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Synthesis of 9-Arylhexahydroacridine-1,8-diones Using Phosphate Fertilizers as Heterogeneous Catalysts

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Abstract—A new strategy has been proposed for the synthesis of 9-arylhexahydroacridine-1,8-diones by three-component condensation of aromatic aldehydes with 5,5-dimethylcyclohexane-1,3-dione and ammonium acetate in ethanol, using nontoxic, available, and inexpensive phosphate fertilizers [monoammonium phosphate (MAP), diammonium phosphate (DAP), and triple super phosphate (TSP)] as heterogeneous catalysts. Notable advantages of the proposed method include operational simplicity, low cost of the reactants, favorable environmental profile, good yield, short reaction time, recovery and reuse of the catalyst, and valorization of Moroccan natural phosphate.

Keywords: heterogeneous catalysts, phosphate fertilizers, MAP, DAP, TSP, 9-arylhexahydroacridine-1,8-diones.

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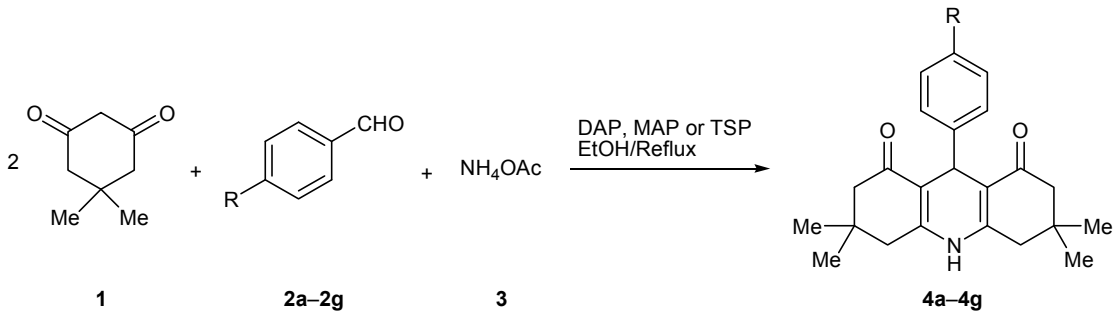
Recently, the development of clean synthetic procedures, which enter into the framework of sustainable chemistry, has become one of the main objectives of organic synthesis in order to reduce or eliminate the use of toxic substances that are harmful to the environment [1]. Undoubtedly, multicomponent reactions are therefore part of sustainable chemistry and constitute a novel way of ideal organic synthesis, since complex structures are rapidly obtained from very simple substrates involving simple synthetic operations and environmentally safe processes, such as the use of eco-friendly solvents and reusable heterogeneous catalysts. This makes these reactions environmentally benign [2–3]. Among the three-component reactions, one can cite the synthesis of acridine-1,8-diones that form an important class of heterocyclic compounds due to their applications in various fields such as biology, pharmacology, and industry [4]. Structural modifications of these heterocycles made it possible to develop products with increased activity and expanded spectrum of therapeutic action. Indeed, acridines are active against malaria [5], cancer [6], and leishmania [7], and they bind to photo-damaged DNA [8]. They also exhibit cytotoxicity [9], block potassium channels [10], show anti-multidrug-resistant [11], antitumor [12], and fungicidal activity [13], and act as potassium channel openers in cardiovascular diseases [14] and antimicrobial agents [15]. In addition, they are also used as laser dyes and possess many important photophysical and electrochemical properties [16].

The importance and usefulness of acridines have led to the development of numerous methods of their synthesis. In general, they are synthesized by the three-component Hantzsch condensation of aldehydes with β-diketones and ammonium acetate or appropriate primary amine on heating in organic solvents [17], under microwave irradiation [18], in ionic liquids [19–20], or using various catalysts such as $FeCl_3/SiO_2$ [21], $In(OTf)_3$ [22], cetyl(trimethyl)ammonium bromide (CTAB) [23], $CeCl_3 \cdot 7H_2O$ [24], tetrabutylammonium hexatungstate [25], nano-Fe_3O_4 [26], nano-ZnO [27], ceric ammonium nitrate (CAN) [28], $InCl_3$ [29], MCM-41-SO_3H [30], oxalic acid [31], $[MIMPS]_3$-$PW_{12}O_{40}$ [32], TsOH [33], and nanorod vanadatesulfuric acid (VSA NRs) [4].

