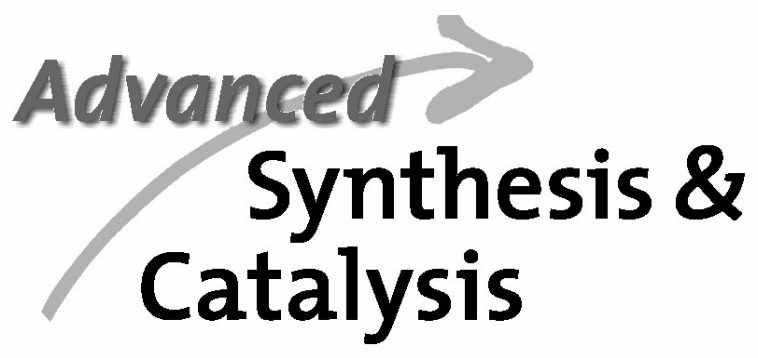

Supporting Information

Synthesis of β-Lactams by 4-Exo-Tet Cyclization Process induced by electrogenerated cyanomethyl anion, Part 2 [1]: Stereochemical Implications

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Supporting Information

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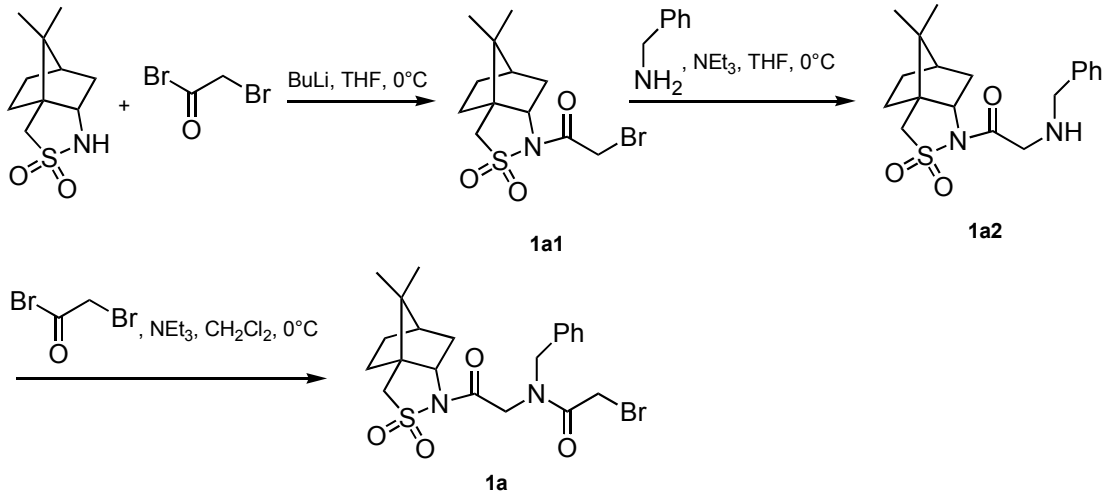
General remarks

Constant current electrolyses were performed under an argon atmosphere, using an Amel Model 552 potentiostat equipped with an Amel Model 731 integrator. All the experiments were carried out in a divided glass cell separated through a porous glass plug filled up with a layer of agar gel (i.e., methyl cellulose 0.5% vol dissolved in DMF-Et_4NClO_4 1.0 mol dm^{-3}); Pt spirals (apparent areas 0.8 cm^2) were used both as cathode and anode. MeCN-Et_4NPF_6 0.1 mol dm^{-3} was used as solvent-supporting electrolyte system (catholite: 20 cm^3; anolite: 5 cm^3).
Flash column chromatography was carried out using Merck 60 kieselgel (230-400 mesh) under pressure. Optical rotations were recorded on a Perkin-Elmer 241 polarimeter. GC-MS measurements were carried out on a SE 54 capillary column using a Fisons 8000 gas chromatograph coupled with a Fisons MD 800 quadrupole mass selective detector. ^1H and ^{13}C NMR spectra were recorded at room temperature using a Bruker AC 200 spectrometer using $CDCl_3$ as internal standard. Where a compound has been characterised as an inseparable mixture of diastereoisomes, the NMR data for the major and minor isomer have been reported as far as was discernable from the spectrum of the mixture.

Starting materials

Acetonitrile was distilled twice from P_2O_5 and CaH_2. Chiral amides **1a-j** were synthesized following literature methods (**1a** and **1f**,[1,2] **1b** and **1g**,[3,2] **1c-e** and **1h-j**[2]) and obtained as a mixture of two rotamers (**1a-e**) or as a mixture of two diastereoisomers and rotamers (**1f-j**). All products were isolated and purified by flash column chromatography using a *n*-hexane/ethyl acetate mixture (95/5 to 7/3) as eluent.

(2*R*)-(-)-Bornane-2,10-sultamyl 2-(*N*-benzyl-2-bromoacetamido) acetate (1a).



1a1 **1a2** **1a**

This compound was obtained by reaction of (2*R*)-(-)-bornane-2,10-sultam with 2-bromoacetyl bromide (obtaining 1-[(2*R*)-(-)-bornane-2,10-

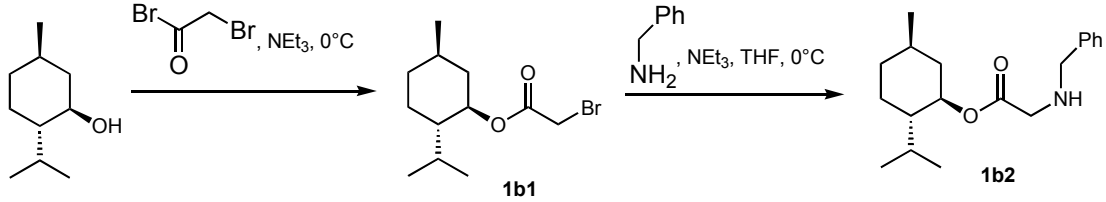
sultamyl]-2-bromoethanone **1a1**), followed by reaction with benzylamine (obtaining 2-(benzylamino)-1-((2*R*)-(-)-bornane-2,10-sultamyl)ethanone **1a2**) and then with 2-bromoacetyl bromide, yelding product **1a**.

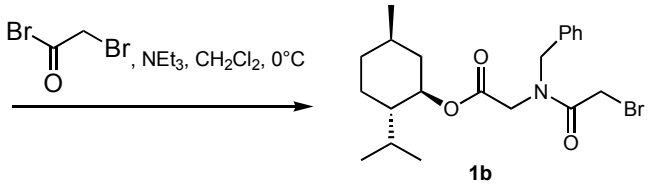
1a1: ^{1}H NMR ($CDCl_3$): δ=4.31 (d, AB, Δν=26.2 Hz, *J*=12.9 Hz, 1H), 4.18 (d, AB, Δν=26.2 Hz, *J*=12.9 Hz, 1H), 3.89 (dd, *J*=7.1-5.5 Hz, 1H), 3.50 (d, AB, Δν=11.6 Hz, *J*=13.8 Hz, 1H), 3.45 (d, AB, Δν=11.6 Hz, *J*=13.8 Hz, 1H), 2.16-2.07 (m, 2H), 1.95-1.81 (m, 3H), 1.42-1.34 (m, 2H), 1.13 (s, 3H), 0.96 (s, 3H). ^{13}C NMR ($CDCl_3$): δ=164.5, 65.4, 52.7, 48.9, 47.8, 44.5, 37.9, 32.8, 27.4, 26.4, 20.7, 19.8. GC-MS: *m/z*=$M^{+\cdot}$ absent, 258 (2%), 256 (3%), 254 (3%), 252 (3%), 214 (2%), 192 (42%), 135 (71%), 134 (100%), 132 (57%). α_D= -90.0 (c=0.071, $CHCl_3$).

