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Synthesis of Liquid-crystalline Substances from the 5-Alkyl-5-arylisoxazole Series

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Abstract—The oxidation of 3-(4-acetoxyphenyl)-5-pentyl-2-isoxazoline with *N*-bromosuccinimide followed by hydrolysis of the oxidation product furnished 3-(4-hydroxyphenyl)-5-pentyl-isoxazole. Its esterification or benzylation afforded the target liquid-crystalline 3-aryl-5-pentyl-isoxazoles.

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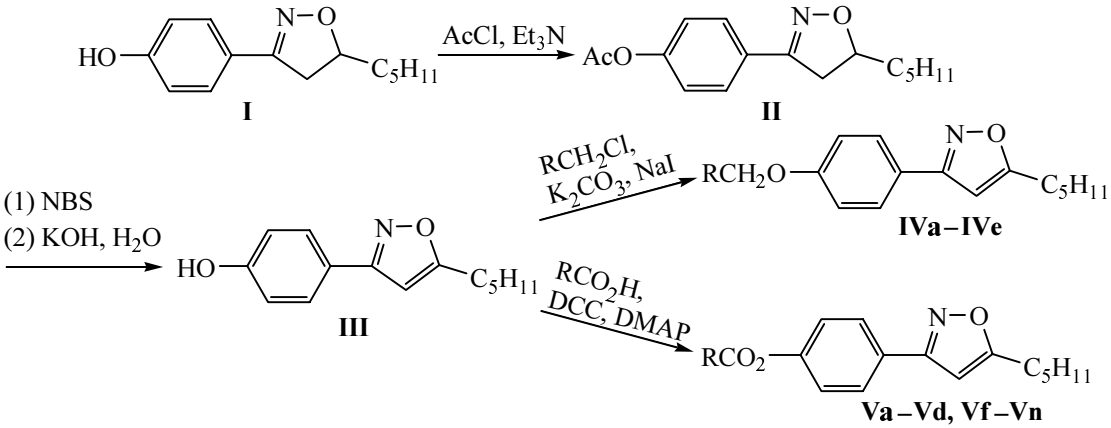
Various five-membered heterocycles are probable promising structural units for liquid-crystalline substances [1–3]. In particular, a synthesis of 2-isoxazoline-containing mesomorphic compounds was formerly reported [4–7]. 2-Isoxazoline ring is well known to possess a wide synthetic potential. For instance, the reductive opening of the heterocycle in substituted 2-isoxazolines may be used in the synthesis of versatile 1,3-bifunctional compounds [8], and its oxidation gives rise to the corresponding isoxazoles [9–11]. The ring opening in the substituted 2-isoxazolines into β-hydroxyketones was recently utilized in the synthesis of new mesomorphic compounds with a functionally modified side chain [4, 12, 13].

Liquid-crystalline compounds containing an isoxazole ring are also known [14–18]. The interest in this kind mesomorphic substances is due to their practically important physical properties. It should also be noted that isoxazole-containing liquid-crystalline compounds known up till now belong to the 3,5-diarylisoxazoles [14–18], whereas mesomorphic substances from the group of 5-alkyl-3-arylisoxazoles are not documented. The goal of the present study was the synthesis of new liquid-crystalline isoxazoles of the latter series. As the key stage in the synthesis of these compounds was chosen the oxidation of 5-alkyl-3-aryl-2-isoxazolines into the corresponding isoxazoles.

One of the key compounds in the previous syntheses [4, 5] of liquid-crystalline 3-aryl-5-pentyl-2-isoxazolines was 3-(4-hydroxyphenyl)-5-pentyl-2-isoxazoline (**I**). This substance was prepared by generating from the 4-hydroxybenzaldehyde oxime the corresponding nitrile oxide with subsequent 1,3-dipolar cycloaddition to 1-hept-



R = 4-CNC_6H_4 (**a**), 4-FC_6H_4 (**b**), 4'-$C_3H_7OC_6H_4C_6H_4$-4 (**c**), 4'-$C_5H_{11}C_6H_4C_6H_4$-4 (**d**), 3-F-4-$C_3H_7OC_6H_3$ (**e**), 4-C_5H_{11}-cyclo-C_6H_{10} (**f**), 4-$C_3H_7OC_6H_4$ (**g**), 4-$C_4H_9OC_6H_4$ (**h**), 4-$C_5H_{11}OC_6H_4$ (**i**), 4-$C_6H_{13}OC_6H_4$ (**j**), 4-$C_7H_{15}OC_6H_4$ (**k**), 4-$C_8H_{17}OC_6H_4$ (**l**), 4-$C_9H_{19}OC_6H_4$ (**m**), 4-$C_{12}H_{25}OC_6H_4$ (**n**).

ene [5]. We guessed that this compound can also be used as an intermediate in the synthesis of liquid-crystalline 5-alkyl-3-arylisoxazoles.

