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An efficient synthetic route to functionalized δ-lactams

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ABSTRACT

This paper describes a convenient synthesis of disubstituted functionalized δ-lactams based on Michael addition of primary amines to dimethyl-*E*-2-alkylidene glutarates **2** followed by an intramolecular cyclisation.

1. Introduction

Six-membered nitrogen-containing heterocycles are common skeletons in many natural products and exhibit diverse and important biological properties.[1,2] Alkaloids that contain the piperidine ring are targets for medicinal chemistry.[3–7] Accordingly, functionalized δ-lactams have been generally used as precursors of the corresponding piperidines.[8–10] Functionalized δ-lactams and structurally related compounds have attracted considerable attention because of various biological activities, including anti-tumour compounds,[11] enzyme inhibitors[12–14] and anti-HIV agents.[15] Over the last few years, there is an increasing interest in the development of general methods for their preparation and several syntheses of these heterocycles, using different approaches, have been reported.[16–24] Our interest in the synthesis of polyfunctionalized heterocyclic compounds [25–30] has led us to the design of a rapid access to these functionalized δ-lactams using dimethyl (*E*)-2-alkylidene glutarates **2** as key intermediates.

2. Results and discussion

We have previously described a highly stereoselective synthesis of dimethyl (*E*)-2-alkylidene glutarates **2** by nucleophilic substitution of the vinylic bromine atom in the diester **1**, by using cuprates as nucleophilic reagents generated in situ at low temperature (Scheme 1).[31] The dimethyl (*E*)-2-bromomethylene glutarate **1** was prepared through a simple tandem-process: bromination–dehydrobromination of dimethyl-2-methylene glutarate **2a**.[32]

As summarized in Scheme 2, we have recently demonstrated that glutarates **2** represent a useful building block for the enantioselective synthesis of functionalized γ-butyrolactones by the utilisation of the Sharpless asymmetric dihydroxylation (AD) and aminohydroxylation (AA) processes,[33] while the silylcupration of the same Michael acceptors **2**, followed by oxidation of the carbon–silicon bond and cyclisation provide the corresponding functionalized δ-lactones.[34]

We now report the use of dimethyl (*E*)-2-alkylidene glutarates **2** as intermediates in the synthesis of functionalized δ-lactams. In our approach, the construction of the nitrogen-containing heterocycle is based on an efficient coupling of primary amines to Michael acceptors **2**. In fact, the condensation of glutarates **2** with primary amines in methanol, as solvent, at reflux proceeds via a two-step sequence: a nucleophilic conjugate addition of amine to the activate ethylenic carbon leading to the β-amino-ester intermediate, which spontaneously undergoes an intramolecular cyclisation through a 6-*exo-trig* process [35] to provide the corresponding functionalized δ-lactam **3** (Scheme 3).

It is interesting to notice that spontaneous lactamization of the resulting β-aminoester intermediate was completely regioselective in the examined cases; only δ-lactams were obtained in moderate and good yields. The disubstituted δ-lactams **3e–g** were obtained as a mixture of two isomers with good diatereoselectivity and satisfactory yields (Table 1). A small coupling constant (4–5 Hz) for the two protons at C-2 (δ 4.2–3.9) and C-3 (δ 2.9–3.1) suggested a cis configuration for the major isomer of the disubstituted lactams **3e–g**. This result allowed us to establish the configuration of the β-amino-ester precursor. The major isomer was found to possess

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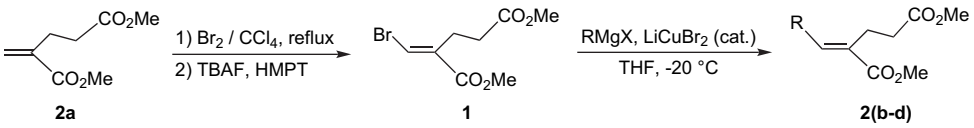


Scheme 1.

the *syn*-relative configuration (Scheme 3). The configuration of the β-amino-ester intermediate is determined during protonation in the aza-Michael addition of primary amines on the alkylidene glutarates **2b–d**. It is well recognized that aza-Michael addition on unsaturated esters is the most direct method used in the preparation of β-aminoesters.[36–40] However, this reaction is limited by steric factors imposed by the presence of α and/or β-substituents, as evidenced by Pfau's pioneering results.[41] Interestingly, we found that diastereoselectivity increases with the size of R group in **2b–d**, consequently we examined two transition state conformations **TS-1** and **TS-2**, in which H^+ would approach the enolate face anti-relative to the bulky amino group (Scheme 4).

