

Vanilline Alkanoates in the Synthesis of Hexahydrobenzacridine and Octahydroxanthene Derivatives

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Abstract—Cascade heterocyclization of 1,3-cyclohexanedione and dimedone with 2-naphthylamine and vanilline esters gave derivatives of 2-methoxy-4-(alkyl-11-oxo-7,8,9,10,11,12-hexahydrobenz[*a*]acridin-12-yl)- and 2-methoxy-4-(alkyl-1,8-dioxo-2,3,4,5,6,7,8,9-octahydro-1*H*-xanthen-9-yl)phenyl esters of aliphatic (C₁–C₄) carboxylic acids.

Introduction of natural fragments, such as esters derived from plant phenols, into benzacridines, analogs of natural alkaloids, is a promising synthetic approach to novel biologically active substances. Moreover, acridine derivatives have been used to synthesize labeled conjugates with medicinals, peptides, proteins, and nucleic acids [1–3], that exhibit antitumor and DNA-binding properties.

In the present work we have studied reactions of 1,3-cyclohexadione (**Ia**) and 5,5-dimethyl-1,3-cyclohexadione (dimedone) (**Ib**) with 2-naphthylamine (**II**) and vanillin esters **IIIa–IIIe**. The reactions were performed in ethanol under reflux. At equimolar reagent ratios, 2-methoxy-4-(11-oxo-7,8,9,10,11,12-hexahydrobenz[*a*]acridin-12-yl)phenyl carboxylates **IVa–IVe** (with dione **Ia**) and 2-methoxy-4-(9,9-dimethyl-11-oxo-7,8,9,10,11,12-hexahydrobenz[*a*]acridin-12-yl)phenyl carboxylates **Va–Ve** (with dione **Ib**) were synthesized.

In the three-component mixture **I–II–III**, where each of the reagents can react with the other two, preferred are the reactions **Ia** (or **Ib**) + **II** → **A** and **Ia** (or **Ib**) + **IIIa–IIIe** → **B**. The reactions of enamine **A** with aldehydes **IIIa–IIIe** and of dione **B** with 2-naphthylamine (**II**) provide intermediate **C** whose dehydration gives rise to benz[*a*]acridin-11-ones **IVa–IVe** and **Va–Ve**. The reactions involve formation of a 1,4-dihydropyridine ring that is likely to have an axial aryl substituent and thus acoplanar to the main ring.

