Chemistry of Heterocyclic Compounds, Vol. 36, No. 4, 2000

PECULIARITIES OF FORMATION OF DECAHYDROACRIDINE-1,8-DIONES ON THE BASIS OF 1,3-DIOXOCYCLOHEXANE COMPOUNDS IN VARIOUS MEDIA

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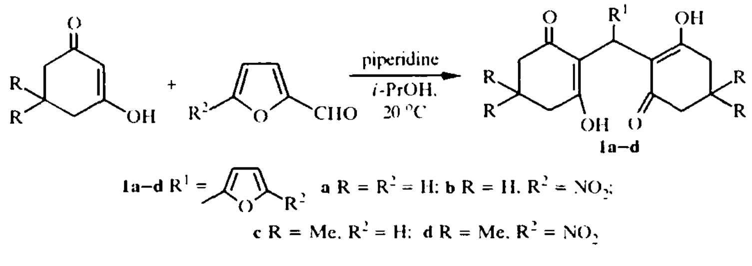
The conditions for the formation of N-substituted decahydroacridine-1,8-diones from α-R-methylenebiscyclohexane-1,3-diones and 5,5-dimethyl-3-(N-methylamino)-2-cyclohexen-1-one in ethanol, isopropanol, and DMSO have been studied. Methods have been developed for preparation of 1,8-dioxo-10-methyldecahydroacridines which contain furyl and 5-nitrofuryl substituents at the 9 position.

Keywords: 1,8-dioxodecahydroacridines, 3-(N-methylamino)-2-cyclohexenones, methylene(arylidene)-biscyclohexane-1,3-diones, 2-cyclohexenones, azacylization, amination.

1,8-Dioxodecahydroacridines contain the pharmacophoric 1,4-dihydropyridine fragment which is a structural unit of the biologically important N-alkylnicotinamides, oxidative-reductive coenzymes NADH and NADPH [1]. In this regard, they represent themselves as a class of potentially physiologically active compounds. As a rule, their preparation involves a number of variants of the Hantzsch synthesis starting from cyclohexane-1,3-diones, α-R-methylenebiscyclohexane-1,3-diones, and 3-amino-2-cyclohexen-1-ones [2-9]. Reaction of the latter with ammonia, aliphatic or aromatic amines occurs upon heating in acetic or formic acid but this limits the use of the methods indicated for the preparation of decahydroacridine-1,8-diones which contain acidophobic furyl substituents. Individual cases of the synthesis of decahydroacridinediones have been reported starting from oxo-1,5-diketones in alcohol solutions in the presence of ammonia under drastic conditions (at 150°C) [7].

In this work we present the results of a search for the optimal conditions for preparing 1,8-dioxodecahydroacridines, including furyl- and 5-nitrofuryl-substituted ones in various media, starting from methylene(arylidene, furfurylidene)biscyclohexane-1,3-diones (**1a-i**) and 5,5-dimethyl-3-(N-methylamino)-2-cyclohexen-1-one (**2a**).



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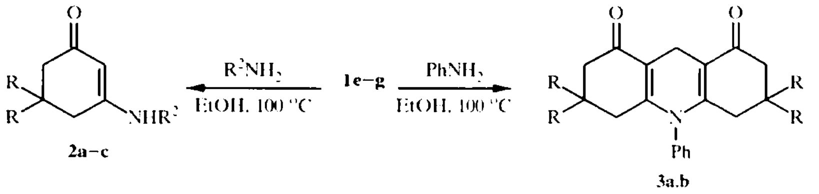
[2] Saratov Higher Military Engineering Chemical Defence School, Saratov 410037, Russia. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 4, pp. 475-481, April, 2000. Original article submitted September 14, 1998; revision submitted February 26, 1999.

