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Effective Synthesis of Optically Pure Macrocyclic Polyesters from Castor Oil and Dichlorides of 2,6-Pyridinedicarboxylic, Oxalic, and Sebacic Acids

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An effective synthesis of optically pure macrocylclic polyesters is proposed, based on consecutive reactions of [1+1]-condensation of natural triol (castor oil) with dichlorides of 2,6-pyridinedicarboxylic or oxalic acids in carbon tetrachloride in the presence of dimethylformamide and the catalyst N,N-dimethylaminopyridine, followed by the reaction of the obtained macrocylclic alcohols with the dichloride of sebacic acid under similar conditions.

Keywords: Castor oil, pyridinedicarboxylic acid, oxalic acid, sebacic acid, condensation reaction, macrocyclic polyesters.

Эффективный синтез оптически чистых макроциклических полиэфиров из касторового масла и дихлорангидридов 2,6-пиридиндикарбоновой, щавелевой и себациновой кислот

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Предложен эффективный синтез оптически чистых макроциклических полиэфиров, основанный на последовательных реакциях [1+1]-конденсации природного триола (касторового масла) с дихлорангидридами 2,6-пиридиндикарбоновой или щавелевой кислот в четыреххлористом углероде в присутствии диметилформамида и катализатора N,N-диметиламинопиридина, а затем полученных макроциклических спиртов с дихлорангидридом себациновой кислоты в аналогичных условиях.

Ключевые слова: Касторовое масло, пиридиндикарбоновая кислота, щавелевая кислота, себациновая кислота, реакция конденсации, макроциклические полиэфиры.

Introduction

Macrocylclic compounds are of great interest to a wide range of researchers.[1-5] The synthesis of these cycles involves reactions of dihydroxy-[1,6] or diamino-[1,7] substituted compounds, where these functional groups are sufficiently distanced from one another, with dichlorides of dicarboxylic acids. Castor oil, which consists of 90–95% triglyceride of 12*R*-hydroxy-9*Z*-octadecenoic (ricinoleic) acid (**1**), meets the requirements for compounds that can be used as one of the components in the synthesis of macroheterocycles. Furthermore, the presence of hydroxyl and carboxyl groups, double bonds, and a long-chain hydrocarbon skeleton in ricinoleic acid opens up broad possibilities for its transformation into various materials. Despite this vast potential, recently, the use of castor oil as a bioresource for the production of functional materials has been very limited.

