# SYNTHESIS OF SYMMETRIC MACROCYCLIC DIESTERDIHYRAZIDES USING SUCCESSIVE [2+1]- AND [1+1]-CONDENSATIONS

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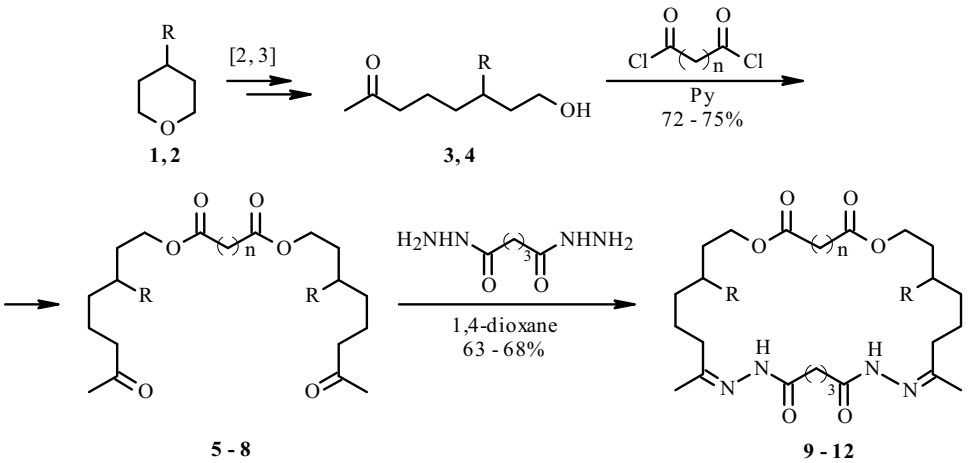
*Potentially useful symmetric macrocyclic diesterdihydrazides were synthesized efficiently from available petrochemical products (tetrahydropyran and 4-methyltetrahydropyran) using successive [2+1]-condensation of 8-hydroxyoctan-2-one and its 6-methyl derivative with glutaric and adipic chlorides and [1+1]-condensation of the intermediate diketodiesters with glutaric dihydrazide.*

**Keywords:** tetrahydropyran, 4-methyltetrahydropyran, 8-hydroxyoctan-2-one, 8-hydroxy-6-methyloctan-2-one, macrocyclic diesterdihydrazides, synthesis.

Most macrocycles have unusual properties and broad applications [1]. Therefore, methods for preparing such compounds with various functional groups must be devised in order to develop their chemistry, modern pharmacology, and advanced technology.

Herein we report the synthesis of potentially biologically and pharmacologically active 30- (**9**, **11**) and 31-membered (**10**, **12**) symmetric macrocycles containing two esters and two hydrazides. Tetrahydropyran (**1**) and 4-methyltetrahydropyran (**2**) were converted in three steps [2, 3] to hydroxyketones **3** and **4**, [2+1]-condensation of which with glutaric and adipic chlorides gave *bis*-derivatives **5–8** in which the two ketone C atoms of **3** or **4** were linked by diester spacers. Ring closure of key diketodiesters **5-8** into the desired macrocycles **9–12** was carried out via [1+1]-condensation with glutaric dihydrazide in dioxane at room temperature under high-dilution conditions.




**1, 2** **3, 4** **5 - 8** **9 - 12**

R = H (**1**, **3**, **5**, **6**, **9**, **10**); R = $CH_3$ (**2**, **4**, **7**, **8**, **11**, **12**)
n = 3 (**5**, **7**, **9**, **11**); n = 4 (**6**, **8**, **10**, **12**)

