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Synthesis of 2-(12-Aryldodecanoyl)cyclohexane-1,3-diones

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Abstract—Synthesis was developed of 2-(10-undecenoyl)cyclohexane-1,3-diones containing in the side chain keto and hydroxy groups and a phenyl substituent. The synthesis is underlain by a nitrile oxide approach. The scheme included isoxazole synthesis for the protection of the β,β'-tricarbonyl fragment, building up of a heterocycle by 1,3-dipolar cycloaddition of nitrile oxide *in situ* to the terminal double bond, cycle opening (of isoxazole and isoxazoline), and alkaline hydrolysis.

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Cyclic β,β'-triketones (2-acylcyclohexane-1,3-diones, 2-acylcyclopentane-1,3-diones) and related to them by structure acylphloroglucinols, flavonoids, and isoflavonoids form a large group of natural polyketides produced by plants, microorganisms, insects and exhibit a wide range of biological activity [1–5]. Many among these compounds and their synthetic derivatives have found application in agrochemistry and pharmcology [6–8]. On the other hand, the polyfunctionality and the high reactivity of the mentioned compounds provide rich possibilities for their modification and application as universal synthons for the preparation of the other classes of biologically active substances (steroids, prostaglandins, antibiotics, pheromones, kairomones of insects, etc.) [9–12].

Formerly a synthesis was described of 2-acylcyclohexane-1,3-diones **1** that were utilized in the preparation of environmentally friendly pheromone traps against various pests [13–16]. The synthesis of β,β'-triketones **1** and their analogs is now relatively simple and is underlain by the previously developed method of O–C-isomerization of monoenol esters of appropriate acids and cyclohexane-1,3-dione under the action of various catalysts [15] (Scheme 1).

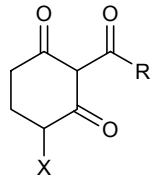
The goal of this work was the synthesis of analogs of natural 2-acylcyclohexane-1,3-diones **1** [R = $(CH_2)_{10}$Ph, X = H] having additional oxygen functions in the side acyl chain proceeding from 2-acylcyclohexane-1,3-diones **2a** and **2b** with a terminal double bond.

The bringing of the terminal double bond in reactions of acyl chain building and introduction of additional functions into its backbone is impossible without protecting the active carbonyl groups of β,β'-triketones. To this end the classic method was chosen of the synthesis of fused isoxazoles from 2-acylcyclohexane-1,3-diones **2a** and **2b** and hydroxylamine protecting two carbonyl groups and providing a possibility to modify the acyl chain with subsequent opportunity of recovering polyfunctionality by the cleavage of isoxazoles [16].

Common strategy of isoxazole (nitrile oxide) method of the synthesis includes three stages: synthesis of the heterocycle by the 1,3-dipolar cycloaddition of nitrile oxide *in situ* to the unsaturated compound; modification of the molecule of the formed isoxazole; ring opening leading to the formation of bifunctional compounds. In our case the second stage is absent.

2-(10-Undecenoyl)cyclohexane-1,3-diones **2a** and **2b** in the reaction with hydroxylamine readily formed isoxazoles **3a** and **3b** containing a terminal double bond in the side chain acyl fragment (Scheme 2) [17].