At conversion of the 2-isoxazoline ring of compound **I** into the isoxazole side processes are presumable consisting in oxidation of the aromatic fragment of the molecule. Therefore in the first stage of the synthesis we carried out a protection of the phenol group. To this end by reaction of phenol **I** with acetyl chloride in a 87% yield was obtained acetate **II**. Its structure is unambiguously proved by the data of IR and ^{1}H NMR spectra. For instance, the ester formation is confirmed by the lack in the IR spectrum of acetate **II** of the absorption band from the stretching vibrations of the hydroxy group in the region 3600–3200 cm^{-1} and the presence of a strong band from vibrations of the C=O bond in the ester moiety at 1765 cm^{-1}. In the ^{1}H NMR spectrum of compound **II** the protons of the acetoxy group give rise to a singlet at δ 2.30 ppm.

Several known preparation methods make it possible to convert the substituted 2-isoxazolines into the corresponding isoxazoles [9–11]. We applied the previously developed method [10] consisting in reaction of 2-isoxazolines with *N*-bromosuccinimide (NBS) followed by dehydrobromination of the arising mixture of 4- and 5-bromo-2-isoxazolines effected by basic reagents to afford isoxazoles. We established that treating compound **II** with *N*-bromosuccinimide and then with triethylamine actually resulted in formation of 3-(4-acetoxyphenyl)-5-pentylisoxazole. However this compound was isolated only in a mixture with the unreacted 4-bromo-2-isoxazoline. Therefore we used for dehydrobromination instead of triethylamine a water-methanol solution of potassium hydroxide. Therewith naturally occurred not only dehydrobromination but also the hydrolysis of the protecting acetoxy group. Thus we obtained as a result isoxazole **III** with a free phenol hydroxy group in a 50% yield.

The structure of compound **III** was proved by the combined data of UV, IR, and ^{1}H NMR spectra. For instance, in the UV spectrum of compound **III** the absorbance of the conjugated system of the phenol and isoxazole rings gives rise to maxima at 260 and 264 nm whereas in the UV spectrum of compound **I** the absorption maximum is observed at 276 nm. In the ^{1}H NMR spectrum of compound **III** the proton of the isoxazole ring C^{4}H appears as a characteristic downfield singlet at δ 6.23 ppm [14–17]. The additional proof of the aromatic heterocycle formation is the considerable downfield shift of the signal from the methylene group protons of the pentyl substituent vicinal to the isoxazole ring as compared to the position of the corresponding proton signal in the ^{1}H NMR spectrum of compound **I**. This proton signal in the spectrum of compound **III** appears as a triplet at δ 2.76 ppm. The signals of aromatic protons in the ^{1}H NMR spectrum of this compound are observed in a weak field as two two-proton doublets characteristic of a 1,4-disubstituted benzene ring. This fact evidences that no transformations of the aromatic system occurred in the course of the reaction. The phenol structure of compound **III** is confirmed by the presence in its IR spectrum of the absorption bands from the stretching vibrations of the OH group at 3590 and 3500–3050 cm^{-1}, and also by the broadened singlet at δ 6.71 ppm in the ^{1}H NMR spectrum of compound **III**.

Based on obtained phenol **III** we further synthesized liquid-crystalline 3-aryl-5-pentylisoxazoles with bridging oximethylene or ester groups. For instance, the reaction of phenol **III** with appropriate benzyl chlorides in the presence of potassium carbonate and sodium iodide afforded benzyl ethers **IVa–IVe** in 76–98% yields. The synthesis of esters was carried out by a direct treatment of compound **III** with carboxylic acids in the presence of *N,N*-dicyclohexylcarbodiimide (DCC) and *N,N*-dimethylaminopyridine (DMAP). The corresponding esters **Va–Vd, Vf–Vn** were obtained in 63–96% yields.

The structure of benzyl ethers **IVa–IVe** and esters **Va–Vd, Vf–Vn** was confirmed by IR and ^{1}H NMR spectra. In the ^{1}H NMR spectra all signals from the fragments of these compounds are present: of the side alkyl chains, of the isoxazole and benzene rings in the central part of the molecule, and also from benzyl and carboxylate moieties (see EXPERIMENTAL)

The study of phase transitions in esters **Va–Vd, Vf–Vn** revealed that the majority of compounds obtained possesses well pronounces mesomorphic properties (see the table).