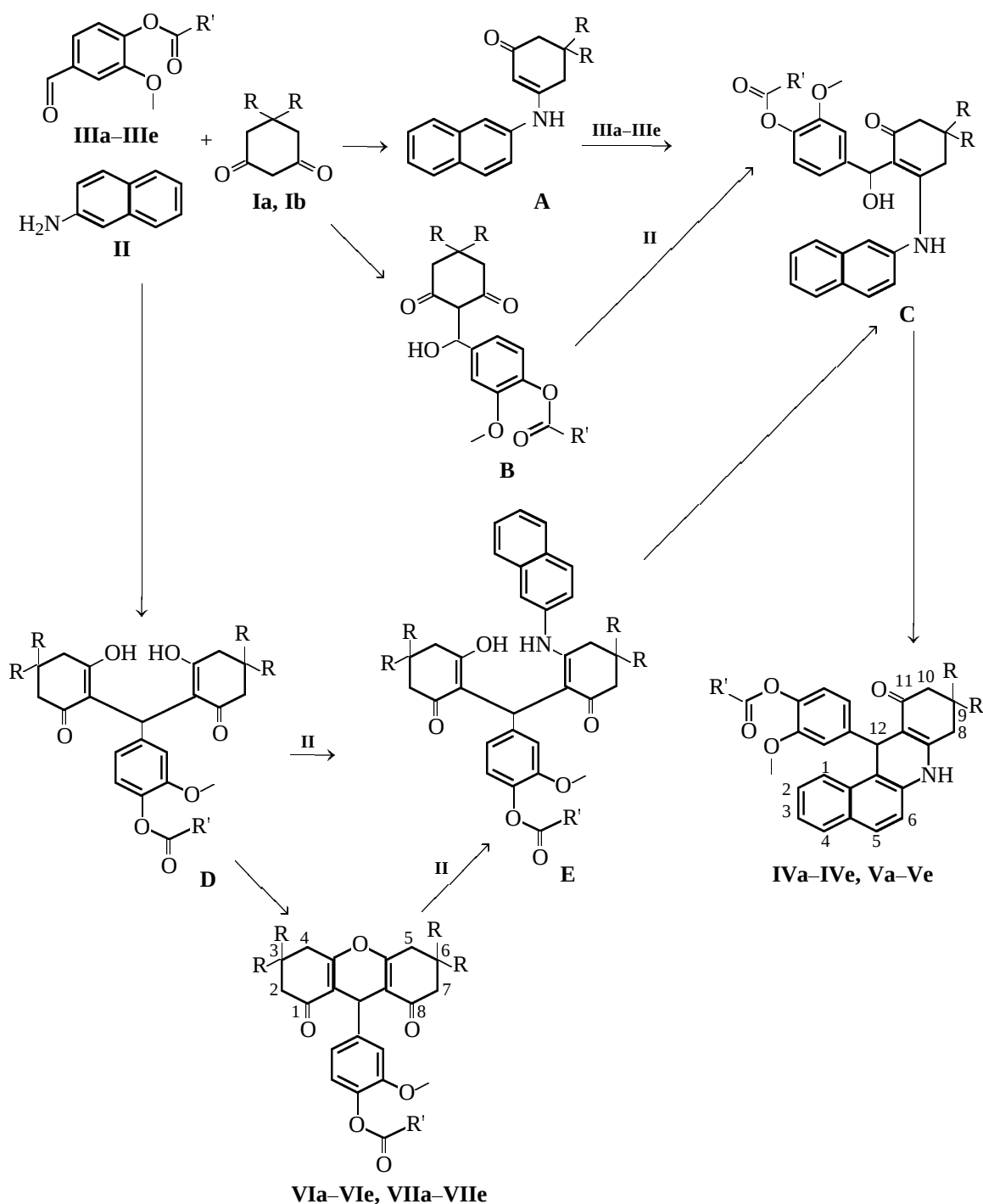
Fused benz[*a*]acrydine derivatives **IVa–IVe** and **Va–Ve** are colorless or light yellow crystals (Tables 1 and 2).

Along with compounds **IVa–IVe** and **Va–Ve**, the reactions gives rise to arylmethylene bisdiketones **D** whose dehydration yields 2-methoxyphenyl 4-(1,8-di-

oxo-2,3,4,5,6,7,8,9-octahydro-1*H*-xanthen-9-yl)- or 4-(3,3,6,6-tetramethyl-1,8-dioxo-2,3,4,5,6,7,8,9-octahydro-1*H*-xanthen-9-yl)carboxylates **VIa–VIe** and **VIIa–VIIe** and the reaction with 2-naphthylamine provides benz[*a*]acridines **IVa–IVe** and **Va–Ve**. Octahydro-1*H*-xanthen-9-yl derivatives **VIa–VIe** and **VIIa–VIIe** that form as by-products in the above reaction were also obtained by refluxing in ethanol of a double excess of diketone **Ia** or **Ib** with aldehydes **IIIa–IIIe** (Tables 1 and 2).

The ¹H NMR spectra of benz[*a*]acridines **IVa–IVe** and **Va–Ve** correspond, in position and multiplicity of aromatic proton signals, to the benz[*a*]acridine structure [4]. The H¹² methine proton of the dihydropyridine ring gives a singlet at 5.88–5.94 ppm. This signal is shifted downfield compared with respective methine proton signals of cyclic compounds [5], which is explained by the anisotropic effect of the neighboring aromatic ring (Table 2). The ¹H NMR spectra of octahydro-1*H*-xanthen-9-yl derivatives **VIa–VIe** and **VIIa–VIIe** (Table 2) are fully supportive of the structure of these compounds.