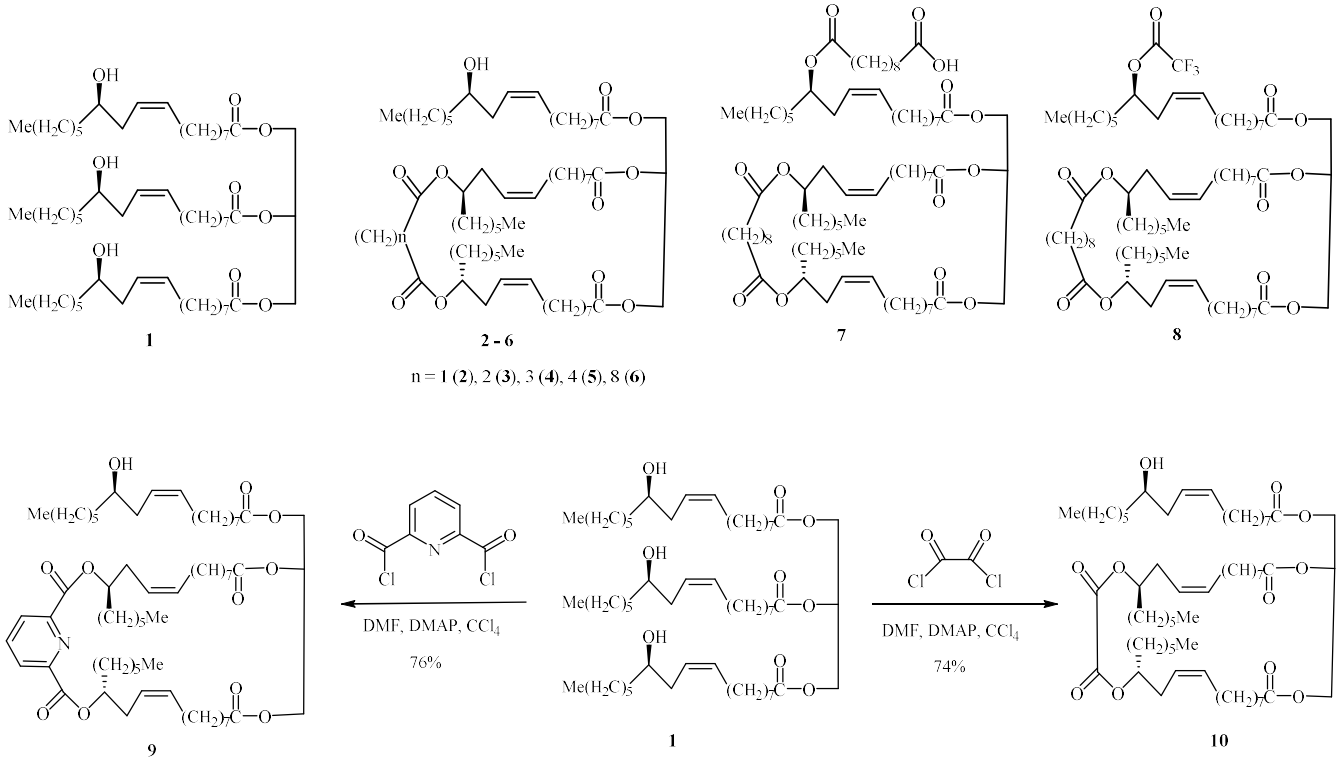
We previously reported efficient one-pot syntheses of a number of macrocyles **2-6** with four ester groups in the rings, based on [1+1]-condensation reactions of castor oil (**1**) with dichlorides of malonic,[8] succinic, glutaric, adipic,[9] and sebacic acids,[10] respectively. We also obtained conjugates of castor oil (**1**) with the dichloride of sebacic acid – macrocyclic polyesters **7** and **8** with four ester groups in the ring, containing in the side chain a residue of (*R*)-ricinoleic acid, where the hydroxyl group is replaced either by a residue of sebacic acid or by a trifluoroacetate group.[10]

Earlier, it was established[11] that compound **6** is toxic *in vitro* to all studied cell lines – HEK293 (human embryonic kidney cells), HepG2 (human hepatocellular carcinoma), A549 (human lung adenocarcinoma), MCF-7 (human breast duct adenocarcinoma), HCT-116 (human colorectal carcinoma). Among these, the cell lines of human breast adenocarcinoma and human colorectal carcinoma were found to be the most sensitive to this compound. Compound **8** exhibited *in vitro* cytotoxicity to the HTC-116 tumor line, while the other studied tumor lines were insensitive to this compound. It should be noted that the cytotoxicity of these compounds is lower compared to 5-fluorouracil (control).

Continuing this research, we planned to conduct reactions of castor oil (**1**) with 1 and 2 equivalents of the dichlorides of 2,6-pyridinedicarboxylic acid and oxalic acid to produce macrocyclic polyesters. These acids were chosen for good reason. It is known that the ester derivatives of 2,6-pyridinedicarboxylic acid, including macrocycles, possess a broad spectrum of pharmacological activities (antibacterial, anti-inflammatory, anticoagulant, and antitumor) and are widely used in complexation chemistry.[12] A number of complex esters of oxalic acid and substituted phenols are used as chemosensitizers in combined antitumor therapy with cytostatics in the treatment of leukemias and drug-resistant leukemias.[13] Therefore, incorporation of residues of 2,6-pyridinedicarboxylic and oxalic acids into the molecule of castor oil could lead to the production of potentially useful materials for medicine and various industries.

