

Synthesis and Functionalization of *trans*-2-(2'-Aminocyclohexyloxy)- and *trans*-2-(2'-Azidocyclohexyloxy)acetic Acid

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Summary. Synthesis of the hydrochloride of *trans*-2-(2'-aminocyclohexyloxy)acetic acid (**4**) from *trans*-2-(2'-azidocyclohexyloxy)acetic acid (**1**) is described. **4** was acylated at the amino group to give compounds **5–8**. **1** was converted into acid chloride (**9**) and amides **10–13**.

Keywords. *trans*-2-(2'-Aminocyclohexyloxy)acetic acid; *trans*-2-(2'-Azidocyclohexyloxy)acetic acid; *trans*-2'-Functionalized 2-cyclohexyloxyacetic acid.

Synthese und Funktionalisierung der *trans*-2-(2'-Aminocyclohexyloxy)- und *trans*-2-(2'-Azidocyclohexyloxy)essigsäure

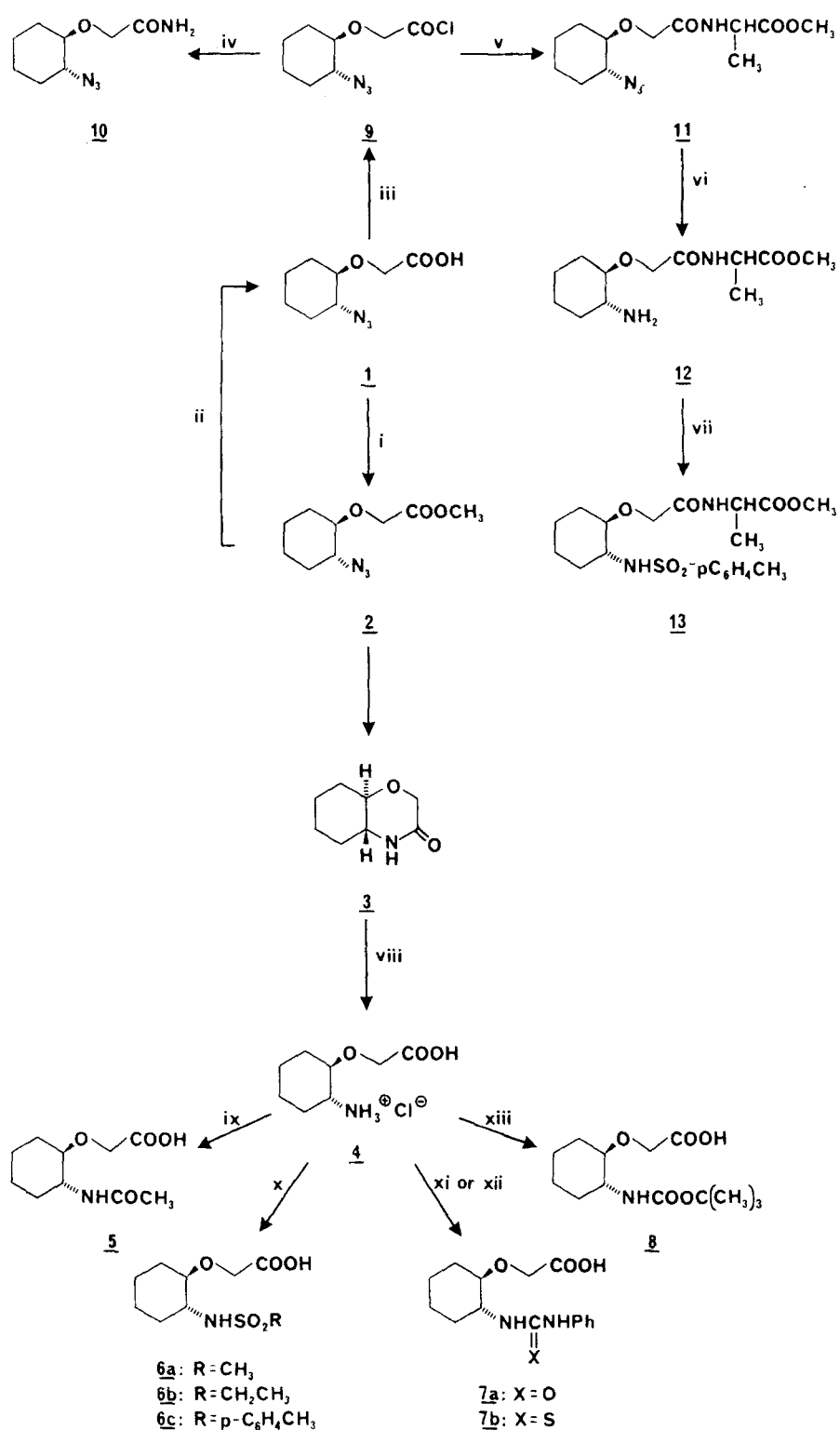
Zusammenfassung. Die Synthese des *trans*-2-(2'-Aminocyclohexyloxy)essigsäurehydrochlorids (**4**), ausgehend von *trans*-2-(2'-Azidocyclohexyloxy)essigsäure (**1**), wird beschrieben. **4** wurde durch Acylierung der Aminogruppe in die Verbindungen **5–8** übergeführt. **1** wurde in das Säurechlorid **9** und die Carbonsäureamide **10–13** umgewandelt.