0009-3122/00/3604-0403$25.00©2000 KluwerAcademic/Plenum Publishers

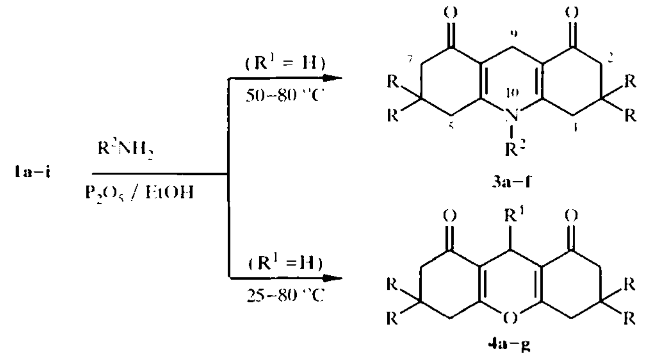
In contrast to methylene- and arylidenebiscyclohexane-1,3-diones, the properties of their furfurylidene analogs **1a-d** have been little studied and methods for their preparation have not been worked out, with the exception of the tetraketone **1c** [10]. We have shown that condensation of cyclohexane-1,3-diones with furan series aldehydes is best carried out in isopropanol medium at 20°C in the presence of catalytic amount of piperidine. Under these conditions 5-R-2-furfurylidenebiscyclohexane-1,3-diones **1a-d** are formed in 76-88% yields (compounds **1b,d** have been synthesized for the first time). Tetraketones **1a-d** are thermally unstable (in particular compounds **1a,c**) and temperature increase at their preparation to 30-50°C causes tarring of the reaction mixture.

Diones **1a-d** exist in the diketodienol form (in agreement with the data of [11]) and this is confirmed by the presence of intense, broad absorption band in the region of 2500-2700 cm^{-1} in their IR spectra, characteristic of the intramolecular hydrogen bond (IHB) between the carbonyl group and its enolized form, and also by the absence of absorption for a nonconjugated carbonyl group at 1700-1720 cm^{-1}. The ^1H NMR spectra of compounds **1a-d** (Table 1) show a broad singlet at 11.96-12.08 ppm with an integrated intensity of two protons and this points to the presence of the OH groups which participate in the formation of IHB.



1 e R = R^1 = H; **f** R = Me, R^1 = H; **g** R = Me, R^1 = C_6H_4-OMe-*p*;
2 a R = R^2 = Me; **b** R = Me, R^2 = CH_2Ph; **c** R = H, R^2 = Me; **3 a** R = H, **b** R = Me

In the amination of tetraketones **1e-g** in alcohol solutions at 100°C under hydrogen atmosphere the factor determining the direction of the reaction is the basicity of amine. Azacyclization occurs only when aniline is used, the target N-phenyldecahydroacridine-1,8-diones **3a,b** being isolated in 45-55% yields. Under the action of strong nucleophiles (methylamine, benzylamine) tetraketones **1e,g** undergo reverse decomposition to the corresponding aldehydes and cyclohexane-1,3-diones.



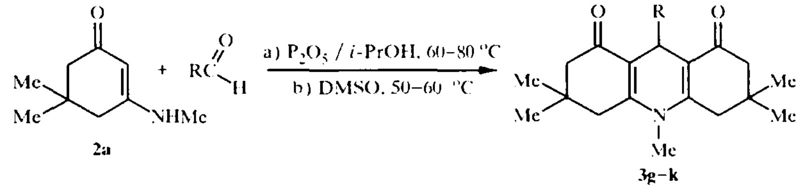
1 h R = H, R^1 = Ph; **i** R = Me, R^1 = Ph; **3c-f** R = Me; **c** R^2 = C_6H_4OH-*o*; **d** R^2 = C_6H_4COOH-*p*; **e** R^2 = CH_2Ph;
f R^2 = NHPh; **4 a** R = H, R^1 = C_4H_3O-2 (2-furyl); **b** R = H, R^1 = 5-$NO_2C_4H_2$O-2; **c** R = Me, R^1 = C_4H_3O-2;
d R = Me, R^1 = 5-$NO_2C_4H_2$O-2; **e** R = H, R^1 = Ph; **f** R = Me, R^1 = Ph; **g** R = Me, R^1 = C_6H_4OMe-*p*

The latter react with amines to give N-methyl(benzyl)-substituted enamino ketones **2a-c** in 38-64% yield. The products of amination of nitrofuryl-substituted tetraketones **1b,d** could not be isolated due to much tarring.

Under mild conditions (25-80°C) and in the presence of P_2O_5 (20% of the mass of the solvent) fulfilling the role of acid catalyst and dehydrating agent the reaction of tetraketones **1a-i** with amines in alcoholic media (absolute ethanol or isopropanol) is determined by the structure of the substrate. Tetraketones **1e,f** without a substituent in the α-methylene unit undergo azacyclization at 50-80°C independently on the strength of the nucleophile (benzylamine, aniline, *o*-hydroxy or *p*-carboxy substituted anilines, or phenylhydrazine) to give the corresponding N-R^2-decahydroacridinediones **3a-f** in high yields (84-94%).

