Supporting Information

N-Heterocyclic carbene catalysed β-lactam synthesis

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I. General Experimental Procedures

General Information

All reactions involving moisture sensitive reagents were performed under an atmosphere of nitrogen via standard vacuum line techniques and with freshly dried solvents. All glassware was flame dried and allowed to cool under vacuum. Toluene, tetrahydrofuran (THF), diethyl ether (Et₂O) and dichloromethane were obtained dry from a solvent purification system (MBraun, SPS-800). Potassium bis(trimethylsilyl)amide (KHMDS) was supplied as a 0.5M solution in toluene (Aldrich), titrated before use, and used as a 0.45M solution. Petrol is defined as petroleum ether 40-60 °C. All solvents and commercial reagents were used as supplied without further purification unless stated otherwise. Room temperature refers to 20-25 °C. Reflux conditions were obtained using an oil bath equipped with a contact thermometer. In vacuo refers to the use of a Büchi Rotavapor R-2000 rotary evaporator with a Vacubrand CVC₂ vacuum controller or a Heidolph Laborota 4001 rotary evaporator with a vacuum controller.

Analytical thin layer chromatography was performed on aluminium sheets coated with 60 F₂₅₄ silica. TLC visualisation was carried out with ultraviolet light (254 nm), followed by staining 1% aqueous KMnO₄ solution. Flash column chromatography was performed on Kieselgel 60 silica in the solvent system stated.

¹H and ¹³C nuclear magnetic resonance (NMR) spectra were acquired on a Bruker Avance II 400 (400 MHz ¹H, 100 MHz ¹³C) spectrometer and in CDCl₃ unless stated otherwise. Coupling constants (J) are reported in Hz.

Infrared spectra (νmax) were recorded on a Perkin-Elmer Spectrum GX FT-IR Spectrometer using KBr discs (KBr disc) as stated. Only the characteristic peaks are quoted. Microanalyses were carried out on a Carlo Erba CHNS analyser. Melting points were recorded on an Electrothermal apparatus and are uncorrected.

Mass spectrometric (m/z) data was acquired by electrospray ionisation (ESI), electron impact (EI) or chemical ionisation (CI), either at the University of St Andrews Mass Spectrometry facility or from the EPSRC National Mass Spectrometry Service Centre, Swansea. At the University of St Andrews low and high resolution ESI MS was carried out on a Micromass LCT spectrometer and low and high resolution CI MS was carried out on a Micromass GCT

II. Synthesis of ketenes

Diphenylketene and isobutylphenylketene were synthesized according to literature procedures.²³

III. Synthesis of imines

N-Tosyl imines were prepared according the procedure described by Proctor.⁴ BF₃·Et₂O (0.016 equiv) was added to a refluxing solution of aldehyde (1.0 equiv.) and para-toluene sulfonamide (1.0 equiv) in toluene (150 mL) using a Dean-Stark apparatus. The mixture was refluxed until the theoretical amount of water had been collected (20 hours). The solution was then cooled and washed with 2M NaOH solution and water. The organic layer was separated, dried (MgSO₄) and the solvent was removed under reduced pressure. The residue was purified by recrystallisation from EtOAc/hexane unless otherwise noted. The following yields have not been optimised.

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\text{N-Benzylidene-4-methylbenzenesulfonamide. The imine was obtained using benzaldehyde (5.1 mL, 50 mmol) and para-toluene sulfonamide (8.56 g, 50 mmol) and following the above general procedure. The residue was purified by recrystallisation to give the title compound (9.83 g, 76% yield) as a white solid.}^{1} \text{H NMR } \delta 2.44 (3H, s), 7.35 (2H, d, } J = 8.0), 7.49 (2H, t, } J = 7.8), 7.62 (2H, tt, } J = 7.6, 2.8), 7.86-7.97 (4H, m), 9.03 (1H, s).
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**N-(2-Naphthylidene)-4-methylbenzenesulfonamide.** The imine was obtained using 2-naphthaldehyde (5 g, 32 mmol) and para-toluenesulfonamide (5.48 g, 32 mmol) and following the above general procedure. The residue was purified by recrystallisation to give the title compound (8.41 g, 85% yield) as a white solid. $^1$H NMR $\delta$ 2.44 (3H, s), 7.36 (2H, d, $J = 8.4$), 7.57 (1H, t, $J = 7.2$), 7.62 (1H, t, $J = 7.0$), 7.87 (2H, d, $J = 8.8$), 7.91-7.98 (3H, m), 8.02 (1H, dd, $J = 8.4$, 1.2), 8.32 (1H, s), 9.17 (1H, s).

**N-(2-Furfurylidene)-4-methylbenzenesulfonamide.** The imine was obtained using furfural (4.15 mL, 50 mmol) and para-toluenesulfonamide (8.56 g, 50 mmol) and following the above general procedure. The residue was purified by recrystallisation to give the title compound (6.69 g, 54% yield) as a yellowish solid. $^1$H NMR $\delta$ 2.42 (3H, s), 6.64 (1H, dd, $J = 3.6$, 1.8), 7.28-7.37 (3H, m), 7.74 (1H, m), 7.86 (2H, d, $J = 8.4$), 8.81 (1H, s).

**N-(4-Bromobenzylidene)-4-methylbenzenesulfonamide.** The imine was obtained using 4-bromobenzaldehyde (9.25 g, 50 mmol) and para-toluenesulfonamide (8.56 g, 50 mmol) and following the above general procedure. The residue was purified by recrystallisation from THF/hexane to give the title compound (13.20 g, 78% yield) as a white solid. $^1$H NMR $\delta$ 2.44 (3H, s), 7.35 (2H, d, $J = 8.0$), 7.63 (2H, d, $J = 8.4$), 7.78 (2H, d, $J = 8.4$), 7.88 (2H, d, $J = 8.4$), 8.98 (1H, s).
**N-(4-Methoxybenzylidene)-4-methylbenzenesulfonamide.** The imine was obtained using 4-methoxybenzaldehyde (6.1 mL, 50 mmol) and *para*-toluenesulfonamide (8.56 g, 50 mmol) and following the above general procedure. The residue was purified by recrystallisation from THF/hexane to give the title compound (10.89 g, 75% yield) as a white solid. $^1$H NMR $\delta$ 2.41 (3H, s), 3.86 (3H, s), 6.95 (2H, d, $J = 8.7$), 7.32 (2H, d, $J = 8.1$), 7.78-7.97 (4H, m), 8.93 (1H, s).