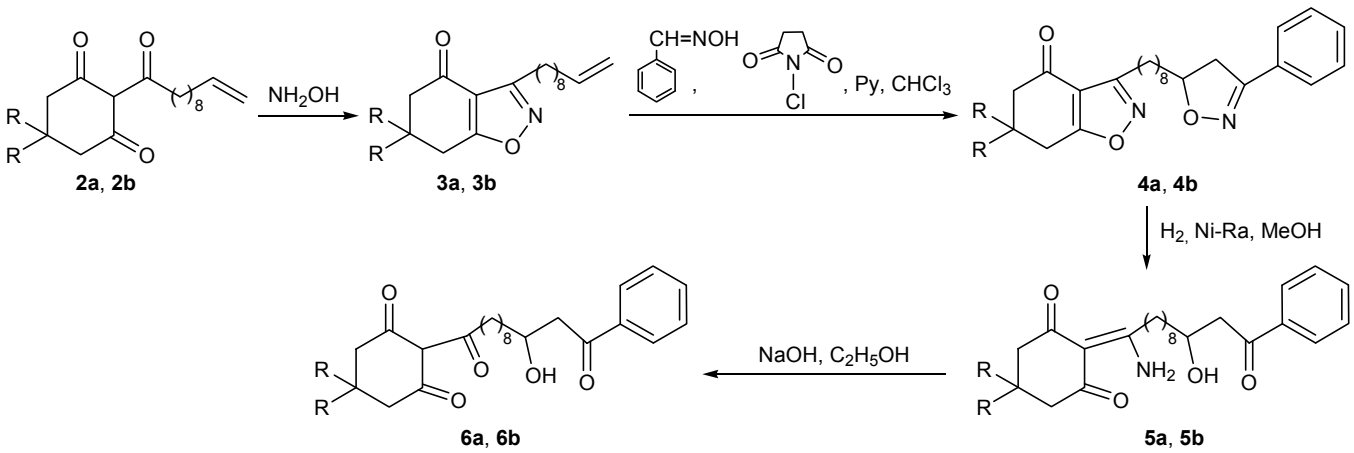
Scheme 1.



1

X = H, OH; R = carbon fragment (C_{11}–C_{17}) of saturated, mono- and dienecarboxylic acids.

Scheme 2.



R = H (**a**), CH_3 (**b**).

The structure of compounds **3a** and **3b** was confirmed by IR and ^{1}H NMR spectra. For instance, in contrast to the spectra of initial β,β'-triketones **2a** and **2b**, in the ^{1}H NMR spectra of isoxazoles **3a** and **3b** the proton of the enol hydroxyl was absent, in the IR spectrum the signal of the C=N bond appeared at 1600 cm^{-1}, and an absorption band of a single carbonyl group was observed at 1700 cm^{-1}. As a result of 1,3-dipolar cycloaddition of benzonitrile oxide *in situ* to the terminal double bond of isoxazoles **3a** and **3b** an isoxazoline ring was formed.

In the ^{1}H NMR spectra of isoxazoles **4a** and **4b** the signals appear of methylene protons from isoxazoline ring as doublets of doublets at 2.95 and 3.38 ppm, of the methine proton as a multiplet at 4.72 ppm, and of aromatic protons at 7.3–7.8 ppm.

Under the hydrogenation conditions in the presence of Raney nickel and catalytic amounts of trimethylamine in methanol both the isoxazole and isoxazoline ring suffer opening affording ketoimine from the first ring and β-hydroxyketone from the second ring in good agreement with the published data [16, 18, 19]. We failed to isolate the intermediately formed oximines since under the conditions of catalytic hydrogenation in the presence of a base they readily suffer hydrolysis giving β-hydroxy ketones **5a** and **5b**. The structure of the latter was confirmed by ^{1}H NMR spectra where signals were present of methine and hydroxyl protons in the position *10* at 4.22 and 4.73 ppm respectively, and also of two protons (free and chelated) of the amino group at 6.56 and 12.26 ppm.

The subsequent hydrolysis of the enaminodiketone moiety of the β-hydroxy ketones **5a** and **5b** resulted in the formation of 2-(10-hydroxy-12-oxo-12-phenyldodecanoyl)cyclohexane-1,3-diones **6a** and **6b** in 70–72% yields. The recovery of the β,β'-tricarbonyl system is confirmed by the presence of a signal of enol proton as a singlet in the region 18.24–18.26 ppm, and the appearance in the IR spectra of absorption bands at 1520 cm^{-1} (chelated carbonyl group) and 1670 cm^{-1} (free carbonyl group).

The synthesis of 2-(10-hydroxy-12-oxo-12-phenyldodecanoyl)cyclohexane-1,3-diones **6a** and **6b** provides an opportunity to prepare analogs of natural β,β'-triketones with a terminal phenyl group and a functionalized acyl fragment [20].