Results and Discussion

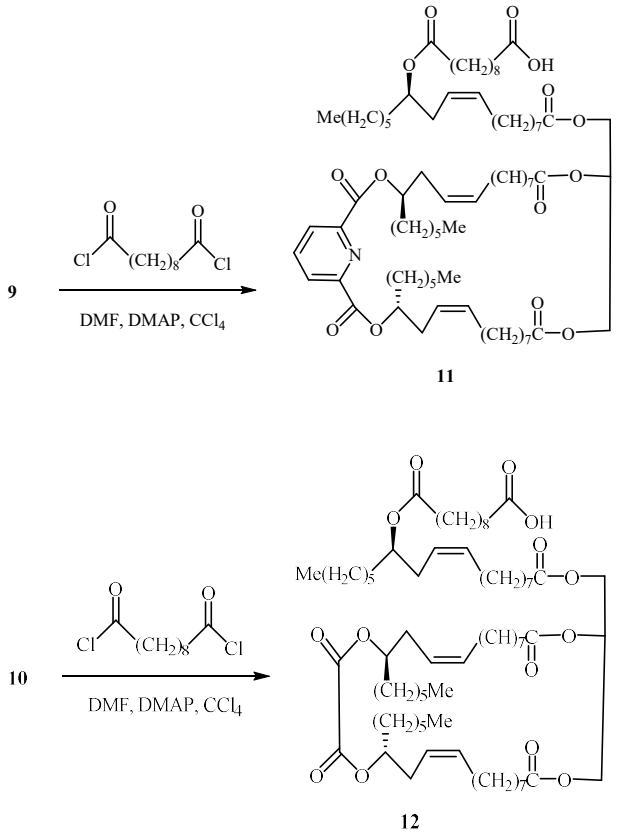
The objective of this work was to study the interaction of castor oil (**1**) with 1 and 2 equivalents of the dichloride of 2,6-pyridinedicarboxylic acid or oxalyl chloride to obtain macrocyclic polyesters. During our research, it was established that during the [1+1]-condensation of castor oil (**1**) with the dichloride of 2,6-pyridinedicarboxylic acid or oxalyl chloride in dry CCl_4 at room temperature in the presence of DMF and the catalyst N,N-dimethylaminopyridine (DMAP), macrocyclic tetraesters **9** and **10** are formed with high yields, containing in the side chain a residue of unreacted ricinoleic acid (Scheme 1). The use of additives of the more basic pyridine (p*K* 5.23)[14] (as in experiments with malonic acid)[8] and Et_3N (p*K* 9.80) (in the case of succinic, glutaric, adipic, and sebacic acids)[9] instead of DMF, which, unlike amines, is a relatively weak base. In this case, it is unacceptable, because it leads to complete tarring of the reaction mass. This is most likely due to the fact that 2,6-pyridindicarboxylic acid (p*K* 2.21) and oxalic acid (p*K* 1.25) are stronger than malonic acid (p*K* 2.85), succinic acid (p*K* 4.19), glutaric acid (p*K* 4.34), adipic acid (p*K* 4.43) and sebacic acid (p*K* 4.55) and therefore they cause rapid polycondensation of substrates.



Scheme 1.

Evidence for the formation of tetraester **10** is provided by the following facts. In the ^{1}H NMR spectrum, along with a multiplet in the range of 3.84–3.90 ppm with an intensity of 1H, corresponding to the fragment >CH–OH, there is a multiplet in the range of 4.87–4.99 with an intensity of 2H, corresponding to two fragments >CH–OCO-. In the ^{13}C NMR spectrum, alongside signals at 127.93 and 130.03 ppm, corresponding to the carbon atoms of the double bonds C-11' and C-12', signals at 123.56 and 133.30 ppm of the carbon atoms of the double bonds C-10, C-11, and C-20, C-21 appear. Additionally, to the signals at 172.82 and 173.22 ppm of the ester groups of ricinoleic acid, a signal at 157.94 ppm of the formed ester groups is added.

Attempts to obtain the product from [1+2]-condensation of castor oil (**1**) with both the dichloride of 2,6-pyridinedicarboxylic acid and oxalyl chloride in the presence of pyridine, triethylamine, or DMF also resulted in complete gelling of the reaction masses. However, the use of the dichloride of sebacic acid in the [1+1]-condensation reaction with macrocyclic alcohols **9** and **10** in the presence of DMF led to the formation of the corresponding esters **11** and **12**, containing in the side chain a residue of ricinoleic acid, where the hydroxyl group is replaced by a residue of sebacic acid (Scheme 2).



Scheme 2.

Since sebacic acid is used in medicine for constructing three-dimensional frames for use as wound dressings and for obtaining biodegradable elastomeric membranes in retinal transplant surgeries, and oral administration of sebacic acid improves glycemic control in type 2 diabetes, and biodegradable polyanhydrides based on betulindisuccinate and sebacic acid exhibited significant antitumor activity against cell lines HeLa, A549, U-87MG, KB, HepG2, incorporating it into the molecule of castor oil is also effective for obtaining functional materials for medicine and various industries.

