamido ketone. The solid product was recrystallized from ethyl acetate and hexane, and fully characterized by recording IR, ^1H NMR, and elemental analysis. Encouraged by this result, a wide variety of aromatic aldehydes, containing both electron withdrawing and donating substituents, were treated under the same experimental conditions and afforded the corresponding β-acetamido ketones (Table 1, entries 2–9) in good to excellent yields. Similarly, α,β-unsaturated

Table 1. $FeCl_3 \cdot 6H_2O$ catalyzed multi-component reaction for the preparation of β-acetamido ketones



Entry	β-Acetamido ketones[a]	Time (h)	Yield[b,c] (%)	Entry	β-Acetamido ketones[a]	Time (h)	Yield[b,c] (%)
1	 **1a**	8	88[6]	8	 **1h**	10	96[6]
2	 **1b**	5	99[8]	9	 **1i**	11	95[6]
3	 **1c**	5	98	10	 **1j**	6	85
4	 **1d**	7	94[10]	11	 **1k**	12	83
5	 **1e**	5	98	12	 **1l**	9	97
6	 **1f**	6	96	13	 **1m**	8	96
7	 **1g**	12	90[6]	14	 **1n**	12	92

[a] All the products were fully characterized by usual spectroscopic techniques and their data were compared with authentic data.
[b] Yields were calculated just after aqueous work up.
[c] Corresponding literature reference.

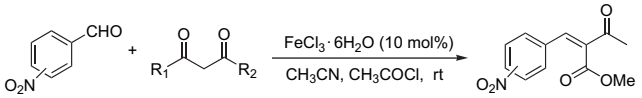
aldehydes such as cinnamaldehyde also reacts under the same experimental conditions and provided the desired product (Table 1, entry 10) without any difficulty. A 1,3-diketone, namely benzoylacetone, was treated under the same experimental conditions to provide β-acetamido diketones with poor diastereoselectivity. Next, by using benzonitrile in place of acetonitrile, a variety of aldehydes were transformed to their corresponding β-benzamido ketones in excellent yields (Table 1, entries 12–14). This clearly demonstrates that in this reaction, the alkyl/aryl nitrile acts as a nucleophile.

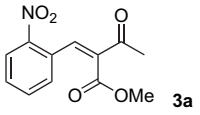
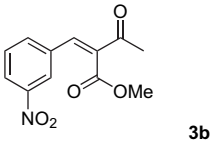
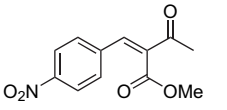
To extend the preparative utility and generality of this multicomponent reaction, a variety of aromatic aldehydes were treated with methyl acetoacetate or ethyl benzoyl acetoacetate under the same experimental conditions, and the corresponding β-acetamido/benzamido keto esters were obtained in good yields with moderate to good diastereoselectivities. These β-acetamido esters are useful precursor for the synthesis of β-aryl homoisothreonine derivatives, which can be used for the preparation of dipeptide isoesters by incorporation with an amino acid residue. The ratio of *syn*:*anti* diastereomers was determined from the ^{1}H NMR spectrum of the crude reaction mixture. The ratio of diastereomers differed with substitution on the aromatic ring (Table 2).

Interestingly, during the course of our study we have noticed that when the aldehyde contains a nitro group either at the ortho, meta or para positions, and is treated with a β-keto ester under the same experimental conditions, it gives only the Knoevenagel condensation product instead of our expected β-acetamido keto ester (Table 3). All these products were fully characterized by recording IR, ^{1}H NMR, and elemental analysis.

To further confirm the formation of these alkenes and to know the geometry of the substituents on the double bond,

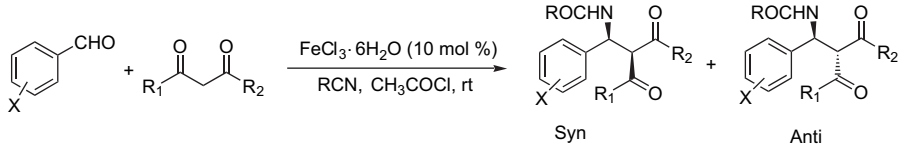
Table 3. $FeCl_3\cdot 6H_2O$ catalyzed Knoevenagel products for nitro benzaldehydes along with trace multi-component products

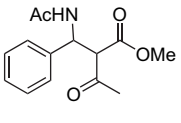
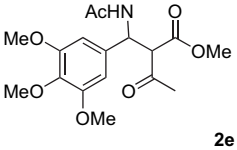
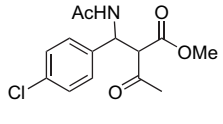
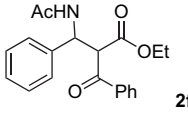
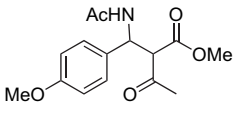
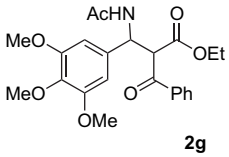
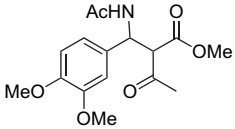
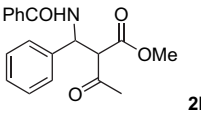


Entry	Product[a]	Yield[b] (%)
1	 **3a**	81
2	 **3b**	80
3	 **3c**	82

[a] All the products were characterized by usual spectroscopic analysis.
[b] Isolated yield.

Table 2. $FeCl_3\cdot 6H_2O$ catalyzed multi-component reaction for the preparation of β-acetamido esters



Entry	Product	Yield[a,c] (%)	*syn*:*anti*[b]	Entry	Product	Yield[a] (%)	*syn*:*anti*[b]
1	 **2a**	81[5]	26:74	5	 **2e**	84	25:75
2	 **2b**	80[5]	27:73	6	 **2f**	78	98:2
3	 **2c**	82[5]	46:54	7	 **2g**	80	95:5
4	 **2d**	86	5:95	8	 **2h**	85	71:29

[a] Yields were calculated without further purification.
[b] The *syn*:*anti* ratio was determined from ^{1}H NMR spectrum of crude reaction mixture.
[c] Corresponding literature reference.