**N-(3-Phenyl-allylidene)-4-methylbenzenesulfonamide.** The imine was obtained using cinnamaldehyde (6.3 mL, 50 mmol) and *para*-toluenesulfonamide (8.56 g, 50 mmol) and following the above general procedure. The residue was purified by recrystallisation to give the title compound (10.92 g, 77% yield) as a pale brown solid. $^1$H NMR $\delta$ 2.43 (3H, s), 6.98 (1H, dd, $J = 15.6, 9.3$), 7.34 (2H, d, $J = 8.1$), 7.37-7.60 (6H, m), 7.86 (2H, d, $J = 8.1$), 8.77 (1H, d, $J = 9.3$).

### IV. Procedures for the Staudinger reaction promoted by NHC 2 (Figure 2)

**Entry 1:**
A 0.45 M solution of KHMDS in toluene (0.16 mL, 0.0733 mmol, 19 mol%) was added to a suspension of triazolium salt 1 (21.1 mg, 0.0771 mmol, 20 mol%) in toluene (1 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of diphenylketene 3 (74.9 mg, 0.386 mmol, 1.0 equiv) in toluene (1 mL) was added, followed by a solution of *N*-benzylidene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) in toluene (1 mL), then the reaction mixture was stirred for 24 hours at room temperature before concentration. The analysis of the crude product by $^1$H NMR revealed...
>95% conversion of the imine to the corresponding β-lactam. The residue was purified by column chromatography (EtOAc/petroleum ether 10:90 → 30:70) to give β-lactam (±)-5 (103.1 mg, 59% yield) as a white solid.

Entries 2 and 3:
A 0.45 M solution of KHMDS in toluene (0.16 mL, 0.0733 mmol, 19 mol%) was added to a suspension of triazolium salt 1 (21.1 mg, 0.0771 mmol, 20 mol%) in toluene (1 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) in toluene (1 mL) was added, followed by a solution of N-benzyldiene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) in toluene (1 mL), then the reaction mixture was stirred for 24 hours at room temperature before concentration. The reaction mixture was sampled after 1 hour and 24 hours and the 1H NMR spectroscopic analysis revealed 66% and >95% conversion of the imine to the corresponding β-lactam, respectively. The residue was purified by column chromatography (EtOAc/petroleum ether 10:90 → 30:70) to give β-lactam (±)-5 (146.3 mg, 84% yield) as a white solid.

Entries 4 and 5:
Same procedure as above, except that CH2Cl2 and THF were used as solvent instead of toluene.

Entry 6:
A 0.45 M solution of KHMDS in toluene (0.16 mL, 0.0733 mmol, 19 mol%) was added to a suspension of triazolium salt 1 (21.1 mg, 0.0771 mmol, 20 mol%) in Et2O (1.5 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) in Et2O (1.5 mL) was added, followed by N-benzyldiene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) as a solid, then the reaction mixture was stirred for 1 hour at room temperature before concentration. The analysis of the crude product by 1H NMR revealed >95% conversion of the imine to the corresponding β-lactam (±)-5.
Entry 7:
A 0.45M solution of KHMDS in toluene (0.08 mL, 0.0347 mmol, 9 mol%) was added to a suspension of triazolium salt 1 (10.5 mg, 0.0386 mmol, 10 mol%) in Et₂O (1.5 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) in Et₂O (1.5 mL) was added, followed by N-benzylidene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) as a solid, then the reaction mixture was stirred for 1 hour at room temperature before concentration. The analysis of the crude product by ¹H NMR revealed >95% conversion of the imine to the corresponding β-lactam (±)-5.

Entry 8:
A 0.45 M solution of KHMDS in toluene (0.04 mL, 0.0174 mmol, 4.5 mol%) was added to a suspension of triazolium salt 1 (5.3 mg, 0.0193 mmol, 5 mol%) in Et₂O (1.5 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) in Et₂O (1.5 mL) was added, followed by N-benzylidene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) as a solid, then the reaction mixture was stirred for 1 hour at room temperature before concentration. The analysis of the crude product by ¹H NMR revealed >95% conversion of the imine to the corresponding β-lactam (±)-5. The residue was purified by column chromatography (EtOAc/petroleum ether 10:90 → 30:70) to give β-lactam (±)-5 (162.6 mg, 93% yield) as a white solid.

Entry 9:
A 0.45 M solution of KHMDS in toluene (0.008 mL, 0.0035 mmol, 0.9 mol%) was added to a suspension of triazolium salt 1 (1.1 mg, 0.0039 mmol, 1 mol%) in Et₂O (1.5 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) in Et₂O (1.5 mL) was added, followed by N-benzylidene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) as a solid, then the reaction mixture was stirred for 24 hours at room temperature before concentration. The analysis of the crude product by ¹H NMR revealed 60% conversion of the imine to the corresponding β-lactam (±)-5.
V. General procedure for the synthesis of β-lactams 5 and 8-12 (Figure 4).

A 0.45 M solution of KHMDS in toluene (0.04 mL, 0.0174 mmol, 4.5 mol% or 0.08 mL, 0.0347 mmol, 9 mol%) was added to a suspension of triazolium salt 1 (5.3 mg, 0.0193 mmol, 5 mol% or 10.5 mg, 0.0386 mmol, 10 mol%) in Et$_2$O (1.5 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) in Et$_2$O (1.5 mL) was added, followed by the imine (0.386 mmol, 1.0 equiv) as a solid, then the reaction mixture was stirred at room temperature before concentration.

