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Supporting Information for:

α -Alkyl- α -Amino- β -Lactam Peptides: Design, Synthesis and Conformational Features.

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The following information is provided:

- 1.- The general procedures for the preparation and a listing of the physical and spectroscopical data of compounds **4a-f** , **6**, **7**, **8** and **10**.
- 2.- ROESY NMR plot of compound **10** in CDCl₃.

EXPERIMENTAL PROCEDURES:

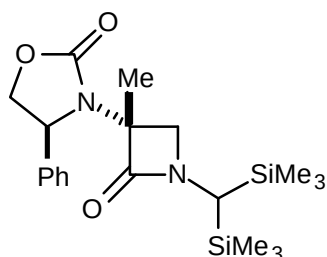
(3R)-3-Alkyl-1-[bis(trimethylsilyl)methyl]-3-[(4*S*)-4-phenyl-2-oxo-oxazolidin-3-yl]azetidin-2-ones (**4a-f**): LDA was formed by slow addition of 1.4 M *n*-butyllithium (3.12 ml, 6 mmol) to a cooled (-78°C) solution of diisopropylamine (0.84 ml, 6 mmol) in THF (10 ml) under a nitrogen atmosphere and stirring at the same temperature for 30 min. Then, a solution of the β -lactam **3** (1.56 g, 4 mmol) in THF (8 ml) was added and the mixture was stirred for 1h at -78°C. Finally, the corresponding alkyl halide (20 mmol, freshly distilled) was dropwise added and the dry-ice/acetone bath was allowed to reach room temperature overnight. The reaction mixture was taken up over CH₂Cl₂ (30 ml), washed successively with sat. NH₄Cl (15 ml) and water (15 ml) and dried over MgSO₄. Evaporation of volatiles afforded crude 3-alkyl-azetidin-2-ones **4a-f**, which were purified by flash chromatography. The transformation of β -lactams **4** into **6–8** is performed following the method described in ref. [12].

(R)-3-Benzyl-3-*tert*-butoxycarbonylamino-1-carboxymethylazetidin-2-one (**9**): To a solution of *(R)*-3-benzyl-3-*tert*-butoxycarbonylaminoazetidin-2-one **8** (5 mmol, 1.38 g) in dry acetonitrile (75 ml) was added Cs₂CO₃ (5.35 mmol, 1.75 g) and freshly distilled benzyl bromoacetate (7.5 mmol, 1.20 ml). The mixture was stirred at room temperature for 90 min. After this time the salts were filtered off from the reaction mixture, water was added (50 ml) and the mixture was extracted with EtOAc (3 x 20 ml). The organic phase was dried with anhydrous MgSO₄ and evaporated under reduced pressure to afford crude *(R)*-3-benzyl-1-benzyloxycarbonylmethyl-3-*tert*-butoxycarbonylaminoazetidin-2-one, which was purified by flash column chromatography (silicagel-60, eluant: EtOAc/hexanes 1:10) (Yield: 87%). This product was dissolved in EtOAc (25 ml) containing Pd(10%)/C (0.20 g) and the mixture was stirred overnight under hydrogen (1 atm.) at room temperature. After this time, the mixture was filtered through a pad of celite and extracted with EtOAc. The solvents were evaporated under reduced pressure to give quantitative yield of **9** (2.10g, 85% overall from **8**).

(R)-3-Benzyl-3-*tert*-butoxycarbonylamino-1-[3-aza-4-carboxy-4-methyl-2-oxo-hexyl]-azetidin-2-one (**10**): To a solution of **9** (10 mmol) in dry CH₂Cl₂ (25 ml) cooled to -20°C was added pyridine (0.8 ml, 10 mmol) and cyanuric fluoride (4.50 ml, 50 mmol). The mixture was stirred at -20°C during 1.5 h. and ice was added. The organic layer was separated, dried over MgSO₄ and evaporated under reduced pressure to afford crude *(R)*-3-benzyl-3-*tert*-butoxycarbonyl-amino-1-fluorocarboxymethyl-azetidin-2-one, which was dissolved in dry CH₂Cl₂ (6 ml). This solution was added to freshly prepared benzyl 2-aminoisobutyrate (0.55g, 2.8 mmol) and *N*-methylmorpholine (0.5 ml, 4.5 mmol). The mixture was stirred at room temperature for 1.5h. After this time, more CH₂Cl₂ (15 ml) was added and the organic layer was washed with water (2 x 10 ml), HCl 1M (10 ml) and NaHCO₃ (sat. soln. 10 ml). Then, it was dried over MgSO₄ and the solvents evaporated under reduced pressure to afford crude *(R)*-3-benzyl-3-*tert*-butoxycarbonylamino-1-[3-aza-5-

