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An investigation towards the diastereoselective synthesis of 3-acetoxy/methoxy/phthalimido-β-lactams using chiral imines

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ABSTRACT

The efficient diastereoselective synthesis of 3-acetoxy/methoxy/phthalimido-β-lactams **2**/**2′**, **3**/**3′** and **4**/**4′** respectively was performed using chiral imines **1** obtained from chiral amines. Factors (solvent, temperature, substituent, steric bulk) influencing the stereoselectivity and the diastereomeric ratio were also studied in detail. The diastereoselectivity of the two isomers was determined from the ratio of integral values of doublets of C3–H and C4–H and from the integral values of H in –*CH*(Me/Et)Ph/Np of the two diastereomers. Representative pairs of *cis*-diastereomers were separated by efficient column chromatography.

1. Introduction

With the discoveries of penicillin and cephalosporin a century ago, antibiotics have emerged as a milestone in medicinal chemistry. The core structure of the majority of antibiotics such as penems, carbapenems, clavulanic acids and monobactams is mainly the β-lactam nucleus. However, for the past 50–60 years, the rapid emergence of bacterial strains resistant against these antibiotics[1] renewed the interest of organic chemists in identifying new families of antibiotics. Thus, there is a relentless demand for more efficient novel β-lactam antibiotics, which are stable to β-lactamases and possess high potency. Literature studies successfully demonstrated that enantiopure β-lactams are versatile intermediates for the asymmetric synthesis of a variety of protein and non-protein amino acids, peptides, peptide turn mimetics, peptidomimetics, taxoid antitumour agents, heterocycles and other types of compounds of biological and medicinal interest.[2]

The Staudinger reaction, a convergent strategy, has endured as a particularly attractive approach for generating this family of compounds. In spite of the high level of achievement reached in the Staudinger reaction,[3] the subject still continues to be an area of active research. Over the past few years, this reaction has been extensively studied using a combination of either chiral ketenes and achiral imines[4] or achiral ketenes and chiral imines[5–7] and also chiral ketenes with chiral imines[8] to achieve good diastereoselectivity. Very recently, Zakaszewska et al.[9] have reported the stereoselective synthesis of 3-acyl-β-lactams from acyl ketenes generated from Meldrum's acid derivatives and chiral aldimines. Roje et al.[10] have carried out the synthesis of chiral 3-amino-β-lactams and β-lactam guanidines *via* cyclocondensation of chiral ester enolate with imines. Kommidi et al.[11] described the one-pot efficient synthesis of highly chiral and reactive carbapenem chalcone derivatives. Chiral imines with a chiral auxiliary placed at the nitrogen have been used in the enantioselective synthesis of versatile 2-isocephem synthon[12] **A** and 7-TES-baccatin III **B**, for the preparation of taxotere, a promising anticancer drug (Fig. 1).

In our previous studies, we reported on novel β-lactam precursors,[13a–c] 3-thio/seleno-β-lactams and their Lewis acid mediated functionalization,[13d–j] stereoselective *cis*- and *trans*-alkoxy-β-lactams,[13k] spirocyclic-β-lactams,[13l–n] α-keto-β-lactams,[13o] bicyclic-β-lactams,[13p] novel 4-pyrazolyl-β-lactams,[13q] 4-pyrazolylspirocyclic-β-lactams[13r] and (*E*)- and (*Z*)-3-allylidene-β-lactams.[13s,t]

In continuation of the above and our interests in the synthesis of novel functionalized β-lactams, we investigated the potential of chiral imines in the synthesis of novel chiral β-lactams. We considered studying the diastereoselective synthesis of 3-acetoxy/methoxy/phthalimido-β-lactams by employing chiral imines, where the chiral auxiliary is placed at the nitrogen atom of the imine. We decided to use chiral auxiliaries, which would facilitate high stereocontrol in the Staudinger reaction and could be removed in one step from the β-lactam nitrogen. Moreover, *N*-unsubstituted (R^2 = H) β-lactams are also important chiral building blocks for the synthesis of β-lactam antibiotics.[14] Various chiral amines were employed for the diastereoselective synthesis of 3-acetoxy/methoxy/phthalimido-β-lactams **2**/**2′**, **3**/**3′** and **4**/**4′**, respectively.

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E-mail address: amanbhalla@pu.ac.in (A. Bhalla).

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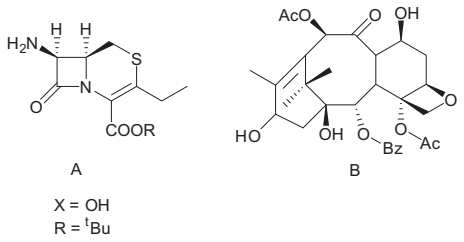


Figure 1.

2. Results and discussion

In the literature, that are various methods available for the synthesis of chiral imines from chiral amines using aldehydes or alcohols under different reaction conditions.[7,15–20] The yields of the chiral imines using these reported methods decreased with prolonged reaction time. Therefore, we pursued the synthesis of the required chiral Schiff's bases **1a–j** from aldehydes and chiral amines using toluene in the presence of molecular sieves 4 Å under reflux conditions. The reaction was completed in less than 2 h and afforded **1a–j** in high yields. Compounds **1a–j** were used as such without further purification (Scheme 1, Table 1). The structures of these chiral Schiff's bases or imines were confirmed on the basis of their spectral data (IR and ^{1}H NMR).

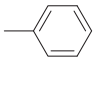
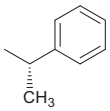
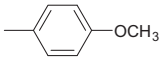
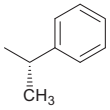
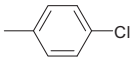
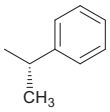
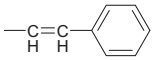
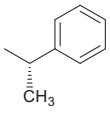
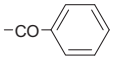
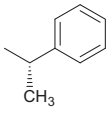
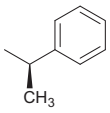
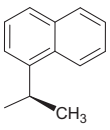
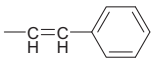
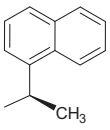
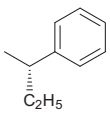
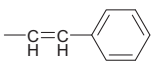
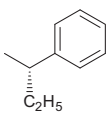
The influence of solvent was investigated on the ketene-imine cycloaddition reaction between acetoxyacetyl chloride and chiral imines **1a** and **1f** in the presence of triethylamine (Scheme 2) and the results are summarized in Table 2 (entries 1–4, 9–11). The reaction resulted in the formation of two diastereomeric *cis*-β-lactams **2a/2′a** [R^2 = –CH(Me)C_6H_5 (*S*)] and **2f/2′f** [R^2 = –CH(Me)C_6H_5 (*R*)] respectively. The chloroform solvent among the other solvents provided the best diastereomeric ratio, with good yield at 0 °C.

With these optimized reaction conditions in hand (chloroform, 0 °C), the scope of this reaction was studied further by using other chiral Schiff's bases **1b–e,g–j** and the results are reported in Table 2 (entries 5–8, 12–17).

Since, the ketene generated *in situ* from acetoxyacetyl chloride and Et_3N is a Bose-Evans ketene,[21] it has a preference to form *cis*-β-lactams. The mixture of *cis*-diastereomers was purified by column chromatography. Further, the pair of *cis*-diastereomers **2h/2′h** were separated using efficient column chromatography. Since, we were only interested in determining the diastereomeric ratio of major and minor diastereomers **2/2′**, no further attempts were made to separate these diastereomers which are easily separable by employing chromatographic techniques except **2h/2′h** as the representative ones. The structures of these β-lactams **2/2′** were established on the basis of spectral data (IR, ^{1}H NMR and ^{13}C NMR) and CHN analysis. IR spectra showed the absorption bands in the range of 1735–1755 cm^{-1} (C=O) and ^{1}H NMR of the β-lactams showed the coupling constant (*J*) between C3–H and C4–H in the range of 4–6 Hz, thereby, confirming the *cis*-stereochemistry of protons at C–3 and C–4 of these β-lactams.

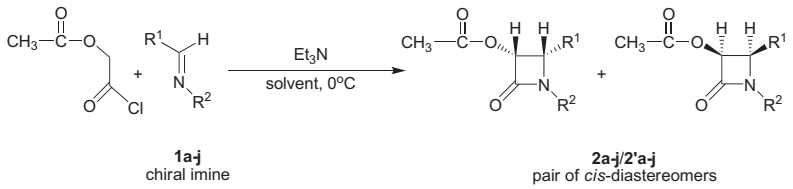
Scheme 1. Preparation of chiral Schiff's bases **1a–j**.

