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1. General

Melting Points: Melting points were recorded on a Griffin melting point apparatus, and are uncorrected.

Infra-red spectra: Infra-red spectra were recorded on a Bruker Tensor 27 Fourier Transform spectrometer, as a thin film between NaCl plates or pressed into a KBr disk. Absorption maxima ($v_{\text{max}}$) are described as s (strong), m (medium), w (weak), br (broad) and are quoted in wavenumbers (cm$^{-1}$).

NMR Spectra: Proton ($^1$H) and Carbon ($^{13}$C) NMR spectra were recorded using either Bruker AC200 (200 MHz), Bruker AMX400 (400 MHz), or Bruker AVII500 (500 MHz) spectrometers. Chemical shifts are quoted in parts per million (ppm) downfield of tetramethylsilane with residual solvent as the internal standard. Assignments were made on the basis of chemical shifts, coupling constants, COSY, HMQC and n.O.e. data, comparison with spectra of related compounds, and comparison with spectra predicted by ACD/NMR predictor v8.08 or later. Resonances are described as s (singlet), d (doublet), t (triplet), q (quartet), quin (quintet), se (sextet), hep (heptet), oct (octet), app (apparent), and br (broad). Coupling constants ($J$) are given in Hz and are rounded to the nearest 0.1 Hz. Where two coupling constants were recorded for a single interaction, the average was recorded. Multiplets structures were confirmed using the J-coupler function on ACD NMR SpecManager v8.08 or later.

Mass spectra: Mass spectra were recorded on a VG Platform spectrometer using atmospheric pressure chemical ionisation from the solvent system 40:40:20 CH$_3$OH:CH$_3$CN:H$_2$O in positive (APCI+) or negative (APCI–) scan mode as
indicated. GC mass spectra were recorded on an Agilent 6890 Series GC System spectrometer. Other mass spectra were recorded on VG Micromass ZAB IF or Masslab 20-250 spectrometers. \( m/z \) values are reported with their percentage abundances, although peaks with a relative intensity less than 10% are not reported. High resolution mass spectra were recorded by the author, Mr. Robin Procter, or Dr. James McCullah at the Chemistry Research Laboratory, and are calculated to four decimal places from the molecular formula.

**Nomenclature:** All compounds were named and numbered using the ChemDraw Ultra 12.0 software package.

**Chromatography techniques:** Thin layer chromatography was performed on Merck DC-Alufolien 60 F\(_{254} \) 0.2 mm precoated plates, and Fluka Alufolien aluminium oxide plates. Product spots were visualised by quenching of UV fluorescence (\( \nu_{\text{max}} = 254 \) nm), then stained and heated using acidic vanillin solution, basic potassium permanganate solution, \( p \)-anisaldehyde, Dragendorff's reagent, iodine on silica, or ninhydrin as appropriate. Flash column chromatography was carried out on silica gel (ICN Silica 32-63 60 Å), or alumina gel (Fluka aluminium oxide for chromatography), and the solvent system used is quoted in parentheses.

**Solvents and reagents:** CH\(_2\)Cl\(_2\), pentane, toluene, THF and Et\(_2\)O were dried by filtration through an activated alumina purification column. Triethylamine, pyridine, and diisopropylamine were dried by distilling over calcium hydride, and stored over calcium hydride or 3 Å molecular sieves. HMPA was dried over 3 Å molecular sieves for 1 month prior to use. Reagents obtained from Acros, Aldrich, Avocado, Fluka and
Lancaster fine chemicals suppliers were used directly as supplied or following purification according to procedures described by Perrin and Armarego. All non-aqueous reactions were carried out under an atmosphere of argon using flame dried glassware. All reactions were carried out at room temperature unless otherwise stated. 'Petrol' refers to the fraction of light petroleum ether boiling in the range 40-60 °C.

2. General Procedures

General Procedure A

NaH (60% by weight in mineral oil, 1.5 eq.) was added portionwise to a solution of alcohol in DMF (1 mL/mmol) under argon at 0 °C. The suspension was stirred at room temperature for 1 hour, before the addition of the benzyl halide (1.5 eq.). The reaction was allowed to warm to room temperature and stirred for 2 hours before being quenched with saturated aqueous NH₄Cl solution. The organic phase was separated, and the aqueous phase was extracted with ethyl acetate (10 mL/mmol ×3). The combined organic layers were dried over MgSO₄, filtered, and the resultant mixture was concentrated in vacuo to afford the crude product.

General Procedure B

n-BuLi (1.6 M solution in hexanes, 1.2 eq.) was added dropwise to a stirred solution of alkyne in THF (4 mL/mmol) at –78 °C under argon. After 1 hour, para-formaldehyde (6 eq.) was added portionwise, and the resultant mixture was allowed to warm to room temperature over 4 hours, before being quenched by addition of an aqueous saturated solution of NH₄Cl (10 mL/mmol). The organic phase was separated, and the aqueous phase was extracted with ethyl acetate (10 mL/mmol ×3). The combined
organic layers were dried over MgSO$_4$, filtered, and the resultant mixture was concentrated in vacuo to afford the crude product.$^3$

**General Procedure C**

Lindlar’s catalyst (50 mg/mmol) was added to a solution of alkyne in ethyl acetate:pyridine (95:5, 5 mL/mmol), and the resultant mixture was degassed and subsequently flushed with hydrogen three times before being stirred at room temperature under an atmosphere of hydrogen for 30 minutes. The reaction mixture was filtered through a thin bed of Celite, and washed with ethyl acetate (50 mL/mmol). The resultant mixture was concentrated in vacuo to afford the crude product.$^4$

**General Procedure D**

$N,N'$-Carbonyldiimidazole (3 eq.) was added to a stirred solution of alcohol in pyridine (2 mL/mmol) at 40 °C. After TLC analysis indicated that complete consumption of the starting material had occurred, the mixture was cooled to 0 °C, and hydroxylamine hydrochloride (10 eq.) was added. After warming to room temperature, the reaction was quenched with 1 M HCl (10 mL/mmol), and the aqueous layer was extracted with diethyl ether (10 mL/mmol) and ethyl acetate (10 mL/mmol). The combined organic layers were then washed with water (10 mL/mmol) and brine (10 mL), dried over Na$_2$SO$_4$ and filtered before the solvents were azeotropically removed with toluene. Triethylamine (0.9 eq.) was then added to a solution of the resultant crude product in diethyl ether (5 mL/mmol) at 0 °C under argon, and trimethylbenzoyl chloride (0.9 eq.) was added dropwise. After warming to room temperature overnight, the reaction was quenched with 1 M HCl (10 mL/mmol), and the aqueous layer was
extracted with Et₂O (10 mL/mmol ×2). The combined organic layers were then washed sequentially with water (10 mL/mmol), an aqueous saturated solution of NaHCO₃ (10 mL/mmol) and brine (10 mL/mmol), before being dried over Na₂SO₄, filtered and concentrated in vacuo to afford the crude product.⁵

**General procedure E**

Potassium osmate dihydrate (4 mol%) in water (0.5 mL/mmol) was added to a stirred solution of benzoyloxy carbamate in tert-butanol:water (3:1, 20 mL/mmol). After TLC analysis indicated that complete consumption of the starting material had occurred, the reaction was quenched by addition of Na₂SO₃ (200 mg/mmol) and the resultant solution was left to stir for 30 minutes. The solvents were then azeotropically removed with toluene to afford the crude product.⁵

**General procedure F**

10% by weight palladium on carbon (30 mol%) was added to a solution of benzyl ether in ethanol (10 mL/mmol), and the resultant mixture was degassed under vacuum for 30 seconds and subsequently flushed with hydrogen three times before being stirred at room temperature under an atmosphere of hydrogen overnight. The reaction mixture was filtered through a thin bed of Celite, and washed with a mixture of dichloromethane:methanol (5:1, 50 mL/mmol). The resultant solution was concentrated in vacuo to afford the crude product.⁶

**General procedure G**

Amberlyst H-15 strong acid cation exchange resin (30 mg/mmol) was added to a solution of diol in acetone (20 mL/mmol) and the resultant solution was stirred at 40
After complete consumption of the starting material was confirmed by TLC analysis, the resultant mixture was concentrated \textit{in vacuo} to afford the crude product.\(^7\)

**General procedure H**

2,3-Dichloro-5,6-dicyanobenzoquinone was added to a stirred solution of \textit{para}-methoxybenzyl ether in CH\(_2\)Cl\(_2\):water (20:1, 5 mL/mmol). After complete consumption of the starting material was confirmed by TLC analysis, the reaction was diluted with water (10 mL/mmol). The organic phase was separated, and the aqueous phase was extracted with ethyl acetate (10 mL/mmol ×3). The combined organic layers were dried over MgSO\(_4\), filtered, and the resultant mixture was concentrated \textit{in vacuo} to afford the crude product.\(^8\)

**General procedure I**

tert-Butyldimethylsilyl chloride (1.5 eq.), imidazole (2.5 eq.), and DMAP (5 mol%) were added to a stirred solution of alcohol in CH\(_2\)Cl\(_2\) (4 mL/mmol) at room temperature under argon. After complete consumption of the starting material was confirmed by TLC, the reaction was quenched by addition of an aqueous saturated solution of NH\(_4\)Cl (10 mL/mmol). The organic phase was separated, and the aqueous phase was extracted with ethyl acetate (10 mL/mmol ×3). The combined organic layers were dried over MgSO\(_4\), filtered, and the resultant mixture was concentrated \textit{in vacuo} to afford the crude product.

**General procedure J**

Silyl ether was dissolved in a mixture of EtOH:concentrated HCl (99:1, 10 mL/mmol) and stirred at 70 °C. After complete consumption of the starting material was
confirmed by TLC analysis, the solvent and silicon residues were removed \textit{in vacuo} to afford the crude product.$^9$

\textbf{General Procedure K}

\textit{n}-BuLi (1.6 M solution in hexanes, 1 equivalent) was added dropwise to a stirred solution of alkyne in THF (4 mL/mmol) at \textdegree 78 °C under argon. After 1 hour, a solution of aldehyde in THF (4 mL/mmol, 1.2 eq.) was added dropwise, and the resultant mixture was allowed to warm to room temperature over 4 hours, before being quenched by addition of an aqueous saturated solution of NH$_4$Cl (10 mL/mmol). The organic phase was separated, and the aqueous phase was extracted with ethyl acetate (10 mL/mmol \times 3). The combined organic layers were dried over MgSO$_4$, filtered, and the resultant mixture was concentrated \textit{in vacuo} to afford the crude product.$^{10}$

\textbf{General Procedure L}

Acetic anhydride (5 mL/mmol) was added to a solution of alcohol in pyridine (5 mL/mmol) and the resulting solution was stirred overnight. After the starting material had been consumed as indicated by TLC analysis, the solvents were removed azeotropically with toluene to afford the crude product.$^{11}$

\textbf{General procedure M}

Triethylamine (2 eq.) and pyridine (2 eq.) were added to the alcohol in diethyl ether (5 ml/mmol) at 0 °C under argon, followed by the dropwise addition of the acid chloride (2 eq.). After warming to room temperature overnight, the reaction was quenched with 1 M HCl (10 mL/mmol) and the aqueous layer was extracted with Et$_2$O (10 mL/mmol \times 3). The combined organic layers were then washed with water (10 mL/mmol) and
brine (10 mL/mmol), before being dried over Na₂SO₄, filtered and concentrated in vacuo to afford the crude product.⁰¹²

**General procedure N**

TBAF (1.0 M solution in THF, 1.5 eq.) was added dropwise to a stirred solution of silyl ether in THF (10 mL/mmol) at 0 °C under argon. After TLC analysis indicated that complete consumption of the starting material had occurred, the resultant mixture was concentrated in vacuo to afford the crude product.⁰¹³

**General procedure O**

Commercially available ethynyl magnesium bromide (0.5 M solution in THF, 1.5 eq.) was added dropwise to a stirred solution of aldehyde in THF (10 mL/mmol) at –78 °C under argon. The resultant solution was allowed to warm to room temperature over 4 hours, before being quenched by addition of a saturated aqueous solution of NH₄Cl (10 mL/mmol). The organic phase was separated, and the aqueous phase was extracted with diethyl ether (10 mL/mmol ×3). The combined organic layers were dried over Na₂SO₄, filtered, and the resultant mixture was concentrated in vacuo to afford the crude product.⁰¹⁴

**General procedure P**

Red-Al (65% by weight in toluene, 2 eq.) was added dropwise to a stirred solution of alkyne in THF (6 mL/mmol) at –20 °C, and the reaction was stirred at the same temperature for 20 hours before being quenched at –78 °C with a saturated aqueous solution of Rochelle’s salt (3 mL/mmol). The mixture was then allowed to warm to room temperature before being stirred vigorously for 16 hours. The aqueous layer was
extracted with Et₂O (10 mL/mmol ×3), and the combined organic layers were washed with water (10 mL/mmol) and brine (10 mL/mmol), before being dried over MgSO₄, filtered, and concentrated in vacuo to afford the crude product.¹⁵

**General procedure Q**

DiBALH (1.0 M solution in hexanes, 2.4 eq.) was added dropwise to a stirred solution of ester in diethyl ether (3 mL/mmol) at −78 °C under argon. After TLC analysis confirmed that complete consumption of the starting material had occurred, the reaction was quenched with ice cold 6 M HCl (3 mL/mmol). The organic phase was separated, and the aqueous phase was extracted with diethyl ether (10 mL/mmol ×3). The combined organic layers were then washed sequentially with water (5 mL/mmol), and brine (5 mL/mmol), before being dried over Na₂SO₄, filtered and concentrated in vacuo to afford the crude product.¹⁶

**General Procedure R**

N,N’-Carbonyldiimidazole (1.5 eq.) was added to a stirred solution of alcohol in THF (10 mL/mmol) at room temperature. After TLC analysis indicated that complete consumption of the starting material had occurred, the reaction mixture was quenched with an aqueous saturated solution of NH₄Cl (4 mL/mmol), and the aqueous layer was extracted with diethyl ether (10 mL/mmol) and ethyl acetate (10 mL/mmol). The combined organic layers were then washed with brine (4 mL/mmol), dried over Na₂SO₄ and filtered before the resultant mixture was concentrated in vacuo to afford the crude intermediate carboxylat compound. This mixture was dissolved in pyridine (2 mL/mmmol) and the solution was cooled to 0 °C, before hydroxylamine hydrochloride (10 eq.) was added. After warming to room temperature, the reaction
was quenched with 1 M HCl (10 mL/mmol), and the aqueous layer was extracted with
diethyl ether (10 mL/mmol) and ethyl acetate (10 mL/mmol). The combined organic
layers were then washed with water (10 mL/mmol) and brine (10 mL), dried over
Na$_2$SO$_4$ and filtered before the solvents were azeotropically removed with toluene.
Triethylamine (0.9 eq.) was then added to a solution of the resultant crude product in
diethyl ether (5 ml/mmol) at 0 °C under argon, and trimethylbenzoyl chloride (0.9
eq.) was added dropwise. After warming to room temperature overnight, the reaction
was quenched with 1 M HCl (10 mL/mmol), and the aqueous layer was extracted with
Et$_2$O (10 mL/mmol ×2). The combined organic layers were then washed sequentially
with water (10 mL/mmol), an aqueous saturated solution of NaHCO$_3$ (10 mL/mmol)
and brine (10 mL/mmol), before being dried over Na$_2$SO$_4$, filtered and concentrated in
vacuo to afford the crude product.$^5,17$

3. Experimental Data

((Hex-1-yn-3-yloxy)methyl)benzene, 5a

![Chemical Structure]

Commercially available 1-hexyn-3-ol (1.00 g, 10.2 mmol) was subjected to general
procedure A. Flash column chromatography on silica gel (99:1 petrol:ethyl acetate)
afforded the title compound as a colourless oil (1.50 g, 7.96 mmol, 78%). Data were
consistent with those previously reported in the literature.$^{18}$

$\delta_H$ (400 MHz, CDCl$_3$) 0.98 (3 H, t, $J$ 7.5), 1.50 - 1.63 (2 H, m), 1.73 - 1.89 (2 H, m),
2.52 (1 H, d, $J$ 2.0), 4.15 (1 H, td, $J$ 6.6, 2.0), 4.56 (1 H, d, $J$ 11.9), 4.87 (1 H, d, $J$
11.9), 7.31 - 7.45 (5 H, m); $\delta_C$ (100 MHz, CDCl$_3$) 13.8, 18.5, 37.8, 68.3, 70.5, 73.8,
83.0, 127.7, 128.0, 128.4, 138.0.
4-(Benzyloxy)hept-2-yn-1-ol, 6a

Alkyne 27 (1.31 g, 6.97 mmol) was subjected to general procedure B. Flash column chromatography on silica gel (94:6 petrol:ethyl acetate) afforded the title compound as a colourless oil (1.19 g, 5.44 mmol, 78%).

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} 3383\text{br s}, 3031\text{m}, 2959\text{w}, 2871\text{s}, 1721\text{w}, 1496\text{m}, 1454\text{s}, 1334\text{s}, 1207\text{m}, 1151\text{s}, 1069\text{s}, 735\text{s}, 698\text{s}; \delta_{\text{H}} \text{ (400 MHz, CDCl}_3\text{)} 0.98 (3 \text{ H, t, } J 7.5), 1.50 - 1.63 (2 \text{ H, m}), 1.73 - 1.89 (3 \text{ H, m}), 4.15 (1 \text{ H, t, } J 6.6), 4.33 (2 \text{ H, s}), 4.56 (1 \text{ H, d, } J 11.9), 4.87 (1 \text{ H, d, } J 11.9), 7.31 - 7.45 (5 \text{ H, m}); \delta_{\text{C}} \text{ (100 MHz, CDCl}_3\text{)} 13.8, 18.6, 37.8, 51.2, 68.6, 70.6, 84.0, 85.0, 127.7, 127.9, 128.4, 138.0; \text{ HRMS (ESI)} \text{ C}_{14}\text{H}_{18}\text{NaO}_2 \text{ requires } 241.1199, \text{ found } 241.1199 \text{ (–0.11 ppm).}

(Z)-4-(Benzyloxy)hept-2-en-1-ol, 7a

Alcohol 5a (764 mg, 3.50 mmol) was subjected to general procedure C. Flash column chromatography on silica gel (94:6 petrol:ethyl acetate) afforded the title compound as a yellow oil (654 mg, 2.97 mmol, 85%).

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} 3385\text{br s}, 3029\text{w}, 2958\text{s}, 2931\text{s}, 2871\text{s}, 1496\text{w}, 1454\text{m}, 1388\text{m}, 1308\text{m}, 1205\text{m}, 1067\text{s}, 735\text{s}, 698\text{s}; \delta_{\text{H}} \text{ (400 MHz, CDCl}_3\text{)} 0.91 (3 \text{ H, t, } J 8.1), 1.26 - 1.50 (3 \text{ H, m}), 1.62 - 1.79 (2 \text{ H, m}), 4.06 - 4.15 (2 \text{ H, m}), 4.20 (1 \text{ H, dd, } J 12.0, 6.5), 4.38 (1 \text{ H, d, } J 11.9), 4.59 (1 \text{ H, d, } J 11.9), 5.49 (1 \text{ H, dd, } J 10.5, 9.5), 5.81 (1 \text{ H, app dt, } J 10.5, 6.5), 7.25 - 7.40 (5 \text{ H, m}); \delta_{\text{C}} \text{ (100 MHz, CDCl}_3\text{)} 14.0, 18.6, 37.7, 58.8,
70.1, 74.0, 127.6, 127.8, 128.4, 131.5, 133.3, 138.6; **HRMS (ESI)** C$_{14}$H$_{20}$NaO$_2$ requires 243.1356, found 243.1355 (0.3 ppm).

(Z)-4-(Benzyloxy)hept-2-enyl mesitoxy carbamate, 8a

![Chemical structure](attachment:image)

Alcohol 682 (637 mg, 2.89 mmol) was subjected to general procedure D. Flash column chromatography on silica gel (99:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (667 mg, 1.62 mmol, 56%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3275br s, 2959s, 1755s, 1611s, 1455s, 1227s, 1158s, 1093s, 851s, 736s; $\delta_H$ (400 MHz, CDCl$_3$) 0.91 (3 H, t, $J$ 7.6), 1.27 - 1.50 (3 H, m), 1.63 - 1.75 (1 H, m), 2.31 (3 H, s), 2.38 (6 H, s), 4.16 (1 H, ddd, $J$ 9.6, 7.4, 7.0), 4.37 (1 H, d, $J$ 11.9), 4.59 (1 H, d, $J$ 11.9), 4.72 (1 H, dd, $J$ 12.6, 6.8), 4.82 (1 H, dd, $J$ 12.6, 6.8), 5.65 (1 H, dd, $J$ 11.0, 9.6), 5.79 (1 H, dt, $J$ 11.0, 6.8), 6.90 (2 H, s), 7.25 - 7.41 (5 H, m), 8.31 (1 H, br s); $\delta_C$ (100 MHz, CDCl$_3$) 14.0, 18.5, 19.9, 21.2, 37.7, 62.4, 70.3, 73.9, 125.5, 127.6, 127.8, 128.4, 128.7, 126.4, 136.6, 136.7, 138.5, 141.0, 156.4, 169.0; **HRMS (ESI)** C$_{25}$H$_{31}$NNaO$_5$ requires 448.2094, found 448.2095 (–0.08 ppm).
Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-2'(benzyloxy)-1'-hydroxypentyl)oxazolidin-2-one (major) with (±) (R)-4-((1'R,2'R)-2'(benzyloxy)-1'-hydroxypentyl)oxazolidin-2-one (minor), anti- and syn-9a

Benzoyloxy carbamate 8a (150 mg, 353 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 170 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 340 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated in vacuo to deliver the title compound as a cream solid (74 mg, 260 µmol, 74%) and a mixture of diastereomers (3:1). Flash column chromatography on silica gel (3:1 to 1:1 petrol:ethyl acetate) allowed isolation of the major (50 mg) and minor (8 mg) diastereomers as a white solid and a colourless oil respectively.

Major Diastereomer

Melting point 97 - 103 °C; ν\textsubscript{max} (KBr disk)/cm\textsuperscript{-1} 3386br s, 3031w, 2958s, 2468w, 1746s, 1454m, 1408m, 1243m, 1093m, 698m; δ\textsubscript{H} (400 MHz, CD\textsubscript{3}OD) 0.96 (3 H, t, J 7.2), 1.34 - 1.70 (4 H, m), 3.44 (1 H, app q, J 5.4), 3.67 (1 H, dd, J 5.4, 3.7), 4.05 (1 H, ddd, J 8.7, 5.9, 3.7), 4.24 (1 H, app t, J 8.7), 4.42 (1 H, dd, J 8.7, 5.9), 4.53 (1 H, d, J 11.4), 4.59 (1 H, d, J 11.4), 7.26 - 7.40 (5 H, m); δ\textsubscript{C} (100 MHz, CD\textsubscript{3}OD) 13.6, 18.1, 32.2, 54.1, 66.4, 72.2, 72.6, 80.1, 127.8, 128.2, 128.4, 138.7, 161.6; HRMS (ESI) C\textsubscript{15}H\textsubscript{21}NNaO\textsubscript{4} requires 302.1363, found 302.1363 (–0.16 ppm).
Major Diastereomer ν\textsubscript{max} (thin film)/cm\textsuperscript{-1} 3384br s, 3031w, 2958s, 2872s, 1743s, 1496s, 1454s, 1409s, 1243s, 1093s, 943w, 736s; δ\textsubscript{H} (400 MHz, CD\textsubscript{3}OD) 0.97 (3 H, t, J 7.3), 1.35 - 1.51 (2 H, m), 1.59 - 1.73 (2 H, m), 3.49 (1 H, app td, J 6.7, 2.6), 3.64 (1 H, dd, J 5.3, 2.6), 4.00 (1 H, app dt, J 8.7, 5.3), 4.38 (1 H, app t, J 8.8), 4.48 (1 H, dd, J 8.8, 5.8), 4.51 (1 H, d, J 11.4), 4.63 (1 H, d, J 11.4), 7.25 - 7.41 (5 H, m); δ\textsubscript{C} (100 MHz, CDCl\textsubscript{3}) 14.6, 20.0, 32.9, 55.9, 68.4, 73.0, 73.5, 80.8, 128.8, 129.2, 129.4, 139.9, 162.5; HRMS (ESI) C\textsubscript{15}H\textsubscript{21}NNaO\textsubscript{4} requires 302.1363, found 302.1363 (–0.3ppm).

(±) (\textsuperscript{R})-4-((1'\textsuperscript{R},2'S)-1',2'-dihydroxypentyl)oxazolidin-2-one, \textit{anti}-10a

\[
\begin{align*}
\text{O} & \quad \text{O} \\
\text{N} & \quad \text{OH} \\
\text{HO} & \quad \text{HO}\n\end{align*}
\]

Carbamate \textit{anti}-9a (20 mg, 66 µmol) was subjected to general procedure H to afford the title compound as a colourless oil (12 mg, 63 µmol, 97%).

ν\textsubscript{max} (thin film)/cm\textsuperscript{-1} 3375br s, 2921s, 1738s, 1416w, 1258w, 1041m; δ\textsubscript{H} (400 MHz, CD\textsubscript{3}OD) 0.97 (3 H, t, J 7.1), 1.28 - 1.47 (2 H, m), 1.50 - 1.71 (2 H, m), 3.39 - 3.48 (2 H, m), 4.13 (1 H, ddd, J 8.8, 5.8, 3.0), 4.41 (1 H, app t, J 8.8), 4.48 (1 H, dd, J 8.8, 5.8); δ\textsubscript{C} (100 MHz, CD\textsubscript{3}OD) 13.4, 18.7, 35.9, 54.4, 66.2, 72.3, 74.7, 161.7; HRMS (ESI) C\textsubscript{8}H\textsubscript{15}NNaO\textsubscript{4} requires 212.0893, found 212.0893 (0.04 ppm).
\((\pm)\, (R)-4-((4'R,5'S)-2',2'-dimethyl-5'-propyl-1,3-dioxolan-4-yl)oxazolidin-2-one,\)  
\(anti-11a\)

Diol \(anti-10\) (12 mg, 63 \(\mu\)mol) was subjected to general procedure \(G\). Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as a white solid (14 mg, 61 \(\mu\)mol, 97%).