benzyloxycarbonyl-4-methyl-2-oxo-pentyl]-azetidin-2-one, which was purified by column chromatography (silicagel-60, eluant: EtOAc/hexanes 1:1) (Yield: 1.53g, 64 %). This product was dissolved in EtOAc (15 ml) containing Pd(10%)/C (0.12 g) and the mixture was stirred overnight under hydrogen (1 atm.) at room temperature. After this time, the mixture was filtered through a pad of celite and extracted with ethyl acetate. The solvents were evaporated under reduced pressure to give **9** (0.76 g, 60% overall from **10**).

(3R)-1-[Bis(trimethylsilyl)methyl]-3-methyl-3-[(4S)-4-phenyl-2-oxo-oxazolidin-3-yl]-azetidin-2-one



Reference n°

4a

Mol. W. (Dalton)

404.64

Yield (%)

65 (3R)+(3S) mixture

Melting.P. (°C)

$[a]_D^{25}$ (c= ,)

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₀ H ₃₂ N ₂ O ₃ Si ₂	C 59.37	H 7.97	N 6.92	C	H	N

IR (cm ⁻¹ , film)	1740 (CO), 842 (C-Si)
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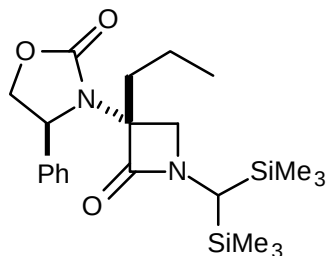
MS m/z (rel. int.)	389(M-15)(5), 276(14), 203(40), 202(26), 138(13), 118 (100), 104(38), 73(55), 68(40)
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¹ H-NMR (δ, ppm) CDCl ₃	¹³ C-NMR (δ, ppm) CDCl ₃
7.47-7.33 (m, 5H, Arom)	165.8
5.01 (dd, 1H, J=2.2Hz, J'=8.7Hz, <u>H</u> CPh)	158.0
4.62 (t, 1H, J=8.7Hz, <u>H</u> CHO)	140.2
4.30 (dd, 1H, J=2.2Hz, J'=8.7Hz, H <u>C</u> HO)	129.0
3.73 (d, 1H, J=6.1Hz, <u>H</u> CH)	128.8
3.27 (d, 1H, J=6.6Hz, H <u>C</u> H)	127.0
2.73 (s, 1H, <u>H</u> CSi)	71.0
1.18 (s, 3H, CH ₃)	66.9
0.15 (s, 9H, Si(CH ₃) ₃)	59.6
0.12 (s, 9H, Si(CH ₃) ₃)	57.1
	36.7
	19.2
	-0.3

Comments:

Spectroscopic data corresponding to the major (3R) isomer.

(3R)-1-[Bis(trimethylsilyl)methyl]-3-[(4S)-4-phenyl-2-oxo-oxazolidin-3-yl]-3-propyl-azetidin-2-one



Reference n°
4b

Mol. W. (Dalton)
432.58

Yield (%)
40

Melting.P. (°C)
Oil

$[\alpha]_D^{25}$ (c=0.61, CH₂Cl₂)
+82.1

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₂ H ₃₆ N ₂ O ₃ Si ₂	C 61.08	H 8.40	N 6.47	C	H	N

IR (cm ⁻¹ , KBr)	1739 (CO), 844 (C-Si)
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MS m/z (rel. int.)	203 (22), 172 (5), 105 (10), 104 (92), 97 (13), 91 (12), 73 (100)
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¹H-NMR (δ, ppm) CDCl₃

7.55-7.30 (m, 5H, Arom)
5.10 (dd, 1H, J=2.1Hz, J'=8.3Hz, HCPh)
4.61 (t, 1H, J=8.4Hz, HCHO)
4.37 (dd, 1H, J=2.2Hz, J'=8.6Hz, HCHO)
3.72 (d, 1H, J=6.6Hz, HCH)
3.43 (d, 1H, J=6.6Hz, HCH)
2.73 (s, 1H, HCSi)
1.41-0.84 (m, 4H, CH₂CH₂CH₃)
0.63 (t, 3H, J=6.7Hz, CH₂CH₂CH₃)
0.13 (s, 9H, Si(CH₃)₃)

