

Supplemental Material

β -Lactam Synthesis from Diazoketones and Imines:

The Use of Microwave Irradiation

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Content

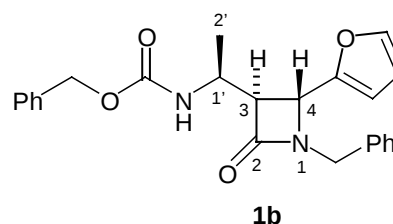
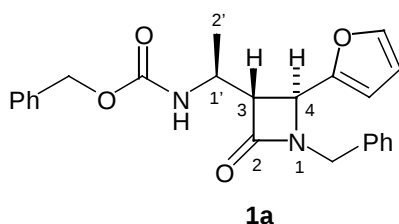
Experimental Procedures for β -Lactams **1** – **7**

X-Ray Data for β -Lactams **1a**, **2a**, and **5a**

General Procedure for the Microwave-induced Syntheses of β -Lactams

1,2-Dichlorobenzene or 1,2-dimethoxyethane were used as solvent. Solvent, amount of starting material and details of the microwave irradiation (irradiation power, temperature, reaction time) are given in the respective procedure. All reactions were performed under nitrogen atmosphere. When 1,2-dichlorobenzene was used as solvent, an oven-dried flask, fitted with a nitrogen inlet was connected via a tube with an external (outside the microwave oven) reflux condenser. Reactions with 1,2-dimethoxyethane were performed in a Teflon autoclave (volume 100 mL) with mounted thermometer.

(3R,4S,1'S)- and (3S,4R,1'S)-1-benzyl-3-[1-(benzyloxycarbonylamino)ethyl]-4-(2-furyl)-azetidin-2-one (1a,b)



Cbz-Ala-CHN₂ (989 mg, 4.00 mmol) and *N*-benzyl-furan-2-carbaldimine (1.61 g, 8.00 mmol) were reacted in 1,2-dichlorobenzene (50 mL) in accordance with the general procedure (500 W, 160 °C, 30 min). The solvent was removed by passing the cooled solution through SiO₂ (120 g, 3 cm × 35 cm; light petroleum/ethyl acetate 5:1 → 3:1 → 1:2). The diastereoisomeric ratio was determined by ¹H NMR spectroscopy and HPLC **1a:1b** 65:35) and the mixture of isomers (1.43 g, 3.54 mmol, 88 %) was separated by medium pressure liquid chromatography (light petroleum/*i*PrOH 97:3) to afford β -lactams **1a** (803 mg, 1.99 mmol, 50 %) and **1b** (415 mg, 1.03 mmol, 26 %) as colorless solids.

1a:

C ₂₄ H ₂₄ N ₂ O ₄	calcd. C 71.27	H 5.98	N 6.93
(404.46 g/mol)	found C 71.04	H 5.99	N 6.81

$[\alpha]_{\text{D}}^{20} = +25$ ($c = 1$, CHCl₃).

Mp.: 110 – 113 °C (light petroleum/*i*PrOH).

^1H NMR (500 MHz, CDCl_3): δ = 1.35 (d, $^3J_{2',1'} = 6.9$ Hz, 3 H, H-2'), 3.51 (s, 1 H, H-3), 3.78 (d, $^2J = 15.1$ Hz, 1 H, NCH_2Ph), 4.18 (m, 1 H, H-1'), 4.30 (s, 1 H, H-4), 4.69 (d, $^2J = 15.1$ Hz, 1 H, NCH_2Ph), 4.89 (d, $^3J_{\text{NH},1'} = 8.7$ Hz, 1 H, NH), 4.94 (d, $^2J = 12.3$ Hz, 1 H, OCH_2Ph), 5.11 (d, $^2J = 12.3$ Hz, 1 H, OCH_2Ph), 7.12 – 7.38 (m, 13 H, Ph, $\text{C}_4\text{H}_3\text{O}$).

^{13}C NMR (126 MHz, CDCl_3): δ = 19.8 (q, C-2'), 44.7 (t, NCH_2Ph), 45.0 (d, C-1'), 50.2 (d, C-4), 61.3 (d, C-3), 66.9 (t, OCH_2Ph), 109.8, 110.5 (2 d, $\text{C}_4\text{H}_3\text{O}$), 127.6, 128.0, 128.2, 128.6, 128.7 (5 d, Ph, 1 d verdeckt), 135.4, 136.3 (2 s, *ipso*-Ph), 143.2 (s, *ipso*- $\text{C}_4\text{H}_3\text{O}$), 149.8 (d, $\text{C}_4\text{H}_3\text{O}$), 156.1 (s, NHCO), 167.1 (s, C-2).

IR (KBr): $\tilde{\nu}$ (cm^{-1}) = 3300 (NH), 3020 (CH), 2960 (CH), 2920 (CH), 1750 (C=O), 1675 (C=O, amide I), 1525 (NHCO, amide II), 1240 (CO), 1040 (CO), 730, 680 (Ph).

MS (EI, 70 eV): m/z (%) 404 (1, M^+), 376 (47, $[\text{M} - \text{CO}]^+$), 313 (19, $[\text{M} - \text{C}_7\text{H}_7]^+$), 226 (50, $[\text{M} - \text{C}_{10}\text{H}_{12}\text{NO}_2]^+$), 91 (100, C_7H_7^+), 28 (3, CO^+).

1b:

$\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_4$	calcd. C 71.27	H 5.98	N 6.93
(404.46 g/mol)	found C 71.13	H 5.98	N 6.83

$[\alpha]_{\text{D}}^{20} = -40$ ($c = 1$, CHCl_3).

Mp.: 111 – 112 °C (light petroleum/*i*PrOH).

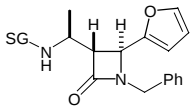
^1H NMR (500 MHz, CDCl_3): δ = 1.28 (d, $^3J_{2',1'} = 6.7$ Hz, 3 H, H-2'), 3.42 (d, $^3J_{3,1'} = 6.6$ Hz, 1 H, H-3), 3.85 (d, $^2J = 15.0$ Hz, 1 H, NCH_2Ph), 4.13 (m, 1 H, H-1'), 4.38 (s, 1 H, H-4), 4.65 (d, $^2J = 15.0$ Hz, 1 H, NCH_2Ph), 4.93 (d, $^3J_{\text{NH},1'} = 8.5$ Hz, 1 H, NH), 5.06 (s, 2 H, OCH_2Ph), 6.15 (s, 1 H, $\text{C}_4\text{H}_3\text{O}$), 6.29 (m, 1 H, $\text{C}_4\text{H}_3\text{O}$), 7.14 (m, 2 H, $\text{C}_4\text{H}_3\text{O}$, Ph), 7.25 – 7.36 (m, 9 H, Ph).

^{13}C NMR (126 MHz, CDCl_3): δ = 18.2 (q, C-2'), 44.8 (t, NCH_2Ph), 46.0 (d, C-1'), 51.3 (d, C-4), 61.7 (d, C-3), 66.7 (t, OCH_2Ph), 109.5, 110.5 (2 d, $\text{C}_4\text{H}_3\text{O}$), 127.7, 128.1, 128.2, 128.3, 128.5, 128.7 (6 d, Ph), 135.4, 136.4 (2 s, *ipso*-Ph), 143.2 (s, *ipso*- $\text{C}_4\text{H}_3\text{O}$), 149.9 (d, $\text{C}_4\text{H}_3\text{O}$), 155.5 (s, NHCO), 167.0 (s, C-2).

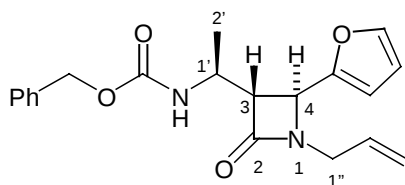
IR (KBr): $\tilde{\nu}$ (cm^{-1}) = 3315 (NH), 1760 (C=O), 1695 (C=O, amide I), 1520 (NHCO, amide II), 1230 (CO), 1050 (CO), 700, 680 (Ph).

MS (EI, 70 eV): m/z (%) 404 (2, M^+), 376 (28, $[\text{M} - \text{CO}]^+$), 313 (15, $[\text{M} - \text{C}_7\text{H}_7]^+$), 226 (42, $[\text{M} - \text{C}_{10}\text{H}_{12}\text{NO}_2]^+$), 91 (100, C_7H_7^+), 28 (4, CO^+).

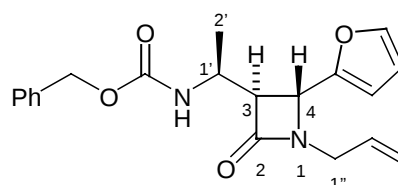
Optical rotary power of β -lactams 1a and 1b

	light induced reaction		microwave induced thermic reaction	
	1a	1b	1a	1b
$[\alpha]_{\text{D}}^{20}$, $c = 1$, CHCl_3	+25	−40	+25	−39

(3R,4S,1'S)- and (3S,4R,1'S)-1-allyl-3-[1-(benzyloxycarbonylamino)-ethyl]-4-(2-furyl)-azetidin-2-one (2a,b)



2a



2b

Cbz-Ala-CHN₂ (1.35 g, 5.47 mmol) and *N*-allyl furan-2-carbaldimine (814 mg, 6.02 mmol) were reacted in 1,2-dichlorobenzene (30 mL) in accordance with the general procedure (500 W, 160 °C, 20 min). The solvent was removed by passing the cooled solution through SiO₂ (100 g, 3 cm × 40 cm; elution with 1 L light petroleum). The products were eluted with light petroleum/ethyl acetate (3:1). The diastereoisomeric ratio was determined by ¹H NMR spectroscopy (**2a:2b** 65:35) and the mixture of isomers (1.79 g, 5.04 mmol, 92 %) was recrystallized from light petroleum/ethyl acetate to afford a pure fraction of isomer **2a** (500 mg, 1.41 mmol, 26 %) as a colorless solid. The mother liquor was evaporated and separated by medium pressure liquid chromatography (light petroleum/*i*PrOH 92:8) to afford a second crystalline fraction of β-lactams **2a** (261 mg, 736 μmol, 13 %) and isomer **2b** (187 mg, 596 μmol, 24 %) as a colorless oil.

2a:

C ₂₀ H ₂₂ N ₂ O ₄	calcd. C 67.78	H 6.26	N 7.90
(354.40 g/mol)	found C 68.02	H 6.33	N 7.90

$$[\alpha]_{\text{D}}^{20} = +71 \text{ (} c = 1, \text{CHCl}_3 \text{)}.$$

Mp.: 100 – 101 °C (light petroleum/ethyl acetate).