Table 1
Synthesis of chiral Schiff's bases **1a–j**

Entry	R^1	R^2	Products	Yield (%)
1			**1a**	98
2			**1b**	97
3			**1c**	98
4			**1d**	99
5			**1e**	92
6			**1f**	93
7			**1g**	98
8			**1h**	98
9			**1i**	97
10			**1j**	99

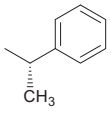
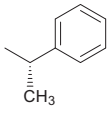
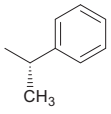
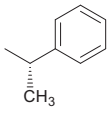
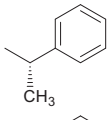
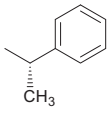
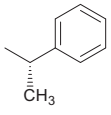
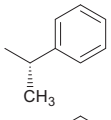
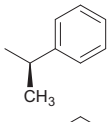
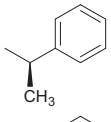
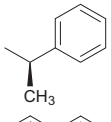
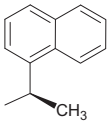
The effect of substituent R^1 *i.e.* the aldehyde component of the Schiff's base on the diastereoselectivity of the *cis*-3-acetoxy-β-lactams **2/2′** [R^2 = CH(Me)C_6H_5 (*S*)] was studied in chloroform at 0 °C (Table 2, entries 5–8). The diastereomeric ratios were found to be almost similar while the chemical yield significantly decreased for R^1 = –C_6H_4Cl (*p*) (Table 2, entry 6). However, both the diastereomeric ratio and chemical yields lowered for R^1 = –COC_6H_5 (Table 2, entry 8).

In order to study the effect of the steric bulk of the alkyl and the aryl groups on the Schiff's base, [2+2] cycloaddition reactions were carried out with chiral imines **1g–j** in benzene and chloroform as the solvent, thus forming *cis*-3-acetoxy-β-lactams **2g–j/2′g–j**; the results are summarized in Table 2 (entries 12–17). It was found that the yield and diastereomeric ratio was observed to drop for **2f/2′f** [R^2 = –CH(Me)C_6H_5 (*R*)] in comparison to **2a/2′a** [R^2 = –CH(Me)C_6H_5 (*S*)] (Table 2, entry 1,9). The diastereoselectivity was improved when we used a bulky group such as naphthyl (Table 2, entries 12–14), however increasing the steric bulk of the alkyl group at the stereogenic center of the imine had no influence on



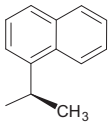
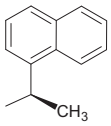
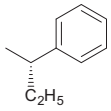
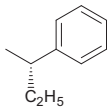
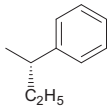
Scheme 2. Synthesis of chiral *cis*-3-acetoxy-β-lactams **2a–j/2′a–j**.

Table 2
Effect of solvent, substituent and steric bulk on the diastereoselective synthesis of chiral *cis*-3-acetoxy-β-lactams **2a–j/2′a–j** at 0 °C

Entry	R^1	R^2	Solvent	Products	Diastereomeric ratio (Major: Minor)	Yield[a] (%)
1	$-C_6H_5$		$CHCl_3$	**2a/2′a**	74:26	95
2	$-C_6H_5$		CH_2Cl_2	**2a/2′a**	68:32	65
3	$-C_6H_5$		$C_6H_5CH_3$	**2a/2′a**	72:28	89
4	$-C_6H_5$		C_6H_6	**2a/2′a**	73:27	91
5	$-C_6H_4OCH_3$ (p)		$CHCl_3$	**2b/2′b**	75:25	90
6	$-C_6H_4Cl$ (p)		$CHCl_3$	**2c/2′c**	71:29	22
7	$-CH{=}CH\text{-}C_6H_5$		$CHCl_3$	**2d/2′d**	75:25	71
8	$-COC_6H_5$		$CHCl_3$	**2e/2′e**	55:45	60
9	$-C_6H_5$		$CHCl_3$	**2f/2′f**	70:30	88
10	$-C_6H_5$		CH_2Cl_2	**2f/2′f**	68:32	61
11	$-C_6H_5$		C_6H_6	**2f/2′f**	70:30	76
12	$-C_6H_5$		$CHCl_3$	**2g/2′g**	79:21	90

(*continued on next page*)

Table 2 (*continued*)

Entry	R^1	R^2	Solvent	Products	Diastereomeric ratio (Major: Minor)	Yield[a] (%)
13	$-C_6H_5$		C_6H_6	**2g/2′g**	73:27	76
14	$-CH{=}CH-C_6H_5$		$CHCl_3$	**2h/2′h**	82:18	44
15	$-C_6H_5$		$CHCl_3$	**2i/2′i**	72:28	66
16	$-C_6H_5$		C_6H_6	**2i/2′i**	71:29	31
17	$-CH{=}CH-C_6H_5$		$CHCl_3$	**2j/2′j**	71:29	50

[a] The yields are reported after column chromatographic purification.

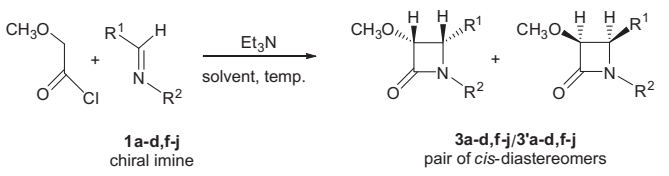
the diastereoselectivity, in addition to decreasing the yield (Table 2, entries 15–17).

To the best of our knowledge, this is the highest diastereoselectivity obtained in the case of *cis*-3-acetoxy-β-lactams. On the basis of the above studies, we concluded that the diastereoselectivity in the 3-acetoxy-β-lactams can be enhanced by performing the reaction in chloroform at lower temperature using sterically hindered Schiff's base with steric bulk being present at the *N*-position.

To further strengthen these results, the diastereoselective synthesis of chiral 3-methoxy-β-lactams was pursued by the [2+2] cycloaddition reactions of chiral imines **1a–d,f–j** with methoxyacetyl chloride in the presence of triethylamine (Scheme 3).

Initially, the solvent and temperature optimization was carried out on the reaction of methoxyacetyl chloride with chiral Schiff's base **1a** the results are summarized in Table 3 (entries 1–7). The reaction yielded two diastereomeric *cis*-β-lactams **3a**/**3′a** (major/minor) respectively. The best diastereomeric ratio (68:32) was obtained in *N,N′*-dimethylformamide but with moderate chemical yield (41%) (Table 3, entry 5). However, the best yield (89%) and a diastereoselectivity of 57:43 were obtained at room temperature using chloroform as the solvent (Table 3, entry 2).

The scope of this reaction was also explored in chloroform at room temperature using other chiral Schiff's bases **1b–d,f–j**. The reaction afforded novel *cis*-3-methoxy-β-lactams **3b–d,f–j/3′b–d, f–j** and the results are reported in Table 3 (entries 8–18). The mixture of *cis*-3-methoxy-β-lactams was purified by column chromatography. In addition, the representative β-lactams **3g/3′g** and **3h/3′h** were separated by column chromatography as pure isomers. The structures of these β-lactams were established on the basis of spectroscopic data (IR, ^{1}H NMR and ^{13}C NMR) and CHN analysis.



Scheme 3. Synthesis of chiral *cis*-3-methoxy-β-lactams **3a–d,f–j/3′a–d,f–j**.

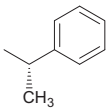
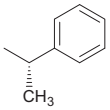
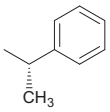
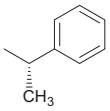
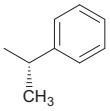
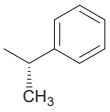
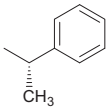
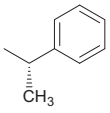
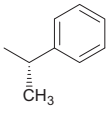
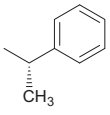
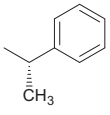
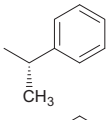
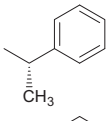
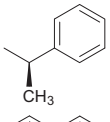
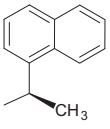
The R^1substituent *i.e.* the aldehyde component of the imine was found to have a profound effect on the stereoselectivity of the Staudinger reaction as shown in Table 3 (entries 8–13). The best diastereoselectivity was achieved with R^1 = -$C_6H_4OCH_3$ (*p*) (72:28) and –CH=CH-C_6H_5 (67:33) in chloroform at room temperature (Table 3, entries 8,12) however replacing the phenyl group with $-C_6H_4Cl$ (*p*) led to a decrease in the yield of *cis*-3-methoxy-β-lactams (Table 3, entries 10,11).

In order to investigate the trend in the diastereomeric ratios, imines with opposite absolute configuration **1f** and sterically demanding imines **1g–j** were made to undergo [2 + 2] cycloaddition reactions with methoxyacetyl chloride in chloroform at rt (Table 3, entries 14–18). It was observed that both the yield and diastereomeric ratio slightly decreased when R^2 = –CH(Me)C_6H_5 (*R*) (Table 3, entry 14) in comparison to R^2 = –CH(Me)C_6H_5 (*S*) (Table 3, entry 2). Replacing the aryl group from phenyl to naphthyl, keeping the alkyl group as methyl, gave excellent diastereoselectivity (84:16) (Table 3, entry 16). To the best of our knowledge, this is the highest diastereomeric ratio obtained for chiral *cis*-3-methoxy-β-lactams synthesized by using chiral imines. Increasing the steric bulk of the alkyl group *i.e.* replacing methyl group by ethyl led to a slightly improved diastereomeric ratio (63:37) (Table 3, entry 18).