Introduction

In the course of our investigations on the synthesis of potential immunomodulators related to muramyl dipeptide (*MDP*) [1, 2], *trans*-2-(2'-aminocyclohexyloxy)acetic acid (**4**) and its synthetic precursor *trans*-2-(2'-azidocyclohexyloxy)acetic acid (**1**) were needed as key intermediates for the synthesis of some model compounds. Since both carboxylic acids have been unknown so far, we report here our approaches to the synthesis of **1** and **4** and the preparation of some derivatives of the title compounds.

Results and Discussion

We have shown previously [3] that *trans*-2-(2'-azidocyclohexyloxy)acetic acid (**1**) can be prepared by the alkylation of *trans*-2-azidocyclohexanol [6] with chloroacetic acid and converted *via* its methyl ester **2** into *trans*-octahydro-2*H*-1,4-benzoxazin-3-one (**3**). However, **1** has not been characterized before, since it could not be



Scheme 1. i: MeOH, H₂SO₄; ii: NaOH/dioxane; iii: SOCl₂; iv: NH₃/dioxane; v: L-Ala-OMe·HCl/dioxane; vi: SnCl₂·2H₂O/MeOH; vii: TsCl, Et₃N/dichloromethane; viii: HCl/H₂O; ix: Ac₂O/NaOH; x: RSO₂Cl/NaOH/ether; xi: PhNCO/NaOH; xii: PhNCS/NaOEt; xiii: BOC₂O, Et₃N/EtOAc

obtained pure with the reported procedure. *Trans*-2-(2'-azidocyclohexyloxy)acetic acid (**1**) was now prepared in pure crystalline form by hydrolysis of its methyl ester (**2**) with 1 *N* sodium hydroxide in dioxane. **2** was obtained in good yield by esterification of the crude carboxylic acid **1** with methanol in the presence of concentrated sulfuric acid. The yield of the ester **2** obtained with this method was comparable to the yield of the previously described method [3] which used the BF_3 -methanol complex for esterification.

Reaction of crude **1** with thionyl chloride afforded acid chloride **9** which is a useful intermediate for coupling with amino acids and peptide fragments. Being unstable, **9** was converted for analytical purposes into the corresponding amide **10** with aqueous ammonia in dioxane. Additionally, the coupling of the acid chloride **10** with *L*-alanine methyl ester in dioxane was performed to give compound **11**, which was unstable and was therefore characterized as *N*-[*trans*-2-(2'-tosylaminocyclohexyloxy)acetyl]-*L*-alanine methyl ester (**13**) upon reduction of the azido group in **11** with tin(II) chloride [4] and tosylation of the resulting amine **12**. Compound **13** was obtained as a mixture of diastereomers which were not separated.