TS-2 shows an increasingly important $A_{1,2}$-allylic strain when the size of R group becomes larger and, therefore it will be disfavoured relatively to **TS-1** in which this interaction is minimised. The diastereocontrol then improves with large R groups and the relative *syn*-diastereomer being largely predominant, which is corroborated by the experiment (Table 1).

Table 1
Functionalized δ-lactams **3a–g** prepared

δ-lactams	R	R′	Cis/trans[a]	Yield (%)[b]
3a	H	$PhCH_2$	—	82
3b	H	*p*-$MeOC_6H_4$-CH_2	—	92
3c	H	*p*-FC_6H_4-CH_2	—	80
3d	H	^{i}Pr	—	65
3e	Me	$PhCH_2$	78:22	55
3f	Et	$PhCH_2$	84:16	63
3g	Pr	$PhCH_2$	92:8	59

[a] Calculated by integration of the OMe signals in **3e–g**.
[b] Yields referred to isolated pure product.

3. Conclusion

In summary, an efficient and rapid method for the synthesis of functionalized disubstituted δ-lactams **3** has been developed by an effective coupling between dimethyl-2-alkylidene glutarates **2** and primary amines. The advantages of this method include using readily inexpensive available starting materials and operational simplicity. Extension of this method to a more functionalised Michael acceptor and its application to the synthesis of alkaloids skeletons of biological relevance is in progress.

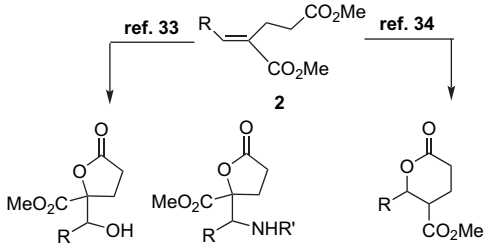
4. Experimental

4.1. General

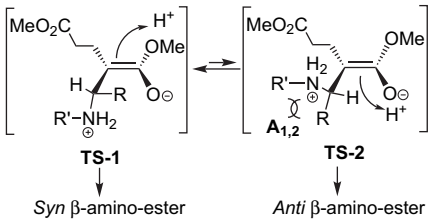
^1H and ^{13}C NMR spectra were recorded on Bruker AC-300 FT (^1H: 300 MHz, ^{13}C: 75 MHz) with $CDCl_3$ as internal reference. The chemical shifts (δ) and coupling constants (J) are, respectively, expressed in parts per million and hertz. IR spectra were recorded with a Perkin–Elmer paragon 1000 FT-IR spectrophotometer. Mass spectra MS were recorded on a Hewlett–Packard 5989A apparatus (EI with 70 eV ionisation potential). Elemental analyses were carried out with a Perkin–Elmer 240 B microanalyser. Merck silica gel 60 (70–230 mesh) and (0.063–0.200 mm) were used for flash chromatography. All reactions involving anhydrous conditions were conducted in dry glassware under a nitrogen atmosphere. Solvents were distilled under nitrogen immediately prior to use. Grignard reagents were prepared by known methods and stored under inert atmosphere. They were titrated prior to use with a 1 M solution of benzyl alcohol in anhydrous toluene and in the presence of 2,2′-bipyridil as indicator.[42]

