Lewis Acid Mediated Cyclization of Epoxy Benzoylcarbamates

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Lewis acid mediated cyclization of 2,3-epoxypropylbenzoylcarbamates is described. *trans*-Aryl-substituted epoxy carbamates cyclized to 4-(α-benzoylbenzyl)-1,3-dioxolan-2-ones and 4-aryl-3-benzoyl-5-hydroxymethyloxazolidin-2-ones. On the contrary, *cis*-aryl- and alkyl-substituted epoxy carbamates gave only 4-[1-(benz-oyloxy)alkyl]oxazolidin-2-ones. Mechanism of this cyclization is also discussed.

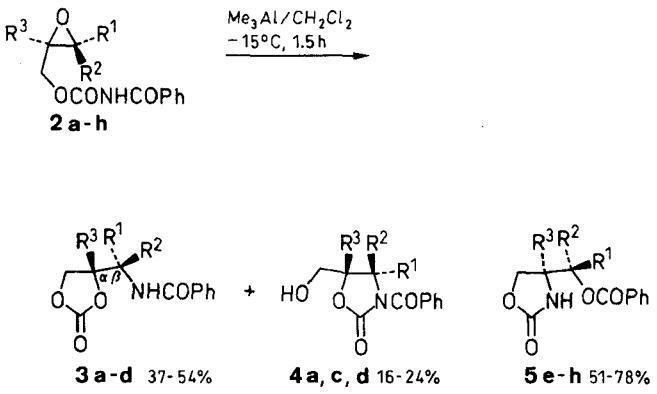
Amino alcohols and β-hydroxyamino acids are an important class of natural products.[1] These compounds have been synthesized by various methods;[2] cyclization of an epoxycarbamate with a base is especially important.[3,4]

The merits of this cyclization are: (a) these epoxycarbamates can be formed readily under neutral conditions; (b) the anion of a benzoylcarbamate is easily generated and undergoes *N*-cyclization; (c) an acid-catalyzed ring opening of the epoxide ring results in the two possible modes of cyclizations, depending on attack by either the nitrogen or the oxygen atom of the carbamate moiety; and (d) one or both carbonyl groups of the cyclized product are removable from the amino nitrogen by a basic hydrolysis step.[5]

While developing a new methodology for the regio- and stereoselective synthesis of cyclic amino alcohol derivatives, we investigated Lewis acid mediated cyclization of 2,3-epoxypropyl benzoylcarbamates, to find a novel cyclization with high regio- and stereoselectivity. It has also been found that only *trans*-aryl-substituted epoxides cyclize smoothly, and the corresponding *cis*-aryl and alkyl-substituted epoxides give only normal Baldwin's cyclization products.

The key substrate 2,3-epoxy-3-phenylpropyl benzoylcarbamate (**2a**) was synthesized from cinnamyl alcohol with benzoyl isocyanate followed by epoxidation with 3-chloroperoxybenzoic acid (MCPBA)[6] (Table 1). Compound **2a** was treated with an equimolar amount of trimethylaluminum as catalyst in dichloromethane at −20 °C for 9 hours under nitrogen. The reaction mixture was worked up as normal to give two crystalline materials, **3a** and **4a**, in 58 and 19% yields. When compound **2a** was treated with four molar equivalents of trimethylaluminum, both products **3a** and **4a** were again obtained (in 54 and 24% yield respectively) but the reaction time could be shortened from 9 to 1.5 hours. For this reason, the other cyclizations were conducted with 4 equivalents of trimethylaluminum.

From the spectroscopic data and the combustion analyses **3a** was assigned as a cyclic carbonate and **4a** as a 5-membered carbamate. The stereochemistry of **3a** was determined by the results of ^{1}H NMR: the coupling constant ($J_{\alpha,\beta}$ = 4.5 Hz) showed a *syn* stereochemistry in **3a** from its comparison with the known value;[7] The coupling constant J_{23} in **4a** (J_{23} = 8.2 Hz) also showed a *syn* stereochemistry.[8]