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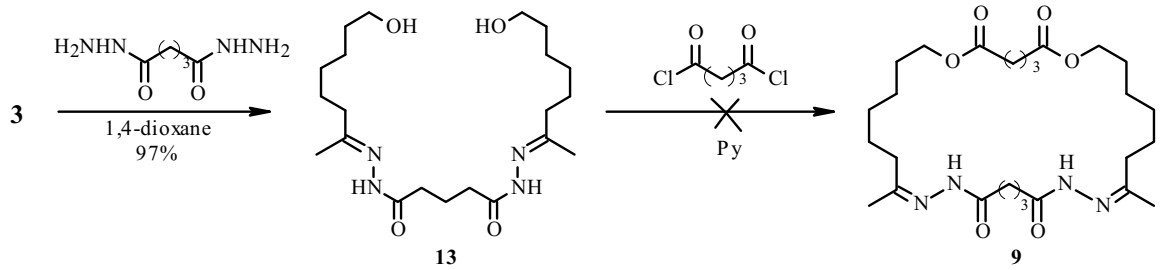
An alternative attempt to synthesize **9** via [2+1]-condensation of **3** with glutaric dihydrazide into acyclic symmetric dihydrazidodiol **13** and subsequent [1+1]-condensation with glutaric chloride under high-dilution conditions was unsuccessful in the second step.




The structures of the resulting macrocycles **9**–**12** were confirmed by IR, PMR, and $^{13}C$ NMR spectroscopy and GC–MS.

IR spectra of **9**–**12** lacked absorption bands (1705 $cm^{-1}$, **5**; 1703, **6**; 1716, **7** and **8**) characteristic of the ketones in the key diketodiesters (**5–8**). Bands in the IR spectra of **9**–**12** at 1639 $cm^{-1}$ (C=N), 1670–1700 (CONH), and 3326–3420 (NH) proved that macrocycles with hydrazides had formed.

NMR spectra of **9–12** were analyzed by comparison with those of the starting compounds, diketodiesters **5–8** and glutaric hydrazide. $^{13}C$ NMR spectra of **9**–**12** lacked resonances for the carbonyls of **5**–**8** (208.57 ppm, **5**; 208.66, **6**; 208.82, **7**, 208.07, **8**). Furthermore, proton spectra of **9–12** lacked a resonance at 4.90 for the hydrazine ($NH_2NH$) group. These facts indicated that the products were not linear substitution products.

$^{13}C$ NMR spectra of **9**–**12** had resonances for the ester C atom (172.81 ppm, **9**; 173.18, **10**; 172.92, **11**; and 173.18, **12**) and resonances that were shifted compared with starting glutaric dihydrazide (171.89) for the C atom of the NH–C=O group (175.35, **9**; 175.58, **10**; 175.33, **11**; and 175.58, **12**) in addition to singlets for the C=N group (151.66, **9**; 152.26, **10**; 151.29, **11**; 152.04, **12**) and two quartets for $CH_3$ groups (14.95, **9**; 15.33, **10**; 14.94, **11**; and 15.19, **12**), the chemical shifts of which corresponded to the C atoms of the two magnetically equivalent $CH_3$–C=N groups. Triplets (43.38, **9**; 43.42, **10**; 39.29, **11**; 38.76, **12**) for the C atoms of two $CH_2C$=N groups also confirmed that the hydrazide group ($CH_2C$=N–NH–C=O) had formed. PMR spectra of **9**–**12** had weak-field resonances (8.38, **9**; 8.40, **10**; 8.15, **11**; 8.60, **12**), the chemical shifts and integrated intensities of which corresponded to the two NHC=O groups of the macrocycles.

Resonances of the C atom of the NHC=O groups in $^{13}C$ NMR spectra of **9–12** were noticeably shifted relative to those of the corresponding C atoms in starting glutaric dihydrazide. This was probably due to tautomeric (hydrazide–diazene) transitions in the macrocycles. All these spectral data indicated that macrocycles **9**–**12** formed. This was confirmed also by mass spectral data.