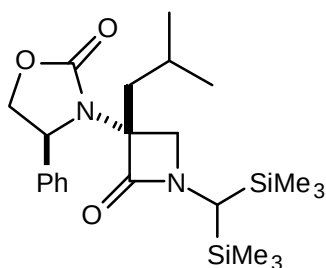
¹³C-NMR (δ, ppm) CDCl₃

165.3
156.9
140.3
128.9
128.8
127.3
71.0
59.5
53.7
36.9
34.8
17.6
13.8
- 0.3
- 0.4

Comments:

Product purified by preparative HPLC (fused silicagel column, Lichrosorb Si 60, 7μm, 250x25mm, eluant AcOEt/Hx, 1/3, v=10ml/min), r.t.: 10.7min

(3R)-1-[Bis(trimethylsilyl)methyl]-3-[2-methyl-propyl]-3-[(4S)-4-phenyl-2-oxo-oxazolidin-3-yl]-azetidin-2-one



Reference n°

4c

Mol. W. (Dalton)

446.74

Yield (%)

62

Melting.P. (°C)

99-100

$[\alpha]_D^{25}$ (c= 0.59, Cl₂CH₂)
-26.78

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₃ H ₃₈ N ₂ O ₃ Si ₂	C 61.84	H 8.57	N 6.27	C 61.51	H 8.25	N 6.40

IR (cm ⁻¹ , film)	1738 (CO), 848 (C-Si)
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MS m/z (rel. int.)	246(37), 205(41), 204(98), 118(39), 104(99), 73(100), 68(96)
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¹H-NMR (δ, ppm) CDCl₃

7.47-7.33 (m, 5H, Arom)
 5.19 (dd, 1H, J=2.3Hz, J'=8.4Hz, HCPh)
 4.61 (t, 1H, J=8.6Hz, HCHO)
 4.41 (dd, 1H, J=2.3Hz, J'=8.4Hz, HCHO)
 3.89 (d, 1H, J=6.8Hz, HCH)
 3.50 (d, 1H, J=6.8Hz, HCH)
 2.70 (s, 1H, HCSi)
 1.63-1.30 (m, 3H, CH₂, CH)
 0.89 (d, 3H, J=6.3Hz, CH₃)
 0.49 (d, 3H, J=6.3Hz, CH₃)
 0.15 (s, 9H, Si(CH₃)₃)
 0.12 (s, 9H, Si(CH₃)₃)

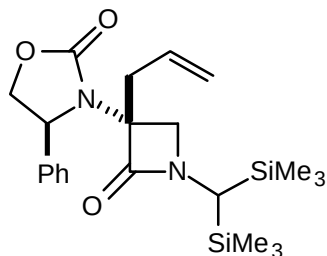
¹³C-NMR (δ, ppm) CDCl₃

165.9
 140.4
 128.8
 127.7
 71.0
 70.7
 59.5
 54.5
 41.9
 37.0
 24.1
 22.9
 -0.2
 -0.3

Comments:

Crystallized from hexane.

(3R)-3-Allyl-1-[bis(trimethylsilyl)methyl]-3-[(4S)-4-phenyl-2-oxo-oxazolidin-3-yl]-azetidin-2-one



Reference n°
4d

Mol. W. (Dalton)
430.56

Yield (%)
70

Melting.P.(°C)
111-112 (Hx)

$[\alpha]_D^{25}$ (c=1.0, Cl₂CH₂)
- 16.8

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₂ H ₃₄ N ₂ O ₃ Si ₂	C 61.37	H 7.97	N 6.50	C 61.55	H 8.15	N 6.23