2, 3, 4	R^1	R^2	R^3	2, 5	R^1	R^2	R^3
a	Ph	H	H	**e**	Me	Me	H
b	Ph	H	Me	**f**	Me	H	H
c	4-ClC_6H_4	H	H	**g**	H	Ph	H
d	4-MeC_6H_4	H	H	**h**	H	Ph	Me

Scheme 1

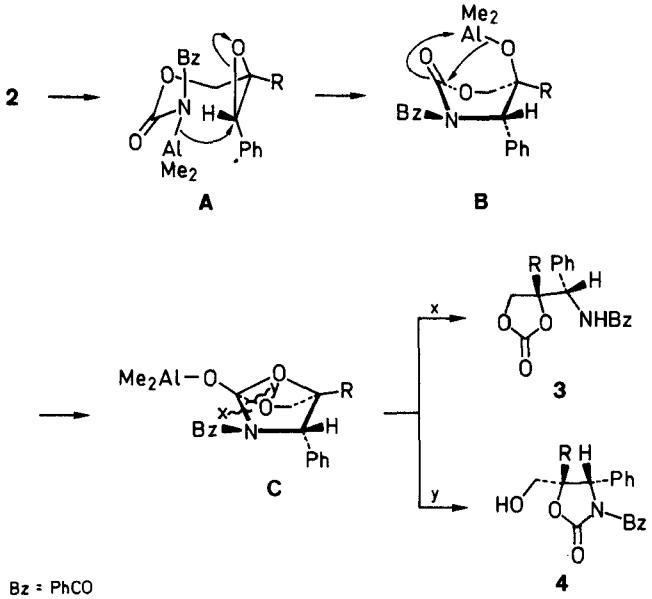
The mechanism of this cyclization is considered to be as follows: Initial removal of an acidic imide proton in **2** with trimethylaluminum gives the aluminum imide **A**. Then the activated imide nitrogen atom attacks at the C-3 epoxide carbon atom to give a 6-membered carbamate **B**. At this stage the other free trimethylaluminum coordinates on the epoxide oxygen atom and assists the epoxide ring cleavage. The resulting aluminum alkoxide again attacks the carbamate group to afford an intermediate **C**. Although the intermediates **A**, **B** and **C** could not be detected, ring cleavage of **C** is presumed to give **3** and **4**. In the cyclization of *trans*-aryl-substituted carbamate **2a** the resulting intermediate **C** underwent cleavage at the x bond to give **3a** and at the y bond to give **4a** (Scheme 2).

This cyclization proceeded with 4 molar equivalents of diethylaluminum chloride as a catalyst. However, boron trifluoride–diethyl ether complex, titanium(IV) chloride or titanium(IV) isopropoxide gave only complex mixtures and no **3a** or **4a** was obtained. Trimethylaluminum mediated cyclization of variously substituted carbamates **2b–h** has been attempted (Table 2). *trans*-Aryl-substituted 2,3-epoxypropyl carbamates **2b**, **2c** and **2d** underwent the cyclization to give the corresponding cyclic carbonates **3b** (37%), **3** (50%) and **3d** (47%), and cyclic carbamates **4c** (21%) and **4d** (16%), respectively. In the case of **2b** only cyclic carbonate **3b** was obtained and **4b** was not detected.

Table 1. Spectral Data of 2,3-Epoxypropyl Benzoylcarbamates **2a–h**[a]