Evidence for the formation of ester **11** is provided by the following facts. In the ^{1}H NMR spectrum, there is only a multiplet in the range of 5.25-5.43 ppm with an intensity of 3H, corresponding to three fragments >CH-OCO-, while in the ^{13}C NMR spectrum, there are only signals at 123.29 and 132.22 ppm for the carbon atoms of the double bonds C-10, C-11, C-11', C-12' and C-21, C-22, and also present is a signal at 176.95 ppm, corresponding to a free carboxyl group.

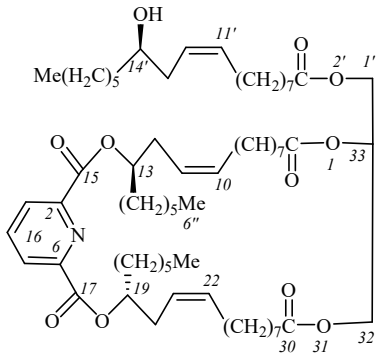
Evidence for the formation of ester **12** can be provided by the following facts. In the ^{1}H NMR spectrum, only a multiplet in the range of 4.85–4.98 ppm with an intensity of 3H appears, corresponding to three fragments >CH-OCO-, and in the ^{13}C NMR spectrum, only signals at 123.29 and 132.22 ppm for the carbon atoms of the double bonds C-10, C-11, C-11', C-12' and C-20, C-21 appear, as well as a signal at 176.95 ppm, corresponding to a free carboxyl group.

Experimental

The equipment of the regional center for collective use "Agidel" of the UFIC RAS was used in the work. IR spectra in thin layers were recorded on IR Prestige-21 Shimadzu instrument. NMR spectra were recorded in $CDCl_3$ with TMS internal standard on a «BRUKER AM-500» spectrometer (operating frequency 500.13 MHz for ^{1}H; 125.76 MHz for ^{13}C). TLC monitoring used Sorbfil SiO_2 (Russia). Elemental analyses of all compounds were agreed with those calculated. Mass spectra were recorded on Shimadzu LCMS 2010 EV instrument using under atmospheric pressure chemical ionization (APCI) with electron energy 20 eV and detection of positive and negative ions. The liquid mobile phase was H_2O and/or CH_3CN at f low rate 0.02 ml/min. Refined unbleached castor oil of grade 1 according to State standard GOST 6757-96 (NMZHK, Nizhny Novgorod, Russia), $[\alpha]_D^{20}$ +2.0 (c 0.1, $CHCl_3$) was used for the work.

*13R,19R-Dihexyl-33-(2'-oxa-3'-oxo-14'R-hydroxyeicos-11'Z-en-1'-yl)-16^1-aza-1,14,18,31-tetraoxa-2,15,17,30-tetraoxo-16(2,6)-pyridinacyclotritriaconta-10Z,21Z-dienaphane (**9**).* Dry castor oil (**1**) (3.00 g, 3.2 mmol) and 0.005 g (0.04 mmol) DMAP in dry CCl_4 (80 mL) and dry DMF (0.51 g, 7.0 mmol) was added to the solution of fresh 2,6-pyridinedicarboxylic acid dichloride (0.71 g, 3.5 mmol) in dry CCl_4 (20 mL) in an inert atmosphere of argon. The reaction mixture was stirred for 48 hours at room temperature (TLC control), then diluted with CH_2Cl_2 (150 mL) and sequentially washed with saturated solution $NaHCO_3$ and brine, dried with Na_2SO_4 and evaporated. Yield: 2.60 g (76%) of macroheterocycle **9**, $[\alpha]_D^{20}$ +13.0 (*c* 0.5, $CHCl_3$). Found: C 72.32, H 9.89, N 1.41. $C_{64}H_{105}NO_{11}$ requires C 72.21, H 9.94, N 1.32. *m/z* (ESI) (%) 1064.8 (100) $[M+H]^+$, 1082.7 (70) $[M+H_2O+H]^+$. IR (KBr) ν_{max} cm^{-1}: 1584 (Ar), 1638 (C=C), 1743 (C=O), 3556 (OH). ^{1}H NMR ($CDCl_3$) δ_H ppm: 0.88 (t, 9H, *J*=6.6 Hz, H-20', H-6''), 1.19–1.79 (m, 60H, H-4–H-8, H-24–H-28, H-5'–H-9', H-15'–H-19', H-1''–H-5''), 2.01–2.12 (m, 12H, H-9, H-12, H-20, H-23, H-10', H-13'), 2.28–2.57 (m, 6H, H-3, H-29, H-4'), 3.62–3.64 (m, 1H, H-14'), 3.90 (br.s, 1H, OH), 4.01–4.55 (m, 7H, H-13, H-19, H-32, H-33, H-1'), 5.19–5.55 (m, 6H, H-10, H-11, H-21, H-22, H-11', H-12'), 7.97–8.10 (m, 1H, H-16(4)), 8.23 (d, 2H, *J*=7.7 Hz, H-16(3), H-16(5)). ^{13}C NMR ($CDCl_3$) δ_C ppm: 13.98 (q, C-20', C-6''), 22.50, 24.74, 25.29, 27.31, 28.96, 29.00, 29.05, 29.09, 29.44, 31.64, 31.81, 33.44, 33.92, 34.08, 35.30 (t, C-3–C-9, C-12, C-20, C-23–C-29, C-4'–C-10', C-13', C-15'–C-19', C-1''–C-5''), 62.02 (t, C-1', C-32), 68.82 (d, C-14'), 71.41 (d, C-33), 76.09 (d,