When using in the benzyl(phenyl) amination reaction the tetraketones **1a-d, g-i** (which contain aryl, furyl, or 5-nitrofuryl substituent) a concurrent O-cyclization occurs giving 9-substituted *sym*-octahydroxanthene-1,8-diones **4a-g** in quantitative yields for the whole range of temperatures studied (25-80°C).

Evidently the presence of a substituent in the α-methylene unit of the indicated tetraketones in these conditions enables their rapid tautomeric conversion to the hemiacetal forms that readily undergo dehydration to give the stable octahydroxanthene-1,8-diones **4a-g**. Hence the reaction of α-R-methylenebiscyclohexane-1,3-diones **1a-i** with amines in alcoholic media (basic or acidic) occurs uniquely and is applicable only for the preparation of 1,8-dioxodecahydroacridines unsubstituted in the 9 position.



3 g R = H, **h** R = C_6H_4OMe-*p*, **i** R = 5-$NO_2C_4H_2O$-2, **j** R = C_4H_3O-2

In order to construct 9-R-10-methyldecahydroacridine-1,8-diones we have chosen another route, *viz*. condensation of 5,5-dimethyl-3-(N-methylamino)-2-cyclohexen-1-one (**2a**) with formaldehylde, *p*-methoxybenzaldehyde, furfural, and 5-nitrofurfural in acidic isopropanol solution containing 20% of P_2O_5. The reaction we found to occur at 60-80°C, the target products **3g-j** (both substituted and unsubstituted in the 9 position) being obtained in 51-86% yields.

The lower yield (51%) of 9-(5-nitro-2-furyl)-10-methyldecahydroacridinedione (**3i**) in the case of 5-nitrofurfural and the strong tarring of the reaction mixture when furfural is used (the target product **3j** was not isolated) are due to the instability of the furan ring in acidic media. In order to exclude the use of the latter, the condensation of enamino ketone **2a** with the indicated aldehydes was carried out in dimethyl sulfoxide acting both as solvent and (due to its bipolarity) catalyst. Under these conditions, at 50-60°C 9-R-10-methyldecahydroacridinediones **3g-j** are formed in 40-90% yield. In the case of 5-nitrofurfural tarring occurs leading to a low yield of product **3i** (40%), hence the synthesis of the latter should be carried out in isopropanol over P_2O_5

The structure of the synthesized compounds **3a-j** and **4a-g** was confirmed by IR and chromato-mass spectrometric data and also by comparing the melting points of compounds **3a-c,g, 4c,e-g** with those reported in the literature.

The IR spectra of decahydroacridinediones **3a-j** and the xanthenediones **4a-d** are characterized by the presence of stretching vibrations for the oxovinylene fragment ($\nu_{C=C-C=O}$) which appear as two strong bands at 1585-1608 and 1630-1660 cm^{-1}. The spectrum of compound **3d**, containing *p*-carboxyphenyl substituent on the nitrogen atom, shows absorption bands assigned to stretching vibrations of the carbonyl (1720 cm^{-1}) and hydroxyl (3150-3350 cm^{-1}) groups. Absorption at 3100-3300 (ν_{NH}) and 3030-3080 cm^{-1} ($\nu_{benzene\ ring}$) in the spectrum of compound **3f** confirms the presence of the NHPh group on the nitrogen atom.

The ^1H NMR spectra of tetraketones **1a-d**, xanthenediones **4b,c**, (Table 1), and decahydroacridinediones **3a-j** (Table 2) are in complete agreement with their structure. In the spectra of decahydroacridinediones, the most characteristic signals are those for the protons on the $C_{(9)}$ atom, i. e., methylene protons (compounds **3a-c,e,g**) at 3.23-3.45, methine proton (compounds **3h-j**) at 5.29-5.4 ppm; the chemical shifts for the protons of the N–Me groups at 3.25-3.31 ppm (N-methyldecahydroacridinediones **3g-j**), and the furan ring protons at 7.16 and 6.43 (compound **3i**), and 7.13, 6.12, and 5.97 ppm (compound **3j**).