(±)-3,3,4-Triphenyl-1-tosylazetidin-2-one (±)-5. The β-lactam (±)-5 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-benzylidene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 20:80 then CH$_2$Cl$_2$/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-5 (162.6 mg, 93% yield) as a white solid (mp = 184-185°C). IR (KBr disk) $\nu$ 3063, 3037, 2928, 1776 (C=O), 1597, 1495, 1452, 1447, 1383, 1370, 1262, 1210, 1188, 1172, 1141, 1090, 1070, 1028; $^1$H NMR δ 2.32 (3H, s), 5.70 (1H, s), 6.77-6.94 (7H, m), 6.98 (2H, t, $J = 7.6$), 7.05 (1H, t, $J = 7.2$), 7.11-7.27 (5H, m), 7.31 (2H, d, $J = 7.2$), 7.65 (2H, d, $J = 8.0$); $^{13}$C NMR δ 21.8 (CH$_3$), 69.3 (CH), 72.9 (C), 127.0 (2 CH), 127.3 (CH), 127.7 (2 CH), 127.9 (3 CH), 128.0 (2 CH), 128.1 (2 CH), 128.4 (2 CH), 128.5 (CH), 129.0 (2 CH), 129.9 (2 CH), 134.0 (C), 135.4 (C), 135.9 (C), 139.0 (C), 145.4 (C), 166.9 (C); CIMS (NH$_3$) m/z 471 (M + NH$_4^+$, 4), 257 (67), 256 (Ph$_2$C=CH$^+$-Ph, 29), 212 (Ph$_2$C=C=O + NH$_4^+$, 27), 106 (O=C-N-SO$_2$), 100, 91 (C$_6$H$_5^+$-CH$_3$, 77); HRMS (EI, NH$_3$) [M + NH$_4^+$] $C_{28}H_{27}N_2O_3S$ requires 471.1737, found, 471.1738; Anal. Calcd. for C$_{28}$H$_{23}$NO$_3$S: C 74.15, H 5.11, N 3.09%, Found: C 73.84, H 4.97, N 3.12%. 

(±)-3,3-Diphenyl-4-(2-naphthyl)-1-tosylazetidin-2-one (±)-8. The β-lactam (±)-8 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-(2-naphthylidene)-4-methylbenzenesulfonamide (119.3 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 20:80 then CH2Cl2/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-8 (177.9 mg, 92% yield) as a white solid (mp = 182-183°C). IR (KBr disk) ν3054, 1792 (C=O), 1595, 1496, 1449, 1374, 1361, 1167, 1128, 1121, 1088; 1H NMR δ 2.39 (3H, s), 5.94 (1H, s), 6.81 (1H, d, J = 8.4, 1.2), 6.83-7.03 (5H, m), 7.18 (2H, d, J = 8.0), 7.22-7.39 (4H, m), 7.39-7.51 (5H, m), 7.56 (1H, s), 7.63 (1H, dd, J = 5.6, 3.2), 7.69 (3H, d, J = 8.0); 13C NMR δ 27.7 (CH3), 69.7 (CH), 72.8 (C), 124.8 (CH), 126.3 (CH), 126.5 (CH), 127.0 (2 CH), 127.3 (CH), 127.7 (4 CH), 128.0 (4 CH), 128.1 (CH), 128.2 (2 CH), 129.0 (2 CH), 129.8 (2 CH), 131.5 (C), 132.7 (C), 133.1 (C), 135.5 (C), 135.7 (C), 139.2 (C), 145.4 (C), 166.8 (C); CIMS m/z 504 (M + H+, 18), 308 (M - Ph2C=C=O, 13), 307 (100), 306 (40), 198 (O=C-N=SO2-tol, 38), 194 (Ph2C=C=O, 18), 155 (SO2-tol, 16), 141 (C11H9, 5); HRMS (CI) [M + H+] C32H26NO3S requires 504.1633, found, 504.1631.

(±)-3,3-Diphenyl-4-(2-furanyl)-1-tosylazetidin-2-one (±)-9. The β-lactam (±)-9 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-(2-furfurylidene)-4-methylbenzenesulfonamide (96.1 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 20:80 then CH2Cl2/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-9 (146.0 mg, 85% yield) as a white solid (mp = 160°C). IR (KBr disk) ν3129, 3090, 3069, 1792 (C=O), 1596, 1496, 1450, 1364, 1257, 1170, 1138, 1089; 1H NMR δ 2.34 (3H, s), 5.69
(±)-3,3-Diphenyl-4-(4-methoxyphenyl)-1-tosylazetidin-2-one (±)-10. The β-lactam (±)-10 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-(4-methoxybenzylidene)-4-methylbenzenesulfonamide (111.6 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 24 hours at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 20:80 then CH₂Cl₂/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-10 (109.2 mg, 59% yield) as a white solid (mp = 183-184°C). IR (KBr disk) ν 3161, 2931, 2834, 1773 (C=O), 1595, 1515, 1496, 1447, 1368, 1248, 1173, 1138, 1089; 1H NMR δ 2.42 (3H, s), 3.73 (3H, s), 5.72 (1H, s), 6.59 (2H, d, J = 8.4), 6.84 (2H, d, J = 8.8), 6.98-7.09 (3H, m), 7.18-7.43 (7H, m), 7.72 (2H, d, J = 8.4); 13C NMR δ 21.7 (CH₃), 55.2 (CH₃), 69.3 (CH), 72.6 (C), 113.5 (2 CH), 125.9 (C), 126.9 (2 CH), 127.2 (CH), 127.7 (2 CH), 127.8 (CH), 128.0 (2 CH), 128.2 (2 CH), 128.9 (2 CH), 129.3 (2 CH), 129.8 (2 CH), 135.6 (C), 135.9 (C), 139.3 (C), 145.3 (C), 159.7 (C), 166.9 (C); CIMS m/z 484 (M + H⁺, 3), 287 (MH⁺ – O=C=N-SO₂-tol, 43), 286 (28), 198 (O=C=N-SO₂-tol, 93), 172 (100), 155 (SO₂-tol, 93), 135 (MeO-C₆H₄-CN, 39); HRMS (CI) [M + H⁺] C₂₉H₂₆NO₄S requires 444.1270, found, 444.1265.
S
O
N
O
Br