1a2: ^{1}H NMR ($CDCl_3$): δ=7.31-7.22 (m, 5H), 3.91-3.70 (m, 5H), 3.45 (d, AB, *J*=13.9 Hz, Δν=9.5 Hz, 1H), 3.41 (d, AB, *J*=13.9 Hz, Δν=9.5 Hz, 1H), 2.24 (bs, 1H), 2.10-2.00 (m, 2H), 1.93-1.87 (m, 3H), 1.45-1.27 (m, 2H), 1.11 (s, 3H), 0.94 (s, 3H). ^{13}C NMR ($CDCl_3$): δ=170.9, 139.3, 128.4, 128.3, 127.1, 65.1, 53.3, 52.7, 51.5, 49.0, 47.8, 44.6, 38.3, 32.8, 26.1, 20.8, 19.8. α_D= -68.3 (c=0.145, CH_3OH).

1a, mixture of two rotamers: ^{1}H NMR ($CDCl_3$): δ=7.36-7.24 (m, 5H), 4.87-4.18 (m, 4H), 3.92-3.83 (m, 3H), 3.49-3.33 (m, 2H), 2.15-1.86 (m, 4H), 1.65-1.48 (m, 3H), 1.43-1.34 (m, 3H), 1.23 (s, 1.1H), 1.13 (s, 1.9H), 1.04 (s, 1.1H), 0.94 (s, 1.9H). ^{13}C NMR ($CDCl_3$): δ= major rotamer: 167.3, 166.5, 135.1, 128.9, 128.5, 127.0, 65.0, 52.5, 50.6, 50.1, 49.2, 49.1, 47.7, 44.5, 38.0, 32.6, 26.3, 20.7, 19.7; minor rotamer: 167.5, 166.9, 135.7, 128.6, 128.1, 127.7, 65.1, 53.4, 50.6, 50.1, 49.2, 49.1, 48.9, 44.5, 37.9, 32.6, 25.7, 20.7, 19.7. Anal. Calcd for $C_{21}H_{27}BrN_2O_4S$: C, 52.18; H, 5.63; N, 5.79; S, 6.63. Found: C, 52.26; H, 5.71; N, 5.76; S, 6.61.

(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl 2-(*N*-benzyl-2-bromo acetamido)acetate (1b).





This compound was obtained by reaction of (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexanol with 2-bromoacetyl bromide (obtaining (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 2-bromoacetate **1b1**), followed by reaction with benzylamine (obtaining (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 2-(benzylamino)acetate **1b2**) and then with 2-bromoacetyl bromide, yelding product **1b**.

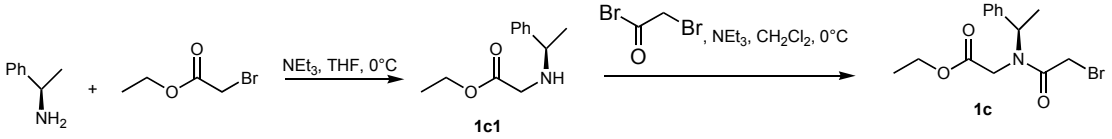
1b1: ^{1}H NMR ($CDCl_3$): δ=4.69 (dt, *J*=10.9-4.4 Hz, 1H), 3.78 (s, 2H), 2.00-1.83 (m, 2H), 1.69-1.63 (m, 2H), 1.47-1.34 (m, 2H), 1.11-0.82 (m, 9H), 0.73 (d,

J=7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ=166.6, 76.2, 46.8, 40.3, 34.0, 31.2, 26.1, 26.0, 23.2, 21.8, 20.6, 16.1. GC-MS: *m/z*= $M^{+\cdot}$ absent, 138 (25%), 95 (100%), 81 (76%), 43 (4%). α_D= -64.9 (c=0.092, $CHCl_3$).

1b2: ^{1}H NMR ($CDCl_3$): δ=7.33-7.26 (m, 5H), 4.74 (dt, *J*=10.8-4.4 Hz, 1H), 3.79 (s, 2H), 3.37 (s, 2H), 2.03-1.62 (m, 6H), 1.48-1.23 (m, 3H), 1.12-0.73 (m, 10H). ^{13}C NMR ($CDCl_3$): δ=171.9, 139.4, 128.3, 128.2, 127.0, 74.6, 53.2, 50.2, 46.9, 48.9, 34.1, 31.3, 26.2, 23.3, 21.9, 20.6, 16.2. GC-MS: *m/z*= $M^{+\cdot}$ absent, 164 (8%), 120 (69%), 106 (26%), 91 (100%). α_D= -56.7 (c=0.067, $CHCl_3$).

1b, mixture of two rotamers: ^{1}H NMR($CDCl_3$): δ=7.4-7.18 (m, 5H), 4.77-4.64 (m, 3H), 4.15-3.85 (m, 4H), 2.02-1.62 (m, 4H), 1.45-1.18 (m, 4H), 1.11-0.71 (m, 10H). ^{13}C NMR ($CDCl_3$): major rotamer: δ=168.1, 167.3, 135.2, 129.0, 128.3, 127.1, 75.5, 53.0, 49.1, 46.9, 40.7, 34.1, 31.3, 26.4, 26.1, 23.3, 21.9, 20.7, 16.2; minor rotamer: 167.4, 167.3, 135.8, 128.7, 128.2, 127.8, 76.2, 53.0, 50.1, 47.4, 40.7, 34.0, 31.3, 26.4, 25.7, 23.3, 20.9, 20.7, 16.2. Anal. Calcd for $C_{21}H_{30}BrNO_3$: C, 59.43; H, 7.13; N, 3.30. Found: C, 59.61; H, 7.23; N, 3.27.

Ethyl 2-(2-bromo-*N*-((*R*)-1-phenylethyl)acetamido)acetate (1c).

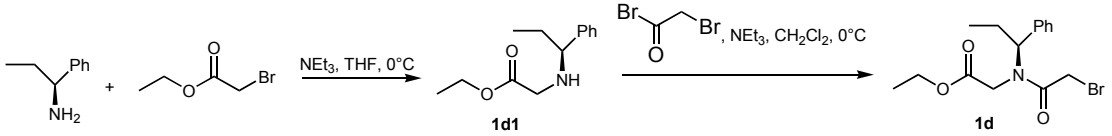


This compound was obtained by reaction of (*R*)-1-phenylethanamine with ethyl bromoacetate (obtaining ethyl 2-((*R*)-1-phenylethylamino)acetate **1c1**), followed by reaction with 2-bromoacetyl bromide, yielding product **1c**.

1c1: ^{1}H NMR ($CDCl_3$): δ=7.31-7.19 (m, 5H), 4.13 (q, *J*=7.1 Hz 2H), 3.77 (q, *J*=6.5 Hz 1H), 3.26 (d, AB, Δν=10.3 Hz, *J*=17.5 Hz, 1H), 3.21 (d, AB, Δν=10.3 Hz, *J*=17.5 Hz, 1H), 1.89 (s, 1H), 1.37 (d, *J*=6.5 Hz, 1H), 1.22 (t, *J*=7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ=172.4, 144.4, 128.4, 127.0, 126.6, 60.5, 57.6, 48.7, 24.0, 14.0. GC-MS: *m/z*= $M^{+\cdot}$ absent, 192 (9%, $M^{+\cdot}$ -CH_3), 134 (16%), 120 (31%), 105 (100%). α_D=+68.1 (c=0.091, $CHCl_3$).