TABLE 1. ^{1}H NMR Spectra of 5-R-2-Furfurylidenebiscyclohexane-1,3-diones **1a-d** and 9-(5-R^1-2-Furyl)-*sym*-octahydroxanthene-1,8-diones **4c,d**

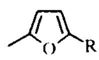
Compound	Chemical shifts, δ, ppm, spin-spin coupling constants (*J*, Hz)				
	CH (1H, s)	CH$_2$	C(CH$_3$)$_2$ (s)		OH (2H, br. s)
1a	5.38	2.06-2.68 (12H, m)	—	7.24 (1H, d, J = 3.8); 6.28 (1H, t); 5.92 (1H, d, J = 3.8)	11.98
1b	5.42	2.13-2.61 (12H, m)	—	7.21 (1H, d , J = 3.8); 6.21 (1H, d, J = 3.8)	11.96
1c	5.38	2.34 (8H, s)	1.19 (12H)	7.26 (1H, d, J = 3.8); 6.29 (1H, t); 5.93 (1H, d, J = 3.8)	12.08
1d	5.45	2.37 (8H, s)	1.16 (12H)	7.24 (1H, d, J = 3.8); 6.20 (1H, d, J = 3.8)	12.04
4c	4.97	2.45 (4H, s), 2.16 (4H, s)	0.93 (6H), 1.03 (6H)	7.24 (1H, d, J = 3.7); 6.25 (1H, t)	—
4d	4.99	2.49 (4H, s), 2.18 (4H, s)	0.91 (6H) 1.09 (6H)	7.20 (1H,d, J = 3.6); 5.99 (1H, d, J = 3.7); 6.58 (1H, d, J = 3.6)	—

TABLE 2. ^{1}H NMR Spectral Characteristics of 9-R^1-10-R^2-Decahydroacridine-1,8-diones

Compound	Chemical shifts, δ, ppm, spin-spin coupling constants (*J*, Hz)				
	2,2-H$_2$;	7,7-H$_2$;	4,4-H$_2$;	5,5-H$_2$ (4H)	9,9-H$_2$/9-H (s)
3a	2.34-2.37 (4H, m)	1.86-1.97 (m)	3.24 (2H)	—	1.56-1.62 (4H, m, 3,3-H$_2$, 6,6-H$_2$); 7.23-7.48 (5H, m, H$_{Ph}$)
3b	2.21 (4H, s)	1.78 (s)	3.23 (2H)	0.93 (12H)	7.19-7.46 (5H, m, H$_{Ph}$)
3c	2.25 (4H, s)	1.83 (s)	3.36 (2H)	0.87 (6H), 0.93 (6H)	6.96-7.10 (4H, m, H$_{Ar}$); 9.50 (1H, br. s, OH)
3e	2.30 (4H, s)	2.17 (s)	3.45 (2H)	1.00 (6H), 1.05 (6H)	7.26-7.23 (5H, m, H$_{Ph}$); 4.65 (2H, s, NCH$_2$)
3g	2.38 (4H, s)	2.26 (s)	3.35 (2H)	1.08 (12H)	3.25 (3H, s, NMe)
3h	2.41 (2H, s), 2.51 (2H, s)	2.20 (s)	5.29 (1H)	1.05 (12H)	6.70 (2H, d, J = 8.5, 2'-, 6'-H); 7.13 (2H, d, J = 8.5, 3'-, 5'-H); 3.25 (3H, s, NMe); 3.70 (3H, s, OMe)
3i	2.45 (2H, s), 2.57 (2H, s)	2.26 (s)	5.47 (1H)	1.10 (12H)	7.15 (1H, d, J = 3.6, H$_{Het}$); 6.43 (1H, d, J = 3.6, H$_{Het}$); 3.31 (3H, s, NMe)
3j	2.41 (2H, s), 2.52 (2H, s)	2.25 (s)	5.42 (1H)	1.07 (12H)	7.13 (1H, d, J = 3.6,H$_{Het}$); 6.12 (1H, t, H$_{Het}$); 5.97 (1H, d, J = 3.6, H$_{Het}$); 3.25 (3H, s, NMe)