¹H NMR (500 MHz, CDCl₃): δ = 1.36 (d, ³J_{2',1'} = 6.9 Hz, 3 H, H-2'), 3.36 (dd, ²J_{1'',1''} = 15.8 Hz, ³J_{1'',2''} = 6.8 Hz, 1 H, H-1''), 3.48 (dd, ³J_{3,4} = ³J_{3,1'} = 3.0 Hz, 1 H, H-3), 4.00 (dd, ²J_{1'',1''} = 15.8 Hz, ³J_{1'',2''} = 5.2 Hz, 1 H, H-1'), 4.32 (m, 1 H, H-1'), 4.44 (s, 1 H, H-4), 4.92 (d, ³J_{NH,1'} = 8.7 Hz, 1 H, NH), 5.04 (d, ³J_{3'',2''} = 10.1 Hz, 1 H, H-3''), 5.08 (dd, ³J_{3'',2''} = 17.1 Hz, ²J_{3'',3''} = 1.5 Hz, 1 H, H-3''), 5.10 (d, ²J = 12.0 Hz, 1 H, OCH₂Ph), 5.14 (d, ²J = 12.0 Hz, 1 H, CH₂Ph), 5.58 (dddd, ³J_{2'',3''} = 17.1 Hz, ³J_{2'',3''} = 10.2 Hz, ³J_{2'',1'} = 6.8 Hz, ³J_{2'',1'} = 5.1 Hz, 1 H, H-2''), 6.32 (dd, ³J = 3.3 Hz, ³J = 1.9 Hz, 1 H, C₄H₃O), 6.34 (d, ³J = 1.8 Hz, 1 H, C₄H₃O), 7.32 – 7.41 (m, 6 H, C₄H₃O, Ph).

¹³C NMR (126 MHz, CDCl₃): δ = 19.8 (q, C-2'), 43.1 (t, C-1''), 45.0 (d, C-1'), 50.5 (d, C-4), 61.4 (d, C-3), 66.8 (t, CH₂Ph), 110.0 (d, C₄H₃O), 110.5 (d, C₄H₃O), 118.1 (t, C-3''), 128.0, 128.2, 128.5 (3 d, Ph), 131.4 (d, C-2''), 136.4 (s, *ipso*-Ph), 143.2, 149.8 (d, s, C₄H₃O, *ipso*-C₄H₃O), 156.2 (s, NHCO), 167.1 (s, C-2).

IR (KBr): $\tilde{\nu}$ (cm⁻¹) = 3295 (NH), 2960 (CH), 1740 (C=O), 1685 (C=O, amide I), 1535 (NHCO, amide II), 1245 (CO), 730, 680 (Ph).

MS (CI, CH₄): *m/z* (%) 355 (21, [M + H]⁺), 206 (5, [M + H – C₈H₇NO₂]⁺), 178 (50, C₁₀H₁₂NO₂⁺), 176 (19, [M – C₁₀H₁₂NO₂]⁺), 149 (22, C₈H₇NO₂⁺), 91 (100, C₇H₇⁺).

2b:

$C_{20}H_{22}N_2O_4$	calcd. C 67.78	H 6.26	N 7.90
(354.40 g/mol)	found C 67.73	H 6.39	N 7.90

$$[\alpha]_D^{20} = -60 \text{ (} c = 1, \text{CHCl}_3 \text{)}.$$

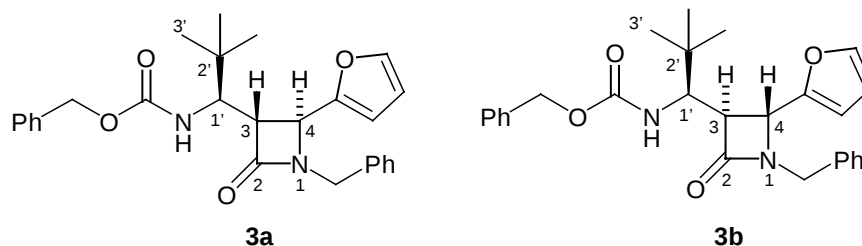
^1H NMR (500 MHz, CDCl_3): δ = 1.34 (d, $^3J_{2',1'} = 6.8$ Hz, 3 H, H-2'), 3.35 (dd, $^3J_{3,1'} = 8.6$ Hz, $^3J_{3,4} = 2.3$ Hz, 1 H, H-3), 3.42 (dd, $^2J_{1'',1'''} = 15.9$ Hz, $^3J_{1'',2'''} = 6.9$ Hz, 1 H, H-1''), 4.00 (dd, $^2J_{1'',1'''} = 15.7$ Hz, $^3J_{1'',2'''} = 5.4$ Hz, 1 H, H-1'''), 4.12 (m, 1 H, H-1'), 4.57 (d, $^3J_{4,3} = 1.6$ Hz, 1 H, H-4), 4.96 (d, $^3J_{\text{NH},1'} = 8.6$ Hz, 1 H, NH), 5.06 (d, $^2J = 12.2$ Hz, 1 H, CH_2Ph), 5.11 (d, $^2J = 12.1$ Hz, 1 H, CH_2Ph), 5.12 (dd, $^3J_{3'',2'''} = 10.3$ Hz, $^2J_{3'',3'''} = 1.3$ Hz, 1 H, H-3''), 5.11 – 5.13 (m, 1 H, H-3'''), 5.65 (dddd, $^3J_{2'',3'''} = 17.0$ Hz, $^3J_{2'',3''} = 10.2$ Hz, $^3J_{2'',1'''} = 6.7$ Hz, $^3J_{2'',1''} = 5.7$ Hz, 1 H, H-2''), 6.21 (d, $^3J = 3.2$ Hz, 1 H, $\text{C}_4\text{H}_3\text{O}$), 6.32 (dd, $^3J = 3.3$ Hz, $^3J = 1.8$ Hz, 1 H, $\text{C}_4\text{H}_3\text{O}$), 7.30 – 7.38 (m, 6 H, $\text{C}_4\text{H}_3\text{O}$, Ph).

^{13}C NMR (126 MHz, CDCl_3): δ = 18.5 (q, C-2'), 43.3 (t, C-1''), 46.3 (d, C-1'), 51.8 (d, C-4), 61.9 (d, C-3), 66.7 (t, CH_2Ph), 109.4 (d, $\text{C}_4\text{H}_3\text{O}$), 110.6 (d, $\text{C}_4\text{H}_3\text{O}$), 118.6 (t, C-3''), 128.1, 128.2, 128.5 (3 d, Ph), 131.3 (d, C-2''), 136.4 (s, *ipso*-Ph), 143.1, 150.1 (d, s, $\text{C}_4\text{H}_3\text{O}$, *ipso*- $\text{C}_4\text{H}_3\text{O}$), 155.7 (s, NHCO), 167.0 (s, C-2).

IR (film): $\tilde{\nu}$ (cm^{-1}) = 3319 (NH), 3034 (CH), 2976 (CH), 2935 (CH), 1747 (C=O), 1740 (C=O, amide I), 1537 (NHCO, amide II), 1248 (CO), 740, 698 (Ph).

MS (CI, CH_4): m/z (%) 709 (1, $[2\text{ M} + \text{H}]^+$), 355 (42, $[\text{M} + \text{H}]^+$), 263 (29, $[\text{M} - \text{C}_7\text{H}_7]^+$), 206 (13, $[\text{M} + \text{H} - \text{C}_8\text{H}_7\text{NO}_2]^+$), 178 (100, $\text{C}_{10}\text{H}_{12}\text{NO}_2^+$), 176 (17, $[\text{M} - \text{C}_{10}\text{H}_{12}\text{NO}_2]^+$), 149 (13, $\text{C}_8\text{H}_7\text{NO}_2^+$), 91 (62, C_7H_7^+).

(3R,4S,1'R)- and (3S,4R,1'R)-1-benzyl-3-[1-(benzyloxycarbonylamino)-2,2-dimethyl-ethyl]-4-(2-furyl)-azetidin-2-one (3a,b)



With microwave irradiation:

Cbz-Tle- CHN_2 (2.02 g, 7.00 mmol) and *N*-benzyl furan-2-carbaldimine (2.59 g, 14.0 mmol) were reacted in 1,2-dichlorobenzene (50 mL) in accordance with the general procedure (500 W, 160 °C, 30 min). The diastereoisomeric ratio was determined by HPLC (**3a:3b** 90:10) and the mixture of isomers was separated by flash chromatography (light petroleum/ethyl acetate 6:1 → 1:4) to afford β -lactam **3a** (2.35 g, 5.26 mmol, 75 %) as a colorless oil and β -lactam **3b** (270 mg, 605 μmol , 9 %) as a colorless solid.

With thermal heating:

Instead of microwave heating, a flask equipped as described above was transferred in a bath kept at 160 °C. When the reaction was finished (after about 15 min, as monitored with tlc), the flask was cooled in a bath kept at room temperature. Workup of the crude material (diastereoisomeric ratio 64 : 36) as described above afforded the β -lactams **3a** (54 %) and **3b** (31 %).

3a:

$C_{27}H_{30}N_2O_4$	calcd. C 72.62	H 6.77	N 6.27
(446.54 g/mol)	found C 72.52	H 6.84	N 6.18

$$[\alpha]_D^{20} = +61 \text{ (} c = 1, \text{CHCl}_3 \text{)}.$$

^1H NMR (500 MHz, CDCl_3): δ = 0.97 [s, 9 H, $\text{C}(\text{CH}_3)_3$], 3.74 (dd, $^3J_{3,4} = ^3J_{3,1'} = 2.5$ Hz, 1 H, H-3), 3.76 (d, $^2J = 15.1$ Hz, 1 H, NCH_2Ph), 3.87 (dd, $^3J_{1',\text{NH}} = 10.7$ Hz, $^3J_{1',3} = 2.4$ Hz, 1 H, H-1'), 4.25 (d, $^3J_{4,3} = 2.5$ Hz, 1 H, H-4), 4.66 (d, $^2J = 15.1$ Hz, 1 H, NCH_2Ph), 4.96 (d, $^2J = 12.3$ Hz, 1 H, OCH_2Ph), 5.04 (d, $^3J_{\text{NH},1'} = 10.7$ Hz, 1 H, NH), 5.17 (d, $^2J = 12.3$ Hz, 1 H, OCH_2Ph), 6.26 (d, $^3J = 3.3$ Hz, 1 H, $\text{C}_4\text{H}_3\text{O}$), 6.33 (dd, $^3J = 3.3$ Hz, $^3J = 1.8$ Hz 1 H, $\text{C}_4\text{H}_3\text{O}$), 7.12 (m, 2 H, $\text{C}_4\text{H}_3\text{O}$, Ph), 7.18 (m, 3 H, Ph), 7.20 – 7.41 (m, 6 H, Ph).

^{13}C NMR (126 MHz, CDCl_3): δ = 26.8 [q, $\text{C}(\text{CH}_3)_3$], 34.8 (s, C-2'), 44.7 (t, NCH_2Ph), 51.6 (d, C-4), 57.4 (d, C-3), 58.0 (d, C-1'), 66.9 (t, OCH_2Ph), 110.2 (d, $\text{C}_4\text{H}_3\text{O}$), 110.5 (d, $\text{C}_4\text{H}_3\text{O}$), 127.5, 127.8, 128.1, 128.2, 128.5, 128.8 (6 d, Ph), 135.4, 136.4, 143.3, 149.6 (1 d, 3 s, $\text{C}_4\text{H}_3\text{O}$, *ipso*- $\text{C}_4\text{H}_3\text{O}$, *ipso*-Ph), 156.8 (s, NHCO), 166.6 (s, C-2).