EXPERIMENTAL

^{1}H and ^{13}C NMR spectra were registered on a spectrometer Bruker BioSpin Avance 500 using 5-mm probe (QNP) with Z-gradient at the sample temperature 193 K. As internal reference the signals served of residual protons and carbon atoms of the solvent $CDCl_3$ (δ_H 7.26, δ_C 77 ppm). IR spectra were recorded on a spectrophotometer FT-IR Perkin Elmer Spectrum 100. Melting points were measured on a Boëtius apparatus. Elemental analysis was carried out on a CHNS–O analyzer Eurovector EA3000. The reaction progress and the homogeneity of compounds synthesized was monitored by TLC on Silufol UV-254 plates (eluent hexane–ethyl ether). In column chromatography was used silica gel (100/160, 230–400 mesh), eluent hexane–ethyl ether.

6,7-Dihydro-3-(dec-9-enyl)benzo[*d*]isoxazol-4(5*H*)-one (3a). A mixture of 0.806 g (2.9 mmol) of β,β'-triketone **2a** in 20 mL of ethanol, 0.206 g (2.97 mmol) of hydroxylamine hydrochloride in 15 mL of water, 0.119 g (29.7 mmol) of sodium hydroxide in 10 mL of water was boiled for 3–5 h, cooled, acidified with sulfuric acid to pH 1 and again boiled at reflux for 36 h. The reaction mixture was diluted with the same volume of water and was extracted with ethyl ether (5 × 100 mL). The organic solution was washed with water, dried with magnesium sulfate, filtered, and ether was evaporated. The residue was subjected to preparative separation. Yield 0.65 g (82%), oily substance. IR spectrum (film), ν, cm^{-1}: 1700 s (C=O), 1600 s (C=N). ^1H NMR spectrum, δ, ppm: 1.24–1.44 m (10H, $5CH_2$), 1.63–1.83 m (2H, CH_2), 1.94–2.14 m (2H, CH_2), 2.12–2.32 m (2H, CH_2), 2.35 t (2H, CH_2, *J* 7.5 Hz), 2.52 t (2H, CH_2, *J* 7.5 Hz), 2.98 t (2H, CH_2, *J* 7.5 Hz), 4.87–5.07 m (2H, CH_2), 5.71–5.91 m (1H, CH). ^{13}C NMR spectrum, δ, ppm: 22.2, 23.4, 28.3, 29.2, 29.6, 29.7, 33.9, 38.9, 113.1, 115.7, 139.1, 161.9, 173.0, 196.8. Found, %: C 74.18; H 9.21; N 5.12. $C_{17}H_{25}NO_2$. Calculated, %: C 74.14; H 9.15; N 5.09.

6,7-Dihydro-3-(dec-9-enyl)-6,6-dimethylbenzo[*d*]-isoxazol-4(5*H*)-one (3b) was obtained similarly from 0.887 g (2.9 mmol) β,β'-triketone **2b** in 20 mL of ethanol. Yield 0.72 g (82%), oily substance. IR spectrum (film), ν, cm^{-1}: 1700 s (C=O), 1600 s (C=N). ^1H NMR spectrum, δ, ppm: 1.16 s (6H, $2CH_3$), 1.22–1.42 m (10H, $5CH_2$), 1.61–1.81 m (2H, CH_2), 1.93–2.13 m (2H, CH_2), 2.39 s (2H, CH_2), 2.83 s (2H, CH_2), 2.85 t (2H, CH_2, *J* 7.5 Hz), 4.87–5.07 m (2H, CH_2), 5.71–5.91 m (1H, CH). ^{13}C NMR spectrum, δ, ppm: 28.2, 28.3, 29.2, 29.6, 29.7, 33.9, 35.5, 36.5, 53.1, 113.1, 115.7, 139.1, 161.9, 180.3, 192.8. Found, %: C 75.30; H 9.68; N 4.67. $C_{19}H_{29}NO_2$. Calculated, %: C 75.21; H 9.63; N 4.62.