TABLE 3. Characteristics of the Synthesized Compounds

Compound	Empirical formula	Found, % / Calculated, % C	H	N	mp, °C (Lit mp, °C)	Yield, %
1a	$C_{17}H_{18}O_5$	$\frac{67.46}{67.54}$	$\frac{5.70}{5.96}$	—	144-146 (144-147 [13])	76
1b	$C_{17}H_{17}NO_7$	$\frac{58.48}{58.79}$	$\frac{4.72}{4.90}$	$\frac{3.85}{4.03}$	169-171	85
1c	$C_{21}H_{26}O_5$	$\frac{70.89}{70.39}$	$\frac{7.70}{7.26}$	—	155-157 (155-157 [10])	76
1d	$C_{21}H_{25}NO_7$	$\frac{62.53}{62.53}$	$\frac{6.48}{6.20}$	$\frac{4.00}{3.97}$	164-166	88
3a	$C_{19}H_{19}NO_2$	$\frac{77.35}{77.81}$	$\frac{6.69}{6.48}$	$\frac{4.36}{4.78}$	259-261 (260 [14])	45 (92)*
3b	$C_{23}H_{27}NO_2$	$\frac{79.32}{79.08}$	$\frac{7.83}{7.74}$	$\frac{4.33}{4.01}$	289-291 (289-291 [15])	55 (94)*
3c	$C_{23}H_{27}NO_3$	$\frac{75.44}{75.62}$	$\frac{7.40}{7.40}$	$\frac{3.42}{3.84}$	277-279 (275-278 [16])	90
3d	$C_{24}H_{27}NO_4$	$\frac{73.45}{73.28}$	$\frac{7.12}{6.87}$	$\frac{3.56}{3.56}$	307-309	98
3e	$C_{24}H_{29}NO_2$	$\frac{79.64}{79.34}$	$\frac{8.44}{7.99}$	$\frac{3.68}{3.86}$	168-170	93
3f	$C_{23}H_{28}N_2O_2$	$\frac{75.57}{75.82}$	$\frac{7.80}{7.69}$	$\frac{7.63}{7.69}$	Above 350	89
3g	$C_{18}H_{25}NO_2$	$\frac{75.39}{75.26}$	$\frac{8.58}{8.71}$	$\frac{5.02}{4.88}$	247-249 (247-249 [2])	86 (90)*^{2}
3h	$C_{25}H_{31}NO_3$	$\frac{76.12}{76.34}$	$\frac{7.72}{7.89}$	$\frac{3.57}{3.56}$	211-213	84 (78)*^{2}
3i	$C_{22}H_{26}N_2O_5$	$\frac{66.25}{66.39}$	$\frac{6.47}{6.53}$	$\frac{7.00}{7.04}$	231-233	51 (40)*^{2}
3j	$C_{22}H_{27}NO_3$	$\frac{74.61}{74.79}$	$\frac{7.38}{7.65}$	$\frac{3.99}{3.97}$	227-229	— (61)*^{2}
4a	$C_{17}H_{16}O_4$	$\frac{71.68}{71.83}$	$\frac{5.94}{5.63}$	—	189-191	99
4b	$C_{17}H_{15}NO_6$	$\frac{61.58}{62.01}$	$\frac{4.92}{4.56}$	$\frac{4.75}{4.26}$	237-239	99
4c	$C_{21}H_{24}O_4$	$\frac{74.17}{74.12}$	$\frac{7.19}{7.06}$	—	160-162 (160-161 [17])	99
4d	$C_{21}H_{23}NO_6$	$\frac{65.44}{65.45}$	$\frac{6.32}{6.02}$	$\frac{3.91}{3.65}$	148-150	99

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* Yields of products obtained in alcohol over P_2O_5.

*^{2} Yields of products obtained in DMSO.

The chromatographic mass spectra of octahydroxanthenediones **4d,g** and decahydroacridinediones **3h-j** give evidence on their purity and contain molecular ion peaks, m/z (M^+): **4d** 385, **4g** 380, **3h** 393, **3i** 398, and **3j** 353. The mass spectra of acridinediones **3h-j** show an intense peak at m/z 286 (M^+-Ar, M^+-5-R-$C_4H_2O_2$) which are typical of 1,4-dihydropyridines substituted at position 4 [1, 12].

Hence a study of the reaction of α-R-methylenebiscyclohexane-1,3-diones with amines and the condensation of 5,5-dimethyl-3-(N-methylamino)-2-cyclohexen-1-one with aldehydes in various solvents has allowed us to establish the conditions for formation of N-substituted 1,8-dioxodecahydroacridines and to develop methods for preparation of derivatives of the latter containing furyl or 5-nitrofuryl substituent in the 9 position.

EXPERIMENTAL

IR spectra were recorded on a Specord M-80 instrument for suspensions in vaseline oil and in hexachlorobutadiene. ^1H NMR spectra were taken on a Varian FT-80 spectrometer in $CDCl_3$ and with TMS as internal standard and chromatographic mass spectra were recorded on an HP 5890A gas chromatograph utilizing an HP 5972A mass-selective detector, 30 m x 0.25 mm column containing 5% of methylphenylsilicone, nitrogen carrier gas, and electron ionization energy of 70 eV.