The attempts to prepare *trans*-2-(2'-aminocyclohexyloxy)acetic acid (**4**) by reduction of **1** with tin(II) chloride were unsuccessful due to the difficulties encountered during the isolation of the product. Therefore we looked for a better method which would allow an efficient preparation of the desired compound **4**. We have found that hydrolytic ring opening of *trans*-octahydro-2*H*-1,4-benzoxazin-3-one (**3**) takes place when it is heated with 18% hydrochloric acid, affording the hydrochloride of *trans*-2-(2'-aminocyclohexyloxy)acetic acid (**4**) in excellent yield. With the acid **4** in hand, some functional derivatives of the amino group which are interesting for the synthesis of potential *MDP* related immunomodulators, were prepared using known procedures [5]. Thus, acylation of **4** with acetic anhydride and sulfonyl chlorides in alkaline solution afforded *N*-acetyl-(**5**) and *N*-sulfonyl derivatives (**6 a, b, c**), respectively. With di-*t*-butyl dicarbonate (BOC_2O) in ethyl acetate the corresponding *N*-*t*-butyloxycarbonyl derivative **8** was obtained in good yield. Reaction of **4** with phenyl isocyanate gave the corresponding *N*-phenylurea (**7 a**). Similarly, with phenyl isothiocyanate *N*-phenylthiourea **7 b** was obtained.

Experimental

Melting points were determined on a Kofler hot stage microscope and are uncorrected. The IR spectra were obtained on a Perkin Elmer 257 spectrometer. Proton NMR spectra were measured at 60 MHz on a Varian EM 360 and at 300 MHz on a Varian VXR 300 spectrometer, respectively, with *TMS* as internal standard. EI mass spectra were measured at 100 eV on a CEC 21-110 B mass spectrometer. Elemental analyses for C, H, and N were performed on a Perkin-Elmer CHN Analyzer 240 C.

trans-2-(2'-Azidocyclohexyloxy)acetic Acid (**1**)

To a solution of *trans*-methyl-2-(2'-azidocyclohexyloxy)acetate **2** (1.065 g, 5 mmol) in dioxane (20 ml) was added 1*N* NaOH (7 ml). The mixture was stirred for 18 h at room temperature, evaporated *in vacuo* and the residue dissolved in water (20 ml). After extraction with ethyl acetate (2 × 20 ml), the aqueous layer was acidified to *pH* 2 with 18% HCl and extracted with ethyl acetate (5 × 20 ml). The organic phase was dried with MgSO_4 , filtered and evaporated *in vacuo* to give **1** as a white solid which was recrystallized from ethyl acetate/*n*-hexane; yield: 0.93 g (86%); m.p. 38–40 °C. IR (KBr): 2115 (N_3), 1740 (CO) cm^{-1} . NMR (60 MHz, CDCl_3): δ = 0.9–2.15 (m, 8 H, 4 CH_2), 2.85–3.55 (m,

2 H, 1'-H, 2'-H), 4.2 (s, 2 H, OCH₂), 9.15 (s br, 1 H, COOH) ppm. MS: calcd. 199.2; found: 155 (0.7%, M⁺-CO₂), 41 (100%). Anal. calcd. for C₈H₁₃N₃O₃: C 48.24, H 6.58, N 21.09; found: C 48.53, H 6.77, N 20.87.

trans-Methyl-2-(2'-Azidocyclohexyloxy) Acetate (**2**)

A solution of crude **1** (5.97 g, 30 mmol) and conc. H₂SO₄ (0.4 ml) in methanol (15 ml) was refluxed for 3 h, poured into ice water (100 ml) and extracted with *n*-hexane (3 × 40 ml). The combined extracts were dried over MgSO₄, filtered and evaporated *in vacuo*. The residual oil was distilled *in vacuo* to give pure **2** which was in all respects (IR, NMR) identical with the ester **2** prepared by us previously [3]; yield: 3.96 g (62%); b.p. 102 °C/0.3 Torr.

trans-2-Carboxymethoxy-cyclohexylammonium Chloride (**4**)

A solution of *trans*-octahydro-2*H*-1,4-benzoxazin-3-one (**3**) (7.75 g, 50 mmol) in 18% aqueous HCl (240 ml) was refluxed for 4 h and evaporated *in vacuo* to a white solid which was recrystallized from ethanol/ether; yield: 9.85 g (94%); m.p. 194–196 °C. IR (KBr): 3 500–2 450 br, 1 745 (CO) cm⁻¹. NMR (60 MHz, D₂O): δ = 1.2–2.55 (m, 8 H, 4 CH₂), 2.9–3.9 (m, 2 H, 1-H, 2-H), 4.4 (s, 2 H, OCH₂) ppm. Anal. calcd. for C₈H₁₆ClNO₃: C 45.83, H 7.69, N 6.68; found: C 45.62, H 7.85, N 6.73.

