# A simple one-pot synthesis of new 9-aroyl-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridinediones

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**Abstract**

A series of new acridinedione derivatives have been synthesized via one-pot three-component reactions of cyclohexane-1,3-dione, ammonium acetate and arylglyoxals in the presence of alginic acid as a bio-polymeric catalyst in ethanol at room temperature or under reflux conditions. The products were characterized by their spectroscopic data and microanalyses.

**Keywords:** Acridinediones, cyclohexane-1,3-dione, arylglyoxals, ammonium acetate, alginic acid, one-pot multi-component reaction

## Introduction

Acridinedione derivatives are a class of nitrogen-containing heterocyclic compounds with significant synthetic potential. The 1,8-acridinediones and their derivatives are known to exhibit a wide range of biological and pharmaceutical properties such as antibacterial,[1] antimicrobial,[2] anticancer,[3-5] antitumor,[6-7] antifungal,[8] antimalarial,[9] antiviral,[10] larvicidal[11] and hypertensive[12] activities. Electroluminescent devices based on decahydroacridinedione (DAD) derivatives exhibit good efficiencies and excellent color purity.[13] There are many reports on the synthesis of acridinedione derivatives including multi-component reactions (MCRs) of aromatic aldehydes, 1,3-diketones and various aromatic amines or ammonium acetate in the presence of different catalysts.[14-20]

Alginic acid is a naturally occurring polysaccharide that can also be produced by a microbial fermentation. It has many carboxylic acid and hydroxyl groups in its backbone.[21,22] Alginic acid can activate the reaction components not only via its Brønsted acid centers but also by hydrogen bonding, as a heterogeneous catalyst.[23,24]

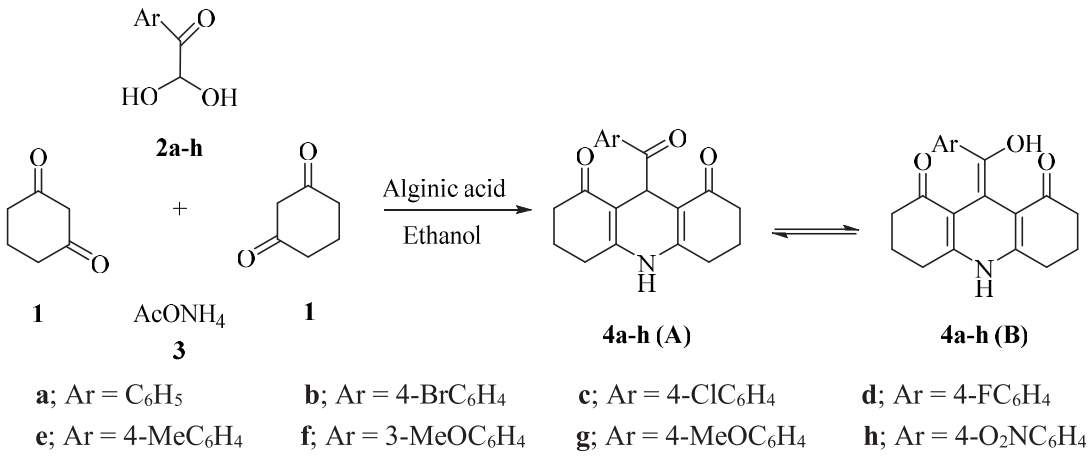
In continuation of our research interests in the synthesis of heterocyclic compounds using arylglyoxals as valuable sources,[25-33] here, we report the synthesis of a series of new 9-aroyl-1,8-acridinediones by a one-pot three-component reaction of cyclohexane-1,3-dione, arylglyoxals

and ammonium acetate in the presence of alginic acid as a catalyst in ethanol at room temperature or under reflux conditions.

## Results and Discussion

The reaction of cyclohexane-1,3-dione (**1**) arylglyoxals as hydrates **2a-h** and ammonium acetate (**3**) in the presence of alginic acid as a catalyst in ethanol at room temperature or under reflux conditions gave a series of new 9-aroyl-1,8-acridinediones **4a-h** by a one-pot, three-component reaction as shown on Scheme 1.