3-[8-(4,5-Dihydro-3-phenylisoxazol-5-yl)octyl]-6,7-dihydrobenzo[*d*]isoxazol-4(5*H*)-one (4a). A solution of 0.121 g (1 mmol) of benzaldoxime in 2.5 mL of anhydrous chloroform was added dropwise to slurry of 0.134 mg (1 mmol) of N-chlorosuccinimide in 2.5 mL of anhydrous chloroform containing 0.0016 mL (0.02 mmol) of pyridine. The reaction mixture was stirred for 15 min, 0.275 g (1 mmol) of compound **3a** in 2.5 mL of anhydrous chloroform was added in one portion. A solution of 0.14 mL (1 mmol) of trimethylamine in 10 mL of chloroform was added (very slowly!) at stirring to the reaction mixture, and it was left standing for 12 h. The mixture was diluted with chloroform, washed with water (20 mL), with a saturated sodium carbonate solution (20 mL), and dried with magnesium sulfate. The residue after evaporation of the solvent was subjected to column chromatography. Yield 0.236 g (60%), mp 53–56°C (C_6H_{14}–$C_2H_5OC_2H_5$). IR spectrum (KBr), ν, cm^{-1}: 3300 br (C–H_{arom}), 1700–1600 s (C=O, C=N), 1480 s (C–C_{arom}). ^1H NMR spectrum, δ, ppm: 1.22–1.44 m (10H, $5CH_2$), 1.65–1.85 m (4H, $2CH_2$), 2.11–2.31 m (2H, CH_2), 2.51 t (2H, CH_2, *J* 7.5 Hz), 2.82 t (2H, CH_2, *J* 7.5 Hz), 2.95 t (2H, CH_2, *J* 8.0 Hz), 2.97 d.d (1H, CH, *J* 16.5, 8.0 Hz), 3.39 d.d (1H, CH, *J* 16.5, 10.5 Hz), 4.63–4.83 m (1H, CH), 7.39 t (3H, H_{arom}, *J* 7.0 Hz), 7.67 d (2H, H_{arom}, *J* 7.0 Hz). ^{13}C NMR spectrum, δ, ppm: 22.2, 23.4, 25.1, 28.3, 29.2, 29.6, 29.9, 37.9, 38.9, 41.0, 68.8, 113.1, 128.2, 128.8, 131.0, 136.0, 156.2, 161.9, 173.0, 196.8. Found, %: C 73.06; H 7.65; N 7.11. $C_{24}H_{30}N_2O_3$. Calculated, %: C 73.07; H 7.66; N 7.10.

5,7-Dihydro-3-[8-(4,5-dihydro-3-phenylisoxazole-5-yl)octyl]-6,6-dimethyl-6,7-dihydrobenzo[*d*]-isoxazol-4(5*H*)-one (4b) was obtained similarly from 0.303 g (1 mmol) of compound **3b**. Yield 0.257 g (61%), mp 47–49°C (C_6H_{14}–$C_2H_5OC_2H_5$). IR spectrum (KBr), ν, cm^{-1}: 3300 br (C–H_{arom}), 1700–1600 s (C=O, C=N), 1480 s (C–C_{arom}), 1370 s (CH_3 *gem*-dimethyl). ^1H NMR spectrum, δ, ppm: 1.15 s (6H, $2CH_3$), 1.22–1.42 m (10H, CH_2), 1.61–1.81 m (4H, $2CH_2$), 2.39 s (2H, CH_2), 2.82 s (2H, CH_2), 2.85 t (2H, CH_2, *J* 8.0 Hz), 2.95 d.d (1H, CH, *J* 16.5, 8.0 Hz), 3.38 d.d (1H, CH, *J* 16.5, 10.5 Hz), 4.62–4.82 m (1H, CH), 7.39 t (3H, H_{arom}, *J* 7.0 Hz), 7.67 d (2H, H_{arom}, *J* 7.0 Hz). ^{13}C NMR spectrum, δ, ppm: 25.1, 28.2, 28.3, 29.2, 29.6, 29.9, 35.5, 36.5, 37.9, 41.0, 53.1, 68.8, 113.1, 128.2, 128.8, 131.0, 136.0, 156.2, 161.9, 180.3, 192.8. Found, %: C 73.90; H 8.12; N 6.62. $C_{26}H_{34}N_2O_3$. Calculated, %: C 73.90; H 8.11; N 6.63.