1c, mixture of two rotamers: ^{1}H NMR ($CDCl_3$): δ=7.40-7.25 (m, 5H), 5.99 (q, *J*=7.0 Hz, 0.4H), 5.24 (q, *J*=6.9 Hz, 0.6H), 4.14-3.96 (m, 5.4H), 3.45 (d, *J*=17.0 Hz, 0.6H), 1.66 (d, *J*=6.9 Hz, 1.6H), 1.43 (d, *J*=7.0 Hz, 1.4H), 1.19 (t, *J*=7.0 Hz, 1.6H), 1.16 (t, *J*=7.1 Hz, 1.4H). ^{13}C NMR ($CDCl_3$) major rotamer: δ=168.6, 166.9, 139.1, 128.8, 127.8, 127.6, 61.1, 56.4, 25.6, 17.9, 15.6, 14.0; minor rotamer: 169.4, 167.4, 139.1, 128.5, 128.0, 126.8, 61.7, 51.7, 27.0, 17.9, 15.6, 14.0. GC-MS: *m/z*= $M^{+\cdot}$ absent, 248 (15%, $M^{+\cdot}$ – Br), 206 (12%), 192 (3%), 119 (10%), 105 (100%). Anal. Calcd for $C_{14}H_{18}BrNO_3$: C, 51.23; H, 5.53; N, 4.27. Found: C, 51.27; H, 5.56; N, 4.26.

Ethyl 2-(2-bromo-*N*-((*S*)-1-phenylpropyl)acetamido)acetate (1d).

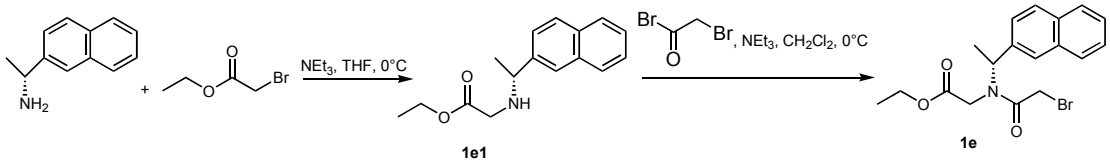


This compound was obtained by reaction of (*S*)-1-phenylpropan-1-amine with ethyl bromoacetate (obtaining ethyl 2-((*S*)-1-phenylpropylamino)acetate **1d1**), followed by reaction with 2-bromoacetyl bromide, yielding product **1d**.

1d1: ^{1}H NMR ($CDCl_3$): δ=7.33-7.16 (m, 5H), 4.11 (q, *J*=7.2 Hz, 2H), 3.48 (dd, *J*=7.5 Hz; 6.1 Hz, 1H), 3.23 (d, AB, Δν=15.9 Hz; *J*=17.4 Hz, 1H), 3.15 (d, AB, Δν=15.9 Hz; *J*=17.4 Hz, 1H), 2.01 (s, 1H), 1.52-1.56 (m, 2H), 1.20 (t, *J*=7.2 Hz, 3H), 0.79 (t, *J*=7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ=172.6, 143.0, 128.2, 127.4, 127.1, 64.4, 60.5, 48.7, 30.9, 14.1, 10.6. GC-MS: *m/z*= $M^{+\cdot}$ absent, 192 ($M^{+\cdot}$-CH_2CH_3, 50%), 164 (6%), 118 (58%), 91 (100%). α_D= - 64.8 (c=0.086, $CHCl_3$).

1d, mixture of two rotamers: ^{1}H NMR ($CDCl_3$): δ=7.46-7.17 (m, 5H), 5.90 (dd, *J*=8.7- 6.8 Hz, 0.6 H), 4.89 (t, *J*= 7.4, 0.4H), 4.28-3.55 (m, 6H), 2.16-2.02 (m, 0.8H), 2.01-1.73 (m, 1.2H), 1.36-0.68 (m, 6H). ^{13}C NMR.($CDCl_3$): major rotamer: δ= 169.0, 167.9, 137.9, 128.4, 128.2, 127.7, 61.5, 57.7, 45.2, 27.2, 22.8, 13.9, 10.7; minor rotamer: 168.4, 167.0, 137.6, 128.7, 128.4, 127.8, 62.8, 60.9, 44.7, 29.6, 24.8, 13.9, 11.3. GC-MS: *m/z*= $M^{+\cdot}$ absent, 262 ($M^{*\cdot}$- Br, 4%), 220 (3%), 192 (12%), 146 (2%), 118 (15%), 91 (100%). Anal. Calcd for $C_{15}H_{20}BrNO_3$: C, 52.64; H, 5.89; N, 4.09. Found: C, 52.84; H, 5.91; N, 4.05.

Ethyl 2-(2-bromo-*N*-((*R*)-1-(naphthalen-3-yl)ethyl)acetamido) acetate (1e).

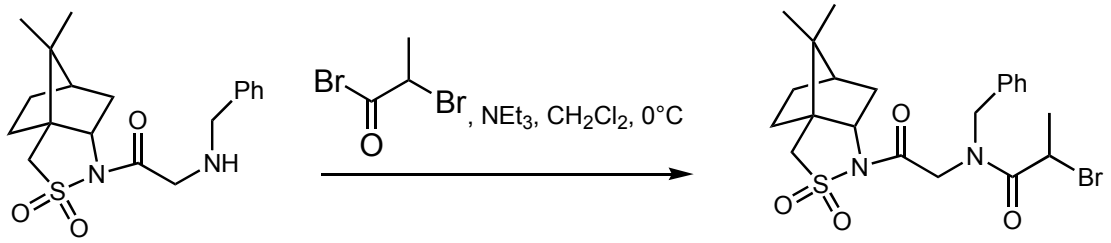


This compound was obtained by reaction of (*R*)-1-(naphthalen-6-yl)ethanamine with ethyl bromoacetate (obtaining ethyl 2-((*R*)-1-(naphthalen-6-yl)ethylamino)acetate **1e1**), followed by reaction with 2-bromoacetyl bromide, yielding product **1e**.

1e1: ^{1}H NMR($CDCl_3$): δ=7.83-7.78 (m, 3H), 7.72 (s, 1H), 7.49-7.41 (m, 3H), 4.13 (q, *J*=7.1 Hz, 2H), 3.95 (q, *J*=6.5 Hz, 1H), 3.29 (d, AB, Δν=10.0 Hz, *J*=17.5 Hz, 1H), 3.24 (d, AB, Δν=10.0 Hz, *J*=17.5 Hz, 1H), 2.21 (s, 1H), 1.45 (d, *J*=6.5 Hz, 3H), 1.21 (t, *J*=7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ=172.5, 142.0, 133.4, 132.9, 128.3, 127.7, 127.6, 125.9, 125.5, 125.4, 124.8, 60.6, 57.8, 48.8, 24.1, 14.1. α_D= + 64.5 (c=0.108, $CHCl_3$).

1e, mixture of two rotamers: ^{1}H NMR ($CDCl_3$): δ= 7.83-7.71 (m, 4H), 7.51-7.32 (m, 3H), 6.16 (q, *J*=7.0 Hz, 0.5H), 5.37 (q, *J*=6.8Hz, 0.5H), 4.12-3.44 (m, 6H), 1.77 (d, *J*=6.8 Hz, 1.5H), 1.55 (d, *J*=7.0 Hz, 1.5H), 1.16 (t, *J*=7.1 Hz, 1.5H), 1.03 (t, *J*=7.1 Hz, 1.5H). ^{13}C NMR ($CDCl_3$): δ= major rotamer: 169.3,167.5, 136.6, 132.7, 128.3, 127.9, 127.5, 127.2, 126.2, 125.9, 125.3, 125.0, 61.6, 51.7, 45.3, 27.1, 15.5, 13.9; minor rotamer: 168.5, 166.8, 136.4, 133.0, 128.6, 127.8, 127.4, 126.4, 126.2, 125.9, 125.3, 125.0, 60.9, 56.4, 44.6, 25.7, 17.9, 13.7. Anal. Calcd for $C_{18}H_{20}BrNO_3$: C, 57.15; H, 5.33; N, 3.70. Found: C, 57.10; H, 5.28; N, 3.68.