Attention should be drown to the fact that for benzyl ethers **IVa–IVe** in general smectic mesophases are characteristic. Only compound **IVa** formed an enantiotropic nematic phase. Among the esters **Va–Vd, Vf–Vn** only compounds **Vb–Vd** proved to be non-mesamorphic. Therewith the liquid-crystalline esters unlike the benzyl ethers characteristically form nematic phase. The data of the table also suggest a conclusion that in the homolog series of 4-alkoxybenzoates **Vg–Vn** the growing alkoxy group length leads to decreasing temperature range of the nematic phase and to appearance of the smectic phase *C* in the higher homologs.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Specord 75-IR from chloroform solutions, UV spectra, on a spectrophotometer Specord M40 from ethanol solutions. ^{1}H NMR spectra of solutions in pyridine-d_5 (if not overwise indicated) were registered on a spectrometer Bruker Avance 400 (400 MHz) using HMDS as an internal reference. The melting points and phase transition temperatures were measured on a heating block coupled with a polarization microscope. The mesophase type was estimated by comparison of the observed texture with the corresponding standards given in a monograph [19]. The reaction progress was monitored and the purity of compounds obtained was checked by TLC on Kieselgel 60 F_{254} (Merck) plates.

3-(4-Acetoxyphenyl)-5-pentyl-2-isoxazoline (II). To a solution of 4.9 g (21.0 mmol) of 3-(4-hydroxyphenyl)-5-pentyl-2-isoxazoline (**I**) (prepared by procedure [5]) in 35 ml of toluene and 30 ml of triethylamine was added dropwise 4.5 ml (63.3 mmol) of acetyl chloride. The reaction mixture obtained was stirred for 3 h 15 min and then 100 ml of water was added thereto. The organic layer was separated, the water layer was extracted with 30 ml of toluene. The combined organic solution was washed with 20% sulfuric acid (100 ml) and with a saturated sodium chliride solution (2 × 40 ml). On drying with magnesium sulfate the solvent was distilled off under a reduced pressure. The residue was recrystallized from methanol. Yield 5.1 g (87%), mp 78–80°C (from methanol). IR spectrum, cm^{-1}: 3035, 3025 (C–H_{arom}), 2970, 2945, 2870 (C–H_{alkyl}), 1765 (C=O), 1605, 1510 (C=C_{arom}), 925 (N–O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.89 t (3H, CH_3, *J* 7 Hz), 1.25–1.53 m (6H), 1.55–1.65 m (1H), 1.72–1.82 m (1H) ($CH_{2\ alkyl}$), 2.30 s (3H, CH_3COO), 2.85 d.d (1H, J_1 8, J_2 16.5 Hz), 3.35 d.d (1H, J_1 10.4, J_2 16.5 Hz) (C^4H_2), 4.68–4.76 m (1H, C^5H), 7.11 d (2H, *J* 9 Hz), 7.67 d (2H, *J* 9 Hz) (H_{arom}).

3-(4-Hydroxyphenyl)-5-pentylisoxazole (III). To 4.892 g (17.79 mmol) of 2-isoxazoline **II** and 3.438 g (19.32 mmol) of *N*-bromosuccinimide was added 90 ml of carbon tetrachloride. The reaction mixture obtained was heated at reflux while irradiated with an UV lamp EDRT-240 for 2 h. Then the reaction mixture was cooled, the separated precipitate of siccinimide was filtered off and washed on the filter with 20 ml of carbon tetrachloride. The filtrate was washed with water (2 × 50 ml), and the solvent was distilled off under a reduced pressure. To the residue was added 30 ml of methanol, 30 ml of water, and 3.0 g (53.6 mmol) of potassium hydroxide. The mixture obtained was boiled at stirring for 1 h, then cooled, and 25 ml of diluted (1:4) hydrochloric acid was added. The reaction product was extracted into ethyl acetate (3 × 50 ml). The combined extracts were washed with a saturated sodium chliride solution (4 × 30 ml) and dried over magnesium sulfate. The solvent was removed at reduced pressure, and the residue was crystallized from a mixture of toluene with petroleum ether. After crystallization the substance obtained was additionally purified by column chromato-graphy on silica gel, eluent ethyl acetate–petroleum ether, 3:1 v/v. On distilling off the eluent the residue was recrystallized from the mixture of toluene with petroleum ether. Yield 2.037 g (50%), mp 82°C (from the mixture of toluene with petroleum ether). UV spectrum, λ_{max}, nm: 260, 264. IR spectrum, cm^{-1}: 3590, 3500–3050 (O–H), 3005 (C–H_{arom}), 2960, 2935, 2865 (C–H_{alkyl}), 1605, 1525 (C=C_{arom}), 1260, 1175 (C–O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.90 t (3H, CH_3, *J* 7.0 Hz), 1.32–1.40 m (4H), 1.73 quintet (2H, *J* 7.0 Hz), 2.76 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 6.23 s (1H, C^4H), 6.71 br.s (1H, OH), 6.89 d (2H, *J* 8.8 Hz), 7.64 d (2H, *J* 8.8 Hz) (H_{arom}).