From these studies, we concluded that the best diastereomeric ratio in case of 3-methoxy-azetidin-2-ones can be achieved at room temperature using *N,N′*-dimethylformamide as the solvent while chloroform gave the best yield. Similarly, the use of chiral Schiff's base **1h** bearing (R^1 = -CH=CH-C_6H_5 and R^2 = -CH(Me) $C_{10}H_7$) provided excellent diastereoselectivity. In comparison to 3-acetoxy-β-lactams, the R^1 component has an impact on the diastereoselectivity of 3-methoxy-β-lactams.

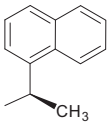
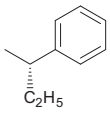
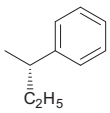
The asymmetric Staudinger reaction of chiral imines derived from (*R*)-(+)-1-(phenyl)ethylamine with phthalimido ketene has also been reported in the literature.[12,22] However, the potential of other chiral imines in the synthesis of chiral 3-phthalimido-β-lactams still remains unexplored. Therefore, the use of various chiral imines **1a–d** was further investigated for the cycloaddition reaction with phthalimidoacetyl chloride (Scheme 4).

Table 3
Effect of solvent, temperature, substituent, steric bulk on the diastereoselective synthesis of chiral *cis*-3-methoxy-β-lactams **3a–d,f–j/3′a–d,f–j**

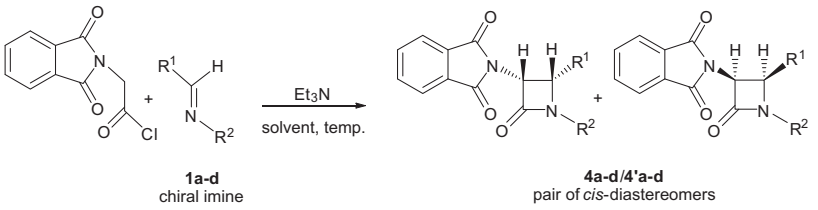
Entry	R^1	R^2	Solvent	Temperature (°C)	Products	Diastereomeric ratio (Major: Minor)	Yield[a] (%)
1	$-C_6H_5$		CH_2Cl_2	rt	**3a/3′a**	55:45	67
2	$-C_6H_5$		$CHCl_3$	rt	**3a/3′a**	57:43	89
3	$-C_6H_5$		$C_6H_5CH_3$	rt	**3a/3′a**	52:48	80
4	$-C_6H_5$		C_6H_6	rt	**3a/3′a**	52:48	62
5	$-C_6H_5$		DMF	rt	**3a/3′a**	68:32	41
6	$-C_6H_5$		CCl_4	rt	**3a/3′a**	50:50	45
7	$-C_6H_5$		$CHCl_3$	Reflux	**3a/3′a**	52:48	67
8	$-C_6H_4OCH_3$ (p)		$CHCl_3$	rt	**3b/3′b**	72:28	71
9	$-C_6H_4OCH_3$ (p)		$CHCl_3$	Reflux	**3b/3′b**	50:50	58
10	$-C_6H_4Cl$ (p)		$CHCl_3$	rt	**3c/3′c**	57:43	52
11	$-C_6H_4Cl$ (p)		$CHCl_3$	Reflux	**3c/3′c**	51:49	39
12	-CH=CH–C_6H_5		$CHCl_3$	rt	**3d/3′d**	67:33	54
13	-CH=CH–C_6H_5		$CHCl_3$	Reflux	**3d/3′d**	54:46	60
14	$-C_6H_5$		$CHCl_3$	rt	**3f/3′f**	52:48	60
15	$-C_6H_5$		$CHCl_3$	rt	**3g/3′g**	56:44	63

(*continued on next page*)

Table 3 (*continued*)

Entry	R^1	R^2	Solvent	Temperature (°C)	Products	Diastereomeric ratio (Major: Minor)	Yield[a] (%)
16	–CH=CH–C_6H_5		$CHCl_3$	rt	**3h/3′h**	84:16	56
17	–C_6H_5		$CHCl_3$	rt	**3i/3′i**	56:44	62
18	–CH=CH–C_6H_5		$CHCl_3$	rt	**3j/3′j**	63:37	47

[a] The yields are reported after column chromatographic purification.



Scheme 4. Synthesis of *cis*-3-phthalimido-β-lactams **4a–d/4′a–d**.

Initial studies involved the synthesis of chiral 3-phthalimido-β-lactams using chiral imine **1a** [R^1 = –C_6H_5; R^2 = –CH(Me)C_6H_5 (*S*)] and phthalimidoacetyl chloride in presence of triethylamine in chloroform at 0 °C (Scheme 4). This resulted in the formation of a pair of *cis*-diastereomers **4a/4′a** (Table 4, entry 2). Other [2+2]cycloadditions involving chiral imines **1a–d** and phthalimidoacetyl chloride were carried out under different reaction conditions to afford *cis*-3-phthalimido-β-lactams **4a–d/4′a–d** and the results are summarized in Table 4.

With our earlier optimized conditions (chloroform, 0 °C), a good diastereomeric ratio (77:23) and yield (72%) were obtained with R^1 = –CH=CH-C_6H_5 (Table 4, entry 8). However, the highest diastereoselectivity was observed with R^1 = –C_6H_5 (83:17) using benzene as a solvent at 0 °C (Table 4, entry 3).

3. Conclusion

In conclusion, we have investigated the potential of chiral Schiff's bases **1** obtained from chiral amines in the diastereoselective synthesis of *cis*-3-acetoxy/methoxy/phthalimido-β-lactams **2/2′**, **3/3′** and **4/4′**. All of the factors which influence the stereoselectivity in the asymmetric Staudinger reaction, *i.e.* the solvent, temperature and substituents, have been studied in detail. Moreover, the role played by the steric effects, *i.e.* the steric bulk of the groups placed at the stereogenic center of the chiral amines, in determining the diastereomeric ratios of resulting chiral β-lactams has also been explored. In addition, the reported pair of *cis* diastereomers were separated with efficient column chromatography to afford the pure isomers.

4. Experimental

4.1. General

^{1}H NMR (400 MHz and 300 MHz), ^{13}C NMR (100 MHz and 75 MHz) were recorded using BRUKER or JEOL 400 MHz and 300 MHz NMR spectrometers respectively. The chemical shifts are ^{1}H NMR (400 MHz and 300 MHz), ^{13}C NMR (100 MHz and 75 MHz) were recorded using BRUKER or JEOL 400 MHz and 300 MHz NMR spectrometers respectively. The chemical shifts are expressed in δ values (ppm) using tetramethylsilane as an internal standard. Infrared spectra were recorded using Perkin-Elmer Model 1430 spectrophotometer with potassium bromide (KBr) plates or Nujol with NaCl optic plates and are reported in cm^{-1}. The elemental analysis (C, H, N) was carried out using a PERKIN–ELMER 2400 elemental analyzer. Optical rotations were measured on Rudolph Autopol V polarimeter at room temperature in chloroform. Column chromatography was performed using Merck

Table 4
Diastereomeric selectivity of *cis*-3-phthalimido-β-lactams **4a–d/4′a–d** using (*S*)-(-)-1-(phenyl)ethylimines

Entry	R^1	Solvent	Temp. (°C)	Products	Diastereomeric ratio (Major: Minor)	Yield[a] (%)
1	–C_6H_5	CH_2Cl_2	0	**4a/4′a**	65:35	60
2	–C_6H_5	$CHCl_3$	0	**4a/4′a**	66:34	72
3	–C_6H_5	C_6H_6	0	**4a/4′a**	83:17	65
4	–$C_6H_4OCH_3$ (*p*)	CH_2Cl_2	0	**4b/4′b**	63:37	88
5	–C_6H_4Cl (*p*)	CH_2Cl_2	0	**4c/4′c**	73:27	70
6	–C_6H_4Cl (*p*)	CH_2Cl_2	rt	**4c/4′c**	61:39	84
7	–C_6H_4Cl (*p*)	CH_2Cl_2	Reflux	**4c/4′c**	72:28	76
8	–CH=CH–C_6H_5	$CHCl_3$	0	**4d/4′d**	77:23	72

[a] The yields are reported after column chromatographic purification.