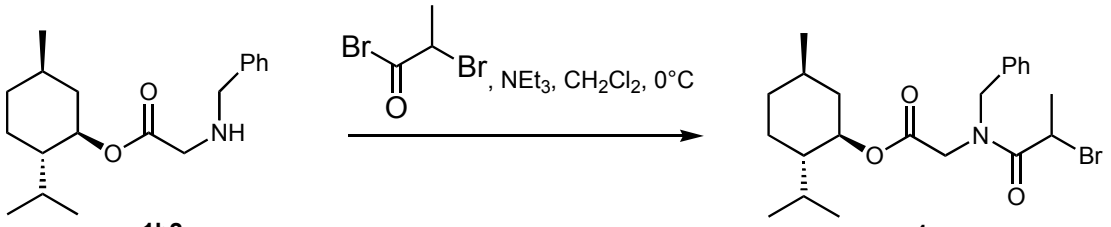
(2*R*)-(-)-Bornane-2,10-sultamyl 2-(*N*-benzyl-2-bromopropanamido) acetate (1f).



1a2 **1f**

This compound was obtained by reaction of 2-(benzylamino)-1-((2*R*)-(-)-bornane-2,10-sultamyl)ethanone **1a2** with 2-bromopropionyl bromide.
1f, mixture of two diastereomers and rotamers: ^{1}H NMR ($CDCl_3$): δ= 7.35-7.16 (m, 5H), 5.13-4.08 (m, 5H), 3.89-3.81 (m, 1H), 3.54-3.33 (m, 2H), 2.15-1.79 (m, 7H), 1.41-1,25 (m, 3H), 1.12 (s, 1.5H), 1.11 (s, 0.7H), 0.99 (s, 0.7H), 0.96 (s, 0.6H), 0.93 (s, 2.5H). ^{13}C NMR ($CDCl_3$) δ=: 170.3, 170.2, 170.1, 167.2, 166.6, 166.5, 136.1, 135.9, 135.5, 135.4, 129.0, 128.9, 128.6, 128.5, 128.3, 128.0, 127.7, 127.6, 127.0, 126.8, 65.1, 65.0, 64.9, 52.9, 52.8, 52.6, 50.9, 50.4, 49.8, 49.7, 49.3, 49.2, 49.2, 49.1, 47.8, 47.7, 44.6, 38.7, 38.5, 38.1, 38.0, 37.9, 37.7, 32.6, 26.4, 26.3, 21.8, 21.7, 21.5, 20.8, 19.8, 19.7. Anal. Calcd for $C_{22}H_{29}BrN_2O_4S$: C, 53.12; H, 5.88; N, 5.63; S, 6.45. Found: C, 53.16; H, 5.91; N, 5.67; S, 6.45.

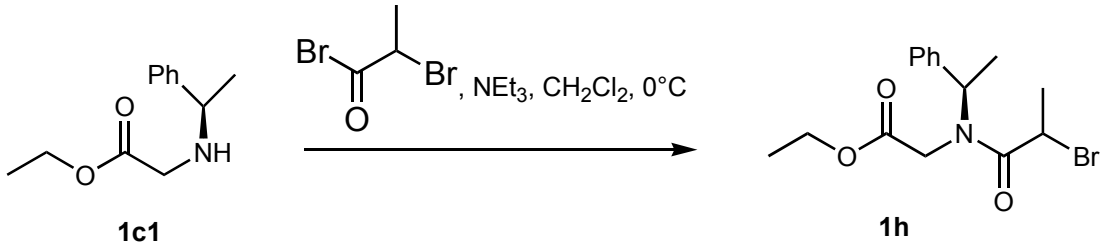
(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl 2-(*N*-benzyl-2-bromo propanamido)acetate (1g).



1b2 **1g**

This compound was obtained by reaction of (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 2-(benzylamino)acetate **1b2** with 2-bromopropionyl bromide.
1g, mixture of two diastereomers and rotamers: ^{1}H NMR ($CDCl_3$): δ= 7.39-7.18 (m, 5H), 5.07-3.71 (m, 6H), 2.15-2.02 (m, 1H), 1.94-1.61 (m, 6H), 1.40-1.20 (m, 4H), 1.10-0.68 (m, 10H). ^{13}C NMR ($CDCl_3$): δ=169.8, 168.4, 168.3, 168.1, 136.0, 135.6, 135.5, 128.9, 128.6, 128.1, 128.0, 127.6, 126.8, 126.7, 76.1, 76.0, 75.4, 75.3, 52.4, 50.2, 48.7, 47.8, 46.8, 40.7, 40.6, 38.4, 37.7, 34.1, 33.9, 31.3, 26.4, 26.3, 26.1, 25.9, 23.3, 23.2, 21.8, 21.6, 21.4, 20.6, 16.2, 16.1, 16.1. Anal. Calcd for $C_{22}H_{32}BrNO_3$: C, 60.27; H, 7.36; N, 3.19. Found: C, 60.26; H, 7.32; N, 3.18.

Ethyl 2-(2-bromo-*N*-((*R*)-1-phenylethyl)propanamido)acetate (1h).

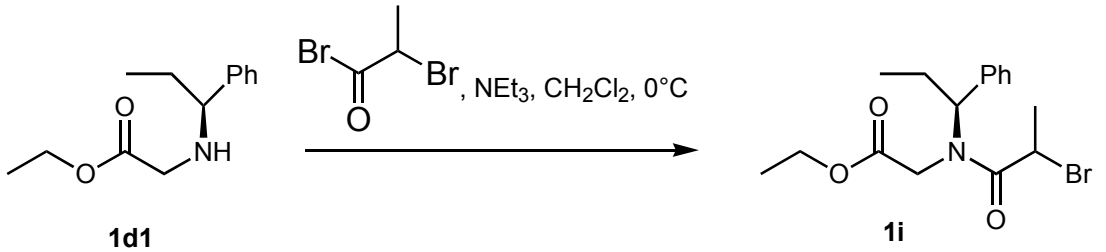


1c1 **1h**

This compound was obtained by reaction of ethyl 2-((*R*)-1-phenylethylamino)acetate **1c1** with 2-bromopropionyl bromide.

1h, mixture of two diastereomers and rotamers: ^{1}H NMR ($CDCl_3$): δ=7.42-7.19 (m, 5H), 6.11-5.95 (m, 0.5H), 5.44-5.25 (m, 0.5H), 4.77-4.59 (m. 0.5H), 4.39-3.32 (m, 4.5H), 1.90-1.80 (m, 3H), 1.67 (d, *J*=7.2 Hz, 0.6H), 1.64 (d, *J*=7.2Hz, 0.8 H), 1.45 (d, *J*=7.3 Hz, 0.9H), 1.41 (d, *J*=7.3 Hz, 0.7 H), 1.25 (t, *J*=7.2 Hz, 1H), 1.22 (t, *J*=7.1 Hz, 1.3H), 0.98 (t, *J*=7.0 Hz, 0.7H). ^{13}C NMR ($CDCl_3$): δ=173.1, 170.3, 170.0, 169.9, 169.7, 168.9, 168.8, 168.6, 139.6, 139.6, 138.2, 138.0, 128.8, 128.6, 128.5, 128.3, 128.1, 128.0, 127.8, 127.5, 127.3, 126.9, 126.1, 61.8, 61.4, 61.0, 55.5, 55.2, 52.1, 51.3, 45.1, 44.9, 44.8, 44.7, 39.8, 39.7, 39.3, 37.6, 21.7, 21.5, 21.4, 21.2, 21.1, 18.0, 17.5, 15.5, 15.0, 13.9, 13.8, 13.7. GC-MS: *m/z*= $M^{+\cdot}$ absent, 262 ($M^{+\cdot}$- Br, 26%), 206 (19%), 192 (5%), 133 (15%), 105 (100%). Anal. Calcd for $C_{15}H_{20}BrNO_3$: C, 52.64; H, 5.89; N, 4.09. Found: C, 52.81; H, 5.99; N, 4.05.

Ethyl 2-(2-bromo-*N*-((*S*)-1-phenylpropyl)propanamido)acetate (1i).