Each of these methods has its own advantages, but some of them suffer from several disadvantages, such as the use of costly catalysts, which requires a lot of

Scheme 1.



R = H (**a**), Me (**b**), Me_2N (**c**), O_2N (**d**), Br (**e**), MeO (**f**), Cl (**g**).

time and money for their preparation, non-reusable toxic solvents, severe operating condition, low yields, and long reaction times. Consequently, effective, inexpensive, available, nontoxic, and recyclable catalysts remain needed for the preparation of 9-arylhexahydroacridine-1,8-diones with excellent yield in short reaction time under green operating conditions to save cost and energy while respecting the environment. This work was aimed at developing a procedure for the synthesis of 9-arylhexahydroacridine-1,8-diones via multicomponent reaction between aromatic aldehydes, dimedone, and ammonium acetate using phosphate fertilizers, monoammonium phosphate (MAP), diammonium phosphate (DAP), and triple super phosphate (TSP) [34–36], as heterogeneous catalysts (Scheme 1). The efficiency of these catalysts has been demonstrated by studying different reaction parameters such as time, solvent nature, and the amount of catalyst.

When the reaction was carried out without a catalyst, no acridine was formed after 60 min. Increase of the reaction time to 8 h led to the formation of the desired product in a low yield (40%). In the presence of a phosphate catalyst, it was possible to obtain the desired product with an excellent yield (94%) after only 60 min, which proved the effectiveness of the three catalysts used (Table 1).

In order to find the optimum conditions, the reaction time was shortened from 60 to 10 min in 10-min steps. The obtained results are presented in Fig. 1. It is seen that the yield increases with time reaching (94%) after 60 min in presence of each catalyst (MAP, DAP, or TSP). The influence of the amount of the catalyst was studied by varying it from 0.002 to 0.01 g in a step of 002 g, other conditions being equal (reaction time 60 min; Table 2). The optimal amounts of the catalysts were 0.01 (8.5 mol %), 0.008 (6.1 mol %), and 0.004 g (1.7 mol %) of MAP, DAP, and TSP, respectively. Further increase of the catalyst amount led to the reduction of the yield, which may be related to the insufficiency of the catalyst surface to adsorb the reagent.

Then, the effect of protic polar (ethanol), aprotic polar (acetonitrile), and nonpolar solvents (dioxane) on the yield of 9-arylhexahydroacridine-1,8-diones was examined. The reaction was also carried out under solvent-free conditions. The results are summarized in Table 3. Ethanol gave high yields, and it appeared to be the most appropriate solvent for this reaction. Ethanol has a high dipole moment and a relatively high

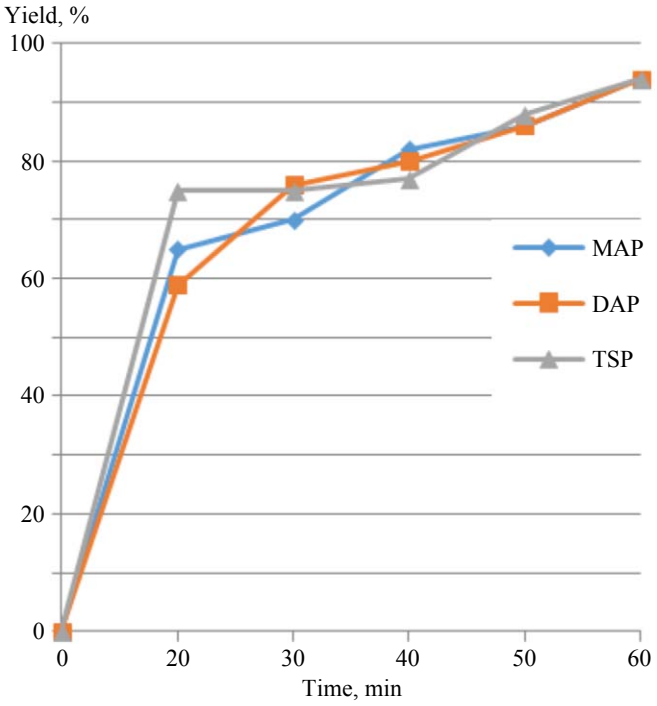


Fig. 1. Yields of 9-phenyl-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**4a**) in the reaction of dimedone (**1**, 2 mmol) with benzaldehyde (**2a**, 1 mmol), and ammonium acetate (1 mmol) in the presence of MAP, DAP, and TSP (0.01 g) in 3 mL of ethanol (reflux) versus reaction time.