Monitoring of the course of the reaction and the purity of the compounds obtained was carried out using TLC on Silufol UV-254 plates with hexane–acetone–chloroform eluent (3: 1: 1) and visualization with iodine vapor.

Tetraketones **1e-g** were prepared by known methods [9-11, 17]; enamino ketone **5** was synthesized as described in [18].

5,5-R$_2$-2-Furfurylidenebiscyclohexane-1,3-diones (1a-d). Piperidine (0.5 ml) was added to solution of cyclohexane-1,3-dione or dimedone (0.1 mol) and furfural or 5-nitrofurfural (0.05 mol) in isopropanol (100 ml). The reaction mixture was stirred for 4 h at 20°C, cooled, and the precipitated products **1a-d** were filtered off and recrystallized from 5:1 mixture of hexane and acetone.

N-Phenyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione (3a). Tetraketone **1e** (2.36 g, 0.01 mol), ethanol (100 ml), and aniline (0.93 g, 0.01 mol) were placed in a steel, rotating autoclave of 150 ml volume. The process was carried out over 6 h at 100°C with hydrogen pressure of 7 MPa. Solvent was then evaporated and the residue was dissolved in chloroform (10 ml) and acetone (40 ml) added. The solid product **3a** was filtered off and recrystallized from ethanol.

In the same way tetraketone **1f** gave decahydroacridine **3b**, tetraketones **1e-g** (in the presence of fivefold excess of methylamine) gave 3-(N-methylamino)-2-cyclohexenones **2a-c**, and tetraketone **1g** (in the presence of equimolar amount of benzylamine) gave 3-(N-benzylamino)-2-cyclohexenone **2b**. The products **2a-c** were recrystallized from 1:1 mixture of hexane and acetone. Compound **1f** gave 38% and compound **1g** gave 64% of product **2a**; mp 149-151°C (lit. mp 148-150°C [18]). Compound **1e** gave 40% of product **2c**; mp 160-162°C (lit. mp 165°C [19]). Compound **1g** gave 41% of product **2b**; mp 128-130°C (lit. mp 124-127°C [20]).

3,3,6,6-Tetramethyl-N-benzyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione (3e). Tetraketone **1f** (2.92 g, 0.01 mol) was added to solution of P_2O_5 (8 g) in absolute isopropanol (or ethanol) (40 ml). The mixture was heated to complete dissolution of tetraketone and then benzylamine (1.07 g, 0.01 mol) was poured in. The reaction mixture was refluxed with a condenser for 10-12 h and then poured into cold water (200 ml). After 12 h, the precipitated product **3e** was filtered off, washed with ether or hexane (for removal of an admixture of the starting tetraketone **1f**), and recrystallized from isopropanol.

Similarly, tetraketone **1f** and aniline, *p*-aminobenzoic acid, *o*-aminophenol, or phenylhydrazine gave decahydroacridinediones **3b-d,f** respectively, tetraketone **1e** and aniline gave decahydroacridinedione **3a**, and 5,5-dimethyl-3-(N-methylamino)-2-cyclohexen-1-one **2a** and formaldehyde, *p*-methoxybenzaldehyde, or 5-nitrofurfural gave decahydroacridinediones **3g-i** respectively. Under the same conditions, the tetraketones **1a-d,g,h** in the presence of aniline (or benzylamine) gave the corresponding *sym*-1,2,3,4,5,6,7,8-octahydroxanthenes **4a-g** (of which compounds **4c,e-g** have been reported previously) and their determined melting points agreed with those given in the literature [17, 21].

9-(2-Furyl)-3,3,6,6,10-pentamethyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-diones (3j). Furfural (0.96 g, 0.01 mol) was added to solution of enamino ketone **2a** (3.06 g, 0.02 mol) in DMSO (20 ml). The reaction mixture was held at 50-60°C for 12 h and then poured into cold 5:1 mixture of isopropanol and water. After 12h the precipitated solid product **3j** was filtered off and recrystallized from isopropanol.

Similarly, compound **2a** and 5-nitrofurfural, formaldehyde or p-methoxybenzaldehyde gave the corresponding decahydroacridinediones **3g-i**.

Characteristics of the synthesized compounds **1a-d, 3a-j, and 4a-d** are given in Table 3.

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