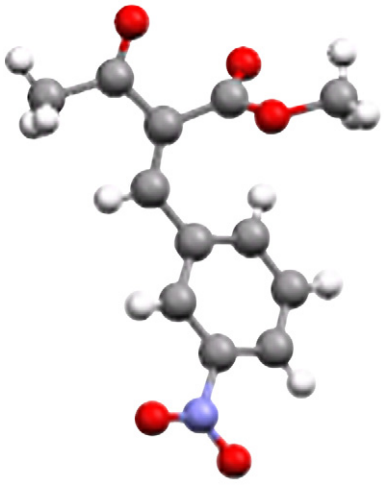


Figure 1. ORTEP plot of compound **3b**.

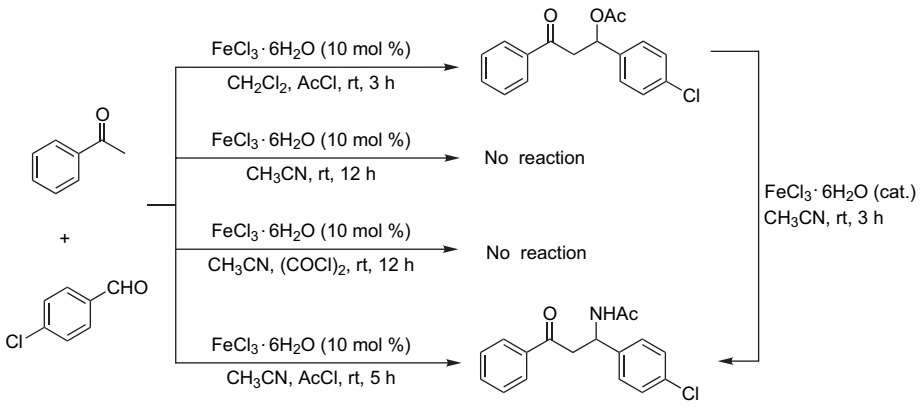
the product **3b** was recrystallized from ethyl acetate–hexane and a single crystal XRD was recorded. It shows the (*Z*)-configuration of the alkene as depicted in Figure 1.

Although the exact explanation of this anomaly is yet to be determined, we assumed that due to electronic effects, i.e., electron withdrawing nature of the nitro group, it provides the Knoevenagel condensation alkenes.

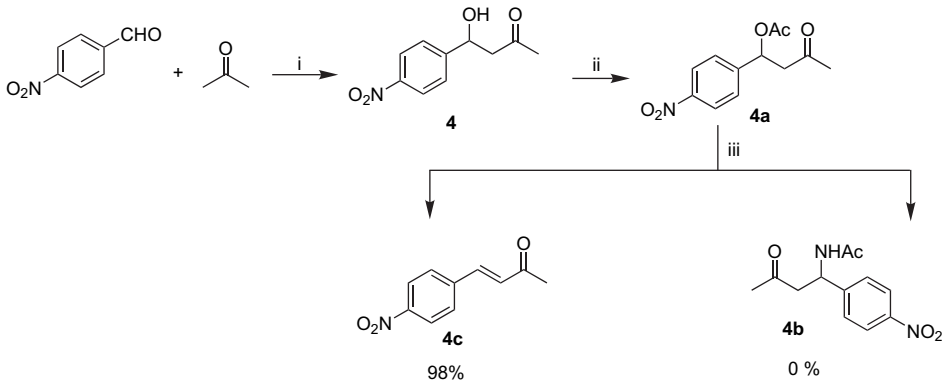
Next we turned our attention to study the mechanistic aspect of this multi-component reaction. Thus, the reaction of 4-chlorobenzaldehyde with acetophenone was chosen as a model reaction for this study. In the absence of acetyl chloride, the reaction failed to provide the desired product, which clearly indicates that it plays a vital role in this reaction, although not directly involved in the final product. Then the same reaction was tried using oxalyl chloride, instead of acetyl chloride, and the reaction was also unsuccessful. From this observation, it is clear that the chloride ion does not have any role in the above transformation. Similarly, the same reaction was carried out in the absence of acetonitrile using dichloromethane as a solvent, and the corresponding β-acetoxy ketone was obtained in 60% yield after 3 h of stirring at room temperature. Consequently, this β-acetoxy ketone as shown in Scheme 2 was once again treated with 10 mol % of the catalyst in acetonitrile as solvent, and the corresponding β-acetamido ketone was obtained in 80% yield under the same experimental conditions. This clearly demonstrates the working hypothesis that the reaction goes via an aldol reaction followed by acetylation and subsequent nucleophilic displacement by the alkyl/aryl nitrile to get the desired product as shown in Scheme 4.

Next, the aldol product **4** was prepared following a literature procedure using proline as an organo catalyst,[24] and acetylated by our self developed BDMS method[25] to get the product **4a**. Subsequently, the compound **4a** was treated separately with acetonitrile under the same experimental conditions using 10 mol % $FeCl_3 \cdot 6H_2O$ as a catalyst. Interestingly, after 12 h of stirring, α,β-unsaturated ketone **4c** was isolated in 98% yield instead of the desired β-acetamido ketone **4b** as shown in Scheme 3. Similarly, in Table 3 we have noted that nitro substituted aldehydes react with methyl acetoacetate and provide the Knoevenagel condensation products under the experimental conditions. These studies lead us to the conclusion that when aliphatic ketones or keto esters react with nitro substituted aldehydes, the intermediate acetylated aldol products (e.g., **4a**) prefer α-H elimination to provide α,β-unsaturated ketones to the formation of desired acetamido ketone by nucleophilic substitution by alkyl or aryl nitrile. This may be attributed to the electronic effect, i.e., the electron withdrawing nature of the substituent as well as, the stability of the elimination products. Later, the Knoevenagel product **3b** was treated further with acetonitrile and acetyl chloride in the presence of 10 mol % catalyst and kept stirring. No detectable amount of the desired β-acetamido keto ester was found, even after stirring for 12 h. Thus it is clear that these unsaturated ketones are more stable compared to the corresponding β-acetamido ketones.