(±)-4-(4-Bromophenyl)-3,3-diphenyl-1-tosylazetidin-2-one (±)-11. The β-lactam (±)-11 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-(4-bromobenzylidene)-4-methylbenzenesulfonamide (130.4 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 20:80 then CH2Cl2/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-11 (181.8 mg, 89% yield) as a white solid (mp = 214-215°C). IR (KBr disk) ν 3161, 2931, 2834, 1773 (C=O), 1595, 1494, 1447, 1367, 1170, 1141, 1089; 1H NMR δ 2.43 (3H, s), 5.70 (1H, s), 6.82 (2H, d, J = 8.4), 6.87-6.94 (2H, m), 6.98-7.10 (3H, m), 7.20 (2H, d, J = 8.4), 7.23-7.38 (7H, m), 7.73 (2H, d, J = 8.4); 13C NMR δ 21.8 (CH3), 68.5 (CH), 73.0 (C), 122.7 (C), 127.0 (2 CH), 127.6 (CH), 127.7 (2 CH), 127.9 (2 CH), 128.0 (CH), 128.4 (2 CH), 129.0 (2 CH), 129.5 (2 CH), 129.9 (2 CH), 131.2 (2 CH), 133.3 (C), 135.2 (C), 135.5 (C), 138.7 (C), 145.6 (C), 166.6 (C); CIMS m/z 534 (MH+ (81Br), 87), 532 (MH+ (79Br), 83), 378 (M (81Br) – 2 * C6H5), 376 (M (79Br) – 2 * C6H5), 337 (M (81Br) – Ph2C=C=O, 33), 336 (M (81Br) – O=C=N-SO2tol, 42), 335 (M (79Br) – Ph2C=O, 36), 334 (M (79Br) – O=C=N-SO2tol, 35), 256 (28), 226 (22), 198 (O=C=N-SO2-tol, 29), 194 (Ph2C=O=O, 100); HRMS (Cl) [M (79Br) + H+] C28H2779BrN2O3S requires 532.0582, found, 532.0471; [M (81Br) + H+] C28H2781BrN2O3S requires 534.0562, found, 534.0457; Anal. Calcd. for C28H22BrNO3S: C 63.16, H 4.16, N 2.63%, Found: C 63.38, H 4.08, N 2.55%.

S
O
N
O

(±)-3,3-Diphenyl-4-(2-phenylvinyl)-1-tosylazetidin-2-one (±)-12. The β-lactam (±)-12 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-(3-phenyl-allylidene)-4-methylbenzenesulfonamide (110.0 mg, 0.386 mmol, 1.0 equiv)
and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 20:80 then CH$_2$Cl$_2$/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-12 (174.2 mg, 94% yield) as a white solid (mp = 164-165°C). IR (KBr disk) ν 3061, 3028, 2360, 1771 (C=O), 1596, 1494, 1448, 1365, 1171, 1147, 1089; $^1$H NMR δ 2.42 (3H, s), 5.34 (1H, d, $J = 9.6$), 5.43 (1H, dd, $J = 15.2, 9.6$), 6.87 (1H, d, $J = 15.6$), 7.04-7.20 (2H, m), 7.20-7.48 (15H, m), 7.68 (2H, d, $J = 8.0$); $^{13}$C NMR δ 21.7 (CH$_3$), 68.6 (CH), 71.0 (C), 123.7 (CH), 126.88 (2 CH), 126.93 (2 CH), 127.7 (2 CH), 127.9 (CH), 127.95 (2 CH), 128.0 (CH), 128.6 (3 CH), 128.7 (2 CH), 129.0 (2 CH), 130.0 (2 CH), 135.5 (C), 136.0 (C), 136.1 (C), 136.6 (C), 138.7 (C), 145.3 (C), 166.1 (C); CIMS m/z 480 (M + H$^+$, 4), 349 (3), 307 (8), 282 (18), 198 (O=C=N-SO$_2$-tol, 100), 172 (13), 155 (SO$_2$tol, 37); Anal. Calcd. for C$_{30}$H$_{25}$NO$_3$S: C 75.13, H 5.25, N 2.92%, Found: C 75.05, H 5.09, N 2.87%.