3-(4-Cyanophenylmethoxy)phenyl-5-pentylisoxazole (IVa). To a solution of 0.100 g (0.43 mmol) of phenol **III** and 0.062 g (0.41 mmol) of 4-cyanobenzyl chloride in 10 ml of acetone was added 0.123 g (0.82 mmol) of sodium iodide, and 0.28 g (2 mmol) of potassium carbonate. The reaction mixture was boiled at stirring for 9.5 h. Then 35 ml of water was added, the

Temperature of phase transitions in compounds **IV** and **V**[a]

Compd. no.	mp, °C	Smectic phase	T_{tr}, °C	Nematic phase	T_{cl}, °C
IVa	86	• (*SmA*)	(72.5)	•	102
IVb	9.5	• *SmC* 105.5 *SmA*	–	–	109.5
IVc	187	• *SmA*	–	–	224
IVd	160	• *SmA*	–	–	210
IVe	94	• (*SmC* 90) *SmA*	–	–	102.5
Va	111.5	• (*SmC*)	(106.5)	•	174
Vf	72	• *SmC*	117	•	144
Vg	118.5	–	–	•	149
Vh	110.5	–	–	•	153.5
Vi	114.5	–	–	•	144.5
Vj	104	• (*SmC*)	(95.5)	•	148
Vk	106	• *SmC*	113	•	143.5
Vl	105.5	• *SmC*	114	•	143
Vm	108	• *SmC*	121.5	•	142
Vn	105	• *SmC*	129	•	135

[a] T_{tr} is temperature of transition smectic phase–nematic phase, T_{cl} is the clarification temperature; the phase types and temperature of monotropic transitions are given in parentheses.

separated precipitate was filtered off and washed with water. Yield 0.138 g (97.5%). An analytically pure sample was obtained by double recrystallization from 2-propanol. UV spectrum λ_{max}, nm: 231, 256. IR spectrum, cm^{-1}: 3000 (C–H_{arom}), 2925, 2860 (C–H_{alkyl}), 2225 (C≡N), 1600, 1520 (C=C_{arom}), 1240, 1170 (C–O). ^{1}H NMR spectrum, δ, ppm: 0.81 t (3H, CH_3, *J* 7.0 Hz), 1.18–1.26 m (4H), 1.61 quintet (2H, *J* 7.0 Hz), 2.70 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 5.19 s (2H, Ar'CH_2OAr), 6.62 s (1H, C^{4}H), 7.23 d (2H, *J* 9.0 Hz), 7.57 d (2H, *J* 9.0 Hz), 7.72 d (2H, *J* 9.0 Hz), 8.09 d (2H, *J* 9.0 Hz) (H_{arom}).

Compounds **IVb–IVe** were obtained in a similar way as compound **IVa**.

3-(4-Fluorophenylmethoxy)phenyl-5-pentylisoxazole (IVb). Yield 90%. UV spectrum, λ_{max}, nm: 259. IR spectrum, cm^{-1}: 3000 (C–H_{arom}), 2950, 2925, 2855 (C–H_{alkyl}), 1600, 1500 (C=C_{arom}), 1170, 1150 (C–O). ^{1}H NMR spectrum, δ, ppm: 0.72 t (3H, CH_3, *J* 7.0 Hz), 1.14–1.18 m (4H), 1.53 quintet (2H, *J* 7.0 Hz), 2.61 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 5.02 s (2H, Ar'CH_2OAr), 6.52 s (1H, C^{4}H), 7.10 t (2H, *J* 8.8 Hz), 7.15 d (2H, *J* 8.8 Hz), 7.42 d.d (2H, J_1 5.2, J_2 8.8 Hz), 7.99 d (2H, *J* 8.8 Hz) (H_{arom}).