Next to exemplify that acetyl chloride is not incorporated in the final product and acetonitrile itself is the N-donor and nucleophile (i.e., it follows the Ritter reaction pathway), we tried the reaction of 4-chlorobenzaldeyde under similar experimental conditions using benzonitrile (1.5 equiv) instead of acetonitrile (Table 1, entry 13). The formation of benzamide clearly indicates that in this reaction nitrile acts not only as a solvent but also as a nitrogen donor. Therefore,

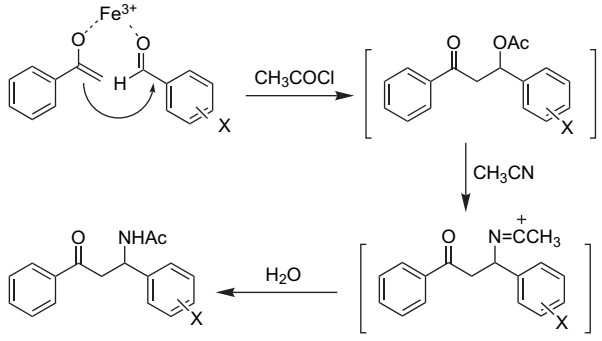


Scheme 2.



Scheme 3. Reagents: (i) proline (20 mol %), H_2O; (ii) BDMS (5 mol %), Ac_2O; (iii) $FeCl_3 \cdot 6H_2O$ (10 mol %), CH_3CN, CH_3COCl.

the most probable mechanism for this reaction is illustrated in Scheme 4.



Scheme 4. Probable mechanism of iron(III) chloride-catalyzed multi-component reactions for the preparation of β-acetamido carbonyl compounds.

3. Conclusion

In summary, we have devised a new synthetic methodology using $FeCl_3 \cdot 6H_2O$, a cheap, readily available, and efficient catalyst for the one-pot synthesis of β-acetamido carbonyl compounds. In the case of nitro aldehydes, the reaction with methyl acetoacetate provides α,β-unsaturated ketones instead of the expected β-acetamido carbonyl compounds. The simplicity of the present protocol, high yields, and efficiency of the catalyst are the key features of this present protocol. Due to the low cost and ready availability of the reagent $FeCl_3 \cdot 6H_2O$, we prefer this protocol than our earlier reported method.[14] In addition we have presented a thorough study on the mechanistic aspect of this multi-component reaction in this article. Thus the present method will be useful for the facile preparation of β-acetamido ketones and keto esters.

4. Experimental

4.1. General

All reagents were of commercial quality and were used as received. Solvents were dried and purified using standard techniques. Melting points were recorded on a Buchi B-545 melting point apparatus and are uncorrected. IR spectra were recorded in KBr on a Nicolet Impact 410 spectrophotometer. ^1H NMR spectra and ^{13}C NMR spectra were recorded on a Varian 400 MHz spectrometer in $CDCl_3$ or DMSO, d_6 using TMS as an internal reference. Elemental analyses were carried out in a Perkin Elmer 2400 automatic carbon, hydrogen, nitrogen, and sulfur analyzer. Column chromatographic separations were done on SRL silica gel (60–120 mesh).

4.2. General procedure for the preparation of β-acetamido/benzamido ketones or keto esters

To a stirred solution of aldehyde (2 mmol) and acetophenone/methyl acetoacetate (2 mmol) in acetonitrile (3 mL) or benzonitrile (3 mmol) were added acetyl chloride (3 mmol) and $FeCl_3 \cdot 6H_2O$ (0.2 mmol) and the reaction stirred at room temperature. The progress of the reaction was monitored by TLC, and after completion of the reaction, crushed (50 mL) ice was added to the reaction mixture and stirred thoroughly. On solidification, the products were filtered off and dried to get the corresponding β-acetamido ketones. For all the substrates of Table 2, the reactions were stirred for 12 h, then 50 mL of water was added. The mixture was extracted with ethyl acetate (3×20 mL), washed with water (3×20 mL), dried over Na_2SO_4, and the solvent was removed using a rotary evaporator. The crude mixture was recrystallized from ethyl acetate–hexane and the solid product (mixture of diastereomers) was isolated. The solid products were recrystallized from the mixture of solvents (ethyl acetate–hexane) and fully characterized by recording IR, NMR, and elemental analysis.

4.2.1. *N*-(3-Oxo-1,3-diphenyl-propyl) acetamide (1a).[6] Yield 88% (470.0 mg), white crystal (EtOAc–hexane), mp 103–105 °C (lit.[6] mp 102–104 °C) [Found: C, 76.49; H, 6.39; N, 5.19. $C_{17}H_{17}NO_2$ requires C, 76.38; H, 6.41; N, 5.24%]; ν_{max} (KBr): 3278, 3099, 2925, 1682, 1646, 1556, 1447, 1372, 1195, 990, 750 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.01 (3H, s, CO*Me*), 3.43 (1H, dd, *J* 6.0, 16.8 Hz, CH_2), 3.75 (1H, dd, *J* 5.2, 16.8 Hz, CH_2), 5.53–5.57 (1H, m, C*H*), 6.68 (1H, d, *J* 7.6 Hz, N*H*), 7.19–7.30 (5H, m, Ph), 7.43 (2H, t, *J* 8.0 Hz, Ph), 7.55 (1H, t, *J* 7.6 Hz, Ph), 7.88 (2H, d, *J* 8.0 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.6, 43.2, 49.9, 126.6, 127.7, 128.3, 128.9, 133.7, 136.4, 140.7, 169.3, 198.3.

4.2.2. *N*-[1-(4-Chlorophenyl)-3-oxo-3-phenyl-propyl] acetamide (1b).[8] Yield 99% (597.0 mg), white crystal

(EtOAc–hexane), mp 146 °C (lit.[8] mp 146–148 °C) [Found: C, 67.53; H, 5.28; N, 4.72. $C_{17}H_{16}ClNO_2$ requires C, 67.66; H, 5.34; N, 4.64%]; ν_{max} (KBr): 3291, 2329, 1687, 1647, 1547, 1445, 1349, 1229, 1009, 754 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.01 (3H, s, CO*Me*), 3.41 (1H, dd, *J* 6.0, 16.8 Hz, CH_2), 3.73 (1H, dd, *J* 5.2, 17.2 Hz, CH_2), 5.50–5.55 (1H, m, C*H*), 6.74 (1H, d, *J* 7.2 Hz, N*H*), 7.25 (4H, d, *J* 4.4 Hz, Ph), 7.44 (2H, t, *J* 8.0 Hz, Ph), 7.56 (1H, t, *J* 7.6 Hz, Ph), 7.87 (2H, d, *J* 8.4 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.0, 43.2, 49.4, 128.1, 128.3, 128.9, 133.6, 133.9, 140.0, 142.0, 148.0, 165.0, 198.7.