4.2. Synthesis of (*E*)-dimethyl 2-alkylidene glutarates 2b–d: general procedure

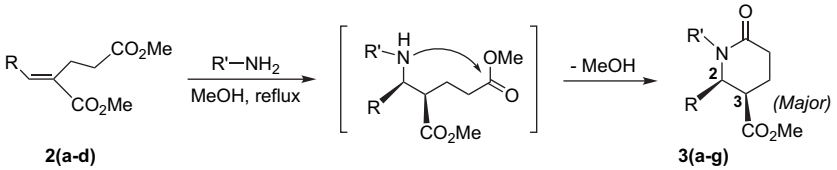
An ether or THF solution of alkylmagnesium halide RMgX (2–3 M) was added dropwise over a period of 20–30 min to a stirred mixture of dimethyl (*E*)-2-bromomethylene glutarate **1** (1.25 g, 5 mmol) and a 1 M solution of $LiCuBr_2$ (0.15 mL, 3 mol %) diluted in dry THF (20 mL) at −20 °C under nitrogen atmosphere. After a few minutes (TLC), the reaction mixture was quenched with a saturated aqueous NH_4Cl solution (10 mL) then extracted with ether (3×20 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude



Scheme 2.



Scheme 4.



Scheme 3.

product was purified by flash chromatography on silica gel (AcOEt/hexane, 1:9) to produce (*E*)-dimethyl-2-alkylidene glutarates **2b–d**.

*4.2.1. (E)-2-Ethylidene pentanedioic acid dimethyl ester **2b***

Colourless oil. IR (film) ν_{max} 1704, 1697, 1642 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 6.91 (q, 1H, *J*=7.0 Hz, CH), 3.70 (s, 3H, CH_3), 3.64 (s, 3H, CH_3), 2.66–2.48 (m, 2H, CH_2), 2.47–2.32 (m, 2H, CH_2), 1.81 (d, 3H, *J*=7.0 Hz, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.4 (C=O), 167.7 (C=O), 139.2 (CH), 131.2 (C), 51.7 (CH_3), 51.5 (CH_3), 33.1 (CH_2), 21.9 (CH_2), 14.2 (CH_3). MS (EI, 70 eV) *m*/*z* (%) 186 (M^+, 2), 171 (48), 154 (100), 127 (72), 113 (64), 99 (30).

*4.2.2. (E)-2-Propylidene pentanedioic acid dimethyl ester **2c***

Colourless oil. IR (film) ν_{max} 1716, 1702, 1651 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 6.84 (t, 1H, *J*=7.3 Hz, CH), 3.78 (s, 3H, CH_3), 3.65 (s, 3H, CH_3), 2.71–2.60 (m, 2H, CH_2), 2.58–2.35 (m, 2H, CH_2), 2.28 (qt, 2H, *J*=7.6, 7.2 Hz CH_2), 1.08 (t, 3H, *J*=7.6 Hz, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.3 (C=O), 167.9 (C=O), 146.2 (CH), 129.7 (C), 51.9 (CH_3), 51.3 (CH_3), 33.4 (CH_2), 23.0 (CH_2), 20.4 (CH_2), 13.9 (CH_3). MS (EI, 70 eV) *m*/*z* (%) 200 (M^+, 4), 171 (62), 168 (100), 141 (74), 140 (42), 127 (39).

*4.2.3. (E)-2-Butylidene pentanedioic acid dimethyl ester **2d***

Viscous yellow oil. IR (film) ν_{max} 1728, 1704, 1642 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 6.81 (q, 1H, *J*=7.6 Hz, CH), 3.71 (s, 3H, CH_3), 3.65 (s, 3H, CH_3), 2.79–2.57 (m, 2H, CH_2), 2.54–2.33 (m, 2H, CH_2), 2.23 (q, 2H, *J*=7.4 Hz, CH_2), 1.42–1.28 (m, 2H, CH_2), 0.96 (t, 3H, *J*=6.8 Hz, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.4 (C=O), 167.2 (C=O), 139.2 (CH), 131.2 (C), 51.5 (CH_3), 51.3 (CH_3), 31.7 (CH_2), 28.8 (CH_2), 24.1 (CH_2), 23.2 (CH_2), 14.2 (CH_3). MS (EI, 70 eV) *m*/*z* (%) 214 (M^+, 2), 185 (50), 182 (100), 155 (39), 141 (61), 73 (54), 59 (68).

4.3. Preparation of δ-lactams 3a–g: general procedure

A solution of (*E*)-dimethyl-2-alkyldiene glutarate **2** (3 mmol) and an excess of primary amine (9 mmol, 3 equiv) in methanol (7 mL) was stirred at reflux for 48–72 h. The reaction mixture was concentrated under reduced pressure to remove methanol, then the crude product was purified by flash chromatography on silica gel (AcOEt/hexane, 1:1) to afford the corresponding δ-lactam **3**.