VI. General procedure for the synthesis of β-lactams 14 to 18 (Figure 5).

A 0.45 M solution of KHMDS in toluene (0.08 mL, 0.0347 mmol, 9 mol%) was added to a suspension of triazolium salt 1 (10.5 mg, 0.0386 mmol, 10 mol%) in Et$_2$O (1.5 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of isobutylphenylketene 13 (87.3 mg, 0.501 mmol, 1.3 equiv) in Et$_2$O (1.5 mL) was added, followed the imine (0.386 mmol, 1.0 equiv) as a solid, then the reaction mixture was stirred at room temperature before concentration. The initial diastereoselectivity was measured by $^1$H NMR on the crude product and can be improved (after purification by column chromatography) by trituration of the product in Et$_2$O and filtration. This procedure allowed the characterisation of the major syn diastereoisomer.
**Syn-3,4-diphenyl-3-isobutyl-1-tosylazetidin-2-one (±)-14.** The β-lactam (±)-14 was obtained using isobutylphenylketene 13 (87.3 mg, 0.501 mmol, 1.3 equiv) and N-benzylidene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour at room temperature (dr = 68:32 syn:anti) and the residue was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH₂Cl₂/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-14 (148.7 mg, 89% yield). Trituration in Et₂O gave a white solid (mp = 156-158°C, dr = 89:11 syn:anti). IR (KBr disk) ν 3064, 3034, 2958, 2927, 2868, 2360, 1770 (C=O), 1597, 1496, 1447, 1364, 1241, 1188, 1170, 1145, 1090; ¹H NMR syn: δ 0.69 (3H, d, J = 6.8), 0.87 (3H, d, J = 6.4), 1.48-1.61 (1H, m), 1.92 (1H, dd, J = 14.6, 6.0), 2.04 (1H, dd, J = 14.0, 6.4), 2.43 (3H, s), 5.00 (1H, s), 6.77 (2H, d, J = 7.6), 6.85-6.92 (2H, m), 6.95-7.05 (5H, m), 7.10 (1H, t, J = 7.4), 7.15-7.30 (2H, m), 7.71 (2H, d, J = 8.4); anti (visible peaks): δ 0.37 (3H, d, J = 6.4), 2.43 (3H, s), 5.05 (1H, s), 7.80 (2H, d, J = 8.4); ¹³C NMR syn: δ 21.7 (CH₃), 23.7 (CH₃), 23.8 (CH₃), 24.9 (CH), 47.5 (CH₂), 69.3 (C), 69.8 (CH), 127.0 (CH), 127.4 (2 CH), 127.6 (2 CH), 127.9 (2 CH), 127.95 (2 CH), 128.0 (2 CH), 128.4 (CH), 129.7 (2 CH), 134.2 (C), 134.9 (C), 135.7 (C), 145.2 (C), 168.1 (C); anti (visible peaks): δ 23.0, 24.0, 24.5, 42.1, 69.9, 126.1, 127.4, 127.8, 128.6, 128.8, 128.9, 129.8, 168.5.

**Syn-3-Isobutyl-4-(2-naphthyl)-3-phenyl-1-tosylazetidin-2-one (±)-15.** The β-lactam (±)-15 was obtained using isobutylphenylketene 13 (87.3 mg, 0.501 mmol, 1.3 equiv) and N-(2-naphthylidene)-4-methylbenzenesulfonamide (119.3 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour at room temperature (dr = 63:37 syn:anti) and the residue
was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH₂Cl₂/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-15 (165.8 mg, 89% yield). Trituration in Et₂O gave a white solid (mp = 146-148°C, dr = 90:10 syn:anti). IR (KBr disk) ν̃ 3060, 2956, 2927, 2870, 2358, 1784 (C=O), 1597, 1496, 1448, 1368, 1187, 1172, 1088; ¹H NMR syn: δ 0.72 (3H, d, J = 6.8), 0.91 (3H, d, J = 6.8), 1.51-1.65 (1H, m), 2.00 (1H, dd, J = 14.4, 6.0), 2.11 (1H, dd, J = 14.0, 6.4), 2.38 (3H, s), 5.18 (1H, s), 6.62 (1H, dd, J = 8.4, 1.2), 6.88-7.00 (5H, m), 7.16 (2H, d, J = 8.0), 7.32 (1H, d, J = 8.8), 7.38-7.48 (3H, m), 7.60 (1H, dd, J = 6.0, 3.2), 7.60-7.69 (3H, m); anti (visible peaks): δ 0.33 (3H, d, J = 6.4), 2.45 (3H, s), 5.21 (1H, s); ¹³C NMR syn: δ 21.7 (CH₃), 23.7 (CH₃), 23.8 (CH₃), 24.9 (CH), 47.8 (CH₂), 69.2 (C), 70.0 (CH), 124.9 (CH), 126.2 (CH), 126.4 (CH), 127.0 (CH), 127.4 (2 CH), 127.5 (CH), 127.6 (2 CH), 127.9 (CH), 128.0 (2 CH), 128.2 (CH), 129.7 (2 CH), 131.7 (C), 132.6 (C), 133.1 (C), 134.8 (C), 135.6 (C), 145.1 (C), 168.0 (C); CIMS m/z 484 (M + H⁺, 100), 287 (MH⁺ - O=C=N-SO₂tol, 37), 198 (O=C=N-SO₂-tol, 40), 174 (Ph(iBu)C=O=C, 75), 154 (SO₂tol, 29); HRMS (ESI⁺) [M + Na⁺] C₃₀H₂₉NNaO₃S requires 506.1766, found, 506.1768.

The β-lactam (±)-16 was obtained using isobutylphenylketene 13 (87.3 mg, 0.501 mmol, 1.3 equiv) and N-(2-furfurylidene)-4-methylbenzenesulfonamide (96.1 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour at room temperature (dr = 81:19 syn:anti) and the residue was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH₂Cl₂/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-16 (141.2 mg, 86% yield). Trituration in Et₂O gave a white solid (mp = 130-132°C, dr = 86:14 syn:anti). IR (KBr disk) ν̃ 3066, 2958, 2872, 2360, 1767 (C=O), 1597, 1497, 1468, 1449, 1367, 1242, 1187, 1170, 1089; ¹H NMR syn: δ 0.74 (3H, d, J = 6.8), 0.87 (3H, d, J = 6.8), 1.43-1.61 (1H, m), 1.95 (1H, dd, J = 14.0, 5.6), 2.01 (1H, dd, J = 14.4, 7.2), 2.41 (3H, s), 5.10 (1H, s), 6.11 (2H, d, J = 0.8), 6.88 (1H, s), 6.96-7.06 (2H, m), 7.07-7.17 (3H, t, J = 3.2), 7.21-7.23 (2H, m), 7.69 (2H, d, J = 8.4); anti (visible
peaks): $\delta$ 0.44 (3H, d, $J = 6.4$), 0.68 (3H, d, $J = 6.4$), 1.18-1.32 (1H, m), 1.73 (1H, dd, $J = 14.4, 7.6$), 2.43 (3H, s), 5.15 (1H, s), 6.39-6.51 (2H, m), 7.72 (2H, d, $J = 8.4$); $^{13}$C NMR syn: $\delta$ 21.7 (CH$_3$), 23.5 (CH$_3$), 23.8 (CH$_3$), 24.9 (CH), 47.6 (CH$_2$), 62.5 (CH), 68.4 (C), 110.4 (CH), 111.4 (CH), 126.5 (2 CH), 127.1 (CH), 127.5 (2 CH), 128.0 (2 CH), 129.7 (2 CH), 135.4 (C), 135.5 (C), 142.9 (CH), 145.0 (C), 147.3 (C), 167.3 (C); anti (visible peaks): 23.1, 24.5, 42.1, 62.7, 110.8, 111.2, 126.3, 126.4, 127.6, 127.7, 128.9, 129.8, 143.3.