trans-2-(2'-Acetylaminocyclohexyloxy)acetic Acid (**5**)

To a stirred precooled solution of **4** (1.048 g, 5 mmol) in 2 *N* NaOH (5 ml) was added first 1 *N* NaOH (1 ml) and then acetic anhydride (0.102 g, 1 mmol). When the anhydride disappeared and a homogeneous solution formed, the addition of the same amounts of 1 *N* NaOH and acetic anhydride was repeated four more times. After the addition of the last portion, stirring was continued for 30 min, the solution was acidified to *pH* 2 and extracted with ethyl acetate (5 × 20 ml). The organic extracts were dried over MgSO₄, filtered and evaporated under reduced pressure. The residue was recrystallized from chloroform/*n*-hexane to give white crystals of **5**; yield: 0.60 g (56%); m.p. 141–143 °C. IR (KBr): 3 305, (NH), 1 750 (COOH), 1 645, 1 560 (CONH) cm⁻¹. NMR (60 MHz, CDCl₃): δ = 0.85–2.65 (m, 8 H, 4 CH₂), 2.07 (s, 3 H, CH₃), 2.8–3.7 (m, 2 H, 1'-H, 2'-H), 4.1 (AB system, 2 H, *J* = 18 Hz, OCH₂), 8.25 (s br, 1 H, NH), 10.6 (s br, 1 H, COOH) ppm. MS: calcd. 215.25; found 215 (M⁺). Anal. calcd. for C₁₀H₁₇NO₄: C 55.80, H 7.96, N 6.51; found: C 55.76, H 8.25, N 6.75.

trans-2-(2'-Tosylaminocyclohexyloxy)acetic Acid (**6c**)

To a solution of **4** (1.048 g, 5 mmol) in 1 *N* NaOH (15 ml) was added a solution of *p*-toluenesulfonyl chloride (1.048 g, 5.5 mmol) in ether (20 ml). The mixture was vigorously stirred for 20 h at room temperature, the aqueous phase was separated, washed with ether (20 ml), acidified with 18% HCl to *pH* 2 and extracted with ether (5 × 20 ml). The organic extracts were dried over MgSO₄, filtered and evaporated *in vacuo*. The residue was recrystallized from diisopropyl ether; yield: 1.09 g (67%), white crystals; m.p. 56–58 °C. IR (KBr): 3 240 (NH), 1 720 (COOH), 1 345, 1 330, 1 170, 1 160 (SO₂NH) cm⁻¹. NMR (300 MHz, DMSO-*d*₆): δ = 0.95–1.20 (m, 4 H, 3', 4', 5', 6'-H_{ax}), 1.4–1.55 (m, 2 H, 4', 5'-H_{eq}), 1.80–1.95 (m, 2 H, 3', 6'-H_{eq}), 2.35 (s, 3 H, CH₃), 2.75–2.85 (m, 1 H, 2'-H), 2.98–3.08 (m, 1 H, 1'-H), 3.4 (s br, NH + COOH + H₂O), 3.92 (AB system, 2 H, *J* = 17.1 Hz, OCH₂), 7.34 (d, 2 H, 2''-H, 6''-H), 7.67 (d, 2 H, 3''-H, 5''-H) ppm. MS: calcd. 327.40; found 327 (M⁺). Anal. calcd. for C₁₅H₂₁NO₅S: C 55.03, H 6.47, N 4.28; found: C 55.15, H 6.61, N 4.03.

trans-2-(2'-Methylsulfonylaminocyclohexyloxy)acetic Acid (**6a**)

Prepared from **4** (1.048 g, 5 mmol) and methanesulfonyl chloride (0.859 g, 7.5 mmol) in analogy to **6c**; yield: 0.38 g (30%), m.p. 119–121 °C (from ethylacetate/*n*-hexane). IR (KBr): 3 250 (NH), 1 735

(COOH), 1 355, 1 340, 1 170, 1 155 (SO_2NH) cm^{-1} . NMR (60 MHz, CDCl_3): δ = 0.83–2.43 (m, 8 H, 4 CH_2), 2.67–3.43 (m, 2 H, 1'-H, 2'-H), 3.0 (s, 3 H, CH_3), 4.13 (AB system, 2 H, J = 18 Hz, OCH_2), 6.5 (s br, 2 H, NH, COOH) ppm. MS: calcd. 251.30; found 251 (M^+). Anal. calcd. for $\text{C}_9\text{H}_{17}\text{NO}_5\text{S}$: C 43.01, H 6.82, N 5.57; found: C 43.28, H 6.95, N 5.41.