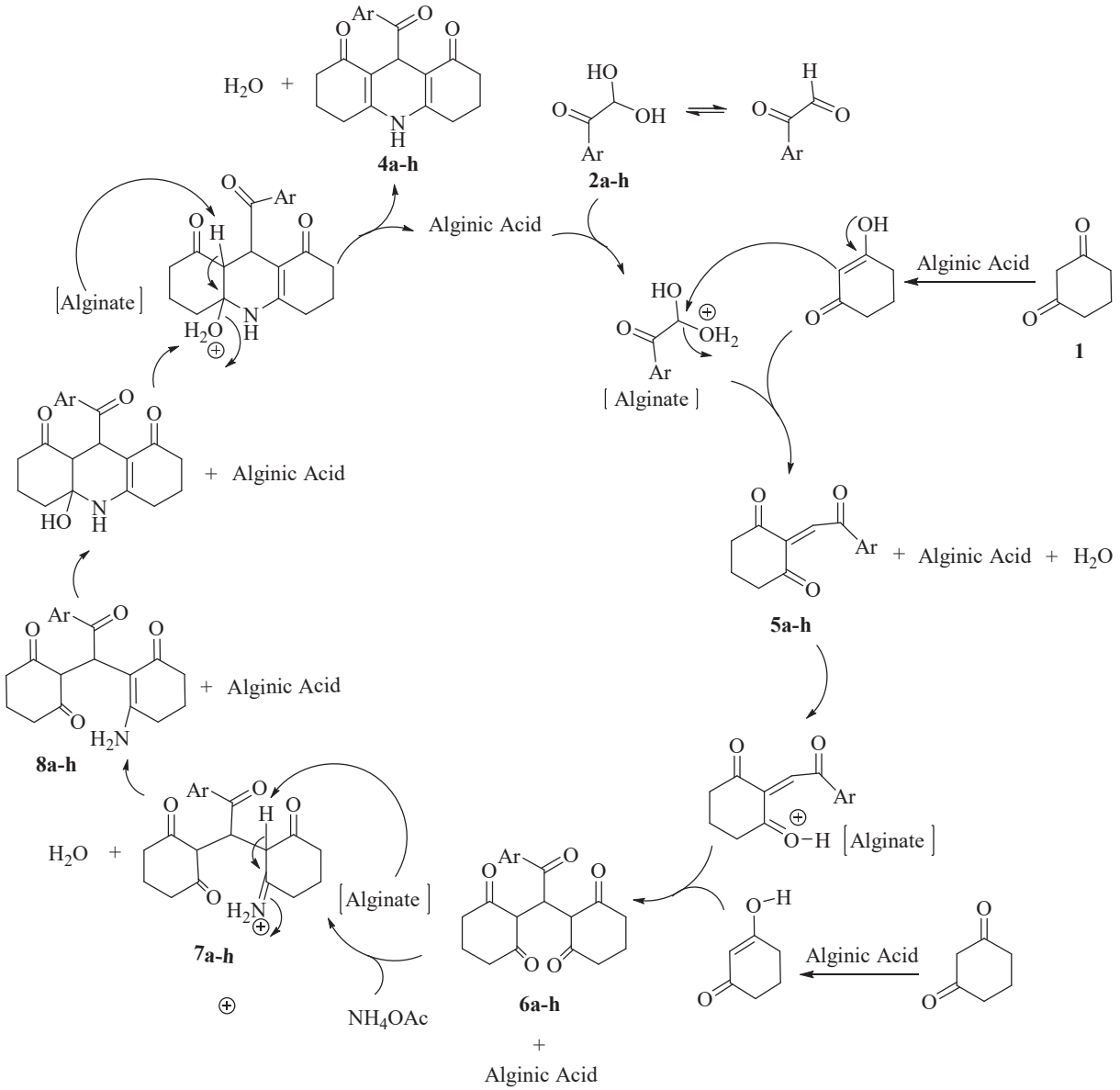



**Scheme 1.** Synthesis of acridinedione derivatives **4a-h**

**Table 1.** The yields and melting points of compounds **4a-h**

| Entry | Substrate | Product | Ar | Time (h) / Conditions | Yield (%) | Mp (°C) |
|---|---|---|---|---|---|---|
| **1** | **2a** | **4a** | $C_6H_5$ | 6 / Reflux | 83 | 248 |
| **2** | **2b** | **4b** | 4-$BrC_6H_4$ | 8 / Reflux | 92 | 245 |
| **3** | **2c** | **4c** | 4-$ClC_6H_4$ | 5 / Reflux | 90 | 242 |
| **4** | **2d** | **4d** | 4-$FC_6H_4$ | 6 / Reflux | 70 | 230 |
| **5** | **2e** | **4e** | 4-$MeC_6H_4$ | 3 / Reflux | 98 | 249 |
| **6** | **2f** | **4f** | 3-$MeOC_6H_4$ | 5 / Reflux | 92 | 230 |
| **7** | **2g** | **4g** | 4-$MeOC_6H_4$ | 5 / RT | 96 | 243 |
| **8** | **2h** | **4h** | 4-$O_2NC_6H_4$ | 7 / Reflux | 79 | 295 |

As the reaction proceeds very slowly in the absence of catalyst, with very low yield, we used algenic acid as a catalyst due to its low-cost, readily availability, easily recoverability, clean and environmentally benign properties.