Compound	Yield (%)	mp (°C)	IR ($\nu_{C=O}$)	^{1}H NMR ($CDCl_3$/TMS), δ, J(Hz)	^{13}C NMR ($CDCl_3$/TMS, δ
2a	80	105.5–107.0	1770, 1690	3.35 (ddd, 1 H, J = 2.0, 3.3, 5.9), 3.88 (d, 1 H, J = 2.0), 4.21 (dd, 1 H, J = 5.9, 12.2), 4.70 (dd, 1 H, J = 3.2, 12.2), 7.95–7.21 (m, 10 H), 8.39 (br, 1 H)	56.35 (d), 58.92 (d), 65.48 (t), 125.7 (d), 127.8 (d), 128.5 (d), 128.8 (d), 132.7 (s), 133.0 (d), 135.8 (s), 150.8 (s), 165.0 (s)
2b	85	glass	1764	1.10 (s, 3 H), 4.10 (s, 1 H), 4.18 (d, 1 H, J = 12.1), 4.51 (d, 1 H, J = 12.1), 7.99–7.18), m, 10 H), 8.83 (br, 1 H)	13.33 (q), 60.92 (s), 60.16 (d), 68.71 (t), 126.3 (d), 127.6 (d), 17.8 (d), 128.0 (d), 128.1 (d), 128.6 (d), 132.7 (s), 132.8 (d), 134.6 (s), 150.9 (s), 165.2 (s)
2c	61	glass	1764, 1692	3.19 (ddd, 1 H, J = 2.1m 3.4, 5.8), 3.78 (d, 1 H, J = 2.1), 4.08 (dd, 1 H, J = 5.8, 12.3), 4.60 (dd, 1 H, J = 3.4, 12.3), 6.99–8.00 (m, 9 H), 9.10 (s, 1 H)	55.46 (d), 58.89 (d), 64.95 (t), 126.9 (d), 127.7 (d), 128.1 (d), 128.5 (d), 132.5 (s), 132.9 (d), 134.0 (s), 134.3 (s), 150.9 (s), 165.2 (s)
2d	40	glass	1786, 1714	2.30 (s, 3 H), 3.24 (ddd, 1 H, J = 2.1, 2.9, 6.0), 3.74 (d, 1 H, J = 2.1), 4.04 (dd, 1 H, J = 6.0, 12.3), 4.57 (dd, 1 H, J = 2.9, 12.3), 6.93–8.02 (m, 9 H), 9.28 (br, 1 H)	20.88 (q), 56.09 (d), 58.65 (d), 65.25 (t), 125.5 (d), 127.7 (d), 128.4 (d), 128.6 (s) 128.9 (d), 132.5 (s), 132.7 (d), 138.1 (s), 150.9 (s), 165.3 (s)
2e	88	91.0–93.0	1770, 1746, 1676	1.27 (s, 6 H), 3.02 (dd, 1 H, J = 4.0, 7.1), 4.05 (dd, 1 H, J = 7.1, 12.2), 4.56 (dd, 1 H, 4.0, 12.2), 7.44–8.02 (m, 5 H), 8.80 (br, 1 H)	18.82 (q), 24.37 (q), 58.44 (s), 60.20 (d), 64.86 (t), 127.8 (d), 128.7 (d), 132.8 (s), 132.9 (d), 150.8 (s), 165.1 (s)
2f	97	82.5–83.5	1794, 1778 1708	1.35 (d, 3 H, J = 5.1), 2.85–3.11 (m, 2 H), 4.02 (dd, 1 H, J = 6.2, 12.3), 4.60 (dd, 1 H, J = 2.7, 3.2), 7.35–8.03 (m, 5 H), 8.88 (s, 1 H)	16.94 (q), 52.45 (d), 55.94 (d), 65.84 (t), 127.7 (d), 128.6 (d), 132.7 (s), 132.9 (d), 150.8 (s), 165.1 (s)
2g	68	106.5–108.5	1766, 1750	3.53 (dt, 1 H, J = 3.8, 7.2), 3.84 (dd, 1 H, J = 7.2, 12.1), 4.20 (dd, 1 H, J = 3.8, 12.1), 4.27 (d, 1 H, J = 3.8), 7.28–8.10 (m, 10 H), 9.98 (br, 1 H)	55.55 (d), 56.50 (d), 64.14 (t), 126.2 (d), 127.6 (d), 128.2 (d), 128.4 (d), 128.9 (d), 132.7 (s), 133.1 (d), 133.8 (s), 150.5 (s), 164.7 (s)
2h	75	140.5–142.5	1785, 1755 1690	1.54 (s, 3 H), 3.88 (d, 1 H, J = 11.8), 3.99 (s, 1 H), 4.18 (d, 1 H, J = 11.8), 7.25–7.95 (m, 10 H), 8.50 (br, 12 H)	19.60 (q), 60.71 (s), 63.60 (d), 66.62 (t), 126.1 (d), 127.7 (d), 127.9 (d), 128.3 (d), 128.6 (d), 132.7 (s), 132.9 (d), 134.3 (s), 150.7 (165.0 (s)