The IR spectra of benz[*a*]acridines **IVa–IVe** and **Va–Ve** display characteristic stretching and deformation NH vibration bands at 3300 and 1655–1650 cm^{–1}, respectively. In the IR spectra of benz[*a*]acridines **IVa–IVe** and **Va–Ve** and octahydro-1*H*-xanthen-9-yl derivatives **VIa–VIe**, stretching vibrations of the carbonyl group conjugated with the enamine fragment appear at 1615–1610 cm^{–1}. The ester carbonyl gives a strong absorption band at 1640–1630 cm^{–1}. This band is shifted red, which is probably associated with the involvement of the alkoxycarbonyl, amino, and enolized ketone carbonyl groups in intermolecular hydrogen bonding. The C–O–C fragments absorb at 1240–1220 cm^{–1}. Stretching vibrations of alkyl and cyclo-



I, **R** = H (**a**), CH₃ (**b**); **III**, **R'** = CH₃ (**a**), C₂H₅ (**b**), C₃H₇ (**c**), *iso*-C₃H₇ (**d**), C₄H₉ (**e**); **IV**, **VI**, **R** = H, **R'** = CH₃ (**a**), C₂H₅ (**b**), C₃H₇ (**c**), *iso*-C₃H₇ (**d**), C₄H₉ (**e**); **V**, **R** = CH₃, **R'** = CH₃ (**a**), C₂H₅ (**b**), C₃H₇ (**c**), *iso*-C₃H₇ (**d**), C₄H₉ (**e**); **VII**, **R** = CH₃, **R'** = CH₃ (**a**), C₂H₅ (**b**), C₃H₇ (**c**), *iso*-C₃H₇ (**d**), C₄H₉ (**e**).

aliphatic CH bonds appear at 2960–2840 cm⁻¹ and of aromatic CH bonds, at 3130–3030 cm⁻¹.

The mass spectra of compounds **IVa-IVe**, **Va-Ve**, **VIIa-VIIe** contain molecular ion peaks

(*I* 11–18%). The base peak in the spectra belongs to an [M – MeO – R'COOC₆H₃] ion. The spectra of benz[a]acridines **IVa-IVe** and **Va-Ve** all show an *m/z* 192 ion peak (15–38%) formed by elimination from the [M – MeO – R'COOC₆H₃]⁺ ion of

Table 1. Yields, melting points, and elemental analyses of 4-(11-oxo-7,8,9,10,11,12-hexahydrobenz[*a*]acridin-12-yl)-(IVa–IVe)-, 4-(9,9-dimethyl-11-oxo-7,8,9,10,11,12-hexahydrobenz[*a*]acridin-12-yl)-(Va–Ve)-, 4-(1,8-dioxo-2,3,4,5,6,7,8,9-octahydro-1*H*-xanthen-9-yl)-(VIa–VIe)-, and 4-(3,3,6,6-tetramethyl-1,8-dioxo-2,3,4,5,6,7,8,9-octahydro-1*H*-xanthen-9-yl)-(VIIa–VIIe)-2-methoxyphenyl esters of aliphatic (C¹–C⁴) carboxylic acids