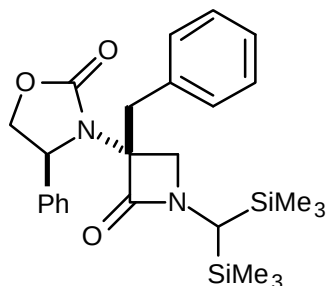
IR (cm ⁻¹ , KBr)	
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MS m/z	229 (11), 170 (7), 104 (45), 67 (14), 91 (13)
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¹ H-NMR (δ, ppm) CDCl ₃	¹³ C-NMR (δ, ppm) CDCl ₃
7.53-7.26 (m, 5H, Arom)	164.4
5.65-5.49 (m, 1H, <u>H</u> C=CH ₂)	156.7
5.10-4.98 (m, 3H, <u>H</u> CPh, <u>H</u> CHO, <u>H</u> CH=CH)	140.1
4.59 (t, 1H, J=8.6Hz, <u>H</u> CHO)	131.7
4.35 (dd, 1H, J=2.0Hz, J'=8.6Hz, <u>H</u> CH=CH)	129.0
3.59 (d, 1H, J=6.4Hz, <u>H</u> CH)	127.4
3.49 (d, 1H, J=6.5Hz, <u>H</u> CH)	119.9
2.70 (s, 1H, <u>H</u> CSi)	71.2
2.31 (dd, 1H, J=8.5Hz, J'=14.2Hz, <u>H</u> CHCH)	70.3
1.83 (dd, 1H, J=6.1Hz, J'=14.1Hz, <u>H</u> CHCH)	59.6
0.13 (s, 9H, Si(CH ₃) ₃)	52.9
0.11 (s, 9H, Si(CH ₃) ₃)	37.1
	36.4
	- 0.2
	- 0.3

Comments:

(3R)-3-Benzyl-1-[bis(trimethylsilyl)methyl]-3-[(4S)-4-phenyl-2-oxo-oxazolidin-3-yl]-azetidin-2-one



Reference n°
4f

Mol. W. (Dalton)
480.62

Yield (%)
90

Melting.P. (°C)
168-169 (Hx)

$[\alpha]_D^{25}$ (c=1.0, CH₂Cl₂)
+ 21.5

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₆ H ₃₆ N ₂ O ₃ Si ₂	C 64.97	H 7.56	N 5.83	C 64.89	H 7.60	N 5.92
IR (cm ⁻¹ , KBr)	1737 (CO), 1730 (CO), 846 (C-Si)					
MS m/z	279 (13), 172 (3), 104 (25), 91 (19), 73 (100)					

¹H-NMR (δ, ppm) CDCl₃

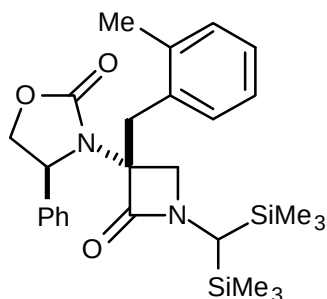
7.70-7.17 (m, 10H, Arom)
5.18 (dd, 1H, J=8.1Hz, J'=1.8Hz, HCPh)
4.64 (t, 1H, J=8.5Hz, HCHO)
4.43(dd, 1H, J=8.7Hz, J'=1.9Hz, HCHO)
3.52 (d, 1H, J=6.6Hz, HCH)
3.44 (d, 1H, J=6.4Hz, HCH)
2.85 (d, 1H, J=13.7Hz, HCHPh)
2.52 (s, 1H, HCSi)
2.24 (d, 1H, J=13.7Hz, HCHPh)
0.00 (s, 9H, Si(CH₃)₃)
- 0.12 (s, 9H, Si(CH₃)₃)

¹³C-NMR (δ, ppm) CDCl₃

164.0 - 0.8
156.7
140.1
134.8
130.5
129.0
128.5
127.7
126.9
72.0
71.1
59.7
52.1
37.4
37.2
- 0.6

Comments:

(3R)-1-[Bis(trimethylsilyl)methyl]-3-[(4S)-4-phenyl-2-oxo-oxazolidin-3-yl]-3-(2-methyl-benzyl)-azetidin-2-one



Reference n°
4e

Mol. W. (Dalton)
494.92

Yield (%)
80

Melting.P. (°C)
152-153 (Hx)

$[\alpha]_D^{25}$ (c=1.0, Cl₂CH₂)
+ 32.1

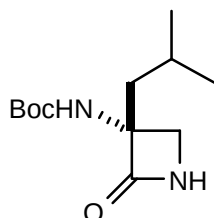
Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₇ H ₃₈ N ₂ O ₃ Si ₂	C 65.52	H 7.75	N 5.66	C 65.05	H 7.94	N 5.80