Table 1. Synthesis of 9-phenyl-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**4a**) in the presence of MAP, DAP, and TSP[a]

Catalyst	Amount of catalyst, g/mol %	Time, min	Yield,[b] g/%
–	0	480	0.139/40
MAP	0.01/8.5	60	0.328/94
DAP	0.01/7.6	60	0.328/94
TSP	0.01/4.3	60	0.328/94

[a] Reaction conditions: 2 mmol of dimedone, 1 mmol of ammonium acetate, 1 mmol of benzaldehyde, 0.01 g of MAP, DAP, or TSP, 3 mL of ethanol, reflux.
[b] Isolated yield.

Table 2. Synthesis of 9-phenyl-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**4a**) in the presence of different amounts of MAP, DAP, and TSP[a]

Amount of catalyst, g/mol %			Yield,[b] g/%		
MAP	DAP	TSP	MAP	DAP	TSP
0.002/1.7	0.002/1.5	0.002/0.8	0.283/81	0.286/82	0.317/91
0.004/3.3	0.004/3.1	0.004/1.7	0.286/82	0.286/82	0.335/96
0.006/5.2	0.006/4.6	0.006/2.6	0.297/85	0.324/93	0.331/95
0.008/6.9	0.008/6.1	0.008/3.4	0.307/88	0.342/98	0.328/94
0.01/8.5	0.01/7.6	0.01/4.3	0.328/94	0.328/94	0.328/94

[a] Reaction conditions: 2 mmol of dimedone, 1 mmol of ammonium acetate, 1 mmol of benzaldehyde, 3 mL of ethanol; reflux, 60 min.
[b] Isolated yield.

Table 3. Synthesis of 9-phenyl-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**4a**) in different solvents

Solvent	Yield,[b] g/%		
	MAP	DAP	TSP
EtOH	0.328/94	0.342/98	0.335/96
MeOH	0.310/89	0.314/90	0.300/86
MeCN	0.293/84	0.272/78	0.244/70
THF	0.289/83	0.265/76	0.251/72
Dioxane	0.240/69	0.219/63	0.226/65
Toluene	0.219/63	0.216/62	0.230/66
No solvent	0.303/87	0.275/79	0.282/81

[a] Reaction conditions: 2 mmol of dimedone, 1 mmol of ammonium acetate, 1 mmol of benzaldehyde, 0.01 g of MAP, 0.008 g of DAP, and 0.004 g of TSP, 3 mL of solvent; reflux, 60 min.
[b] Isolated yield.

dielectric constant which allow it to separate charges and thus favor interactions between the active sites of the reagent, so the products can be easily formed. Although methanol is a more polar solvent, ethanol remains the best one. The yields obtained in these two solvents allowed us to conclude that the polarity is the main factor; in addition, protic character of the solvent also plays an important role in proton exchange. The reaction in nonpolar solvents (dioxane, toluene) gave the final product in lower yields than in polar solvents. Thus, the synthesis of 9-arylhexahydroacridine-1,8-dione was found to be favored in high-polarity solvents, more precisely polar protic solvents.

An important factor of the catalytic efficiency is the ability of the catalyst to be reused several times. Figure 2 represents the yields after each cycle of the

model reaction. It was found that the yields decreased insignificantly after four cycles; thus the catalysts can be reused at least four times without appreciable loss of their activity.