Comp. no.	Yield, %	mp, °C ^a	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IVa	75	298	75.63	5.56	3.37	C ₂₆ H ₂₃ NO ₄	75.55	5.57	3.39
IVb	59	270	75.90	5.82	3.29	C ₂₇ H ₂₅ NO ₄	75.88	5.85	3.28
IVc	46	258–260	76.14	6.14	3.19	C ₂₈ H ₂₇ NO ₄	76.19	6.12	3.17
IVd	43	302–304	76.21	6.10	3.17	C ₂₈ H ₂₇ NO ₄	76.19	6.12	3.17
IVe	70	256	76.49	6.38	3.10	C ₂₉ H ₂₉ NO ₄	76.48	6.37	3.07
Va	94	292	76.22	6.15	3.20	C ₂₈ H ₂₇ NO ₄	76.19	6.12	3.17
Vb	96	252–254	76.51	6.42	3.11	C ₂₉ H ₂₉ NO ₄	76.48	6.37	3.07
Vc	95	256	76.80	6.63	3.01	C ₃₀ H ₃₁ NO ₄	76.76	6.61	2.98
Vd	77	248–250	76.78	6.59	2.97	C ₃₀ H ₃₁ NO ₄	76.76	6.61	2.98
Ve	92	234	77.00	6.85	2.91	C ₃₁ H ₃₃ NO ₄	77.02	6.83	2.90
VIa	43	176–177	69.10	5.74	–	C ₂₂ H ₂₂ O ₆	69.11	5.76	–
VIb	46	141–142	69.68	6.03	–	C ₂₃ H ₂₄ O ₆	69.70	6.06	–
VIc	22	182	70.22	6.35	–	C ₂₄ H ₂₆ O ₆	70.24	6.34	–
VI d	15	179	70.19	6.35	–	C ₂₄ H ₂₆ O ₆	70.24	6.34	–
VIe	19	184–185	70.73	6.60	–	C ₂₅ H ₂₈ O ₆	70.75	6.60	–
VIIa	58	141–142	71.21	6.85	–	C ₂₆ H ₃₀ O ₆	71.23	6.84	–
VIIb	31	167–168	71.65	7.10	–	C ₂₇ H ₃₂ O ₆	71.68	7.08	–
VIIc	36	156–158	72.13	7.28	–	C ₂₈ H ₃₄ O ₆	72.10	7.30	–
VII d	45	150	72.08	7.32	–	C ₂₈ H ₃₄ O ₆	72.10	7.30	–
VIIe	29	139	72.48	7.53	–	C ₂₉ H ₃₆ O ₆	72.50	7.50	–

CH₂CH₂CO (IVa–IVe) or (CH₃)₂CCH₂CO (Va–Ve) fragments. The mass spectra of octahydro-1*H*-xanthen-9-yl derivatives VIa–VIe and VIIa–VIIe lack such peaks.

EXPERIMENTAL

The mass spectra were measured on a Finnigan-MAT Incos-50 instrument, ionizing energy 70 eV. The IR spectra were obtained on a Nicolet Protege-460 Fourier spectrometer. The ¹H NMR spectra were taken on Bruker AC-500 (500 MHz) and Tesla BS-567 (100 MHz) spectrometers in DMSO-*d*₆, internal reference TMS.

The melting points were measured in a Kofler hot stage.

Vanillin alkanates IIIa–IIIe. To a solution of 0.2 mol of vanillin in 500 ml of absolute CH₂Cl₂, absolute pyridine, 0.25 mol, and, in small portions with stirring, 0.2 mol of acid chloride prepared by 6-h refluxing of a mixture of 1 mol of carboxylic acid, 1.3 mol of SOCl₂, and 500 ml of absolute benzene, followed by removal of the solvent and distillation of

the residue. The reaction mixture was refluxed for 1 h, CH₂Cl₂ was removed, and the residue was dissolved in 500 ml of benzene. The solution was washed with three portions of water, three portions of 5% aqueous NaHCO₃, and dried with CaCl₂. The solvent was removed, and the residue was distilled in a vacuum or recrystallized from a 1 : 1 benzene–hexane mixture.

Compound IIIa, yield 92%, mp 78–79°C (published data [6]: mp 77–79°C). ¹H NMR spectrum, δ, ppm: 2.32 s (Me), 3.92 s (OMe), 7.18 d, 7.48 m (3H, H_{arom}), 9.92 s (CH).