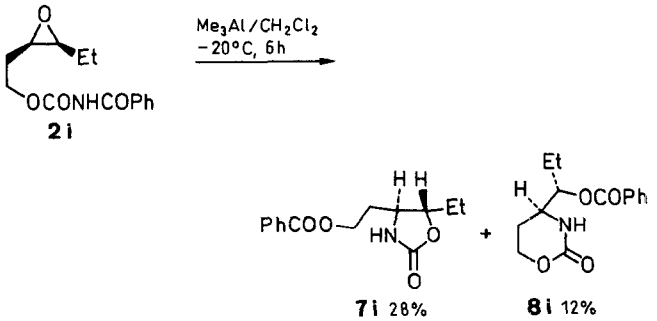
[a] Satisfactory microanalyses: C ± 0.14, H ± 0.08, N ± 0.17 and HRMS: m/z (M^+) ± 0.0006 obtained for all products.



Scheme 2

Alkyl-substituted epoxy carbamates **2e** and **2f** did not undergo this type of cyclization, but a 5-*exo*-tetrahedral mode of cyclization proceeded, followed by benzoyl group migration, to give **5e** and **5f** in 51 and 67% yield, respectively. Cyclization of *cis*-aryl-substituted compounds **2g** and **2h** also afforded only the 5-*exo*-tetrahedral products **5g** (78%) and **5h** (65%). The stereochemistry of **5e** was proved to be *anti* by comparison of its ^{1}H NMR spectra with those of known value.[9]

These carbamate molecules bearing a *cis*-aryl-substituent hardly forms the intermediate **B** in Scheme 2 due to an eclipsed interaction between the benzoyl and R^2 groups. This results in the normal Baldwin's 5-*exo*-tetrahedral mode of cyclization. Alkyl-substituted epoxy carbamates **2e** and **2f** were also governed by a 5-*exo* over a 6-*endo* mode of cyclization to give **5e** and **5f**. We have also attempted the cyclization of **2i**. Two products **7i** and **8i** were obtained in 28 and 12% yield, respectively.



Scheme 3

Table 2. Spectroscopic Data of the Cyclized Products **3a–d**, **4a–d** and **5e–h**[a]