trans-2-(2'-Ethylsulfonylaminocyclohexyloxy)acetic Acid (**6b**)

Prepared from *trans*-2-carboxymethoxy-cyclohexylammonium chloride **4** (1.048 g, 5 mmol) and ethanesulfonyl chloride (0.964 g, 7.5 mmol) in analogy to **6c**; yield: 0.56 g (42%); m.p. 120–122 °C (from ethyl acetate/*n*-hexane). IR (KBr): 3 310 (NH), 1 740 (COOH), 1 330, 1 305, 1 140 (SO_2NH) cm^{-1} . NMR (60 MHz, CDCl_3): δ = 0.87–2.4 (m, 8 H, 4 CH_2), 1.30 (t, 3 H, J = 7 Hz, CH_3), 2.67–3.23 (m, 4 H, SO_2CH_2 , 1'-H, 2'-H), 4.03 (AB system, 2 H, J = 18 Hz, OCH_2), 5.86 (s br, 1 H, NH), 7.95 (s br, 1 H, COOH) ppm. MS: calcd. 265.33; found 265 (M^+). Anal. calcd. for $\text{C}_{10}\text{H}_{19}\text{NO}_5\text{S}$: C 45.27, H 7.22, N 5.28; found: C 45.65, H 7.47, N 5.16.

trans-2-[2'-(*N'*-Phenylureido)cyclohexyloxy]acetic Acid (**7a**)

Phenyl isocyanate (0.476 g, 4 mmol) was added to a stirred solution of **4** (0.419 g, 2 mmol) in 2*N* NaOH (10 ml). After 1 h the solution was extracted with ether (10 ml), the aqueous phase was acidified with 18% HCl to *pH* 3 and **7a**, which separated in form of white crystals, was filtered off and dried over P_2O_5 ; yield: 0.23 g (40%); m.p. 166–168 °C. IR (KBr): 3 350, 3 300 (NH), 1 740 (COOH), 1 640, 1 565 (CONH) cm^{-1} . NMR (300 MHz, $\text{DMSO}-d_6$): δ = 1.1–2.0 (m, 8 H, 4 CH_2), 3.15–3.25 (m, 1 H, 2'-H), 3.4–3.55 (m, 1 H, 1'-H), 4.05 (AB system, 2 H, OCH_2), 6.3 (d br, 1 H, NH), 6.85 (m, 1 H, H-arom.), 6.95 (m, 2 H, 2 H-arom.), 7.15 (m, 2 H, 2 H-arom.), 8.7 (s br, 1 H, NH), 10.4 (s br, 1 H, COOH) ppm. MS: calcd. 292.34; found 292 (M^+). Anal. calcd. for $\text{C}_{15}\text{H}_{20}\text{N}_2\text{O}_4$: C 61.63, H 6.90, N 9.58; found: C 61.36, H 6.96, N 9.43.

trans-2-[2'-(*N'*-Phenylthioureido)cyclohexyloxy]acetic Acid (**7b**)

To a stirred solution of sodium ethoxide, prepared from Na (0.046 g, 2 mmol) and ethanol (10 ml) were added first **4** (0.419 g, 2 mmol) and then phenyl isothiocyanate (0.270 g, 2 mmol). The mixture was refluxed for 1 h, evaporated and the residue dissolved in 1*N* NaOH (25 ml). The solution was extracted with ether (2 $\times$ 20 ml), the aqueous phase was acidified with 18% HCl to *pH* 2 and extracted again with ether (3 $\times$ 20 ml). The organic phase was dried over MgSO_4 , filtered and evaporated *in vacuo*. The residue was recrystallized from ethanol/ether to give white crystals of **7b**; yield: 0.185 g (30%); m.p. 147–149 °C. IR (KBr): 3 300, 3 250 (NH), 1 750 (COOH), 1 545, 1 515 cm^{-1} . NMR (300 MHz, $\text{DMSO}-d_6$): δ = 1.05–1.40 (m, 4 H, 3', 4', 5', 6'- H_{ax}), 1.50–1.75 (m, 2 H, 4', 5'- H_{eq}), 2.00–2.12 (m, 2 H, 3', 6'- H_{eq}), 3.35–3.50 (m, 1 H, 2'-H), 4.0–4.1 (m, 3 H, OCH_2 , 1'-H), 7.10 (m, 1 H, H-arom.), 7.29 (m, 2 H, 2 H-arom.), 7.39 (m, 2 H, H-arom.), 7.81 (d br, 1 H, NH), 9.49 (s br, 1 H, NH), 12.65 (s br, 1 H, COOH) ppm. MS: calcd. 308.4; found 309 ($M^+ + 1$); Anal. calcd. for $\text{C}_{15}\text{H}_{20}\text{N}_2\text{O}_3\text{S}$: C 58.42, H 6.54, N 9.08; found: C 58.63, H 6.81, N 9.42.