IR (cm ⁻¹ , KBr)	1728(CO), 840(C-Si)
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MS m/z	73(100), 91(13), 103(15), 104(25), 130(82), 158(7), 293(34), 294(8)
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¹ H-NMR (δ, ppm) CDCl ₃	¹³ C-NMR (δ, ppm) CDCl ₃	
7.66-7.62 (m, 3H, Arom)	164.2	34.3
7.41-7.35 (m, 2H, Arom)	156.8	19.5
7.05- 7.00 (m, 2H, Arom)	140.3	- 0.6
5.15 (dd, 1H, J=1.5Hz, J'=8.1Hz, <u>H</u> CPh)	137.5	- 0.7
4.64 (t, 1H, J=8.3Hz, <u>H</u> CH)	133.4	
4.45 (dd, 1H, J=1.5Hz, J'=8.8Hz, H <u>C</u> H)	131.4	
3.48 (d, 1H, J=6.6Hz, <u>H</u> CH)	130.5	
3.34 (d, 1H, J=6.6Hz, H <u>C</u> H)	129.0	
2.88 (d, 1H, J=14.1Hz, <u>H</u> CHPh)	127.8	
2.51 (s, 1H, HCSiMe ₃)	127.2	
2.20 (d, 1H, J=13.4Hz, H <u>C</u> HPh)	126.1	
2.18 (s, 3H, CH ₃)	71.9	
- 0.02 (s, 9H, SiMe ₃)	71.2	
- 0.19 (s, 9H, SiMe ₃)	59.9	
	52.2	
	37.5	

Comments:

(R)-3-*tert*-Butoxycarbonylamino-3-[2-methyl-propyl]-azetidin-2-oneReference n°
6P. Mol. (g/mol)
242.31Melt. Point.(°C)
118(Hx) $[\alpha]_D^{25}$ (c=1.19, Cl₂CH₂)
-4.29

Empirical Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₁₂ H ₂₂ N ₂ O ₃	C 59.48	H 9.15	N 11.56	C 59.43	H 9.12	N 11.51

IR (cm ⁻¹ , KBr)	3250.8, 2962 cm ⁻¹ (NH) ; 1711.1 cm ⁻¹ (C=O)
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MS m/z	141 (51.1); 101 (100); 57 (71.5)
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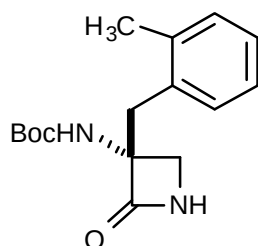
¹H-RMN (δ, ppm) CDCl₃

6.05ppm (s, 1H, NHBoc)
 4.94ppm (s, 1H, NH)
 3.75ppm (s(ancho), 1H, HCHNH)
 3.31ppm (d, 1H, HCHNH) J = 5.16Hz
 1.89-1.69ppm (m, 3H, HCHCH(CH₃)₂)
 1.44ppm (s, 9H, (CH₃)₃CCOO)
 0.98ppm (t, 6H, CH(CH₃)₂)

¹³C-RMN (δ, ppm) CDCl₃

170.9
 154.2
 80.2
 68.7
 49.0
 42.5
 28.2
 24.5
 24.0
 22.9

Comments:

(R)-3-*tert*-Butoxycarbonylamino-3-(2-methyl-benzyl)-azetidin-2-one

Reference n°

7

P. Mol. (g/mol)

290.36

Melt. Point.(°C)

130-131 (Hx)

 $[\alpha]_{\text{D}}^{25}$ (c=1.09, Cl₂CH₂)

+55.7

Empirical Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₁₆ H ₂₂ N ₂ O ₃	C 66.19	H 7.64	N 9.65	C 66.18	H 7.48	N 9.50

IR (cm ⁻¹ , KBr)	3314, 2979.1 cm ⁻¹ (NH); 1750.6, 1725.1, 1699 cm ⁻¹
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MS m/z	
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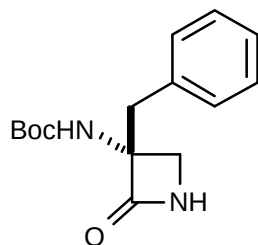
¹H-RMN (δ, ppm) CDCl₃