Subsequently, a number of 9-aryl-acridine-1,8-dione derivatives **4a**–**4g** with various substituents in the aryl ring were prepared from the corresponding aldehydes, dimedone, and ammonium acetate under the optimized conditions (Table 4). All compounds **4a**–**4g** were reported previously, and their melting points were consistent with published data. In the presence of MAP as a catalyst, high yields (94, 96, and 97%) were obtained for aldehydes **2a**, **2b**, and **2e** (R = H, Me, Br), respectively. The best yields (98, 98, and 95%) were achieved in the reactions with **2a**, **2b**, and **2c** (R = Me_2N), respectively, catalyzed by DAP. The third catalyst, TSP, ensured the best yields (96 and 92%) with unsubstituted benzaldehyde (**2a**) and 4-methylbenzaldehyde (**2b**), respectively. Thus, the catalytic activity depended on the structures of both catalyst and initial aldehyde, as well as on the nature of the catalyst surface. In fact, these parameters are responsible for the interactions which take place in the catalyst surface–reagent interface.

Scheme 2 shows a plausible mechanism for the formation of 9-arylhexahydroacridine-1,8-diones in the presence of MAP. The reaction begins with protonation of the carbonyl group in aldehyde **2** by the action of MAP, followed by condensation with dimedone molecule to produce intermediate **6** which loses water molecule to form **7**. Next, enaminone **5** formed by the reaction of dimedone with ammonium acetate adds to the =CH carbon atom of **7** to give intermediate **8**. Finally, intramolecular cyclization via nucleophilic attack of the amino group on the carbonyl carbon atom and removal of water molecule lead to the formation of acridine-1,8-dione **4**. This mechanism is valid for the three catalysts used.

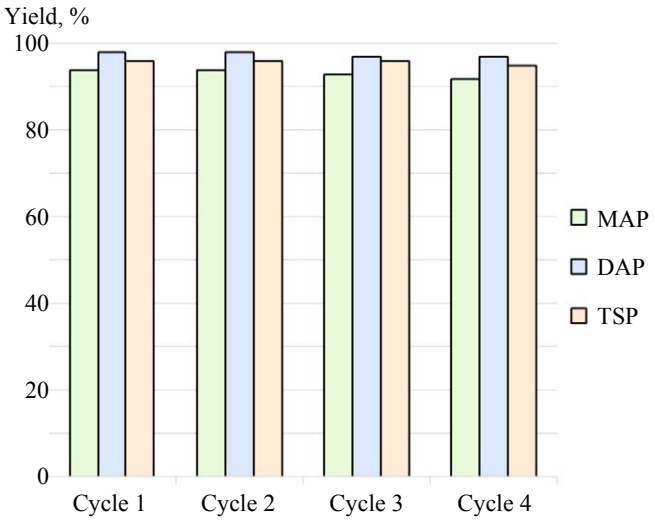


Fig. 2. Reusability of MAP, DAP, and TSP catalysts in the model reaction.

The catalytic activity of MAP, DAP, and TSP was compared with the activity of other catalysts used previously for the same reaction (Table 5). The obtained data indicate that MAP, DAP, and TSP catalysts give better yields and shorter reaction times than the other reported catalysts.