Product	Yield (%)	mp (°C)	IR ($\nu_{C=O}$)	^{1}H NMR ($CDCl_3$/TMS), δ, J(Hz)	^{13}C NMR ($CDCl_3$/TMS), δ
3a	54	147–149	1800, 1780, 1635	4.23 (dd, 1 H, J = 6.4, 8.8), 4.53 (dd, 1 H, J = 8.4, 8.8), 5.26 (ddd, 1 H, J = 4.4, 6.4, 8.4), 5.36 (dd, 1 H, J = 4.4, 7.9), 6.86 (d, 1 H, J = 7.9), 7.40–7.80 (m, 10 H)	55.95 (d), 67.76 (t), 77.97 (d), 128.2 (d), 128.7 (d), 128.8 (d), 129.0 (d), 129.3 (d), 132.3 (d), 135.0 (s), 138.2 (s), 155.1 (s), 167.7 (s)
3b	37	200–202	1800, 1780, 1655	1.67 (s, 3 H), 4.32 (d, 1 H, J = 8.9), 4.59 (d, 1 H, J = 8.9), 5.64 (d, 1 H, J = 9.6), 7.28–8.00 (m, 10 H), 8.27 (d, J = 9.6)	23.38 (q), 59.42 (d), 73.29 (t), 85.70 (s), 128.9 (d), 129.5 (d), 129.6 (d), 129.8 (d), 130.0 (d), 132.7 (d), 135.7 (s), 138.3 (s), 155.1 (s), 168.3 (s)
3c	50	170–172	1810, 1780, 1635	4.48 (dd, 1 H, J = 6.1, 8.8), 4.71 (dd, 1 H, J = 7.7, 8.8), 5.32 (ddd, 1 H, J = 6.1, 7.1, 7.7), 5.58 (dd, 1 H, J = 7.1, 8.6), 7.27–8.00 (m, 9 H), 8.39 (d, 1 H, J = 8.6)	55.87 (d), 68.22 (t), 78.13 (d), 128.6 (d), 129.4 (d), 129.7 (d), 130.8 (d), 132.7 (d), 134.7 (s), 135.1 (s), 137.5 (s), 155.4 (s), 168.1 (s)
3d	47	178–180	1810, 1765, 1628	2.90 (s, 3 H), 4.47 (dd, 1 H, J = 6.1, 8.9), 4.69 (dd, 1 H, J = 7.7, 8.9), 5.30 (ddd, 1 H, J = 6.1, 6.8, 7.7), 5.54 (dd, 1 H, J = 6.8, 8.1), 7.10–7.98 (m, 9 H), 8.27 (d, 1 H, J = 8.1)	21.53 (q), 56.32 (d), 68.34 (t), 78.69 (d), 128.8 (d), 129.3 (d), 129.6 (d), 130.6 (d), 132.8 (d), 135.7 (s), 135.8 (s), 139.1 (s), 155.8 (s), 168.1 (s)
4a	24	156.5–158.5	1780, 1740, 1680	3.32 (ddd, 1 H, J = 5.0, 9.0, 16.0), 3.40 (ddd, 1 H, J = 3.9, 5.8, 12.3), 4.12 (dd, 1 H, J = 5.0, 5.8), 5.08 (ddd, 1 H, J = 3.9, 6.9, 8.4), 5.84 (d, 1 H, J = 8.4), 7.30–7.77 (m, 10 H)	61.19 (d), 61.88 (t), 79.55 (d), 127.5 (d), 128.4 (d), 129.0 (d), 129.3 (d), 129.7 (d), 132.6 (d), 134.8 (s), 136.1 (s), 154.1 (s), 169.4 (s)
4c	21	130.5–132.5	1785, 1692	0.03 (s, s, 6 H), 0.91 (s, 9 H), 3.52 (d, 2 H, J = 5.0), 4.86 (dd, 1 H, J = 5.0, 8.1), 5.69 (d, 1 H, J = 8.1), 7.22–7.84 (m, 9 H)	5.70 (q), 18.17 (s), 25.68 (q), 60.52 (d), 60.86 (t), 77.42 (d), 127.9 (d), 128.3 (d), 128.8 (d), 129.2 (d), 132.6 (d), 132.7 (s), 132.8 (s), 134.5 (s), 153.1 (s), 168.9 (s)
4d	16	142–144	1780, 1688	2.95 (s, 3 H), 3.31 (s, 1 H), 3.36 (t, 1 H, J = 5.5), 4.13 (t, 1 H, J = 5.5), 5.03 (ddd, 1 H, J = 4.8, 5.9, 8.1), 5.77 (d, 1 H, 8.1), 7.11–7.79 (m, 9 H)	21.56 (q), 61.54 (d), 62.52 (t), 80.27 (d), 128.0 (d), 129.0 (d), 130.2 (d), 130.5 (d), 133.1 (d), 133.8 (s), 135.5 (s), 139.3 (s), 154.7 (s), 169.9 (s)
5e	51	88.0–89.5	1756, 1716	1.60 (s, 3 H), 1.16 (s 3 H), 4.10 (ddd, 1 H, J = 1.1, 4.7, 9.0), 4.43 (dd, 1 H, J = 4.7, 9.2), 4.50 (dd, 1 H, J = 9.0, 9.2), 6.98 (s, 1 H), 7.27–7.95 (m, 5 H)	21.39 (q), 21.39 (q), 60.26 (d), 65.99 (t), 81.92 (s), 128.2 (d), 128.4 (d), 129.4 (d), 130.6 (s), 133.0 (d), 160.4 (s), 165.3 (s)
5f	67	134–136	1740, 1720	1.35 (d, 3 H, J = 6.6), 4.04 (m, 1 H), 4.36 (dd, 1 H, J = 4.8, 8.8), 4.53 (t, 1 H, J = 8.8), 4.53 (t, 1 H, J = 8.8), 5.19 (m, 1 H), 6.52 (s, 1 H), 7.30–8.15 (m, 5 H)	15.06 (q), 55.55 (d), 66.20 (t), 71.15 (d), 128.4 (d), 129.5 (s), 129.6 (d), 133.2 (d), 160.1 (s), 165.7 (s)
5g	78	140.0–141.5	1750, 1720	4.21 (dd, 1 H, J = 9,4, 13.6), 4.32 (ddd, 1 H, J = 5.1, 8.6, 9.4), 4.33 (dd, 1 H, J = 8.6, 13.6), 5.92 (d, 1 H, J = 5.1), 6.14 (s, 1 H), 7.28–8.08 (m, 10 H)	56.30 (d), 66.35 (t), 77.27 (d), 126.7 (d), 128.6 (d), 129.1 (d), 129.2 (d), 129.3 (d), 129.8 (d), 133.5 (d), 135.6 (s), 159.5 (s), 165.5 (s)
5h	65	oil	1754, 1602	1.39 (s, 3 H), 4.11 (d, 1 H, J = 9.4), 4.51 (d, 1 H, J = 9.4), 5.70 (s, 1 H), 5.89 (s, 1 H), 7.22–8.20 (m, 10 H)	23.15 (q), 73.24 (t), 79.24 (d), 127.1 (d), 128.6 (d), 128.7 (d), 128.9 (d), 129.2 (s), 129.7 (d), 133.5 (d), 135.2 (s), 158.8 (s), 165.3 (s)