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Silica Gel (60–120 mesh) and eluted with ethyl acetate: hexanes mixtures. Thin-layer chromatography (TLC) was performed using Merck Silica Gel G. For visualization, TLC plates were stained with iodine vapours. All the melting points are uncorrected and are expressed in degree centigrade (°C). Melting points were determined with a Thomas- Hoover capillary melting point apparatus. The synthesis and reactions of β-lactams were carried out under dry and deoxygenated nitrogen atmosphere. Phosphorus oxychloride (Merck), triethylamine (Qualigen), ethyl acetoacetate (Merck), chiral amines (Sigma Aldrich), acetoxy/methoxy/phthalimidoacetyl chloride (Sigma Aldrich) and all other commercially available compounds/reagents/solvents were of reagent grade quality and used without any further purification. Dimethylformamide was dried and distilled over anhydrous calcium chloride ($CaCl_2$). Dichloromethane, chloroform and carbon tetrachloride were dried and distilled over anhydrous phosphorus pentoxide (P_2O_5). Toluene and benzene were distilled under N_2 from sodium-benzophenone immediately before use.

4.2. General procedure for the preparation of chiral imines 1a–j

To a solution of chiral amine (10 mmol) in toluene (50 mL) was added the appropriate aldehyde (10 mmol) and the reaction mixture was allowed to reflux for 2 h using Dean–Stark apparatus. The progress of the reaction was checked by TLC. After the completion of the reaction, the solvent was evaporated under vacuum to yield imine **1**. The spectroscopic data of imines **1a**,**f**,[23] **1b**-**d**,[8] **1e**,[24] **1g**,[16] **1h**[6] have been reported previously in the cited references.

4.2.1. Benzylidene-[(*S*)-1′-phenylpropyl]amine 1i

Yield 97%; thick oil; IR (ATR) ν_{max} 1702 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 8.19 (1H, s, *H*C═N), 7.06–7.75 (10H, m, ArH), 4.04 (1H, t, J = 6.9 Hz, C*H*CH_2), 1.79–1.89 (2H, m, C*H*$_2$$CH_3$), 0.80 (3H, t, J = 7.2 Hz, CH_2C*H*$_3$); Anal. Calcd. for $C_{16}H_{17}N$: C, 86.05; H, 7.67; N, 6.27. Found: C, 86.03; H, 7.65; N, 6.26.

4.2.2. (3′-Phenylallylidene)-[(*S*)-1′-phenylpropyl]amine 1j

Yield 99%; thick yellow oil; IR (ATR) ν_{max} 1680, 1634 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.91 (1H, d, J = 8.1 Hz, *H*C = N), 6.73–7.32 (12H, m, ArH, CH═C*H*Ph and C*H*═CHPh), 3.89 (1H, t, J = 6.9 Hz, C*H*CH_2), 1.76–1.86 (2H, m, C*H*$_2$$CH_3$), 0.74 (3H, t, J = 7.2 Hz, CH_2C*H*$_3$); Anal. Calcd. for $C_{18}H_{19}N$: C, 86.70; H, 7.68; N, 5.62. Found: C, 86.68; H, 7.67; N, 5.60.

4.3. General procedure for the preparation of chiral 3-acetoxy/methoxy/phthalimidoazetidin-2-ones 2a–j/2′a–j, 3a–d,f–j/3′a–d, f–j and 4a–d/4′a–d

To a solution of chiral imine **1a–j** (1.20 mmol) and triethylamine (5.98 mmol) in 20 mL dry solvent was added dropwise under a nitrogen atmosphere, at 0 °C/rt/reflux, a solution of acetoxy/methoxy/phthalimidoacetyl chloride (1.19 mmol) in 5 mL of dry solvent with constant stirring. The reactants were stirred overnight at room temperature. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was washed successively with water (3 × 10 mL), 5% $NaHCO_3$ (10 mL) and brine (10 mL). The organic layer was separated and dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the crude product was purified by column chromatography using silica gel eluting with ethyl acetate: hexane.

4.3.1. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-acetoxy-4-phenyl-azetidin-2-one 2a/2′a

IR (ATR) ν_{max} 1752, 1603 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 7.05–7.27 (10H, m, ArH), 5.54 (1H, d, J = 4.8 Hz, C3-H), 4.96 (1H, q, J = 7.2 Hz, C*H*CH_3), 4.54 (1H, d, J = 4.8 Hz, C4-H), 1.60 (3H, s, C*H*$_3$COO), 1.34 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 7.05–7.27 (10H, m, ArH), 5.58 (1H, d, J = 4.8 Hz, C3-H), 4.58 (1H, d, J = 4.8 Hz, C4-H), 4.28 (1H, q, J = 6.9 Hz, C*H*CH_3), 1.80 (3H, d, J = 7.2 Hz, CHC*H*$_3$), 1.58 (3H, s, C*H*$_3$COO); ^{13}C NMR (75 MHz, $CDCl_3$, Both isomers) δ 169.1, 164.8, 139.1, 134.0, 128.8, 128.8, 128.4,128.2, 128.1, 127.2, 126.8, 76.4, 60.9, 54.8, 52.4, 19.9, 19.8, 19.1; Anal. Calcd. for $C_{19}H_{19}NO_3$: C, 73.77; H, 6.19; N, 4.53. Found: C, 73.75; H, 6.18; N, 4.51.

4.3.2. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-acetoxy-4-(4′-methoxyphenyl)azetidin-2-one 2b/2′b

IR (ATR) ν_{max} 1752, 1663, 1612 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 6.72–7.29 (9H, m, ArH), 5.54 (1H, d, J = 4.5 Hz, C3-H), 4.98 (1H, q, J = 7.2 Hz, C*H*CH_3), 4.53 (1H, d, J = 4.5 Hz, C4-H), 3.74 (3H, s, OC*H*$_3$), 1.64 (3H, s, C*H*$_3$COO), 1.30 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 6.72–7.29 (9H, m, ArH), 5.60 (1H, d, J = 4.5 Hz, C3-H), 4.57 (1H, d, J = 4.5 Hz, C4-H), 4.31 (1H, q, J = 7.2 Hz, C*H*CH_3), 3.72 (3H, s, OC*H*$_3$), 1.75 (3H, d, J = 7.2 Hz, CHC*H*$_3$), 1.62 (3H, s, C*H*$_3$COO); ^{13}C NMR (75 MHz, $CDCl_3$, Both isomers) δ 169.1, 169.0, 164.7, 159.8, 140.4, 139.1, 129.9, 129.7, 128.7, 128.7, 128.0, 127.8, 127.2, 126.7, 125.8, 124.5, 113.5, 113.4, 76.4, 60.4, 60.3, 55.2, 54.4, 52.3, 19.9, 19.6, 19.0; Anal. Calcd. for $C_{20}H_{21}NO_4$: C, 70.78; H, 6.24; N, 4.13. Found: C, 70.75; H, 6.24; N, 4.11.

4.3.3. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-acetoxy-4-(4′-chlorophenyl)azetidin-2-one 2c/2′c

IR (ATR) ν_{max} 1754, 1598 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, Major isomer) δ 7.04–7.33 (9H, m, ArH), 5.64 (1H, d, J = 4.7 Hz, C3-H), 5.05 (1H, q, J = 7.2 Hz, C*H*CH_3), 4.62 (1H, d, J = 4.7 Hz, C4-H), 1.72 (3H, s, C*H*$_3$COO), 1.40 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^1H NMR (400 MHz, $CDCl_3$, Minor isomer) δ 7.04–7.33 (9H, m, ArH), 5.69 (1H, d, J = 4.7 Hz, C3-H), 4.68 (1H, d, J = 4.7 Hz, C4-H), 4.45 (1H, q, J = 6.8 Hz, C*H*CH_3), 1.81 (3H, d, J = 7.2 Hz, CHC*H*$_3$), 1.70 (3H, s, C*H*$_3$COO); ^{13}C NMR (100 MHZ, $CDCl_3$, Both isomers) δ 169.1, 169.0, 164.5, 164.5, 138.9, 134.6, 134.4, 132.7, 131.4, 130.0, 129.7, 128.8, 128.8, 128.3, 128.2, 128.0, 127.1, 126.8, 76.4, 76.2, 60.3, 60.1, 54.4, 52.6, 20.5, 19.9, 19.4, 19.0; Anal. Calcd. for $C_{19}H_{18}NO_3Cl$: C, 66.38; H, 5.28; N, 4.07. Found: C, 66.34; H, 5.25; N, 4.05.

4.3.4. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-acetoxy-4-styrylazetidin-2-one 2d/2′d

IR (ATR) ν_{max} 1748, 1653, 1601, 1522 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 7.10–7.28 (10H, m, ArH), 6.40 (1H, d, CH═C*H*Ph), 5.91 (1H, dd, J = 15.9, 8.7 Hz, C*H*═CHPh), 5.57 (1H, d, C3-H), 4.88 (1H, q, J = 6.9 Hz, C*H*CH_3), 4.17 (1H, dd, J = 9.0, 4.8 Hz, C4-H), 1.957 (3H, s, CH_3COO), 1.57 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 7.10–7.28 (10H, m, ArH), 6.35 (1H, d, CH═C*H*Ph), 5.72 (1H, dd, J = 15.9, 8.7 Hz, C*H*═CHPh), 5.59 (1H, d, C3-H), 4.55 (1H, q, J = 7.2 Hz, C*H*CH_3), 4.23 (1H, dd, J = 8.7, 4.8 Hz, C4-H), 1.951 (3H, s, CH_3COO), 1.70 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^{13}C NMR (75 MHz, $CDCl_3$, Both isomers) δ 169.7, 169.6, 164.5, 164.3, 140.8, 139.7, 137.0, 136.6, 136.1, 136.0, 129.1, 129.1, 129.0, 128.9, 128.7, 128.7, 128.3, 128.2, 127.5, 127.3, 126.9, 123.7, 122.5, 76.5, 76.5, 60.1, 59.6, 53.5, 52.6, 20.6, 19.7, 19.1; Anal. Calcd. for $C_{21}H_{21}NO_3$: C, 75.20; H, 6.31; N, 4.18. Found: C, 75.15; H, 6.26; N, 4.14.