**Melting point** 106 - 107 °C; \(\nu_{\text{max}}\) (KBr disk)/cm\(^{-1}\) 3277br s, 2935s, 1749s, 1371m, 1242s, 1110w, 1066w; \(\delta_H\) (400 MHz, CDCl\(_3\)) 0.98 (3 H, t, \(J\) 7.2, C(8')H\(_3\)), 1.34 (3 H, s, CH\(_3\)), 1.43 (2 H, m, C(7')H\(_2\)), 1.41 (3 H, s, CH\(_3\)), 1.54 - 1.73 (2 H, m, C(6')H\(_2\)), 3.86 - 3.93 (1 H, m, C(4)H), 4.01 (1 H, dd, \(J\) 7.5, 5.8, C(4')H), 4.17 - 4.24 (1 H, m, C(5')H), 4.41 (1 H, dd, \(J\) 8.9, 5.8, C(5)H), 4.46 (1 H, dd, \(J\) 8.9, 8.4, C(5)H), 6.08 (1 H, br s, NH); \(\delta_C\) (100 MHz, CD\(_3\)OD) 14.2 (C(8')), 21.0 (C(7')), 25.3 (CH\(_3\)), 27.6 (CH\(_3\)), 32.8 (C(6')), 53.5 (C(4)), 68.3 (C(5)), 77.8 (C(5')), 80.0 (C(4')), 109.4 (C(2')CH\(_3\)), 162.6 (NC(O)O); **HRMS (ESI)** \(C_{11}H_{19}NNaO_4\) requires 252.1206, found 252.1207 (0.1 ppm).

\((\pm)\, (R)-4-((1'R,2'R)-1',2'-dihydroxypentyl)oxazolidin-2-one, syn-10\)

Carbamate \(syn-9a\) (10 mg, 33 \(\mu\)mol) was subjected to general procedure \(F\) to afford the title compound as a yellow oil (5 mg, 26 \(\mu\)mol, 77%).

\(\nu_{\text{max}}\) (thin film)/cm\(^{-1}\) 3375br s, 2921s, 1738s, 1416w, 1258m, 1041m; \(\delta_H\) (400 MHz, ...
\( \text{CD}_3\text{OD} \) 0.98 (3 H, t, \( J \) 7.3), 1.34 - 1.64 (4 H, m), 3.46 (1 H, dd, \( J \) 5.7, 2.2), 3.56 (1 H, ddd, \( J \) 7.9, 5.0, 2.2), 4.01 (1 H, app dt, \( J \) 8.8, 5.7), 4.46 (1 H, app t, \( J \) 8.8), 4.52 (1 H, dd, \( J \) 8.8, 6.0); \( \delta \text{C} \) (100 MHz, \( \text{CD}_3\text{OD} \)) 14.4, 20.1, 36.8, 56.0, 68.7, 72.2, 75.2, 162.6; HRMS (ESI) \( \text{C}_8\text{H}_{15}\text{NNaO}_4 \) requires 212.0893, found 212.0894 (–0.3 ppm).

\((\pm) \text{ (R)-4-((4'}^R,5'}^R)-2',2'-\text{dimethyl-5'-propyl-1,3-dioxolan-4-yl)oxazolidin-2-one, syn-11}

Diol syn-10 (5 mg, 30 \( \mu \)mol) was subjected to general procedure G. Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as a white solid (4 mg, 20 \( \mu \)mol, 66%).

\( \nu_{\text{max}} \) (thin film)/cm\(^{-1}\) 3278br s, 2935s, 1752s, 1371w, 1242m, 1110w, 1066m; \( \delta \text{H} \) (700 MHz, \( \text{C}_6\text{D}_6 \)) 1.09 (3 H, t, \( J \) 6.0, C(8')H\(_3\)), 1.30 (3 H, s, CH\(_3\)), 1.37 (3 H, s, CH\(_3\)), 1.40 - 1.54 (2 H, m, C(7')H\(_2\)), 1.54 - 1.64 (1 H, m, C(6')H), 1.65 - 1.79 (1 H, m, C(6')H), 3.24 (1 H, app q, \( J \) 7.5, C(4')H), 3.41 (1 H, app t, \( J \) 7.5, C(4')H), 3.68 (1 H, m, C(5')H), 3.91 (1 H, app t, \( J \) 7.5, C(5')H), 4.00 - 4.12 (1 H, m, C(5')H), 7.47 (1 H, br s, NH); \( \delta \text{C} \) (126 MHz, \( \text{C}_6\text{D}_6 \)) 14.5 (C(8')), 20.2 (C(7')), 27.3 (CH\(_3\)), 27.6 (CH\(_3\)), 36.6 (C(6')), 55.2 (C(4)), 68.5 (C(5)), 79.2 (C(5')), 83.1 (C(4')), 111.2 (C(2')CH\(_3\)), 162.3 (NC(O)O); HRMS (ESI) \( \text{C}_{11}\text{H}_{19}\text{NNaO}_4 \) requires 252.1212, found 252.1211 (–1.7 ppm).
1'-((Hex-1-yn-3-yloxy)methyl)-4'-methoxybenzene, 5b

![Chemical Structure](image)

Alcohol 27 (654 mg, 6.67 mmol) was subjected to general procedure A. Flash column chromatography on silica gel (97:3 petrol:ethyl acetate) afforded the title compound as a colourless oil (930 mg, 4.26 mmol, 64%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3290br s, 2959s, 2871s, 1612s, 1586w, 1514s, 1464m, 1388m, 1302s, 1248m, 1173s, 1113s, 1077s, 1036s, 823w, 759s, 637m; $\delta_H$ (400 MHz, CDCl$_3$)

0.92 (3 H, t, $J$ 7.5), 1.43 - 1.56 (2 H, m), 1.64 - 1.84 (2 H, m), 2.47 (1 H, d, $J$ 1.6), 3.82 (3 H, s), 4.07 (1 H, td, $J$ 6.6, 1.6), 4.46 (1 H, d, $J$ 11.4), 4.75 (1 H, d, $J$ 11.4), 6.89 (2 H, d, $J$ 8.6), 7.30 (2 H, d, $J$ 8.6); $\delta_C$ (100 MHz, CDCl$_3$) 13.7, 18.5, 37.7, 55.3, 67.8, 70.1, 73.6, 83.1, 113.8, 129.6, 130.0, 159.2; HRMS (ESI) $C_{14}H_{18}O_2$ requires 241.1204, found 241.1203 (–1.7 ppm).

4-(4'-Benzyloxy)hept-2-yn-1-ol, 6b

![Chemical Structure](image)

Alkyne 5b (500 mg, 2.29 mmol) was subjected to general procedure B. Flash column chromatography on silica gel (89:11 petrol:ethyl acetate) afforded the title compound as a colourless oil (472 mg, 1.90 mmol, 83%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3417br m, 2931s, 1612m, 1514s, 1464m, 1248m, 1174w, 1034m; $\delta_H$ (400 MHz, CDCl$_3$) 0.91 (3 H, t, $J$ 7.3), 1.40 - 1.55 (2 H, m), 1.62 - 1.82 (2 H, m), 2.07 (1 H, br s), 3.81 (3 H, s), 4.10 (1 H, t, $J$ 6.6), 4.33 (2 H, s), 4.44 (1 H, d, $J$ 11.5), 4.71 (1 H, d, $J$ 11.5), 6.88 (2 H, d, $J$ 8.6), 7.29 (2 H, d, $J$ 8.5); $\delta_C$ (100 MHz,
(Z)-4-(4'-Methoxybenzyl oxy)hept-2-en-1-ol, 7b

Alcohol 6b (160 mg, 645 µmol) was subjected to general procedure C. Flash column chromatography on silica gel (9:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (127 mg, 496 µmol, 77%).

ν<sub>max</sub> (thin film)/cm<sup>-1</sup> 3385br s, 2958s, 1612m, 1513s, 1464m, 1302w, 1249s, 1173w, 1035s, 821w; δ<sub>H</sub> (400 MHz, CDCl<sub>3</sub>) 0.88 (3 H, t, J 7.3), 1.22 - 1.48 (3 H, m), 1.58 - 1.71 (1 H, m), 2.47 (1 H, br s, OH), 3.78 (3 H, s), 4.02 - 4.11 (2 H, m), 4.17 (1 H, dd, J 12.9, 6.6), 4.28 (1 H, d, J 11.4), 4.50 (1 H, d, J 11.4), 5.45 (1 H, dd, J 11.5, 9.3), 5.77 (1 H, dt, J 11.5, 6.6), 6.87 (2 H, d, J 8.5), 7.25 (2 H, d, J 8.6); δ<sub>C</sub> (100 MHz, CDCl<sub>3</sub>) 14.0, 18.6, 37.6, 55.2, 58.6, 69.7, 73.7, 113.7, 129.3, 130.6, 131.7, 133.0, 159.1;

HRMS (ESI) C<sub>15</sub>H<sub>22</sub>O<sub>3</sub> requires 250.1567, found 279.1569 (0.8 ppm).

(Z)-4-(4'-Methoxybenzyl oxy)hept-2-enyl mesitoyloxycarbamate, 8b

Alcohol 7b (120 mg, 468 µmol) was subjected to general procedure D. Flash column chromatography on silica gel (9:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (138 mg, 303 µmol, 65%).
\( \nu_{\text{max}} \text{ (thin film)/cm}^{-1} \) 3233 br m, 2950s, 1752s, 1611s, 1513s, 1465s, 1366m, 1232s, 1158m, 1093s, 878m, 851m; \( \delta_{\text{H}} (400 \text{ MHz, CDCl}_3) \) 0.83 - 0.95 (3 H, m), 1.21 - 1.50 (3 H, m), 1.56 - 1.73 (1 H, m), 2.31 (3 H, s), 2.38 (6 H, s), 3.80 (3 H, s), 4.09 - 4.17 (1 H, m), 4.30 (1 H, d, \( J = 11.6 \)), 4.52 (1 H, d, \( J = 11.6 \)), 4.68 - 4.75 (1 H, m), 4.78 - 4.85 (1 H, m), 5.63 (1 H, dd, \( J = 11.4, 9.6 \)), 5.78 (1 H, dt, \( J = 11.4, 6.8 \)), 6.84 - 6.93 (4 H, m), 7.26 (2 H, d, \( J = 8.8 \)), 8.42 (1 H, br s); \( \delta_{\text{C}} (100 \text{ MHz, CDCl}_3) \) 14.0, 18.5, 19.9, 21.2, 37.6, 55.2, 62.4, 69.9, 73.5, 113.8, 125.4, 126.5, 128.7, 129.3, 130.5, 136.7, 136.7, 140.9, 156.4, 159.1, 169.0; \textbf{HRMS (ESI)} C_{26}H_{33}NNaO_6 requires 478.2200, found 478.2194 (1.2 ppm).
Diastereomeric mixture of (±) \((R)-4-((1'R,2'S)-(2'-(4''-methoxybenzyl)-1'\text{-hydroxypentyl)}oxazolidin-2-one\) (major isomer) with (±) \((R)-4-((1'R,2'R)-(2'-(4''-methoxybenzyl)-1'\text{-hydroxypentyl)}oxazolidin-2-one\) (minor isomer), \textit{anti-} and \textit{syn-}

Benzoyloxy carbamate \textbf{8b} (30 mg, 66 µmol) was subjected to general procedure G. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 50 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 100 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated \textit{in vacuo} to deliver the title compound as a colourless oil and a mixture of diastereomers (3:1, 16 mg, 52 µmol, 79%). The NMR data below are given for the major isomer.

\(\nu_{\text{max}}\)\hspace{1cm} (thin film)/cm\(^{-1}\) 3396br s, 2959s, 1747s, 1612w, 1514m, 1465s, 1249s, 1175m, 1032s; \(\delta_H\) \(\textbf{(400 MHz, CD}_3\text{OD)}\) 0.95 (3 H, s, \(J 7.3\)), 1.21 - 1.76 (4 H, m), 3.42 (1 H, app q, \(J 5.6\)), 3.60 - 3.66 (1 H, m), 3.79 (3 H, s), 4.02 (1 H, ddd, \(J 8.7, 5.4, 4.0\)), 4.22 (1 H, app t, \(J 8.7\)), 4.32 - 4.43 (1 H, m), 4.45 (1 H, d, \(J 11.4\)), 4.51 (1 H, d, \(J 11.4\)), 6.91 (2 H, d, \(J 8.5\)), 7.27 (2 H, d, \(J 8.5\)); \(\delta_C\) \(\textbf{(100 MHz, CD}_3\text{OD)}\) 13.7, 18.1, 32.5, 54.1, 54.7, 66.4, 71.8, 72.7, 79.6, 113.8, 129.9, 130.7, 159.9, 161.6; \textbf{HRMS (ESI)} \(\text{C}_{16}\text{H}_{23}\text{NNaO}_5\) requires 332.1468, found 332.1466 (0.8 ppm).
Commercially available 1-hexyn-3-ol (1.73 g, 17.6 mmol) was subjected to general procedure I. Flash column chromatography on silica gel (99:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (3.16 g, 14.8 mmol, 84%). Data were consistent with those previously reported in the literature.\textsuperscript{19}

\[ \delta_H \text{ (400 MHz, CDCl}_3\text{) 0.06 - 0.18 (6 H, s), 0.84 - 1.00 (12 H, s), 1.35 - 1.54 (2 H, m), 1.57 - 1.74 (2 H, m), 2.37 (1 H, d, } J 2.0) \text{, 4.27 - 4.41 (1 H, m); } \delta_C \text{ (100 MHz, CDCl}_3\text{) } -4.6, 13.8, 18.2, 18.4, 25.8, 40.7, 62.6, 71.8, 85.8. \]

\[ \text{4-}(\text{tert-Butyldimethylsilyloxy})\text{hept-2-yn-1-ol, 6c} \]

Alkyne 5c (800 mg, 3.76 mmol) was subjected to general procedure B. Flash column chromatography on silica gel (10:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (680 mg, 2.82 mmol, 75%).

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} \text{ 3333br m, 2958s, 2858s, 1463m, 1361m, 1255s, 1147s, 1073s, 1038s, 977s, 896m, 837s, 777s; } \delta_H \text{ (400 MHz, CDCl}_3\text{) 0.10 (3 H, s), 0.12 (3 H, s), 0.82 - 0.95 (12 H, m), 1.36 - 1.50 (2 H, m), 1.60 - 1.66 (2 H, m), 2.02 (1 H, br s), 4.28 (2 H, s), 4.38 (1 H, t, } J 6.4) \text{; } \delta_C \text{ (100 MHz, CDCl}_3\text{) } -5.1, -4.5, 13.7, 18.2, 18.5, 25.8, 40.7, 51.1, 62.7, 82.0, 87.5; \text{ HRMS (ESI) } C_{13}H_{26}O_2NaSi \text{ requires 265.1594, found 265.1595 (–0.13 ppm).} \]

\[ (Z)-4-\text{(tert-Butyldimethylsilyloxy})\text{hept-2-en-1-ol, 7c} \]
Alcohol 6c (500 mg, 2.05 mmol) was subjected to general procedure C. Flash column chromatography on silica gel (10:1 petrol:ethyl acetate) afforded the title compound as a yellow oil (374 mg, 1.52 mmol, 74%).

\[ \text{\(v_{\text{max}}\) (thin film)/cm\(^{-1}\)} \] 3333br m, 2958s, 2858s, 1463m, 1254s, 1038s, 901w, 836s, 775s; \(\delta_H\) (400 MHz, CDCl\(_3\)) 0.03 - 0.07 (6 H, m), 0.87 - 0.93 (12 H, m), 1.23 - 1.43 (2 H, m), 1.47 - 1.69 (2 H, m), 2.04 (1 H, br s), 4.12 (1 H, dd, \(J\) 13.3, 5.3), 4.22 - 4.29 (1 H, m), 4.37 (1 H, dt, \(J\) 7.4, 5.2), 5.46 - 5.59 (2 H, m); \(\delta_C\) (100 MHz, CDCl\(_3\)) –4.8, –4.4, 14.0, 18.2, 18.6, 25.8, 40.7, 59.0, 68.9, 127.7, 135.9; HRMS (ESI) C\(_{13}\)H\(_{28}\)NaO\(_2\)Si requires 267.1751, found 267.1752 (–0.56 ppm).

(Z)-4-(tert-Butyldimethylsilyloxy)hept-2-enyl mesityloxycarbamate, 8c

\[ \text{\(v_{\text{max}}\) (thin film)/cm\(^{-1}\)} \] 3278br m, 2957s, 1754s, 1612m, 1579w, 1462s, 1307w, 1227s, 1158s, 1092s, 901w, 837s, 776s; \(\delta_H\) (400 MHz, CDCl\(_3\)) 0.01 - 0.08 (6 H, m), 0.88 - 0.93 (12 H, m), 1.22 - 1.43 (3 H, m), 1.48 - 1.60 (1 H, m), 2.31 (3 H, s), 2.37 (6 H, s), 4.42 (1 H, td, \(J\) 7.6, 4.8), 4.76 (1 H, ddd, \(J\) 12.7, 7.0, 1.3), 4.84 (1 H, ddd, \(J\) 12.7, 7.0, 1.3), 5.52 (1 H, app dt, \(J\) 11.1, 7.0), 5.65 (1 H, dd, \(J\) 11.1, 7.0), 6.90 (2 H, s), 8.26 (1 H, br s); \(\delta_C\) (100 MHz, CDCl\(_3\)) –4.8, –4.4, 14.0, 18.2, 18.4 (C(6)), 19.9, 21.3, 25.8, 40.6, 62.6, 68.7, 121.4, 126.5, 128.7, 136.7, 139.5, 140.9, 156.4, 169.0; HRMS (ESI) C\(_{24}\)H\(_{39}\)NNaOSi requires 472.2490, found 472.2485 (1.08 ppm).
Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-2'-(tert-butyldimethylsilyloxy)-1'-hydroxypentyl)oxazolidin-2-one (major) with (±) (R)-4-((1'R,2'R)-2'-(tert-butyldimethylsilyloxy)-1'-hydroxypentyl)oxazolidin-2-one (minor), anti- and syn-9c

Benzoyloxy carbamate 8c (75 mg, 170 µmol) was subjected to general procedure G. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 100 mL of solution petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 200 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated in vacuo to deliver the title compound as a mixture of diastereomers (3:1, 39 mg, 130 µmol, 78%). Flash column chromatography on silica gel (3:1 to 1:1 petrol:ethyl acetate) allowed isolation of the major (24 mg) and minor (12 mg) diastereomers as white solids.

**Major Diastereomer; Melting point** 124 - 126 °C; $\nu_{\text{max}}$ (KBr disk)/cm$^{-1}$ 3300 br w, 2930s, 1741s, 1513s, 1249s, 1095s; δ$\text{H}$ (400 MHz, CD$_3$OD) 0.10 (6 H, m), 0.88 - 0.98 (12 H, m), 1.31 - 1.53 (3 H, m), 1.53 - 1.69 (1 H, m), 3.60 (1 H, dd, $J$ 4.9, 3.3), 3.72 (1 H, app q), 4.07 (1 H, ddd, $J$ 8.6, 6.1, 3.3), 4.35 (1 H, app t, $J$ 8.6), 4.50 (1 H, dd, $J$ 8.6, 5.8); δ$\text{C}$ (100 MHz, CD$_3$OD) –5.3, –5.3, 13.7, 17.9, 25.4, 35.9, 53.7, 66.3, 73.4, 74.0, 161.7; HRMS (ESI) C$_{14}$H$_{29}$NNaO$_4$Si requires 326.1758, found 326.1760 (–0.61 ppm).

**Minor Diastereomer; Melting point** 111 - 115 °C; $\nu_{\text{max}}$ (KBr disk)/cm$^{-1}$ 3385 br m, 2930m, 1742s, 1472m, 1254m, 1099m, 1028m, 965m, 936m, 835w, 776w; δ$\text{H}$ (400 MHz, CD$_3$OD) 0.11 - 0.16 (6 H, m), 0.95 (12 H, m), 1.30 - 1.50 (4 H, m), 3.57 (1 H, dd, $J$ 5.0, 3.0), 3.77 (1 H, td, $J$ 6.0, 3.0), 4.02 (1 H, dt, $J$ 8.5, 5.0), 4.45 (1 H, app t, $J$
8.5), 4.50 (1 H, dd, J 8.8, 5.0); δ_C (100 MHz, CD_3OD) −4.3, −4.1, 14.5, 19.0, 20.2, 26.4, 36.3, 55.2, 68.2, 74.5, 74.8, 162.4; HRMS (ESI) C_{14}H_{29}NNaO_{4}Si requires 326.1758, found 326.1757 (0.4 ppm).

3-tert-Butoxyhex-1-yne, 5d

Amberlyst H-15 acidic strong cation exchange resin was added to a solution of commercially available alcohol 1-hexyn-3-ol (1.00 g, 10.2 mmol) in hexane (5 mL). Isobutene was bubbled through the solution for 8 hours before the resultant mixture was concentrated in vacuo. Flash column chromatography on silica gel (petrol) afforded the title compound as a colourless oil (834 mg, 5.41 mmol, 53%). Data were consistent with those previously reported in the literature.\textsuperscript{21}

δ_H (400 MHz, CDCl_3) 0.92 (3 H, t, J 7.3), 1.25 (9 H, s), 1.36 - 1.54 (2 H, m), 1.59 - 1.67 (2 H, m), 2.34 (1 H, s), 4.09 (1 H, t, J 6.6); δ_C (100 MHz, CDCl_3) 13.8, 18.7, 28.2, 39.7, 61.4, 71.7, 74.7, 86.6.
4-(*tert*-Butoxy)hept-2-yn-1-ol, 6d

Alkyne 5d (100 mg, 649 µmol) was subjected to general procedure B. Flash column chromatography on silica gel (95:5 petrol:ethyl acetate) afforded the title compound as a yellow oil (88 mg, 487 µmol, 75%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3374br s, 2964s, 1462s, 1390s, 1366m, 1335m, 1255m, 1234s, 1194s, 1144s, 1100s, 1022m, 905m, 867w, 835w, 728w; $\delta_H$ (400 MHz, CDCl$_3$) 0.89 (3 H, t, $J$ 7.6), 1.22 (9 H, s), 1.31 - 1.50 (2 H, m), 1.53 - 1.64 (2 H, m), 2.34 - 2.64 (1 H, br s), 4.11 (1 H, tt, $J$ 6.6, 1.5), 4.24 (2 H, s); $\delta_C$ (100 MHz, CDCl$_3$) 13.8, 18.7, 28.1, 39.7, 51.0, 61.7, 74.7, 82.1, 87.9; HRMS (ESI) C$_{11}$H$_{20}$NaO$_2$ requires 207.1356, found 207.1346 (4.7 ppm).

(Z)-4-*tert*-Butoxyhept-2-en-1-ol, 7d

Alkyne 6d (330 mg, 1.79 mmol) was subjected to general procedure C. Flash column chromatography on silica gel (95:5 petrol:ethyl acetate) afforded the title compound as a colourless oil (230 mg, 1.24 mmol, 69%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3375br m, 2972s, 2873m, 1464m, 1389m, 1365s, 1194s, 1036s; $\delta_H$ (400 MHz, CDCl$_3$) 0.89 (3 H, t, $J$ 6.9), 1.18 (9 H, s), 1.22 - 1.43 (3 H, m), 1.45 - 1.57 (1 H, m), 2.54 (1 H, br s), 4.08 - 4.19 (2 H, m), 4.28 (1 H, dd, $J$ 14.4, 5.3), 5.46 -
5.59 (2 H, m);  δ_C (100 MHz, CDCl₃) 14.0, 18.9, 28.5, 39.7, 59.2, 68.5, 74.1, 127.1, 136.6; HRMS (ESI) C₁₁H₂₂NaO₂ requires 209.1512, found 209.1513 (–0.3 ppm).

(Z)-4-tert-Butoxyhept-2-enyl mesitoyloxycarbamate, 8d

Alcohol 7d (220 mg, 1.18 mmol) was subjected to general procedure D. Flash column chromatography on silica gel (98:2 petrol:ethyl acetate) afforded the title compound as a yellow oil (333 mg, 840 µmol, 71%).

ν_max (thin film)/cm⁻¹ 3261 br s, 2969 s, 1753 s, 1611 m, 1460 m, 1365 m, 1228 s, 1158 m, 1093 m, 850 w; δ_H (400 MHz, CDCl₃) 0.90 (3 H, t, J 7.1), 1.17 (9 H, s), 1.22 - 1.44 (3 H, m), 1.45 - 1.58 (1 H, m), 2.29 (3 H, s), 2.36 (6 H, s), 4.24 (1 H, dt, J 8.5, 7.0), 4.76 (1 H, dd, J 13.0, 8.1), 5.47 (1 H, ddd, J 11.1, 8.1, 6.1), 5.69 (1 H, dd, J 11.1, 8.5), 6.88 (2 H, s), 8.53 (1 H, br s); δ_C (100 MHz, CDCl₃) 14.0, 18.8, 19.9, 21.2, 28.7, 39.5, 62.7, 68.1, 74.0, 120.7, 126.6, 128.6, 136.6, 140.5, 140.8, 156.5, 168.9; HRMS (ESI) C₂₂H₃₃NNaO₅ requires 414.2251, found 414.2257 (–1.4 ppm).

Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-(2'(tert-butoxy)-1'-hydroxypentyl)oxazolidin-2-one (major isomer) with (±) (R)-4-((1'R,2'R)-(2'(tert-butoxy)-1'-hydroxypentyl)oxazolidin-2-one (minor isomer), anti- and syn-9c

Benzoyloxycarbamate 8d (44 mg, 110 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic
acid byproduct was removed by washing with 50 mL of solution petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 100 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated in vacuo to deliver the title compound as a colourless oil and a mixture of diastereomers (3:1, 25 mg, 100 µmol, 93%). The NMR data below are given for the major isomer.

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} \] 3386br s, 3031w, 2958s, 2468w, 1746s, 1454m, 1408m, 1243m, 1093m, 698m; \[ \delta_{\text{H}} \text{ (400 MHz, CD}_{3}\text{OD)} \] 0.90 - 1.00 (3 H, t, J 7.3), 1.18 - 1.25 (9 H, m), 1.29 - 1.64 (4 H, m), 3.54 - 3.66 (2 H, m), 4.02 (1 H, ddd, J 9.6, 5.8, 4.0), 4.39 (1 H, t, J 8.8), 4.51 (1 H, dd, J 8.8, 5.8); \[ \delta_{\text{C}} \text{ (100 MHz, CD}_{3}\text{OD)} \] 13.8, 18.4, 28.1, 34.9, 53.8, 67.1, 73.1, 73.7, 74.2, 161.7; HRMS (ESI) \text{C}_{12}\text{H}_{23}\text{NNaO}_{4} \text{ requires 268.1519, found 268.1518 (0.3 ppm).}

![Chemical Diagram]

1'-Methoxy-4'-(prop-2-ynyloxy)methyl)benzene, 28

Commerially available propargylic alcohol (1.00 g, 17.8 mmol) was subjected to general procedure A. Flash column chromatography on silica gel (99:1 petrol:ethyl
acetate) afforded the title compound as a colourless oil (3.05 g, 17.3 mmol, 97%).

Data were consistent with those previously reported in the literature.\textsuperscript{20}

$\delta_H$ (400 MHz, CDCl$_3$) 2.48 (1 H, t, $J$ 2.3), 3.82 (3 H, s), 4.16 (2 H, d, $J$ 2.3), 4.56 (2 H, s), 6.90 (2 H, d, $J$ 8.6), 7.30 (2 H, d, $J$ 8.6); $\delta_C$ (100 MHz, CDCl$_3$) 55.3, 56.7, 71.2, 74.5, 79.8, 113.9, 129.3, 129.8, 159.5.

1-(4'-methoxybenzyloxy)hept-2-yn-4-ol, 29

\[
\text{Alkyne } 28 \text{ (1000 mg, 5.64 mmol) was subjected to general procedure K. Flash column chromatography on silica gel (1:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (800 mg, 3.22 mmol, 57%).}
\]

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3416br s, 2955s, 2860s, 1612s, 1586m, 1514s, 1465m, 1353m, 1302m, 1250s, 1175m, 1141s, 1036s; $\delta_H$ (400 MHz, CDCl$_3$) 0.96 (3 H, t, $J$ 7.3), 1.42 - 1.58 (2 H, m), 1.61 - 1.79 (2 H, m), 2.31 (1 H, br s), 3.80 (3 H, s), 4.17 (2 H, d, $J$ 1.8), 4.39 - 4.47 (1 H, m), 4.52 (2 H, s), 6.88 (2 H, d, $J$ 8.3), 7.28 (2 H, d, $J$ 8.4); $\delta_C$ (100 MHz, CDCl$_3$) 13.7, 18.4, 39.8, 55.3, 57.0, 62.2, 71.2, 80.6, 87.7, 113.8, 129.4, 129.8, 159.4; HRMS (ESI) C$_{15}$H$_{20}$NaO$_3$ requires 271.1305, found 271.1312 (2.8 ppm).

(Z)-1-(4'-Methoxybenzyloxy)hept-2-en-4-ol, 30

\[
\text{Alcohol } 29 \text{ (630 mg, 2.54 mmol) was subjected to general procedure C. Flash column chromatography on silica gel (96:4 petrol:ethyl acetate) afforded the title compound as a colourless oil (470 mg, 1.88 mmol, 74%).}
\]
(Z)-1-(4'-Methoxybenzyloxy)hept-2-en-4-yl acetate, 31

![Chemical Structure](image)

Alcohol 30 (50 mg, 200 µmol) was subjected to general procedure L to deliver the title compound as a colourless oil (49 mg, 170 µmol, 84%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3421br m, 2930s, 2857s, 1733m, 1612m, 1513s, 1464m, 1302w, 1248s, 1174m, 1036m, 821m; $\delta_{H}$ (400 MHz, CDCl$_3$) 0.90 (3 H, t, $J$ 7.1), 1.21 - 1.46 (3 H, m), 1.48 - 1.63 (1 H, m), 2.47 (1 H, br s), 3.79 (3 H, s), 3.99 (1 H, dd, $J$ 12.1, 5.6), 4.11 (1 H, dd, $J$ 12.1, 6.8), 4.34 (1 H, ddd, $J$ 8.0, 6.8, 6.4), 4.46 (1 H, d, $J$ 12.1), 4.43 (1 H, d, $J$ 12.1), 5.57 (1 H, dd, $J$ 10.4, 8.0), 5.66 (1 H, ddd, $J$ 10.4, 6.8, 5.6), 6.88 (2 H, d, $J$ 8.6), 7.26 (2 H, d, $J$ 8.6); $\delta_{C}$ (100 MHz, CDCl$_3$) 14.0, 18.5, 39.3, 55.2, 65.5, 67.4, 72.1, 113.8, 127.4, 129.5, 130.0, 136.6, 159.3; HRMS (ESI) C$_{15}$H$_{22}$NaO$_3$ requires 273.1461, found 273.1462 (–0.2 ppm).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 2956s, 2860s, 1721s, 1612s, 1585w, 1513s, 1464w, 1379w, 1263s, 1170s, 1080s, 1036s, 956m, 851m, 821m, 757m; $\delta_{H}$ (400 MHz, CDCl$_3$) 0.90 (3 H, t, $J$ 7.3), 1.21 - 1.40 (2 H, m), 1.42 - 1.56 (1 H, m), 1.57 - 1.73 (1 H, m), 2.02 (3 H, s), 3.80 (3 H, s), 4.17 (2 H, d, $J$ 6.3), 4.41 (1 H, d, $J$ 11.4), 4.47 (1 H, d, $J$ 11.4), 5.42 - 5.52 (2 H, m), 5.72 (1 H, dt, $J$ 9.9, 6.3), 6.88 (2 H, d, $J$ 8.6), 7.27 (2 H, d, $J$ 8.6); $\delta_{C}$ (100 MHz, CDCl$_3$) 13.8, 18.3, 21.2, 36.7, 55.2, 65.8, 70.2, 72.1, 113.8, 129.4, 130.1, 130.3, 130.8, 159.2, 170.3; HRMS (ESI) C$_{17}$H$_{24}$NaO$_4$ requires 315.1567, found 315.1564 (0.7 ppm).
(Z)-1-Hydroxyhept-2-en-4-yl acetate, 7e

```
                   OH
                 O
            O
```

Ester 31 (236 mg, 808 µmol) was subjected to general procedure H. Flash column chromatography on silica gel (5:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (110 mg, 640 µmol, 79%).

\( \nu_{\text{max}} \) (thin film)/cm\(^{-1}\) 3422 br s, 2928 s, 1736 s, 1238 s, 1025 m; \( \delta_H \) (400 MHz, CDCl\(_3\))

0.91 (3 H, t, \( J = 7.3 \)), 1.20 - 1.40 (2 H, m), 1.42 - 1.55 (1 H, m), 1.59 - 1.72 (1 H, m), 2.02 (3 H, s), 2.69 (1 H, br s), 4.03 (1 H, dd, \( J = 13.1, 5.8 \)), 4.41 (1 H, dd, \( J = 13.1, 8.1 \)), 5.35 (1 H, app t, \( J = 10.0 \)), 5.49 (1 H, dt, \( J = 10.0, 7.3 \)), 5.80 (1 H, ddd, \( J = 10.0, 8.1, 5.8 \)); \( \delta_C \)
(100 MHz, CDCl\(_3\)) 13.8, 18.3, 21.3, 36.3, 58.4, 70.4, 130.1, 132.2, 171.3; HRMS (ESI) \( C_{9}H_{16}NaO_{3} \) requires 195.0992, found 195.0992 (–2.3 ppm).

(Z)-1-(Mesitoyloxycarbamoyloxy)hept-2-en-4-yl acetate, 8e

```
                O
             N\( \text{O} \)
        O
```

Alcohol 7e (95 mg, 550 µmol) was subjected to general procedure R. Flash column chromatography on silica gel (94:6 petrol:ethyl acetate) afforded the title compound as a colourless oil (103 mg, 280 µmol, 50%).

\( \nu_{\text{max}} \) (thin film)/cm\(^{-1}\) 3264 br s, 2961 s, 1737 s, 1611 w, 1458 m, 1372 m, 1229 s, 1158 m, 1092 m, 1020 w, 851 w; \( \delta_H \) (400 MHz, CDCl\(_3\))

0.92 (3 H, t, \( J = 7.3 \)), 1.23 - 1.41 (2 H, m), 1.44 - 1.58 (1 H, m), 1.61 - 1.72 (1 H, m), 2.03 (3 H, s), 2.29 (3 H, s), 2.35 (6 H, s), 4.85 (1 H, dd, \( J = 13.0, 6.5 \)), 4.97 (1 H, dd, \( J = 13.0, 6.5 \)), 5.45 - 5.58 (2 H, m), 5.70 (1 H, dt, \( J = 10.4, 6.5 \)), 6.87 (2 H, s), 8.54 (1 H, s); \( \delta_C \) (100 MHz, CDCl\(_3\)) 13.8, 18.2, 19.8,
21.1, 21.2, 36.5, 62.5, 70.0, 126.4, 126.6, 128.6, 132.8, 136.6, 140.8, 156.4, 168.9, 170.4; **HRMS (ESI)** C_{20}H_{27}NNaO_{6} requires 400.1731, found 400.1723 (2.0 ppm).

Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-2'-acetate-1'-hydroxypentyl oxazolidin-2-one (major isomer) with (±) (R)-4-((1'R,2'R)-2'-acetate-1'-hydroxypentyl oxazolidin-2-one (minor isomer), *anti-* and *syn-* 9e.

![Diagram](image)

Benzoyloxycarbamate 8e (50 mg, 130 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 75 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 150 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated *in vacuo* to deliver the title compound as a mixture of diastereomers (1.5:1, 20 mg). No yield is given due to impurities in the NMR spectra. The NMR data below are given for the major isomer.

\[ \nu_{\text{max}} \,(\text{thin film})/\text{cm}^{-1} \, 3382\text{br s}, 2960\text{s}, 1740\text{s}, 1375\text{m}, 1241\text{w} \]

\[ \delta_{\text{H}} (400 \text{ MHz, } CD_{3}\text{OD}) \]

1.00 (3 H, t, \( J \, 7.3 \)), 1.36 - 1.62 (2 H, m), 1.63 - 1.87 (2 H, m), 2.22 - 2.25 (3 H, s), 3.82 (1 H, t, \( J \, 4.0 \)), 4.04 (1 H, ddd, \( J \, 9.0, 5.6, 4.0 \)), 4.34 (1 H, app t, \( J \, 9.0 \)), 4.47 (1 H, dd, \( J \, 9.0, 5.6 \)), 5.20 (1 H, td, \( J \, 8.3, 4.3 \)); \[ \delta_{\text{C}} (63 \text{ MHz, } CD_{3}\text{OD}) \]

13.1, 18.7, 19.9, 32.6, 54.4, 66.1, 72.7, 74.1, 161.2, 171.7; **HRMS (ESI)** C_{10}H_{17}NNaO_{5} requires 254.0999, found 254.1000 (–0.2 ppm).
Alcohol 30 (200 mg, 797 µmol) was subjected to general procedure M with mesitoyl chloride. Flash column chromatography on silica gel (99:1 petrol:ethyl acetate) afforded the title compound as a yellow oil (316 mg). No yield is given due to impurities in the NMR spectra.

\[ \text{\( \nu_{\max} \) (thin film)/cm}^{-1} \] 2956s, 2860s, 1721s, 1612s, 1585w, 1513s, 1464w, 1379w, 1263s, 1170s, 1080s, 1036s, 956m, 851m, 821m, 757m; \[ \delta_{\text{H}} \] (400 MHz, CDCl\(_3\)) 1.02 (3 H, t, \( J \) 7.3), 1.41 - 1.54 (2 H, m), 1.63 - 1.69 (2 H, m), 2.34 (3 H, s), 2.48 (6 H, s), 3.85 (3 H, s), 4.40 (2 H, d, \( J \) 6.3), 4.58 (1 H, d, \( J \) 11.4), 4.61 (1 H, d, \( J \) 11.4), 5.66 (1 H, dd, \( J \) 10.9, 10.1), 5.81 - 5.97 (2 H, m), 6.94 (2 H, s), 6.96 (2 H, d, \( J \) 8.8), 7.38 (2 H, d, \( J \) 8.6); \[ \delta_{\text{C}} \] (100 MHz, CDCl\(_3\)) 13.9, 18.5, 19.7, 20.4, 36.8, 55.2, 66.0, 70.9, 72.2, 113.9, 128.8, 129.6, 130.6, 130.8, 134.8, 136.1, 139.5, 139.9, 159.3, 169.8; HRMS (ESI) \( C_{25}H_{32}NaO_4 \) requires 419.2193, found 419.2189 (0.8 ppm).
(Z)-1-Hydroxyhept-2-en-4-yl mesitoate, 7f

Ester 32 (104 mg, 263 µmol) was subjected to general procedure H. Flash column chromatography on silica gel (9:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (55 mg, 196 µmol, 75%).

ν<sub>max</sub> (thin film)/cm<sup>-1</sup> 3463br s, 2924s, 1720s, 1612m, 1456m, 1266s, 1170m, 1079s, 852w; δ<sub>H</sub> (400 MHz, CDCl<sub>3</sub>) 0.96 (3 H, t, J 7.3), 1.33 - 1.49 (2 H, m), 1.53 - 1.65 (1 H, m), 1.72 - 1.87 (1 H, m), 2.28 (9 H, m), 2.25 - 2.85 (1 H, br s), 4.13 (1 H, dd, J 12.9, 6.1), 4.56 (1 H, dd, J 12.9, 8.3), 5.49 (1 H, dd, J 10.6, 10.0), 5.82 (1 H, ddd, J 10.0, 7.3, 6.8), 5.92 (1 H, ddd, J 10.6, 8.3, 6.1), 6.85 (2 H, s); δ<sub>C</sub> (100 MHz, CDCl<sub>3</sub>) 13.8, 18.4, 19.6, 21.1, 36.3, 58.5, 71.0, 128.4, 130.0, 130.9, 132.6, 134.8, 139.3, 170.5;

HRMS (ESI) C<sub>17</sub>H<sub>24</sub>NaO<sub>3</sub> requires 299.1618, found 299.1613 (1.7 ppm).

(Z)-1-(Mesitoyloxycarbamoyloxy)hept-2-en-4-yl mesitoate, 8f

Alcohol 7f (41 mg, 150 µmol) was subjected to general procedure R. Flash column chromatography on silica gel (97:3 petrol:ethyl acetate) afforded the title compound as a colourless oil (30 mg, 64 µmol, 43%).

ν<sub>max</sub> (thin film)/cm<sup>-1</sup> 3276br s, 2961s, 1722s, 1611m, 1456m, 1261s, 1168m, 1078s, 851w; δ<sub>H</sub> (400 MHz, CDCl<sub>3</sub>) 0.96 (3 H, t, J 7.3), 1.33 - 1.52 (2 H, m), 1.55 - 1.68 (1 H, m), 1.76 - 1.87 (1 H, m), 2.28 (9 H, s), 2.31 (3 H, s), 2.38 (6 H, s), 4.96 (1 H, dd, J 10.6, 6.1), 5.35 - 5.82 (1 H, ddd, J 10.6, 8.3, 6.1), 5.89 (1 H, ddd, J 10.0, 7.3, 6.8), 6.85 (2 H, s); δ<sub>C</sub> (100 MHz, CDCl<sub>3</sub>) 13.8, 18.4, 19.6, 21.1, 36.3, 58.5, 71.0, 128.4, 130.0, 130.9, 132.6, 134.8, 139.3, 170.5;
Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-2'-mesitoate-1'-hydroxypentyl oxazolidin-2-one (major isomer) with (±) (R)-4-((1'R,2'R)-2'-mesitoate-1'-hydroxypentyl oxazolidin-2-one (minor isomer), anti- and syn-9f

Benzoyloxy carbamate 8f (25 mg, 52 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 50 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 100 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated in vacuo to deliver the title compound as a yellow oil and a mixture of diastereomers (1.5:1, 14 mg, 42 µmol, 80%). The NMR data below are given for the major isomer.

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3374br s, 2961s, 1727s, 1611w, 1427s, 1261m, 1170s, 1077w, 800w; $\delta_H$ (400 MHz, CD$_3$OD) 1.00 (3 H, t, $J$ 6.5), 1.37 - 1.62 (2 H, m), 1.65 - 1.89 (2 H, m), 2.26 - 2.29 (3 H, m), 2.30 (6 H, s), 3.82 (1 H, app t, $J$ 4.5), 4.04 (1 H, ddd, $J$ 9.1, 5.1, 4.5), 4.35 (1 H, app t, $J$ 9.1), 4.47 (1 H, dd, $J$ 9.1, 5.1), 5.20 (1 H, ddd, $J$ 8.3, 4.5, 4.3), 6.90 (2 H, s); $\delta_C$ (100 MHz, CD$_3$OD) 14.3, 19.9, 20.1, 21.2, 33.1, 54.9, 67.3, 73.3, 76.7, 129.5, 132.3, 136.0, 140.7, 162.5, 171.5; HRMS (ESI) C$_{18}$H$_{25}$NNaO$_5$ requires 504.2355, found 504.2356 (0.2 ppm).
requires 358.1625, found 358.1625 (0.0 ppm).

**tert-Butyldimethyl(prop-2-ynyloxy)silane, 33**

Commercially available propargylic alcohol (750 mg, 13.4 mmol) was subjected to general procedure I. Flash column chromatography on silica gel (98:2 petrol:ethyl acetate) afforded the title compound as a yellow oil (1.57 g, 9.24 mmol, 69%). Data were consistent with those previously reported in the literature.\(^{22}\)

\[
\begin{align*}
\delta_H (400 MHz, CDCl_3) & \quad 0.14 (6 H, s), 0.92 (9 H, s), 2.40 (1 H, t, J 2.4), 4.32 (2 H, d, J 2.4); \\
\delta_C (100 MHz, CDCl_3) & \quad -5.2, 18.3, 25.8, 51.5, 72.8, 82.4.
\end{align*}
\]

**1-(tert-butyldimethylsilyloxy)hept-2-yn-4-ol, 34**

Alkyne 714 (680 mg, 4.00 mmol) was subjected to general procedure K. Flash column chromatography on silica gel (98:2 petrol:ethyl acetate) afforded the title compound as
a colourless oil (668 mg, 2.76 mmol, 69%).

\( \nu_{\text{max}} \) (thin film)/cm\(^{-1}\) 3444br s, 2969s, 2235w, 1716s, 1471s, 1389s, 1256s, 1160s, 1078m, 837m, 777m; \( \delta \)\( _{H} \) (400 MHz, CDCl\(_3\)) 0.11 (6 H, s), 0.90 (9 H, s), 0.93 (3 H, t, J 7.6), 1.39 - 1.53 (2 H, m), 1.57 - 1.75 (2 H, m), 2.16 (1 H, br s), 4.33 (2 H, s), 4.39 (1 H, t, J 6.3); \( \delta \)\( _{C} \) (100 MHz, CDCl\(_3\)) –5.2, 13.7, 18.2, 18.4, 25.8, 39.7, 51.7, 62.2, 83.3, 85.9; HRMS (ESI) C\(_{13}\)H\(_{26}\)NaO\(_2\)Si requires 265.1602, found 265.1594 (–3.0 ppm).

\((Z)-1-(\text{tert-Butyldimethylsilyloxy})\)hept-2-en-4-ol, 35

Alcohol 34 (750 mg, 3.09 mmol) was subjected to general procedure C. Flash column chromatography on silica gel (97:3 petrol:ethyl acetate) afforded the title compound as a colourless oil (677 mg, 2.78 mmol, 90%).

\( \nu_{\text{max}} \) (thin film)/cm\(^{-1}\) 3376br s, 2958s, 1716s, 1464s, 1362w, 1255s, 1007s, 837s, 777s; \( \delta \)\( _{H} \) (400 MHz, CDCl\(_3\)) 0.09 (6 H, s), 0.94 (3 H, t, J 7.3), 0.91 (9 H, s), 1.25 - 1.50 (3 H, m), 1.50 - 1.66 (1 H, m), 1.95 (1 H, br s), 4.21 (1 H, dd, J 13.4, 5.6), 4.32 (1 H, dd, J 13.4, 6.1), 4.42 (1 H, td, J 6.8, 6.1), 5.46 - 5.55 (1 H, m), 5.64 (1 H, ddd, J 11.4, 6.1, 5.6); \( \delta \)\( _{C} \) (100 MHz, CDCl\(_3\)) –5.2, 14.0, 18.3, 18.6, 25.9, 39.4, 59.5, 67.6, 130.8, 134.3; HRMS (ESI) C\(_{18}\)H\(_{28}\)NaO\(_2\)Si requires 267.1751, found 267.1752 (0.1 ppm).

\((Z)-\text{tert-Butyl}(4\text{-methoxyhept-2-enyloxy})\)dimethylsilane, 36

NaH (60% by weight dispersion in mineral oil, 80 mg, 4.1 mmol) was added to a solution of alcohol 35 (100 mg, 410 µmol) in THF (4 mL) under argon. The
suspension was stirred at room temperature for 1 hour, before the addition of iodomethane (510 µL, 410 µmol). The reaction was heated at reflux for 1.5 hours and then quenched with a saturated aqueous solution of NH₄Cl. The organic phase was separated, and the aqueous phase was extracted with ethyl acetate (5 mL ×3). The combined organic layers were dried over MgSO₄, filtered, and the resultant mixture was concentrated in vacuo to afford the crude product. Flash column chromatography on silica gel (99:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (79 mg, 310 µmol, 75%).

ν max (thin film)/cm⁻¹ 3385br w, 2958s, 1723s, 1496m, 1470s, 1387m, 1255s, 1093s, 837m, 778m; δ H (400 MHz, CDCl₃) 0.08 (6 H, s), 0.83 - 0.96 (12 H, m), 1.22 - 1.45 (3 H, m), 1.50 - 1.59 (1 H, m), 3.26 (3 H, s), 3.85 - 3.95 (1 H, m), 4.20 (1 H, ddd, J 13.4, 5.8, 1.4), 4.30 (1 H, ddd, J 13.4, 6.5, 1.4), 5.29 (1 H, app dd, J 11.2, 9.3), 5.73 (1 H, ddd, J 11.2, 6.5, 5.8); δ C (100 MHz, CDCl₃) –5.2, 14.0, 18.3, 18.5, 25.9, 37.7, 56.0, 59.4, 76.4, 131.5, 132.8; HRMS (ESI) C₁₄H₃₀NaO₂Si requires 281.1913, found 281.1906 (0.4 ppm).

(Z)-4-Methoxyhept-2-en-1-ol, 7g

Alkene 36 (79 mg, 310 µmol) was subjected to general procedure N. Flash column chromatography on silica gel (4:1 petrol:ethyl acetate) afforded the title compound as a yellow oil (23 mg, 160 µmol, 52%).

ν max (thin film)/cm⁻¹ 3383br s, 3030m, 2959s, 1693w, 1496m, 1454s, 1383m, 1205m, 1067s, 976m, 736m; δ H (400 MHz, CDCl₃) 0.90 (3 H, t, J 7.3), 1.21 - 1.46 (3 H, m), 1.55 - 1.63 (1 H, m), 2.03 (1 H, br s), 3.26 (3 H, s), 3.85 - 3.97 (1 H, m), 4.16 (1 H,
dd, J 13.0, 6.3), 4.27 (1 H, dd, J 13.0, 6.8), 5.38 (1 H, dd, J 11.0), 5.80 (1 H, ddd, J 11.0, 6.8, 6.3); δ_c (100 MHz, CDCl_3) 14.0, 18.4, 37.4, 56.0, 58.8, 76.5, 131.7, 132.9; HRMS (ESI) C_8H_{15}O_2 requires 143.1078, found 143.1077 (0.1 ppm).

(Z)-4-Methoxyhept-2-enyl mesitoyloxycarbamate, 8g

\[
\begin{align*}
\text{Alcohol 7g (28 mg, 230 µmol) was subjected to general procedure D. Flash column} \\
\text{chromatography on silica gel (96:4 petrol:ethyl acetate) afforded the title compound as} \\
a \text{a colourless oil (40 mg, 120 µmol, 50%).}
\end{align*}
\]

ν_max (thin film)/cm⁻¹ 3252br m, 2960s, 1754s, 1611m, 1461m, 1228s, 1158m, 1094s, 851w; δ_H (400 MHz, CDCl₃) 0.94 (3 H, t, J 7.3), 1.23 - 1.48 (3 H, m), 1.56 - 1.68 (1 H, m), 2.30 (3 H, s), 2.37 (6 H, s), 3.27 (3 H, s), 3.97 (1 H, dt, J 8.8, 6.4), 4.78 (1 H, dd, J 12.6, 6.5), 4.89 (1 H, dd, J 12.6, 7.0), 5.54 (1 H, dd, J 10.8, 8.8), 5.77 (1 H, ddd, J 10.8, 7.0, 6.5), 6.89 (2 H, s), 8.44 (1 H, br s); δ_c (100 MHz, CDCl₃) 14.0, 18.4, 19.9, 21.2, 37.4, 56.3, 62.3, 76.4, 125.6, 126.5, 128.7, 136.3, 136.6, 140.9, 156.4, 169.0; HRMS (ESI) C_{19}H_{27}NNaO_5 requires 372.1781, found 372.1778 (0.8 ppm).
Diastereomeric mixture of (±) \((R)-4-((1' R, 2'S)-2'-\text{methoxy}-1'\text{-hydroxypentyl oxazolidin-2-one (major isomer) with (±) \((R)-4-((1'R, 2'R)-2'-\text{methoxy}-1'\text{-hydroxypentyl oxazolidin-2-one (minor isomer), anti- and syn-}\)9g

Benzoyloxy carbamate \(8g\) (30 mg, 86 \(\mu\)mol) was subjected to general procedure \(E\). The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 50 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 100 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated \(\textit{in vacuo}\) to deliver the title compound as a yellow oil and a mixture of diastereomers (3:1, 14 mg, 70 \(\mu\)mol, 82%). The NMR data below are given for the major isomer.