*4.3.1. 1-Benzyl-6-oxo-piperidine-3-carboxylic acid methyl ester **3a***

Viscous colourless oil. IR (film) ν_{max} 1735, 1696 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 7.81–6.94 (m, 5H, aromatic H), 4.6 (AB, 2H, J_{AB}=14.7 Hz, CH_2), 3.6 (s, 3H, CH_3), 3.4 (t, 2H, *J*=8.5 Hz, CH_2), 2.89–2.74 (m, 1H, CH), 2.62–2.48 (m, 2H, CH_2), 2.2–1.9 (m, 2H, CH_2). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.3 (C=O), 168.8 (C=O), 136.8 (aromatic C), 128.5 (aromatic C), 128.4 (aromatic CH), 128.1 (aromatic CH), 52.0 (CH_3), 50.1 (CH_2), 47.9 (CH_2), 39.0 (CH), 30.6 (CH_2), 23.8 (CH_2). Anal. Calcd for $C_{14}H_{17}NO_3$: C, 68.00; H, 6.93; N, 5.66. Found: C, 68.05; H, 6.98; N, 5.59.

*4.3.2. 1-(4-Methoxy-benzyl)-6-oxo-piperidine-3-carboxylic acid methyl ester **3b***

Viscous yellow oil. IR (film) ν_{max} 1731, 1695 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 7.1 (d, 2H, *J*=8.8 Hz, aromatic H), 6.8 (d, 2H, *J*=8.8 Hz, aromatic H), 4.6 (AB, 2H, J_{AB}=14.9 Hz, CH_2), 3.7 (s, 3H, CH_3), 3.6 (s, 3H, CH_3), 3.4 (t, 2H, *J*=8.5 Hz, CH_2), 2.9–2.7 (m, 1H, CH), 2.65–2.45 (m, 2H, CH_2), 2.2–1.9 (m, 2H, CH_2). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6 (C=O), 168.7 (C=O), 159.0 (aromatic C), 129.5 (aromatic C), 128.9 (aromatic CH), 113.9 (aromatic CH), 55.2 (CH_3), 52.1 (OCH_3), 49.5 (CH_2), 39.1 (CH), 30.7 (CH_2), 23.9 (CH_2), 21.0 (CH_2). Anal. Calcd for $C_{15}H_{19}NO_4$: C, 64.97; H, 6.91; N, 5.05. Found: C, 64.89; H, 7.08; N, 5.11.

*4.3.3. 1-(4-Fluoro-benzyl)-6-oxo-piperidine-3-carboxylic acid methyl ester **3c***

Colourless oil. IR (film) ν_{max} 1734, 1692 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 7.2 (dd, 2H, *J*=8.3, 5.5 Hz, aromatic H), 7.0 (t, 2H, *J*=8.4 Hz, aromatic H), 4.6 (AB, 2H, J_{AB}=14.7 Hz, CH_2), 3.6 (s, 3H, CH_3), 3.4 (t, 2H, *J*=8.5 Hz, CH_2), 2.85–2.64 (m, 1H, CH), 2.5–2.3 (m, 2H, CH_2), 2.1–1.9 (m, 2H, CH_2). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.5 (C=O), 168.8 (C=O), 163.8 (aromatic C, J_{C-F}=245.7 Hz), 132.7 (aromatic C), 129.9 (aromatic CH), 115.5 (aromatic CH), 52.1 (CH_3), 49.5 (CH_2), 48.0 (CH_2), 39.0 (CH), 30.6 (CH_2), 23.8 (CH_2).

*4.3.4. 1-Isopropyl-6-oxo-piperidine-3-carboxylic acid methyl ester **3d***

Viscous oil. IR (film) ν_{max} 1704, 1697, 1642 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 3.8 (qt, 1H, *J*=7.3 Hz, CH), 3.6 (s, 3H, CH_3), 3.5 (AB, 2H, J_{AB}=14.7 Hz, CH_2), 2.9–2.7 (m, 1H, CH), 2.45–2.26 (m, 2H, CH_2), 2.2–1.85 (m, 2H, CH_2), 1.3 (d, 6H, *J*=7.3 Hz, $2CH_3$). ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.6 (C=O), 172.5 (C=O), 55.2 (CH_3), 49.5 (CH_2), 44.2 (CH), 43.8 (CH), 33.9 (CH_2), 28.0 (CH_2), 22.5 ($2CH_3$).