**syn-4-(4-Bromophenyl)-3-isobutyl-3-phenyl-1-tosylazetidin-2-one (±)-17.** The β-lactam (±)-17 was obtained using isobutylphenylketene 13 (87.3 mg, 0.501 mmol, 1.3 equiv) and N-(4-bromobenzylidene)-4-methylbenzenesulfonamide (130.4 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 16 hours (dr = 57:43 syn:anti) at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH$_2$Cl$_2$/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-17 (186.4 mg, 94% yield). Trituration in Et$_2$O gave a white solid (mp = 184-186°C, dr = 82:18 syn:anti). IR (KBr disk) $\nu$ 3027, 2960, 2869, 2869, 2360, 1784 (C=O), 1596, 1487, 1468, 1448, 1407, 1362, 1231, 1168, 1139, 1087; $^1$H NMR syn: $\delta$ 0.67 (3H, d, $J = 6.4$), 0.87 (3H, d, $J = 6.8$), 1.47-1.61 (1H, m), 1.90 (1H, dd, $J = 14.4, 6.0$), 2.03 (1H, dd, $J = 14.4, 6.4$), 2.44 (3H, s), 4.93 (1H, s), 6.65 (2H, d, $J = 8.4$), 6.83-6.92 (2H, m), 7.01-7.09 (3H, m), 7.13 (2H, d, $J = 8.4$), 7.28 (2H, d, $J = 8.0$), 7.71 (2H, d, $J = 8.4$); anti (visible peaks): $\delta$ 0.41 (3H, d, $J = 6.4$), 0.71 (3H, d, $J = 6.4$), 4.96 (1H, s), 6.71 (2H, d, $J = 8.4$), 7.55 (2H, d, $J = 8.4$), 7.80 (2H, d, $J = 8.0$); $^{13}$C NMR syn: $\delta$ 21.8 (CH$_3$), 23.7 (CH$_3$), 23.8 (CH$_3$), 24.9 (CH), 47.4 (CH$_2$), 69.0 (CH), 69.4 (C), 122.5 (C), 127.3 (CH), 127.4 (2 CH), 127.6 (2 CH), 128.2 (2 CH), 129.6 (2 CH), 129.9 (2 CH), 131.1 (2 CH), 133.4 (C), 134.5 (C), 135.5 (C), 145.5 (C), 167.7 (C); anti (visible peaks): 22.9, 24.0, 24.6, 42.1, 69.3, 126.0, 127.8, 129.0, 129.4, 129.9, 131.9, 168.2; CIMS m/z 514 (MH$^+$ ($^{81}$Br), 28), 512 (MH$^+$ ($^{79}$Br), 32), 358 (M ($^{81}$Br) – SO$_2$tol, 6), 356 (M ($^{79}$Br) – SO$_2$tol, 6), 316 (M ($^{81}$Br) – O=C=N-SO$_2$tol, 4), 314 (M ($^{79}$Br) – Ph$_2$C=C=O, 4), 198 (O=C=N-SO$_2$tol, 20), 174.
syn-3-Isobutyl-3-phenyl-4-(2-phenylvinyl)-1-tosylazetidin-2-one (±)-18. The β-lactam (±)-18 was obtained using isobutylphenylketene 13 (87.3 mg, 0.501 mmol, 1.3 equiv) and N-(3-phenyl-allylidene)-4-methylbenzenesulfonamide (110.0 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 3 hours (dr = 76:24 syn:anti) at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH2Cl2/petroleum ether/EtOAc 50:45:5) to give β-lactam (±)-18 (128.4 mg, 72% yield). Trituration in Et2O gave a white solid (mp = 130-132°C, dr = 94:6 syn:anti). IR (KBr disk) ν 3085, 3060, 3028, 2928, 2871, 2360, 1775 (C=O), 1597, 1496, 1449, 1367, 1187, 1174, 1152, 1135, 1087; 1H NMR: δ 0.73 (3H, d, J = 6.4), 0.88 (3H, d, J = 6.8), 1.52-1.64 (1H, m), 1.91 (1H, dd, J = 14.0, 5.6), 1.99 (1H, dd, J = 14.0, 6.8), 2.38 (3H, s), 4.62 (1H, d, J = 9.6), 5.21 (1H, dd, J = 15.6, 9.6), 6.70 (1H, d, J = 16.0), 7.07 (2H, dd, J = 7.6, 3.6), 7.18-7.32 (10H, m), 7.79 (2H, d, J = 8.4); anti (visible peaks): δ 0.60 (3H, d, J = 6.8), 0.80 (3H, d, J = 6.4), 2.42 (3H, s), 6.16 (1H, dd, J = 16.0, 8.8), 6.80 (2H, d, J = 16.0); 13C NMR syn: δ 21.7 (CH3), 23.6 (CH3), 23.8 (CH3), 24.7 (CH), 47.2 (CH2), 67.4 (C), 68.8 (CH), 124.2 (CH), 126.8 (2 CH), 127.4 (2 CH), 127.5 (CH), 127.6 (3 CH), 128.5 (2 CH), 128.6 (2 CH), 129.9 (2 CH), 135.0 (C), 135.6 (C), 136.1 (C), 136.3 (C), 145.1 (C), 167.4 (C); anti (visible peaks): 122.4, 126.0, 126.9, 128.8, 128.9.
VII. Preparation of a C$_2$-symmetric imidazolinium salt