C-13, C-19), 123.95 (d, C-11, C-21), 127.38 (d, C-16(3), C-16(5)), 127.83 (d, C-12'), 130.14 (d, C-11'), 133.78 (d, C-10, C-22), 137.78 (d, C-16(4)), 149.02 (d, C-16(2), C-16(6)), 163.96 (s, C-15, C-17), 172.73 (s, C-3', C-30), 173.14 (s, C-2).



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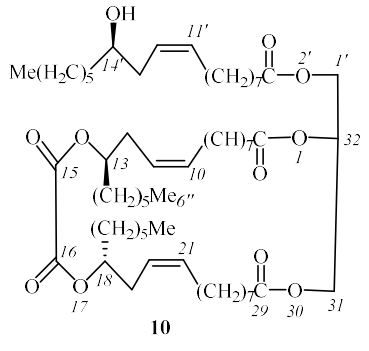
*13R,18R-Dihexyl-32-(2'-oxa-3'-oxo-14'R-hydroxyeicos-11'Z-en-1'-yl)-1,14,17,30-tetraoxa-2,15,16,29-tetraoxocyclodotriaconta-10Z,20Z-dien (**10**).* Dry castor oil (**1**) (3.00 g, 3.2 mmol) and 0.005 g (0.04 mmol) DMAP in dry CCl_4 (80 mL) and dry DMF (0.51 g, 7.0 mmol) was added to the solution of fresh oxalic chloride (0.45 g, 3.5 mmol) in dry CCl_4 (20 mL) in an inert atmosphere of argon. The reaction mixture was stirred for 48 hours at room temperature (TLC control), then diluted with CH_2Cl_2 (150 mL) and sequentially washed with saturated solution $NaHCO_3$ and brine, dried with Na_2SO_4 and evaporated. Yield: 2.34 g (74%) of macroheterocycle **10**, $[\alpha]_D^{20}$ +167.0 (*c* 0.05, $CHCl_3$). Found: C 72.02, H 10.39. $C_{59}H_{102}O_{11}$ requires C 71.77, H 10.41. *m/z* (ESI) (%) 986.91 (25) $[M]^+$, 969.90 (100) $[M-H_2O]^+$. IR (KBr) ν_{max} cm^{-1}: 1650 (C=C), 1742 (C=O), 3550 (OH). ^1H NMR ($CDCl_3$) δ_H ppm: 0.86 (t, 9H, *J*=6.6 Hz, H-20', H-6''), 1.15-1.51 (m, 60H, H-4–H-8, H-23–H-27, H-5'–H-9', H-15'–H-19', H-1"–H-5"), 1.90–2.01 (m, 12H, H-9, H-12, H-19, H-22, H-10', H-13'), 2.37 (t, 6H, *J*=7.3 Hz, H-3, H-28, H-4'), 2.85 (br.s, 1H, OH), 3.84–3.90 (m, 1H, H-14'), 4.05–4.27 (m, 4H, H-31, H-1'), 4.87–4.99 (m, 3H, H-13, H-18, H-32), 5.20–5.51 (m, 6H, H-10, H-11, H-20, H-21, H-11', H-12'). ^{13}C NMR ($CDCl_3$) δ_C ppm: 14.06 (q, C-20', C-6"), 22.56, 24.85, 25.21, 25.65, 27.22, 27.29, 27.37, 29.03, 29.14, 29.51, 29.72, 31.72, 31.95, 33.33, 33.53, 34.18, 34.26 (t, C-3–C-9, C-12, C-19, C-22–C-28, C-4'–C-10', C-13', C-15'–C-19', C-1''–C-5''), 62.01 (t, C-1', C-31), 68.85 (d, C-32), 71.37 (d, C-14'), 76.81 (d, C-13, C-18), 123.56 (d, C-11, C-20), 127.93 (d, C-12'), 130.03 (d, C-11'), 133.30 (d, C-10, C-21), 157.94 (s, C-15, C-16), 172.82 (s, C-3', C-29), 173.22 (s, C-2).