Compound IIIb, yield 79%, mp 33–34°C. ¹H NMR spectrum, δ, ppm: 1.25 t (Me), 2.52 q (CH₂), 3.84 s (OMe), 7.13 d, 7.42 m (3H, H_{arom}), 9.88 s (CH). Found, %: C 63.29; H 5.73. C₁₁H₁₂O₄. Calculated, %: C 63.46; H 5.77.

Compound IIIc, yield 81%, bp 137–138°C (0.5 mm Hg), *n*_D²⁰ 1.5281. ¹H NMR spectrum, δ, ppm: 1.02 t (Me), 1.63 m (CH₂), 2.51 t (CH₂), 3.84 s (OMe), 7.15 d, 7.40 m (3H, H_{arom}), 9.90 s (CH). Found, %: C 64.71; H 6.32. C₁₂H₁₄O₄. Calculated, %: C 64.86; H 6.31.

Table 2. ^1H NMR spectra of compounds **IVa–IVd–VIIa–VIIe**, δ , ppm (J , Hz)

Comp. no.	OMe, s	CH, s	Cycloaliphatic, R, and NH protons	Aromatic and R' protons
IVa	3.69	5.88	1.90–2.00 m, 2.20–2.30 m, 2.60–2.65 m, 9.70 s (NH)	6.55 d (8.0), 6.73 d (8.2), 7.13 s, 7.29–7.34 m, 7.43 t, 7.73–7.80 m, 7.95 d (7.6), 2.15 s (3H, CH_3)
IVb	3.72	5.90	1.90–2.00 m, 2.10–2.15 m, 2.60–2.65 m, 9.50 s (NH)	6.49 d (7.8), 6.62 d (8.0), 7.08 s, 7.24–7.30 m, 7.38 t, 7.68 d (8.8), 7.71 d (8.6), 7.79 d (7.0), 1.19 t (3H, CH_2CH_3), 1.26 q (2H, CH_2CH_3)
IVc	3.75	5.90	1.90–2.10 m, 2.20–2.28 m, 2.50–2.58 m, 9.48 s (NH)	6.48 d (8.0), 6.65 d (8.7), 7.14 s, 7.22–7.30 m, 7.35–7.40 m, 7.68 d (6.9), 7.73 d (7.2), 7.95 d (7.5), 1.00 t (3H, $\text{CH}_2\text{CH}_2\text{CH}_3$), 1.70 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_3$), 2.65 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_3$)
IVd	3.74	5.79	1.87–1.98 m, 2.15–2.28 m, 2.58–2.63 m, 9.63 s (NH)	6.47 d (8.6), 6.70 d (8.8), 7.16 s, 7.20–7.40 m, 7.70 d (7.6), 7.90 d (7.3), 1.35 d (7.0) [6H, $\text{CH}(\text{CH}_3)_2$], 2.88 m [H, $\text{CH}(\text{CH}_3)_2$]
IVe	3.76	5.94	1.98–2.09 m, 2.20–2.30 m, 2.59–2.64 m, 9.49 s (NH)	6.50 d (8.3), 6.64 d (8.9), 7.10 s, 7.20–7.30 m, 7.35–7.40 m, 7.68–7.75 m, 7.90 d (6.8), 0.96 t (3H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.43 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.65 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 2.45 t (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$)
Va	3.74	5.87	2.15–2.20 m, 2.30–2.49 m, 9.37 s (NH), 0.95 s (3H, CH_3), 1.10 s (3H, CH_3)	6.80 d (8.4), 7.12 d (8.3), 7.18 s, 7.28–7.34 m, 7.40 t, 7.60 d (7.8), 7.90 d (7.9), 2.10 s (3H, CH_3)