3-(4'-Propoxy-4-biphenylmethoxy)phenyl-5-pentylisoxazole (IVc). Yield 92%. UV spectrum λ_{max}, nm: 266. IR spectrum, cm^{-1}: 3000 (C–H_{arom}), 2950, 2925, 2860 (C–H_{alkyl}), 1600, 1510, 1495 (C=C_{arom}), 1170 (C–O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.90 t (3H, CH_3, *J* 7.0 Hz), 1.05 t (3H, C_3H_7OAr', *J* 7.0 Hz), 1.32–1.40 m (4H), 1.73 quintet (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 1.82 sextet (2H, C_3H_7OAr', *J* 7.0 Hz), 2.76 t (2H, $CH_{2\ alkyl}$, *J* 7.0 Hz), 3.95 t (2H, C_3H_7OAr', *J* 7.0 Hz), 5.12 s (2H, Ar'CH_2OAr), 6.22 s (1H, C^{4}H), 6.96 d (2H, *J* 9.0 Hz), 7.04 d (2H, *J* 9.0 Hz), 7.47 d (2H, *J* 9.0 Hz), 7.51 d (2H, *J* 9.0 Hz), 7.57 d (2H, *J* 9.0 Hz), 7.72 d (2H, *J* 9.0 Hz) (H_{arom}).

3-(4'-Pentyl-4-biphenylmethoxy)phenyl-5-pentylisoxazole (IVd). Yield 76%. UV spectrum, λ_{max}, nm: 263. IR spectrum, cm^{-1}: 3000 (C–H_{arom}), 2950, 2925, 2850 (C–H_{alkyl}), 1600, 1515 (C=C_{arom}), 1170 (C–O). ^{1}H NMR spectrum, δ, ppm: 0.72 t (3H, C_5H_{11}Ar, *J* 7.0 Hz), 0.75 t (3H, CH_3, *J* 7.0 Hz), 1.10–1.20 m (8H), 1.47–1.56 m (4H), 2.52 t (2H, *J* 7.0 Hz), 2.61 t (2H, *J* 7.0 Hz) (CH_{2alkyl}), 5.13 s (2H, Ar'CH_2OAr), 6.52 s (1H, C^{4}H), 7.20 d (2H, *J* 9.0 Hz), 7.26 d (2H, *J* 9.0 Hz), 7.55 d (2H, *J* 9.0 Hz), 7.62 d (2H, *J* 9.0 Hz), 7.69 d (2H, *J* 9.0 Hz), 8.00 d (2H, *J* 9.0 Hz) (H_{arom}).

3-(3-Fluoro-4-propoxyphenylmethoxy)phenyl-5-pentylisoxazole (IVe). Yield 98%. UV spectrum, λ_{max}, nm: 258. IR spectrum, cm^{-1}: 3000 (C–H_{arom}), 2950, 2925, 2865 (C–H_{alkyl}), 1600, 1505 (C=C_{arom}), 1170, 1115 (C–O). ^{1}H NMR spectrum, δ, ppm: 0.81 t (3H, CH_3, *J* 7.0 Hz), 0.94 t (3H, C_3H_7OAr, *J* 7.0 Hz), 1.19–1.26 m (4H), 1.61 quintet (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 1.72 sextet (2H, C_3H_7OAr, *J* 7.0 Hz), 2.70 t (2H, CH_{2alkyl}, *J* 7.0 Hz), 3.90 t (2H, C_3H_7OAr, *J* 7.0 Hz), 5.11 s (2H, Ar'CH_2OAr), 6.60 s (1H, C^{4}H), 7.08 t (1H, *J* 8.3 Hz), 7.26 d (2H, *J* 9.0 Hz), 7.29 d (1H, *J* 8.3 Hz), 7.42 d.d (1H, J_1 1.9, J_2 12.2 Hz), 8.09 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-cyanobenzoate (Va). To a mixture of 0.060 g (0.41 mmol) of 4-cyanobenzoic acid, 0.085 g (0.37 mmol) of phenol **III**, and 0.085 g (0.41 mmol) of *N,N*-dicyclohexylcarbodiimide in 10 ml of dichloromethane was added a catalytic amount of *N,N*-dimethylaminopyridine. The reaction mixture was stirred for 25 h. The separated precipitate was filtered off through a bed of aluminum oxide, the sorbent was additionally washed with dichloromethane. The combined filtrate was evaporated under a reduced pressure, the residue was recrystallized from 2-propanol. Yield 0.083 g (63%). An analytically pure sample was obtained by double recrystallization from 2-propanol. UV spectrum, λ_{max}, nm: 243. IR spectrum, cm^{-1}: 3000 (C–H_{arom}), 2950, 2925, 2855 (C–H_{alkyl}), 2230 (C≡N), 1730, 1250, 1065 (COOAr), 1600, 1505 (C=C_{arom}). ^{1}H NMR spectrum, δ, ppm: 0.74 t (3H, CH_3, *J* 7.0 Hz), 1.15–1.19 m (4H), 1.53 quintet (2H, *J* 7.0 Hz), 2.63 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 6.57 s (1H, C^{4}H), 7.50 d (2H, *J* 9.0 Hz), 7.80 d (2H, *J* 9.0 Hz), 8.10 d (2H, *J* 9.0 Hz), 8.19 d (2H, *J* 9.0 Hz) (H_{arom}).