Chemical ionization at atmospheric pressure (APCI) with detection of positive and negative ions (20 eV) was used to study **5–12**. Protonation and solvation by water are well known phenomena in the chemistry of amides and hydrazides [4], which made the APCI method necessary. The mass spectrometric study of **5–12** detected exceedingly strong peaks for protonated $MH^+$ and deprotonated $[M - H]^-$ ions in addition to their ionic associates with 1–3 water molecules. This was considered additional proof of the existence of compounds with the corresponding molecular weights.

## EXPERIMENTAL

IR spectra were recorded in a thin layer on an IR Prestige-21 instrument (Shimadzu). NMR spectra were recorded in $CDCl_3$ on a Bruker AM-300 spectrometer (operating frequency 300.13 MHz for $^1H$; 75.47, $^{13}C$) with TMS or MeOH+$C_6D_6$ internal standard. Chromatography was performed in a Chrom-5 instrument [1.2 m column length, SE-30 silicone (5%) stationary phase on Chromaton N-AW-DMCS (0.16–0.20 mm), 50–300°C] with He carrier gas. HPLC analysis was carried out in an LC-20AD liquid chromatograph (Shimadzu) with an SPD-M20A diode-matrix detector (Shimadzu, Japan) using a Phenomenex 250 × 4.6 mm column packed with Luna C18 sorbent (5 μm). The mobile phase was $H_2O$:$CH_3CN$ at flow rate 1 mL/min. The analytical wavelength was 215 nm. We used $SiO_2$ (70–230) for column chromatography (Lancaster, England). TLC monitoring used Sorbfil $SiO_2$ (Russia). Elemental analyses of all compounds agreed with those calculated. Mass spectra

of **5–12** were taken on an LCMS 2010 EV instrument (Shimadzu) under APCI conditions at electron energy 20 eV with detection of positive and negative ions. The liuqid mobile phase was water at flow rate 0.03 mL/min. We used petroleum ether (40–70°C), $CH_2Cl_2$, $Et_2O$, and pyridine for the reactions.

**General Method for Preparing 5–8.** A solution in Py (1 mL, anhydrous) of **3** or **4** (2.0 mmol) prepared from the corresponding tetrahydropyran **1** or **2** [2, 3] was stirred, treated with a solution in $Et_2O$ (1 mL) of the corresponding dicarboxylic acid chloride (1.0 mmol) prepared according to the literature [5], diluted after 48 h (TLC monitoring) with $Et_2O$ (10 mL), washed successively with HCl (5%, 3 × 1.5 mL) and saturated NaCl solution (3 × 1.5 mL), dried over $MgSO_4$, and evaporated. The solid was chromatographed ($SiO_2$, petroleum ether:$Et_2O$, 5:2) to afford the corresponding diketodiester.

***bis*(7-Oxooctyl)pentanedioate (5).** Yield 0.13 g (72%), $R_f$ 0.80. IR spectrum (ν, $cm^{-1}$): 1735 (O=C–O), 1705 (C=O), 1030 (C–O–C).

PMR spectrum ($CDCl_3$, δ, ppm, J/Hz): 1.25–1.30 (4H, m, H-4′), 1.45–1.60 (12H, m, H-2′, H-3′, H-5′), 1.87 (2H, t, J = 7.3, H-3), 2.06 (6H, s, H-8′), 2.29 (4H, t, J = 7.3, H-6′), 2.37 (4H, t, J = 7.4, H-2, H-4), 3.98 (4H, t, J = 6.7, H-1′).

$^{13}C$ NMR spectrum ($CDCl_3$): 19.93 (t, C-3), 23.31 (t, C-3′), 25.44 (t, C-5′), 28.16 (t, C-2′), 28.44 (t, C-4′), 29.54 (t, C-8′), 33.00 (t, C-2, C-4), 43.20 (t, C-6′), 64.05 (t, C-1′), 172.64 (s, C-1, C-5), 208.57 (s, C-7′).

Mass spectrum (APCI, 20 eV, *m*/*z*): 385 $[M + H]^+$, 402 $[M + H + H_2O]^+$, 383 $[M – H]^–$.