IR (film): $\tilde{\nu}$ (cm^{-1}) = 3317 (NH), 3031 (CH), 2962 (CH), 1750 (C=O), 1713 (C=O, amide I), 1504 (NHCO, amide II), 1234 (CO), 736, 698 (Ph).

MS (FAB): m/z (%) 469 (8, $[\text{M} + \text{Na}]^+$), 448 (15, $[\text{M} + 2 \text{H}]^+$), 447 (50, $[\text{M} + \text{H}]^+$), 228 (100, $[\text{M} + 2 \text{H} - \text{C}_{13}\text{H}_{18}\text{NO}_2]^+$), 220 (12, $\text{C}_{13}\text{H}_{18}\text{NO}_2^+$), 91 (100, C_7H_7^+).

3b:

$C_{27}H_{30}N_2O_4$	calcd. C 72.62	H 6.77	N 6.27
(446.54 g/mol)	found C 72.57	H 6.80	N 6.23

$$[\alpha]_D^{20} = -103 \text{ (} c = 1, \text{CHCl}_3 \text{)}.$$

Mp.: 160 – 161 °C (light petroleum/ethyl acetate).

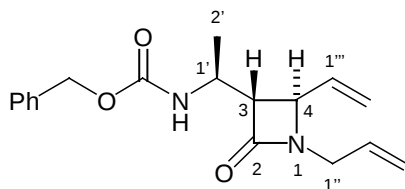
^1H NMR (500 MHz, CDCl_3): δ = 0.88 [s, 9 H, $\text{C}(\text{CH}_3)_3$], 3.70 (dd, $^3J_{3,1'} = 4.7$ Hz, $^3J_{3,4} = 2.6$ Hz, 1 H, H-3), 3.72 (d, $^2J = 15.2$ Hz, 1 H, NCH_2Ph), 4.15 (dd, $^3J_{1',\text{NH}} = 10.6$ Hz, $^3J_{1',3} = 4.7$ Hz, 1 H, H-1'), 4.17 (d, $^3J_{4,3} = 2.4$ Hz, 1 H, H-4), 4.65 (d, $^3J_{\text{NH},1'} = 10.6$ Hz, 1 H, NH), 4.72 (d, $^2J = 15.2$ Hz, 1 H, NCH_2Ph), 5.11 (d, $^2J = 12.1$ Hz, 1 H, OCH_2Ph), 5.19 (d, $^2J = 12.1$ Hz, 1 H, OCH_2Ph), 6.13 (d, $^3J = 3.0$ Hz, 1 H, $\text{C}_4\text{H}_3\text{O}$), 6.31 (dd, $^3J = 3.0$ Hz, $^3J = 1.8$ Hz 1 H, $\text{C}_4\text{H}_3\text{O}$), 7.10 (m, 2 H, $\text{C}_4\text{H}_3\text{O}$, Ph), 7.16 (m, 3 H, Ph), 7.33 – 7.38 (m, 6 H, Ph).

^{13}C NMR (126 MHz, CDCl_3): δ = 26.7 [q, $\text{C}(\text{CH}_3)_3$], 34.7 (s, C-2'), 44.6 (t, NCH_2Ph), 50.0 (d, C-4), 57.1 (d, C-3), 58.0 (d, C-1'), 67.1 (t, OCH_2Ph), 109.6 (d, $\text{C}_4\text{H}_3\text{O}$), 110.5 (d, $\text{C}_4\text{H}_3\text{O}$), 127.6, 128.2, 128.3, 128.4, 128.6, 128.7 (6 d, Ph), 135.5, 136.4, 143.3, 149.8 (1 d, 3 s, $\text{C}_4\text{H}_3\text{O}$, *ipso*- $\text{C}_4\text{H}_3\text{O}$, *ipso*-Ph), 156.0 (s, NHCO), 167.4 (s, C-2).

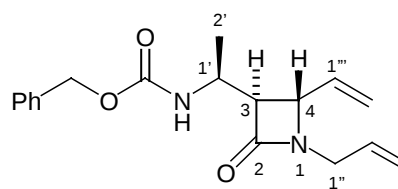
IR (KBr): $\tilde{\nu}$ (cm^{-1}) = 3315 (NH), 2940 (CH), 1735 (C=O), 1705 (C=O, amide I), 1523 (NHCO, amide II), 1235 (CO), 750, 680 (Ph).

MS (EI, 70 eV): m/z (%) 446 (1, M^+), 361 (35, $[\text{M} - \text{C}_5\text{H}_{11}\text{N}]^+$), 311 (2, $[\text{M} - \text{C}_8\text{H}_7\text{O}_2]^+$), 226 (14, $[\text{M} - \text{C}_{13}\text{H}_{18}\text{NO}_2]^+$), 220 (12, $\text{C}_{13}\text{H}_{18}\text{NO}_2^+$), 91 (100, C_7H_7^+).

(3R,4R,1'S)- and (3S,4S,1'S)-1-allyl-3-[1-(benzyloxycarbonylamino)-ethyl]-4-(2-vinyl)-azetidin-2-one (4a,b)



4a



4b

Cbz-Ala-CHN₂ (612 mg, 2.48 mmol) and 4-aza-hepta-1,3,6-triene (235 mg, 2.48 mmol) were reacted in 1,2-dichlorobenzene (50 mL) in accordance with the general procedure (500 W, 160 °C, 30 min). The solvent was removed by passing the cooled solution through SiO₂ (100 g, 3 cm × 40 cm; elution with 1.5 L light petroleum). The products were eluted with light petroleum/ethyl acetate (3:1). The diastereoisomeric ratio was determined by ¹H NMR spectroscopy (**4a:4b** 67:33) and the mixture of isomers (581 mg, 1.58 mmol, 75 %) was separated by medium pressure liquid chromatography (light petroleum/*i*PrOH 97:3) to afford β-lactams **4a** (277 mg, 882 μmol, 36 %) and **4b** (187 mg, 596 μmol, 24 %) as colorless oils.

4a:

C ₁₈ H ₂₂ N ₂ O ₃	calcd. C 68.77	H 7.05	N 8.91
(314.38 g/mol)	found C 68.44	H 7.06	N 8.90

$$[\alpha]_D^{20} = +79 (c = 1, \text{CHCl}_3).$$

¹H NMR (500 MHz, CDCl₃): δ = 1.33 (d, ³J_{2',1'} = 6.9 Hz, 3 H, H-2'), 2.99 (dd, ³J_{3,1'} = 4.0 Hz, ³J_{3,4} = 2.2 Hz, 1 H, H-3), 3.43 (dd, ²J_{1'',1'} = 15.7 Hz, ³J_{1'',2''} = 6.9 Hz, 1 H, H-1''), 3.85 (d, ³J_{4,1''} = 8.2 Hz, 1 H, H-4), 4.00 (dd, ²J_{1'',1'} = 15.7 Hz, ³J_{1'',2''} = 5.1 Hz, 1 H, H-1''), 4.18 (m, 1 H, H-1'), 4.95 (d, ³J_{NH,1'} = 8.1 Hz, 1 H, NH), 5.09 (d, ³J = 11.6 Hz, 1 H, OCH₂Ph), 5.13 (d, ³J = 12.2 Hz, 1 H, OCH₂Ph), 5.14 (dd, ³J_{3'',2''} = 17.1 Hz, ²J_{3'',3''} = 1.5 Hz, 1 H, H-3''), 5.08 – 5.16 (m, 1 H, H-3''), 5.25 (d, ³J_{2'',1''} = 10.2 Hz, 1 H, H-2''), 5.35 (d, ³J_{2'',1''} = 17.1 Hz, 1 H, H-2''), 5.64 (dddd, ³J_{2'',3''} = 17.1 Hz, ³J_{2'',3''} = 10.2 Hz, ³J_{2'',1''} = 6.9 Hz, ³J_{2'',1''} = 5.1 Hz, 1 H, H-2''), 5.75 (ddd, ³J_{1'',2''} = 17.1 Hz, ³J_{1'',2''} = 10.2 Hz, ³J_{1'',4} = 8.2 Hz, 1 H, H-1''), 7.31 – 7.36 (m, 5 H, Ph).

¹³C NMR (126 MHz, CDCl₃): δ = 19.8 (q, C-2'), 42.9 (t, C-1''), 45.0 (d, C-1'), 56.7 (d, C-4), 62.0 (d, C-3), 66.8 (t, OCH₂Ph), 118.3 (t, C-3''), 119.9 (t, C-2''), 128.1, 128.2, 128.5 (3 d, Ph), 131.6 (d, C-2''), 135.2 (d, C-1''), 136.4 (s, *ipso*-Ph), 156.1 (s, NHCO), 167.0 (s, C-2).

IR (film): $\tilde{\nu}$ (cm⁻¹) = 3307 (NH), 2979 (CH), 1744 (C=O), 1644 (C=O, amide I), 1536 (NHCO, amide II), 1236 (CO), 1016 (C=C), 739, 698 (Ph).

MS (EI, 70 eV): *m/z* (%) 314 (12, M⁺), 286 (1, [M – CO]⁺), 223 (23, [M – C₇H₇]⁺), 179 (5, C₁₀H₁₂NO₂⁺), 136 (40, [M – C₁₀H₁₂NO₂]⁺), 108 (16, C₇H₈O⁺), 95 (9, [M – C₁₀H₁₂NO₂ – C₃H₅]⁺), 91 (100, C₇H₇⁺), 41 (14, C₃H₅⁺), 28 (20, CO⁺).

4b:

C ₁₈ H ₂₂ N ₂ O ₃	calcd. C 68.77	H 7.05	N 8.91
(314.38 g/mol)	found C 68.43	H 7.10	N 8.85

$[\alpha]_D^{20} = -70$ ($c = 1$, CHCl_3).