Compounds **Vb–Vd, Vf–Vn** were obtained by procedures similar to that used to prepare compound **Va**.

4-(5-Pentylisoxazol-3-yl)phenyl 4-fluorobenzoate (Vb). Yield 71%, mp 126.5°C (from 2-propanol). UV spectrum, λ_{max}, nm: 243. IR spectrum, cm^{-1}: 3000 (C–H_{arom}), 2950, 2925, 2860 (C–H_{alkyl}), 1735, 1255, 1065 (COOAr), 1600, 1500 (C=C_{arom}). ^{1}H NMR spectrum, δ, ppm: 0.73 t (3H, CH_3, *J* 7.0 Hz), 1.15–1.19 m (4H), 1.53 quintet (2H, *J* 7.0 Hz), 2.62 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 6.55 s (1H, C^{4}H), 7.20 t (2H, *J* 8.8 Hz), 7.47 d (2H, *J* 8.8 Hz), 8.08 d (2H, *J* 8.8 Hz), 8.18 d.d (2H, J_1 5.2, J_2 8.8 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4'-propyloxybiphenyl-4-carboxylate (Vc). Yield 66%, mp 180°C (from methyl ethyl ketone). UV spectrum, λ_{max}, nm: 302. IR spectrum, cm^{-1}: 3000 (C–H_{arom}), 2950, 2925, 2865 (C–H_{alkyl}), 1725, 1255, 1060 (COOAr), 1600, 1490 (C=C_{arom}), 1175 (C–O). ^{1}H NMR spectrum δ, ppm: 0.92 t (3H, CH_3, *J* 7.0 Hz), 1.06 t (3H, C_3H_7OAr,