trans-2-(2'-*t*-Butyloxycarbonylaminocyclohexyloxy)acetic Acid (**8**)

To a solution of **4** (0.42 g, 2 mmol) in dry dioxane were added triethylamine (0.61 g, 6 mmol) and BOC_2O (0.52 g, 2.4 mmol). The mixture was stirred at 50 °C for 6 h and then evaporated *in vacuo*. The residue was dissolved in water (5 ml), the solution acidified to *pH* 2 with 10% citric acid and extracted with ethyl acetate (3 $\times$ 10 ml). The organic solution was dried over MgSO_4 , filtered and evaporated under reduced pressure to a white solid. Recrystallization from ethyl acetate afforded **8**; yield: 0.36 g (66%); m.p. 205–207 °C. IR (KBr): 3 380 (NH), 1 750 (COOH), 1 685, 1 540 (NHCOO) cm^{-1} . NMR (60 MHz, CDCl_3): δ = 0.87–2.3 (m, 8 H, 4 CH_2), 1.47 (s, 9 H, 3 CH_3), 2.8–3.63 (m, 2 H, 1'-H, 2'-H), 4.06 (AB system, 2 H, J = 18 Hz, OCH_2), 5.20 (s br, 1 H, NH), 7.03 (s br, 1 H, COOH)

ppm. MS: calcd. 273.33; found 273 (M^+). Anal. calcd. for $C_{13}H_{23}NO_5$: C 57.13, H 8.48, N 5.12; found: C 57.37, H 8.71, N 4.93.

trans-2-(2'-Azidocyclohexyloxy)acetyl Chloride (**9**)

A mixture of crude **1** (11.94 g, 60 mmol) and thionyl chloride (10.71 g, 90 mmol) were heated for 30 min at 70 °C. The excess thionyl chloride was removed under reduced pressure and the residual oil distilled *in vacuo* to give pure **9** as a pale yellow oil; yield: 8.62 g (66%); b.p. 104–105 °C / 0.2 Torr. IR (film): 2110 (N_3), 1810 (CO) cm^{-1} . NMR (60 MHz, $CDCl_3$): δ = 1.15–2.25 (m, 8 H, 4 CH_2), 3.03–3.52 (m, 2 H, 1'-H, 2'-H), 4.60 (s, 2 H, OCH_2), ppm. MS: calcd. 217.65; found 112 (86%), 69 (75%), 41 (100%).

Trans-2-(2'-Azidocyclohexyloxy)acetamide (**10**)

25% Aqueous ammonia (4 ml) was added to a stirred solution of **9** (1.088 g, 5 mmol) in dioxane (20 ml). After 15 min water (150 ml) was added, the resulting solution was saturated with NaCl and extracted with ethyl acetate (4 $\times$ 50 ml). The organic phase was dried over $MgSO_4$, filtered and evaporated *in vacuo*. Recrystallization of the residue from ethanol afforded white crystals of **10**; yield: 0.870 g (88%); m.p. 79–80 °C. IR (KBr): 3450, 3180 (NH_2), 2110 (N_3), 1655, 1600 ($CONH_2$) cm^{-1} . NMR (60 MHz, $CDCl_3$): δ = 0.93–2.2 (m, 8 H, 4 CH_2), 2.87–3.45 (m, 2 H, 2'-H, 1'-H), 4.0 (AB system, 2 H, J = 16 Hz, OCH_2), 5.9 (s br, 1 H, NH), 6.75 (s br, 1 H, NH) ppm. MS: calcd. 198.22; found 199 (1.7%, $M^+ + 1$), 155 (100%). Anal. calcd. for $C_8H_{14}N_4O_2$: C 48.47, H 7.12, N 28.27; found: C 48.49, H 7.29, N 28.29.