4.2.3. *N*-[1-(4-Bromophenyl)-3-oxo-3-phenyl-propyl] acetamide (1c). Yield 98% (678.0 mg), white crystal (EtOAc–hexane), mp 148–150 °C [Found: C, 58.85; H, 4.70; N, 4.12. $C_{17}H_{16}BrNO_2$ requires C, 58.98; H, 4.66; N, 4.05%]; ν_{max} (KBr): 3285, 2923, 1684, 1651, 1550, 1374, 1292, 1006, 757 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.02 (3H, s, CO*Me*), 3.40 (1H, dd, *J* 6.0, 17.2 Hz, CH_2), 3.72 (1H, dd, *J* 5.2, 16.8 Hz, CH_2), 5.49–5.51 (1H, m, C*H*), 6.73 (1H, d, *J* 6.8, N*H*), 7.19 (2H, d, *J* 8.4 Hz, Ph), 7.40 (2H, d, *J* 8.4 Hz, Ph), 7.44 (2H, d, *J* 8.0 Hz, Ph), 7.55 (1H, t, *J* 7.2 Hz, Ph), 7.87 (2H, d, *J* 7.2 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.7, 42.9, 49.4, 128.3, 128.4, 129.0, 131.9, 134.0, 136.2, 140.2, 166.5, 196.5.

4.2.4. *N*-[1-(4-Methoxyphenyl)-3-oxo-3-phenyl-propyl] acetamide (1d).[10] Yield 94% (560.0 mg), white crystal (EtOAc–hexane), mp 110–111 °C (lit.[10] mp 110–112 °C) [Found: C, 72.59; H, 6.49; N, 4.79. $C_{18}H_{19}NO_3$ requires C, 72.71; H, 6.44; N, 4.71%]; ν_{max} (KBr): 3301, 2928, 1688, 1648, 1545, 1372, 1238, 1033, 754 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.0 (3H, s, CO*Me*), 3.39 (1H, dd, *J* 6.4, 16.8 Hz, CH_2), 3.72 (1H, dd, *J* 5.2, 17.2 Hz, CH_2), 3.74 (3H, s, O*Me*), 5.46–5.51 (1H, m, C*H*), 6.57 (1H, d, *J* 8.0 Hz, N*H*), 6.81 (2H, d, *J* 8.4 Hz, Ph), 7.23 (2H, d, *J* 8.0 Hz, Ph), 7.42 (2H, t, *J* 7.6 Hz, Ph), 7.54 (1H, t, *J* 7.2 Hz, Ph), 7.89 (2H, d, *J* 7.2 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.6, 43.5, 49.7, 55.4, 114.2, 127.9, 128.3, 128.9, 133.1, 133.7, 136.7, 158.9, 169.7, 198.8.

4.2.5. *N*-[1-(3,4-Dimethoxyphenyl)-3-oxo-3-phenyl-propyl] acetamide (1e). Yield 98% (640.0 mg), brownish solid, mp 118–119 °C [Found: C, 69.58; H, 6.40; N, 4.37. $C_{19}H_{21}NO_4$ requires C, 69.71; H, 6.47; N, 4.28%]; ν_{max} (KBr): 3247, 3076, 2917, 2846, 1681, 1637, 1519, 1462, 1253, 1026, 751 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.01 (3H, s, CO*Me*), 3.39 (1H, dd, *J* 6.4, 16.8 Hz, CH_2), 3.72 (1H, dd, *J* 5.2, 16.4 Hz, CH_2), 3.81 (3H, s, O*Me*), 3.82 (3H, s, O*Me*), 5.45–5.50 (1H, m, C*H*), 6.58 (1H, d, *J* 8.0 Hz, N*H*), 6.76 (1H, d, *J* 8.0 Hz, Ph), 6.83 (2H, s+d, *J* 8.0 Hz, Ph), 7.43 (2H, t, *J* 7.6 Hz, Ph), 7.55 (1H, t, *J* 7.6 Hz, Ph), 7.89 (2H, d, *J* 7.2 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.7, 43.4, 50.1, 56.1, 110.5, 111.3, 118.3, 128.3, 128.9, 133.6, 133.7, 149.2, 169.6, 199.0.

4.2.6. *N*-[1-(3,4,5-Trimethoxyphenyl)-3-oxo-3-phenyl-propyl] acetamide (1f). Yield 96% (685.0 mg), white solid, mp 168–169 °C [Found: C, 67.34; H, 6.42; N, 3.99. $C_{20}H_{23}NO_5$ requires C, 67.21; H, 6.49; N, 3.92%]; ν_{max} (KBr): 3276, 2928, 1688, 1648, 1592, 1458, 1339, 1246, 1001, 754 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.01 (3H, s, CO*Me*), 3.37 (1H, dd, *J* 6.0, 16.8 Hz, CH_2), 3.69 (1H, dd, *J* 5.2, 16.8 Hz, CH_2), 3.76 (3H, s, O*Me*), 3.78 (6H, s, 2×O*Me*), 5.44 (1H, q, *J* 7.6 Hz, C*H*), 6.52 (2H, s, Ph), 6.67 (1H, d, *J* 7.6 Hz, N*H*), 7.43 (2H, t, *J* 7.6 Hz, Ph), 7.55 (1H, t, *J* 7.2 Hz, Ph), 7.88 (2H, d, *J* 8.0 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.7, 43.4, 50.7, 56.3, 60.9, 103.9, 128.3, 128.9, 133.8, 136.8, 136.9, 153.5, 156.2, 169.5, 199.4.