\(\nu_{\text{max}} \text{(thin film)/cm}^{-1}\) 2923\(m\), 1684\(s\), 1609\(m\), 1434\(m\), 1292\(s\), 1178\(w\), 1097\(w\), 856\(w\); \(\delta_{\text{H}} \text{(400 MHz, CD}_{3}\text{OD)}\) 0.97 (3 H, t, \(J\) 7.1), 1.32 - 1.67 (4 H, m), 3.20 (1 H, q, \(J\) 5.3), 3.37 (3 H, s), 3.61 (1 H, dd, \(J\) 5.3, 4.0), 4.03 (1 H, ddd, \(J\) 9.0, 6.0, 4.0), 4.41 (1 H, app t, \(J\) 9.0), 4.48 (1 H, dd, \(J\) 9.0, 6.0); \(\delta_{\text{C}} \text{(126 MHz, CD}_{3}\text{OD)}\) 14.6, 19.1, 33.3, 55.3, 58.3, 67.7, 73.6, 83.7, 162.7; \textbf{HRMS (ESI)} \(\text{C}_{9}\text{H}_{17}\text{NNaO}_{4}\) requires 226.1050, found 226.1059 (–4.3 ppm).
**Z-4-Hydroxyhept-2-enyl mesitoyloxycarbamate 8h**

Benzoyloxycarbamate 8c (30 mg, 67 µmol) was subjected to general procedure N. Flash column chromatography on silica gel (4:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (20 mg, 60 µmol, 89%).

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} \]
\[
3444\text{br s}, 2959\text{m}, 1738\text{m}, 1612\text{w}, 1458\text{w}, 1230\text{m}, 1158\text{w,}
1095\text{w}, 850\text{w} ;
\]
\[ \delta \text{H (400 MHz, CDCl}_3 \text{)} \]
\[
0.94 \text{ (3 H, t, J 7.0), 1.23 - 1.52 (3 H, m) 1.54 -}
1.73 \text{ (2 H, m), 2.30 (3 H, s), 2.37 (6 H, s), 4.53 (1 H, ddd, J 7.9, 7.0, 6.1), 4.70 (1 H,}
\]
\[ \delta \text{C (100 MHz, CDCl}_3 \text{)} \]
\[
14.0, 18.5, 19.9, 21.2, 39.0, 62.4, 67.3, 123.8, 128.7,
138.6, 126.3, 136.7, 141.0, 156.5, 168.9;
\]
\[ \text{HRMS (ESI)} \]
\[
\text{C}_{18}\text{H}_{25}\text{NNaO}_5 \text{ requires 358.1630, found 358.1624 (0.3 ppm).}
\]

The title compound, benzoyloxycarbamate 8h (15 mg, 45 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 50 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 100 mL of dichloromethane:methanol (10:1), and the resultant filtrate was concentrated \textit{in vacuo} to deliver \textit{anti-} and \textit{syn-9} as a colourless oil and a mixture of diastereomers (1.5:1, 6 mg, 32 µmol, 68%).

41
(E)-4-(Benzyloxy)hept-2-en-1-ol, 13a

Alcohol 6a (500 mg, 2.27 mmol) was subjected to general procedure Q. Flash column chromatography on silica gel (92:8 petrol:ethyl acetate) afforded the title compound as a colourless oil (420 mg, 1.91 mmol, 84%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3383br m, 3031s, 2959s, 2871s, 1693m, 1496w, 1454s, 1378m, 1205w, 1089s, 1027m, 975m, 736m; $\delta_H$ (400 MHz, CDCl$_3$) 0.87 - 0.98 (3 H, m), 1.30 - 1.55 (3 H, m), 1.60 - 1.74 (1 H, m), 2.35 (1 H, br s), 3.80 (1 H, app q, J 7.4), 4.14 (2 H, d, J 5.3), 4.38 (1 H, d, J 11.9), 4.60 (1 H, d, J 11.9), 5.62 (1 H, dd, J 15.4, 7.4), 5.80 (1 H, dt, J 15.4, 5.3), 7.16 - 7.46 (5 H, m); $\delta_C$ (100 MHz, CDCl$_3$) 14.0, 18.6, 37.8, 62.8, 70.1, 79.3, 127.4, 127.7, 128.2, 132.1, 138.8; HRMS (ESI) C$_{14}$H$_{20}$NaO$_4$ requires 243.1361, found 243.1353 (1.2 ppm).

(E)-4-(Benzyloxy)hept-2-enyl mesitoyloxycarbamate, 14a

Alcohol 13a (420 mg, 1.91 mmol) was subjected to general procedure D. Flash column chromatography on silica gel (97:3 petrol:ethyl acetate) afforded the title compound as a colourless oil (530 mg, 1.28 mmol, 67%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3386br s, 3031w, 2958s, 2468w, 1754s, 1454m, 1229m, 1096m; $\delta_H$ (400 MHz, CDCl$_3$) 0.91 (3 H, t, J 6.8), 1.26 - 1.58 (3 H, m), 1.59 - 1.76 (1 H, m), 2.31 (3 H, s), 2.38 (6 H, s), 3.81 (1 H, app q, J 6.1), 4.37 (1 H, d, J 11.9), 4.60 (1 H, d, J 11.9), 4.77 (2 H, d, J 4.5), 5.71 - 5.86 (2 H, m), 6.90 (2 H, s), 7.24 - 7.39 (5 H, m), 7.44 - 7.46 (2 H, m).
8.41 (1 H, s); δC (100 MHz, CDCl3) 14.0, 18.5, 19.9, 21.2, 37.6, 66.5, 70.3, 78.8, 125.6, 126.5, 127.5, 127.7, 128.3, 128.7, 136.6, 136.7, 138.6, 140.9, 156.4, 169.0; HRMS (ESI) C25H31NNaO5 requires 448.2100, found 448.2084 (2.4 ppm).

Diastereomeric mixture of (±) (R)-4-((1'S,2'S)-2'((benzyloxy)-1'-hydroxypentyl)oxazolidin-2-one, (major) with (±) (R)-4-((1'S,2'R)-2'((benzyloxy)-1'-hydroxypentyl)oxazolidin-2-one (minor), anti- and syn-17a

Benzoyloxy carbamate 14a (200 mg, 471 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 250 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 500 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated in vacuo to deliver the title compound as a white solid (96 mg, 370 µmol, 79%) and a mixture of diastereomers (5:1). The NMR data below are given for the major isomer.

**Melting point** 88 - 91 °C; νmax (KBr disk)/cm⁻¹ 3343br m, 2925s, 1747s, 1415m, 1259m, 1093s; δH (400 MHz, CD3OD) 0.96 (3 H, t, J 7.3), 1.34 - 1.57 (2 H, m), 1.58 - 1.79 (2 H, m), 3.40 - 3.51 (2 H, m), 4.10 (1 H, ddd, J 9.0, 6.1, 4.0), 4.24 (1 H, dd, J 9.0, 6.1), 4.43 (1 H, app t, J 9.0), 4.52 (1 H, d, J 11.4), 4.61 (1 H, d, J 11.4), 7.25 - 7.40 (5 H, m); δC (100 MHz, CD3OD) 14.7, 18.4, 33.4, 55.8, 69.3, 73.0, 74.0, 81.3, 128.8, 129.1, 129.4, 139.9, 162.8; HRMS (ESI) C15H21NNaO4 requires 279.1477, found 279.1471 (2.3 ppm).
Diastereomeric mixture of (±) \((R)-4-((1'S,2'\,R)-(1',2'-\text{dihydroxypentyl})\text{oxazolidin-2-one})\), \textit{anti-} and \textit{syn-45}

Carbamates \textit{anti-} and \textit{syn-17a} (50 mg, 19 µmol) was subjected to general procedure F to afford the title compound as a colourless oil (30 mg, 16 µmol, 84%) and a mixture of diastereomers (5:1).

\begin{align*}
\nu_{\text{max}} \text{(thin film)} / \text{cm}^{-1} &= 3316\text{br s}, 2957\text{s}, 1752\text{w}, 1406\text{w}, 1254\text{m}, 1034\text{m}, 927\text{m}, 837\text{m}; \\
\delta_H \text{(400 MHz, CD}_3\text{OD)} &= 0.97 \text{ (3 H, t, } J = 7.2), 1.28 - 1.45 \text{ (2 H, m), 1.48 - 1.65} \text{ (1 H, m), 1.66 - 1.83} \text{ (1 H, m), 3.21} \text{ (1 H, dd, } J = 8.1, 4.3), 3.45 \text{ (1 H, app td, } J = 8.1, 2.7), 4.15 \text{ (1 H, ddd, } J = 8.7, 6.3, 4.3), 4.30 \text{ (1 H, dd, } J = 8.7, 6.3), 4.49 \text{ (1 H, app t, } J = 8.7); \\
\delta_C \text{(100 MHz, CD}_3\text{OD)} &= 13.4, 18.6, 36.3, 54.8, 68.2, 72.3, 75.3, 161.8; \text{ HRMS (ESI) } C_8H_{15}NNaO_4 \text{ requires 212.0893, found 212.0896} (\pm 1.4 \text{ ppm}).
\end{align*}

Diastereomeric mixture of (±) \((R)-4-((4'S,5'R)-2',2'-\text{dimethyl-5'-propyl-1,3-dioxolan-4-yl})\text{oxazolidin-2-one})\) (major) with (±) \((R)-4-((4'S,5'S)-2',2'-\text{dimethyl-5'-propyl-1,3-dioxolan-4-yl})\text{oxazolidin-2-one})\) (minor), \textit{anti-} and \textit{syn-18a}

Diols \textit{anti-} and \textit{syn-45} (15 mg, 79 µmol) was subjected to general procedure G. Flash column chromatography on silica gel (3:1 petrol:ethyl acetate) afforded the title...
compound as a colourless oil (12 mg, 52 µmol, 67%) and a mixture of diastereomers (>5:1). The NMR data below are given for the major isomer.

**Melting point** 103 - 108 °C; $\nu_{\text{max}}$ (KBr disk)/cm$^{-1}$ 3235br s, 2933s, 1741w, 1242m, 1110m, 1066m, 1022m, 808w; $\delta_H$ (400 MHz, CDCl$_3$) 0.93 (3 H, t, $J$ 7.1, C(8')H$_3$), 1.17 - 1.28 (2 H, m, C(7')H$_2$), 1.30 (3 H, s, C(2')CH$_3$), 1.54 (3 H, s, C(2')CH$_3$), 1.56 - 1.66 (2 H, m, C(6')H$_2$), 3.28 (1 H, dt, $J$ 8.3, 6.2, C(4)H), 3.40 (1 H, app t, $J$ 6.2, C(4')H), 3.62 (1 H, dd, $J$ 8.3, 6.2, C(5)H), 3.75 (1 H, app t, $J$ 8.3, C(5)H), 3.76 - 3.85 (1 H, m, C(5'H), 6.63 (1 H, br s, NH); $\delta_C$ (100 MHz, CDCl$_3$) 13.9 (C(8')), 20.3 (C(7')), 25.5 (CH$_3$), 27.7 (CH$_3$), 31.2 (C(6')), 52.4 (C(4)), 66.4 (C(5)), 76.3 (C(5')), 79.3 (C(4')), 108.5 (C(2')), 159.7 (OC(O)N); **HRMS (ESI)** C$_{11}$H$_{19}$NNaO$_4$ requires 252.1206, found 252.1206 (1.0 ppm).

(E)-4-(Benzyloxy)-5-methylhex-2-en-1-ol, 13b

![Chemical Structure](image)

Alcohol 12b (780 mg, 3.58 mmol) was subjected to general procedure P. Flash column chromatography on silica gel (92:8 petrol:ethyl acetate) afforded the title compound as a colourless oil (568 mg, 2.58 mmol, 73%).

$v_{\text{max}}$ (thin film)/cm$^{-1}$ 3385br s, 3029s, 2959s, 1694w, 1496m, 1454s, 1383m, 1067s, 736s; $\delta_H$ (400 MHz, CDCl$_3$) 0.89 (3 H, d, $J$ 6.8), 0.98 (3 H, d, $J$ 6.8), 1.59 (1 H, br s), 1.83 (1 H, app oct, $J$ 6.8), 3.49 (1 H, dd, $J$ 8.1, 6.8), 4.10 - 4.21 (2 H, m), 4.37 (1 H, d, $J$ 12.0), 4.60 (1 H, d, $J$ 12.0), 5.62 (1 H, dd, $J$ 15.7, 8.1), 5.81 (1 H, dt, $J$ 15.7, 6.3), 7.24 - 7.38 (5 H, m); $\delta_C$ (100 MHz, CDCl$_3$) 18.4, 18.7, 32.8, 63.0, 70.3, 84.8, 127.3, 127.6, 128.3, 130.6, 132.9, 139.0; **HRMS (ESI)** C$_{14}$H$_{20}$NaO$_2$ requires 243.1356, found 243.1357 (–0.7 ppm).
\( (E)-4-(\text{Benzyloxy})-5\text{-methylhex-2-enyl mesityloxy} \) \text{carbamate, 14b} \)

\[
\text{\includegraphics[width=0.5\textwidth]{image}}
\]

Alcohol 13b (440 mg, 2.00 mmol) was subjected to general procedure D. Flash column chromatography on silica gel (96:4 petrol:ethyl acetate) afforded the title compound as a colourless oil (537 mg, 1.26 mmol, 63%).

\( \nu_{\text{max}} \) (thin film) cm\(^{-1}\) 3423br s, 1753s, 1612m, 1454m, 1227s, 1158m; \( \delta_H \) (400 MHz, CDCl\(_3\)) 0.89 (3 H, d, J 6.8), 0.97 (3 H, d, J 6.8), 1.83 (1 H, m, J 6.8, app oct), 2.31 (3 H, s), 2.38 (6 H, s), 3.50 (1 H, app t, J 6.8), 4.35 (1 H, d, J 12.1), 4.60 (1 H, d, J 12.1), 4.78 (2 H, d, J 4.5), 5.73 - 5.80 (2 H, m), 6.90 (2 H, s), 7.24 - 7.39 (5 H, m), 8.38 (1 H, br s); \( \delta_C \) (100 MHz, CDCl\(_3\)) 18.4, 18.6, 19.9, 21.2, 32.7, 66.5, 70.4, 84.3, 126.5, 126.7, 127.4, 127.7, 128.3, 128.7, 134.9, 136.7, 138.7, 140.9, 156.4, 169.0; HRMS (ESI) \( \text{C}_{25}\text{H}_{31}\text{NNaO}_5 \) requires 448.2094, found 448.2091 (0.8 ppm).

Diastereomeric mixture of (±) \( (R)-4-((1'S,2'R)-2'-(\text{benzyloxy})-1'\text{-hydroxy}-3'\text{-methylbutyl})\text{oxazolidin-2-one} \) (major) with (±) \( (R)-4-((1'S,2'S)-2'-(\text{benzyloxy})-1'\text{-hydroxy}-3'\text{-methylbutyl})\text{oxazolidin-2-one} \) (minor), \textit{anti} - and \textit{syn} - 17b

Benzoyloxy carbamate 14b (200 mg, 471 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 250 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 500 mL of petrol:ethyl acetate (1:3),
and the resultant filtrate was concentrated in vacuo to deliver the title compound as a colourless oil and a mixture of diastereomers (7.2:1, 96 mg, 340 µmol, 71%). The NMR data below are given for the major isomer.

\[
\begin{align*}
\nu_{\text{max}} \text{ (thin film)/cm}^{-1} & \quad 3385 \text{br s, } 2957 \text{m, } 1741 \text{s, } 1643 \text{m, } 1393 \text{m, } 1190 \text{m, } 1026 \text{m}; \\
\delta_H \text{ (400 MHz, CD}_3\text{OD)} & \quad 1.00 \text{ (3 H, d, } J 7.0), \quad 1.08 \text{ (3 H, d, } J 7.0), \quad 2.12 \text{ (1 H, septd, } J 7.0, 3.3), \quad 3.30 - 3.37 \text{ (1 H, m), } 3.47 \text{ (1 H, dd, } J 8.3, 3.8), \quad 4.08 \text{ (1 H, ddd, } J 8.8, 5.9, 3.8), \quad 4.22 \text{ (1 H, dd, } J 8.8, 5.9, 4.38 \text{ (1 H, app t, } J 8.8), \quad 4.62 \text{ (1 H, d, } J 11.1), \quad 4.65 \text{ (1 H, d, } J 11.1), \quad 7.24 - 7.44 \text{ (5 H, m)}; \\
\delta_C \text{ (100 MHz, CD}_3\text{OD)} & \quad 15.9, \quad 19.3, \quad 29.6, \quad 54.9, \quad 68.4, \quad 72.2, \quad 74.8, \quad 85.4, \quad 127.8, \quad 127.9, \quad 128.4, \quad 138.9, \quad 161.8; \quad \text{HRMS (ESI)} \quad C_{15}H_{21}NNaO_4, \quad \text{requires 302.1363, found 302.1363 (0.0 ppm).}
\end{align*}
\]

**Diastereomeric mixture of (±) (R)-4-((1'S,2'R)-(1',2'-dihydroxy-3'-methylbutyl)oxazolidin-2-one (major) with (±) (R)-4-((1'S,2'S)-(1',2'-dihydroxy-3'-methylbutyl)oxazolidin-2-one and (minor), anti- and syn-46**

Alcohols *anti-* and *syn-17b* (26 mg, 93 µmol) was subjected to general procedure F, to afford the title compound as a yellow oil (15 mg, 79 µmol, 83%) and a mixture of diastereomers (major:minor 7:1). The NMR data below are given for the major isomer.

\[
\begin{align*}
\nu_{\text{max}} \text{ (thin film)/cm}^{-1} & \quad 3254 \text{br s, } 2961 \text{s, } 1755 \text{s, } 1381 \text{w, } 1217 \text{m, } 1039 \text{s}; \\
\delta_H \text{ (400 MHz, CD}_3\text{OD)} & \quad 0.81 - 0.96 \text{ (3 H, m), } 0.96 - 1.10 \text{ (3 H, m), } 1.94 - 2.10 \text{ (1 H, m), } 3.25 - 3.45 \text{ (2 H, m), } 4.08 - 4.23 \text{ (1 H, m), } 4.27 - 4.38 \text{ (1 H, m), } 4.43 - 4.58 \text{ (1 H, m)}; \\
\delta_C \text{ (100 MHz, CD}_3\text{OD)} & \quad 14.2, \quad 19.3, \quad 29.2, \quad 55.2, \quad 68.2, \quad 72.5, \quad 76.3, \quad 161.8; \quad \text{HRMS (ESI)} \quad C_{8}H_{15}NNaO_4 \text{ requires 212.0893, found 212.0895 (–0.7 ppm).}
\end{align*}
\]
Diastereomeric mixture of (±) \((R)-4-((4'R,5'S)-5'-\text{isopropyl}-2,2\text{-dimethyl}-1,3\text{-dioxolan-4-yl})\text{oxazolidin-2-one}\) (major) with (±) \((R)-4-((4'R,5'R)-5'-\text{isopropyl}-2,2\text{-dimethyl}-1,3\text{-dioxolan-4-yl})\text{oxazolidin-2-one}\) (minor), \textit{anti-} and \textit{syn-18b}

\[
\text{\begin{center}
\includegraphics[width=0.5\textwidth]{diagram.png}
\end{center}}
\]

Diols \textit{anti-} and \textit{syn-46} (12 mg, 63 µmol) was subjected to general procedure G. Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (11 mg, 48 µmol, 76%) and a mixture of diastereomers (>7:1). The NMR data below are given for the major isomer.

\[
\begin{align*}
\nu_{\text{max}} \text{(thin film)/cm}^{-1} & \quad 3321\text{m}, 2961\text{m}, 1755\text{s}, 1381\text{m}, 1217\text{m}, 1039\text{m}; \\
\delta_{\text{H}} \text{(400 MHz, C}_6\text{D}_6) & \quad 0.49 \text{ (3 H, d,} J_6 6.6, \text{C'(7)H}), 1.06 \text{ (3 H, d, } J_6 6.3, \text{C'(7)H}), 1.26 \text{ (3 H, s,} C(2')\text{CH}_3), 1.26 - 1.37 \text{ (1 H, m,} C(6')\text{H}), 1.55 \text{ (3 H, s,} C(2')\text{CH}_3), 3.21 \text{ (1 H, ddd,} J_8 8.4, 6.0, 3.3, \text{C(4)H}), 3.31 \text{ (1 H, dd,} J_5 5.7, 3.3, \text{C(4')H}), 3.45 \text{ (1 H, dd,} J_5 10.2, 5.7, \text{C(5')H}), 3.72 \text{ (1 H, app t,} J_8 8.4, \text{C(5)H}), 3.85 \text{ (1 H, dd,} J_8 8.4, 6.0, \text{C(5)H}), 5.61 \text{ (1 H, br s,} \text{NH}); \\
\delta_{\text{C}} \text{(100 MHz, CD}_3\text{OD}) & \quad 19.5 \text{ (C(7')}, 21.0 \text{ (C'7)'), 25.6 \text{ (CH}_3), 27.0 \text{ (CH}_3), 29.0 \text{ (C(6')), 53.2 \text{ (C(4')}, 69.1 \text{ (C(5')}, 79.4 \text{ (C(5), 84.0 \text{ (C(4), 109.6 \text{ (C(2), 162.6 \text{ (OC(O)N; HRMS (ESI) C}_{11}\text{H}_{19}\text{NNaO}_4 \text{requires 252.1206, found 252.1214 (3.0 ppm).}}}
\end{align*}
\]

\[
\text{\begin{center}
\includegraphics[width=0.8\textwidth]{reactions.png}
\end{center}}
\]
(E)-4-(tert-Butyldimethylsilyloxy)-1-phenylbut-2-en-1-ol, 47

Alcohol 39 (990 mg, 3.59 mmol) was subjected to general procedure P. Flash column chromatography on silica gel (96:4 petrol:ethyl acetate) afforded the title compound as a colourless oil (860 mg, 3.12 mmol, 87%).

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} \]
3396 s, 3063 w, 3030 w, 2929 s, 2856 s, 1645 m, 1493 s, 1454 s, 1377 w, 1333 w, 1255 s, 1096 s, 913 w, 836 s, 777 s, 699 s; \[ \delta_{\text{H}} \text{ (400 MHz, CDCl}_3\text{)} \]
0.08 (6 H, s), 0.93 (9 H, s), 2.33 (1 H, br s), 4.22 (2 H, d, J 4.0), 5.22 (1 H, d, J 5.6), 5.85 (1 H, dt, J 15.4, 4.0), 5.93 (1 H, dd, J 15.4, 5.6), 7.23 - 7.46 (5 H, m); \[ \delta_{\text{C}} \text{ (100 MHz, CDCl}_3\text{)} \]
–5.2, 18.4, 26.0, 63.1, 74.5, 126.3, 127.6, 128.5, 130.7, 132.1, 143.0; HRMS (ESI) C\text{16}H\text{26}NaO\text{2}Si requires 301.1594, found 301.1580 (4.7 ppm).

(E)-(4-(Benzyloxy)-4-phenylbut-2-enyloxy)(tert-butyl)dimethylsilane, 48

Alcohol 47 (644 mg, 2.31 mmol) was subjected to general procedure A. Flash column chromatography on silica gel (99:1 petrol:ethyl acetate) afforded the title compound as a yellow oil (786 mg). No yield is given due to impurities in the NMR spectra.