[a] Satisfactory microanalyses: C ± 0.18, H ± 0.07, N ± 0.03, and HRMS: (M^+) ± 0.0013 obtained for all products.
[b] The structure of **4c** was determined as its *tert*-butyldimethylsilyl ether.

Thus, we have demonstrated a novel Lewis acid mediated cyclization reaction of 2,3-epoxypropyl benzoylcarbamates. Whether the normal Baldwin's cyclization or the present one occurs, depends on the steric and electronic effects of its substituent at C-3 of the 2,3-epoxypropyl group.

Melting points are uncorrected. ^{1}H and ^{13}C NMR spectra were observed with JEOL JNM-GX 270, JNM-FX, GSX-400 and GSX-500 spectrometers. IR spectra were obtained on a JASCO A-202 and Hitachi I-2000 spectrophotometers. High pressure liquid chromatography (HPLC) was performed on a Merck Lichrosorb Si 60 column and Hitachi L-6000 Pump. HRMS data wer obtained on JMS-HX 110A spectrometer. THF was distilled from $LiAlH_4$. CH_2Cl_2 and DMF were distilled over CaH_2.

2,3-Epoxypropyl Benzoylcarbamates; General Procedure:
The benzoylcarbamates were synthesized from the corresponding alkenyl alcohols and benzoyl isocyanate by the known method.[6] To a solution of a benzoylcarbamate in CH_2Cl_2 at 0 °C was added MCPBA (1.5–2.0 molar equiv) and the mixture was stirred for 24 h, quenched with sat. aq $NaHCO_3$, brine, dried ($MgSO_4$) and concentrated. The residue was purified by flash column chromatography or recrystallization (Table 1).

Treatment of 2,3-Epoxy-3-phenylpropyl Benzoylcarbamate (2a); Typical Procedure:
To a solution of **2a** (102 mg, 0.34 mmol) in dry CH_2Cl_2 (3 mL) at −15 °C was added dropwise Me_3Al (0.99 M solution in hexane, 1.36 mL, 1.36 mmol) and the mixture was stirred for 1.5 h. The reaction was monitored by TLC. The reaction was quenched by the addition of aqueous MeOH (10% in H_2O) and to this was added

CH_2Cl_2 (20 mL). The mixture was dried ($MgSO_4$) and concentrated in vacuo. Purification by flash column chromatography on silica gel using hexane/EtOAc (1 : 1) as eluant gave 95 mg of a mixture of products mixture. The mixture was further separated by HPLC using hexane/EtOAc (1 : 1) as eluant to give 54 mg (54%) of white crystals of **3a**, mp 147–149°C (from hexane/EtOAc), and 24 mg (24%) of **4a**, mp 156.5–158.5°C (from hexane/EtOAc).