4.2.7. *N*-[1-(2-Nitrophenyl)-3-oxo-3-phenyl-propyl] acetamide (1g).[6] Yield 90% (560.0 mg), white solid, mp 190–191 °C (lit.[6] mp 186–188 °C) [Found: C, 65.49; H, 5.23; N, 8.88. $C_{17}H_{16}N_2O_4$ requires C, 65.38; H, 5.16; N, 8.97%]; ν_{max} (KBr): 3326, 2379, 1686, 1651, 1544, 1517, 1357, 1337, 1059, 682 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.00 (3H, s, CO*Me*), 3.63 (1H, dd, *J* 5.6, 16.8 Hz, CH_2), 3.71 (1H, dd, *J* 6.4, 17.2 Hz, CH_2), 5.93–5.97 (1H, m, C*H*), 7.07 (1H, d, *J* 5.6 Hz, N*H*), 7.39 (1H, t, *J* 8.0 Hz, Ph), 7.46 (2H, t, *J* 8.0 Hz, Ph), 7.57 (2H, t, *J* 7.6 Hz, Ph), 7.71 (1H, d, *J* 8.0 Hz, Ph), 7.92 (2H, d, *J* 7.2 Hz), 7.94 (1H, d, *J* 6.8, Ph); δ_C (100 MHz, $CDCl_3$) 23.5, 42.4, 47.7, 125.3, 128.5, 128.6, 129.0, 130.1, 133.7, 134.1, 136.5, 137.1, 148.7, 169.5, 198.5.

4.2.8. *N*-[1-(3-Nitrophenyl)-3-oxo-3-phenyl-propyl] acetamide (1h).[6] Yield 96% (593.0 mg), white solid, mp 139–140 °C (lit.[10] mp 139–140 °C) [Found: C, 65.29; H, 5.22; N, 8.88. $C_{17}H_{16}N_2O_4$ requires C, 65.38; H, 5.16; N, 8.97%]; ν_{max} (KBr): 3306, 1693, 1644, 1545, 1522, 1347, 983, 684 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.08 (3H, s, CO*Me*), 3.51 (1H, dd, *J* 5.6, 17.6 Hz, CH_2), 3.79 (1H, dd, *J* 5.2, 17.6 Hz, CH_2), 5.62–5.67 (1H, m, C*H*), 6.91 (1H, d, *J* 7.6 Hz, N*H*), 7.42–7.49 (3H, m, Ph), 7.57 (1H, t, *J* 7.6 Hz, Ph), 7.68 (1H, d, *J* 7.6 Hz, Ph), 7.87 (2H, d, *J* 7.6 Hz, Ph), 8.06 (1H, d, *J* 6.8 Hz, Ph), 8.19 (1H, s, Ph); δ_C (100 MHz, $CDCl_3$) 23.6, 42.9, 49.3, 121.5, 122.6, 128.3, 129.1, 129.8, 130.8, 133.1, 134.2, 136.4, 143.7, 169.9, 198.3.

4.2.9. *N*-[1-(4-Nitrophenyl)-3-oxo-3-phenyl-propyl] acetamide (1i).[6] Yield 95% (593.0 mg), yellow crystalline solid, mp 153 °C (lit.[10] mp 154 °C) [Found: C, 65.29; H, 5.23; N, 8.99. $C_{17}H_{16}N_2O_4$ requires C, 65.38; H, 5.16; N, 8.97%]; ν_{max} (KBr): 3306, 1696, 1646, 1595, 1537, 1350, 988, 755 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.10 (3H, s, CO*Me*), 3.51 (1H, dd, *J* 5.6, 17.6 Hz, CH_2), 3.81 (1H, dd, *J* 5.2, 17.6 Hz, CH_2), 5.65–5.67 (1H, m, C*H*), 6.96 (1H, d, *J* 8.0 Hz, N*H*), 7.47 (2H, t, *J* 8.0 Hz, Ph), 7.51 (2H, d, *J* 8.8 Hz, 2H, Ph), 7.60 (1H, t, *J* 7.2 Hz, Ph), 7.89 (2H, d, *J* 7.2 Hz, Ph), 8.17 (2H, d, *J* 8.8 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.5, 42.6, 49.2, 123.7, 127.2, 127.9, 128.7, 131.0, 133.9, 136.0, 138.4, 169.5, 197.8.

4.2.10. *N*-[1-(4-Styryl)-3-oxo-3-phenyl-propyl] acetamide (1j). Yield 85% (497.0 mg), light yellow solid, mp 120–121 °C [Found: C, 77.68; H, 6.45; N, 4.83. $C_{19}H_{19}NO_2$ requires C, 77.79; H, 6.53; N, 4.77%]; ν_{max} (KBr): 3291, 3065, 2928, 1687, 1648, 1635, 1547, 1445, 1366, 1083, 751 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.04 (3H, s, CO*Me*), 3.35 (1H, dd, *J* 5.6, 17.6 Hz, CH_2), 3.53 (1H, dd, *J* 4.4, 17.6 Hz, CH_2), 5.09–5.13 (1H, m, C*H*), 6.33 (1H, dd, *J* 6.8, 16.0 Hz, C*H*═), 6.54 (1H, d, *J* 15.6 Hz, C*H*═), 7.20 (1H, d, *J* 6.8 Hz, N*H*), 7.25 (2H, t, *J* 6.8 Hz, Ph), 7.29 (3H, d, *J* 7.2 Hz, Ph), 7.46 (2H, t, *J* 7.6 Hz, Ph), 7.57 (1H, t, *J* 7.6 Hz, Ph), 7.93 (2H, d, *J* 7.2 Hz, Ph); δ_C (100 MHz,

$CDCl_3$) 23.8, 42.8, 48.3, 126.7, 127.9, 128.3, 128.6, 128.7, 128.9, 1301.7, 133.9, 169.5, 199.7.

4.2.11. *N*-[1-(4-Chlorophenyl)-2-acetyl-3-oxo-3-phenyl-propyl] acetamide (1k). Yield 83% (571.0 mg), white solid (mixture of diastereomers): (*syn*:*anti*=40:60), mp 172–174 °C [Found: C, 66.49; H, 5.21; N, 4.18. $C_{19}H_{18}NClO_3$ requires C, 66.38; H, 5.28; N, 4.07%]; ν_{max} (KBr): 3301, 1704, 1688, 1648, 1527, 1370, 1087 cm^{-1}; Data for the major isomer (*anti*): δ_H (400 MHz, $CDCl_3$) 1.95 (3H, s, CO*Me*), 2.13 (3H, s, CO*Me*), 5.05 (1H, d, *J* 7.6 Hz, C*H*), 5.85 (1H, t, *J* 8.4 Hz, C*H*), 6.57 (1H, d, *J* 8.4 Hz, N*H*), 7.23 (4H, d, *J* 6.4 Hz, Ph), 7.47 (2H, t, *J* 7.2 Hz, Ph), 7.59 (1H, t, *J* 8.8 Hz, Ph), 7.73 (1H, d, *J* 8.0 Hz, Ph), 7.90 (1H, d, *J* 8.4 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.5, 29.2, 52.4, 66.7, 128.5, 128.8, 129.2, 129.3, 133.9, 134.5, 135.9, 138.5, 169.8, 193.8, 204.0.