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} \]
3030 m, 2929 s, 2857 s, 1693 s, 1495 m, 1453 s, 1255 s, 1098 s, 837 s, 749 m; \[ \delta_{\text{H}} \text{ (400 MHz, CDCl}_3\text{)} \]
0.14 (6 H, s), 0.99 (9 H, s), 4.29 (2 H, d, J 4.3), 4.58 (1 H, d, J 12.6), 4.62 (1 H, d, J 12.6), 4.95 (1 H, d, J 6.1), 5.89 (1 H, dt, J 15.5, 4.3), 5.96 (1 H, dd, J 15.5, 6.1), 7.30 - 7.54 (10 H, m); \[ \delta_{\text{C}} \text{ (100 MHz, CDCl}_3\text{)} \]
–5.1, 18.5, 26.0, 63.3, 70.1, 81.2, 127.0, 127.5, 127.6, 127.7, 128.4, 128.5, 130.7, 131.9, 138.6, 141.4; HRMS (ESI) C\text{23}H\text{32}NaO\text{2}Si requires 391.2064, found (–0.1 ppm).
(E)-4-(Benzyloxy)-4-phenylbut-2-en-1-ol, 49

\[
\begin{align*}
\text{O} & \quad \text{\_\_\_} \\
& \quad \text{\_\_\_} \\
& \quad \text{\_\_\_} \\
& \quad \text{\_\_\_} \\
& \quad \text{\_\_\_} \\
\end{align*}
\]

Alkene 48 (875 mg, 2.37 mmol) was subjected to general procedure N. Flash column chromatography on silica gel (88:12 petrol:ethyl acetate) afforded the title compound as a yellow oil (505 mg, 1.99 mmol, 82%).

\[
\text{\_\_\_}
\]

\[
\begin{align*}
\nu_{\text{max}} (\text{thin film})/\text{cm}^{-1} & \quad 3386\text{br s, } 3062\text{m, } 3029\text{m, } 2866\text{s, } 1602\text{w, } 1494\text{m, } 1453\text{s, } 1388\text{w, } \\
& \quad 1302\text{w, } 1205\text{w, } 1027\text{s, } 845\text{w, } 740\text{s;} \\
\delta_{\text{H}} (400 \text{ MHz, CDCl}_3) & \quad 1.81 (1 \text{ H, br s}), 4.07 - 4.24 (2 \text{ H, m}), 4.51 (1 \text{ H, d, } J 12.0), 4.55 (1 \text{ H, d, } J 12.0), 4.90 (1 \text{ H, d, } J 3.5), 5.73 - 6.06 (2 \text{ H, m}), 7.21 - 7.53 (10 \text{ H, m}); \\
\delta_{\text{C}} (100 \text{ MHz, CDCl}_3) & \quad 62.9, 70.2, 81.1, 127.0, 127.6, 127.7, 127.8, 128.4, 128.6, 131.3, 132.1, 138.4, 141.0; \\
\text{HRMS (ESI)} & \quad \text{C}_{17}\text{H}_{18}\text{NaO}_2 \text{ requires } 277.1199, \text{ found } 277.1198 \text{ (0.5 ppm).}
\end{align*}
\]

(E)-4-(Benzyloxy)-4-phenylbut-2-en-1-yl mesitoxyloxy carbamate, 14c

\[
\begin{align*}
\text{O} & \quad \text{\_\_\_} \\
& \quad \text{\_\_\_} \\
\end{align*}
\]

Alcohol 49 (505 mg, 1.99 mmol) was subjected to general procedure D. Flash column chromatography on silica gel (98:2 petrol:ethyl acetate) afforded the title compound as a yellow oil (455 mg, 989 µmol, 50%).

\[
\begin{align*}
\nu_{\text{max}} (\text{thin film})/\text{cm}^{-1} & \quad 3265\text{br s, } 2924\text{s, } 1753\text{s, } 1611\text{m, } 1453\text{s, } 1227\text{s, } 1158\text{m, } 1090\text{s, } \\
& \quad 850\text{m; } \delta_{\text{H}} (400 \text{ MHz, CDCl}_3) 2.33 (3 \text{ H, s}), 2.40 (6 \text{ H, s}), 4.54 (1 \text{ H, d, } J 12.6), 4.54 (1 \text{ H, d, } J 12.6), 4.76 (2 \text{ H, d, } J 5.8), 4.92 (1 \text{ H, d, } J 5.8), 5.91 (1 \text{ H, dt, } J 15.4, 5.8), 6.02 (1 \text{ H, dd, } J 15.4, 5.8), 6.90 (2 \text{ H, s}), 7.28 - 7.50 (10 \text{ H, m}), 8.44 (1 \text{ H, br s}); \\
\delta_{\text{C}} (100 \text{ MHz, CDCl}_3) & \quad 19.9, 21.2, 66.4, 70.2, 80.6, 124.9, 126.5, 127.1, 127.6, 127.7, 127.9,
\end{align*}
\]
128.4, 128.6, 128.7, 136.0, 136.7, 138.2, 140.4, 140.9, 156.4, 169.0; **HRMS (ESI)**

C$_{29}$H$_{29}$NNaO$_5$ requires 482.1938, found 482.1937 (0.3 ppm).

**Diastereomeric mixture of (±) (R)-4-((1'S,2'S)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one (major) with (±) (R)-4-((1'S,2'R)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one (minor) anti- and syn-17c**

Benzoyloxy carbamate 14c (200 mg, 435 µmol) was subjected to general procedure F. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 200 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 400 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated *in vacuo* to deliver the title compound as a white solid (110 mg, 351 µmol, 81%) and a mixture of diastereomers (1.5:1). The NMR data below are given for the major isomer.

**Melting point** 161 - 164 °C; $\nu_{\text{max}}$ (KBr disk)/cm$^{-1}$ 3423br s, 2975m, 1736s, 1637m, 1535m, 1401m, 1203m, 978s; $\delta$$_H$ (400 MHz, CDCl$_3$) 1.96 (1 H, br s), 3.47 - 3.53 (1 H, m), 3.65 - 3.75 (1 H, m), 3.92 - 4.12 (1 H, m), 4.17 - 4.37 (2 H, m), 4.43 - 4.55 (2 H, m), 6.53 (1 H, s), 7.20 - 7.50 (10 H, m); $\delta$$_C$ (100 MHz, CDCl$_3$) 53.8, 67.1, 70.7, 75.9, 82.1, 127.3, 127.6, 128.1, 128.2, 128.6, 129.0, 137.3, 137.7, 160.2; **HRMS (ESI)**

C$_{18}$H$_{19}$NNaO$_4$ requires 336.1206, found 336.1211 (–1.5 ppm).
(E)-4-(Benzyloxy)-5,5-dimethylhex-2-en-1-ol, 13d

Alcohol 12d (300 mg, 1.28 mmol) was subjected to general procedure P. Flash column chromatography on silica gel (93:7 petrol:ethyl acetate) afforded the title compound as a yellow oil (231 mg, 987 µmol, 77%).

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} 3356 \text{br s, 3064w, 3031w, 2955s, 2867s, 1606w, 1496m, 1478s, 1455s, 1391s, 1363s, 1206m, 1067s, 977s, 735s, 697s; } \delta_{\text{H}} \text{ (400 MHz, CDCl}_3\text{) 0.95 (9 H, s), 2.03 (1 H, br s), 3.40 (1 H, d, } J 8.1\text{), 4.19 (2 H, d, } J 5.3\text{), 4.35 (1 H, d, } J 12.1\text{), 4.62 (1 H, d, } J 12.1\text{), 5.67 (1 H, dd, } J 15.7, 8.1\text{), 5.80 (1 H, dt, } J 15.7, 5.3\text{), 7.23 - 7.44 (5 H, m); } \delta_{\text{C}} \text{ (100 MHz, CDCl}_3\text{) 26.2, 34.9, 63.0, 70.5, 87.5, 127.2, 127.5, 128.2, 129.2, 133.6, 139.2; HRMS (ESI) } C_{15}H_{22}NO_2 \text{ requires } 257.1517, \text{ found } 257.1512 (–1.9 ppm). \]

(E)-4-(Benzyloxy)-5,5-dimethylhex-2-enyl mesityloxy carbamate, 14d

Alcohol 13d (96 mg, 41 µmol) was subjected to general procedure D. Flash column chromatography on silica gel (93:7 petrol:ethyl acetate) afforded the title compound as a yellow oil (98 mg, 22 µmol, 54%).

\[ \nu_{\text{max}} \text{ (thin film)/cm}^{-1} 3278 \text{br m, 2956s, 1753s, 1611m, 1454m, 1226s, 1158m, 1091s, 850m, 699m; } \delta_{\text{H}} \text{ (400 MHz, CDCl}_3\text{) 0.94 (9 H, s), 2.32 (3 H, s), 2.39 (6 H, s), 3.40 (1 H, d, } J 6.8\text{), 4.32 (1 H, d, } J 12.1\text{), 4.61 (1 H, d, } J 11.9\text{), 4.79 (2 H, d, } J 4.8\text{), 5.72 - 5.88 \]

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(2 H, m), 6.90 (2 H, s), 7.35 (5 H, d, J 4.5), 8.44 (1 H, br s); $\delta_C$ (100 MHz, CDCl$_3$)
19.9, 21.2, 26.1, 34.9, 66.6, 70.6, 87.0, 126.5, 127.3, 127.3, 127.6, 128.2, 128.7, 133.8,
136.7, 138.9, 140.9, 156.4, 169.0; **HRMS (ESI)** $C_{26}H_{33}NNaO_5$ requires 462.2251,
found 462.2255 (–3.8 ppm).

(±) (*R*)-(1'S,2'R)-(2'-(Benzyloxy)-1'-hydroxy-3',3'-dimethylbutyl)oxazolidin-2-one 17d

Benzoyloxy carbamate 14d (15 mg, 34 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 25 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 50 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated *in vacuo* to deliver the title compound (>20:1 d.r., 9 mg, 31 µmol, 90%).

$\nu_{max}$ (thin film)/cm$^{-1}$ 3356br s, 2957s, 1743s, 1396m, 1246m, 1067m, 939m, 698m; $\delta_H$
(400 MHz, CD$_3$OD) 1.04 (9 H, s), 3.18 (1 H, d, J 6.7), 3.64 (1 H, app t, J 6.7), 4.05 (1 H, ddd, J 8.8, 6.7, 6.3), 4.11 (1 H, dd, J 8.8, 6.3), 4.29 (1 H, app t, J 8.8), 4.66 (2 H, s), 7.27 - 7.40 (5 H, m); $\delta_C$ (100 MHz, CD$_3$OD) 26.0, 35.7, 56.0, 68.6, 74.1, 76.3, 90.2,
127.7, 127.8, 128.5, 138.6, 161.5; **HRMS (ESI)** $C_{16}H_{23}NNaO_4$ requires 316.1519,
found 316.1523 (–1.2 ppm).
(R)-4-((1'S,2'R)-(1,2-Dihydroxy-3,3-dimethylbutyl)oxazolidin-2-one, anti-50

![Chemical structure]

Alcohol 17d (10 mg, 34 µmol) was subjected to general procedure F, to afford the title compound as a colourless oil (7 mg, 34 µmol, 99%).

\[ \nu_{\text{max}} (\text{thin film})/\text{cm}^{-1} \] 3384br s, 2961s, 1731s, 1417w, 1260s, 1018s; \[ \delta_H (400 \text{ MHz}, \text{CD}_3\text{OD}) \] 0.98 (9 H, s), 3.18 (1 H, d, J 7.6), 3.53 (1 H, dd, J 7.6, 5.5), 4.15 (1 H, ddd, J 8.8, 6.1, 5.5), 4.25 (1 H, dd, J 8.8, 6.1), 4.48 (1 H, app t, J 8.8); \[ \delta_C (100 \text{ MHz}, \text{CD}_3\text{OD}) \] 26.8, 35.8, 57.3, 69.5, 74.3, 81.1, 162.8; HRMS (ESI) \( C_9H_{17}NNaO_4 \) requires 226.1050, found 226.1052 (–1.1 ppm).

(±) (4R,8R,9R)-8-(tert-Butyl)-9-hydroxy-6,6-dimethyltetrahydrooxazolo-oxazin-2-one, anti-18d

![Chemical structure]

Diol anti-50 (10 mg, 49 µmol) was subjected to general procedure G. Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (6 mg, 26 µmol, 50%).

\[ \nu_{\text{max}} (\text{thin film})/\text{cm}^{-1} \] 3327br m, 2955m, 2360m, 1741s, 1384m, 1254m, 1049m; \[ \delta_H \] (400 MHz, \( \text{CDCl}_3 \)) 0.96 (9 H, s, C(CH\(_3\))\(_3\)), 1.52 (3 H, s, CH\(_3\)), 1.69 (3 H, s, CH\(_3\)), 3.23 (1 H, d, J 4.5, C(8)H), 3.79 - 3.88 (1 H, m, C(9)H), 4.05 (1 H, ddd, J 9.3, 7.3, 3.5, C(4)H), 4.31 (1 H, app t, J 9.3, C(5)H), 4.46 (1 H, dd, J 9.3, 7.3, C(5)H); \[ \delta_C \] (126 MHz, \( \text{CDCl}_3 \)) 21.1 (CH\(_3\)), 25.4 (C(CH\(_3\))\(_3\)), 25.5 (CH\(_3\)), 33.5 (C(CH\(_3\))), 54.5 (C(4)), 54
61.4 (C(5)), 68.0 (C(9)), 83.9 (C(8)), 87.0 (C(6)), 155.3 (NC(O)O); HRMS (ESI)
C_{12}H_{21}NNaO_4 requires 266.1363, found 266.1365 (–0.8 ppm).

((4-Methylpent-1-yn-3-ylxy)methyl)benzene, 37

Commercially available isobutyraldehyde (2.34 g, 33.3 mmol) was subjected to general procedure O. The crude product was taken and subjected directly to general procedure A. Flash column chromatography on silica gel (4:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (1.69 g, 10.2 mmol, 31% over two steps).

\[ \nu_{\text{max}}(\text{thin film})/\text{cm}^{-1} \]
3304br w, 3030s, 2963s, 2926m, 1496m, 1454s, 1384m, 1260w, 1068s, 801w, 736m; \[ \delta_{\text{H}}(400 \text{ MHz, CDCl}_3) \]
1.04 (3 H, d, J 6.0), 1.07 (3 H, d, J 6.0), 2.01 (1 H, app oct, J 6.0), 2.48 (1 H, d, J 2.0), 3.89 (1 H, dd, J 6.0, 2.0), 4.54 (1 H, d, J 12.0), 4.86 (1 H, d, J 12.0), 7.23 - 7.44 (5 H, m); \[ \delta_{\text{C}}(100 \text{ MHz, CDCl}_3) \]
17.9, 18.5, 33.0, 70.6, 74.0, 74.5, 81.7, 127.6, 127.9, 128.3, 138.1; HRMS (ESI) C_{13}H_{16}O requires 188.1201, found 188.1202 (0.5 ppm).
4-(Benzyloxy)-5-methylhex-2-yn-1-ol, 12b

Alkyne 37 (960 mg, 5.81 mmol) was subjected to general procedure B. Flash column chromatography on silica gel (20:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (792 mg, 3.63 mmol, 63%).

\[ \text{ν}_{\text{max}} \text{(thin film)/cm}^{-1} 3383\text{br s}, 3031\text{m}, 2959\text{s}, 2871\text{w}, 1721\text{m}, 1496\text{m}, 1454\text{s}, 1334\text{s}, 1207\text{m}, 1151\text{s}, 1069\text{s}, 735\text{s}, 698\text{s}; \delta_H (400 \text{ MHz, CDCl}_3) 1.01 \text{ (3 H, d, J 6.8)}, 1.03 \text{ (3 H, d, J 6.8)}, 1.67 \text{ (1 H, br s)}, 1.98 \text{ (1 H, app oct, J 6.6)}, 3.91 \text{ (1 H, d, J 6.6)}, 4.32 - 4.37 \text{ (2 H, m)}, 4.52 \text{ (1 H, d, J 12.0)}, 4.81 \text{ (1 H, d, J 12.0)}, 7.25 - 7.42 \text{ (5 H, m)}; \delta_C (400 \text{ MHz, CDCl}_3) 18.0, 18.5, 33.1, 51.2, 70.7, 74.3, 83.7, 84.7, 127.6, 127.8, 128.3, 138.2; \]

HRMS (ESI) \(C_{14}H_{18}NaO_2\) requires 241.1199, found 241.1200 (–0.6ppm).

(Z)-4-(Benzyloxy)-5-methylhex-2-en-1-ol, 15b

Alcohol 12b (490 mg, 2.24 mmol) was subjected to general procedure C. Flash column chromatography on silica gel (92:8 petrol:ethyl acetate) afforded the title compound as a colourless oil (400 mg, 1.82 mmol, 81%).

\[ \text{ν}_{\text{max}} \text{(thin film)/cm}^{-1} 3405\text{br s}, 2959\text{s}, 1692\text{w}, 1496\text{m}, 1454\text{m}, 1383\text{m}, 1067\text{s}, 976\text{s}, 736\text{s}, 698\text{s}; \delta_H (400 \text{ MHz, CDCl}_3) 0.89 \text{ (3 H, d, J 6.8)}, 1.00 \text{ (3 H, d, J 6.8)}, 1.83 \text{ (1 H, app oct, J 7.0)}, 2.39 \text{ (1 H, br s)}, 3.78 \text{ (1 H, dd, J 9.0, 7.0)}, 4.01 - 4.10 \text{ (1 H, m)}, 4.13 - 4.25 \text{ (1 H, m)}, 4.36 \text{ (1 H, d, J 12.0)}, 4.61 \text{ (1 H, d, J 12.0)}, 5.47 \text{ (1 H, dd, J 10.9, 9.0)}, \]
5.87 (1 H, dt, J 10.9, 6.6), 7.25 - 7.42 (5 H, m); δC (100 MHz, CDCl₃) 18.3, 18.8, 32.8, 58.7, 70.2, 79.3, 127.5, 127.7, 128.3, 131.1, 132.8, 138.7; HRMS (ESI) C_{14}H_{20}NaO₂ requires 243.1356, found 243.1350 (2.2 ppm).

(Z)-4-(Benzyloxy)-5-methylhex-2-enyl mesitoyloxy carbamate, 16b

![Chemical structure](image)

Alcohol 15b (366 mg, 1.66 mmol) was subjected to general procedure D. Flash column chromatography on silica gel (97:3 petrol:ethyl acetate) afforded the title compound as a yellow oil (445 mg, 1.05 mmol, 63%).

νmax (thin film)/cm⁻¹ 3267br s, 2960s, 1753s, 1611s, 1454s, 1228s, 1158s, 1094s, 851m, 737m; δH (400 MHz, CDCl₃) 0.87 (3 H, d, J 6.8), 0.98 (3 H, d, J 6.8), 1.82 (1 H, app oct, J 6.8), 2.31 (3 H, s), 2.38 (6 H, s), 3.81 (1 H, dd, J 10.0, 6.8), 4.35 (1 H, d, J 11.9), 4.59 (1 H, d, J 11.9), 4.69 (1 H, dd, J 12.6, 6.0), 4.83 (1 H, dd, J 12.6, 8.0), 5.63 (1 H, dd, J 11.0, 10.0), 5.86 (1 H, ddd, J 11.0, 8.0, 6.0), 6.90 (2 H, s), 7.22 - 7.38 (5 H, m), 8.25 (1 H, br s); δC (100 MHz, CDCl₃) 18.2, 18.6, 19.9, 21.2, 32.9, 62.5, 70.4, 79.2, 126.4, 126.6, 127.5, 127.7, 128.3, 128.7, 134.6, 136.7, 138.6, 141.0, 156.3, 169.0; HRMS (ESI) C_{25}H_{31}NNaO₅ requires 448.2094, found 448.2100 (–1.3 ppm).
Diastereomeric mixture of (±) \((R)-4-((1'R,2'S)-(2'-(benzylxy)-1'-hydroxy-2'-methylbutyl)oxazolidin-2-one\) (major) with (±) \((R)-4-((1'R,2'R)-(2'-(benzylxy)-1'-hydroxy-2'-methylbutyl)oxazolidin-2-one\) (minor), anti- and syn-19b

Benzoyloxycarbamate \(18b\) (200 mg, 471 \(\mu\)mol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 250 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 500 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated \textit{in vacuo} to deliver the title compound as a mixture of diastereomers (4:1, 116 mg, 414 \(\mu\)mol, 85%). Flash column chromatography on silica gel (3:1 to 1:1 petrol:ethyl acetate) allowed isolation of the major (75 mg) and minor (21 mg) diastereomers as colourless oils.

**Major diastereomer; \(\nu_{\text{max}}\) (thin film)/cm\(^{-1}\) 3251br s, 2975s, 2870s, 1742s, 1520m, 1433m, 1267m, 1065m, 1026s; \(\delta_{\text{H}}\) (400 MHz) 1.00 (3 H, d, \(J = 6.8\)), 1.03 (3 H, d, \(J = 6.8\)), 2.00 (1 H, sptd, \(J = 6.8, 5.9\)), 3.23 (1 H, t, \(J = 5.9\)), 3.76 (1 H, dd, \(J = 5.9, 2.5\)), 4.06 (1 H, ddd, \(J = 9.0, 6.2, 2.5\)), 4.20 (1 H, t, \(J = 9.0\)), 4.47 (1 H, dd, \(J = 9.0, 6.1\)), 4.57 (1 H, d, \(J = 11.4\)), 4.61 (1 H, d, \(J = 11.4\)), 7.24 - 7.39 (5 H, m); \(\delta_{\text{C}}\) (100 MHz, CD\(_3\)OD) 17.1, 19.0, 30.1, 54.3, 66.1, 71.5, 74.7, 85.8, 127.8, 128.2, 128.5, 138.8, 161.7; HRMS (ESI) C\(_{15}\)H\(_{21}\)NNaO\(_4\) requires 302.1363, found 302.1364 (0.4 ppm).

**Minor diastereomer; \(\nu_{\text{max}}\) (thin film)/cm\(^{-1}\) 3248br s, 2956s, 2886s, 1745s, 1260m, 1069m; \(\delta_{\text{H}}\) (400 MHz, CD\(_3\)OD) 1.02 (3 H, d, \(J = 6.8\)), 1.07 (3 H, d, \(J = 6.8\)), 2.04 - 2.18 (1 H, m), 3.21 (1 H, dd, \(J = 7.2, 2.4\)), 3.70 (1 H, dd, \(J = 5.6, 2.3\)), 3.98 (1 H, dt, \(J = 8.8, 5.6\)), 4.37 (1 H, app t, \(J = 8.8\)), 4.49 (1 H, dd, \(J = 8.8, 5.8\)), 4.58 (1 H, d, \(J = 11.3\)), 4.69 (1 H, d, \(J = 11.3\)).
11.3), 7.25 - 7.43 (5 H, m); δC (100 MHz, CD$_3$OD) 18.4, 18.5, 30.0, 55.2, 67.5, 72.5, 74.2, 84.8, 127.7, 128.0, 128.4, 139.1, 161.6; HRMS (ESI) C$_{15}$H$_{21}$NNaO$_4$ requires 302.1363, found 302.1363 (0.1 ppm).

(±) (R)-4-((1'R,2'S)-1',2'-Dihydroxy-3'-methylbutyl)oxazolidin-2-one, $anti$-38

Alcohol $anti$-19b (26 mg, 93 µmol) was subjected to general procedure F, to afford the title compound as a colourless oil (17 mg, 89 µmol, 94%).

ν$_{\text{max}}$ (thin film)/cm$^{-1}$ 3314br m, 2957s, 1752s, 1471w, 1406w, 1254m, 1033m, 926m, 837m; δH (400 MHz, CD$_3$OD) 0.91 (3 H, d, $J$ 6.8), 0.98 (3 H, d, $J$ 6.8), 1.97 (1 H, sptd, $J$ 6.8, 3.8), 3.24 (1 H, dd, $J$ 8.2, 3.8), 3.57 (1 H, dd, $J$ 8.2, 3.0), 4.16 (1 H, ddd, $J$ 8.8, 5.9, 3.0), 4.40 (1 H, app t, $J$ 8.8), 4.49 (1 H, dd, $J$ 8.8, 5.8); δC (100 MHz, CD$_3$OD) 14.7, 19.1, 29.6, 54.8, 65.9, 71.7, 76.5, 161.8; HRMS (ESI) C$_8$H$_{13}$NNaO$_4$ requires 212.0893, found 212.0892 (0.4 ppm).
(±) (R)-4-((4'R,5'S)-5'-Isopropyl-2,2-dimethyl-1,3-dioxolan-4-yl)oxazolidin-2-one, 

*anti*-20b

![Structure](image)

Diol *anti*-38 (10 mg, 53 µmmol) was subjected to general procedure G. Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as a white solid (7 mg, 31 µmol, 58%).

**Melting point** 117 - 120 °C; \(\nu_{\text{max}} (\text{KBr disk})/\text{cm}^{-1}\) 3361br s, 2961s, 1755s, 1450 m, 1397m, 1217m, 1039s; \(\delta_{\text{H}} (400 \text{ MHz, CDCl}_3)\) 0.99 (3 H, d, \(J\) 6.6, C(7')H), 1.09 (3 H, d, \(J\) 6.5, C(7')H), 1.34 - 1.38 (3 H, m, CH\(_3\)), 1.43 (3 H, s, CH\(_3\)), 1.56 - 1.72 (1 H, m, C(6')H), 3.79 (1 H, dd, \(J\) 10.0, 5.5, C(5')H), 3.97 (1 H, app q, \(J\) 6.2, C(4)H), 4.08 (1 H, dd, \(J\) 6.2, 5.5, C(4')H), 4.40 - 4.53 (2 H, m, C(5)H\(_2\)), 5.46 (1 H, br s, NH); \(\delta_{\text{C}} (100 \text{ MHz, CDCl}_3)\) 19.6 (C(7')), 20.3 (C(7')), 25.2 (CH\(_3\)), 27.4 (CH\(_3\)), 27.5 (C(4')), 51.7 (C(4)), 67.5 (C(5)), 78.7 (C(4')), 82.6 (C(5')), 108.1 (C(2')), 160.1 (NC(O)O); \(\text{HRMS (ESI)}\) C\(_{11}\)H\(_{19}\)NNaO\(_4\) requires 252.1206, found 252.1213 (2.6 ppm).

(±) (R)-4-((1'R,2'R)-1',2'-Dihydroxy-3'-methylbutyl)oxazolidin-2-one, *syn*-38

![Structure](image)

Alcohol *syn*-19b (26 mg, 93 µmol) was subjected to general procedure F, to afford the title compound as a colourless oil (15 mg, 79 µmol, 83%).

\(\nu_{\text{max}} (\text{thin film})/\text{cm}^{-1}\) 3331br s, 2957s, 1751s, 1406w, 1254m, 1032m, 837m, 775m; \(\delta_{\text{H}} (400 \text{ MHz, D}_2\text{O})\) 0.81 (3 H, d, \(J\) 6.6), 0.87 (3 H, d, \(J\) 6.6), 1.71 (1 H, dspt, \(J\) 8.3, 6.6),
3.11 (1 H, dd, J 8.3, 2.5), 3.74 (1 H, dd, J 5.1, 2.5), 4.02 (1 H, ddd, J 8.3, 6.1, 5.1), 4.45 (2 H, m); δ<sub>C</sub> (126 MHz, D<sub>2</sub>O) 18.0, 18.3, 29.8, 54.7, 67.5, 71.3, 76.5, 162.1; HRMS (ESI) C<sub>8</sub>H<sub>13</sub>NNaO<sub>4</sub> requires 212.0893, found 212.0899 (–2.8 ppm).