J 7.0 Hz), 1.34–1.42 m (4H), 1.76 quintet (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 1.85 sextet (2H, C_3H_7OAr, *J* 7.0 Hz), 2.79 t (2H, $CH_{2\ alkyl}$, *J* 7.0 Hz), 3.98 t (2H, C_3H_7OAr, *J* 7.0 Hz), 6.29 s (1H, C^4H), 7.00 d (2H, *J* 9.0 Hz), 7.32 d (2H, *J* 9.0 Hz), 7.60 d (2H, *J* 9.0 Hz), 7.70 d (2H, *J* 9.0 Hz), 7.86 d (2H, *J* 9.0 Hz), 8.24 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4'-pentylbiphenyl-4-carboxylate (Vd). Yield 86%, mp 143.5°C (from a mixture of 2-propanol with methyl ethyl ketone). UV spectrum, λ_{max}, nm: 289. IR spectrum, cm^{-1}: 3000 ($C–H_{arom}$), 2950, 2925, 2850 ($C–H_{alkyl}$), 1725, 1255, 1065 (COOAr), 1600, 1510 ($C=C_{arom}$). ^1H NMR spectrum, δ, ppm: 0.74 t (3H, CH_3, *J* 7.0 Hz), 0.77 t (3H, $C_5H_{11}Ar$, *J* 7.0 Hz), 1.12–1.22 m (8H), 1.50–1.59 m (4H), 2.55 t (2H, *J* 7.0 Hz), 2.63 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 6.56 s (1H, C^4H), 7.31 d (2H, *J* 9.0 Hz), 7.50 d (2H, *J* 9.0 Hz), 7.68 d (2H, *J* 9.0 Hz), 7.80 d (2H, *J* 9.0 Hz), 8.10 d (2H, *J* 9.0 Hz), 8.32 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl *trans*-4-pentylcyclohexanecarboxylate (Vf). Yield 78%. UV spectrum, λ_{max}, nm: 245. IR spectrum, cm^{-1}: 2995 ($C–H_{arom}$), 2920, 2850 ($C–H_{alkyl}$), 1740, 1115 (COOAr), 1600, 1505 ($C=C_{arom}$). ^1H NMR spectrum, δ, ppm: 0.73 t (3H, CH_3, *J* 7.0 Hz), 0.81 t (3H, C_5H_{11}, *J* 7.0 Hz), 1.00–1.24 m (15H), 1.44–1.56 m (4H), 1.64–1.72 m (2H), 2.08–2.14 m (2H) ($CH_{2\ alkyl}$), 2.45 t.t (1H, CHCOO, J_1 3.6, J_2 12.0 Hz), 2.61 t (2H, CH_{2alkyl}, *J* 7.0 Hz), 6.53 s (1H, C^4H), 7.36 d (2H, *J* 9.0 Hz), 8.05 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-propyl-oxybenzoate (Vg). Yield 79%. UV spectrum, λ_{max}, nm: 263. IR spectrum, cm^{-1}: 2955, 2925, 2870 ($C–H_{alkyl}$), 1725, 1250, 1060 (COOAr), 1600, 1500 ($C=C_{arom}$), 1165 (C–O). ^1H NMR spectrum, δ, ppm: 0.73 t (3H, CH_3, *J* 7.0 Hz), 0.86 t (3H, C_3H_7OAr, *J* 7.0 Hz), 1.15–1.19 m (4H), 1.53 quintet (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 1.64 sextet (2H, C_3H_7OAr, *J* 7.0 Hz), 2.62 t (2H, $CH_{2\ alkyl}$, *J* 7.0 Hz), 3.82 t (2H, C_3H_7OAr, *J* 7.0 Hz), 6.54 s (1H, C^4H), 7.05 d (2H, *J* 9.0 Hz), 7.46 d (2H, *J* 9.0 Hz), 8.07 d (2H, *J* 9.0 Hz), 8.25 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-butyloxybenzoate (Vh). Yield 68%. UV spectrum, λ_{max}, nm: 264. IR spectrum, cm^{-1}: 2940 ($C–H_{alkyl}$), 1720, 1260, 1060 (COOAr), 1600, 1505 ($C=C_{arom}$), 1165 (C–O). ^1H NMR spectrum, δ, ppm: 0.82 t (3H, CH_3, *J* 7.0 Hz), 0.88 t (3H, C_4H_9OAr, *J* 7.0 Hz), 1.20–1.26 m (4H), 1.41 sextet (2H, *J* 7.0 Hz), 1.62 quintet (2H, *J* 7.0 Hz), 1.70 quintet (2H, *J* 7.0 Hz), 2.70 t (2H, *J* 7.0 Hz) (CH_{2alkyl}), 3.97 t (2H, C_4H_9OAr, *J* 7.0 Hz), 6.63 s (1H, C^4H), 7.15 d (2H, *J* 9.0 Hz), 7.55 d (2H, *J* 9.0 Hz), 8.16 d (2H, *J* 9.0 Hz), 8.34 d (2H, *J* 9.0 Hz), (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-pentyloxybenzoate (Vi). Yield 78.5%. UV spectrum, λ_{max}, nm: 266. IR spectrum, cm^{-1}: 3000 ($C–H_{arom}$), 2950, 2925, 2860 ($C–H_{alkyl}$), 1725, 1250, 1065 (COOAr), 1600, 1500 ($C=C_{arom}$), 1160 (C–O). ^1H NMR spectrum, δ, ppm: 0.73 t (3H, CH_3, *J* 7.0 Hz), 0.77 t (3H, $C_5H_{11}OAr$, *J* 7.0 Hz), 1.10–1.32 m (8H), 1.53 quintet (2H, *J* 7.0 Hz), 1.64 sextet (2H, *J* 7.0 Hz), 2.62 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 3.89 t (2H, $C_5H_{11}OAr$, *J* 7.0 Hz), 6.54 s (1H, C^4H), 7.08 d (2H, *J* 9.0 Hz), 7.46 d (2H, *J* 9.0 Hz), 8.07 d (2H, *J* 9.0 Hz), 8.26 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-hexyloxybenzoate (Vj). Yield 68%. UV spectrum, λ_{max}, nm: 263. IR spectrum, cm^{-1}: 3000 ($C–H_{arom}$), 2950, 2920, 2855 ($C–H_{alkyl}$), 1720, 1245, 1065 (COOAr), 1600, 1500 ($C=C_{arom}$), 1155 (C–O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.91 t (6H, CH_3, *J* 7.0 Hz), 1.32–1.42 m (8H), 1.47 quintet (2H, *J* 7.0 Hz), 1.75 quintet (2H, *J* 7.0 Hz), 1.81 quintet (2H, *J* 7.0 Hz), 2.78 t (2H, *J* 7.0 Hz) (CH_{2alkyl}), 4.04 t (2H, $C_6H_{13}OAr$, *J* 7.0 Hz), 6.28 s (1H, C^4H), 6.97 d (2H, *J* 9.0 Hz), 7.29 d (2H, *J* 9.0 Hz), 7.84 d (2H, *J* 9.0 Hz), 8.14 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-heptyloxybenzoate (Vk). Yield 65%. UV spectrum, λ_{max}, nm: 265. IR spectrum, cm^{-1}: 3000 ($C–H_{arom}$), 2950, 2925, 2855 ($C–H_{alkyl}$), 1725, 1250, 1065 (COOAr), 1600, 1500 ($C=C_{arom}$), 1160 (C–O). ^1H NMR spectrum, δ, ppm: 0.82 t (3H, CH_3, *J* 7.0 Hz), 0.87 t (3H, $C_7H_{15}OAr$, *J* 7.0 Hz), 1.20–1.30 m (10H), 1.40 quintet (2H, *J* 7.0 Hz), 1.62 quintet (2H, *J* 7.0 Hz), 1.75 quintet (2H, *J* 7.0 Hz), 2.70 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 4.01 t (2H, $C_7H_{15}OAr$, *J* 7.0 Hz), 6.62 s (1H, C^4H), 7.18 d (2H, *J* 9.0 Hz), 7.54 d (2H, *J* 9.0 Hz), 8.15 d (2H, *J* 9.0 Hz), 8.35 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-octyloxybenzoate (Vl). Yield 72%. UV spectrum, λ_{max}, nm: 265. IR spectrum, cm^{-1}: 3000 ($C–H_{arom}$), 2925, 2855 ($C–H_{alkyl}$), 1725, 1250, 1065 (COOAr), 1600, 1500 ($C=C_{arom}$), 1160 (C–O). ^1H NMR spectrum, δ, ppm: 0.82 t (3H, CH_3, *J* 7.0 Hz), 0.87 t (3H, $C_8H_{17}OAr$, *J* 7.0 Hz), 1.18–1.30 m (12H), 1.37–1.44 m (2H), 1.58–1.64 m (2H), 1.72–1.79 m (2H), 2.70 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 4.01 t (2H, $C_8H_{17}OAr$, *J* 7.0 Hz), 6.61 s (1H, C^4H), 7.17 d (2H, *J* 9.0 Hz), 7.52 d (2H, *J* 9.0 Hz), 8.14 d (2H, *J* 9.0 Hz), 8.33 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-nonyloxybenzoate (Vm). Yield 85%. UV spectrum, λ_{max}, nm: 264. IR spectrum, cm^{-1}: 2935 ($C–H_{alkyl}$), 1725, 1250, 1065 (COOAr), 1600, 1505 ($C=C_{arom}$), 1165 (C–O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.88 t (3H, CH_3, *J* 7.0 Hz),