4.2.12. *N*-(3-Oxo-1,3-diphenyl-propyl) benzamide (1l). Yield 97% (639.0 mg), yellowish solid, mp 153–154 °C [Found: C, 80.33; H, 5.76; N, 4.31. $C_{22}H_{19}NO_2$ requires C, 80.22; H, 5.81; N, 4.25%]; ν_{max} (KBr): 3306, 3062, 1681, 1634, 1599, 1488, 1357, 981, 754 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 3.52 (1H, dd, *J* 6.0, 16.4 Hz, CH_2), 3.87 (1H, dd, *J* 4.8, 16.8 Hz, CH_2), 5.73–5.78 (1H, m, C*H*), 7.22 (1H, t, *J* 7.2 Hz, Ph), 7.30 (2H, t, *J* 7.6 Hz, Ph), 7.37–7.45 (5H, m, Ph), 7.49 (2H, t, *J* 7.2 Hz, Ph), 7.55 (1H, t, *J* 7.2 Hz, Ph), 7.60 (1H, d, *J* 8.0 Hz, Ph), 7.82 (2H, d, *J* 8.0 Hz, Ph), 7.90 (2H, d, *J* 8.0 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 43.1, 50.4, 126.6, 127.2, 127.6, 128.3, 128.7, 128.8, 128.9, 131.8, 133.8, 134.4, 136.7, 141.1, 166.9, 199.3.

4.2.13. *N*-[1-(4-Chlorophenyl)-3-oxo-3-phenyl-propyl] benzamide (1m). Yield 96% (699.0 mg), yellow solid, mp 180–182 °C [Found: C, 72.74; H, 4.93; N, 3.93. $C_{22}H_{18}ClNO_2$ requires C, 72.63; H, 4.99; N, 3.85%]; ν_{max} (KBr): 3276, 2928, 2372, 1683, 1641, 1542, 1490, 1362, 1087, 689 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 3.52 (1H, dd, *J* 5.6, 17.2 Hz, CH_2), 3.86 (1H, dd, *J* 4.8, 17.2 Hz, CH_2), 5.71–5.75 (1H, m, C*H*), 7.28 (2H, d, *J* 8.4 Hz, Ph), 7.35 (2H, d, *J* 8.4 Hz, Ph), 7.46 (2H, t, *J* 7.6 Hz, Ph), 7.47 (2H, t, *J* 7.6 Hz, Ph), 7.53 (1H, t, *J* 7.2 Hz, Ph), 7.59 (1H, t, *J* 7.2 Hz, Ph), 7.69 (1H, d, *J* 8.0 Hz, N*H*), 7.84 (2H, d, *J* 7.6 Hz, Ph), 7.91 (2H, d, *J* 8.0 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 42.6, 49.6, 127.0, 127.9, 128.1, 128.6, 128.8, 131.7, 133.1, 133.8, 136.4, 139.5, 166.7, 198.9.

4.2.14. *N*-[1-(4-Nitrophenyl)-3-oxo-3-phenyl-propyl] benzamide (1n). Yield 92% (689.0 mg), solid, mp 142–144 °C [Found: C, 70.69; H, 4.80; N, 7.39. $C_{22}H_{18}N_2O_4$ requires C, 70.58; H, 4.85; N, 7.48%]; ν_{max} (KBr): 3313, 3060, 2934, 1684, 1626, 1517, 1399, 1111, 688 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 3.57 (1H, dd, *J* 4.4, 18.0 Hz, CH_2), 3.90 (1H, dd, *J* 4.4, 16.8 Hz, CH_2), 5.82–5.86 (1H, m, C*H*), 7.46 (4H, t, *J* 7.6 Hz, Ph), 7.52 (1H, d, *J* 6.8 Hz, N*H*), 7.55–7.59 (4H, m, Ph), 7.84 (2H, d, *J* 7.6 Hz, Ph), 7.89 (2H, d, *J* 7.6 Hz, Ph), 8.16 (2H, d, *J* 8.8 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 42.7, 49.9, 124.1, 127.3, 127.7, 128.4, 128.9, 129.1, 132.2, 133.8, 134.3, 136.4, 147.2, 148.8, 167.0, 198.0.

4.2.15. Methyl 2-acetyl-3-acetamido-3-phenyl propionate (2a).[5] Yield 81% (426.0 mg), white solid, mp 128–130 °C (lit.[5] mp 129–131 °C) [Found: C, 63.74; H, 6.45; N, 5.43. $C_{14}H_{17}NO_4$ requires C, 63.87; H, 6.51; N, 5.32%]; ν_{max} (KBr): 3329, 3049, 2961, 1747, 1717, 1643, 1528, 1451, 1371, 1037, 754 cm^{-1}. Data for the major isomer (*anti*): δ_H (400 MHz, $CDCl_3$) 1.99 (3H, s, CO*Me*), 2.10 (3H, s, CO*Me*), 3.69 (3H, s, O*Me*), 4.07 (1H, d, *J* 5.6 Hz, C*H*), 5.73 (1H, dd, *J* 5.6, 9.2 Hz, C*H*), 6.91 (1H, d, *J* 8.4 Hz, N*H*), 7.23–7.31 (5H, m, Ph); δ_C (100 MHz, $CDCl_3$) 23.5, 31.1, 52.5, 53.0, 62.5, 126.6, 128.0, 128.9, 139.3, 166.1, 169.9, 203.8.