(±) (R)-4-((4'R,5'S)-5'-Isopropyl-2,2-dimethyl-1,3-dioxolan-4-yl)oxazolidin-2-one, syn-20b

Diol syn-38 (10 mg, 53 µmol) was subjected to general procedure G. Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (7 mg, 30 µmol, 58%).

ν<sub>max</sub> (thin film)/cm<sup>-1</sup> 3250br m, 2961s, 1753s, 1447m, 1386m, 1258m, 1038m; δ<sub>H</sub> (400 MHz, CDCl<sub>3</sub>) 0.96 - 1.00 (3 H, m, C(7')H<sub>3</sub>), 1.03 (3 H, d, J 6.6, C(7')H<sub>3</sub>), 1.28 (3 H, s, C(2')CH<sub>3</sub>), 1.34 (3 H, s, C(2')CH<sub>3</sub>), 1.64 - 1.78 (1 H, m, C(6')H), 3.27 - 3.34 (1 H, m, C(4)H), 3.42 (1 H, app t, J 6.6, C(5')H), 3.64 (1 H, app t, J 6.6, C(4')H), 3.92 (1 H, app t, J 8.6, C(5)H), 4.07 - 4.14 (1 H, m, C(5)H), 7.13 (1 H, br s, NH); δ<sub>C</sub> (100 MHz, CDCl<sub>3</sub>) 17.5 (C(7')), 19.5 (C(7')), 27.3 (CH<sub>3</sub>), 27.5 (CH<sub>3</sub>), 31.4 (C(6')), 54.6 (C(4)), 67.1 (C(5)), 79.9 (C(4')), 84.1 (C(5')), 109.6 (C(2')), 159.9 (NC(O)O); HRMS (ESI) C<sub>11</sub>H<sub>19</sub>NNaO<sub>4</sub> requires 252.1206, found 252.1213 (–2.6 ppm).
Alkyne 33 (1.00 g, 5.88 mmol) was subjected to general procedure K. Flash column chromatography on silica gel (97:3 petrol:ethyl acetate) afforded the title compound as a colourless oil (1.29 g, 4.67 mmol, 82%).

\[ \nu_{\text{max}} \text{(thin film)/cm}^{-1} \]

3384br m, 2929s, 2857s, 1649w, 1494m, 1454m, 1364m, 1256s, 1127s, 1084s, 1005m, 836s, 779m; \( \delta_H \) (400 MHz, CDCl\textsubscript{3}) 0.13 (6 H, s), 0.93 (9 H, s), 2.58 - 2.87 (1 H, br s), 4.41 (2 H, s), 5.43 - 5.55 (1 H, m), 7.29 - 7.44 (3 H, m), 7.49 - 7.60 (2 H, m); \( \delta_C \) (100 MHz, CDCl\textsubscript{3}) -5.1, 18.3, 25.8, 51.8, 64.5, 84.6, 85.3, 126.7, 128.3, 128.5, 140.5; HRMS (ESI) C\textsubscript{16}H\textsubscript{24}NaO\textsubscript{2}Si requires 299.1438, found 299.1437 (–0.2 ppm).
(Z)-4-(tert-Butyldimethylsilyloxy)-1-phenylbut-2-en-1-ol, 40

Alcohol 39 (600 mg, 2.17 mmol) was subjected to general procedure C. Flash column chromatography on silica gel (97:3 petrol:ethyl acetate) afforded the title compound as a yellow oil (600 mg, 2.16 mmol, 99%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3412br s, 2956s, 1720s, 1598w, 1494w, 1450s, 1259s, 1095m, 836s, 697s; $\delta_H$ (400 MHz, CDCl$_3$) 0.11 (6 H, s), 0.94 (9 H, s), 2.55 (1 H, br s), 4.31 (1 H, dd, J 13.0, 4.4), 4.45 (1 H, dd, J 13.0, 4.4), 5.48 - 5.60 (1 H, m), 5.67 - 5.79 (2 H, m), 7.23 - 7.32 (1 H, m), 7.32 - 7.45 (4 H, m); $\delta_C$ (100 MHz, CDCl$_3$) –5.2, 18.3, 25.9, 59.7, 70.0, 126.0, 127.5, 128.5, 130.8 and 133.5, 143.2; HRMS (ESI) C$_{16}$H$_{26}$NaO$_2$ requires 301.1594, found 301.1585 (3.0 ppm).

(Z)-(4-(Benzyloxy)-4-phenylbut-2-enyloxy)(tert-butyl)dimethylsilane, 41

Alcohol 40 (600 mg, 2.16 mmol) was subjected to general procedure A. Flash column chromatography on silica gel (97:3 petrol:ethyl acetate) afforded the title compound as a colourless oil (661 mg). No yield is given due to impurities in the NMR spectra.

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3030w, 2929s, 2857s, 1694s, 1494w, 1453s, 1254s, 1090s, 837s, 778m; $\delta_H$ (400 MHz, CDCl$_3$) 0.11 (3 H, s), 0.12 (3 H, s), 0.92 - 1.01 (9 H, m), 4.27 (1 H, dd, J 13.6, 6.0), 4.40 (1 H, dd, J 13.6, 6.0), 4.55 (1 H, d, J 12.5), 4.60 (1 H, d, J 12.5), 5.22 (1 H, d, J 8.9), 5.72 (1 H, dd, J 11.1, 8.9), 5.80 (1 H, dt, J 11.1, 6.0), 7.23 - 7.57 (10 H, m); $\delta_C$ (100 MHz, CDCl$_3$) –5.1, –5.1, 18.3, 25.9, 59.6, 69.9, 76.2, 126.7,
127.6, 127.8, 127.8, 128.4, 128.5, 130.9, 132.3, 138.3, 141.4; **HRMS (ESI)**

C$_{23}$H$_{32}$NaO$_2$Si requires 391.2064, found 391.2063 (0.3 ppm).

**(Z)-4-(Benzyloxy)-4-phenylbut-2-en-1-ol, 42**

Alcohol 41 (650 mg, 1.77 mmol) was subjected to general procedure N. Flash column chromatography on silica gel (85:5 petrol:ethyl acetate) afforded the title compound as a yellow oil (325 mg, 1.28 mmol, 71%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3386br s, 3062w, 3029m, 2866m, 1602w, 1494m, 1453s, 1388m, 1302w, 1205w, 1027s, 845w, 739s; $\delta$H (400 MHz, CDCl$_3$) 1.87 - 2.18 (1 H, br s), 4.17 (1 H, dd, J 13.5, 4.4), 4.30 (1 H, dd, J 13.5, 6.3), 4.51 (1 H, d, J 12.2), 4.54 (1 H, d, J 12.2), 5.20 (1 H, d, J 7.3), 5.46 - 5.99 (2 H, m), 7.11 - 7.62 (10 H, m); $\delta$C (100 MHz, CDCl$_3$) 58.8, 70.1, 76.5, 126.8, 127.8, 127.9, 127.9, 128.5, 128.7, 131.1, 132.7, 138.1, 141.1; **HRMS (ESI)** C$_{17}$H$_{18}$NaO$_2$ requires 277.1199, found 277.1198 (0.5 ppm).

**(Z)-4-(Benzyloxy)-4-phenylbut-2-enyl mesitoyloxy carbamate, 16c**

Alcohol 42 (310 mg, 1.22 mmol) was subjected to general procedure D. Flash column chromatography on silica gel (98:2 petrol:ethyl acetate) afforded the title compound as a colourless oil (345 mg, 750 µmol, 62%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3265br m, 2924m, 1753s, 1611m, 1453m, 1227s, 1158m, 1089m, 850w; $\delta$H (400 MHz, CDCl$_3$) 2.34 (3 H, s), 2.42 (6 H, s), 4.55 (1 H, d, J 12.0), 4.58 (1
H, d, J 12.0), 4.86 (1 H, dd, J 12.9, 6.3), 4.97 (1 H, dd, J 12.9, 7.3), 5.26 (1 H, d, J 9.0), 5.80 (1 H, ddd, J 10.9, 7.3, 6.3), 5.93 (1 H, dd, J 10.9, 9.0), 6.93 (2 H, s), 7.30 - 7.48 (10 H, m), 8.49 (1 H, s); δC (100 MHz, CDCl₃) 20.0, 21.3, 62.5, 70.2, 76.4, 125.0, 126.6, 126.8, 127.2, 127.7, 127.9, 127.9, 128.5, 128.7, 135.5, 136.7, 138.1, 140.7, 140.9, 156.5, 169.0; HRMS (ESI) C₂₈H₂₉NNaO₅ requires 482.1938, found 482.1935 (0.5 ppm).

Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one with (±) (R)-4-((1'R,2'R)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one, anti- and syn-19c

Benzoyloxycarbamate 16c (200 mg, 435 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 200 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 400 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated in vacuo to deliver the title compound as a colourless oil and a mixture of diastereomers (2:1, 120 mg, 383 µmol, 88%). The NMR data below are given for the major isomer.

$\nu_{\text{max}}$ (thin film)/cm⁻¹: 3385br s, 2875s, 2796s, 1737s, 1512m, 1430w, 1256w, 977s; δH (400 MHz, CDCl₃) 1.98 - 2.38 (1 H, br s), 3.77 (1 H, t, J 6.3), 3.95 (1 H, ddd, J 7.8, 6.3, 6.1), 4.22 (1 H, d, J 11.6), 4.30 (1 H, dd, J 9.3, 7.8), 4.26 - 4.31 (1 H, m), 4.40 (1 H, dd, J 9.3, 6.1), 4.51 (1 H, d, J 11.6), 5.68 (1 H, br s), 7.20 - 7.53 (10 H, m); δC (100
MHz, CDCl₃) 54.0, 66.2, 70.8, 74.6, 82.2, 127.5, 127.7, 128.1, 128.6, 128.8, 128.9, 137.4, 137.9, 160.5; HRMS (ESI) C₁₈H₁₉NNaO₄ requires 336.1206, found 336.1210 (–1.1 ppm).

(±) (R)-4-((4'R,5'S)-2,2-Dimethyl-5-phenyl-1,3-dioxolan-4-yl)oxazolidin-2-one with (±) (R)-4-((4'R,5'R)-2,2-dimethyl-5-phenyl-1,3-dioxolan-4-yl)oxazolidin-2-one, anti- and syn-20c

Carbamates anti- and syn-19c (13 mg, 42 µmol, 6:1 d.r) was subjected to general procedure F followed directly by general procedure I. Flash column chromatography on silica gel (98:2 petrol:ethyl acetate) afforded the title compound as a colourless oil (4 mg, 20 µmol, 36%) and a mixture of diastereomers (>5:1). The NMR data below are given for the major isomer.

νmax (thin film)/cm⁻¹ 3318br s, 2924s, 1748s, 1383s, 1213s, 1162s, 1041s, 879m, 702m; δH (400 MHz, CDCl₃) 1.49 (3 H, s, CH₃), 1.58 (3 H, s, CH₃), 3.68 (1 H, dt, J 9.2, 7.1, C(4)H), 4.25 (1 H, dd, J 9.2, 6.2, C(4')H), 4.33 (2 H, d, J 7.1, C(5)H₂), 5.40 (1 H, d, J 6.2, C(5')H₂), 7.33 - 7.50 (5 H, m, PhH ×5); δC (126 MHz, CDCl₃) 25.0 (CH₃), 27.6 (CH₃), 51.9 (C(4)), 67.7 (C(5)), 78.4 (C(5')), 80.5 (C(4')), 109.3 (C(2')), 126.4 (Ph), 129.2 (Ph), 129.3 (Ph), 134.6 (i-Ph), 158.7 (NC(O)O); HRMS (ESI) C₁₄H₁₇NNaO₄ requires 286.1050, found 286.1059 (–3.2 ppm).
Commercially available trimethylacetaldehyde (2.86 g, 33.3 mmol) was subjected to general procedure O. The crude product was subjected directly to general procedure A. Flash column chromatography on silica gel (4:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (2.16 g, 12.1 mmol, 36% over two steps).

ν_{max} (thin film)/cm\(^{-1}\) 3304s, 3065w, 3031w, 2957s, 2869s, 1497m, 1479m, 1455s, 1387s, 1364m, 1323m, 1196w, 1073s, 1028m, 988w, 935w, 876w, 736m, 697s, 656s; δ_H (400 MHz, CDCl\(_3\)) 1.03 (9 H, s), 2.46 (1 H, d, J 2.0), 3.70 (1 H, d, J 2.0), 4.50 (1 H, d, J 12.0), 4.88 (1 H, d, J 12.0), 7.25 - 7.43 (5 H, m); δ_C (100 MHz, CDCl\(_3\)) 25.7, 35.3, 70.9, 77.3, 80.9, 81.7, 127.5, 127.8, 128.2, 138.2; HRMS (FI) C\(_{14}\)H\(_{18}\)O requires 202.1358, found 202.1358 (0.2 ppm).

4-(Benzyloxy)-5,5-dimethylhex-2-yn-1-ol, 12d

Alkyne 43 (1.375 mg, 6.81 mmol) was subjected to general procedure B. Flash column chromatography on silica gel (94:6 petrol:ethyl acetate) afforded the title compound as a colourless oil (635 mg, 2.74 mmol, 56%), and 500 mg returned starting material.

ν_{max} (thin film)/cm\(^{-1}\) 3383br s, 3031m, 2959s, 2871s, 1721w, 1496m, 1454s, 1334s, 1207m, 1151s, 1069s, 735s, 698s; δ_H (400 MHz, CDCl\(_3\)) 1.03 (9 H, s), 1.98 - 2.25 (1
H, br s), 3.75 (1 H, s), 4.35 (2 H, s), 4.51 (1 H, d, J 12.1), 4.85 (1 H, d, J 12.1), 7.25 - 7.42 (5 H, m); \( \delta_C \) (100 MHz, CDCl\(_3\)) 25.9, 35.6, 51.1, 71.0, 77.7, 83.6, 84.8, 127.5, 127.8, 128.2, 138.3; HRMS (ESI) \( C_{15}H_{20}NaO_2 \) requires 255.1356, found 255.1355 (0.3 ppm).

(Z)-4-(Benzyloxy)-5,5-dimethylhex-2-en-1-ol, 15d

\[
\begin{align*}
\text{Alcohol 12d (169 mg, 728 µmol) was subjected to general procedure C. Flash column chromatography on silica gel (94:6 petrol:ethyl acetate) afforded the title compound as a colourless oil (168 mg, 718 µmol, 99%).}
\end{align*}
\]
\[
\nu_{\text{max}} \text{(thin film)}/\text{cm}^{-1} \quad 3362\text{br m}, 2957\text{s}, 1738\text{w}, 1455\text{m}, 1362\text{m}, 1260\text{m}, 1069\text{s}, 798\text{m}, 734\text{m}; \delta_H \text{(400 MHz, CDCl}_3) \quad 0.92 \text{ (9 H, s), 1.60 (1 H, br s), 3.68 (1 H, d, J 10.2), 4.07 (1 H, dd, J 13.1, 5.6), 4.22 (1 H, dd, J 13.1, 7.8), 4.34 (1 H, d, J 12.2), 4.61 (1 H, d, J 12.2), 5.51 (1 H, app t, J 10.2), 5.82 - 5.95 (1 H, m), 7.23 - 7.44 (5 H, m); } \delta_C \text{(100 MHz, CDCl}_3) \quad 26.0, 35.2, 58.9, 70.4, 81.3, 127.4, 127.6, 128.2, 130.3, 132.9, 139.0; \text{ HRMS (ESI) } C_{15}H_{22}NaO_2 \text{ requires } 257.1512, \text{ found } 257.1513 (-0.6 ppm).}
\]

(Z)-4-(Benzyloxy)-5,5-dimethylhex-2-ene mesitoxyloxy carbamate, 16d

\[
\begin{align*}
\text{Alcohol 15d (225 mg, 962 µmol) was subjected to general procedure D. Flash column chromatography on silica gel (98:2 petrol:ethyl acetate) afforded the title compound as a colourless oil (245 mg, 557 µmol, 58%).}
\end{align*}
\]
\( v_{\text{max}} \) (thin film)/cm\(^{-1}\) 3276br m, 2956s, 1753s, 1611m, 1454m, 1384m, 1304m, 1226w, 1158s, 1092s, 851m, 736m, 699m; \( \delta_H \) (400 MHz, CDCl\(_3\)) 0.88 - 1.00 (9 H, m), 2.32 (3 H, s, ArCH\(_3\)), 2.36 - 2.47 (6 H, m), 3.72 (1 H, d, J 9.7), 4.32 (1 H, d, J 12.1), 4.61 (1 H, d, J 12.1), 4.68 (1 H, ddd, J 13.0, 5.6, 1.4), 4.86 (1 H, ddd, J 13.0, 7.7, 1.4), 5.67 (1 H, ddd, J 11.4, 9.7, 1.4), 5.89 (1 H, ddd, J 11.4, 7.7, 5.6), 6.90 (2 H, s), 7.19 - 7.47 (5 H, m), 8.32 (1 H, br s); \( \delta_C \) (100 MHz, CDCl\(_3\)) 19.9, 21.2, 26.0, 35.3, 62.6, 70.5, 81.4, 126.5, 127.1, 127.4, 127.6, 128.2, 128.7, 133.5, 136.7, 138.8, 140.9, 156.4, 169.0; HRMS (ESI) \( C_{26}H_{33}NNaO_5 \) requires 462.2251, found 462.2256 (–1.0 ppm).

\((\pm) \ (R)-4-((1'R,2'S)-2'-(Benzyloxy)-1'-hydroxy-3',3'-dimethylbutyl)oxazolidin-2-one \) (major), with \((\pm) \ (S)-4-((1'S,2'S)-2'-(Benzyloxy)-1'-hydroxy-3',3'-dimethylbutyl)oxazolidin-2-one \) (minor), \( \text{anti- and syn-19d} \)

Benzoyloxy carbamate 18d (50 mg, 110 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 50 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 100 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated \textit{in vacuo} to deliver the title compound as a colourless oil and a mixture of diastereomers (6:1, 32 mg, 110 µmol, 95%). Flash column chromatography on silica gel (3:1 to 1:1 petrol:ethyl acetate) allowed isolation of the major (20 mg) diastereomer. The NMR data below are given for the major isomer.
$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3383br m, 2956m, 1742s, 1396m, 1246m; $\delta_H$ (400 MHz, CD$_3$OD) 1.02 (9 H, s), 3.24 (1 H, d, $J$ 3.4), 3.89 (1 H, t, $J$ 3.6), 4.12 (1 H, ddd, $J$ 9.1, 6.1, 3.5), 4.28 (1 H, t, $J$ 9.1), 4.51 (1 H, dd, $J$ 9.1, 6.1), 4.63 (1 H, d, $J$ 11.1), 4.66 (1 H, d, $J$ 11.1), 7.23 - 7.45 (5 H, m); $\delta_C$ (100 MHz, CD$_3$OD) 26.1, 35.7, 55.1, 66.8, 72.5, 76.2, 91.0, 127.7, 127.8, 128.4, 138.7, 161.5; HRMS (ESI) $\text{C}_{16}\text{H}_{23}\text{NNaO}_4$ requires 316.1526, found 316.1519 (−2.2 ppm).

(±) (R)-4-((1'R,2'S)-(1,2-dihydroxy-3,3-dimethylbutyl)oxazolidin-2-one, anti-44

Carbamate anti-19d (20 mg, 68 µmol) was subjected to general procedure F to afford the title compound as a colourless oil (12 mg, 59 µmol, 86%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3462br s, 2975s, 1734s, 1501m, 1435s, 1328m, 1041m; $\delta_H$ (400 MHz, D$_2$O) 0.83 (9 H, s), 3.19 (1 H, d, $J$ 6.1), 3.78 (1 H, dd, $J$ 6.1, 2.8), 4.13 (1 H, ddd, $J$ 9.1, 5.6, 2.8), 4.39 (1 H, dd, $J$ 9.1, 5.6), 4.43 (1 H, app t, $J$ 9.1); $\delta_C$ (100 MHz, D$_2$O) 25.7, 34.6, 55.2, 67.0, 71.6, 80.3, 162.7; HRMS (ESI) $\text{C}_{9}\text{H}_{17}\text{NNaO}_4$ requires 226.1050, found 226.1056 (−2.5 ppm).
(±) (4R,8R,9S)-8-(tert-butyl)-9-hydroxy-6,6-dimethyltetrahydrooxazolo-oxazin-2-one, anti-20d

Diol anti-44 (12 mg, 59 µmol) was subjected to general procedure H. Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (11 mg, 45 µmol, 76%).

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$ 3245br m, 2955m, 1741s, 1253m, 1049m; $\delta_H$ (400 MHz, CDCl$_3$) 1.01 (9 H, s, C(CH$_3$)$_3$), 1.49 (3 H, s, CH$_3$), 1.77 (3 H, s, CH$_3$), 3.25 (1 H, d, $J$ 9.3, C(8)H), 3.47 - 3.60 (1 H, m, C(9)H), 3.79 (1 H, dt, $J$ 9.3, 8.0, C(4)H), 4.07 (1 H, app t, $J$ 8.0, C(5)H), 4.46 (1 H, app t, $J$ 8.0, C(5)H); $\delta_C$ (126 MHz, CDCl$_3$) 21.3 (CH$_3$), 26.4 (CH$_3$), 26.8 (C(CH$_3$)$_3$), 34.1 (C(CH$_3$)$_3$), 56.9 (C(4)), 66.4 (C(5)), 70.3 (C(9)), 79.6 (C(8)), 84.9 (C(6)), 154.6 (NC(O)O); HRMS (ESI) C$_{12}$H$_{21}$NNaO$_4$ requires 266.1363, found 266.1368 (−2.1 ppm).

Ethyl 4-(benzyloxy)hept-2-ynoate, 21

$n$-BuLi (1.6 M solution in hexanes, 1.11 mL, 1.76 mmol) was added dropwise to a stirred solution of alkyne 5a (300 mg, 1.48 mmol) in THF (11 mL) at −78 °C under argon. After 1 hour, ethylchloroformate (0.420 mL, 4.41 mmol) was added dropwise, and the resultant mixture was allowed to warm to room temperature over 4 h, before being quenched by addition of a saturated aqueous solution of NH$_4$Cl (11 mL). The organic phase was separated, and the aqueous phase was extracted with ethyl acetate.
(11 mL ×3). The combined organic layers were dried over MgSO₄, filtered, and the solvents were removed in vacuo to afford the crude product. Flash column chromatography (99:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (351 mg, 1.35 mmol, 87%).

ν_max (thin film)/cm⁻¹ 2962s, 2873s, 2235m, 1714m, 1496m, 1454m, 1367m, 1248s, 1074m, 861w, 751m, 699m; δ_H (400 MHz, CDCl₃) 0.94 (3 H, t, J 7.8), 1.34 (3 H, t, J 7.2), 1.44 - 1.60 (2 H, m), 1.70 - 1.90 (2 H, m), 4.22 (1 H, t, J 6.6), 4.27 (2 H, q, J 7.2), 4.51 (1 H, d, J 11.6), 4.82 (1 H, d, J 11.6), 7.28 - 7.44 (5 H, m); δ_C (100 MHz, CDCl₃) 13.7, 14.0, 18.4, 37.0, 62.1, 68.1, 71.1, 77.7, 86.2, 127.9, 128.0, 128.4, 137.4, 153.3; HRMS (ESI) C₁₁H₂₀NaO₃ requires 283.1310, found 283.1309 (–1.4 ppm).

(Z)-Ethyl 4-(benzyloxy)-3-methylhept-2-enoate, 22-Z

Methyllithium (1.5 M solution in diethyl ether, 4.65 mL, 6.98 mmol) was added dropwise to a stirred suspension of copper (I) iodide (710 mg, 3.84 mmol) in THF (21 mL) at 0 °C under argon. The resultant solution was stirred for one hour, cooled to –20 °C and added dropwise via cannula to a stirred solution of ester 21 (970 mg, 3.49 mmol) in THF (21 mL). The reaction was stirred for 2 hours before being cooled to –78 °C, quenched with ethanol (6 mL) followed by a saturated aqueous solution of NH₄Cl (21 mL). The organic phase was separated, and the aqueous phase was extracted with ethyl acetate (50 mL ×3). The combined organic layers were dried over MgSO₄, filtered and the resultant mixture was concentrated in vacuo to afford the crude product as a single isomer. Flash column chromatography (99:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (691 mg, 2.50 mmol, 66%).
ν_max (thin film)/cm⁻¹ 3406br m, 2962s, 1726s, 1455m, 1217s, 1095s, 756m, 697m; δ_H (400 MHz, CDCl₃) 0.93 (3 H, t, J 7.2), 1.28 (3 H, t, J 7.1), 1.31 - 1.59 (3 H, m), 1.65 - 1.78 (1 H, m, C(6)H), 1.91 (3 H, s), 4.15 (2 H, q, J 7.1), 4.34 (1 H, d, J 11.9), 4.44 (1 H, J 11.9), 5.30 (1 H, dd, J 8.3, 4.5), 5.83 (1 H, s), 7.23 - 7.38 (5 H, m); δ_C (100 MHz, CDCl₃) 14.0, 14.3, 18.6, 19.1, 36.2, 59.9, 71.3, 76.3, 118.8, 127.5, 127.8, 128.3, 138.6, 160.0, 165.9; HRMS (ESI) C₁₇H₂₄NaO₃ requires 299.1622, found 299.1618 (–1.4 ppm).