(1R,2R)-Bis(benzylidene)cyclohexyl-1,2-diamine. A mixture of (1R,2R)-trans-cyclohexane-1,2-diammonium (S)-tartrate$^5$ (1.00 g, 3.77 mmol), K$_2$CO$_3$ (1.05 g, 7.58 mmol) and H$_2$O (5 mL) were stirred until complete dissolution occurred, to which was then added EtOH (20 mL). The mixture was heated at reflux (80 °C) and a solution of benzaldehyde (0.77 mL, 7.58 mmol) in EtOH (8 mL) was added over 30 minutes. The mixture was heated at reflux for a further 2 hours, then when the solution had cooled to ambient temperature. Water (5 mL) was added and the mixture cooled to 0 °C for 3 hours. The mixture was concentrated in vacuo to afford the crude diimine. The residue was redissolved in CH$_2$Cl$_2$ (15 mL), washed with water (2 × 10 mL) and brine (10 mL), dried (Na$_2$SO$_4$), filtered and concentrated in vacuo to give the title compound (1.05 g, 96%) as a pale cream solid (mp = 133-135°C). $^1$H NMR: δ 1.45–1.57 (2H, m), 1.63–1.89 (6H, m), 3.36–3.41 (2H, m), 3.35–3.42 (2H, m), 7.28–7.36 (6H, m), 7.61 (4H, dd, $J = 7.6, 2.1$), 8.20 (2H, s); [$\alpha$]$^20_D$ -261.4 (c 0.05, CH$_3$OH), lit. -263.0 (c 0.19, CH$_3$OH). Data are in accordance with the literature.$^6,^7$

(1R,2R)-Dibenzylcyclohexyl-1,2-diamine. To a solution of (1R,2R)-bis(benzylidene)cyclohexyl-1,2-diimine (1.05 g, 3.62 mmol) in MeOH (8 mL) was added NaBH$_4$ (287 mg, 7.59 mmol) portionwise over 30 minutes. The solution was heated at reflux for 15 minutes then cooled to ambient temperature. Water (8 mL) was added and the mixture extracted with CH$_2$Cl$_2$ (3 × 10 mL). The organics were combined, washed with brine (10 mL), dried (MgSO$_4$), filtered and concentrated in vacuo to give the title compound (1.00 g, 94%) as a colourless oil. $^1$H NMR: δ: 1.45–1.57 (2H, m), (1.63–1.89 (6H, m), (1.78 (2H, br s), 3.36–3.41 (2H, m), 3.58 (2H, q, $J = 13.2$), 3.82 (2H, q, $J = 13.2$), 7.10–7.31 (10H, m); [$\alpha$]$^20_D$ -69.2

(3aR,7aR)-1,3-Dibenzyl-3a,4,5,6,7,7a-hexahydro-1H-benzo[d]imidazolium tetrafluoroborate 19. A mixture of (1R,2R)-dibenzylecyclohexyl-1,2-diamine (500 mg, 1.70 mmol), triethyl orthoformate (0.70 mL, 4.25 mmol), MeOH (5 mL) and ammonium tetrafluoroborate (180 mg, 1.72 mmol) were heated at 110 ºC for 5 hours. Hexane (15 mL) was added to the mixture to precipitate the product as a colourless solid. Recrystallisation from CH₂Cl₂ afforded 19 (550 mg, 82%) as a colourless solid (mp = 150-152°C). [α]²⁰D -66.8 (c 0.05, MeOH); IR (KBr disk): 3108 (=C-H), 3072 (=C-H), 1611 (conj cyclic C=N), 1586 (conj cyclic C=N), 1062 (C-N stretch) and 1059 (C-N stretch); ¹H NMR (CD₃OD) δ: 1.28–1.03 (4H, m), 1.66–1.64 (2H, m), 1.98–1.95 (2H, m), 3.31–3.18 (2H, m), 4.63 (4H, dd, J = 15.1, 6.9), 7.33–7.24 (10H, m), 8.57 (1H, s); ¹³C NMR (CD₃OD) δ: 24.5 (2 CH₂), 28.5 (2 CH₂), 51.5 (2 CH₂), 69.0 (2 CH), 129.4 (2 CH), 129.7 (CH), 130.1 (2 CH), 134.8 (C), 161.9 (CH); HRMS (ESI⁺) [M⁺] C₂₁H₂₅N₂⁺ requires 305.2012, found 305.2019.

VIII. General procedure for the enantioselective synthesis of β-lactams (R)-5, (R)-8, (S)-9 and (R)-11 (Figure 6).

A 0.45 M solution of KHMDS in toluene (0.08 mL, 0.0347 mmol, 9 mol%) was added to a suspension of chiral imidazolinium salt 19 (15.2 mg, 0.0386 mmol, 10 mol%) or chiral triazolium salt 20 (14.5 mg, 0.0386 mmol, 10 mol%) in Et₂O (1.5 mL) under nitrogen atmosphere and the mixture was stirred for 30 minutes at room temperature. A solution of diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) in Et₂O (1.5 mL) was added, followed by the imine (0.386 mmol, 1.0 equiv) as a solid, then the reaction mixture was stirred at room temperature before concentration.