4.2.16. Methyl 2-acetyl-3-acetamido-3-(*p*-chlorophenyl) propionate (2b).[5] Yield 80% (477.0 mg), white solid (mixture of diastereomers), mp 131–133 °C (lit.[5] mp 130–132 °C) [Found: C, 56.60; H, 5.36; N, 4.82. $C_{14}H_{16}ClNO_4$ requires C, 56.49; H, 5.42; N, 4.71%]; ν_{max} (KBr): 3324, 1744, 1714, 1646, 1541, 1486, 1371, 1091, 724 cm^{-1}. Data for the major isomer (*anti*): δ_H (400 MHz, $CDCl_3$) 1.99 (3H, s, CO*Me*), 2.13 (3H, s, CO*Me*), 3.70 (3H, s, O*Me*), 4.04 (1H, d, *J* 5.6 Hz, C*H*), 5.68 (1H, dd, *J* 5.6, 9.2 Hz, C*H*), 6.94 (1H, d, *J* 8.4 Hz, N*H*), 7.20 (2H, d, *J* 8.8 Hz, Ph), 7.25 (2H, d, *J* 8.0 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.5, 31.0, 52.0, 53.1, 62.4, 128.2, 129.1, 133.9, 138.0, 167.8, 169.9, 203.8.

4.2.17. Methyl 2-acetyl-3-acetamido-3-(*p*-methoxy-phenyl) propionate (2c).[5] Yield 82% (480.0 mg), light yellow solid (mixture of diastereomers), mp 140–143 °C (lit.[5] mp 142–144 °C) [Found: C, 61.53; H, 6.48; N, 4.86. $C_{15}H_{19}NO_5$ requires C, 61.42; H, 6.53; N, 4.77%]; ν_{max} (KBr): 3335, 2967, 1739, 1714, 1648, 1602, 1517, 1434, 1369, 1028, 587 cm^{-1}. Data for the major isomer (*anti*): δ_H (400 MHz, $CDCl_3$) 1.97 (3H, s, CO*Me*), 2.14 (3H, s, CO*Me*), 3.68 (3H, s, O*Me*), 3.76 (3H, s, O*Me*), 3.86 (1H, d, *J* 6.0 Hz, C*H*), 5.61 (1H, dd, *J* 6.0, 9.2 Hz, C*H*), 6.81 (2H, d, *J* 8.0 Hz, Ph), 6.98 (1H, d, *J* 8.4 Hz, N*H*), 7.18 (2H, d, *J* 8.4 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 23.3, 30.7, 52.8, 55.2, 55.4, 62.7, 127.7, 131.2, 131.7, 131.9, 160.3, 169.5, 187.8.

4.2.18. Methyl 2-acetyl-3-acetamido-3-(dimethoxy-phenyl) propionate (2d). Yield 86% (556.0 mg), white solid (mixture of diastereomers), mp 125–127 °C [Found: C, 59.55; H, 6.49; N, 4.42. $C_{16}H_{21}NO_6$ requires C, 59.43; H, 6.55; N, 4.33%]; ν_{max} (KBr): 3331, 2928, 1745, 1718, 1648, 1542, 1458, 1372, 1023, 546 cm^{-1}. Data for the major isomer (*anti*): δ_H (400 MHz, $CDCl_3$) 1.98 (3H, s, CO*Me*), 2.15 (3H, s, CO*Me*), 3.68 (3H, s, O*Me*), 3.83 (3H, s, O*Me*), 3.85 (3H, s, O*Me*), 4.03 (1H, d, *J* 6.0 Hz, C*H*), 5.66 (1H, dd, *J* 6.4, 9.2 Hz, C*H*), 6.78 (1H, s, Ph), 6.79 (2H, d, *J* 7.6 Hz, Ph), 6.84 (1H, d, *J* 9.2 Hz, N*H*); δ_C (100 MHz, $CDCl_3$) 23.6, 30.9, 52.4, 53.0, 56.1, 63.0, 110.3, 111.3, 118.8, 131.9, 149.0, 149.2, 167.9, 169.7, 204.2.

4.2.19. Methyl 2-acetyl-3-acetamido-3-(trimethoxy-phenyl) propionate (2e). Yield 84% (593.0 mg), brown solid (mixture of diastereomers), mp 130–133 °C [Found: C, 57.89; H, 6.50; N, 4.09. $C_{17}H_{23}NO_7$ requires C, 57.78; H, 6.56; N, 3.96%]; ν_{max} (KBr): 3326, 2928, 1747, 1718, 1683, 1651, 1592, 1458, 1339, 1003, 670 cm^{-1}. Data for the major isomer (*anti*): δ_H (400 MHz, $CDCl_3$) 2.00 (3H, s, CO*Me*), 2.16 (3H, s, CO*Me*), 3.69 (3H, s, O*Me*), 3.78 (3H, s, O*Me*), 3.82 (6H, s, 2×O*Me*), 4.01 (1H, d, *J* 6.0 Hz,

C*H*), 5.64 (1H, dd, *J* 5.6, 8.8 Hz, C*H*), 6.47 (2H, s, Ph), 6.90 (1H, d, *J* 8.8 Hz, N*H*); δ_C (100 MHz, $CDCl_3$) 23.5, 31.1, 31.2, 52.9, 53.0, 56.3, 60.9, 62.8, 103.9, 135.1, 153.5, 153.6, 167.9, 169.8, 200.0.

4.2.20. Ethyl 2-benzoyl-3-acetamido-3-phenyl propionate (2f). Yield 78% (529.0 mg), white solid (mixture of diastereomers), mp 133–134 °C [Found: C, 70.89; H, 6.29; N, 4.03. $C_{20}H_{21}NO_4$ requires C, 70.78; H, 6.24; N, 4.13%]; ν_{max} (KBr): 3311, 1724, 1691, 1651, 1597, 1352, 1097, 695, 543 cm^{-1}. Data for the major isomer (*syn*): δ_H (400 MHz, $CDCl_3$) 1.20 (3H, t, *J* 7.2 Hz, *Me*), 2.08 (3H, s, *Me*), 4.18 (2H, q, *J* 7.2 Hz, OCH_2), 4.98 (1H, d, *J* 4.0 Hz, C*H*), 5.91 (1H, dd, *J* 4.0, 9.2 Hz, C*H*), 7.15 (1H, t, *J* 7.2 Hz, Ph), 7.22 (3H, m, Ph), 7.30 (2H, d, *J* 7.6 Hz, Ph), 7.40 (1H, t, *J* 7.6 Hz, Ph), 7.47 (1H, d, *J* 9.2 Hz, N*H*), 7.54 (1H, t, *J* 8.4 Hz, Ph), 7.79 (2H, d, *J* 8.4 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 31.8, 41.8, 53.0, 53.3, 62.2, 126.6, 127.3, 128.0, 128.8, 129.0, 131.9, 134.2, 139.3.