(Z)-4-(Benzyloxy)-3-methylhept-2-en-1-ol, 23-Z

Ester 22-Z (130 mg, 471 µmol) was subjected to general procedure Q. Flash column chromatography on silica gel (91:9 petrol:ethyl acetate) afforded the title compound as a colourless oil (102 mg, 436 µmol, 94%).

ν_max (thin film)/cm⁻¹ 3383br m, 2958s, 2870m, 1668w, 1496w, 1454s, 1380m, 1069s, 1027s, 803w, 734s; δ_H (400 MHz, CDCl₃) 0.90 (3 H, t, J 6.8), 1.13 - 1.61 (4 H, m), 1.67 (1 H, br s), 1.75 (3 H, s), 4.03 (1 H, dd, J 12.9, 6.0), 4.10 - 4.23 (2 H, m), 4.26 (1 H, d, J 11.9), 4.52 (1 H, d, J 11.9), 5.64 (1 H, t, J 6.0), 7.20 - 7.50 (5 H, m); δ_C (100 MHz, CDCl₃) 10.0, 13.0, 18.1, 34.8, 58.0, 69.0, 83.3, 126.3, 126.4, 126.7, 127.3, 137.0, 137.7; HRMS (ESI) C₁₅H₂₂NaO₂ requires 257.1511, found 257.1512 (0.5 ppm).
(Z)-4-(Benzyloxy)-3-methylhept-2-enyl mesitoxyloxy carbamate, 24-Z

Alcohol 23-Z (255 mg, 1.09 mmol) was subjected to general procedure D. Flash column chromatography on silica gel (98:2 petrol:ethyl acetate) afforded the title compound as a colourless oil (339 mg, 772 µmol, 71%).

ν_max (thin film)/cm⁻¹ 3322br s, 2972s, 1753s, 1454s, 1227s, 1158m, 1093s; δ_H (400 MHz, CDCl₃) 0.91 (3 H, t, J 7.1), 1.20 - 1.52 (3 H, m), 1.68 - 1.77 (1 H, m), 1.79 (3 H, s), 2.31 (3 H, s), 2.38 (6 H, s), 4.22 (1 H, dd, J 7.7, 5.7), 4.27 (1 H, d, J 11.9), 4.50 (1 H, d, J 11.9), 4.68 (1 H, dd, J 12.4, 6.1), 4.82 (1 H, dd, J 12.4, 8.1), 5.63 (1 H, dd, J 8.1, 6.1), 6.90 (2 H, s), 7.24 - 7.42 (5 H, m), 8.35 (1 H, br s); δ_C (100 MHz, CDCl₃) 14.0, 17.9, 19.0, 19.9, 21.2, 35.9, 62.1, 70.2, 76.1, 122.1, 126.5, 127.6, 127.8, 128.4, 128.7, 136.7, 138.5, 140.9, 142.7, 156.5, 169.0; HRMS (ESI) C₂₆H₃₃NNaO₅ requires 462.2256, found 462.2260 (–2.0 ppm).

Diastereomeric mixture of (±) (R)-4-((2'R,3'R)-(3'-(benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one (major diastereomer) with (±) (R)-4-((2'R,3'S)-(3'-(benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one (minor diastereomer), anti- and syn-25-Z

Benzoyloxy carbamate 24-Z (320 mg, 739 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the
carboxylic acid byproduct was removed by washing with 170 mL of petrol:ethyl acetate:acetic acid (94:5:1). The silica was then washed with 340 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated in vacuo to deliver the title compound as a colourless oil and a mixture of diastereomers (2:1, 181 mg, 618 µmol, 85%). Flash column chromatography on silica gel (3:1 to 1:1 petrol:ethyl acetate) allowed isolation of the major (88 mg) and minor (14 mg) diastereomers.

**Major diastereomer;** \( \nu_{\text{max}} \) (thin film)/cm\(^{-1} \) 3415br s, 2958s, 1747s, 1454s, 1410m, 1248m, 1108m, 942w; \( \delta_H \) (400 MHz, CD\(_3\)OD) 0.96 (3 H, t, \( J 7.1 \)), 1.18 (3 H, s), 1.28 - 1.76 (4 H, m), 3.27 - 3.43 (1 H, m), 3.98 (1 H, dd, \( J 9.1, 6.1 \)), 4.22 (1 H, dd, \( J 9.1, 8.8 \)), 4.51 (1 H, dd), 4.63 (2 H, m), 7.25 - 7.43 (5 H, m); \( \delta_C \) (100 MHz, CD\(_3\)OD) 14.8, 20.0, 21.7, 34.2, 59.3, 67.6, 75.2, 75.6, 85.2, 128.8, 129.2, 129.5, 139.9, 162.8; HRMS (ESI) \( \text{C}_{16}\text{H}_{23}\text{NNaO}_4 \) requires 316.1516, found 316.1519 (1.0 ppm). **Minor diastereomer;** \( \nu_{\text{max}} \) (thin film)/cm\(^{-1} \) 3414br s, 2959s, 2872s, 1748s, 1454m, 1409s, 1248s, 1108s, 986s, 942m, 736s; \( \delta_H \) (400 MHz, CD\(_3\)OD) 0.94 - 1.00 (3 H, \( J 7.3 \)), 1.18 (3 H, s), 1.33 - 1.72 (4 H, m), 3.35 (1 H, dd, \( J 7.8, 3.0 \)), 4.08 (1 H, dd, \( J 9.1, 5.3 \)), 4.36 (1 H, app t, \( J 9.1 \)), 4.51 (1 H, dd, \( J 9.1, 5.3 \)), 4.64 (1 H, d, \( J 11.8 \)), 4.67 (1 H, d, \( J 11.8 \)), 7.23 - 7.43 (5 H, m); \( \delta_C \) (100 MHz, CD\(_3\)OD) 13.7, 18.6, 20.2, 32.6, 57.0, 66.7, 74.3, 75.3, 84.0, 127.7, 128.0, 128.4, 138.9, 161.6; HRMS (ESI) \( \text{C}_{16}\text{H}_{23}\text{NNaO}_4 \) requires 316.1516, found 316.1519 (1.0 ppm).
(±) \((R)-4-((2'R,3'R)-(2',3'-\text{Dihydroxyhexan-2-yl})\text{oxazolidin-2-one and (S)-4-}\
(2'S,3'S)-(2',3'-\text{dihydroxyhexan-2-yl})\text{oxazolidin-2-one, }\text{anti-51-Z}\)

Carbamate \text{anti-25-Z} (16 \text{ mg, 55 µmol}) was subjected to general procedure F, to afford
the title compound as a colourless oil (10 mg, 49 µmol, 91%).

\(\nu_{\text{max}} \text{(thin film)/cm}^{-1} 3386\text{br s, 2958s, 1739s, 1415m, 1252m, 1110m; }\delta_{\text{H}} \text{(400 MHz, D}_2\text{O)} 0.72 - 0.86 (3 \text{ H, m), 1.07 (3 H, s), 1.13 - 1.32 (2 H, m), 1.36 - 1.52 (2 H, m),}
3.32 (1 \text{ H, t, } J 9.6), 3.96 - 4.10 (1 \text{ H, m), 4.27 - 4.51 (2 H, m); }\delta_{\text{C}} \text{(126 MHz, D}_2\text{O)}
13.1, 15.0, 18.9, 31.9, 58.0, 67.0, 74.2, 74.5, 162.2; \text{HRMS (ESI) C}_{9}\text{H}_{17}\text{NNaO}_4
\text{requires 226.1050, found 226.1056 (–2.7 ppm).}

(±) \((R)-4-((4'R,5'R)-(2',2',4'-\text{Trimethyl-5'-propyl-1,3-dioxolan-4-yl})\text{oxazolidin-2-}
one, \text{anti-26-Z}\)

Diol \text{anti-51-Z} (10 \text{ mg, 49 µmol}) was subjected to general procedure G. Flash column
chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as
a yellow oil (8 mg, 34 µmol, 66%).

\(\nu_{\text{max}} \text{(thin film)/cm}^{-1} 3323\text{br m, 2962s, 1755s, 1380w, 1260m, 1101m, 800w; }\delta_{\text{H}} \text{(400 MHz, C}_6\text{D}_6) 1.12 (3 \text{ H, t, } J 7.1, C(8')H), 1.18 (3 \text{ H, s, C(4')CH}_3), 1.23 (3 \text{ H, s,}
C(2')CH}_3), 1.25 (3 \text{ H, s, C(2')CH}_3), 1.31 - 1.57 (3 \text{ H, m, C(6')H and C(7')H}_2), 1.66 -
1.81 (1 \text{ H, m, C(6')H), 3.50 (1 \text{ H, dd, } J 9.2, 5.3, C(4)H), 3.63 - 3.70 (1 \text{ H, m, C(5')H),}

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3.93 (1 H, app t, J 9.2, C(5)H), 4.40 (1 H, dd, J 9.2, 5.3, C(5)H), 7.62 (1 H, s, NH); δC (100 MHz, C₆D₆) 14.1 (C(8')), 18.8 (C(4')CH₃), 20.9 (C(7')), 26.1 (C(2')CH₃), 28.1 (C(2')CH₃), 30.4 (C(6')), 54.3 (C(4)), 66.6 (C(5)), 82.4 (C(4')), 83.2 (C(5')), 107.2 (C(2')), 161.5 (NC(O)O); HRMS (ESI) C₁₂H₂₁NNaO₄ requires 266.1363, found 266.1366 (–1.3 ppm).

(±) (R)-4-((2'R,3'S)-(2',3'-Dihydroxyhexan-2-yl)oxazolidin-2-one, syn-51-Z

![Chemical structure](image)

Carbamate synb-25-Z (14 mg, 48 µmol) was subjected to general procedure F, to afford the title compound as a colourless oil (8 mg, 40 µmol, 81%).

νmax (thin film)/cm⁻¹ 3387br s, 2959s, 1739s, 1416m, 1253m, 1110m, 942w; δH (400 MHz, D₂O) 0.81 (3 H, t, J 7.1), 1.05 (3 H, s), 1.14 - 1.53 (4 H, m), 3.36 (1 H, dd, J 10.1, 2.0), 4.06 (1 H, dd, J 8.5, 6.2), 4.36 - 4.47 (2 H, m); δC (126.0 MHz, D₂O) 13.4, 18.2, 19.2, 32.5, 57.5, 67.2, 74.4, 75.6, 162.6; HRMS (ESI) C₉H₁₇NNaO₄ requires 226.1050, found 226.1051 (–0.4 ppm).

(±) (R)-4-((4'R,5'S)-(2',2',4'-Trimethyl-5'-propyl-1,3-dioxolan-4-yl)oxazolidin-2-one, syn-26-Z

![Chemical structure](image)

Diol syn-51-Z (8 mg, 40 µmol) was subjected to general procedure G. Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as
a colourless oil (5 mg, 20 µmol, 53%).

νmax (thin film)/cm⁻¹ 3324 br m, 2963 s, 1756 s, 1380 w, 1260 m, 1106 m, 799 w; δH (400 MHz, C₆D₆) 1.04 - 1.12 (6 H, m, C(4’)CH₃ and C(8’)H₂), 1.24 (3 H, s, C(2’)CH₃), 1.35 - 1.60 (3 H, m, C(6’)H and C(7’)H₂), 1.40 (3 H, m, C(2’)CH₃), 1.63 - 1.82 (1 H, m, C(6’)H), 3.27 (1 H, dd, J 9.0, 5.3, C(4)H), 3.67 (1 H, dd, J 9.7, 2.4, C(5’)H), 3.87 (1 H, app t, J 9.0, C(5)H), 4.19 (1 H, dd, J 9.0, 5.3, C(5)H), 7.69 (1 H, br s, NH); δC (100 MHz, C₆D₆) 14.2 (C(8’)), 16.5 (C(4’)CH₃), 20.7 (C(7’)), 26.8 (C(2’)CH₃), 28.7 (C(2’)CH₃), 32.3 (C(6’)), 58.3 (C(4’)), 66.3 (C(5’)), 81.1 (C(5’)), 82.3 (C(4’)), 107.5 (C(2’)), 161.0 (NC(O)O); HRMS (ESI) C₁₂H₂₁NNaO₄ requires 266.1363, found 266.1376 (–4.9 ppm).

(E)-Ethyl 4-(benzyloxy)-3-methylhept-2-enoate, 22-E

Methyllithium (1.5 M solution in diethyl ether, 3.49 mL, 5.24 mmol) was added dropwise to a stirred suspension of copper (I) iodide (501 mg, 2.88 mmol) in THF (15 mL) at 0 °C under argon. The resultant solution was stirred for one hour, cooled to –20 °C and added dropwise via cannula to a stirred solution of ester 5a (680 mg, 2.62 mmol) in THF (15 mL). The reaction was allowed to warm to room temperature overnight before being heated at 40 °C for one hour, quenched with ethanol (5 mL) and extracted with a saturated aqueous solution of NH₄Cl (15 mL). The organic phase was separated, and the aqueous phase was extracted with ethyl acetate (30 mL ×3). The combined organic layers were dried over MgSO₄ and the solvents were removed in vacuo to afford the crude product as a mixture of isomers (E:Z 4.2:1). Flash column chromatography (99:1 petrol:ethyl acetate) afforded the title compound as a colourless
oil (470 mg, 1.70 mmol, 64%).

\( \nu_{\text{max}} \text{ (thin film)/cm}^{-1} \) 3387br m, 2963s, 1455m, 1218m, 1095m, 756w, 697w;

\( \delta_H \) (400 MHz, CDCl\(_3\)) 0.89 (3 H, t, J 6.8), 1.31 (3 H, t, J 7.2), 1.36 - 1.57 (3 H, m), 1.57 - 1.72 (1 H, m), 2.14 (3 H, s), 3.74 (1 H, dd, J 7.3, 5.3), 4.19 (2 H, q, J 7.2), 4.26 (1 H, d, J 11.9), 4.51 (1 H, d, J 11.9), 5.87 (1 H, s), 7.24 - 7.44 (5 H, m); \( \delta_C \) (100 MHz, CDCl\(_3\)) 13.9, 14.0, 14.3, 18.9, 36.0, 59.8, 70.7, 83.9, 117.1, 127.6, 127.7, 128.4, 138.2, 158.4, 166.5; HRMS (ESI) \( \text{C}_{17}\text{H}_{24}\text{NaO}_3 \) requires 299.1623, found 299.1617 (0.1 ppm).

(E)-4-(Benzyloxy)-3-methylhept-2-en-1-ol, 23-\( E \)

\[
\begin{align*}
\text{HO-} & \quad \text{O-} \quad \text{Ph} \\
\end{align*}
\]

Ester 23-\( E \) (180 mg, 652 \( \mu \)mol) was subjected to general procedure Q. Flash column chromatography on silica gel (91:9 petrol:ethyl acetate) afforded the title compound as a colourless oil (102 mg, 436 \( \mu \)mol, 68%).

\( \nu_{\text{max}} \text{ (thin film)/cm}^{-1} \) 3387br s, 2959s, 1454m, 1261m, 1069s, 801w, 734m, 698s; \( \delta_H \) (400 MHz, CDCl\(_3\)) 0.90 (3 H, t, J 7.3), 1.19 - 1.34 (2 H, m), 1.34 - 1.55 (3 H, m), 1.64 (3 H, s), 3.69 (1 H, t, J 6.7), 4.23 (1 H, dd, J 12.6, 6.6), 4.25 - 4.32 (2 H, m), 4.48 (1 H, d, J 11.9), 5.60 (1 H, t, J 6.4), 7.19 - 7.44 (5 H, m); \( \delta_C \) (100 MHz, CDCl\(_3\)) 11.0, 14.0, 19.1, 35.9, 59.1, 70.0, 84.3, 127.1, 127.4, 127.7, 128.3, 138.3, 138.8; HRMS (ESI) \( \text{C}_{15}\text{H}_{22}\text{NaO}_2 \) requires 257.1517, found 257.1512 (1.9 ppm).
(E)-4-(Benzyloxy)-3-methylhept-2-enyl mesitoxyloxy carbamate, 24-E

Alcohol 23-E (96 mg, 410 µmol) was subjected to general procedure D. Flash column chromatography on silica gel (91:9 petrol:ethyl acetate) afforded the title compound as a colourless oil (85 mg, 190 µmol, 47%).

νmax (thin film)/cm⁻¹ 3267br m, 2958s, 2870s, 1753s, 1454s, 1381m, 1311m, 1227s, 1158m, 1089s, 851m, 735s; δH (400 MHz, CDCl₃) 0.90 (3 H, t, J 7.3), 1.18 - 1.53 (3 H, m), 1.61 - 1.72 (1 H, m), 1.73 (3 H, s), 2.31 (3 H, s), 2.38 (6 H, s), 3.71 (1 H, dd, J 7.0, 6.1), 4.25 (1 H, d, J 11.9), 4.49 (1 H, d, J 11.9), 4.78 - 4.85 (1 H, m, J 12.4, 6.8), 4.88 (1 H, dd, J 12.4, 6.8), 5.60 (1 H, t, J 6.8), 6.89 (2 H, s), 7.25 - 7.38 (5 H, m), 8.41 (1 H, br s); δC (100 MHz, CDCl₃) 11.3, 14.0, 19.0, 19.9, 21.2, 35.8, 63.0, 70.1, 83.9, 121.0, 126.5, 127.4, 127.8, 128.3, 128.7, 136.7, 138.6, 140.9, 142.2, 156.7, 169.0; HRMS (ESI) C₂₆H₃₃NNaO₅ requires 462.2256, found 462.2251 (–1.1 ppm).

Diastereomeric mixture of (±) (R)-4-((2'S,3'R)-(3'-(benzyloxy)-2'-hydroxy hexan-2-yl)oxazolidin-2-one (major) with (±) (R)-4-((2'S,3'S)-(3'-(benzyloxy)-2'-hydroxy hexan-2-yl)oxazolidin-2-one (minor), anti and syn-25-E

Benzoyloxy carbamate 24-E (180 mg, 410 µmol) was subjected to general procedure E. The crude reaction mixture was dry loaded onto a thin pad of silica, and the carboxylic acid byproduct was removed by washing with 250 mL of petrol:ethyl
acetate:acetic acid (94:5:1). The silica was then washed with 500 mL of petrol:ethyl acetate (1:3), and the resultant filtrate was concentrated in vacuo to deliver the title compound as a yellow oil and a mixture of diastereomers (4:1, 97 mg, 330 µmol, 81%). The NMR data below are given for the major isomer.

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$: 3386br s, 3031w, 2958s, 2468w, 1746s, 1454m, 1408m, 1243m, 1093m, 698m; $\delta_H$ (400 MHz, CD$_3$OD) 0.99 (3 H, t, $J$ 7.1), 1.07 (3 H, s), 1.39 - 1.83 (4 H, m), 3.41 (1 H, dd, $J$ 7.8, 3.3), 4.08 (1 H, dd, $J$ 9.1, 7.1), 4.33 - 4.40 (2 H, m), 4.63 (1 H, m, $J$ 11.0), 4.66 (1 H, d, $J$ 11.0), 7.25 - 7.41 (5 H, m); $\delta_C$ (100 MHz, CDCl$_3$) 14.8, 17.6, 21.8, 34.0, 59.6, 67.7, 75.0, 75.2, 85.4, 128.7, 128.8, 129.5, 140.0, 162.7; HRMS (ESI) C$_{16}$H$_{23}$NNaO$_4$ requires 316.1516, found 316.1519 (0.9 ppm).

Diastereomeric mixture of (±) ($R$)-4-((2'S,3'R)-(2',3'-dihydroxyhexan-2-yl)oxazolidin-2-one (major) with (±) ($R$)-4-((2'S,3'S)-(2',3'-dihydroxyhexan-2-yl)oxazolidin-2-one (minor), anti- and syn-51-Z

Alcohols anti and syn-25-E (16 mg, 54 µmol) was subjected to general procedure F, to afford a mixture of the title compounds as a yellow oil (11 mg, 54 µmol, 99%). The NMR data below are given for the major isomer.

$\nu_{\text{max}}$ (thin film)/cm$^{-1}$: 3387br s, 2958s, 1738m, 1416m, 1252m, 1110m; $\delta_H$ (400 MHz, D$_2$O) 0.84 (3 H, t, $J$ 7.1), 0.96 (3 H, s), 1.23 - 1.33 (2 H, m), 1.37 - 1.63 (2 H, m), 3.37 - 3.44 (1 H, m), 4.08 (1 H, dd, $J$ 9.3, 6.3), 4.35 (1 H, dd, $J$ 9.3, 6.3), 4.46 (1 H, app t, $J$ 9.3); $\delta_C$ (126.0 MHz, D$_2$O) 13.1, 16.7, 17.8, 32.1, 58.1, 66.6, 74.0, 74.4, 162.3; HRMS (ESI) C$_{9}$H$_{17}$NNaO$_4$ requires 226.1050, found 226.1053 (–1.3 ppm).
Diastereomeric mixture of (±) \((R)-4-((4'S,5'R)-(2',2',4'-\text{trimethyl}-5'-\text{propyl}-1,3-dioxolan-4-yl)\text{oxazolidin-2-one}}\) (major) with (±) \((R)-4-((4'S,5'S)-(2',2',4'-\text{trimethyl}-5'-\text{propyl}-1,3-dioxolan-4-yl)\text{oxazolidin-2-one}}\) (minor), \textit{anti} and \textit{syn-26-E}

Diols \textit{anti} and \textit{syn-51-E} (12 mg, 59 µmol) was subjected to general procedure G. Flash column chromatography on silica gel (2:1 petrol:ethyl acetate) afforded the title compound as a colourless oil (8 mg, 33 µmol, 56%) and a mixture of diastereomers (10:1). The NMR data below are given for the major isomer.

\[\nu_{\text{max}} (\text{thin film})/\text{cm}^{-1} \] 3299br m, 2962s, 1756s, 1380w, 1260s, 1106s, 924m, 800w; \[\delta_{\text{H}} (400 \text{ MHz, } C_6D_6) \] 0.76 - 0.80 (3 H, s, C(4')CH₃), 0.86 - 1.32 (3 H, m, C(6')H and C(7')H), 0.95 (3 H, t, \(J\) 7.3, C(8')H₃), 1.26 (3 H, s, C(2')CH₃), 1.51 (3 H, s, C(2')CH₃), 1.54 - 1.69 (1 H, m, C(6')H), 3.23 (1 H, dd, \(J\) 8.7, 6.3, C(4)H), 3.58 (1 H, dd, \(J\) 10.5, 2.9, C(5')H), 3.73 (1 H, app t, \(J\) 8.7, C(5)H), 3.88 (1 H, dd, \(J\) 8.7, 6.3, C(5)H), 6.10 (1 H, br s, NH); \[\delta_{\text{C}} (100 \text{ MHz, } C_6D_6) \] 14.0 (C(8')), 19.1 (C(7')), 20.7 (C(4')CH₃), 26.2 (C(2')CH₃), 27.6 (C(2')CH₃), 30.4 (C(6')), 55.6 (C(4)), 65.8 (C(5)), 81.5 (C(4')), 83.0 (C(5')), 107.9 (C(2')), 159.5 (NC(O)O); \textbf{HRMS (ESI)} \(C_{12}H_{21}N\text{NaO}_4\) requires 266.1363, found 266.1370 (–2.7 ppm).
4. $^{13}$C and $^1$H NMR Spectra of selected compounds

(Z)-4-(Benzyloxy)hept-2-enyl mesitoyloxycarbamate, 8a

[Diagram of the compound]
(±) (R)-4-((1'R,2'S)-2'((benzyloxy)-1'-hydroxypentyl)oxazolidin-2-one (major)

anti-9a
(R)-4-((1'R,2'R)-2'((benzyloxy)-1'-hydroxypentyl)oxazolidin-2-one (minor) syn-9a
(Z)-4-(4'-Methoxybenzyloxy)hept-2-enyl mesityloxycarbamate 8b
Diastereomeric mixture of (±) \((R)-4-((1'R,2'S)-(2''-(4''-methoxybenzyl)-1'-'hydroxypentyl)oxazolidin-2-one\) (major isomer) with (±) \((R)-4-((1'R,2'R)-(2''-(4''-methoxybenzyl)-1'-'hydroxypentyl))oxazolidin-2-one\) (minor isomer) \textit{anti}-9b and \textit{syn} -9b
(Z)-4-(tert-Butyldimethylsilyloxy)hept-2-enyl mesityloxycarbamate, 9c
Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-2'-(((tert-butylimethylsilyl)oxy)-1'-hydroxypentyl)oxazolidin-2-one (major) anti-9c
(±) (R)-4-((1'R,2'R)-2'-(~tert~butyldimethylsilyloxy)-1'-hydroxypentyl)oxazolidin-2-one (minor) syn-9c
(Z)-1-(Mesitoxy carbamoyloxy) hept-2-en-4-yl acetate 8e
(Z)-4-tert-Butoxyhept-2-enyl mesitoxyoxycarbamate 8d
Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-(2'-(tert-butoxy)-1'-hydroxypentyl)oxazolidin-2-one (major isomer) with (±) (R)-4-((1'R,2'R)-(2'-(tert-butoxy)-1'-hydroxypentyl)oxazolidin-2-one (minor isomer), anti-9d and ayn-9d
Diastereomeric mixture of (±) \((R)-4-((1'\,R,2'S)-2'-acetate-1'-hydroxypentyl oxazolidin-2-one\ (major\ isomer)\) with (±) \((R)-4-((1'\,R,2'R)-2'-acetate-1'-hydroxypentyl oxazolidin-2-one\ (minor\ isomer)\) \textit{anti-9e and syn-9e}

\[\text{major} \quad \text{minor}\]