***bis*(7-Oxooctyl)hexanedioate (6).** Yield 0.14 g (74%), $R_f$ 0.83. IR spectrum (ν, $cm^{-1}$): 1735 (O=C–O), 1703 (C=O), 1026 (C–O–C).

PMR spectrum ($CDCl_3$, δ, ppm, J/Hz): 1.29–1.38 (8H, m, H-3′, H-3, H-4), 1.53-1.68 (12H, m, H-2′, H-4′, H-5′), 2.13 (6H, s, H-8′), 2.31 (4H, t, J = 7.1, H-2, H-5), 2.43 (4H, t, J = 7.3, H-6′), 4.07 (4H, t, J = 6.7, H-1′).

$^{13}C$ NMR spectrum ($CDCl_3$): 23.36 (t, 2C-3′), 24.17 (t, C-3, C-4), 25.49 (t, 2C-5′), 28.21 (t, 2C-2′), 28.50 (t, 2C-4′), 29.62 (t, 2C-8′), 33.66 (t, C-2, C-5), 43.29 (t, 2C-6′), 64.05 (t, 2C-1′), 173.10 (s, C-1, C-6), 208.66 (s, 2C-7′).

Mass spectrum (APCI, 20 eV, *m*/*z*): 399 $[M + H]^+$, 416 $[M + H + H_2O]^+$, 397 $[M – H]^–$.

***bis*(3-Methyl-7-oxooctyl)pentanedioate (7).** Yield 0.14 g (73%), $R_f$ 0.81. IR spectrum (ν, $cm^{-1}$): 1732 (O=C–O), 1716 (C=O), 1062 (C–O–C).

PMR spectrum ($CDCl_3$, δ, ppm, J/Hz): 0.91 (6H, d, J = 6.3, $CH_3$-3′), 1.07–1.20, 1.20–1.33 (4H, m, H-2′, H′-2′), 1.35–1.68 (10H, m, H-3′, H-4′, H-5′), 1.92 (2H, t, J = 7.1, H-3), 2.11 (6H, s, H-8′), 2.33 (4H, t, J = 7.3, H-2, H-4), 2.39 (4H, t, J = 7.3, H-6′), 4.08, 4.09 (4H, t, J = 6.8, H-1′, H′-1′).

$^{13}C$ NMR spectrum ($CDCl_3$): 19.15 (q, 2$CH_3$-3′), 20.00 (t, C-3), 20.91 (t, 2C-5′), 29.34 (q, 2C-8′), 29.56 (d, 2C-3′), 33.16 (t, 2C-2′), 35.14 (t, C-2, C-4), 36.07 (t, 2C-4′), 43.64 (t, 2C-6′), 62.64 (t, 2C-1′), 172.83 (s, C-1, C-5), 208.82 (s, 2C-7′).

Mass spectrum (APCI, 20 eV, *m*/*z*): 413 $[M + H]^+$, 430 $[M + H + H_2O]^+$, 411 $[M – H]^–$, 447 $[M + 2H_2O – H]^–$.

***bis*(3-Methyl-7-oxooctyl)hexanedioate (8).** Yield 0.15 g (75%), $R_f$ 0.83. IR spectrum (ν, $cm^{-1}$): 1734 (O=C–O), 1716 (C=O), 1062 (C–O–C).

PMR spectrum ($CDCl_3$, δ, ppm, J/Hz): 0.91 (6H, d, J = 6.4, $CH_3$-3′), 1.10-1.22, 1.25-1.38 (4H, m, H-2′, H′-2′), 1.39–1.49, 1.50–1.61 (6H, m, H-4′, H′-4′, H-3′), 1.62–1.68 (8H, m, H-5′, H-3, H-4), 2.20 (6H, s, H-8′), 2.31 (4H, t, J = 6.9, H-2, H-5), 2.41 (4H, t, J = 7.4, H-6′), 4.08, 4.09 (4H, t, J = 6.9, H-1′, H′-1′).