0.91 t (3H, $C_9H_{19}OAr$, *J* 7.0 Hz), 1.24–1.40 m (14H), 1.42–1.52 m (2H), 1.75 quintet (2H, *J* 7.0 Hz), 1.81 quintet (2H, *J* 7.0 Hz), 2.78 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 4.04 t (2H, $C_9H_{19}OAr$, *J* 7.0 Hz), 6.27 s (1H, C^4H), 6.96 d (2H, *J* 9.0 Hz), 7.29 d (2H, *J* 9.0 Hz), 7.84 d (2H, *J* 9.0 Hz), 8.13 d (2H, *J* 9.0 Hz) (H_{arom}).

4-(5-Pentylisoxazol-3-yl)phenyl 4-dodecyloxybenzoate (Vn). Yield 96%. UV spectrum, λ_{max}, nm: 263. IR spectrum, cm^{-1}: 3005 (C–H_{arom}), 2915, 2850 (C–H_{alkyl}), 1720, 1250, 1065 (COOAr), 1600, 1500 (C=C_{arom}), 1160 (C–O). ^1H NMR spectrum, δ, ppm: 0.74 t (3H, CH_3, *J* 7.0 Hz), 0.79 t (3H, $C_{12}H_{25}OAr$, *J* 7.0 Hz), 1.15–1.28 m (20H), 1.37 quintet (2H, *J* 7.0 Hz), 1.53 quintet (2H, *J* 7.0 Hz), 1.71 quintet (2H, *J* 7.0 Hz), 2.62 t (2H, *J* 7.0 Hz) ($CH_{2\ alkyl}$), 3.95 t (2H, $C_{12}H_{25}OAr$, *J* 7.0 Hz), 6.54 s (1H, C^4H), 7.10 d (2H, *J* 9.0 Hz), 7.46 d (2H, *J* 9.0 Hz), 8.07 d (2H, *J* 9.0 Hz), 8.27 d (2H, *J* 9.0 Hz) (H_{arom}).

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