Chemical Shift (ppm)
(Z)-1-(Mesityloxy carbamoyloxyl) hept-2-en-4-yl mesitoate 8f
Diastereomeric mixture of (±) \((R)-4-((1'R,2'S)-2'\text{-mesitoate-1'}\text{-hydroxypentyl oxazolidin-2-one (major isomer) with (±) \((R)-4-((1'R,2'R)-2'\text{-mesitoate-1'}\text{-hydroxypentyl oxazolidin-2-one (minor isomer), ant}^{-9f} \text{ and syn}^{-9f}\)
(Z)-4-Methoxyhept-2-enyl mesityloxycarbamate 8g
Diastereomeric mixture of (±) \((R)-4-((1'R,2'S)-2'-\text{methoxy}-1'-\text{hydroxypentyl oxazolidin}-2\text{-one}\) (major isomer) with (±) \((R)-4-((1'R,2'R)-2'-\text{methoxy}-1'-\text{hydroxypentyl oxazolidin}-2\text{-one}\) (minor isomer), \text{anti-9g} and \text{syn-9g}
(Z)-4-Hydroxyhept-2-enyl mesitoyloxycarbamate 8h
(±) (R)-4-((1'R,2'S)-1',2'-dihydroxypentyl)oxazolidin-2-one anti-9h
(±) (R)-4-((1'R,2'R)-1',2'-dihydroxypentyl)oxazolidin-2-one syn-9h
(E)-4-(Benzyloxy)hept-2-enyl mesityloxycarbamate 14a
Diastereomeric mixture of (±) \((R)-4-((1'S,2'S)-2'((benzyloxy)-1'-hydroxy)pentyl)oxazolidin-2-one, (major) with (±) \((R)-4-((1'S,2'R)-2'((benzyloxy)-1'-hydroxy)pentyl)oxazolidin-2-one (minor), anti-17a and syn-17a
(E)-4-(Benzylox)-5-methylhex-2-enyl mesitoyloxycarbamate, 14b
Diastereomeric mixture of (±) (R)-4-((1'S,2'R)-2'-(benzyloxy)-1'-hydroxy-3'-methylbutyl)oxazolidin-2-one (major) with (±) (R)-4-((1'S,2'S)-2'-(benzyloxy)-1'-hydroxy-3'-methylbutyl)oxazolidin-2-one (minor), anti-17b and syn-17b
(E)-4-(Benzyloxy)-4-phenylbut-2-enyl mesityloxy carbamate, 14c
Diastereomeric mixture of (±) $(R)$-4-((1'S,2'S)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one (major) with (±) $(R)$-4-((1'S,2'R)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one (minor), $anti$-17c and $syn$-17c
(E)-4-((Benzyloxy)-5,5-dimethylhex-2-enyl mesityloxy)carbamate, 14d
(±) (R)-4-((1'S,2'R)-(2'-(Benzyloxy)-1'-hydroxy-3',3'-dimethylbutyl)oxazolidin-2-one, \textit{anti}-17d and \textit{syn}-17d
(Z)-4-(Benzyloxy)-5-methylhex-2-enyl mesitoyloxycarbamate, 16b
(±) \((R)-4'-(1'R,2'S)-(2'-benzyloxy)-1'-hydroxy-2'-methylbutyl)oxazolidin-2-one\)

(major) \textit{anti-19b}\)
(±) \((R)-4-\left((1'R,2'R)-(2'-(benzyloxy)-1'-hydroxy-2'-methylbutyl)oxazolidin-2\text{-one,}\right)

\text{syn-19b}
(Z)-4-(Benzyloxy)-4-phenylbut-2-enyl mesitoyloxycarbamate, 16c
Diastereomeric mixture of $\pm$ (±) $(R)-4-((1'R,2'S)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one$ with $\pm$ (±) $(R)-4-((1'R,2'R)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one$, anti-19c and syn-19c
(Z)-4-(Benzyloxy)-5,5-dimethylhex-2-enyl mesityloxy carbamate, 16d
(±) \((R)-4-((1'R,2'S)-2'-(benzyloxy)-1'-hydroxy-3',3'-dimethylbutyl)oxazolidin-2-one, \textit{anti-19d}\)
(Z)-4-(Benzyloxy)-3-methylhept-2-enyl mesitoyloxycarbamate, 24-Z

[Chemical structure diagram]
(±) (R)-4-((2'R,3'R)-(3'-(benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one

(major diastereomer), anti-25-Z
(±) \((R)-4-((2'R,3'S)-(3'-(benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one\)

(minor diastereomer) syn-25-E
(E)-4-(Benzyloxy)-3-methylhept-2-enyl mesityloxy carbamate, 24-E
Diastereomeric mixture of (±) (R)-4-((2'S,3'R)-(3'-benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one (major) with (±) (R)-4-((2'S,3'S)-(3'-benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one (minor), anti-25-E and syn-25-E.
5. n.O.e. Spectra

\((\pm)\) \((R)-4-((4'R,5'S)-2',2'-\text{dimethyl}-5'-\text{propyl}-1,3\text{-dioxolan-4-yl})\text{oxazolidin-2-one}\)

\textit{anti-11}
(±) (R)-4-((4′R,5′R)-2′,2′-dimethyl-5′-propyl-1,3-dioxolan-4-yl)oxazolidin-2-one

syn-11
Diastereomeric mixture of (±) \((R)-4-((4'S,5'R)-2',2'-\text{dimethyl-5'}-\text{propyl-1,3-dioxolan-4-yl})\text{oxazolidin-2-one}\) (major) with (±) \((R)-4-((4'S,5'S)-2',2'-\text{dimethyl-5'}-\text{propyl-1,3-dioxolan-4-yl})\text{oxazolidin-2-one}\) (minor) anti-18a and syn-18a
Diastereomeric mixture of (±) \((R)-4-((4'R,5'S)-5'-isopropy1-2,2-dimethyl-1,3-dioxolan-4-yl)oxazolidin-2-one\) (major) with (±) \((R)-4-((4'R,5'R)-5'-isopropy1-2,2-dimethyl-1,3-dioxolan-4-yl)oxazolidin-2-one\) (minor) \(anti-18b\) and \(syn-18b\)
(±) (4R,8R,9R)-8-(tert-Butyl)-9-hydroxy-6,6-dimethyltetrahydrooxazolo-oxazin-2-one anti-18d and syn-18d
(±) (R)-4-((4'R,5'S)-5'-Isopropyl-2,2-dimethyl-1,3-dioxolan-4-yl)oxazolidin-2-one

anti-20b
(±) (R)-4-((4'R,5'S)-5'-Isopropyl-2,2-dimethyl-1,3-dioxolan-4-yl)oxazolidin-2-one

\[ \text{syn-20b} \]
(±) \((R)-4-((4'R,5'S)-2,2\text{-}\text{Dimethyl}-5\text{-}\text{phenyl}-1,3\text{-}\text{dioxolan-4-yl})\text{o}xazolidin\text{-}2\text{-}\text{one}\) with (±) \((R)-4-((4'R,5'R)-2,2\text{-}\text{dimethyl}-5\text{-}\text{phenyl}-1,3\text{-}\text{dioxolan-4-yl})\text{o}xazolidin\text{-}2\text{-}\text{one} \text{anti-20c}\)
(±) (4R,8R,9S)-8-(tert-butyl)-9-hydroxy-6,6-dimethyltetrahydrooxazolo-oxazin-2-one, anti-20d
(Z)-4-(Benzyloxy)-3-methylhept-2-enyl mesityloxycarbamate, 24-Z
(±) (R)-4-((4'R,5'R)-(2',2',4'-Trimethyl-5'-propyl-1,3-dioxolan-4-yl)oxazolidin-2-one, *anti*-25-Z
(±) \((R)-4-((4'R,5'S)-(2',2',4'-\text{Trimethyl-5'-propyl-1,3-dioxolan-4-yl})\text{oxazolidin-2-one, syn-24-Z}\)
(E)-4-(Benzyloxy)-3-methylhept-2-en-1-ol, 23-\(E\)
Diastereomeric mixture of (±) (R)-4-((4'S,5'R)-(2',2',4'-trimethyl-5'-propyl-1,3-dioxolan-4-yl)oxazolidin-2-one (major) with (±) (R)-4-((4'S,5'S)-(2',2',4'-trimethyl-5'-propyl-1,3-dioxolan-4-yl)oxazolidin-2-one (minor), anti-25-E and syn-25-E
6. Crude NMR Spectra

Diastereomeric mixture of (±) \((R)-4-((1' R, 2'S)-(2'-(4''-methoxybenzyl)-1'-hydroxypentyl)oxazolidin-2-one\) (major isomer) with (±) \((R)-4-((1'R, 2'R)-(2'-(4''-methoxybenzyl)-1'-hydroxypentyl))oxazolidin-2-one\) (minor isomer), \textit{anti}-9b and \textit{syn}-9b
Diastereomeric mixture of (±) (R)-4-((1′R,2′S)-2′-((tert-butyl(dimethyl)silyl)oxy)-1′-hydroxypentyl)oxazolidin-2-one (major) with (±) (R)-4-((1′R,2′R)-2′-((tert-butyl(dimethyl)silyloxy)-1′-hydroxypentyl)oxazolidin-2-one (minor), anti-9c and syn-9c
Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-(2'-tert-butoxy)-1'-hydroxypentyl)oxazolidin-2-one (major isomer) with (±) (R)-4-((1'R,2'R)-(2'-tert-butoxy)-1'-hydroxypentyl)oxazolidin-2-one (minor isomer), anti-9d and syn-9d
Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-2'-acetate-1'-hydroxypentyl oxazolidin-2-one (major isomer) with (±) (R)-4-((1'R,2'R)-2'-acetate-1'-hydroxypentyl oxazolidin-2-one (minor isomer) anti-9e and syn-9e.
Diastereomeric mixture of (±) $(R)$-4-((1'R,2'S)-2'-mesitoate-1'hydroxypentyl oxazolidin-2-one (major isomer) with (±) $(R)$-4-((1'R,2'R)-2'-mesitoate-1'-hydroxypentyl oxazolidin-2-one (minor isomer), *anti*-9f and *syn*-9f
Diastereomeric mixture of (±) (R)-4-((1'R,2'S)-2'-methoxy-1'-hydroxypentyl oxazolidin-2-one (major isomer) with (±) (R)-4-((1'R,2'R)-2'-methoxy-1'-hydroxypentyl oxazolidin-2-one (minor isomer), anti-9g and syn-9g
Deprotection of Hydroxycarbamates to Confirm Relative Stereochemistry

\[ \text{anti-10} \]

\[ \text{syn-10} \]

From 9b

From 9c

From 9d
Diastereomeric mixture of (±) (R)-4-((1'S,2'S)-2'((benzyloxy)-1'-hydroxypentyl)oxazolidin-2-one, (major) with (±) (R)-4-((1'S,2'R)-2'((benzyloxy)-1'-hydroxypentyl)oxazolidin-2-one (minor) anti- and syn-17a
Diastereomeric mixture of (±) \((R)-4'-(1'S,2'R)-2'-\text{benzyloxy})-1'\text{-hydroxy-3'}\text{-methylbutyl})\text{oxazolidin-2-one}\) (major) with (±) \((R)-4'-(1'S,2'S)-2'-\text{benzyloxy})-1'\text{-hydroxy-3'}\text{-methylbutyl})\text{oxazolidin-2-one}\) (minor) \(\text{anti}\)- and \(\text{syn}\)-17b
Diastereomeric mixture of (±) (R)-4-((1'S,2'S)-2'-benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one (major) with (±) (R)-4-((1'S,2'R)-2'-benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one (minor) anti- and syn-17c
(±) (R)-4-((1'S,2'R)-(2'-(Benzyloxy)-1'-hydroxy-3',3'-dimethylbutyl)oxazolidin-2-one, *anti*-17d
Diastereomeric mixture of (±) \((R)-4-((1'R,2'S)-(2'-(benzyloxy)-1'-hydroxy-2'-methylbutyl)oxazolidin-2-one\) (major) with (±) \((R)-4-((1'R,2'R)-(2'-(benzyloxy)-1'-hydroxy-2'-methylbutyl)oxazolidin-2-one\) (minor), anti- and syn-19b
Diastereomeric mixture of (±) \((R)-4-((1'R,2'S)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one\) with (±) \((R)-4-((1'R,2'R)-2'-(benzyloxy)-1'-hydroxy-2'-phenylethyl)oxazolidin-2-one\), \textit{anti}- and \textit{syn}-19c
(±) \((R)-4-((1'R,2'S)-2'-(Benzylolxy)-1'-hydroxy-3',3'-dimethylbutyl)oxazolidin-2-one\) anti- and syn-19d
(Z)-Ethyl 4-(benzyloxy)-3-methylhept-2-enoate, 22-Z
Diastereomeric mixture of (±) \((R)-4-((2'R,3'R)-(3'-(benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one \) (major diastereomer) with (±) \((R)-4-((2'R,3'S)-(3'-(benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one \) (minor diastereomer) anti- and syn-25-Z

[Chemical structures and spectra]
(E)-Ethyl 4-(benzyloxy)-3-methylhept-2-enoate, 22-\(E\)
Diastereomeric mixture of (±) \((R)-4-((2'S,3'R)-(3'-(benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one\) (major) with (±) \((R)-4-((2'S,3'S)-(3'-(benzyloxy)-2'-hydroxyhexan-2-yl)oxazolidin-2-one\) (minor) \textit{anti} and \textit{syn}\textsuperscript{-25-E}

\[\text{major} \quad \text{minor}\]
7 HPLC Data

HPLC was carried out on a Dionex Ultimate 3000 system, with an ACE 5 Sil 150 x 4.6 mm column, at a flow rate of 1.5 mL/minute of 7:3 heptane:THF.

9a room temperature

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9a 80 °C

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Peak Retention Time Area Height Rel. Area Resolution

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3    | 12.413         | 16.1362  | 19.423  | 4.02      | n.a.      

9a MeOH:H₂O 3:1
**9a MeCN:H₂O 3:1**

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9a tBuOH:H₂O 1:1

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9a 5 mol% Hunig’s Base

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9a 5 mol% (DHQD)$_2$AQN

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\textbf{9a 5 mol\% citric acid}
*anti-* and *syn-9b*

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anti- and syn-9f

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anti- and syn-19c

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**syn-25-Z**

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8. X Ray Diffraction Data

\((\pm)\) \((R)-4-((4'R,5'S)-2',2'-\text{dimethyl-5'-propyl-1,3-dioxolan-4-yl})\text{oxazolidin-2-one}\)

anti-11

\begin{center}
\begin{tikzpicture}
\end{tikzpicture}
\end{center}

**Computing details**

Data collection: *COLLECT* (Nonius, 1997-2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare et al., 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge et al., 2003); molecular graphics: *CAMERON* (Watkin et al., 1996); software used to prepare material for publication: *CRYSTALS* (Betteridge et al., 2003).

**References**


Crystal data

\[
\begin{align*}
C_{11}H_{19}NO_4 & \quad Z = 2 \\
M_r = 229.28 & \quad F(000) = 248 \\
\text{Triclinic, } P1 & \quad D_x = 1.268 \text{ Mg m}^{-3} \\
\text{Hall symbol: } -P 1 & \quad \text{Mo } K\alpha \text{ radiation, } \lambda = 0.71073 \text{ Å} \\
\alpha = 5.7635 (3) \text{ Å} & \quad \text{Cell parameters from 2455 reflections} \\
b = 9.0193 (4) \text{ Å} & \quad \theta = 5–27^\circ \\
c = 11.9660 (7) \text{ Å} & \quad \mu = 0.10 \text{ mm}^{-1} \\
\alpha = 80.997 (2)^\circ & \quad T = 150 \text{ K} \\
\beta = 82.328 (2)^\circ & \quad \text{Plate, Colourless} \\
\gamma = 79.362 (2)^\circ & \quad 0.44 \times 0.44 \times 0.20 \text{ mm} \\
V = 600.29 (5) \text{ Å}^3 & \quad \\
\end{align*}
\]

Data collection

Area diffractometer

2026 reflections with \( I > 2.0\sigma(I) \)
graphite  
\( R_{int} = 0.025 \)

\( \omega \) scans  
\( \theta_{\text{max}} = 27.6^\circ, \theta_{\text{min}} = 5.2^\circ \)

Absorption correction: Multi-scan

\textit{DENZO/SCALEPACK} (Otwinowski & Minor, 1997)

\( T_{\text{min}} = 0.96, T_{\text{max}} = 0.98 \)

4409 measured reflections

2742 independent reflections

**Refinement**

**Refinement on \( F^2 \)**

Primary atom site location: Structure-invariant direct methods

Least-squares matrix: Full

Hydrogen site location: Inferred from neighbouring sites

\( R[F^2 > 2\sigma(F^2)] = 0.053 \)

H-atom parameters constrained

Method = Modified Sheldrick

\( wR(F^2) = 0.135 \)

\( w = 1/[\sigma^2(F^2) + (0.06P)^2 + 0.41P] \),

where \( P = (\text{max}(F_o^2,0) + 2F_c^2)/3 \)

\( S = 0.94 \)

\( (\Delta/\sigma)_{\text{max}} = 0.0003 \)

2742 reflections

145 parameters

0 restraints

\( \Delta \rho_{\text{max}} = 0.53 \) e Å\(^{-3}\)

\( \Delta \rho_{\text{min}} = -0.35 \) e Å\(^{-3}\)

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å\(^2\))**

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*Atomic displacement parameters (Å²)*

|  | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
| | | | | | | |
| O1 | 0.0415 (7) | 0.0293 (6) | 0.0283 (7) | −0.0136 (5) | −0.0024 (5) | −0.0024 (5) |
| C2 | 0.0292 (9) | 0.0266 (8) | 0.0311 (9) | −0.0066 (7) | −0.0027 (7) | −0.0047 (7) |
| C3 | 0.0298 (9) | 0.0277 (8) | 0.0292 (9) | −0.0060 (7) | −0.0037 (7) | −0.0027 (7) |
| O4 | 0.0417 (7) | 0.0250 (6) | 0.0282 (6) | −0.0083 (5) | −0.0017 (5) | −0.0032 (5) |
| C5 | 0.0337 (9) | 0.0276 (9) | 0.0261 (9) | −0.0085 (7) | −0.0008 (7) | −0.0028 (7) |
| C6 | 0.0326 (10) | 0.0413 (11) | 0.0413 (11) | −0.0069 (8) | 0.0012 (8) | −0.0018 (8) |
| C7 | 0.0376 (10) | 0.0270 (9) | 0.0384 (10) | −0.0055 (7) | −0.0025 (8) | −0.0049 (7) |
| C8 | 0.0354 (9) | 0.0298 (9) | 0.0294 (9) | −0.0012 (7) | −0.0037 (7) | −0.0031 (7) |
| C9 | 0.0515 (12) | 0.0298 (9) | 0.0320 (10) | 0.0056 (8) | −0.0005 (8) | −0.0041 (8) |
| C10 | 0.0520 (12) | 0.0350 (10) | 0.0441 (12) | 0.0020 (9) | 0.0050 (9) | −0.0079 (9) |
| C11 | 0.0330 (9) | 0.0303 (9) | 0.0297 (9) | −0.0084 (7) | 0.0002 (7) | −0.0065 (7) |
| N12 | 0.0356 (8) | 0.0266 (7) | 0.0303 (8) | −0.0093 (6) | 0.0021 (6) | −0.0071 (6) |
| C13 | 0.0347 (9) | 0.0276 (8) | 0.0269 (9) | −0.0070 (7) | −0.0034 (7) | −0.0054 (7) |
### Geometric parameters (Å, °)

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H73—C7—H71  110.3  C11—C15—O14  104.74 (14)
C5—C7—H72  109.7  C11—C15—H151  111.3
H73—C7—H72  110.9  O14—C15—H151  110.4
H71—C7—H72  109.0  C11—C15—H152  111.0
C3—C8—C9  112.31 (15)  O14—C15—H152  108.7
C3—C8—H81  106.6  H151—C15—H152  110.5
C9—C8—H81  109.4

Hydrogen-bond geometry (Å, °)

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Symmetry code: (i) −x+1, −y, −z+2.

(±) (R)-4-((4'R,5'S)-5'-Isopropyl-2,2-dimethyl-1,3-dioxolan-4-yl)oxazolidin-2-one

*anti*-20b
Computing details

Data collection: COLLECT (Nonius, 1997-2001); cell refinement: DENZO/SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO/SCALEPACK (Otwinowski & Minor, 1997); program(s) used to solve structure: SIR92 (Altomare et al., 1994); program(s) used to refine structure: CRYSTALS (Betteridge et al., 2003); molecular graphics: CAMERON (Watkin et al., 1996); software used to prepare material for publication: CRYSTALS (Betteridge et al., 2003).

References


**Crystal data**

C_{11}H_{19}NO_4  

\[ F(000) = 496 \]

\[ M_r = 229.28 \]

\[ D_x = 1.292 \text{ Mg m}^{-3} \]

Monoclinic, \( P2_1/c \)

Mo Ka radiation, \( \lambda = 0.71073 \text{ Å} \)

Hall symbol: -P 2ybc

Cell parameters from 2816 reflections

\[ a = 13.4443 \ (3) \text{ Å} \]

\[ \theta = 5–27^\circ \]

\[ b = 8.0126 \ (2) \text{ Å} \]

\[ \mu = 0.10 \text{ mm}^{-1} \]

\[ c = 11.2541 \ (3) \text{ Å} \]

\[ T = 150 \text{ K} \]

\[ \beta = 103.5780 \ (14)^\circ \]

Plate, Colourless

\[ V = 1178.45 \ (5) \text{ Å}^3 \]

\[ 0.40 \times 0.30 \times 0.20 \text{ mm} \]

\[ Z = 4 \]

**Data collection**

Area
diffractometer

2075 reflections with \( I > 2.0\sigma(I) \)

graphite

\[ R_{int} = 0.041 \]

\( \omega \) scans

\[ \theta_{max} = 27.5^\circ, \theta_{min} = 5.3^\circ \]

Absorption correction: Multi-scan

\textit{DENZO/SCALEPACK} (Otwiowski & Minor, 1997)

\[ h = -17 \rightarrow 17 \]

\[ k = -10 \rightarrow 9 \]

\[ l = -14 \rightarrow 14 \]

16300 measured reflections

2682 independent reflections

**Refinement**
Refinement on $F^2$

Primary atom site location: Structure-invariant direct methods

Least-squares matrix: Full

Hydrogen site location: Inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.038$

H-atom parameters constrained

Method = Modified Sheldrick

$wR(F^2) = 0.073$

$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.46P]$

where $P = (\text{max}(F_o^2,0) + 2F_c^2)/3$

$S = 0.98$

$(\Delta/\sigma)_{\text{max}} = 0.0002$

2682 reflections

$\Delta \rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$

145 parameters

$\Delta \rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$

0 restraints

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

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<th>$x$</th>
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Hydrogen-bond geometry (Å, °)

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Symmetry codes: (i) x, y+3/2, z+1/2; (ii) x, y+1, z; (iii) −x, y+1/2, −z+1/2; (iv) −x, −y+1, −z+1.

(±) (R)-4-((4’S,5’R)-2’,2’-dimethyl-5’-propyl-1,3-dioxolan-4-yl)oxazolidin-2-one

anti-18a

Computing details
Data collection: COLLECT (Nonius, 2001); cell refinement: DENZO/SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO/SCALEPACK (Otwinowski & Minor, 1997); program(s) used to solve structure: SIR92 (Altomare et al., 1994); program(s) used to refine structure: CRYSTALS (Betteridge et al., 2003); molecular graphics: CAMERON (Watkin et al., 1996); software used to prepare material for publication: CRYSTALS (Betteridge et al., 2003).

References


Crystal data

\[ \text{C}_{11}\text{H}_{19}\text{NO}_4 \]  

\[ F(000) = 496 \]

\[ M_r = 229.28 \]  

\[ D_x = 1.277 \text{ Mg m}^{-3} \]
Monoclinic, $P2_1/c$  
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

$a = 8.8709 (3)$ Å
Cell parameters from 2776 reflections

$b = 10.0472 (4)$ Å  
$\theta = 5$–$27^\circ$

$c = 13.3746 (6)$ Å  
$\mu = 0.10$ mm$^{-1}$

$\beta = 90.3115 (14)^\circ$  
$T = 150$ K

$V = 1192.03 (8)$ Å$^3$
Block, Colourless

$Z = 4$
$0.32 \times 0.16 \times 0.06$ mm

**Data collection**

Nonius KappaCCD
diffractometer

graphite  
$R_{int} = 0.035$

$\omega$ scans  
$\theta_{max} = 27.5^\circ$, $\theta_{min} = 5.2^\circ$

Absorption correction: Multi-scan

*DENZO/SCALEPACK* (Otwinowski & Minor, 1997)

$h = -11 \rightarrow 11$

$k = -13 \rightarrow 11$

$12796$ measured reflections

$l = -17 \rightarrow 17$

$2719$ independent reflections

**Refinement**

Refinement on $F^2$

Primary atom site location: Structure-invariant direct methods

Least-squares matrix: Full

Hydrogen site location: Inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.046$
No H atoms present
Method, part 1, Chebychev polynomial, (Watkin, 1994, Prince, 1982) [weight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) + ... + A_n*T_n(x)]

where A_i are the Chebychev coefficients listed below and x

\[ wR(F^2) = 0.074 \]

Weighting (Prince, 1982) W = [weight] * [1 - (\Delta F / 6 \times \sigma F)^2]^2

A_i are: 3.69 3.22 0.59

\[ S = 1.07 \]

\[ (\Delta \sigma)_{\text{max}} = 0.0002 \]

2719 reflections \[ \Delta \rho_{\text{max}} = 0.48 \text{ e Å}^{-3} \]

145 parameters \[ \Delta \rho_{\text{min}} = -0.49 \text{ e Å}^{-3} \]

0 restraints

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

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### Geometric parameters (Å, °)

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**Hydrogen-bond geometry (Å, °)**

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Symmetry codes: (i) −x+1, y+1/2, −z+3/2; (ii) −x+2, −y+1, −z+2; (iii) −x+1, −y+1, −z+2.
9. References