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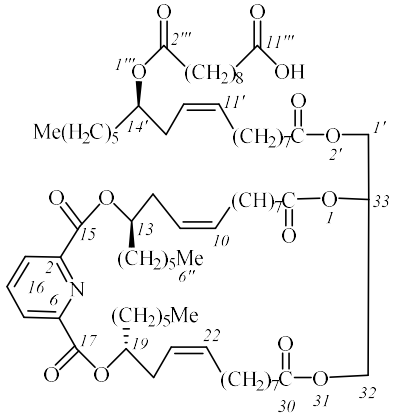
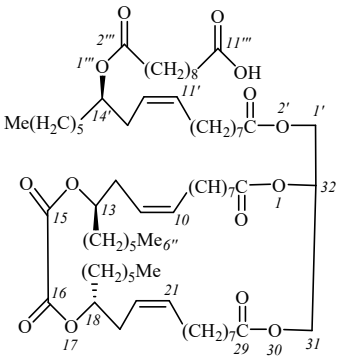
*13R,19R-Dihexyl-33-(2'-oxa-3'-oxo-14'R-(1'''-oxa-2''',11'''-dioxo-11'''-hydroxyundecan-1'''-yl)-16[1]-aza-1,14,18,31-tetraoxa-2,15,17,30-tetraoxo-16(2,6)-pyridinacyc-lotritriaconta-10Z,21Z-dienaphane (**11**).* Fresh sebacic dichloride (0.18 g, 0.53 mmol) in dry CCl_4 (20 mL) was added to the solution of macrocycle **9** (0.51 g, 0.48 mmol), 0.005 g (0.04 mmol) DMAP and dry DMF (1.90 g, 26.0 mmol) in dry CCl_4 (100 mL) in an inert atmosphere of argon. The reaction mixture was stirred for 48 hours at room temperature (TLC control), then diluted with CH_2Cl_2 (150 mL) and sequentially washed with saturated solution $NaHCO_3$ and brine, dried with Na_2SO_4 and evaporated. Yield: 0.49 g (82%) of macroheterocycle **11**, $[\alpha]_D^{20}$ +59.0 (*c* 0.1, $CHCl_3$). Found: C 71.42, H 9.61, N 1.15. $C_{74}H_{121}NO_{14}$ requires C 71.17; H 9.77; N 1.12. IR (KBr) ν_{max} cm^{-1}: 1580 (Ar), 1640 (C=C), 1700 (C=O acid), 1744 (C=O ester), 3550 (OH). ^1H NMR ($CDCl_3$) δ_H ppm: 0.76 (t, 9H, *J*=6.6 Hz, H-20', H-6''), 1.12–1.68 (m, 72H, H-4–H-8, H-24–H-28, H-5'–H-9', H-15'–H-19', H-1"–H-5", H-4'''–H-9'''), 2.01–2.13 (m, 12H, H-9, H-12, H-20, H-23, H-10', H-13'), 2.22 (t, 8H, *J*=7.3 Hz, H-3, H-29, H-4', H-3'''), 2.32 (t, 2H, *J*=5.4 Hz, H-10'''), 4.04–4.55 (m, 8H, H-13, H-19, H-32, H-33, H-1', H-14'), 5.25–5.43 (m, 6H, H-10, H-11, H-21, H-22, H-11', H-12'), 7.97–8.10 (m, 1H, H-16(4)), 8.23 (d, 2H, *J*=7.7 Hz, H-16(3), H-16(5)). ^{13}C NMR ($CDCl_3$) δ_C ppm: 13.96 (q, C-20', C-6"), 22.46, 24.74, 25.10, 25.63, 27.11, 27.27, 27.30, 28.89, 29.06, 29.28, 29.40, 29.61, 31.54, 31.84, 33.47, 33.92, 34.01, 34.93, 36.67 (t, C-3–C-9, C-12, C-20, C-23–C-29, C-4'–C-10', C-13', C-15'–C-19', C-1''–C-5'', C-3'''–C-10'''), 62.02 (t, C-1', C-32), 74.06 (d, C-14'), 76.91 (d, C-13, C-18), 123.29 (d, C-11, C-21, C-12'), 132.22 (d, C-10, C-22, C-11'), 157.97 (s, C-15, C-17), 162.97 (s, C-2'''), 173.23, 172.79 (s, C-2, C-30, C-3'), 176.95 (s, C-11''').