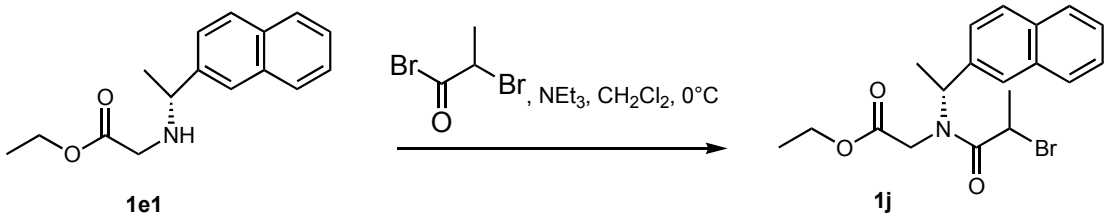


1d1 **1i**

This compound was obtained by reaction of ethyl 2-((*S*)-1-phenylpropylamino)acetate **1d1** with 2-bromopropionyl bromide.

1i, mixtures of two diastereomers and rotamers: ^{1}H NMR ($CDCl_3$): δ=7.48-7.26 (m, 5H), 8.89-5.78 (m, 0.6H), 5.07-4.89 (m, 0.4H), 4.84-4.75 (m, 0.4H), 4.43-4.34 (m, 0.6H), 4.31-3.40 (m, 4H), 2.31-1.59 (m, 5H), 1.29-0.91 (m, 6H). ^{13}C NMR ($CDCl_3$): δ=170.6, 170.5, 169.9, 168.9, 168.7, 168.5, 138.8, 137.7, 137.5, 128.9, 128.6, 128.2, 128.1, 127.9, 127.8, 127.7, 127.2, 61.8, 61.6, 61.4, 61.0, 60.9, 58.0, 57.4, 45.1, 44.8, 44.5, 39.8, 39.7, 37.8, 37.5, 24.6, 22.9, 22.8, 21.9, 21.8, 21.6, 21.4, 21.3, 14.0, 13.8, 11.4, 11.2, 11.0, 10.4. GC-MS: *m/z*= $M^{+\cdot}$absent, 276 ($M^{+\cdot}$-Br 37%), 220 (14%), 192 (55%), 119 (42%), 91 (100%). Anal. Calcd for $C_{16}H_{22}BrNO_3$: C, 53.94; H, 6.22; N, 3.93. Found: C, 54.03; H, 6.27; N, 3.91.

Ethyl 2-(2-bromo-*N*-((*R*)-1-(naphthalen-3-yl)ethyl)propanamido)acetate (1j).



1e1 **1j**

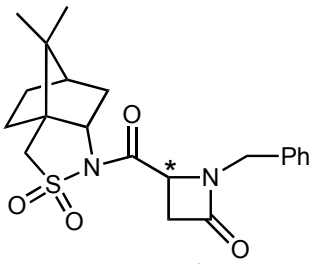
This compound was obtained by reaction of ethyl 2-((*R*)-1-(naphthalen-6-yl)ethylamino)acetate **1e1** with 2-bromopropionyl bromide.

1j, mixtures of two diastereomers and rotamers: ^{1}H NMR ($CDCl_3$):δ =7.84-7.78 (m, 3H), 7.69-7.65 (m, 1H), 7.51-7.45 (m, 2H), 7.37-7.32 (m, 1H), 6.22 (q, *J*=7.0 Hz, 0.7H), 5.45 (q, *J*=6.8 Hz, 0.3H), 4.75 (q, *J*=6.6 Hz, 0.3H), 4.32 (q, *J*=6.4 Hz, 0.7H), 4.25-4.03 (m, 3H), 3.66-3.51 (m, 1H), 1.92-1.81 (m, 3H), 1.78 (d, *J*=6.8 Hz, 0.9H), 1.53 (d, *J*=7.0 Hz, 2.1H), 1.28-1.16 (m, 3H). ^{13}C NMR ($CDCl_3$): δ=170.1, 169.9, 169.4, 168.5, 137.4, 133.0, 132.6, 128.6, 128.5, 127.8, 127.4, 126.5, 126.3, 126.1, 126.0, 125.6, 125.3, 124.7, 124.4, 61.7, 60.9, 55.6, 51.3, 44.8, 44.7, 39.5, 37.7, 21.7, 21.2, 17.7, 15.6, 13.9. Anal. Calcd for $C_{19}H_{22}BrNO_3$: C, 58.17; H, 5.65; N, 3.57. Found: C, 58.19; H, 5.69; N, 3.55.

General Procedure for Electrochemically Promoted Diastereoselective Synthesis of β-Lactams

A constant current electrolysis was carried out (*I* = 75 mA cm^{-2}) in a MeCN-Et_4NPF_6 0.1 mol dm^{-3} (20 cm^3) solution as catholite, at 0°C, under argon atmosphere. After 193 C were passed, the current was switched off and amide **1** (1 mmol) was added to the catholite and the solution was allowed to stand under stirring at 0°C for 3 h. The solvent was then evaporated under reduced pressure and the residue extracted with diethyl ether (3 x 30 cm^3). The extracts were analysed by thin layer chromatography, GC-MS and ^{1}H NMR; all products were purified by flash chromatography, using *n*-hexane-ethyl acetate 95:5 to 7:3 as eluent. The two β-lactams produced are named β^1 and β^2, being β^1 the first isomer reported in the d.r. in Tables 1 and 2, and β^2 the second one.

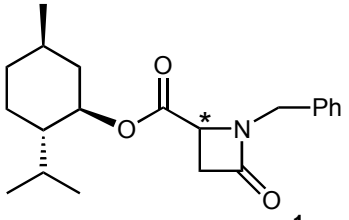
(2*R*)-(-)-Bornane-2,10-sultamyl 1-benzyl-4-oxoazetidine-2-carboxylate (2a).



β^1 and β^2 in mixture*:* ^{1}H NMR ($CDCl_3$): **β^1** δ= 7.30-7.15 (m, 5H), 4.80 (d, AB, Δν= 132.6 Hz, *J*=15.0 Hz, 1H), 4.53 (dd, *J*=5.8-2.3 Hz, 1H), 4.44 (d, AB, Δν=11.0 Hz, *J*=13.9 Hz, 1H), 4.38 (d, AB, Δν=11.0 Hz, *J*=13.9 Hz, 1H), 4.14 (d, AB, Δν= 132.6 Hz, *J*= 15.0 Hz, 1H), 3.82 (t, *J*=6.2 Hz, 1H), 3.27 (dd,

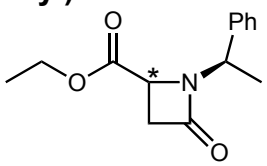
J=14.6 -5.8 Hz, 1H), 2.87 (dd, *J*=14.6 -2.3 Hz, 1H), 2.07-1.75 (m, 5H), 1.45-1.16 (m, 2H), 1.05 (s, 3H), 0.92 (s, 3H). **β²**: δ= 7.30-7.15 (m, 5H), 4.69 (d, AB, Δν=103.8 Hz, *J*=14.6 Hz, 1H), 4.45 (dd, *J*=5.4-2.5 Hz, 1H), 4.17 (d, AB, Δν=103.8 Hz, *J*=14.6 Hz, 1H), 3.81 (dd, *J*=7.4-4.7 Hz, 1H), 3.47-3.31 (m, 2H), 3.23 (dd, *J*=14.8-5.4 Hz, 1H), 2.99 (dd, *J*=14.8-2.5 Hz, 1H), 2.15-1.85 (m, 5H), 1.42-1.14 (m, 2H), 0.93 (s, 3H), 0.92 (s, 3H). ^{13}C NMR ($CDCl_3$): **β¹**: δ= 169.1, 165.2, 135.0, 128.7, 128.4, 127.7, 64.9, 52.6, 50.6, 49.1, 47.7, 45.6, 44.5, 43.4, 37.9, 32.6, 26.3, 20.7, 19.7. **β²**: δ= 169.2, 165.7, 134.4, 128.8, 128.7, 127.8, 65.2, 52.8, 50.4, 49.2, 47.8, 45.8, 44.6, 43.0, 38.2, 32.8, 26.4, 20.7, 19.8. Anal. Calcd for $C_{21}H_{26}N_2O_4S$: C, 62.66; H, 6.51; N, 6.96; S, 7.97. Found: C, 62.71; H, 6.61; N, 6.94; S, 7.95.