Vb	3.70	5.89	2.10–2.123 m, 2.30–2.35 m, 9.37 s (NH), 0.95 s (3H, CH_3), 1.10 s (3H, CH_3)	7.00 d (7.9), 7.14 d (7.3), 7.10 s, 7.30–7.35 m, 7.37 t, 7.68 d (6.7), 7.75 d (7.4), 7.95 d (6.9), 1.17 t (3H, CH_3), 2.50 q (2H, CH_2CH_3)
Vc	3.76	5.88	2.10–2.20 m, 2.25–2.40 m, 9.55 s (NH), 0.98 s (1H, CH_3), 1.10 s (1H, CH_3)	6.60–6.80 m, 7.10–7.30 m, 7.60–7.80 m, 7.90–8.10 m, 1.15 s (3H, $\text{CH}_2\text{CH}_2\text{CH}_3$), 1.76 q (2H, $\text{CH}_2\text{CH}_2\text{CH}_3$), 2.50 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_3$)
Vd	3.69	5.82	2.03–2.09 m, 2.10–2.13 m, 9.58 s (NH), 0.90 s (3H, CH_3), 1.08 s (3H, CH_3)	6.55 d (8.1), 6.60 d (7.3), 7.10 s, 7.29–7.34 m, 7.45–7.49 m, 7.72–7.80 m, 7.90 d (6.8), 1.20 d (6.5) [6H, $\text{CH}(\text{CH}_3)_2$], 2.90 m [1H, $\text{CH}(\text{CH}_3)_2$]
Ve	3.72	5.89	2.15–2.20 m, 2.30–2.35 m, 9.35 s (NH), 0.90 s (3H, CH_3), 1.10 s (3H, CH_3)	6.52 d (6.5), 6.64 d (7.2), 7.10 s, 7.20–7.30 m, 7.35–7.40 m, 7.65 d (6.9), 7.08 d (7.5), 7.90 d (8.3), 0.90 t (3H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.40 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.60 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 2.45 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$)
Vla	3.80	4.65	1.90–2.10 m, 2.24 s, 2.25–2.40 m	6.85 d (8.9), 6.75 d (8.0), 7.00 s, 2.20 s (3H, CH_3)
Vlb	3.82	4.64	1.90–2.10 m, 2.25–2.30 m, 2.65–2.76 m	2.65–2.70 m 6.65 d (9.1), 6.79 d (8.3), 7.00 s, 1.20 t (3H, CH_2CH_3), 2.50 q (2H, CH_2CH_3)
Vlc	3.80	4.63	1.95–2.10 m, 2.26–2.40 m, 2.65–2.7 m	6.67 d (8.6), 6.78 d (8.0), 6.69 s, 1.10 t (3H, $\text{CH}_2\text{CH}_2\text{CH}_3$), 1.75 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_3$), 2.45 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_3$)
Vld	3.75	4.67	1.90–2.15 m, 2.28–2.40 m, 2.60–2.75 m	6.68 d (8.4), 6.70 d (7.6), 6.70 s, 1.25 d (6.4) [6H, $\text{CH}(\text{CH}_3)_2$], 2.85 m [1H, $\text{CH}(\text{CH}_3)_2$]
Vle	3.80	4.64	1.90–2.10 m, 2.30–2.40 m, 2.65–2.70 m	6.65 d (9.0), 6.80 d (7.8), 6.97 s, 0.90 t (3H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.48 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.65 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 2.50 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$)
VIIa	3.76	4.70	2.10–2.20 m, 2.35–2.50 m, 0.90 s (6H, 2CH_3), 1.10 s (6H, 2CH_3)	6.80 d (9.3), 6.85 s, 7.15 d (7.9), 1.25 s (3H, CH_3)

Table 2. (Contd.)