$^{13}C$ NMR spectrum ($CDCl_3$): 18.85 (q, 2$CH_3$-3′), 20.56 (t, C-3, C-4), 23.88 (t, 2C-5′), 29.22 (d, 2C-3′), 29.41 (q, 2C-8′), 33.36 (t, C-2, C-5), 34.85 (t, 2C-4′), 35.72 (t, 2C-2′), 43.20 (t, 2C-6′), 62.11 (t, 2C-1′), 172.66 (s, C-1, C-6), 208.07 (s, 2C-7′).

Mass spectrum (APCI, 20 eV, *m*/*z*): 427 $[M + H]^+$, 444 $[M + H + H_2O]^+$, 425 $[M – H]^–$.

**General Method for Preparing 9–12.** Diketodiester **5-8** (1.0 mmol) in anhydrous dioxane (30 mL) was stirred vigorously, treated slowly with glutaric dihydrazide (0.16 g, 1.0 mmol) prepared according to the literature [5], and stirred for 48 h (TLC monitoring). The dioxane was evaporated. The solid was dissolved in $CH_2Cl_2$ (20 mL), washed with $H_2O$ (3 × 5 mL), dried over $MgSO_4$, and evaporated. The resulting mass was stirred, treated successively with anhydrous $CH_2Cl_2$ (1 mL) and hexane (10 mL), and stored until the layers separated. The upper layer was decanted. The remainder was washed with hexane (5 mL) and evaporated.

**14,24-Dimethyl-1,7-dioxo-15,16,22,23-tetraazacyclotriaconta-14,23-dien-2,6,17,21-tetraone (9).** Yield 0.33 g (65%). IR spectrum (ν, $cm^{-1}$): 1735 (O=C–O), 1705 (C=O), 1030 (C–O–C), 1639 (C=N), 1670–1700 (CONH), 3326–3420 (NH).

PMR spectrum ($CDCl_3$, δ, ppm, J/Hz): 1.28–1.43 (8H, m, H-9, H-10, H-28, H-29), 1.46–1.68 (8H, m, H-11, H-12, H-26, H-27), 1.78 (6H, s, $CH_3$-14, $CH_3$-24), 1.91 (4H, t, J = 7.2, H-4, H-19), 2.33 (8H, t, J = 7.2, H-3, H-5, H-18, H-20), 2.70 (4H, t, J = 6.9, H-13, H-25), 4.08 (4H, t, J = 6.8, H-8, H-30), 8.38 (2NH, br.s).

$^{13}$C NMR spectrum ($CDCl_3$): 14.95 (q, $CH_3$-14, $CH_3$-24), 19.29 (t, C-4), 20.00 (t, C-19), 25.81 (t, C-10, C-28), 28.24 (t, C-11, C-27), 28.56 (t, C-12, C-26), 28.82 (t, C-18, C-20), 32.06 (t, C-9, C-29), 38.55 (t, C-3, C-5), 43.48 (t, C-13, C-25), 65.96 (t, C-8, C-30), 151.66 (s, C-14, C-24), 172.81 (s, C-2, C-6), 175.35 (s, C-17, C-21).

Mass spectrum (APCI, 20 eV, *m*/*z*): 509 $[M + H]^+$, 527 $[M + H + H_2O]^+$, 507 $[M - H]^-$, 525 $[M + H_2O - H]^-$, 543 $[M + 2H_2O - H]^-$.

**15,25-Dimethyl-1,8-dioxo-16,17,23,24-tetraazacyclohentriaconta-15,24-dien-2,7,18,22-tetraone (10).** Yield 0.32 g (63%). IR spectrum (ν, $cm^{-1}$): 1735 (O=C–O), 1705 (C=O), 1030 (C–O–C), 1639 (C=N), 1670–1700 (CONH), 3326–3420 (NH).