4.3.5. (1′*S*,3*R*,4*R*)- and (1′*S*,3*S*,4*S*)-1-(1′-Phenylethyl)-3-acetoxy-4-benzoylazetidin-2-one 2e/2′e

IR (ATR) ν_{max} 1760, 1694, 1658, 1598 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, Major isomer) δ 7.26–7.77 (10H, m, ArH), 6.13 (1H, d, J = 5.3 Hz, C3-H), 5.00 (1H, d, J = 5.3 Hz, C4-H), 4.72 (1H, q, J = 7.3 Hz, C*H*CH_3), 1.72 (3H, d, J = 7.0 Hz, CHC*H*$_3$), 1.64 (3H, s, C*H*$_3$COO); ^1H NMR (400 MHz, $CDCl_3$, Minor isomer) δ 7.26–7.77

(10H, m, ArH), 6.21 (1H, d, J = 5.3 Hz, C3-H), 5.18 (1H, q, J = 7.4 Hz, C*H*CH$_3$), 4.97 (1H, d, J = 5.3 Hz, C4-H), 1.95 (3H, d, J = 7.1 Hz, CHC*H*$_3$), 1.65 (3H, s, C*H*$_3$COO); ^{13}C NMR (75 MHz, CDCl$_3$, Both isomers) δ 169.7, 169.6, 164.5, 164.3, 140.8, 139.7, 137.0, 136.6, 136.1, 136.0, 129.1, 129.1, 129.0, 128.9, 128.7, 128.7, 128.3, 128.2, 127.5, 127.3, 126.9, 123.7, 122.5, 76.5, 76.5, 60.1, 59.6, 53.5, 52.6, 20.6, 19.7, 19.1; Anal. Calcd. for $C_{20}H_{19}NO_4$: C, 71.20; H, 5.68; N, 4.15. Found: C, 71.15; H, 5.64; N, 4.11.

4.3.6. (1′*R*,3*R*,4*S*)- and (1′*R*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-acetoxy-4-phenylazetidin-2-one 2f/2′f

IR (ATR) ν_{max} 1753, 1602 cm^{-1} ^{1}H NMR (400 MHz, CDCl$_3$, Major isomer) δ 7.15–7.34 (10H, m, ArH), 5.65 (1H, d, J = 4.7 Hz, C3-H), 5.07 (1H, q, J = 7.1 Hz, C*H*CH$_3$), 4.66 (1H, d, J = 4.7 Hz, C4-H), 1.66 (3H, s, C*H*$_3$COO), 1.38 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^{1}H NMR (400 MHz, CDCl$_3$, Minor isomer) δ 7.15–7.34 (10H, m, ArH), 5.70 (1H, d, J = 4.6 Hz, C3-H), 4.70 (1H, d, J = 4.7 Hz, C4-H), 4.41 (1H, q, J = 7.3 Hz, C*H*CH$_3$), 1.84 (3H, d, J = 7.2 Hz, CHC*H*$_3$), 1.64 (3H, s, C*H*$_3$COO), ^{13}C NMR (100 MHz, CDCl$_3$, Both isomers) δ 169.1, 169.0, 164.7, 140.3, 139.1, 134.0, 132.7, 128.8, 128.7, 128.6, 128.5, 128.4, 128.1, 128.0, 127.9, 127.1, 126.7, 76.5, 76.3, 60.9, 60.7, 54.6, 52.4, 19.8, 19.7, 19.0; Anal. Calcd. for $C_{19}H_{19}NO_3$: C, 73.77; H, 6.19; N, 4.53. Found: C, 73.70; H, 6.20; N, 4.55.

4.3.7. (1′*R*, 3*R*, 4*S*)- and (1′*R*, 3*S*, 4*R*)-1-(1′-Naphthylethyl)-3-acetoxy-4-phenylazetidin-2-one 2g/2′g

White solid; IR (ATR) ν_{max} 1748, 1711, 1599 cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$, Major isomer) δ 6.86–8.01 (12H, m, ArH), 5.87 (1H, q, J = 6.9 Hz, C*H*CH$_3$), 5.42 (1H, d, J = 4.8 Hz, C3-H), 4.01 (1H, d, J = 4.8 Hz, C4-H), 1.58 (3H, s, C*H*$_3$COO), 1.48 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^{1}H NMR (300 MHz, CDCl$_3$, Minor isomer) δ 6.85–8.01 (12H, m, ArH), 5.66 (1H, d, J = 4.5 Hz, C3-H), 5.29 (1H, q, J = 6.9 Hz, C*H*CH$_3$), 4.66 (1H, d, J = 4.8 Hz, C4-H), 1.90 (3H, d, J = 7.2 Hz, CHC*H*$_3$), 1.54 (3H, s, C*H*$_3$COO); ^{13}C NMR (75 MHz, CDCl$_3$, Both isomers) δ 169.5, 169.3, 165.4, 165.1, 135.5, 134.5, 134.2, 133.6, 132.8, 131.4, 129.4, 129.4, 129.3, 129.1, 129.0, 128.9, 128.7, 128.6, 128.3, 128.3, 128.2, 127.6, 127.6, 126.9, 126.5, 126.4, 126.1, 125.6, 125.3, 125.2, 124.6, 124.6, 124.3, 122.9, 76.5, 76.5, 61.0, 60.9, 50.2, 47.5, 28.5, 20.2, 20.2, 19.3, 19.3; Anal. Calcd. for $C_{23}H_{21}NO_3$: C, 76.86; H, 5.89; N, 3.90. Found: C, 76.84; H, 5.84, N, 3.91.

4.3.8. (1′*R*, 3*R*, 4*S*)- and (1′*R*, 3*S*, 4*R*)-1-(1′-Naphthylethyl)-3-acetoxy-4-styrylazetidin-2-one 2h/2′h

IR (ATR) ν_{max} 1750, 1652, 1599, 1512 cm^{-1}; ^{1}H NMR (400 MHz, CDCl$_3$, Major isomer, Pure) δ 7.18–7.52 (12H, m, ArH), 6.16 (1H, d, J = 15.8 Hz, CH = C*H*Ph), 5.98 (1H, dd, J = 15.8, 9.0 Hz, C*H* = CHPh), 5.79 (1H, q, J = 6.9 Hz, C*H*CH$_3$), 5.49 (1H, d, J = 4.8 Hz, C3-H), 3.68 (1H, dd, J = 9.0, 4.8 Hz, C4-H), 1.95 (3H, s, C*H*$_3$COO), 1.71 (3H, d, J = 7.0 Hz, CHC*H*$_3$); ^{1}H NMR (300 MHz, CDCl$_3$, Minor isomer, Pure) δ 6.73–8.11 (12H, m, ArH), 6.05 (1H, d, J = 15.9 Hz, CH = C*H*Ph), 5.60 (1H, d, J = 4.8 Hz, C3-H), 5.40 (1H, q, J = 6.9 Hz, C*H*CH$_3$), 5.27 (1H, dd, J = 15.9, 8.7 Hz, C*H* = CHPh), 4.22 (1H, dd, J = 8.7, 4.5 Hz, C4-H), 1.87 (3H, s, C*H*$_3$COO), 1.80 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^{13}C NMR (100 MHz, CDCl$_3$, Major isomer) δ 169.3, 164.0, 136.5, 135.8, 133.9, 133.2, 131.1, 129.2, 129.1, 128.8, 128.5, 127.3, 126.6, 126.1, 124.9, 124.4, 123.1, 122.6, 76.1, 59.3, 47.0, 20.4, 19.1; ^{13}C NMR (100 MHz, CDCl$_3$, Both isomers) δ 169.2, 164.2, 163.9, 136.3, 136.1, 135.7, 135.5, 134.6, 133.8, 133.1, 131.0, 129.0, 129.0, 128.9, 128.8, 128.6, 128.4, 128.2, 128.0, 127.1, 126.7, 126.5, 126.3, 126.0, 125.8, 125.1, 124.8, 124.3, 124.1, 123.0, 122.7, 122.5, 121.7, 77.1, 76.1, 76.0, 59.2, 59.0, 48.3, 46.9, 20.2, 19.0, 18.3; Anal. Calcd. for $C_{25}H_{23}NO_3$: C, 77.90; H, 6.01; N, 3.63. Found: C, 77.84; H, 5.99; N, 3.60.