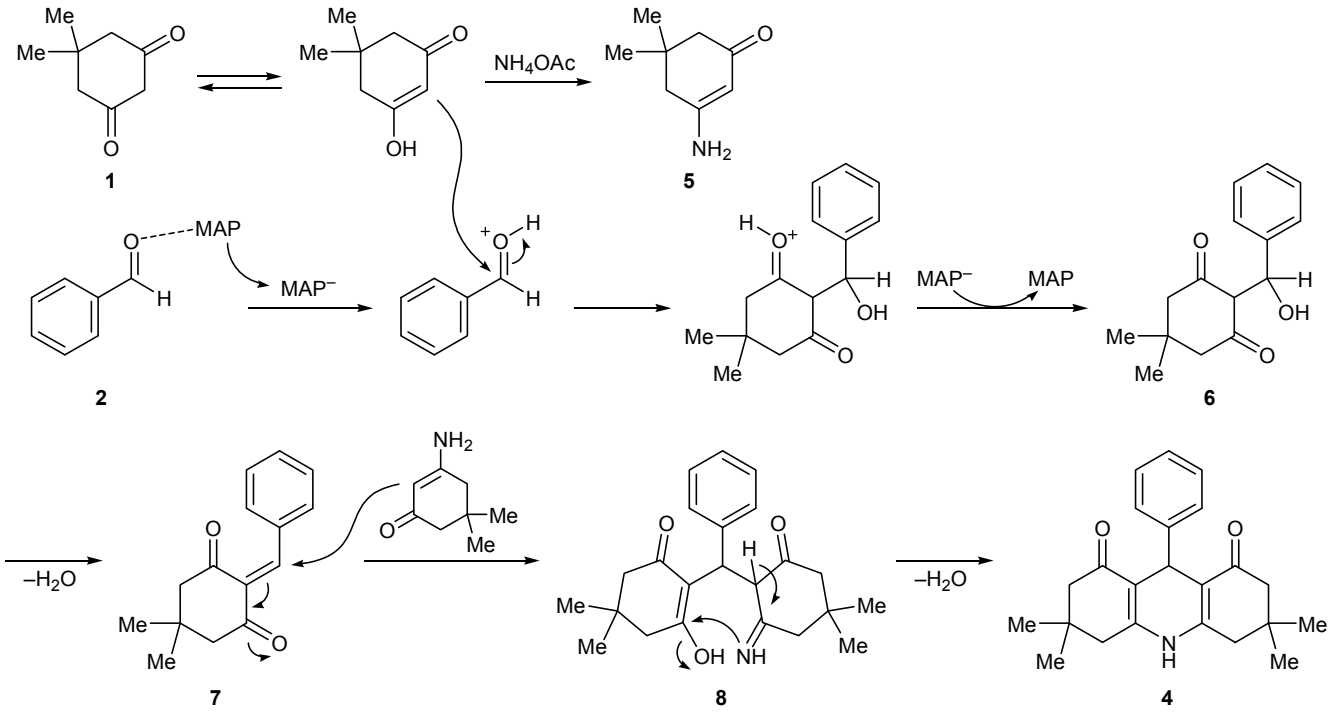
To sum up, the use of MAP, DAP, and TSP phosphate fertilizers as heterogeneous catalysts provide a very effective method for the synthesis of 9-arylacridine-1,8-diones with very good yields and in shorter reaction times. Furthermore, these catalysts

Table 4. Synthesis of acridines **4a**–**4g** under the optimized conditions[a]

Compound no.	R	Yield,[b] g/%			melting point, °C	
		MAP	DAP	TSP	found	reported
4a	H	0.328/94	0.342/98	0.335/96	258–260	258–260 [37]
4b	4-Me	0.348/96	0.356/98	0.334/92	>260	318–320 [24]
4c	4-Me_2N	0.337/86	0.372/95	0.349/89	>260	280–282 [37]
4d	4-O_2N	0.347/88	0.351/89	0.309/88	>260	287–290 [37]
4e	4-Br	0.414/97	0.325/76	0.325/76	>260	312–315 [24]
4f	4-MeO	0.307/81	0.235/62	0.273/72	>260	269–271 [37]
4g	4-Cl	0.329/86	0.147/52	0.276/72	>260	300–302 [37]

[a] Reaction conditions: 2 mmol of dimedone, 1 mmol of ammonium acetate, 1 mmol of aldehyde **2a**–**2g**, 0.01 g of MAP, 0.008 g of DAP, or 0.004 g of TSP, 3 mL of ethanol; reflux, 60 min.
[b] Isolated yield.

Scheme 2.



possess a remarkable recycling capacity. The possibility of using recyclable catalysts under optimum conditions to synthesize a large number of organic compounds is the main contribution of this work to preserving our environment from toxic pollutants.

EXPERIMENTAL

All chemicals were purchased from Sigma–Aldrich. The progress of reactions was monitored by TLC on silica gel F254 plates (Merck) using ethyl acetate–hexane as eluent. The melting points were measured on a Kofler hot stage. The ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Brucker AV II spectrometer at 300 and 75 MHz, respectively,

General procedure for the synthesis of 9-arylacridine-1,8-diones 4a–4g. A mixture of 0.28 g (2 mmol) of 5,5-dimethylcyclohexane-1,3-dione, 0.154 g (1 mmol) of ammonium acetate, and 1 mmol of aromatic aldehyde **2a**–**2g** in 3 ml of ethanol containing MAP, DAP, or TSP as catalyst was heated

Table 5. Synthesis of 9-phenyl-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**4a**) in the presence of phosphate fertilizers and other catalysts