2-(1-Amino-10-hydroxy-12-oxo-12-phenyldodecylidene)cyclohexane-1,3-dione (5a). To a slurry of Raney nickel (~0.2 g) in 2-propanol was added 0.394 g (1 mmol) of isoxazolone **4a** in the same solvent (10 mL) and 2–3 drops of triethylamine. The obtained solution was stirred under a hydrogen atmosphere till the gas consumption finished (~4 h). The reaction mixture was filtered, the solvent was removed on a rotary evaporator, the residue was crystallized from ethyl ether. Yield 0.367 g (92%), mp 77–82°C (ethyl ether). IR spectrum (KBr), ν, cm^{-1}: 3300 br (C–H_{arom}), 1700–1600 s (C=O, C=N), 1465 s (C–C_{arom}). ^1H NMR spectrum, δ, ppm: 1.24–1.44 m (10H, $5CH_2$), 1.61–1.81 m (4H, $2CH_2$), 2.11–2.31 m (2H, CH_2), 2.51 t

(2H, CH_2, *J* 7.5 Hz), 2.82 t (2H, CH_2, *J* 7.5 Hz), 2.95 t (2H, CH_2, *J* 8.0 Hz), 2.96 d.d (1H, CH, *J* 16.5, 8.0 Hz), 3.40 d.d (1H, CH, *J* 16.5, 10.5 Hz), 4.12–4.32 m (1H, CH), 4.63–4.83 m (1H, OH), 6.56 br.s (1H, NH), 7.39 t (3H, H_{arom}, *J* 7.0 Hz), 7.67 d (2H, H_{arom}, *J* 7.0 Hz), 12.26 br.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 15.1, 25.1, 28.9, 29.6, 29.7, 29.9, 31.3, 37.3, 38.8, 48.8, 66.8, 110.5, 128.6, 128.8, 133.1, 136.7, 171.0, 194.4, 196.2. Found, %: C 72.14; H 8.33; N 3.52. $C_{24}H_{33}NO_4$. Calculated, %: C 72.15; H 8.33; N 3.51.

2-(1-Amino-10-hydroxy-12-oxo-12-phenyldodecylidene)-5,5-dimethylcyclohexane-1,3-dione (5b) was obtained similarly from 0.427 g (1 mmol) of isoxazolone **4b**. Yield 0.393 g (92%), mp 87–89°C (ethyl ether). IR spectrum (KBr), ν, cm^{-1}: 3300 br (C–H_{arom}), 1700–1600 s (C=O, C=N), 1465 s (C–C_{arom}), 1370 s (CH_3 *gem*-dimethyl). ^1H NMR spectrum, δ, ppm: 1.03 s (6H, $2CH_3$), 1.24–1.44 m (10H, $5CH_2$), 1.61–1.81 m (4H, $2CH_2$), 2.35 s (2H, CH_2), 2.41 s (2H, CH_2), 2.91 t (2H, CH_2, *J* 8.0 Hz), 2.96 d.d (1H, CH, *J* 16.5, 8.0 Hz), 3.40 d.d (1H, CH, *J* 16.5, 10.5 Hz), 4.12–4.32 m (1H, CH), 4.63–4.83 m (1H, OH), 6.56 br.s (1H, NH), 7.39 t (3H, H_{arom}, *J* 7.0 Hz), 7.67 d (2H, H_{arom}, *J* 7.0 Hz), 12.26 br.s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 25.1, 26.6, 28.9, 29.6, 29.7, 29.9, 30.4, 31.3, 37.3, 48.8, 51.6, 66.8, 110.5, 128.6, 128.8, 133.1, 136.7, 171.0, 194.4, 196.2. Found, %: C 73.04; H 8.71; N 3.28. $C_{26}H_{37}NO_4$. Calculated, %: C 73.03; H 8.72; N 3.28.