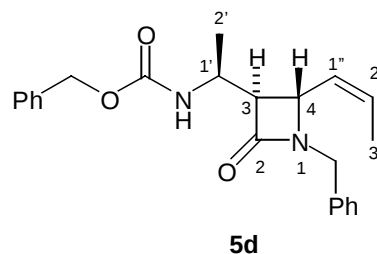
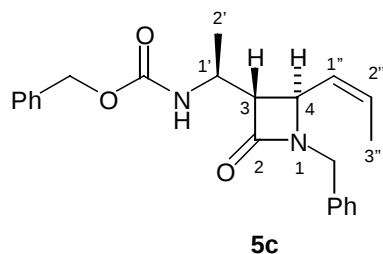
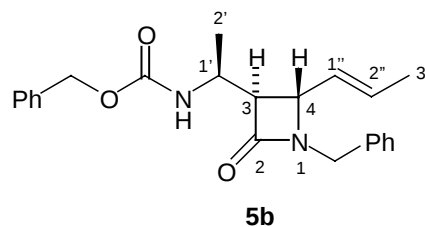
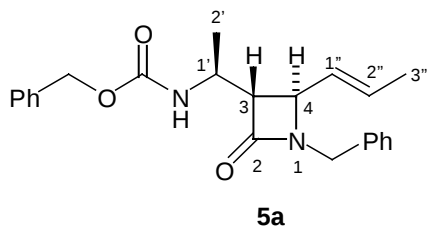
^1H NMR (500 MHz, CDCl_3): $\delta = 1.30$ (d, $^3J_{2',1'} = 6.8$ Hz, 3 H, H-2'), 2.90 (d, $^3J_{3,1'} = 7.1$ Hz, 1 H, H-3), 3.50 (dd, $^2J_{1'',1'} = 15.6$ Hz, $^3J_{1'',2''} = 6.9$ Hz, 1 H, H-1''), 3.96 (d, $^3J_{4,1''} = 7.7$ Hz, 1 H, H-4), 4.01 (dd, $^2J_{1'',1'} = 15.6$ Hz, $^3J_{1'',2''} = 5.4$ Hz, 1 H, H-1''), 4.11 (ddd, $^3J_{1',2'} = ^3J_{1',3} = 7.0$ Hz, $^3J_{1',\text{NH}} = 8.2$ Hz, 1 H, H-1'), 5.00 (d, $^3J_{\text{NH},1'} = 8.3$ Hz, 1 H, NH), 5.09 (m, 2 H, OCH_2Ph), 5.10 (m, 1 H, H-3''), 5.18 (dd, $^3J_{3'',2''} = 10.3$ Hz, $^2J_{3'',3'''} = 1.1$ Hz, 1 H, H-3''), 5.20 (dd, $^3J_{2''',1'''} = 10.1$ Hz, $^2J_{2''',2'''} = 1.4$ Hz, 1 H, H-2'''), 5.25 (d, $^3J_{2''',1'''} = 17.1$ Hz, 1 H, H-2'''), 5.71 (dd, $^3J_{1''',2'''} = 17.1$ Hz, $^3J_{1''',2''} = 10.1$ Hz, 1 H, H-1'''), 5.72 (dd, $^3J_{2'',3'''} = 17.1$ Hz, $^3J_{2'',3''} = 10.1$ Hz, 1 H, H-2''), 7.30 – 7.37 (m, 5 H, Ph).

^{13}C NMR (126 MHz, CDCl_3): $\delta = 18.5$ (q, C-2'), 43.1 (t, C-1''), 46.1 (d, C-1'), 57.9 (d, C-4), 62.4 (d, C-3), 66.7 (t, OCH_2Ph), 118.6 (t, C-3''), 119.7 (t, C-2'''), 128.0, 128.1, 128.5 (3 d, Ph), 131.7 (d, C-2''), 135.5 (d, C-1'''), 136.4 (s, *ipso*-Ph), 155.9 (s, NHCO), 166.8 (s, C-2).

IR (film): $\tilde{\nu}$ (cm^{-1}) = 3318 (NH), 3066 (CH), 2981 (CH), 2935 (CH), 1746 (C=O), 1644 (C=O, amide I), 1537 (NHCO, amide II), 1242 (CO), 1028 (C=C), 739, 698 (Ph).

MS (EI, 70 eV): m/z (%) 314 (10, M^+), 286 (2, $[\text{M} - \text{CO}]^+$), 223 (12, $[\text{M} - \text{C}_7\text{H}_7]^+$), 179 (8, $\text{C}_{10}\text{H}_{13}\text{NO}_2^+$), 136 (38, $[\text{M} - \text{C}_{10}\text{H}_{12}\text{NO}_2]^+$), 108 (32, $\text{C}_7\text{H}_7\text{O}^+$), 95 (19, $[\text{M} - \text{C}_{10}\text{H}_{12}\text{NO}_2 - \text{C}_3\text{H}_5]^+$), 91 (100, C_7H_7^+), 41 (25, C_3H_5^+), 28 (45, CO^+).

(3R,4R,1'S,E)-, (3S,4S,1'S,E)-, (3R,4R,1'S,Z)- and (3S,4S,1'S,Z)-1-benzyl-3-[1-(benzyloxycarbonylamino)-ethyl]-4-(1-propenyl)-azetidin-2-one (5a–d)



Cbz-Ala- CHN_2 (1.00 g, 4.04 mmol) and *N*-benzyl crotonaldimine (650 mg, 4.08 mmol) were reacted in 1,2-dichlorobenzene (50 mL) in accordance with the general procedure (500 W, 160 °C, 30 min). The solvent was removed *in vacuo*. The diastereoisomeric ratio was determined by ^1H NMR spectroscopy (**5a**:**5b**:**5c**:**5d** 36:36:15:13) and the mixture of isomers was filtered through a pad of SiO_2 (5 g) and separated by medium pressure liquid chromatography (light petroleum/*i*PrOH 99:1) to afford β -lactams **5a** (326 mg, 867 μmol , 21 %), **5b** (326 mg, 867 μmol , 21 %), **5c** (133 mg, 350 μmol , 9 %), and **5d** (109 mg, 287 μmol , 7 %). Isomers **5a** and **5c** are colorless solids, isomers **5b** and **5d** were isolated as colorless oils.

5a:

$C_{23}H_{26}N_2O_3$	calcd. C 72.99	H 6.92	N 7.40
(378.46 g/mol)	found C 72.96	H 6.92	N 7.41

$$[\alpha]_D^{20} = +66 \text{ (} c = 1, \text{CHCl}_3 \text{)}.$$

Mp.: 112 – 114 °C (light petroleum/*i*PrOH).

^1H NMR (500 MHz, CDCl_3): δ = 1.31 (d, $^3J_{2',1'} = 6.9$ Hz, 3 H, H-2'), 1.68 (d, $^3J_{3'',2''} = 6.3$ Hz, 3 H, H-3''), 2.97 (dd, $^3J_{3,4} = 3.9$ Hz, $^3J_{3,1'} = 2.3$ Hz, 1 H, H-3), 3.68 (d, $^3J_{4,1''} = 9.1$ Hz, 1 H, H-4), 3.88 (d, $^2J = 15.1$ Hz, 1 H, NCH_2Ph), 4.11 (m, 1 H, H-1'), 4.65 (d, $^2J = 15.1$ Hz, 1 H, NCH_2Ph), 4.81 (d, $^3J_{\text{NH},1'} = 9.0$ Hz, 1 H, NH), 4.91 (d, $^2J = 12.2$ Hz, 1 H, OCH_2Ph), 5.09 (d, $^2J = 12.3$ Hz, 1 H, OCH_2Ph), 5.33 (dd, $^3J_{1'',2''} = 15.0$ Hz, $^3J_{1'',4} = 9.2$ Hz, 1 H, H-1''), 5.69 (dq, $^3J_{2'',1''} = 15.0$ Hz, $^3J_{2'',3''} = 7.0$ Hz, 1 H, H-2''), 7.18 – 7.37 (m, 10 H, Ph).

^{13}C NMR (126 MHz, CDCl_3): δ = 17.8 (q, C-3''), 19.6 (q, C-2'), 44.2 (t, NCH_2Ph), 45.0 (d, C-1'), 56.1 (d, C-4), 62.0 (d, C-3), 66.8 (t, OCH_2Ph), 127.5 (d, C-1'), 127.9, 128.0, 128.1, 128.2, 128.5, 128.7 (6 d, Ph), 131.9, 135.9 (2 s, *ipso*-Ph), 136.4 (d, C-2''), 156.0 (s, NHCO), 167.1 (s, C-2).

IR (KBr): $\tilde{\nu}$ (cm^{-1}) = 3300 (NH), 3005 (CH), 2960 (CH), 2910 (CH), 2860 (CH), 1735 (C=O), 1673 (C=O, amide I), 1520 (NHCO, amide II), 1240 (CO), 1035 (C=C), 703, 678 (Ph).

MS (CI, NH_3): m/z (%) 380 (8, $[\text{M} + 2 \text{H}]^+$), 379 (45, $[\text{M} + \text{H}]^+$), 350 (47, $[\text{M} - \text{C}_2\text{H}_4]^+$), 287 (20, $[\text{M} - \text{C}_7\text{H}_7]^+$), 245 (40, $[\text{M} + 2 \text{H} - \text{C}_8\text{H}_7\text{O}_2]^+$), 91 (100, C_7H_7^+).

5b:

$C_{23}H_{26}N_2O_3$	calcd. C 72.99	H 6.92	N 7.40
(378.46 g/mol)	found C 72.68	H 6.97	N 7.42

$$[\alpha]_D^{20} = -64 \text{ (} c = 1, \text{CHCl}_3 \text{)}.$$

^1H NMR (500 MHz, CDCl_3): δ = 1.22 (d, $^3J_{2',1'} = 6.6$ Hz, 3 H, H-2'), 1.65 (d, $^3J_{3'',2''} = 6.5$ Hz, 3 H, H-3''), 2.93 (d, $^3J_{3,1'} = 6.8$ Hz, 1 H, H-3), 3.73 (d, $^3J_{4,1''} = 8.6$ Hz, 1 H, H-4), 3.93 (d, $^2J = 15.0$ Hz, 1 H, NCH_2Ph), 4.06 (m, 1 H, H-1'), 4.61 (d, $^2J = 15.0$ Hz, 1 H, NCH_2Ph), 5.06 (d, $^3J_{\text{NH},1'} = 8.6$ Hz, 1 H, NH), 5.05 (d, $^2J = 12.5$ Hz, 1 H, OCH_2Ph), 5.08 (d, $^2J = 12.2$ Hz, 1 H, OCH_2Ph), 5.29 (dd, $^3J_{1'',2''} = 15.1$ Hz, $^3J_{1'',4} = 8.8$ Hz, 1 H, H-1''), 5.60 (dq, $^3J_{2'',1''} = 15.1$ Hz, $^3J_{2'',3''} = 6.6$ Hz, 1 H, H-2''), 7.19 – 7.35 (m, 10 H, Ph).

^{13}C NMR (126 MHz, CDCl_3): δ = 17.8 (q, C-3''), 18.2 (q, C-2'), 44.3 (t, NCH_2Ph), 45.9 (d, C-1'), 57.1 (d, C-4), 62.1 (d, C-3), 66.7 (t, OCH_2Ph), 127.7, 127.8, 128.1, 128.2, 128.3, 128.4, 128.5 (6 d, 1 s, Ph), 128.8 (d, C-1'), 131.6 (s, *ipso*-Ph), 136.2 (d, C-2''), 155.5 (s, NHCO), 167.0 (s, C-2).