11

*13R,18R-Dihexyl-32-(2'-oxa-3'-oxo-14'R-(1'''-oxa-2''',11'''-dioxo-11'''-hydroxyundecan-1'''-yl)eicos-11'Z-en-1'-yl)-1,14,17,30-tetraoxa-2,15,16,29-tetraoxocyclodotriaconta-10Z,20Z-dien (**12**).* Fresh sebacic dichloride (0.18 g, 0.53 mmol) in dry CCl_4 (20 mL) was added to the solution macrocycle of **10** (0.50 g, 0.51 mmol), 0.005 g (0.04 mmol) DMAP and dry DMF (1.90 g, 26.0 mmol) in dry CCl_4 (100 mL) in an inert atmosphere of argon. The reaction mixture was stirred for 48 hours at room temperature (TLC control), then diluted with CH_2Cl_2 (150 mL) and sequentially washed with saturated solution $NaHCO_3$ and brine, dried with Na_2SO_4 and evaporated. Yield: 0.41 g (72%) of macroheterocycle **12**, $[\alpha]_D^{20}$ +12.0 (*c* 0.05, $CHCl_3$). Found: C 71.32, H 9.56. $C_{69}H_{118}O_{14}$ requires C 70.73; H 10.15. IR (KBr) ν_{max} cm^{-1}: 1650 (C=C), 1695 (C=O acid), 1742 (C=O ester), 3447 (OH). ^1H NMR ($CDCl_3$) δ_H ppm: 0.80 (t, 9H, *J*=6.5 Hz, H-20', H-6''), 1.13–1.60 (m, 72H, H-4–H-8, H-23–H-27, H-5'–H-9', H-15'–H-19', H-1"–H-5", H-4'''–H-9'''), 1.90–2.16 (m, 12H, H-9, H-12, H-19, H-22, H-10', H-13'), 2.21 (t, 8H, *J*=7.3 Hz, H-3, H-28, H-4', H-3'''), 2.65 (t, 2H, *J*=5.4 Hz, H-10'''), 4.04–4.21 (m, 4H, H-31, H-1'), 4.85–4.98 (m, 4H, H-13, H-18, H-32, H-14'), 5.25–5.43 (m, 6H, H-10, H-11, H-20, H-21, H-11', H-12'). ^{13}C NMR ($CDCl_3$) δ_C ppm: 14.03 (q, C-20', C-6"), 22.46, 24.74, 25.10, 25.63, 27.11, 27.27, 27.30, 28.89, 29.06, 29.28, 29.40, 29.61, 31.54, 31.84, 33.47, 33.92, 34.01, 34.93, 36.67 (t, C-3–C-9, C-12, C-19, C-22–C-28, C-4'–C-10', C-13', C-15'÷C-19', C-1''–C-5'', C-3'''–C-10'''), 62.02 (t, C-1', C-31), 71.43 (d, C-32), 74.06 (d, C-14'), 76.91 (d, C-13, C-18), 123.29 (d, C-11, C-20, C-12'), 132.22 (d, C-11', C-10, C-21), 157.97 (s, C-15, C-16),

162.97 (s, C-2’’’), 172.79 (s, C-29, C-3’), 173.23 (s, C-2), 176.95 (s, C-11’’’).



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Conclusion

Thus, based on the available optically pure triol – castor oil – methods have been developed for the synthesis with high yields of four macrocylclic polyesters, based on consecutive [1+1]-condensation reactions of it with the dichlorides of 2,6-pyridine dicarboxylic or oxalic acids, followed by the reaction with the dichloranhydride of sebacic acid in carbon tetrachloride in the presence of dimethylformamide and N,N-dimethylaminopyridine.

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