Treatment of 2a with Equimolar Trimethylaluminum:
Compound **2a** was also treated with Me_3Al (1 equiv) at −20°C. Both produts, **3a** (58%) and **4a** (19%), were obtained, but the reaction required 9 h.

Treatment of 2a with Various Lewis Acids:
Treatment of **2a** with 4 molar equivalents of Et_2AlCl (4 equiv) gave 22% of **3a** and 19% of **4a**. Epoxy carbamate **2a** was also treated with 4 equiv of $TiCl_4$, $Ti(OPr\text{-}i)_4$, $Et_2O \cdot BF_3$ as described above. In the case of $TiCl_4$ and $Et_2O \cdot BF_3$ none of **3a** or **4a** was detected. From the reaction with $Ti(OPr\text{-}i)_4$ isopropyl benzoylcarbamate was isolated.

The Reaction of 3,4-Epoxyhexyl Benzoylcarbamate (2i) with Trimethylaluminum:
To a solution of **2i** (576 mg, 2.19 mmol) in dry CH_2Cl_2 (20 mL) at −20°C was added dropwise Me_3Al (0.99 M solution in hexane, 8.84 mL, 8.76 mmol) and the mixture was stirred for 6 h. The mixture was quenched with 1 N HCl and extracted with CH_2Cl_2. The organic layer was dried ($MgSO_4$) and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel using hexane/EtOAc (1 : 2) as eluant to give 238 mg (41%) of **7i** and 83 mg (14%) of **8i**.

4-[2-(Benzoyloxy)ethyl]-5-ethyloxazolidin-2-one (**7i**):

IR (neat): ν = 3284, 2972, 1740, 1600, 1398, 1112, 992 and 714 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.01 (t, 3H, J = 7.5 Hz), 1.67–1.82 (m, 2H), 1.92–2.10 (m, 2H), 3.61 (ddt, 1H, J = 0.7, 5.5, 6.1 Hz), 4.21 (dt, 1H, J = 5.5, 7.1 Hz), 4.39 (dt, 1H, J = 5.6, 11.7 Hz), 4.53 (ddd, 1H, J = 4.9, 7.7, 11.6 Hz), 6.26 (s, 1H), 7.44–7.48 (m, 2H), 7.56–7.61 (m, 1H), 8.00–8.03 (m, 2H).

^{13}C NMR (100.4 MHz, $CDCl_3$): δ = 9.05 (q), 27.64 (t), 34.69 (t), 55.13 (d), 61.34 (t), 83.53 (d), 128.50 (d), 129.60 (d), 129.70 (s), 133.30 (d), 159.30 (s, C=O), 166.50 (s, C=O).

HRMS (FAB): m/z, $C_{14}H_{18}N_4O$ calc: 264.1236. (M + 1); found: 264.1249 (M^+ + 1).

4-[1-(Benzoyloxy)propyl]tetrahydro-2H-1,3-oxazin-2-one (**8i**):

IR (neat): ν = 3260, 2972, 1714, 1602, 1264, 1098, 948 and 716 cm^{-1}.

^1H NMR (270 MHz, $CDCl_3$): δ = 0.98 (t, 3H, J = 7.4 Hz), 1.65–1.94 (m, 3H), 1.96–2.14 (m, 1H), 3.77 (dddd, 1H, J = 1.2, 5.6, 5.9, 8.9 Hz), 4.20 (ddd, 1H, J = 3.2, 10.0, 11.1 Hz), 4.32 (dt, 1H, J = 4.6, 11.1 Hz), 5.05 (ddd, 1H, J = 4.8, 5.9, 7.6 Hz), 6.32 (d, 1H, J = 1.2 Hz), 7.38–7.62 (m, 3H), 7.99–8.09 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 9.41 (t), 23.59 (t), 23.98 (t), 52.37 (d), 65.10 (t), 76.79 (d), 128.50 (d), 129.50 (s), 129.80 (d), 133.40 (d), 154.20 (s, C=O), 166.10 (s, C=O).

HRMS (FAB): m/z, $C_{14}H_{18}NO_4$, calc.: 264.1236 (M^+ + 1); found: 264, 1267 (M^+ + 1).

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