N-[*trans*-2-(2'-Azidocyclohexyloxy)acetyl]-*L*-alanine methyl Ester (**11**)

To a stirred suspension of *L*-alanine methyl ester hydrochloride (4.19 g, 30 mmol) in dry dioxane (30 ml), which was cooled in a water-ice bath, were added first triethylamine (6.06 g, 60 mmol) and then **9** (6.53 g, 30 mmol). After 40 h stirring at room temperature, dioxane was removed under reduced pressure, the residue was suspended in water (150 ml), acidified to pH 3 with 1N HCl and extracted with ethyl acetate (3 $\times$ 100 ml). The organic phase was dried ($MgSO_4$), filtered and evaporated to give crude **11** as a yellow oil which was purified by chromatography on silica gel (chloroform/methanol/*n*-hexane = 9/1/5); yield: 5.11 g (60%), yellow oil. IR (film): 3440, 3360 (NH), 2120 (N_3), 1760 ($COOCH_3$), 1680, 1535 (CONH) cm^{-1} . NMR (60 MHz, $DMSO-d_6$): δ = 0.95–2.25 (m, 8 H, 4 CH_2), 1.31 (d, 3 H, J = 7.3 Hz, CH_3), 2.95–3.60 (m, 2 H, 1'-H, 2'-H), 3.64 (s, 3 H, OCH_3), 4.01 (s, 2 H, OCH_2), 4.54 (q, 1 H, J = 7.3 Hz, $CHCH_3$), 7.88 (d br, 1 H, J = 7.2 Hz, NH) ppm. MS: calcd. 284.31; found 284 (3%, M^+), 241 (56%), 145 (100%).

N-[*trans*-2-(2'-Tosylaminocyclohexyloxy)acetyl]-*L*-alanine Methyl Ester (**13**)

Compound **11** (3.40 g, 11.96 mmol) dissolved in methanol (6 ml) was added dropwise over 15 min to a stirred solution of $SnCl_2 \times 2H_2O$ (4.05 g, 17.95 mmol) in methanol (6 ml). Stirring was continued for 5 h, methanol was removed *in vacuo*, the residue suspended in 10% Na_2CO_3 and extracted with ethyl acetate (5 $\times$ 50 ml). The organic extract was dried over $MgSO_4$, filtered and evaporated under reduced pressure to give crude **12** as a yellow oil; yield: 1.88 g, 61%.

To the solution of crude **12** (1.88 g, 7.29 mmol) in dichloromethane (50 ml) was added first tosyl chloride (1.39 g, 7.29 mmol) and then triethylamine (0.736 g, 7.29 mmol). The solution was stirred for 18 h at room temperature, evaporated *in vacuo* and the residual oil purified by column chromatography on silica gel (chloroform/methanol/*n*-hexane = 9/1/5); yield: 2.40 g (80%) of **13** (diastereomeric mixture), yellow oil. IR (film): 3380, 3300 (NH), 1740 ($COOCH_3$), 1685, 1520 (CONH), 1345, 1335, 1170 (SO_2NH) cm^{-1} . NMR (60 MHz, $DMSO-d_6$): δ = 0.90–2.20 (m, 8 H, 4 CH_2), 1.34 (d, 3 H, J = 7.3 Hz, CH_3CH), 2.37 (s, 3 H, CH_3), 2.70–3.25 (m, 2 H, 1'-H, 2'-H), 3.65 (3.66) (s, 3 H, OCH_3),

3.85 (3.90) (AB system, 2 H, $J=15$ Hz, OCH_2), 4.34 (q, 1 H, $J=7.3$ Hz, CHCH_3), 7.52 (d, 2 H, 2 H-arom.), 7.86 (d, 2 H, 2 H-arom.), 8.0 (m br, 1 H, NH) ppm. MS: calcd. 412.51; found 412 (M^+). Anal. calcd. for $\text{C}_{19}\text{H}_{28}\text{N}_2\text{O}_6\text{S}$: C 55.32, H 6.84, N 6.79; found: C 55.45, H 7.10, N 6.81.

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