7.24-7.16ppm (m, 4H, ar.)
 5.58ppm (s, 1H, NH_{Boc})
 5.02ppm (s, 1H, NH)
 3.75ppm (d, 1H, J = 5.1Hz, H_{CH}Ph(Me))
 3.36ppm (d, 1H, J = 5.5Hz, H_{CH}Ph(Me))
 3.26ppm (d, 1H, J = 14.6Hz, H_{CH}NH)
 3.15ppm (d, 1H, J = 14.8Hz, H_{CH}NH)
 2.37ppm (s, 3H, CH₃)
 1.46ppm (s, 9H, C(CH₃)₃)

¹³C-RMN (δ, ppm) CDCl₃

169.5
 154.3
 137.3
 133.1
 130.6
 129.9
 127.2
 126.1
 80.5
 69.2
 47.8
 35.9
 28.3
 19.9

Comments:

(3R)-3-Benzyl-3-*tert*-butoxycarbonylamino-azetidin-2-one

Reference n°

8Mol. W. (g/mol)
276.35Yield (%)
70Melting.P. (°C)
108-110 (Hx/Et₂O)[α]_D²⁵ (c=1.0, Cl₂CH₂)
+ 30.7

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₁₅ H ₂₀ N ₂ O ₃	C 65.19	H 7.31	N 10.13	C 65.13	H 7.44	N 9.60

IR (cm ⁻¹ , KBr)	3273, 2961 (NH); 1759, 1701 (CO)
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MS m/z	265(1), 233 (4), 177 (100), 133 (12), 116 (10), 92 (29), 91 (24), 57 (9)
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¹H-NMR (δ, ppm) DMSO 90°C

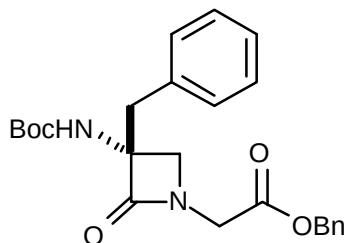
7.43 (s, 1H, NH)
 7.29 (s, 5H, arom)
 6.61 (s, 1H, NH)
 3.38 (d, 1H, J=5.3Hz, HCH)
 3.12 (d, 1H, J=5.3Hz, HCH)
 3.06 (s, 2H, CH₂Ph)
 1.41 (s, 9H, (CH₃)₃)

¹³C-NMR (δ, ppm) CDCl₃

169.6
 154.3
 134.7
 130.0
 128.6
 127.2
 80.4
 69.2
 47.7
 39.5
 28.2

Comments:

(3R)-3-Benzyl-1-benzyloxycarbonylmethyl-3-tert-butoxycarbonylamino-azetidin-2-one



Reference n°

Mol. W. (Dalton)
424.52

Yield (%)
50

Melting.P. (°C)
Oil

$[\alpha]_D^{25}$ (c=1.0, Cl₂CH₂)

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₄ H ₂₈ N ₂ O ₅	C 59.44	H 9.11	N 8.15	C	H	N

IR (cm ⁻¹ , KBr)	3350, 2961 (NH); 1763, 1743, 1708 (CO)
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MS m/z	
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¹H-NMR (δ, ppm) CDCl₃

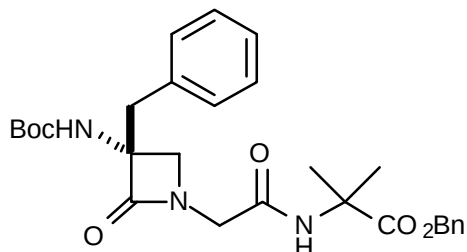
7.35-7.20 (m, 10H, Arom)
5.18 (d, 1H, J=12.0Hz, OHCHPh)
5.10 (d, 1H, J=12.1Hz, OHCHPh)
4.92 (s, 1H, NH)
4.20 (d, 1H, J=18.1Hz, HCHCO)
3.83 (d, 1H, J=18.1Hz, HCCHCO)
3.74 (d, 1H, J=4.9Hz, HCH)
3.62 (d, 1H, J=5.1Hz, HC)
3.26 (d, 1H, J=14.1Hz, HCHPh)
3.14 (d, 1H, J=14.1Hz, HCPh)
1.42 (s, 9H, (CH₃)₃)

¹³C-NMR (δ, ppm) CDCl₃

168.8
167.7
154.2
134.8
129.8
128.6
128.4
127.2
80.3
68.3
67.2
52.9
42.6
39.3
28.2

Comments:

(R)-3-Benzyl-3-*tert*-butoxycarbonylamino-1-[3-aza-5-benzyloxy-carbonyl-4-methyl-2-oxo-pentyl]-azetidin-2-one



Reference n°

Mol. W. (Dalton)
509.63

Yield (%)
64 (from 7)

Melting.P. (°C)
Oil

$[\alpha]_D^{25}$ (c=0.95, Cl₂CH₂)
+ 81.8

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₈ H ₃₅ N ₃ O ₆	C 65.98	H 6.94	N 8.24	C	H	N

IR (cm ⁻¹ , KBr)	3274, 2966(NH), 1757, 1735, 1658(CO)
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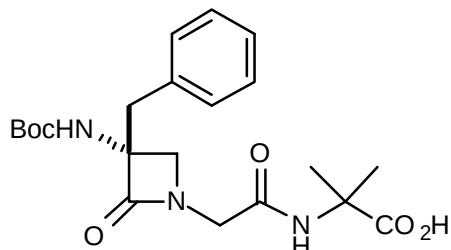
MS m/z	
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¹ H-NMR (δ, ppm) CDCl ₃	¹³ C-NMR (δ, ppm) CDCl ₃	
7.79 (s, 1H, NH)	173.9	44.9
7.39-7.20 (m, 10H, Arom)	168.5	38.3
5.17 (d, 1H, J=12.4Hz, <u>H</u> CHPh)	166.8	28.1
5.09 (d, 1H, J=12.4Hz, HCH <u>Ph</u>)	154.3	25.9
4.82 (s, 1H, NHBoc)	136.0	24.4
4.37 (d, 1H, J=17.4Hz, NH <u>CH</u>)	133.6	
3.75 (d, 1H, J=5.3Hz, <u>H</u> CH)	129.6	
3.25(d, 1H, J=4.4Hz, HCH <u>)</u>	128.9	
3.20 (d, 1H, J=17.2Hz, NHCH <u>)</u>	128.3	
3.16 (d, 1H, J=14.1Hz, <u>H</u> CH)	127.9	
3.00 (d, 1H, J=14.13Hz, HCH <u>)</u>	127.7	
1.59 (s, 3H, CH ₃)	80.9	
1.52 (s, 3H, CH ₃)	68.3	
1.40 (s, 9H, (CH ₃) ₃)	66.8	
	56.4	
	51.7	

Comments:

Product purified by preparative HPLC (fused silicagel column, Lichrosorb Si 60, 7μm, 250x25mm, eluant AcOEt, v=10ml/min) r.t.:11.3min

(R)-3-Benzyl-3-*tert*-butoxycarbonylamino-1-[3-aza-5-carboxy-4-methyl-2-oxo-pentyl]-azetidin-2-one



Reference n°
10

Mol. W. (Dalton)
419.5

Yield (%)
60 (from 7)

Melting.P. (°C)
183-185(Hx/AcOEt)

$[\alpha]_D^{25}$ (c=0.24, Cl₂CH₂)
+ 89.7

Molecular Formula	Elemental Analysis					
	Calculated(%)			Found(%)		
C ₂₁ H ₂₉ N ₃ O ₆	C 60.12	H 6.98	N 10.01	C 60.32	H 7.12	N 9.96