4.3.9. (1′*S*, 3*R*, 4*S*)- and (1′*S*, 3*S*, 4*R*)-1-(1′-Phenylpropyl)-3-acetoxy-4-phenylazetidin-2-one 2i/2′i

IR (ATR) ν_{max} 1751, 1602, 1527 cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$, Major isomer) δ 7.06–7.27 (10H, m, ArH), 5.56 (1H, d, J = 4.5 Hz, C3-H), 4.56–4.58 (1H, m, C*H*CH$_2$), 4.52 (1H, d, J = 4.8 Hz, C4-H), 1.78–1.88 (1H, m, CHC*H*$_2$), 1.62–1.69 (1H, m, CHC*H*$_2$), 1.59 (3H, s, C*H*$_3$COO), 0.78 (3H, t, J = 7.2 Hz, CH$_2$C*H*$_3$); ^{1}H NMR (300 MHz, CDCl$_3$, Minor isomer) δ 7.06–7.27 (m, 10H, ArH), 5.61 (1H, d, J = 4.5 Hz, C3-H), 4.56–4.58 (1H, m, C4-H), 3.97 (1H, t, J = 8.1 Hz, C*H*CH$_2$), 2.42–2.47 (1H, m, CHC*H*$_2$), 2.00–2.04 (1H, m, CHC*H*$_2$), 1.57 (3H, s, C*H*$_3$COO), 0.92 (3H, t, J = 7.2 Hz, CH$_2$C*H*$_3$), ^{13}C NMR (75 MHz, CDCl$_3$, Both isomers) δ 169.1, 169.0, 164.8, 164.6, 139.2, 137.7, 133.9, 133.7, 132.7, 128.7, 128.6, 128.5, 128.4, 128.1, 128.0, 127.9, 127.9, 127.8, 127.2, 76.4, 76.2, 61.6, 61.6, 60.4, 60.1, 26.7, 26.3, 19.8, 19.8, 11.7, 11.1; Anal. Calcd. for $C_{20}H_{21}NO_3$: C, 74.28; H, 6.55; N, 4.33. Found: C, 74.22; H, 6.50; N, 4.29.

4.3.10. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylpropyl)-3-acetoxy-4-styrylazetidin-2-one 2j/2′j

White needles; m.p.: 132–133 °C; IR (ATR) ν_{max} 1752, 1650, 1601 cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$, Major isomer, Pure) δ 7.18–7.30 (10H, m, ArH), 6.45 (1H, d, J = 15.9 Hz, CH=C*H*Ph), 5.91 (1H, dd, J = 15.6, 9.0 Hz, C*H*=CHPh), 5.59 (1H, d, J = 4.8 Hz, C3-H), 4.51 (1H, t, J = 7.5 Hz, C*H*CH$_2$), 4.17 (1H, dd, J = 8.7, 4.8 Hz, C4-H), 1.98–2.10 (1H, m, C*H*$_2$CH$_3$), 1.96 (3H, s, C*H*$_3$COO), 1.83–1.93 (1H, m, C*H*$_2$CH$_3$), 0.91 (3H, t, J = 7.2 Hz, CH$_2$C*H*$_3$); ^{1}H NMR (300 MHz, CDCl$_3$, Minor isomer) δ 7.15–7.48 (10H, m, ArH), 6.40 (1H, d, J = 16.2 Hz, CH=C*H*Ph), 5.73 (1H, dd, J = 16.2, 8.4 Hz, C*H*=CHPh), 5.58 (1H, d, J = 4.8 Hz, C3-H), 4.12–4.17 (1H, t, J = 8.1 Hz, C*H*CH$_2$), 4.12–4.17 (1H, dd, J = 8.7, 4.8 Hz, C4-H), 1.98–2.08 (1H, m, C*H*$_2$-CH$_3$), 1.90–1.92 (1H, m, C*H*$_2$CH$_3$), 1.95 (3H, s, C*H*$_3$COO), 0.82 (3H, t, J = 6.9 Hz, CH$_2$C*H*$_3$); Anal. Calcd. for $C_{22}H_{23}NO_3$: C, 75.62; H, 6.63; N, 4.01. Found: C, 75.60; H, 6.59; N, 3.99.

4.3.11. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-methoxy-4-phenyl-azetidin-2-one 3a/3′a

IR (ATR) ν_{max} 1746, 1648, 1521 cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$, Major isomer) δ 7.05–7.29 (10H, m, ArH), 4.43 (1H, d, J = 4.5 Hz, C3-H), 4.33 (1H, d, J = 4.5 Hz, C4-H), 4.13 (1H, q, J = 7.2 Hz, C*H*CH$_3$), 2.99 (3H, s, OC*H*$_3$), 1.73 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^{1}H NMR (300 MHz, CDCl$_3$, Minor isomer) δ 7.05–7.29 (10H, m, ArH), 4.92 (1H, q, J = 7.5 Hz, C*H*CH$_3$), 4.37 (1H, d, J = 4.5 Hz, C3-H), 4.28 (1H, d, J = 4.5 Hz, C4-H), 3.03 (3H, s, OC*H*$_3$), 1.24 (3H, d, J = 7.2 Hz, CHC*H*$_3$); ^{13}C NMR (75 MHz, CDCl$_3$, Both isomers) δ 166.8, 140.9, 139.4, 135.1, 133.8, 128.8, 128.7, 128.6, 128.6, 128.5, 128.4, 128.2, 128.1, 127.9, 127.7, 127.3, 126.8, 84.8, 61.1, 61.0, 58.1, 58.0, 54.3, 51.8, 19.9, 19.1; Anal. Calcd. for $C_{18}H_{19}NO_2$: C, 76.84; H, 6.81; N, 4.98. Found: C, 76.80; H, 6.80; N, 4.95.

4.3.12. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-methoxy-4-(4′-methoxyphenyl)azetidin-2-one 3b/3′b

Yellow oil; IR (ATR) ν_{max} 1750, 1615 cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$, Major isomer) δ 6.69–7.25 (9H, m, ArH), 4.40 (1H, d, J = 4.5 Hz, C3-H), 4.29 (1H, d, J = 4.5 Hz, C4-H), 4.14 (1H, q, J = 7.2 Hz, C*H*CH$_3$), 3.72 (3H, s, OCH$_3$), 3.04 (3H, s, OC*H*$_3$), 1.72 (1H, d, J = 7.2 Hz, CHC*H*$_3$); ^{1}H NMR (300 MHz, CDCl$_3$, Minor isomer) δ 6.69–7.25 (9H, m, ArH), 4.94 (1H, q, J = 7.2 Hz, C*H*CH$_3$), 4.34 (1H, d, J = 4.5 Hz, C3-H), 4.24 (1H, d, J = 4.5 Hz, C4-H), 3.73 (3H, s, OCH$_3$), 3.00 (3H, s, OC*H*$_3$), 1.24 (1H, d, J = 7.2 Hz, CHC*H*$_3$); ^{13}C NMR (100 MHz, CDCl$_3$, Both isomers) δ 166.9, 166.8, 159.8, 159.7, 141.0, 139.5, 130.0, 129.8, 128.7, 128.6, 127.9, 127.7, 127.3, 126.9, 126.8, 125.6, 113.7, 113.5, 84.8, 60.5, 60.4, 58.1, 58.0, 55.2, 54.1, 51.6, 19.9, 19.1; Anal. Calcd. for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.25; H, 6.78; N, 4.52.

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4.3.13. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-methoxy-4-(4′-chlorophenyl)azetidin-2-one 3c/3′c

Yellowish solid; IR (ATR) ν_{max} 1750, 1615 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 7.03–7.27 (9H, m, ArH), 4.45 (1H, d, J = 4.8 Hz, C3-H), 4.36 (1H, d, J = 4.5 Hz, C4-H), 4.22 (1H, q, J = 7.1 Hz, $CHCH_3$), 3.05 (3H, s, OCH_3), 1.71 (3H, d, J = 6.9 Hz, $CHCH_3$); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 7.03–7.27 (9H, m, ArH), 4.95 (1H, q, J = 7.1 Hz, $CHCH_3$), 4.40 (1H, d, J = 4.5 Hz, C3-H), 4.28 (1H, d, J = 4.5 Hz, C4-H), 2.99 (3H, s, OCH_3), 1.26 (3H, d, J = 7.2 Hz, $CHCH_3$); ^{13}C NMR (75 MHz, $CDCl_3$, Both isomers) δ 166.7, 140.5, 139.2, 134.5, 134.3, 133.8, 132.5, 130.1, 129.9, 128.8, 128.7, 128.5, 128.4, 128.0, 127.9, 127.3, 126.9, 84.8, 60.3, 60.3, 58.2, 58.2, 54.1, 51.9, 19.6, 19.1; Anal. Calcd. for $C_{18}H_{18}NO_2Cl$: C, 68.46; H, 5.75; N, 4.44. Found: C, 68.40; H, 5.69; N, 4.41.