2-(10-Hydroxy-12-oxo-12-phenyldodecanoyl)-cyclohexane-1,3-dione (6a). To a solution of 0.599 g (1.5 mmol) of enamine **5a** in 20 mL of ethanol was added 0.072 g (1.8 mmol) of sodium hydroxide dissolved in 5 mL of water. The reaction mixture was boiled for ~2 h, cooled, acidified with a solution of hydrochloric acid (1 : 5) to pH 3, diluted with 100 mL of water, and extracted with ethyl ether (3 × 75 mL). The extract was dried with magnesium sulfate. The residue after evaporation of the solvent was subjected to column chromatography. Yield 0.40 g (70%). IR spectrum (film), ν, cm^{-1}: 3300 br (C–H_{arom}), 1670 s (C=O), 1520 s (C=$O_{chelate}$), 1465 s (C–C_{arom}). ^1H NMR spectrum, δ, ppm: 1.24–1.44 m (12H, $6CH_2$), 1.61–1.81 m (2H, CH_2), 2.14–2.34 m (2H, CH_2), 2.56 t (2H, CH_2, *J* 7.5 Hz), 2.92 t (2H, CH_2, *J* 7.5 Hz), 2.96 d.d (1H, CH, *J* 16.5, 8.0 Hz), 3.05 t (2H, CH_2, *J* 8.0 Hz), 3.40 d.d (1H, CH, *J* 16.5, 10.5 Hz), 4.22 m (1H, CH), 4.63–4.83 m (1H, OH), 7.39 t (3H, H_{arom}, *J* 7.0 Hz), 7.67 d (2H, H_{arom}, *J* 7.0 Hz), 18.24 s (1H, OH). ^{13}C NMR spectrum, δ, ppm: 20.4, 25.1, 25.2, 29.1, 29.3, 29.6, 29.9, 33.3, 37.3, 38.0, 40.2, 48.8, 66.8, 112.2, 128.6, 128.8, 133.1, 136.7, 194.4, 196.2, 194.4, 196.3, 205.4. Found, %: C 71.94; H 8.04. $C_{24}H_{32}O_5$. Calculated, %: C 71.97; H 8.05.

2-(10-Hydroxy-12-oxo-12-phenyldodecanoyl)-5,5-dimethylcyclohexane-1,3-dione (6b) was obtained similarly from 0.640 g (1.5 mmol) of enamine **5b**. Yield 0.434 g (71%). IR spectrum (film), ν, cm^{-1}: 3300 br (C–H_{arom}), 1670 s (C=O), 1520 s (C=$O_{chelate}$), 1465 s (C–C_{arom}), 1370 s (CH_3 *gem*-dimethyl). ^1H NMR spectrum, δ, ppm: 1.08 s (6H, $2CH_3$), 1.16–1.36 m (12H, $6CH_2$), 1.51–1.71 m (2H, CH_2), 2.35 s (2H, CH_2), 2.53 s (2H, CH_2), 2.96 d.d (1H, CH, *J* 16.5, 8.0 Hz), 3.01 t (2H, CH_2, *J* 8.0 Hz), 3.39 d.d (1H, CH, *J* 16.5, 10.5 Hz), 4.62–4.82 m (1H, CH), 5.24–5.44 m (1H, OH), 7.39 t (3H, H_{arom}, *J* 7.0 Hz), 7.67 d (2H, H_{arom}, *J* 7.0 Hz), 18.26 s (1H, OH). ^{13}C NMR spectrum, δ, ppm: 25.1, 25.2, 27.5, 29.1, 29.3, 29.6, 29.9, 30.6, 37.3, 40.2, 45.8, 48.8, 51.7, 66.8, 112.2, 128.6, 128.8, 133.1, 136.7, 196.2, 197.8, 200.1, 205.4. Found, %: C 72.81; H 8.43. $C_{26}H_{36}O_5$. Calculated, %: C 72.87; H 8.47.

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