Catalyst	Solvent	Temperature, °C	Time, h	Yield, %	Reference
MAP	EtOH	Reflux	1	94	This study
DAP	EtOH	Reflux	1	98	This study
TSP	EtOH	Reflux	1	96	This study
SiO_2–I	EtOH	80	2.5	90	[37]
FSG–$Hf(NPf_2)_4$	EtOH–H_2O	Reflux	4	82	[38]
[CMIM][CF_3COO]	EtOH–H_2O	80	1.33	87	[39]
SiO_2–$PrSO_3H$	Solvent free	120	2	85	[40]
KH_2PO_4	EtOH–H_2O	120	5	94	[41]
SSA	H_2O	70	1.5	95	[42]
SPNP	H_2O	Reflux	2	91	[43]

under reflux until the reaction was complete (TLC). The mixture was cooled to room temperature, 5 mL of chloroform was added, the undissolved catalyst was filtered off, the filtrate was evaporated, and the residue was recrystallized from ethanol. Compounds **4a–4g** were characterized on the basis of their melting points and NMR spectra, which showed a good agreement with published data [24, 37].

3,3,6,6-Tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (4a). ^{1}H NMR spectrum, δ ppm: 0.98 s and 1.09 s (6H each, CH_3), 2.14–2.46 m (8H, CH_2), 5.10 s (1H, 9-H), 7.13–7.37 m (5H, H_{arom}), 11.92 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 27.20, 29.49, 31.43, 32.76, 46.47, 47.09, 115.60, 125.85, 126.79, 128.08, 138.08,189.40, 195.20.

3,3,6,6-Tetramethyl-9-(4-methylphenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (4b). ^{1}H NMR spectrum, δ, ppm: 0.97 s and 1.07 s (6H each, CH_3), 2.13–2.27 m (8H, CH_2), 2.22 s (3H, 4′-CH_3), 5.31 s (1H, 9-H), 7.00–7.28 m (4H, H_{arom}), 8.05 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 21.10, 27.13, 29.55, 32.60, 33.23, 40.67, 50.94, 113.31, 127.91, 128.70, 135.20, 143.78, 149.19, 195.98.

3,3,6,6-Tetramethyl-9-(4-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (4d). ^{1}H NMR spectrum, δ, ppm: 0.97 s and 1.14 s (6H each, CH_3), 2.19–2.46 m (8H, CH_2), 5.17 s (1H, 9-H), 6.51 s (1H, NH), 7.54 d (2H, H_{arom}), 8.10 d (2H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 27.14, 29.44, 32.72, 34.43, 41.20, 50.57, 112.74, 123.39, 129.04, 148.11, 153.76, 195.19.

9-(4-Bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (4e). ^{1}H NMR spectrum, δ, ppm: 0.88 s and 1.01 s (6H each, CH_3), 2.09–2.31 m (8H, CH_2), 4.95 s (1H, 9-H), 7.14–7.28 m (4H, H_{arom}), 8.43 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 27.06, 29.53, 32.50, 33.43, 40.56, 50.77, 112.55, 129.90, 130.15, 145.98, 149.23, 195.55.

9-(4-Methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (4f). ^{1}H NMR spectrum, δ, ppm: 0.96 s and 1.07 s (6H each, CH_3), 2.15–2.48 m (8H, CH_2), 3.70 s (3H, OCH_3), 5.05 s (1H, 9-H), 6.72–6.78 d (2H, H_{arom}), 7.23–7.28 d (2H, H_{arom}), 7.59 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 27.34, 29, 55, 30.98, 32.78, 40.87, 50.81, 55.11, 113.52, 115.79, 128.96, 129.30, 139.19, 148.49, 162.22, 196.65.

9-(4-Chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (4g). ^{1}H NMR spectrum, δ, ppm: 0.99 s and 1.11 s (6H each, CH_3), 2.19–2.48 m (8H, CH_2), 4.72 s (1H, 9-H), 7.15–7.31 m (4H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 27.28, 29.28, 31.48, 32.21, 40.83, 50.72, 115.22, 128.61, 129.79, 132.01, 142.75, 162.58, 196.45.

CONFLICT OF INTERESTS

The authors declare the absence of conflict of interests.

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