4.3.14. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-methoxy-4-styrylazetidin-2-one 3d/3′d

IR (ATR) ν_{max} 1744, 1521 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 7.07–7.27 (10H, m, ArH), 6.40 (1H, d, J = 15.9 Hz, CH = C*H*Ph), 6.16 (1H, dd, J = 15.9, 9.3 Hz, C*H* = CHPh), 4.92 (1H, q, J = 7.2 Hz, $CHCH_3$), 4.41 (1H, d, J = 4.5 Hz, C3-H), 4.03 (1H, dd, J = 9.3, 4.5 Hz, C4-H), 3.332 (3H, s, OCH_3), 1.64 (3H, d, J = 7.2 Hz, $CHCH_3$); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 7.07–7.27 (10H, m, ArH), 6.43 (1H, d, J = 15.9 Hz, CH=C*H*Ph), 5.96 (1H, dd, J = 15.9, 9.3 Hz, C*H*=CHPh), 4.55 (1H, q, J = 7.2 Hz, $CHCH_3$), 4.45 (1H, d, J = 4.2 Hz, C3-H), 4.11 (1H, dd, J = 9.6, 4.8 Hz, C4-H), 3.337 (3H, s, OCH_3), 1.51 (3H, d, J = 6.9 Hz, $CHCH_3$); ^{13}C NMR (75 MHz, $CDCl_3$, Both isomers) δ 166.1, 165.9, 140.9, 139.5, 136.0, 136.0, 135.8, 135.1, 128.6, 128.6, 128.5, 128.1, 128.1, 127.7, 127.6, 127.2, 127.1, 126.9, 126.6, 125.2, 123.8, 84.8, 84.6, 60.1, 59.7, 58.5, 52.8, 51.5, 19.3, 18.9; Anal. Calcd. for $C_{20}H_{21}NO_2$: C, 78.15; H, 6.89; N, 4.56. Found: C, 78.13; H, 6.87; N, 4.55.

4.3.15. (1′*R*,3*R*,4*S*)- and (1′*R*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-methoxy-4-phenylazetidin-2-one 3f/3′f

IR (ATR) ν_{max} 1746, 1519 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 7.06–7.21 (10H, m, ArH), 4.41 (1H, d, J = 4.5 Hz, C3-H), 4.31 (1H, d, J = 4.5 Hz, C4-H), 4.21 (1H, q, J = 7.2 Hz, $CHCH_3$), 2.98 (3H, s, OCH_3), 1.75 (3H, d, J = 6.9 Hz, $CHCH_3$); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 7.06–7.21 (10H, m, ArH), 4.99 (1H, q, J = 6.9 Hz, $CHCH_3$), 4.47 (1H, d, J = 4.5 Hz, C3-H),), 4.37 (1H, d, J = 4.5 Hz, C4-H), 3.03 (3H, s, OCH_3), 1.25 (3H, d, J = 7.2 Hz, $CHCH_3$); ^{13}C NMR (100 MHz, $CDCl_3$, Both isomers) δ 166.9, 166.8, 140.9, 139.5, 135.1, 133.9, 128.8, 128.7, 128.7, 128.6, 128.6, 128.5, 128.3, 128.2, 127.9, 127.7, 127.3, 126.9, 84.9, 76.7, 61.1, 61.0, 58.1, 58.1, 54.3, 51.8, 19.9, 19.1; Anal. Calcd. for $C_{18}H_{19}NO_2$: C, 76.84; H, 6.81; N, 4.98. Found: C, 76.83; H, 6.78, N, 4.95.

4.3.16. (1′*R*,3*R*,4*S*)- and (1′*R*,3*S*,4*R*)-1-(1′-Naphthylethyl)-3-methoxy-4-phenylazetidin-2-one 3g/3′g

IR (ATR) ν_{max} 1740 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer, Pure) δ 6.94–8.03 (12H, m, ArH), 5.14 (1H, q, J = 7.2 Hz, $CHCH_3$), 4.49 (1H, d, J = 4.8 Hz, C3-H), 4.39 (1H, d, J = 4.5 Hz, C4-H), 3.02 (3H, s, OCH_3), 1.88 (3H, d, J = 7.2 Hz, $CHCH_3$); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer, Pure) δ 7.09–8.02 (12H, m, ArH), 5.84 (1H, q, J = 7.5 Hz, $CHCH_3$), 4.29 (1H, d, J = 4.5 Hz, C3-H), 3.77 (1H, d, J = 4.5 Hz, C4-H), 2.93 (3H, s, OCH_3), 1.42 (3H, d, J = 7.0 Hz, $CHCH_3$); ^{13}C NMR (75 MHz, $CDCl_3$, Major isomer) δ 166.7, 135.1, 133.8, 133.6, 131.3, 129.1, 128.9, 128.5, 128.0, 127.1, 126.0, 125.0, 124.3, 122.8, 84.6, 60.5, 58.0, 46.5, 19.0; ^{13}C NMR (75 MHz, $CDCl_3$, Minor isomer) δ 166.7, 135.1, 133.8, 133.7, 131.2, 129.0, 128.9, 128.6, 128.0, 127.1, 126.1, 125.0, 124.4, 122.8, 84.7, 60.6, 58.0, 46.5, 19.0; Anal. Calcd. for $C_{22}H_{21}NO_2$: C, 79.73; H, 6.39; N, 4.23. Found: C, 79.70; H, 6.28; N, 4.30.

4.3.17. (1′*R*,3*R*,4*S*)- and (1′*R*,3*S*,4*S*)-1-(1′-Naphthylethyl)-3-methoxy-4-styrylazetidin-2-one 3h/3′h

IR (ATR) ν_{max} 1741, 1646 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, Major isomer, Pure) δ 7.25–8.07 (12H, m, ArH), 6.19–6.27 (2H, m, C*H*=CHPh and CH=C*H*Ph), 5.85 (1H, q, J = 7.3 Hz, $CHCH_3$), 4.35 (1H, d, J = 4.5 Hz, C3-H), 3.59 (1H, dd, J = 8.4, 4.5 Hz, C4-H), 3.36 (3H, s, OCH_3), 1.75 (3H, d, J = 6.9 Hz, $CHCH_3$); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer, Pure) δ 6.91–8.18 (12H, m, ArH), 6.19 (1H, d, J = 15.9 Hz, CH=C*H*Ph), 5.64 (1H, dd, J = 15.9 Hz, 9.3 Hz, C*H*=CHPh), 5.53 (1H, q, J = 6.6 Hz, $CHCH_3$), 4.54 (1H, d, J = 4.5 Hz, C3-H), 4.16 (1H, dd, J = 9.3, 4.5 Hz, C4-H), 3.37 (3H, s, OCH_3), 1.86 (3H, d, J = 6.9 Hz, $CHCH_3$); ^{13}C NMR (100 MHz, $CDCl_3$, Major isomer) δ 165.9, 136.2, 135.4, 133.9, 133.6, 131.3, 129.0, 128.7, 128.3, 127.1, 126.7, 126.0, 124.9, 124.5, 122.9, 84.8, 59.8, 58.6, 46.4, 19.2; ^{13}C NMR (100 MHz, $CDCl_3$, Minor isomer) δ 165.8, 136.1, 135.3, 133.8, 133.5, 131.1, 129.0, 128.8, 128.4, 127.2, 126.6, 126.0, 124.9, 124.4, 122.9, 84.8, 59.8, 58.6, 46.5, 19.4; Anal. Calcd. for $C_{24}H_{23}NO_2$: C, 80.64; H, 6.49; N, 3.92. Found: C, 80.40; H, 6.46; N, 3.90.

4.3.18. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylpropyl)-3-methoxy-4-phenylazetidin-2-one 3i/3′i

Oil; IR (ATR) ν_{max} 1741 cm^{-1} ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 7.04–7.26 (10H, m, ArH), 4.47 (1H, d, J = 4.5 Hz, C3-H), 4.35 (1H, d, J = 4.5 Hz, C4-H), 3.85 (1H, t, J = 7.5 Hz, $CHCH_2$), 3.00 (3H, s, OCH_3), 1.93–2.02 (1H, m, CH_2CH_3), 1.66–1.80 (1H, m, CH_2CH_3), 0.89 (3H, t, J = 7.2 Hz, CH_2CH_3); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 7.04–7.26 (10H, m, ArH), 4.57 (1H, t, J = 8.1 Hz, $CHCH_2$), 4.42 (1H, d, J = 4.2 Hz, C3-H), 4.27 (1H, d, J = 4.5 Hz, C4-H), 2.96 (3H, s, OCH_3), 2.38–2.47 (1H, m, CH_2CH_3), 1.52–1.61 (1H, m, CH_2CH_3), 0.76 (3H, t, J = 7.2 Hz, CH_2CH_3); ^{13}C NMR (75 MHz, $CDCl_3$, Both isomers) δ 166.6, 139.9, 138.1, 135.0, 133.8, 128.8, 128.6, 128.6, 128.5, 128.4, 128.2, 128.0, 128.0, 127.9, 127.7, 127.3, 126.5, 84.8, 72.0, 84.8, 61.5, 61.4, 60.7, 59.3, 59.0, 58.0, 57.9, 54.1, 26.8, 26.2, 11.8, 11.2, 10.7; Anal. Calcd. for $C_{19}H_{21}NO_2$: C, 77.26; H, 7.17; N, 4.74. Found: C, 77.24; H, 7.14; N, 4.72.