4.2.21. Ethyl 2-benzoyl-3-acetamido-3-(trimethoxyphenyl) propionate (2g). Yield 80% (687 mg), white solid (mixture of diastereomers), mp 150–152 °C [Found: C, 64.21; H, 6.39; N, 3.15. $C_{23}H_{27}NO_7$ requires C, 64.32; H, 6.34; N, 3.26%]; ν_{max} (KBr): 3306, 2931, 1729, 1686, 1648, 1592, 1456, 1370, 1001, 733 cm^{-1}. Data for the major isomer (*syn*): δ_H (400 MHz, $CDCl_3$) 1.21 (3H, t, *J* 7.2 Hz, *Me*), 2.07 (3H, s, CO*Me*), 3.74 (3H, s, O*Me*), 3.75 (6H, s, 2×O*Me*), 4.17 (2H, q, *J* 7.2 Hz, OCH_2), 4.93 (1H, d, *J* 4.0 Hz, C*H*), 5.80 (1H, dd, *J* 4.0, 8.8 Hz, C*H*), 6.49 (2H, s, Ph), 7.41 (2H, t, *J* 8.0 Hz, Ph), 7.50 (1H, d, *J* 8.8 Hz, N*H*), 7.55 (1H, t, *J* 7.2 Hz, Ph), 7.77 (2H, d, *J* 7.2 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 14.2, 23.6, 53.5, 56.3, 56.6, 60.9, 62.4, 104.3, 128.5, 129.1, 134.3, 135.4, 153.4, 167.0, 199.5.

4.2.22. Methyl 2-acetyl-3-benzamido-3-phenyl propionate (2h). Yield 85% (553.0 mg), light yellow solid (mixture of diastereomers), mp 151–153 °C [Found: C, 70.25; H, 5.84; N, 4.39. $C_{19}H_{19}NO_4$ requires C, 70.14; H, 5.89; N, 4.30%]; ν_{max} (KBr): 3346, 2941, 1744, 1719, 1633, 1527, 1458, 1360, 1031, 700 cm^{-1}. Data for the major isomer (*syn*): δ_H (400 MHz, $CDCl_3$) 2.15 (3H, s, CO*Me*), 3.71 (3H, s, O*Me*), 4.21 (1H, d, *J* 4.4 Hz, C*H*), 5.91 (1H, dd, *J* 4.4, 9.6 Hz, C*H*), 7.30–7.32 (4H, m, Ph), 7.43 (2H, t, *J* 7.6 Hz, Ph), 7.49 (2H, d, *J* 7.6 Hz, Ph), 7.80 (2H, d, *J* 7.2 Hz, Ph), 7.88 (1H, d, *J* 9.6 Hz, N*H*); δ_C (100 MHz, $CDCl_3$) 30.1, 53.0, 53.3, 62.2, 126.6, 127.3, 128.0, 128.8, 129.0, 131.9, 134.2, 139.0, 166.9, 167.9, 204.5.

4.2.23. Methyl 2-(*o*-nitrobenzylidene) acetoacetate (3a). Yield 81% (404.0 mg), crystalline white solid (EtOAc–hexane), mp 90–93 °C (lit.[27] mp 102 °C) [Found: C, 57.71; H, 4.39; N, 5.74. $C_{12}H_{11}NO_5$ requires C, 57.83; H, 4.45; N, 5.62%]; ν_{max} (KBr): 3062, 2953, 1733, 1683, 1599, 1448, 1362, 1053, 736, 605 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.49 (3H, s, CO*Me*), 3.61 (3H, s, O*Me*), 7.43 (1H, t, *J* 7.6 Hz, Ph), 7.59 (1H, d, *J* 7.6 Hz, Ph), 7.69 (1H, t, *J* 7.6 Hz, Ph), 8.08 (1H, s, =C*H*), 8.24 (1H, d, *J* 7.6 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 26.3, 51.5, 124.3, 129.6, 129.8, 132.0, 133.3, 135.4, 139.8, 147.1, 166.9, 193.8.

4.2.24. Methyl 2-(*m*-nitrobenzylidene) acetoacetate (3b). Yield 80% (399.0 mg), crystalline solid (EtOAc–hexane), mp 151–152 °C (lit.[26] mp 148 °C) [Found: C, 57.72; H, 4.39; N, 5.71. $C_{12}H_{11}NO_5$ requires C, 57.83; H, 4.45; N, 5.62%]; ν_{max} (KBr): 3065, 1732, 1651, 1618, 1525, 1443, 1390, 1091, 735, 688 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.43 (3H, s, O*Me*), 3.87 (3H, s, O*Me*), 7.58 (1H, s, Ph), 7.59 (1H, t, *J* 7.6 Hz, Ph), 7.72 (1H, d, *J* 7.6 Hz, Ph), 8.25 (1H, d, *J* 8.4 Hz, Ph), 8.29 (1H, s, =C*H*); δ_C (100 MHz, $CDCl_3$) 27.0, 53.1, 123.9, 125.2, 130.2, 134.8, 135.2, 136.7, 138.6, 147.5, 168.0, 194.0.

4.2.25. Methyl 2-(*p*-nitrobenzylidene) acetoacetate (3c). Yield 82% (408.0 mg), yellow crystalline solid (EtOAc–hexane), mp 110–114 °C [Found: C, 57.74; H, 4.40; N, 5.54. $C_{12}H_{11}NO_5$ requires C, 57.83; H, 4.45; N, 5.62%]; ν_{max} (KBr): 1736, 1659, 1624, 1599, 1440, 1349, 1042, 817, 537 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 2.43 (3H, s, CO*Me*), 3.82 (3H, s, OMe), 7.56 (2H, d, *J* 8.8 Hz, Ph), 7.59 (1H, s, =C*H*), 8.23 (2H, d, *J* 8.8 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 27.1, 53.1, 109.9, 124.2, 130.1, 138.7, 139.6, 148.1, 167.0, 194.1.

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