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**(R)-3,3,4-Triphenyl-1-tosylazetidin-2-one (R)-5.** The β-lactam (R)-5 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-benzylidene-4-methylbenzenesulfonamide 4 (100 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour (using the NHC from 19) or 3 hours (using the NHC from 20) at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH₂Cl₂/petroleum ether/EtOAc 50:45:5) to give β-lactam (R)-5 as a yellowish solid (158.0 mg, 90% yield, from chiral imidazolinium salt 19 and 167.2 mg, 96% yield from chiral triazolium salt 20). HPLC analysis: 64% ee (Daicel CHIRALCEL OD-H column, eluent: hexane/PrOH 90:10, flow: 1 mL/min, wavelength: 254 nm, retention times: 9.4 min (major, R) and 16.5 min (minor, S)). $[\alpha]_{D}^{20} + 17.7$ (c 1.00, CHCl₃, 64% ee). The initial e.e. can be improved by crystallisation (CH₂Cl₂/Hexane) and re-isolation of the β-lactam from the mother liquor, giving (R)-5 as a further enantiomerically enriched product (mp = 152-154°C, 98% e.e.). $[\alpha]_{D}^{20} + 22.0$ (c 0.63, CHCl₃, 98% ee).

**HPLC trace (±)-5**

![HPLC Trace](image)
HPLC trace (R)-5 (64% ee)

HPLC trace (R)-5 (98% ee)
(R)-3,3-Diphenyl-4-(2-naphthyl)-1-tosylazetidin-2-one  (R)-8.

The β-lactam (R)-8 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-(2-naphthylidene)-4-methylbenzenesulfonamide (119.3 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour (using the NHC from 19) or 3 hours (using the NHC from 20) at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH2Cl2/petroleum ether/EtOAc 50:45:5) to give β-lactam (R)-8 as a yellowish solid (184.1 mg, 95% yield, from chiral imidazolinium salt 19 and 178.5 mg, 92% yield from chiral triazolium salt 20). HPLC analysis: 75% ee (Daicel CHIRALCEL OD-H column, eluent: hexane/iPrOH 90:10, flow: 1 mL/min, wavelength: 254 nm, retention times: 10.0 min (major, R) and 20.5 min (minor, S)). $[\alpha]^{20}_D - 5.0$ (c 1.00, CHCl3, 75% ee). The initial e.e. can be improved by crystallisation (CH2Cl2/Hexane) and re-isolation of the β-lactam from the mother liquor, giving (R)-8 as a further enantiomerically enriched product (mp = 136-138°C, >99% e.e.). $[\alpha]^{20}_D - 5.9$ (c 0.69, CHCl3, >99% ee).

HPLC trace (±)-8
HPLC trace (R)-8 (75% ee)

HPLC trace (R)-8 (>99% ee)
(S)-3,3-Diphenyl-4-(2-furanyl)-1-tosylazetidin-2-one (S)-9. The β-lactam (S)-9 was obtained using diphenylketene 3 (97.4 mg, 0.501 mmol, 1.3 equiv) and N-(2-furfurylidene)-4-methylbenzenesulfonamide (96.1 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 1 hour (using the NHC from 19) or 3 hours (using the NHC from 20) at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH₂Cl₂/petroleum ether/EtOAc 50:45:5) to give β-lactam (S)-9 as a yellowish solid (146.0 mg, 85% yield, from chiral imidazolinium salt 19 and 158.8 mg, 93% yield from chiral triazolium salt 20). HPLC analysis: 61% ee (Daicel CHIRALCEL OD-H column, eluent: hexane/iPrOH 90:10, flow: 1 mL/min, wavelength: 254 nm, retention times: 8.5 min (major, S) and 10.6 min (minor, R)). [α]_D^{20} + 32.6 (c 1.00, CHCl₃, 61% ee). The initial e.e. can be improved by crystallisation (CH₂Cl₂/Hexane) and re-isolation of the β-lactam from the mother liquor, giving (S)-9 as a further enantiomerically enriched product (mp = 144-146°C, 92% e.e.). [α]_D^{20} + 45.7 (c 0.91, CHCl₃, 92% ee).

**HPLC trace (±)-9**

![HPLC trace image]
HPLC trace (S)-9 (61% ee)

HPLC trace (S)-9 (92% ee)
The β-lactam (R)-11 was obtained using diphenylketene (97.4 mg, 0.501 mmol, 1.3 equiv) and N-(4-bromobenzylidene)-4-methylbenzenesulfonamide (130.4 mg, 0.386 mmol, 1.0 equiv) and following the above general procedure. The reaction mixture was concentrated after stirring for 2 hours (using the NHC from 19) or 3 hours (using the NHC from 20) at room temperature and the residue was purified by column chromatography (EtOAc/petroleum ether 10:90 then CH$_2$Cl$_2$/petroleum ether/EtOAc 50:45:5) to give β-lactam (R)-11 as a yellowish solid (163.2 mg, 79% yield, from chiral imidazolinium salt 19 and 197.8 mg, 96% yield from chiral triazolium salt 20). HPLC analysis: 57% ee (Daicel CHIRALCEL OD-H column, eluent: hexane/PrOH 90:10, flow: 1 mL/min, wavelength: 254 nm, retention times: 8.8 min (major, R) and 21.1 min (minor, S)). [α]$^{20}_D$ + 21.8 (c 1.00, CHCl$_3$, 57% ee). The initial e.e. can be improved by crystallisation (CH$_2$Cl$_2$/Hexane) and re-isolation of the β-lactam from the mother liquor, giving (R)-11 as a further enantiomERICALLY enriched product (mp = 138-140°C, >99% e.e.). [α]$^{20}_D$ + 25.2 (c 0.44, CHCl$_3$, >99% ee).

HPLC trace (±)-11
HPLC trace \((R)-11\) (57\% ee)

HPLC trace \((R)-11\) (>99\% ee)
IX. Spectral Data

[Diagram of spectral data]