IR (film): $\tilde{\nu}$ (cm^{-1}) = 3313 (NH), 3064 (CH), 3030 (CH), 2937 (CH), 2855 (CH), 1737 (C=O), 1586 (NHCO, amide II), 1251 (CO), 1029 (C=C), 752, 698 (Ph).

MS (CI, NH_3): m/z (%) 380 (8, $[\text{M} + 2 \text{H}]^+$), 379 (35, $[\text{M} + \text{H}]^+$), 245 (100, $[\text{M} + 2 \text{H} - \text{C}_8\text{H}_7\text{O}_2]^+$), 287 (8, $[\text{M} - \text{C}_7\text{H}_7]^+$), 91 (73, C_7H_7^+).

5c:

$C_{23}H_{26}N_2O_3$	calcd. C 72.99	H 6.92	N 7.40
(378.46 g/mol)	found C 72.90	H 6.92	N 7.34

$$[\alpha]_D^{20} = +91 \text{ (} c = 0.5, \text{CHCl}_3 \text{)}.$$

Mp.: 119 – 120 °C (light petroleum/*i*PrOH).

^1H NMR (500 MHz, CDCl_3): δ = 1.33 (d, $^3J_{2',1'} = 6.9$ Hz, 3 H, H-2'), 1.50 (d, $^3J_{3'',2''} = 6.5$ Hz, 3 H, H-3''), 2.97 (dd, $^3J_{3,4} = ^3J_{3,1'} = 3.0$ Hz, 1 H, H-3), 3.85 (d, $^2J = 15.0$ Hz, 1 H, NCH_2Ph), 4.09 (d, $^3J_{4,1''} = 10.2$ Hz, 1 H, H-4), 4.13 (ddd, $^3J_{1',\text{NH}} = 9.4$ Hz, $^3J_{1',2'} = 6.7$ Hz, $^3J_{1',3} = 3.7$ Hz, 1 H, H-1'), 4.67 (d, $^2J = 15.1$ Hz, 1 H, NCH_2Ph), 4.90 (d, $^3J_{2',1'} = 9.1$ Hz, 1 H, NH), 4.95 (d, $^2J = 12.2$ Hz, 1 H, OCH_2Ph), 5.09 (d, $^2J = 12.3$ Hz, 1 H, OCH_2Ph), 5.30 (dd, $^3J_{1'',2''} = 10.8$ Hz, $^3J_{1'',4} = 10.3$ Hz, 1 H, H-1''), 5.29 (dq, $^3J_{2'',1''} = 10.7$ Hz, $^3J_{2'',3''} = 7.0$ Hz, 1 H, H-2''), 7.18 – 7.36 (m, 10 H, Ph).

^{13}C NMR (126 MHz, CDCl_3): δ = 13.1 (q, C-3''), 19.9 (q, C-2'), 44.3 (t, NCH_2Ph), 45.1 (d, C-1'), 50.2 (d, C-4), 62.1 (d, C-3), 66.7 (t, OCH_2Ph), 126.9 (d, C-1'), 127.5, 127.9, 128.1, 128.3, 128.5, 128.7 (6 d, Ph), 131.2, 135.8 (2 s, *ipso*-Ph), 136.4 (d, C-2''), 156.0 (s, NHCO), 167.2 (s, C-2).

IR (KBr): $\tilde{\nu}$ (cm^{-1}) = 3283 (NH), 3005 (CH), 2940 (CH), 2897 (CH), 1720 (C=O), 1650 (C=O, amide I), 1529 (NHCO, amide II), 1230 (CO), 1040 (C=C), 735, 685 (Ph).

MS (EI, 70 eV): m/z (%) 378 (1, M^+), 350 (15, $[\text{M} - \text{CO}]^+$), 287 (8, $[\text{M} - \text{C}_7\text{H}_7]^+$), 200 (18, $\text{C}_{10}\text{H}_{12}\text{NO}_2^+$), 137 (35, $\text{C}_8\text{H}_9\text{O}_2^+$), 108 (30, $\text{C}_7\text{H}_8\text{O}^+$), 91 (100, C_7H_7^+), 41 (9, C_3H_5^+), 28 (19, CO^+).

5d:

$C_{23}H_{26}N_2O_3$	calcd. C 72.99	H 6.92	N 7.40
(378.46 g/mol)	found C 72.76	H 6.94	N 7.31

$$[\alpha]_D^{20} = -69 \text{ (} c = 1, \text{CHCl}_3 \text{)}.$$

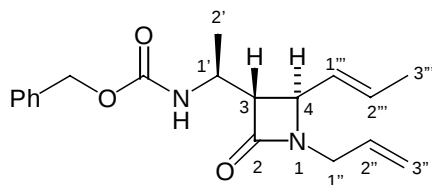
^1H NMR (500 MHz, CDCl_3): δ = 1.27 (d, $^3J_{2',1'} = 6.6$ Hz, 3 H, H-2'), 1.43 (d, $^3J_{3'',2''} = 6.7$ Hz, 3 H, H-3''), 2.90 (d, $^3J_{3,1'} = 7.3$ Hz, 1 H, H-3), 3.89 (d, $^2J = 15.0$ Hz, 1 H, NCH_2Ph), 4.08 (m, 1 H, H-1'), 4.13 (d, $^3J_{4,1''} = 10.0$ Hz, 1 H, H-4), 4.64 (d, $^2J = 15.1$ Hz, 1 H, NCH_2Ph), 5.00 (d, $^3J_{\text{NH},1'} = 9.2$ Hz, 1 H, NH), 5.05 (s, 2 H, OCH_2Ph), 5.24 (dd, $^3J_{1'',2''} = ^3J_{1'',4} = 10.3$ Hz, 1 H, H-1''), 5.67 (dq, $^3J_{2'',1''} = 10.8$ Hz, $^3J_{2'',3''} = 7.0$ Hz, 1 H, H-2''), 7.18 – 7.36 (m, 10 H, Ph).

^{13}C NMR (126 MHz, CDCl_3): δ = 17.8 (q, C-3''), 18.6 (q, C-2'), 44.3 (t, NCH_2Ph), 45.0 (d, C-1'), 51.3 (d, C-4), 62.3 (d, C-3), 66.6 (t, OCH_2Ph), 127.5 (d, C-1'), 127.7, 128.0, 128.1, 128.4 128.5, 128.7 (6 d, Ph), 130.9, 135.8 (2 s, *ipso*-Ph), 136.4 (d, C-2''), 155.7 (s, NHCO), 167.1 (s, C-2).

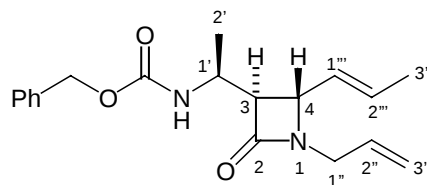
IR (film): $\tilde{\nu}$ (cm^{-1}) = 3311 (NH), 3064 (CH), 3030 (CH), 2972 (CH), 2934 (CH), 1718 (C=O), 1527 (NHCO, amide II), 1249 (CO), 1028 (C=C), 751, 699 (Ph).

MS (EI, 70 eV): m/z (%) 378 (1, M^+), 350 (20, $[\text{M} - \text{CO}]^+$), 287 (10, $[\text{M} - \text{C}_7\text{H}_7]^+$), 200 (21, $\text{C}_{10}\text{H}_{12}\text{NO}_2^+$), 137 (9, $\text{C}_8\text{H}_9\text{O}_2^+$), 108 (8, $\text{C}_7\text{H}_8\text{O}^+$), 91 (100, C_7H_7^+), 41 (4, C_3H_5^+), 28 (9, CO^+).

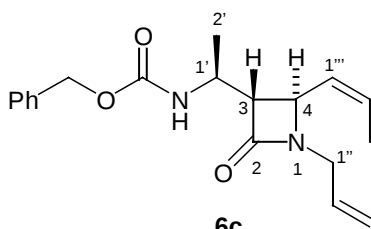
(3R,4R,1'S,E)-, (3S,4S,1'S,E)-, (3R,4R,1'S,Z)- and (3S,4S,1'S,Z)-1-allyl-3-[1-(benzyloxycarbonylamino)-ethyl]-4-(1-propenyl)-azetidin-2-one (**6a** – **d**)



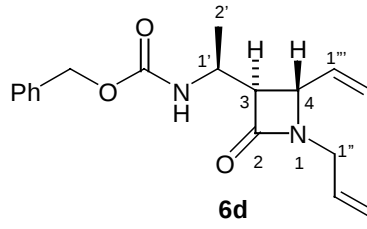
6a



6b



6c



6d

Cbz-Ala-CHN₂ (495 mg, 2.00 mmol) and *N*-allyl crotonaldimine (215 mg, 2.00 mmol) were reacted in 1,2-dichlorobenzene (50 mL) in accordance with the general procedure (800 W, 160 °C, 30 min). The solvent was removed by passing the cooled solution through SiO₂ (100 g, 3 cm × 40 cm; elution with 1 L light petroleum). The products were eluted with light petroleum/ethyl acetate (2:1). The products were eluted with light petroleum/ethyl acetate (3:1). The diastereoisomeric ratio was determined by ¹H NMR spectroscopy (**6a**:**6b**:**6c**:**6d** 38:36:17:9). Medium pressure liquid chromatography (light petroleum/*i*PrOH 95:5) of the mixture of isomers (553 mg, 1.68 mmol, 84 %) afforded the major isomers **6a** (151 mg, 460 μmol, 23 %) and **6b** (142 mg, 378 μmol, 19 %) as colorless oils. The isomers **6c** and **6d** could only be isolated as mixtures of isomers (100 mg, 304 μmol, 15 %).

6a:

C ₁₉ H ₂₄ N ₂ O ₃	calcd. C 69.49	H 7.37	N 8.53
(328.41 g/mol)	found C 69.39	H 7.41	N 8.51

[α]_D²⁰ = +92 (*c* = 0.5, CHCl₃).

¹H NMR (500 MHz, CDCl₃): δ = 1.32 (d, ³*J*_{2',1'} = 7.0 Hz, 3 H, H-2'), 1.71 (d, ³*J*_{3'',2''} = 6.3 Hz, 3 H, H-3'''), 2.94 (dd, ³*J*_{3,4} = ³*J*_{3,1'} = 2.3 Hz, 1 H, H-3), 3.41 (dd, ²*J*_{1'',1''} = 15.8 Hz, ³*J*_{1'',2''} = 6.8 Hz, 1 H, H-1''), 3.80 (d, ³*J*_{4,1'''} = 8.1 Hz, 1 H, H-4), 3.98 (dd, ²*J*_{1'',1''} = 15.8 Hz, ³*J*_{1'',2''} = 5.1 Hz, 1 H, H-1''), 4.15 (m, 1 H, H-1'), 4.90 (d, ³*J*_{NH,1'} = 9.1 Hz, 1 H, NH), 5.06 – 5.10 (m, 2 H, H-3''), 5.11 (d, ²*J* = 12.4 Hz, 1 H, CH₂Ph), 5.15 (d, ²*J* = 12.2 Hz, 1 H, CH₂Ph), 5.35 (dd, ³*J*_{1'',2'''} = 15.0 Hz, ³*J*_{1'',4} = 8.1 Hz, 1 H, H-1''), 5.63 (dddd, ³*J*_{2'',3''} = 17.0 Hz, ³*J*_{2'',3''} = 10.1 Hz, ³*J*_{2'',1''} = 6.7 Hz, ³*J*_{2'',1''} = 5.2 Hz, 1 H, H-2''), 5.76 (dq, ³*J*_{2'',1'''} = 14.9 Hz, ³*J*_{1''',4} = 6.1 Hz, 1 H, H-2'''), 7.30 – 7.37 (m, 5 H, Ph).