Comp. no.	OMe, s	CH, s	Cycloaliphatic, R, and NH protons	Aromatic and R' protons
VIIb	3.80	4.72	2.15–2.20 m, 2.40–2.60 m, 1.00 s (6H, 2CH ₃), 1.08 s (6H, 2CH ₃)	6.75 d (8.3), 6.80 s, 7.10 d (7.2), 1.22 t (3H, CH ₂ ·CH ₃ , 2.40 q (2H, CH ₂ CH ₃)
VIIc	3.70	4.68	2.20–2.50 m, 1.00 s (6H, 2CH ₃), 1.20 s (6H, 2CH ₃)	6.77 d (8.5), 6.79 d (6.5), 6.90 s, 1.10 t (3H, CH ₂ ·CH ₂ CH ₃), 1.75 q (2H, CH ₂ CH ₂ CH ₃), 2.45 m (2H, CH ₂ CH ₂ CH ₃)
VIIId	3.73	4.55	2.10–2.19 m, 0.90 s (6H, 2CH ₃), 1.10 s (6H, 2CH ₃)	6.70 d (8.4), 6.85–6.92 m, 1.25 d [6H, CH(CH ₃) ₂], 2.70 m [1H, CH(CH ₃) ₂]
VIIe	3.80	4.59	2.15–2.20 m, 1.00 s (6H, 2CH ₃), 1.10 s (6H, 2CH ₃)	6.65 d (7.2), 6.80 d (6.2), 6.94 s, 0.95 t (3H, CH ₂ CH ₂ CH ₂ CH ₃), 1.40 m (2H, CH ₂ CH ₂ CH ₂ CH ₃), 1.65 m (2H, CH ₂ CH ₂ CH ₂ CH ₃), 2.59 m (2H, CH ₂ ·CH ₂ CH ₂ CH ₃)

Compound IIIId, yield 89%, mp 29–30°C. ¹H NMR spectrum, δ, ppm: 1.35 d, 2.88 m (7H, *i*-Pr), 3.90 s (OMe), 7.21 d, 7.50 m (3H, H_{arom}), 9.96 s (CH). Found, %: C 64.72; H 6.19. C₁₂H₁₄O₄. Calculated, %: C 64.86; H 6.31.

Compound IIIe, yield 76%, bp 149–150°C (0.5 mm Hg), *n*_D²⁰ 1.5273. ¹H NMR spectrum, δ, ppm: 0.96 t (Me), 1.20–1.90 m (2CH₂), 2.62 t (CH₂), 3.96 s (OMe), 7.15 d, 7.38 m (3H, H_{arom}), 9.90 s (CH). Found, %: C 66.01; H 6.62. C₁₃H₁₆O₄. Calculated, %: C 66.10; H 6.77.

2-Methoxy-4-(11-oxo-7,8,9,10,11,12-hexahydrobenz[*a*]acridin-12-yl)phenyl esters IVa–IVe of aliphatic (C₁–C₄) carboxylic acids. To a solution of 0.01 mol of 2-naphthylamine (**II**) in 50 ml of ethanol, 0.01 mol of vanillin alkanate **IIIa–IIIe** and 0.01 mol of 1,3-cyclohexanedione (**Ia**) were added, and the mixture was refluxed for 1–3 h (depending on the radical chain length in the vanillin ester). After cooling, crystal formed and were separated, washed with hotmethanol, and recrystallized from benzene.

2-Methoxy-4-(9,9-dimethyl-11-oxo-7,8,9,10,11,12-hexahydrobenz[*a*]acridin-12-yl)phenyl esters Va–Ve of aliphatic (C₁–C₄) carboxylic acids were prepared by the same procedure using dimedone (**Ib**) under reflux for 4–6 h.

2-Methoxy-4-(1,8-dioxo-2,3,4,5,6,7,8,9-octahydro-1*H*-xanthen-9-yl)- (VIa–VIf) and 2-methoxy-4-(3,3,6,6-tetramethyl-1,8-dioxo-2,3,4,5,6,7,8,9-octahydro-1*H*-xanthen-9-yl)-2-methoxyphenyl esters

(VIIa–VIIe) of aliphatic (C₁–C₄) carboxylic acids. To a solution of 0.01 mol of vanillin alkanate **IIIa–IIIe** in 50 ml of ethanol, 0.02 mol of 1,3-cyclohexanedione (**Ia**) or dimedone (**Ib**) was added, and the mixture was refluxed for 2–6 h. Crystals formed and were separated, washed with diethyl ether, dried, and recrystallized from methanol.

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