PMR spectrum ($CDCl_3$, δ, ppm, J/Hz): 1.38–1.43 (8H, m, H-10, H-11, H-29, H-30), 1.44–1.73 (12H, m, H-4, H-5, H-12, H-13, H-27, H-28), 1.80 (6H, s, $CH_3$-15, $CH_3$-25), 1.81 (2H, t, J = 7.0, H-20), 2.28–2.37 (8H, m, J = 7.2, H-3, H-6, H-18, H-21), 2.71 (4H, t, J = 6.9, H-14, H-26), 4.04 (4H, t, J = 6.6, H-9, H-31), 8.40 (2NH, br.s).

$^{13}$C NMR spectrum ($CDCl_3$): 15.33 (q, $CH_3$-15, $CH_3$-25), 19.47 (t, C-20), 23.49 (t, C-4, C-5), 24.30 (t, C-13, C-27), 25.63 (t, C-11, C-29), 26.52 (t, C-12, C-28), 28.89 (t, C-19, C-21), 32.16 (t, C-10, C-30), 38.68 (t, C-3, C-6), 38.97 (t, C-9, C-31), 43.42 (t, C-14, C-26), 152.26 (s, C-15, C-25), 173.18 (s, C-2, C-7), 175.58 (s, C-18, C-22).

Mass spectrum (APCI, 20 eV, *m*/*z*): 523 $[M + H]^+$, 541 $[M + H + H_2O]^+$, 521 $[M - H]^-$, 539 $[M + H_2O - H]^-$, 557 $[M + 2H_2O - H]^-$, 575 $[M + 3H_2O - H]^-$.

**10,14,24,28-Tetramethyl-1,7-dioxo-15,16,22,23-tetraazacyclotriaconta-14,32-dien-2,6,17,21-tetraone (11).** Yield 0.36 g (68%). IR spectrum (ν, $cm^{-1}$): 1735 (O=C–O), 1705 (C=O), 1030 (C–O–C), 1639 (C=N), 1670-1700 (CONH), 3326–3420 (NH).

PMR spectrum ($CDCl_3$, δ, ppm, J/Hz): 0.89 (6H, d, J = 6.5, $CH_3$-10, $CH_3$-28), 1.10–1.20, 1.25–1.30 (4H, m, H-9, H-29), 1.40–1.72 (6H, m, H-10, H-11, H-27, H-28), 1.70 (6H, s, $CH_3$-14, $CH_3$-24), 1.87–1.95 (4H, m, H-4, H-12, H-19, H-26), 2.31 (8H, t, J = 7.3, H-3, H-5, H-18, H-20), 2.73 (4H, t, J = 6.7, H-13, H-25), 4.08, 4.10 (4H, t, J = 6.8, H-8, H-30), 8.15 (2NH, br.s).

$^{13}$C NMR spectrum ($CDCl_3$): 14.94 (q, $CH_3$-14, $CH_3$-24), 19.41 (q, $CH_3$-10, $CH_3$-28), 20.12 (t, C-4), 20.24 (t, C-19), 29.67 (d, C-10, C-28), 32.15 (t, C-18, C-20), 33.29 (t, C-12, C-26), 35.36 (t, C-9, C-29), 36.33 (t, C-11, C-27), 38.93 (t, C-3, C-5), 39.29 (t, C-13, C-25), 62.65 (t, C-8, C-30), 151.29 (s, C-14, C-24), 172.92 (s, C-2, C-6), 175.33 (s, C-17, C-21).

Mass spectrum (APCI, 20 eV, *m*/*z*): 537 $[M + H]^+$, 535 $[M - H]^-$.

**11,15,25,29-Tetramethyl-1,8-dioxo-16,17,23,24-tetraazacyclohentriaconta-15,24-dien-2,7,18,22-tetraone (12).** Yield 0.35 g (64%). IR spectrum (ν, $cm^{-1}$): 1735 (O=C–O), 1705 (C=O), 1030 (C–O–C), 1639 (C=N), 1670–1700 (CONH), 3326–3420 (NH).