4.3.19. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylpropyl)-3-methoxy-4-styrylazetidin-2-one 3j/3′j

IR (ATR) ν_{max} 1740, 1520 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 6.98–7.33 (10H, m, ArH), 6.43 (1H, d, J = 15.9 Hz, CH=C*H*Ph), 6.11 (1H, dd, J = 15.9, 9.3 Hz, C*H*=CHPh), 4.51 (1H, t, J = 7.5 Hz, $CHCH_2$), 4.33 (1H, d, J = 4.5 Hz, C3-H), 3.94 (1H, dd, J = 9.6, 4.8 Hz, C4-H), 3.35 (3H, s, OCH_3), 1.92–2.01 (1H, m, CH_2CH_3), 1.81–1.88 (1H, m, CH_2CH_3), 0.90 (3H, t, J = 7.2 Hz, CH_2CH_3); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 6.98–7.33 (10H, m, ArH), 6.39 (1H, d, J = 15.9 Hz, CH = C*H*Ph), 5.96 (1H, dd, J = 15.9, 9.0 Hz, C*H*=CHPh), 4.38 (1H, d, J = 4.8 Hz, C3-H), 3.97–4.06 (2H, m, $CHCH_2$ and C4-H), 3.37 (3H, s, OCH_3), 2.28–2.31 (1H, m, CH_2CH_3), 1.61–2.75 (1H, m, CH_2CH_3), 0.90 (3H, t, J = 7.2 Hz, CH_2CH_3); Anal. Calcd. for $C_{21}H_{23}NO_2$: C, 78.47; H, 7.21; N, 4.36. Found: C, 78.45; H, 7.12; N, 4.31.

4.3.20. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-phthalimido-4-phenylazetidin-2-one 4a/4′a

IR (ATR) ν_{max} 1781, 1756, 1720 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, Major isomer) δ 7.10–7.67 (14H, m, ArH), 5.33 (1H, d, J = 5.4 Hz, C3-H), 5.24 (1H, q, J = 7.2 Hz, $CHCH_3$), 4.76 (1H, d, J = 5.4 Hz, C4-H), 1.59 (3H, d, J = 7.2 Hz, $CHCH_3$); ^1H NMR (400 MHz, $CDCl_3$, Minor isomer) δ 7.10–7.67 (14H, m, ArH), 5.41 (1H, d, J = 5.3 Hz, C3-H), 4.85 (1H, d, J = 5.3 Hz, C4-H), 4.60 (1H, q, J = 7.2 Hz, $CHCH_3$), 2.00 (3H, d, J = 7.2 Hz, $CHCH_3$); ^{13}C NMR (100 MHz, $CDCl_3$, Both isomers) δ 166.8, 164.0, 163.7, 141.0, 139.5, 134.1, 134.0, 133.0, 131.2, 128.8, 128.4, 128.3, 128,2, 128.0, 127.9, 127.7, 127.4, 127.3, 126.8, 123.3, 60.9, 60.4, 59.2, 55.3, 53.1, 19.9, 19.3; Anal. Calcd. for $C_{25}H_{20}N_2O_3$: C, 75.74; H, 5.08; N, 7.07. Found: C, 75.71; H, 5.10; N, 7.00.

4.3.21. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-phthalimido-4-(4′-methoxyphenyl)azetidin-2-one 4b/4′b

White solid; IR (ATR) ν_{max} 1779, 1754, 1721 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, Major isomer) δ 6.66–7.68 (13H, m, ArH), 5.29 (1H, d, J = 5.2 Hz, C3-H), 5.20 (1H, q, J = 7.2 Hz, C*H*CH_3), 4.73 (1H, d, J = 5.3 Hz, C4-H), 3.64 (3H, s, OCH_3), 1.56 (3H, t, J = 7.2 Hz, CHCH_3); ^1H NMR (400 MHz, $CDCl_3$, Minor isomer) δ 6.66–7.68 (13H, m, ArH), 5.37 (1H, d, J = 5.2 Hz, C3-H), 4.81 (1H, d, J = 5.2 Hz, C4-H), 4.58 (1H, q, J = 7.2 Hz, C*H*CH_3), 3.63 (3H, s, OCH_3), 1.97 (3H, d, J = 7.2 Hz, CHCH_3); ^{13}C NMR (100 MHz, $CDCl_3$, Both isomers) δ 163.9, 163.7, 159.4, 159.3, 140.9, 139.4, 134.6, 134.4, 134.1, 131.1, 128.9, 128.4, 128.3, 128.0, 127.8, 127.7, 127.5, 127.2, 126.7, 126.1, 125.6, 124.5, 123.9, 123.7, 123.5, 123.3, 114.2, 113.6, 113.5, 60.5, 60.1, 59.2, 59.1, 55.2, 55.0, 54.8, 53.4, 52.9, 19.7, 19.28; Anal. Calcd. for $C_{26}H_{22}N_2O_4$: C, 73.23; H, 5.20; N, 6.57. Found: C, 73.17; H, 5.11; N, 6.54.

4.3.22. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-phthalimido-4-(4′-chlorophenyl)azetidin-2-one 4c/4′c

White solid; IR (ATR) ν_{max} 1781, 1756, 1720 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 6.97–7.80 (13H, m, ArH), 5.23 (1H, d, J = 5.4 Hz, C3-H), 5.12 (1H, q, J = 7.2 Hz, C*H*CH_3), 4.63 (1H, d, J = 5.1 Hz, C4-H), 1.51 (3H, d, J = 7.2 Hz, CHCH_3); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 6.97–7.80 (13H, m, ArH), 5.31 (1H, d, J = 5.4 Hz, C3-H), 4.72 (1H, d, J = 5.1 Hz, C4-H), 4.51 (1H, q, J = 7.2 Hz, C*H*CH_3), 1.90 (3H, d, J = 7.2 Hz, CHCH_3); ^{13}C NMR (75 MHz, $CDCl_3$, Both isomers) δ 166.7, 163.6, 139.4, 134.3, 134.2, 132.8, 131.7, 131.2, 129.2, 129.1, 128.9, 128.6, 128.5, 128.1, 128.0, 127.8, 127.2, 126.8, 123.7, 123.5, 60.4, 59.9, 59.2, 55.1, 53.2, 19.6, 19.3; Anal. Calcd. for $C_{25}H_{19}N_2O_3Cl$: C, 69.69; H, 4.44; N, 6.50. Found: C, 69.63; H, 4.38, N, 6.48.

4.3.23. (1′*S*,3*R*,4*S*)- and (1′*S*,3*S*,4*R*)-1-(1′-Phenylethyl)-3-phthalimido-4-styrylazetidin-2-one 4d/4′d

Yellow oil; IR (ATR) ν_{max} 1752, 1726 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, Major isomer) δ 6.99–7.79 (14H, m, ArH), 6.35 (1H, d, J = 15.6 Hz, CH = C*H*Ph), 6.14 (1H, dd, J = 15.9, 9.0 Hz, C*H*=CHPh), 5.37 (1H, d, J = 5.4 Hz, C3-H), 5.06 (1H, q, J = 7.2 Hz, C*H*CH_3), 4.32 (1H, dd, J = 9.3, 5.1 Hz, C4-H), 1.65 (3H, d, J = 7.2 Hz, CHCH_3); ^1H NMR (300 MHz, $CDCl_3$, Minor isomer) δ 6.99–7.79 (14H, m, ArH), 6.40 (1H, d, J = 15.9 Hz, CH=C*H*Ph), 5.90 (1H, dd, J = 15.6, 9.0 Hz, C*H*=CHPh), 5.42 (1H, d, J = 5.4 Hz, C3-H), 4.76 (1H, q, J = 7.2 Hz, C*H*CH_3), 4.43 (1H, dd, J = 9.3, 5.1 Hz, C4-H), 1.75 (3H, d, J = 7.2 Hz, CHCH_3); ^{13}C NMR (75 MHz, $CDCl_3$, Both isomers) δ 167.3, 163.3, 140.6, 139.4, 136.4, 135.5, 134.3, 131.5, 131.4, 128.7, 128.6, 128.5, 128.4, 128.2, 127.8, 127.7, 127.2, 126.9, 126.5, 124.4, 123.6, 60.2, 56.7, 52.8, 52.0, 51.5, 19.2, 19.2; Anal. Calcd. for $C_{27}H_{22}N_2O_3$: C, 76.76; H, 5.25; N, 6.63. Found: C, 76.71; H, 5.22; N, 6.60.

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A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.tetasy.2016.12.007.

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