(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl 1-benzyl-4-oxoazetidine-2-carboxylate (2b).



β¹ and β² in mixture*:* ^{1}H NMR ($CDCl_3$): δ = **β¹**: 7.32-7.18 (m, 5H), 4.81 (d, *J*=14.9 Hz, 1H), 4.79-4.64 (m, 1H), 4.08 (d, *J*=14.8 Hz, 1H), 3.18 (dd, ABX, Δν=40.2 Hz, *J*=14.2-5.6 Hz, 1H), 2.98 (dd, ABX, Δν=40.2 Hz, *J*=14.2-2.5 Hz, 1H), 2.19-2.15 (m, 1H), 1.92-1.64 (m, 3H), 1.45-1.23 (m, 3H), 1.00-0.81 (m, 8H), 0.73 (d, *J*=7.0 Hz, 3H). **β²**: 7.32-7.18 (m, 5H), 4.79 (d, *J*=15.0 Hz, 1H), 4.79-4.64 (m, 1H), 4.08 (d, *J*=14.8 Hz, 1H), 3.87-3.83 (m, 1H), 3.17 (dd, ABX, Δν=44.5 Hz, *J*=14.3-5.6 Hz, 1H), 2.95 (dd, ABX, Δν=44.5 Hz, *J*=14.3-2.6 Hz, 1H), 2.19-2.15 (m, 1H), 1.92-1.64 (m, 3H), 1.45-1.23 (m, 3H), 1.00-0.81 (m, 8H), 0.73 (d, *J*=7.0 Hz, 3H). ^{13}CNMR ($CDCl_3$): δ= **β¹**:170.0, 165.8, 135.0, 128.6, 128.5, 127.9, 75.8, 50.2, 46.9, 45.7, 41.8, 40.7, 34.1, 31.4, 26.4, 23.3, 21.9, 20.7, 16.1. **β²**:170.0, 165.7, 134.9, 128.9, 128.5, 127.9, 75.7, 50.2, 46.9, 45.7, 42.0, 40.7, 34.1, 31.4, 26.4, 23.4, 21.9, 20.7, 16.3. GC-MS: *m/z*= $M^{+\cdot}$absent, 210 (22%), 204 (13%), 160 (22%), 133 (10%), 91 (100%). Anal. Calcd for $C_{21}H_{29}NO_3$: C, 73.44; H, 8.51; N, 4.08. Found: C, 73.51; H, 8.69; N, 4.05.

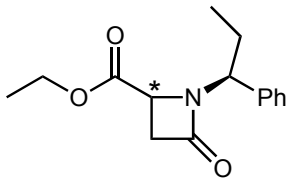
Ethyl 4-oxo-1-((*R*)-1-phenylethyl)azetidine-2-carboxylate (2c).[4]



β¹ and β² in mixture*:* ^{1}H NMR ($CDCl_3$): δ= **β²**: 7.37-7.26 (m, 5H), 4.95 (q, *J*=7.1 Hz, 1H), 4.18 (q, *J*=7.1 Hz, 1H), 3.80 (dd, *J*=5.2-2.6 Hz, 1H), 3.03 (ABX, Δν=23.0 Hz, *J*=14.9-5.2 Hz, 1H), 2.91 (ABX, Δν=23.0 Hz, *J*=14.9-2.5 Hz, 1H), 1.57 (d, *J*=7.1 Hz, 3H), 1.16 (t, *J*=7.1 Hz, 3H). **β¹**: 7.37-7.26 (m, 5H), 4.67 (q, *J*=7.0 Hz, 1H), 3.89 (dd, *J*=5.0-2.8 Hz, 1H), 3.15-3.00 (m, 2H), 1.89 (d, *J*=7.0 Hz, 3H), 1.10 (t, *J*=7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ= **β²**: 171.1, 165.8, 139.5, 128.7, 127.9, 127.2, 61.5, 52.8, 50.1, 41.1, 18.4, 13.9. **β¹**: 170.6, 165.8, 140.4, 128.7, 127.8, 127.0, 60.3, 54.8, 49.7, 41.0, 19.5, 14.2. GC-MS: *m/z*=

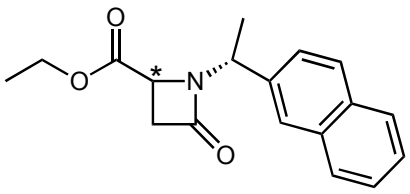
$M^{+\cdot}$absent, 232 ($M^{+\cdot}$- CH_3, 2%), 175 (6%), 105 (100%). Anal. Calcd for $C_{14}H_{17}NO_3$: C, 68.00; H, 6.93; N, 5.66. Found: C, 68.12; H, 6.98; N, 5.62.

Ethyl 4-oxo-1-((*S*)-1-phenylpropyl)azetidine-2-carboxylate (2d).



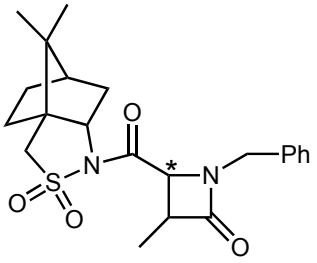
β^1 and β^2 in mixture*:* ^{1}H NMR ($CDCl_3$): δ= **β^1**: 7.37-7.23 (m, 5H), 4.60 (t, *J*=7.8 Hz, 1H), 4.11(dq, *J*=7.1-1.6 Hz, 2H), 3.74 (dd, *J*=5.2-3.0 Hz, 1H), 3.01 (dd, ABX, $\Delta\nu$=12.9 Hz, *J*=14.6-5.2 Hz, 1H), 2.95 (dd, ABX, $\Delta\nu$=12.9 Hz, *J*=14.6-3.0 Hz, 1H), 2.48-1.76 (m, 2H), 1.22 (t, *J*=7.1 Hz, 3H), 0.93 (t, *J*=7.3 Hz, 3H). **β^2**: 7.37-7.23 (m, 5H), 4.31 (t, *J*=7.8 Hz, 1H), 4.07-3.95 (m, 2H), 3.85 (dd, *J*=5.5-2.5 Hz, 1H), 3.08 (dd, ABX, $\Delta\nu$=35.8 Hz, *J*=14.3-5.5 Hz, 1H), 2.90 (dd, ABX, $\Delta\nu$=35.8 Hz, *J*=14.3-2.5 Hz, 1H), 2.48-1.76 (m, 2H), 1.15 (t, *J*=7.2 Hz, 3H), 0.92 (t, *J*=7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ= **β^1**: 170.9, 166.2, 138.3, 128.8, 127.9, 127.8, 61.5, 60.1, 50.5, 40.9, 25.9, 13.9, 11.4. **β^2**: 170.5, 165.8, 139.3, 128.7, 127.9, 127.6, 62.0, 61.4, 49.7, 40.9, 26.5, 13.9, 11.2. GC-MS: *m/z*= $M^{+\cdot}$absent, 232 ($M^{+\cdot}$-29, 2%), 204 (7%), 132 (8%), 104 (15%), 91 (100%). Anal. Calcd for $C_{15}H_{19}NO_3$: C, 68.94; H, 7.33; N, 5.36. Found: C, 69.10; H, 7.41; N, 5.32.

Ethyl 1-((*R*)-1-(naphthalen-2-yl)ethyl)-4-oxoazetidine-2-carboxylate (2e).