PMR spectrum ($CDCl_3$, δ, ppm, J/Hz): 0.87 (6H, d, J = 6.8, $CH_3$-11, $CH_3$-29), 1.05–1.15, 1.25–1.34 (4H, m, H-10, H-30), 1.45–1.60 (6H, m, H-11, H-12, H-28, H-29), 1.63–1.68 (8H, m, H-4, H-5, H-13, H-27), 1.79 (2H, t, J = 6.6, H-20), 1.90 (6H, s, $CH_3$-15, $CH_3$-25), 2.28–2.35 (4H, m, H-3, H-6), 2.40 (4H, t, J = 7.4, H-19, H-21), 2.73 (4H, t, J = 6.8, H-14, H-26), 4.08, 4.09 (4H, t, J = 6.7, H-9, H-31), 8.60 (2NH, br.s).

$^{13}$C NMR spectrum ($CDCl_3$): 15.19 (q, $CH_3$-15, $CH_3$-25), 19.26 (q, $CH_3$-11, $CH_3$-29), 19.35 (t, C-20), 22.46 (t, C-4, C-5), 23.76 (t, C-13, C-27), 29.92 (d, C-11, C-29), 31.68 (t, C-19, C-21), 36.27 (t, C-12, C-28), 36.76 (t, C-10, C-30), 38.76 (t, C-14, C-26), 38.87 (t, C-3, C-6), 62.59 (t, C-9, C-31), 152.04 (s, C-15, C-25), 173.18 (s, C-2, C-7), 175.58 (s, C-18, C-22).

Mass spectrum (APCI, 20 eV, *m*/*z*): 551 $[M + H]^+$, 569 $[M + H + H_2O]^+$, 549 $[M - H]^-$, 567 $[M + H_2O - H]^-$, 585 $[M + 2H_2O - H]^-$.

**7,17-Dimethyl-1,23-dihydroxy-10,14-dioxo-8,9,15,16-tetraazatricosa-7,16-diene (13).** A solution of hydroxyketone (**3**, 0.50 g, 3.4 mmol) in anhydrous dioxane (5 mL) was stirred and treated with glutaric dihydrazide (0.27 g, 1.7 mmol) prepared according to the literature [5]. After 4 h (TLC monitoring) the precipitate was filtered off on a Schott filter, washed with anhydrous $Et_2O$ (10 mL), and evaporated to afford **9** (0.66 g, 97%). IR spectrum (ν, $cm^{-1}$): 1635 (C=N), 1660 (CONH), 3209–3234 (NH), 3406 (OH).

PMR spectrum (MeOH+$C_6D_6$, δ, ppm, J/Hz): 1.15–1.33 (8H, m, H-4′, H-5′), 1.35–1.50 (8H, m, H-3′, H-6′), 1.79 (6H, s, $2CH_3$), 2.12 (2H, t, J = 7.5, H-3), 2.18 (4H, t, J = 7.5, H-2, H-4), 2.24 (4H, t, J = 7.4, H-2′), 3.47 (4H, t, J = 7.1, H-7′), 4.79 (2NH + 2OH + $H_2O$, br.s).

$^{13}$C NMR spectrum (MeOH+$C_6D_6$): 15.40 (q, $CH_3$C-1′), 21.26 (t, C-3), 25.56 (t, C-5′), 28.37 (t, C-3′), 28.89 (t, C-2, C-4), 29.28 (t, C-4′), 32.50 (t, C-6′), 38.34 (t, C-2′), 62.19 (t, C-7′), 161.18 (s, C-1′), 171.05 (s, C-1, C-5).

Mass spectrum (APCI, 20 eV, *m*/*z*): 413 $[M + H]^+$, 411 $[M - H]^-$, 447 $[M + 2H_2O - H]^-$.

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