¹³C NMR (126 MHz, CDCl₃): δ = 18.0 (q, C-2'), 19.9 (q, C-3'''), 42.7 (t, C-1''), 45.0 (d, C-1'), 56.4 (d, C-4), 62.0 (d, C-3), 66.7 (t, CH₂Ph), 118.0 (t, C-3''), 127.1 (d, C-1'''), 128.0, 128.1, 128.5 (3 d, Ph), 131.0 (d, H-2'''), 131.9 (d, C-2''), 136.4 (s, *ipso*-Ph), 156.1 (s, NHCO), 167.1 (s, C-2).

IR (KBr): $\tilde{\nu}$ (cm⁻¹) = 3302 (NH), 3033 (CH), 2976 (CH), 2938 (CH), 1644 (C=O, amide I), 1538 (NHCO, amide II), 1244 (CO), 738, 679 (Ph).

MS (FAB): m/z (%) 351 (8, $[M + Na]^+$), 330 (20, $[M + 2 H]^+$), 329 (100, $[M + H]^+$), 178 (9, $C_{10}H_{12}NO_2^+$), 152 (19, $[M + 2 H - C_{10}H_{12}NO_2]^+$), 91 (60, $C_7H_7^+$).

6b:

$C_{19}H_{24}N_2O_3$	calcd. C 69.49	H 7.37	N 8.53
(328.41 g/mol)	found C 69.19	H 7.41	N 8.50

$[\alpha]_D^{20} = -41$ ($c = 0.5$, $CHCl_3$).

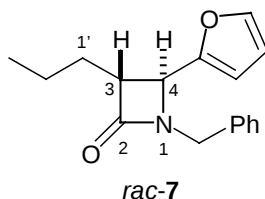
1H NMR (500 MHz, $CDCl_3$): $\delta = 1.28$ (d, $^3J_{2',1'} = 6.7$ Hz, 3 H, H-2'), 1.67 (d, $^3J_{3'',2''} = 6.5$ Hz, 3 H, H-3''), 2.87 (d, $^3J_{3,1'} = 6.5$ Hz, 1 H, H-3), 3.46 (dd, $^2J_{1'',1'} = 15.7$ Hz, $^3J_{1'',2''} = 6.8$ Hz, 1 H, H-1''), 3.90 (d, $^3J_{4,1''} = 8.6$ Hz, 1 H, H-4), 3.99 (dd, $^2J_{1'',1'} = 15.7$ Hz, $^3J_{1'',2''} = 5.3$ Hz, 1 H, H-1'), 4.09 (m, 1 H, H-1'), 5.07 (d, $^2J = 12.0$ Hz, 1 H, CH_2Ph), 5.10 (d, $^2J = 12.2$ Hz, 1 H, CH_2Ph), 5.14 – 5.18 (m, 2 H, NH, H-3''), 5.22 (dd, $^3J_{3'',2''} = 10.2$ Hz, $^2J_{3'',3''} = 1.4$ Hz, 1 H, H-3''), 5.32 (dd, $^3J_{1'',2''} = 15.2$ Hz, $^3J_{1'',4} = 8.8$ Hz, 1 H, H-1''), 5.67 (m, 1 H, H-2''), 5.75 (dd, $^3J_{2'',3''} = 10.2$ Hz, $^3J_{2'',1''} = 5.2$ Hz, 1 H, H-2''), 7.31 – 7.37 (m, 5 H, Ph).

^{13}C NMR (126 MHz, $CDCl_3$): $\delta = 18.2$ (q, C-3''), 18.8 (q, C-2'), 38.2 (d, C-3), 42.2 (t, C-1''), 46.5 (d, C-1'), 57.9 (d, C-4), 67.0 (t, CH_2Ph), 118.6 (t, C-3''), 128.4, 128.5, 128.6 (3 d, Ph), 128.9 (d, C-1''), 131.8 (d, C-2''), 132.6 (d, C-2'), 136.9 (s, *ipso*-Ph), 156.0 (s, NHCO), 167.4 (s, C-2).

IR (KBr): $\tilde{\nu}$ (cm^{-1}) = 3313 (NH), 2970 (CH), 2937 (CH), 1742 (C=O), 1670 (C=O, amide I), 1533 (NHCO, amide II), 1251 (CO), 738, 698 (Ph).

MS (FAB): m/z (%) 351 (9, $[M + Na]^+$), 330 (22, $[M + 2 H]^+$), 329 (100, $[M + H]^+$), 178 (9, $C_{10}H_{12}NO_2^+$), 152 (28, $[M + 2 H - C_{10}H_{12}NO_2]^+$), 91 (70, $C_7H_7^+$).

(3R,4S)- und (3S,4R)-1-benzyl-4-(2-furyl)-3-propyl-azetidin-2-one (7)



1-Diazopentane-2-one (167 mg, 1.49 mmol) and *N*-benzyl furan-2-carbaldimine (302 mg, 1.63 mmol) were reacted in 1,2-dichlorobenzene (50 mL) in accordance with the general procedure (800 W, 160 °C, 30 min). The crude product was purified on SiO_2 (25 g, 2 cm $\times$ 20 cm) by passing of light petroleum (500 mL) and elution of the product with light petroleum/ethyl acetate (4:1). Medium pressure liquid chromatography (light petroleum/ethyl acetate 85:15) afforded β -lactam 7 (249 mg, 810 μ mol, 54 %) as colorless oil.

7:

$C_{17}H_{19}NO_2$	calcd. C 75.81	H 7.11	N 5.20
(269.34 g/mol)	found C 75.66	H 7.12	N 5.34

1H NMR (500 MHz, $CDCl_3$): $\delta = 0.89$ (t, $^3J_{3',2'} = 7.5$ Hz, 3 H, H-3'), 1.42 (m, 2 H, H-2'), 1.63 (m, 1 H, H-1'), 1.81 (m, 1 H, H-1'), 3.36 (ddd, $^3J_{3,1'} = 9.0$ Hz, $^3J_{3,1'} = 5.8$ Hz, $^3J_{3,4} = 2.3$ Hz, 1 H, H-3), 3.84 (d, $^2J = 15.1$ Hz, 1 H, CH_2Ph), 4.11 (d, $^3J_{4,3} = 2.3$ Hz, 1 H, H-4), 4.71 (d, $^2J = 15.1$ Hz, 1 H,

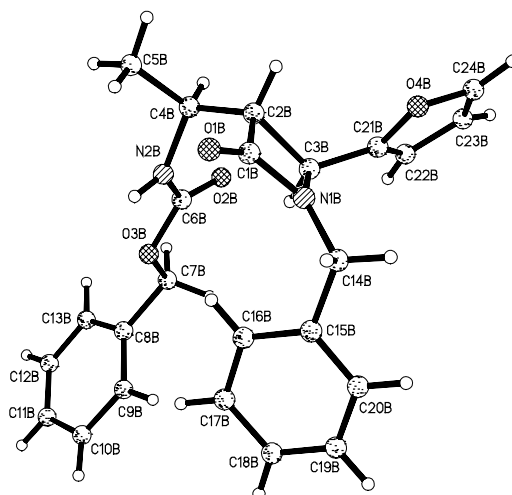
CH₂Ph), 6.21 (d, ³J = 3.2 Hz, 1 H, C₄H₃O), 6.32 (dd, ³J = 3.2 Hz, ³J = 1.8 Hz, 1 H, C₄H₃O), 7.18 (m, 2 H, C₄H₃O, Ph), 7.25 – 7.33 (m, 4 H, Ph).

¹³C NMR (126 MHz, CDCl₃): δ = 13.9 (q, C-3'), 20.4 (t, C-2'), 30.4 (t, C-1'), 44.5 (t, CH₂Ph), 53.6 (d, C-4), 56.9 (d, C-3), 108.8 (d, C₄H₃O), 110.4 (d, C₄H₃O), 127.6, 128.3, 128.7 (3 d, Ph), 135.8 (s, *ipso*-Ph), 143.0 (d, C₄H₃O), 150.9 (s, *ipso*-C₄H₃O), 170.0 (s, C-2).

IR (film): $\tilde{\nu}$ (cm⁻¹) = 2958 (CH), 2930 (CH), 2872 (CH), 1755 (C=O), 739, 701 (Ph).

MS (FAB): *m/z* (%) 270 (1, [M + H]⁺), 269 (2, M⁺), 136 (100, [M + H – C₇H₇ – C₃H₇]⁺), 91 (50, C₇H₇⁺).

Crystal Structure of β-Lactam 1a



Crystal data and structure refinement.