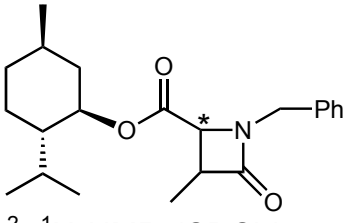
β^1 and β^2 in mixture*.* **β^1**: ^{1}H NMR ($CDCl_3$): δ= 7.84-7.78 (m, 3H), 7.71-7.68 (m, 1H), 7.51-7.39 (m, 3H), 5.16 (q, *J*=7.0 Hz, 1H), 4.07 (q, *J*= 7.1 Hz, 2H), 3.81 (dd, *J*= 5.4-2.8 Hz, 1H), 3.05 (dd, ABX, $\Delta\nu$= 22.2 Hz, *J*= 14.4-5.4 Hz, 1H), 2.94 (dd, ABX, $\Delta\nu$=22.2 Hz, *J*= 14.4-2.8 Hz, 1H), 1.68 (d, *J*= 7.0 Hz, 3H), 1.19 (t, *J*=7.1 Hz, 3H). **β^2**: ^{1}H NMR ($CDCl_3$): δ= 7.84-7.78 (m, 3H), 7.71-7.68 (m, 1H), 7.51-7.39 (m, 3H), 4.84 (q, *J*=7.0 Hz, 1H), 3.94-3.87 (m, 3H), 3.17-2.90 (m, 2H), 2.94 (dd, ABX, $\Delta\nu$=22.2 Hz, *J*= 14.4-2.8 Hz, 1H β^1), 1.82 (d, *J*= 7.0 Hz, 3H), 1.03 (t, *J*=7.2 Hz, 3H). **β^1**: ^{13}C NMR ($CDCl_3$): δ= 171.1, 165.9, 136.8, 133.3, 133.0, 128.6, 128.0, 127.6, 126.4, 126.1, 125.9, 125.3, 61.5, 52.9, 50.1, 41.2, 18.4, 13.9. **β^2**: ^{13}C NMR ($CDCl_3$): δ= 170.6, 165.9, 137.8, 133.3, 133.0, 128.6, 127.9, 127.6, 126.3, 126.2. 125.7, 125.0, 61.4, 55.0, 49.8, 41.1, 19.5, 13.8. Anal. Calcd for $C_{18}H_{19}NO_3$: C, 72.71; H, 6.44; N, 4.71. Found: C, 72.81; H, 6.46; N, 4.68.

***cis* (2*R*)-(-)-Bornane-2,10-sultamyl 1-benzyl-3-methyl-4-oxoazetidine-2-carboxylate (2f)**.



***cis* β^2** (15%): ^{1}H NMR ($CDCl_3$): δ= 7.32-7.20 (m, 3H), 7.16-7.12 (m, 2H), 4.56 (d, *J*=4.8 Hz, 1H), 4.68 (d, AB, Δν=126.3 Hz, *J*=14.2 Hz, 1H)*, 4.05 (d, AB, Δν=126.3 Hz, *J*=14.2 Hz, 1H)*, 3.80 (t, *J*=6.2 Hz, 1H), 3.57-3.43 (m, 1H), 3.40 (d, AB, Δν=10.8 Hz, *J*=14.2 Hz, 1H), 3.34 (d, AB, Δν=10.8 Hz, *J*=14.2 Hz, 1H), 2.11-1.84 (m, 5H), 1.39-1.31 (m, 2H), 1.15 (d, *J*=7.2 Hz, 3H), 0.94 (s, 3H), 0.91 (s, 3H). ^{13}C NMR ($CDCl_3$): δ=169.4, 167.6, 134.1, 128.9, 128.8, 127.9, 65.1, 55.4, 52.8, 49.6, 49.1, 47.7, 45.3, 44.7, 38.3, 32.8, 26.3, 20.8, 19.8, 9.7. α_D=+15.3 (c=0.170, $CHCl_3$). Anal. Calcd for $C_{22}H_{28}N_2O_4S$: C, 63.44; H, 6.78; N, 6.73; S, 7.70. Found: C, 63.56; H, 6.90; N, 6.67; S, 7.68.
*Δν/*J*=8.86 I consider this system an AB system (Δν/*J*<10).
***cis* β^1** in mixture with *cis* β^2: ^{1}H NMR ($CDCl_3$): δ=7.36-7.18 (m, 5H), 4.84 (d, *J*=14.8 Hz, 1H), 4.68 (d, *J*=5.6 Hz, 1H), 4.02 (d, *J*=14.8 Hz, 1H), 3.89 (t, *J*=6.4 Hz, 1H), 3.63-3.53 (m, 1H), 3.45 (d, AB, Δν=7.1 Hz, *J*=14.0 Hz, 1H), 3.41 (d, AB, Δν=7.1 Hz, *J*=14.0 Hz, 1H), 2.15-1.98 (m, 2H), 1.91-1.81 (m, 3H), 1.47-1.22 (m, 5H), 1.10 (s, 3H), 0.94 (s, 3H). ^{13}C NMR ($CDCl_3$): δ=168.9, 168.4, 135.8, 128.8, 128.6, 127.8, 65.3, 55.9, 52.8, 50.1, 49.0, 47.8, 45.3, 44.5, 38.4, 32.9, 26.4, 20.6, 19.9, 10.0. Anal. Calcd for $C_{22}H_{28}N_2O_4S$: C, 63.44; H, 6.78; N, 6.73; S, 7.70. Found: C, 63.51; H, 6.88; N, 6.68; S, 7.70.

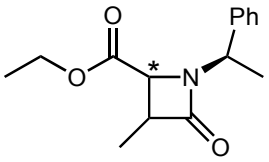
***cis* (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 1-benzyl-3-methyl-4-oxoazetidine-2-carboxylate (2g)**.



cis β^1 in mixture with *cis* β^2. ^{1}H NMR ($CDCl_3$): δ= ***cis* β^1**: 7.37-7.16 (m, 5H), 4.85 (d, *J*=14.7 Hz, 1H), 4.80-4.66 (m, 1H), 4.11 (d, *J*=14.7 Hz, 1H), 3.94 (d, *J*=5.8 Hz, 1H), 3.53-3.35 (m, 1H), 1.98-1.88 (m, 1H), 1.71-1.64 (m, 3H), 1.48-1.30 (m, 2H), 1.20 (d, *J*=7.8 Hz, 3H), 1.11-1.00 (m, 3H), 0.97-0.82 (m, 6H), 0.71(d, *J*=6.8 Hz, 3H). ***cis* β^2**: 7.37-7.16 (m, 5H), 4.81 (d, *J*=14.8 Hz, 1H), 4.80-4.66 (m, 1H), 4.04 (d, *J*=14.8 Hz, 1H), 3.90 (d, *J*=5.7 Hz, 1H), 3.53-3.35 (m, 1H), 1.98-1.88 (m, 1H), 1.71-1.64 (m, 3H), 1.48-1.30 (m, 2H), 1.19 (d, *J*=7.4 Hz, 3H), 1.11-1.00 (m, 3H), 0.97-0.82 (m, 6H), 0.72 (d, *J*=6.8 Hz, 3H). ^{13}C NMR ($CDCl_3$): ***cis* β^1**: δ= 169.3, 135.0, 75.6, 55.4, 48.6, 46.7, 45.2, 40.9, 34.0, 31.4, 25.9, 22.8, 21.9, 20.8, 15.6, 10.8. ***cis* β^2**:169.2, 169.1, 134.9, 128.9, 128.8, 128.7, 128.4, 127.9, 76.0, 54.9, 48.2, 46.9, 45.2, 41.0, 34.1, 31.4, 26.3, 23.4, 21.9, 20.6, 16.2, 10.1. GC-MS: *m/z*= $M^{+\cdot}$absent, 224 (3%),

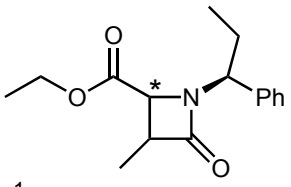
218 (4%), 174 (6%), 139 (5%), 91 (100%), 43 (18%). Anal. Calcd for $C_{22}H_{31}NO_3$: C, 73.91; H, 8.74; N, 3.92. Found: C, 73.99; H, 8.90; N, 3.88.