*4.3.5. 1-Benzyl-2-methyl-6-oxo-piperidine-3-carboxylic acid methyl ester **3e** (major isomer)*

Yellow oil. IR (film) ν_{max} 1733, 1692 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 7.8–6.8 (m, 5H, aromatic H), 4.4 (AB, 2H, J_{AB}=15.8 Hz, CH_2), 3.9 (dq, 1H, *J*=6.9, 5.7 Hz, CH), 3.7 (s, 3H, CH_3), 3.1–2.75 (m, 1H, CH), 2.72–2.56 (m, 2H, CH_2), 2.48–2.22 (m, 2H, CH_2), 1.9 (d, 3H, *J*=6.9 Hz, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.3 (C=O), 168.1 (C=O), 139.0 (aromatic C), 131.5 (aromatic C), 128.7 (aromatic CH), 126.4 (aromatic CH), 51.7 (CH_2), 50.2 (CH_3), 46.1 (CH), 43.5 (CH), 30.7 (CH_2), 23.9 (CH_2), 15.8 (CH_3). Anal. Calcd for $C_{15}H_{19}NO_3$: C, 68.94; H, 7.33; N, 5.36. Found: C, 68.99; H, 7.34; N, 5.42.

*4.3.6. 1-Benzyl-2-ethyl-6-oxo-piperidine-3-carboxylic acid methyl ester **3f** (major isomer)*

Colourless oil. IR (film) ν_{max} 1746, 1712 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 7.4–7.2 (m, 5H, aromatic H), 4.6 (AB, 2H, J_{AB}=15.7 Hz, CH_2), 4.2 (dt, 1H, *J*=7.3, 4.5 Hz, CH), 3.6 (s, 3H, CH_3), 3.3–2.9 (m, 1H, CH), 2.28–2.25 (m, 2H, CH_2), 2.2–1.9 (m, 2H, CH_2), 1.75–1.4 (m, 2H, CH_2), 0.8 (t, 3H, *J*=7.6 Hz, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.4 (C=O), 171.9 (C=O), 138.3 (aromatic C), 132.7 (aromatic C), 129.8 (aromatic CH), 127.1 (aromatic CH), 51.8 (CH), 51.3 (CH_3), 49.7 (CH_2), 45.7 (CH), 34.9 (CH_2), 25.6 (CH_2), 23.0 (CH_2), 17.8 (CH_3). Anal. Calcd for $C_{16}H_{21}NO_3$: C, 69.79; H, 7.69; N, 5.09. Found: C, 69.72; H, 7.73; N, 5.12.

*4.3.7. 1-Benzyl-2-propyl-6-oxo-piperidine-3-carboxylic acid methyl ester **3g** (major isomer)*

Viscous oil. IR (film) ν_{max} 1731, 1695 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 7.3–7.1 (m, 5H, aromatic H), 4.5 (AB, 2H, J_{AB}=13.9 Hz, CH_2), 3.9 (dt, 1H, *J*=7.8, 5.4 Hz, CH), 3.7 (s, 3H, CH_3), 3.1–2.8 (m, 1H, CH), 2.45–2.25 (m, 2H, CH_2), 2.15–1.72 (m, 2H, CH_2), 1.7–1.5 (m, 2H, CH_2), 1.45–1.35 (m, 2H, CH_2), 0.9 (t, 3H, *J*=7.0 Hz, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.0 (C=O), 172.4 (C=O), 144.7 (aromatic C), 138.2 (aromatic CH), 130.4 (aromatic CH), 128.7 (aromatic CH), 52.0 (CH_3), 50.1 (CH_2), 48.9 (CH), 43.0 (CH), 35.6 (CH_2), 29.5 (CH_2), 24.3 (CH_2), 21.0 (CH_2), 13.8 (CH_3).

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