Identification code	s563rm1
Empirical formula	C ₂₄ H ₂₄ N ₂ O ₄
Formula weight	404.45
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	tetragonal
Space group	P4(1)
Unit cell dimensions	a = 9.6260(10) Å alpha = 90 deg. b = 9.6260(10) Å beta = 90 deg. c = 47.354(11) Å gamma = 90 deg.
Volume, Z	4387.8(12) Å ³ , 8
Density (calculated)	1.224 Mg/m ³
Absorption coefficient	0.084 mm ⁻¹
F(000)	1712
Crystal size	0.5 x 0.35 x 0.20 mm
Theta range for data collection	1.29 to 24.00 deg.
Limiting indices	0 ≤ h ≤ 11, 0 ≤ k ≤ 11, 0 ≤ l ≤ 54
Reflections collected	3968
Independent reflections	3483 [R(int) = 0.0264]
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3248 / 1 / 543
Goodness-of-fit on F ²	1.107
Final R indices [I > 2σ(I)]	R1 = 0.0535, wR2 = 0.0859
R indices (all data)	R1 = 0.0930, wR2 = 0.1032

Absolute structure parameter 0(3)
 Extinction coefficient 0.0025(3)
 Largest diff. peak and hole 0.132 and -0.131 e. Å⁻³

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
N(1A)	4813(6)	6892(7)	515(1)	61(2)
C(1A)	3633(8)	7302(10)	643(2)	66(2)
O(1A)	2937(6)	8345(7)	612(1)	82(2)
N(2A)	4658(6)	6885(6)	1262(1)	58(2)
O(2A)	5854(8)	4959(6)	1387(1)	98(2)
C(2A)	3584(8)	5993(8)	823(2)	58(2)
O(3A)	6656(6)	7099(5)	1492(1)	75(2)
C(3A)	5003(8)	5638(8)	691(2)	60(2)
C(4A)	3495(8)	6135(8)	1146(2)	61(2)
C(5A)	2130(9)	6887(9)	1231(2)	78(3)
C(6A)	5749(9)	6210(9)	1382(2)	65(2)
C(7A)	7823(11)	6413(11)	1625(3)	132(5)
C(8A)	8670(11)	7458(10)	1780(3)	80(3)
C(9A)	8731(13)	7441(14)	2072(3)	125(4)
C(10A)	9633(23)	8333(23)	2208(4)	202(11)
C(11A)	10289(23)	9229(18)	2057(7)	193(14)
C(12A)	10296(18)	9338(22)	1770(6)	183(11)
C(13A)	9403(15)	8386(16)	1640(3)	116(4)
C(14A)	5727(9)	7622(9)	321(2)	72(2)
C(15A)	7067(8)	8067(8)	457(2)	53(2)
C(16A)	7014(10)	8830(10)	699(2)	87(3)
C(17A)	8245(12)	9261(11)	835(2)	105(3)
C(18A)	9453(13)	8946(13)	721(2)	110(4)
C(19A)	9536(10)	8214(16)	481(3)	114(4)
C(20A)	8331(10)	7780(10)	347(2)	86(3)
C(21A)	5190(9)	4311(9)	539(2)	59(2)
O(4A)	4437(6)	4110(6)	302(1)	73(2)
C(22A)	5966(10)	3198(10)	597(2)	81(3)
C(23A)	5614(12)	2218(9)	379(2)	88(3)
C(24A)	4738(11)	2793(10)	212(2)	86(3)
N(1B)	5045(7)	1665(7)	2143(1)	62(2)
O(1B)	6842(6)	3289(7)	2082(1)	83(2)
C(1B)	6231(9)	2212(9)	2038(2)	61(2)
N(2B)	5397(6)	1921(6)	1392(1)	55(2)
C(2B)	6426(7)	963(7)	1836(2)	51(2)
O(2B)	4299(7)	-29(6)	1263(1)	80(2)
O(3B)	3464(6)	2070(6)	1148(1)	74(2)
C(3B)	5002(8)	475(8)	1953(2)	59(2)
C(4B)	6625(8)	1233(7)	1522(2)	57(2)
C(5B)	7908(8)	2058(9)	1456(2)	75(2)
C(6B)	4401(9)	1219(8)	1270(2)	59(2)
C(7B)	2336(12)	1348(11)	1014(3)	117(4)
C(8B)	1482(10)	2374(10)	849(2)	74(3)
C(9B)	617(12)	3311(13)	974(2)	101(3)
C(10B)	-152(12)	4258(14)	815(4)	118(4)
C(11B)	-65(15)	4096(15)	523(4)	129(6)
C(12B)	726(17)	3183(18)	404(3)	139(5)

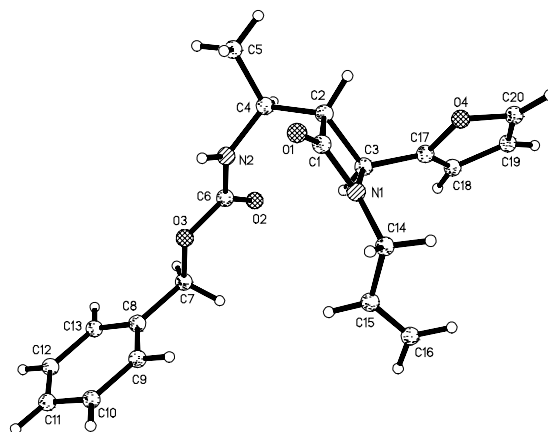
C(13B)	1528(11)	2317(12)	565(3)	111(4)
C(14B)	4087(8)	2211(8)	2350(2)	61(2)
C(15B)	2779(8)	2794(8)	2218(2)	58(2)
C(16B)	2793(10)	3420(10)	1958(2)	89(3)
C(17B)	1567(13)	3924(11)	1844(2)	116(4)
C(18B)	358(12)	3792(12)	1997(3)	108(4)
C(19B)	354(11)	3162(14)	2241(3)	108(4)
C(20B)	1563(9)	2660(9)	2358(2)	78(3)
C(21B)	4856(9)	-907(9)	2083(2)	62(2)
O(4B)	5672(7)	-1191(7)	2305(1)	105(2)
C(22B)	4139(13)	-1999(12)	2007(2)	106(4)
C(23B)	4484(21)	-3081(15)	2177(3)	154(7)
C(24B)	5401(18)	-2595(14)	2360(3)	153(7)

Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$).

	x	y	z	U(eq)
H(2A)	4663(6)	7777(6)	1255(1)	69
H(2A1)	2865(8)	5358(8)	753(2)	69
H(3A)	5751(8)	5779(8)	828(2)	71
H(4A)	3485(8)	5201(8)	1228(2)	74
H(5A1)	2088(9)	6969(9)	1433(2)	117
H(5A2)	1347(9)	6361(9)	1165(2)	117
H(5A3)	2112(9)	7797(9)	1148(2)	117
H(7A1)	8388(11)	5962(11)	1482(3)	159
H(7A2)	7491(11)	5709(11)	1755(3)	159
H(9A)	8172(13)	6837(14)	2176(3)	150
H(10A)	9762(23)	8288(23)	2402(4)	243
H(11A)	10820(23)	9877(18)	2155(7)	232
H(12A)	10836(18)	9976(22)	1672(6)	219
H(13A)	9322(15)	8411(16)	1445(3)	139
H(14A)	5251(9)	8436(9)	249(2)	86
H(14B)	5934(9)	7021(9)	162(2)	86
H(16A)	6157(10)	9069(10)	776(2)	104
H(17A)	8205(12)	9760(11)	1003(2)	126
H(18A)	10266(13)	9239(13)	809(2)	132
H(19A)	10399(10)	7998(16)	405(3)	137
H(20A)	8393(10)	7283(10)	179(2)	103
H(22A)	6591(10)	3083(10)	745(2)	97
H(23A)	5962(12)	1321(9)	362(2)	105
H(24A)	4361(11)	2375(10)	52(2)	104
H(2B)	5341(6)	2812(6)	1398(1)	66
H(2B1)	7168(7)	357(7)	1907(2)	61
H(3B)	4279(8)	612(8)	1811(2)	71
H(4B)	6729(8)	327(7)	1430(2)	69
H(5B1)	7976(8)	2194(9)	1256(2)	112
H(5B2)	8711(8)	1564(9)	1522(2)	112
H(5B3)	7857(8)	2944(9)	1549(2)	112
H(7B1)	1762(12)	898(11)	1155(3)	140
H(7B2)	2698(12)	639(11)	888(3)	140
H(9B)	538(12)	3318(13)	1170(2)	122
H(10B)	-687(12)	4951(14)	899(4)	142
H(11B)	-601(15)	4671(15)	409(4)	155

H(12B)	750(17)	3114(18)	208(3)	167
H(13B)	2113(11)	1680(12)	477(3)	133
H(14C)	4545(8)	2937(8)	2457(2)	73
H(14D)	3836(8)	1476(8)	2480(2)	73
H(16B)	3622(10)	3506(10)	1859(2)	106
H(17B)	1562(13)	4345(11)	1667(2)	140
H(18B)	-465(12)	4155(12)	1926(3)	129
H(19B)	-480(11)	3050(14)	2337(3)	129
H(20B)	1544(9)	2229(9)	2534(2)	94
H(22B)	3495(13)	-2030(12)	1861(2)	127
H(23B)	4145(21)	-3984(15)	2167(3)	184
H(24B)	5806(18)	-3110(14)	2504(3)	184

Crystal Structure of β -Lactam 2a



Crystal data and structure refinement.

Identification code	s689rm
Empirical formula	C ₂₀ H ₂₂ N ₂ O ₄
Formula weight	354.40
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	P2(1)
Unit cell dimensions	a = 9.170(2) Å alpha = 90 deg. b = 9.496(2) Å beta = 111.81(2) deg. c = 12.036(2) Å gamma = 90 deg.
Volume, Z	973.1(4) Å ³ , 2
Density (calculated)	1.210 Mg/m ³
Absorption coefficient	0.085 mm ⁻¹
F(000)	376
Crystal size	0.95 x 0.95 x 0.40 mm
Theta range for data collection	1.82 to 30.00 deg.
Limiting indices	0 ≤ h ≤ 12, 0 ≤ k ≤ 13, -16 ≤ l ≤ 15
Reflections collected	3163
Independent reflections	2997 [R(int) = 0.0450]
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2895 / 1 / 236
Goodness-of-fit on F ²	1.130
Final R indices [I > 2sigma(I)]	R1 = 0.0552, wR2 = 0.1524
R indices (all data)	R1 = 0.0655, wR2 = 0.1695
Absolute structure parameter	2(2)

Extinction coefficient 0.045(8)
 Largest diff. peak and hole 0.223 and -0.177 e. Å⁻³

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

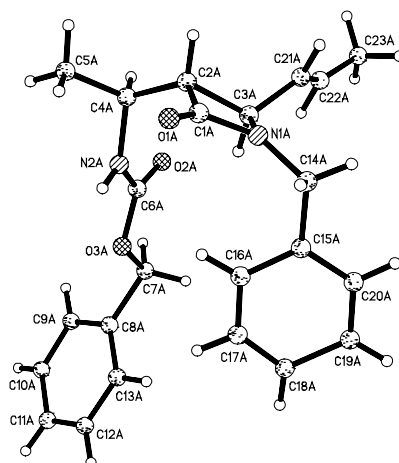
	x	y	z	U(eq)
O(1)	1209(3)	695(3)	2346(2)	77(1)
N(1)	702(3)	2825(3)	3096(2)	64(1)
C(1)	1429(3)	1929(3)	2605(2)	57(1)
N(2)	4518(3)	2115(2)	4414(2)	49(1)
O(2)	4953(3)	4123(2)	5506(2)	79(1)
C(2)	2569(3)	3066(3)	2541(2)	48(1)
O(3)	5046(3)	2000(2)	6360(2)	66(1)
C(3)	1656(3)	4061(3)	3074(2)	54(1)
C(4)	4296(3)	2766(2)	3269(2)	45(1)
O(4)	-172(3)	4927(4)	1169(2)	94(1)
C(5)	4968(4)	1802(4)	2564(3)	66(1)
C(6)	4847(3)	2852(3)	5418(2)	53(1)
C(7)	5367(6)	2735(3)	7470(3)	83(1)
C(8)	5565(4)	1662(3)	8436(2)	62(1)
C(9)	4356(4)	799(5)	8427(3)	82(1)
C(10)	4579(5)	-176(5)	9324(4)	88(1)
C(11)	5999(5)	-292(4)	10236(3)	81(1)
C(12)	7190(4)	570(4)	10265(3)	80(1)
C(13)	6980(4)	1546(4)	9364(3)	69(1)
C(14)	-696(4)	2698(6)	3381(4)	86(1)
C(15)	-439(6)	3152(7)	4607(4)	102(2)
C(16)	-1232(8)	4114(8)	4856(6)	131(2)
C(17)	764(4)	5239(3)	2318(3)	64(1)
C(18)	759(6)	6621(4)	2542(5)	88(1)
C(19)	-263(8)	7254(7)	1489(8)	138(3)
C(20)	-836(5)	6211(8)	688(6)	130(3)

Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$).

	x	y	z	U(eq)
H(2)	4433(3)	1215(2)	4444(2)	59
H(2A)	2398(3)	3314(3)	1712(2)	57
H(3)	2307(3)	4375(3)	3882(2)	65
H(4)	4873(3)	3658(2)	3418(2)	54
H(5A)	6055(4)	1618(4)	3027(3)	100
H(5B)	4877(4)	2252(4)	1826(3)	100
H(5C)	4396(4)	931(4)	2394(3)	100
H(7A)	4505(6)	3365(3)	7402(3)	99
H(7B)	6317(6)	3290(3)	7663(3)	99
H(9)	3378(4)	874(5)	7810(3)	98
H(10)	3755(5)	-758(5)	9306(4)	106
H(11)	6150(5)	-958(4)	10835(3)	97

H(12)	8156(4)	506(4)	10896(3)	96
H(13)	7809(4)	2127(4)	9390(3)	82
H(14A)	-1040(4)	1724(6)	3283(4)	103
H(14B)	-1528(4)	3262(6)	2822(4)	103
H(15)	348(6)	2710(7)	5237(4)	122
H(16A)	-2027(8)	4573(8)	4244(6)	157
H(16B)	-1018(8)	4358(8)	5649(6)	157
H(18)	1325(6)	7073(4)	3258(5)	105
H(19)	-499(8)	8208(7)	1368(8)	165
H(20)	-1576(5)	6333(8)	-82(6)	157

Crystal Structure of β -Lactam 5a



Crystal data and structure refinement.

Identification code	s688rm	
Empirical formula	C ₂₃ H ₂₆ N ₂ O ₃	
Formula weight	378.46	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	tetragonal	
Space group	P4(3)	
Unit cell dimensions	a = 9.6411(11) Å	alpha = 90 deg.
	b = 9.6411(11) Å	beta = 90 deg.
	c = 46.197(9) Å	gamma = 90 deg.
Volume, Z	4294.0(11) Å ³ , 8	
Density (calculated)	1.171 Mg/m ³	
Absorption coefficient	0.078 mm ⁻¹	
F(000)	1616	
Crystal size	1.3 x 1.1 x 0.3 mm	
Theta range for data collection	1.32 to 24.00 deg.	
Limiting indices	0<=h<=11, 0<=k<=11, 0<=l<=52	
Reflections collected	3886	
Independent reflections	3416 [R(int) = 0.0523]	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3291 / 13 / 507	
Goodness-of-fit on F ²	1.038	
Final R indices [I>2sigma(I)]	R1 = 0.1153, wR2 = 0.2920	
R indices (all data)	R1 = 0.1365, wR2 = 0.3684	
Absolute structure parameter	0(5)	
Extinction coefficient	0.031(6)	

Largest diff. peak and hole

0.589 and -0.358 e. Å⁻³

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
N(1A)	2920(10)	4609(9)	7979(2)	56(2)
O(1A)	1240(11)	2952(10)	8096(2)	81(3)
C(1A)	2409(13)	3530(11)	8123(2)	58(3)
N(2A)	2883(9)	4562(10)	8758(2)	54(2)
O(2A)	4837(9)	5733(12)	8848(2)	84(3)
C(2A)	3650(14)	3353(12)	8303(3)	61(3)
O(3A)	2752(8)	6587(9)	8976(2)	68(2)
C(3A)	4168(12)	4714(13)	8154(3)	61(3)
C(4A)	3573(14)	3322(13)	8636(2)	62(3)
C(5A)	2779(16)	2022(13)	8739(3)	76(3)
C(6A)	3612(12)	5623(13)	8860(2)	62(3)
C(7A)	3483(18)	7820(16)	9076(4)	92(5)
C(8A)	2597(14)	8596(16)	9292(3)	75(3)
C(9A)	2919(18)	8523(19)	9581(3)	85(4)
C(10A)	2083(26)	9337(28)	9791(4)	119(7)
C(11A)	975(26)	10061(26)	9692(5)	118(7)
C(12A)	688(20)	10084(20)	9417(7)	118(8)
C(13A)	1469(16)	9391(15)	9213(3)	77(3)
C(14A)	2318(15)	5602(13)	7783(2)	64(3)
C(15A)	1948(13)	6957(13)	7917(2)	57(3)
C(16A)	1346(16)	6998(15)	8189(3)	78(4)
C(17A)	980(21)	8252(18)	8313(4)	94(5)
C(18A)	1229(23)	9525(21)	8176(5)	106(6)
C(19A)	1870(22)	9436(17)	7901(5)	105(6)
C(20A)	2248(16)	8198(12)	7778(3)	70(3)
C(21A)	5599(18)	4731(17)	8013(3)	80(4)
C(22A)	6613(17)	5443(29)	8066(4)	119(8)
C(23A)	8024(29)	5622(42)	7975(6)	154(10)
N(1B)	-2025(12)	15163(11)	9635(2)	66(3)
O(1B)	-3700(12)	16825(11)	9524(2)	81(3)
C(1B)	-2576(14)	16249(13)	9499(3)	66(3)
N(2B)	-2109(10)	15290(10)	8875(2)	57(2)
O(2B)	-151(10)	14079(11)	8772(2)	81(3)
C(2B)	-1246(13)	16457(11)	9317(3)	60(3)
O(3B)	-2242(9)	13214(9)	8660(2)	66(2)
C(3B)	-736(15)	15078(12)	9460(3)	65(3)
C(4B)	-1381(13)	16497(12)	8979(3)	62(3)
C(5B)	-2094(16)	17805(14)	8874(3)	76(3)
C(6B)	-1380(11)	14174(13)	8765(2)	53(2)
C(7B)	-1623(17)	11932(16)	8560(4)	87(5)
C(8B)	-2542(14)	11251(14)	8352(3)	70(3)
C(9B)	-3628(21)	10452(18)	8453(3)	90(5)
C(10B)	-4532(21)	9798(22)	8260(5)	110(6)
C(11B)	-4197(38)	9925(33)	7969(8)	164(15)
C(12B)	-3139(32)	10735(25)	7872(4)	121(8)
C(13B)	-2386(22)	11389(20)	8054(5)	105(6)
C(14B)	-2631(18)	14171(16)	9831(3)	78(4)
C(15B)	-2988(12)	12781(12)	9674(3)	60(3)

C(16B)	-2646(18)	11533(17)	9799(4)	87(4)
C(17B)	-3020(21)	10338(16)	9676(5)	102(6)
C(18B)	-3781(19)	10333(16)	9430(4)	89(5)
C(19B)	-4077(18)	11589(19)	9285(4)	92(5)
C(20B)	-3735(17)	12770(15)	9425(3)	75(4)
C(21B)	579(18)	15198(21)	9625(3)	91(5)
C(22B)	1642(20)	14375(31)	9567(4)	119(8)
C(23B)	3067(22)	14637(35)	9728(7)	159(11)

Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$).

	x	y	z	U(eq)
H(2A)	1992(9)	4596(10)	8764(2)	65
H(2A1)	4192(14)	2560(12)	8233(3)	74
H(3A)	4084(12)	5494(13)	8289(3)	73
H(4A)	4520(14)	3278(13)	8713(2)	75
H(5A1)	2739(16)	2015(13)	8947(3)	114
H(5A2)	3251(16)	1204(13)	8673(3)	114
H(5A3)	1855(16)	2037(13)	8662(3)	114
H(7A1)	3693(18)	8416(16)	8913(4)	110
H(7A2)	4351(18)	7551(16)	9167(4)	110
H(9A)	3651(18)	7973(19)	9644(3)	102
H(10A)	2315(26)	9349(28)	9986(4)	143
H(11A)	417(26)	10544(26)	9822(5)	142
H(12A)	-78(20)	10591(20)	9355(7)	142
H(13A)	1230(16)	9462(15)	9019(3)	92
H(14A)	1487(15)	5201(13)	7700(2)	77
H(14B)	2967(15)	5769(13)	7627(2)	77
H(16A)	1187(16)	6176(15)	8289(3)	94
H(17A)	552(21)	8250(18)	8493(4)	113
H(18A)	990(23)	10368(21)	8260(5)	127
H(19A)	2042(22)	10250(17)	7799(5)	126
H(20A)	2707(16)	8189(12)	7601(3)	84
H(21A)	5716(18)	4097(17)	7863(3)	96
H(22A)	6404(17)	6074(29)	8212(4)	143
H(23A)	8443(29)	6354(42)	8085(6)	231
H(23B)	8047(29)	5855(42)	7773(6)	231
H(23C)	8527(29)	4775(42)	8006(6)	231
H(2B)	-3000(10)	15265(10)	8881(2)	68
H(2B1)	-710(13)	17251(11)	9388(3)	73
H(3B)	-737(15)	14293(12)	9326(3)	78
H(4B)	-443(13)	16480(12)	8897(3)	74
H(5B1)	-2154(16)	17787(14)	8666(3)	114
H(5B2)	-1569(16)	18601(14)	8933(3)	114
H(5B3)	-3010(16)	17855(14)	8954(3)	114
H(7B1)	-1461(17)	11322(16)	8724(4)	104
H(7B2)	-736(17)	12126(16)	8469(4)	104
H(9B)	-3759(21)	10348(18)	8651(3)	108
H(10B)	-5309(21)	9310(22)	8322(5)	132
H(11B)	-4718(38)	9435(33)	7834(8)	197
H(12B)	-2966(32)	10811(25)	7675(4)	145
H(13B)	-1705(22)	11984(20)	7984(5)	127
H(14C)	-3470(18)	14559(16)	9914(3)	94

H(14D)	-1987(18)	13987(16)	9988(3)	94
H(16B)	-2147(18)	11521(17)	9971(4)	105
H(17B)	-2755(21)	9502(16)	9759(5)	123
H(18B)	-4111(19)	9498(16)	9356(4)	106
H(19B)	-4487(18)	11601(19)	9103(4)	111
H(20B)	-4022(17)	13611(15)	9347(3)	90
H(21B)	658(18)	15858(21)	9771(3)	109
H(22B)	1554(20)	13657(31)	9433(4)	143
H(23D)	3737(22)	13966(35)	9665(7)	239
H(23E)	2932(22)	14550(35)	9933(7)	239
H(23F)	3396(22)	15553(35)	9684(7)	239