***cis* ethyl 3-methyl-4-oxo-1-((*R*)-1-phenylethyl)azetidine-2-carboxylate (2h)**.[5]



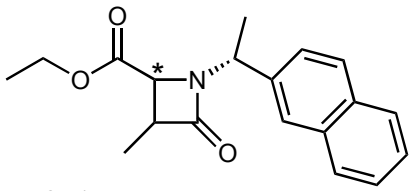
***cis* β¹** (24%): ^{1}H NMR ($CDCl_3$): δ= 7.36-7.13 (m, 5H), 4.98 (q, *J*=7.1 Hz, 1H), 4.17 (dq, *J*=7.1-1.6 Hz, 2H), 3.90 (d, *J*=6.0 Hz, 1H), 3.34 (dq, *J*=7.4-6.0 Hz, 1H), 1.50 (d, *J*=7.1 Hz, 3H), 1.24 (t, *J*=7.1 Hz, 3H), 1.15 (d, *J*=7.4 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ= 170.0, 169.3, 139.3, 128.7, 127.9, 127.2, 61.2, 55.4, 52.0, 47.5, 18.6, 14.2, 9.6. GC-MS: *m/z*= $M^{+\cdot}$ absent, 188 (2%), 160 (4%), 132 (20%), 119 (2%), 105 (100%). α_D= - 62.5 (c=0.047 $CHCl_3$). Anal. Calcd for $C_{15}H_{19}NO_3$: C, 68.94; H, 7.33; N, 5.36. Found: C, 69.11; H, 7.50; N, 5.35.
***cis* β²** in mixture with *cis* β¹: ^{1}H NMR ($CDCl_3$): δ= 7.36-7.24 (m, 5H), 4.56 (q, *J*=7.1 Hz, 1H), 4.10 (q, *J*=7.1 Hz, 2H), 3.96 (d, *J*=5.9 Hz, 1H), 3.40-3.30 (m, 1H), 1.76 (d, *J*=7.1 Hz, 3H), 1.28 (d, *J*=7.3 Hz, 3H), 1.19 (t, *J*=7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ= 169.7, 169.2, 141.0, 127.8, 127.7, 126.9, 61.1, 55.7, 55.3, 47.3, 20.3, 14.2, 9.7. GC-MS: *m/z*= $M^{+\cdot}$absent, 188 (2%), 160 (4%), 132 (20%), 119 (2%), 105 (100%). Anal. Calcd for $C_{15}H_{19}NO_3$: C, 68.94; H, 7.33; N, 5.36. Found: C, 69.07; H, 7.48; N, 5.37.

***cis* ethyl 3-methyl-4-oxo-1-((*S*)-1-phenylpropyl)azetidine-2-carboxylate (2i)**.



***cis* β¹** in mixture with *cis* β²: ^{1}H NMR ($CDCl_3$): δ= 7.37-7.20 (m, 5H), 4.21 (t, *J*=7.8 Hz, 1H), 4.10 (q, *J*=7.2 Hz, 2H), 3.93 (d, *J*=5.9 Hz, 1H), 3.38-3.28 (dq, *J*=7.4-5.9 Hz, 1H), 3.58-2.40 (m, 1H), 2.01-1.87 (m, 1H), 1.19 (t, *J*=7.2 Hz, 3H), 1.16 (d, *J*=7.4 Hz, 3H), 0.91 (t, *J*=7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ=169.6, 169.3, 139.8, 128.6, 127.7, 127.4, 62.7, 61.0, 55.1, 47.2, 27.2, 14.2, 11.4, 9.7. GC-MS: *m/z*= $M^{+\cdot}$absent, 246 ($M^{+\cdot}$-29, 4%), 218 (9%), 132 (48%), 119 (32%), 91 (100%). Anal. Calcd for $C_{16}H_{21}NO_3$: C, 69.79; H, 7.69; N, 5.09. Found: C, 69.81; H, 7.73; N, 5.08.
***cis* β²** (28%): ^{1}H NMR ($CDCl_3$): δ= 7.38-7.21 (m, 5H), 4.63 (t, *J*=7.8 Hz, 1H), 4.18 (dq, *J*=7.3-1.0 Hz, 2H), 3.81 (d, *J*=6.0 Hz, 1H), 3.38-3.24 (m, 1H), 2.32-1.67 (m, 2H), 1.23 (t, *J*=7.3 Hz, 3H), 1.15 (d, *J*=7.6 Hz, 3H), 0.95 (t, *J*=7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ=170.0, 169.7, 138.2, 128.8, 127.9, 61.2, 59.3, 55.9, 47.5, 26.2, 14.2, 11.2, 9.9. GC-MS: *m/z*= $M^{+\cdot}$ absent, 246 ($M^{+\cdot}$-29, 6%), 218 (9%), 132 (47%), 119 (32%), 91 (100%). α_D= - 2.3 (c=0.026, $CHCl_3$). Anal. Calcd for $C_{16}H_{21}NO_3$: C, 69.79; H, 7.69; N, 5.09. Found: C, 69.80; H, 7.73; N, 5.09.

***cis* ethyl 3-methyl-1-((*R*)-1-(naphthalen-2-yl)ethyl)-4-oxoazetidine-2-carboxylate (2j)**.



***cis* β[1]** in mixture with *cis* β[2]: ^{1}H NMR ($CDCl_3$): δ=7.84-7.77 (m, 3H), 7.67 (s, 1H), 7.48-7.38 (m, 3H), 4.73 (q, *J*= 7.1 Hz, 1H), 4.07 (dq, *J*=7.3-0.9 Hz, 2H), 3.96 (d, *J*=6.0 Hz, 1H), 3.40 (dq, *J*=7.3-6.0 Hz, 1H), 1.87 (d, *J*=7.1 Hz, 3H), 1.17 (d, *J*=7.3 Hz, 3H), 1.15 (t, *J*=7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ=169.7, 169.5, 138.5, 133.3, 132.9, 128.7, 127.8, 127.6, 126.3, 126.1, 125.7, 124.8, 61.1, 55.9, 55.3, 47.4, 20.3, 14.1, 9.7. Anal. Calcd for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.33; H, 6.85; N, 4.51.

***cis* β[2]** (29%): ^{1}H NMR ($CDCl_3$): δ=7.83-7.79 (m, 3H), 7.71 (s, 1H), 7.50-7.45 (m, 2H), 7.42-7.37 (m, 1H), 5.17 (q, *J*= 7.1 Hz, 1H), 4.15 (dq, *J*=7.1-3.0 Hz, 2H), 3.89 (d, *J*=5.9 Hz, 1H), 3.36 (dq, *J*=7.3-5.9 Hz, 1H), 1.70 (d, *J*=7.1 Hz, 3H), 1.20 (t, *J*=7.1 Hz, 3H), 1.16 (d, *J*=7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$): δ=170.0, 169.4, 136.8, 133.3, 132.9, 128.6, 128.0, 127.6, 126.3, 126.2, 126.0, 125.5, 61.2, 55.4, 52.1, 47.7, 18.6, 14.2, 9.7. α_D= + 0.75 (c=0.040, $CHCl_3$). Anal. Calcd for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.34; H, 6.84; N, 4.48.

References

[1] J. B. Sweeney, A. A. Cantrill, A. B. McLaren, S. Thobhnani, *Tetrahedron* **2006**, *62*, 3681-3693.
[2] T. Kawabata, K. Sumi, T. Hiyama, *J. Am. Chem. Soc.* **1989**, *111*, 6843-6845.
[3] T. Hudlicky, L. Radesca, H. L. Rigby, *J. Org. Chem.* **1987**, *52*, 4397-4399.
[4] F. M. Cordero, M. Salvati, F. Pisaneschi, A. Brandi, *Eur. J. Org. Chem.* **2004**, 2205-2213.
[5] N. Miyachi, F. Kanda, M. Shibasaki, *J. Org. Chem.* **1989**, *54*, 3511-3513.