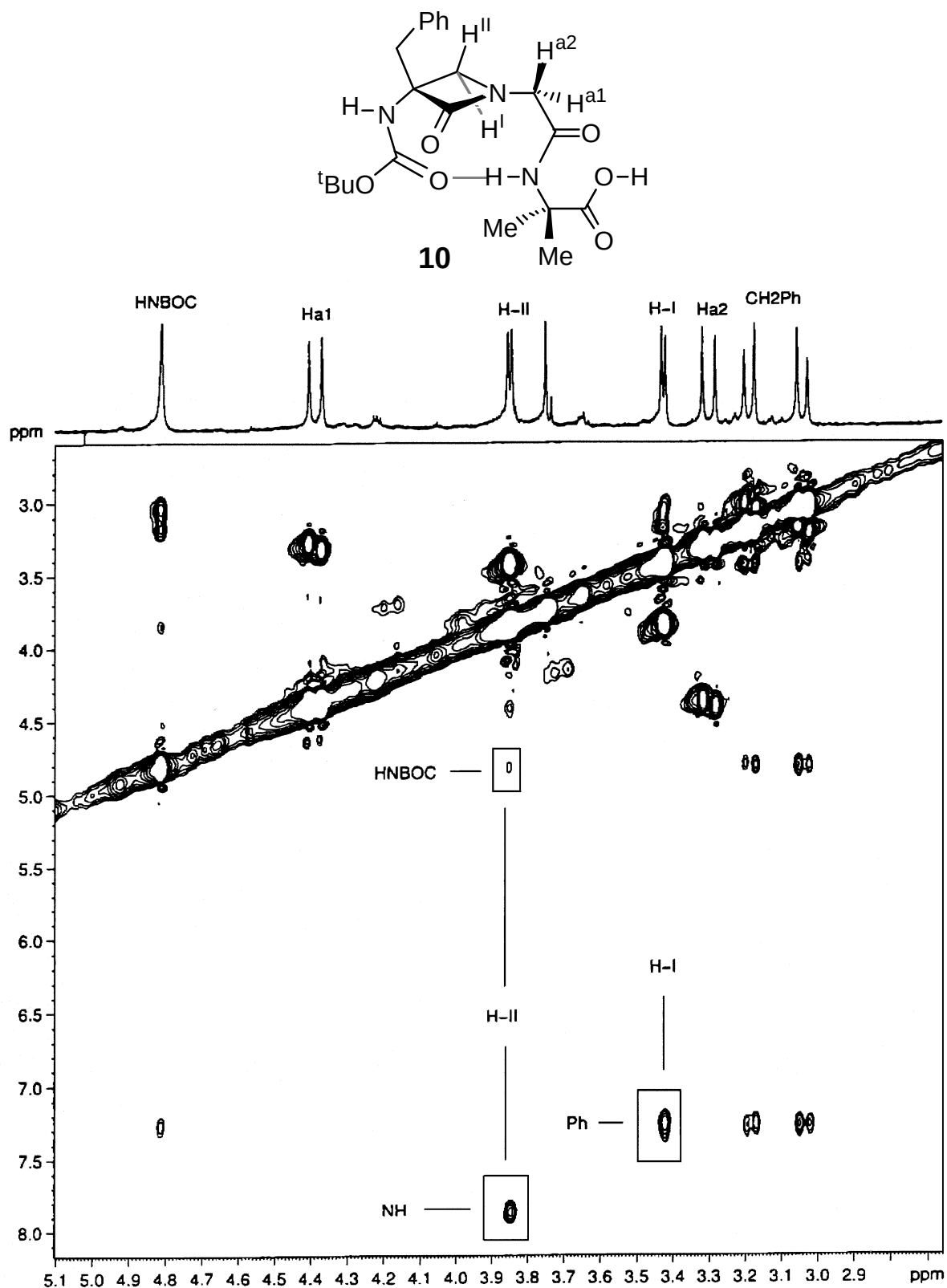
IR (cm ⁻¹ , KBr)	3076, 2992, 2950(NH, OH); 1766, 1700(CO)
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MS m/z	
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¹ H-NMR (δ, ppm) CDCl ₃	¹³ C-NMR (δ, ppm) CDCl ₃
7.90 (s, 1H, NH)	177.4
7.36-7.22 (m, 5H, arom)	24.4
5.35 (s _a , 1H, COOH)	168.9
4.89 (s, 1H, NH)	167.9
4.36 (d, 1H, J=17.6Hz, HNHCHCO)	154.4
3.86 (d, 1H, J=5.3Hz, NHCH)	133.6
3.42 (d, 1H, J=5.3Hz, NHCH)	129.7
3.29 (d, 1H, J=17.9Hz, HNHCHCO)	129.0
3.18 (d, 1H, J=14.4Hz, HCHPh)	127.8
3.02 (d, 1H, J=14.3Hz, HCHPh)	81.1
1.54 (s, 3H, CH ₃)	68.4
1.52 (s, 3H, CH ₃)	56.7
1.40 (s, 9H, (CH ₃) ₃)	52.1
	44.9
	39.3
	28.2
	25.8

Comments:

NMR-DATA:



Expansion of a ROESY experiment of **10** in CDCl₃ [10⁻³ M], 400ms mixing time, 2.4kHz spinlock power collected at 500Mhz with 2K datablocks for 256 *tl*- increments and 40 transients for each *tl*- increment. Marked crosspeaks indicate a type-II *b*-turn conformation.