**a**; Ar = $C_6H_5$ **b**; Ar = 4-$BrC_6H_4$ **c**; Ar = 4-$ClC_6H_4$ **d**; Ar = 4-$FC_6H_4$
**e**; Ar = 4-$MeC_6H_4$ **f**; Ar = 3-$MeOC_6H_4$ **g**; Ar = 4-$MeOC_6H_4$ **h**; Ar = 4-$O_2NC_6H_4$

**Scheme 2.** The proposed mechanism for the one-pot three-component reaction.

The proposed mechanism of reaction is shown in Scheme 2. The first step involves the attack of the enol form of cyclohexane-1,3-dione (**1**) on arylglyoxal **2a-h** catalyzed by alginic acid to

form the corresponding condensation intermediate **5a-h** by loss of a molecule of water. Reaction of this intermediate, protonated by alginic acid, with a second molecule of cyclohexane-1,3-dione (**1**) in its enol form gives the corresponding intermediate **6a-h**. This intermediate **6a-h** is also activated through proton transfer from alginic acid to react with ammonium acetate to form an imine intermediate **7a-h**, which by subsequent tautomerisation through a proton transfer to alginate anion forms the corresponding enamine **8a-h**. In the next step, alginic acid activates the remaining ketone group for ring closure by amino group of the enamine moiety, followed by final elimination of another molecule of water catalyzed by alginic acid to afford the desired products **4a-h**.

Owing to the extreme insolubility of products **4a-h**, their $^{1}$H and $^{13}$C-NMR spectra could not be measured in $CDCl_3$, except for compound **4f**, which showed a singlet at δ = 3.81 ppm for the hydrogen next to aroyl group (H-9) in its keto form (**A**). The $^{1}$H and $^{13}$C-NMR spectra of all products **4a-h** were measured in DMSO-$d_6$, showing singlets for hydroxyl groups at δ = 11.32-11.83 in the enol form (**B**), which were exchanged by $D_2O$ addition. It seems that the products exist as enol form (**B**) in more polar solvent (DMSO-$d_6$) and as keto form in a less polar solvent ($CDCl_3$) as shown on Scheme 1. The C=O absorptions in FT-IR spectra was observed at 1606-1618 cm$^{-1}$.

The reaction conditions, yields and melting points for the synthesis of 9-aroyl-1,8-acridinedione derivatives are shown in Table 1.

In conclusion, we have successfully developed a simple, cheap, efficient and ecofriendly method for the synthesis of 9-aroyl-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridinediones **4a-h** from a one-pot, three-component reaction of various arylglyoxals **2a-h** with cyclohexane-1,3-dione (**1**) and ammonium acetate using the readily available alginic acid as a catalyst.

## Experimental Section

**General.** The chemicals used in this work were purchased from Acros Organics or from Merck, and were used without purification. Melting points were measured on a Philip Harris C4954718 apparatus. $^{1}$H and $^{13}$C NMR spectra were recorded on a Bruker Avance AQS 300 MHz spectrometer at 300 and 75.5 MHz, respectively. Chemical shifts were measured in DMSO-$d_6$ as solvent relative to TMS as the internal standard. Infrared spectra were recorded on a Thermo-Nicolet Nexus 670 FT-IR instrument using KBr discs. Elemental analyses were performed using a Leco Analyzer 932.

**General procedure for synthesis of 9-aroyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-diones (4a-h).** A mixture of cyclohexane-1,3-dione (2 mmol), arylglyoxal (1 mmol) and $NH_4OAc$ (1 mmol) in presence of alginic acid (6 mg) in EtOH (95%, 2 mL) was stirred under reflux or at rt for the appropriate time (Table 1). The precipitate was separated by filtration,

washed with EtOH/$H_2O$ (1:1) (3-4 mL) and recrystallized from absolute EtOH to give the desired products in 70-98% yields.

**9-Benzoyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione** (**4a**). White needles; yield 83%; mp 248 °C. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta_H$ 11.45 (s, 1H, exchanged by $D_2O$ addition, OH), 9.81 (bs, 1H, exchanged by $D_2O$ addition, NH), 7.39 (d, *J* 7.8 Hz, 2H, Ar), 7.29 (t, *J* 7.2 Hz, 2H, Ar), 7.12 (t, *J* 6.9 Hz, 1H, Ar), 2.80 (t, *J* 6 Hz, 2H, $CH_2$), 2.20-2.10 (m, 4H, $CH_2$), 2.21 (t, *J* 5.4 Hz, 2H, $CH_2$), 1.95 (t, *J* 6.3 Hz, 2H, $CH_2$), 1.90-1.82 (m, 2H, $CH_2$). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta_C$ 192.6, 144.0, 133.5, 129.3, 128.8, 127.8, 120.1, 111.6, 111.1, 23.9, 22.9, 20.9. FT-IR (KBr): 3419, 3235, 3152, 2939, 1608, 1463, 1375, 983, 754, 695 cm$^{-1}$. Anal. Calcd for $C_{20}H_{19}NO_3$: C, 74.75; H, 5.96; N, 4.36. Found: C, 74.88; H, 5.87; N, 4.45%.

**9-(4-Bromobenzoyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione** (**4b**)**.** White needles; yield 92%; mp 245 °C. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta_H$ 11.51 (s, 1H, exchanged by $D_2O$ addition, OH), 10.02 (bs, 1H, exchanged by $D_2O$ addition, NH), 7.49 (d, *J* 8.3 Hz, 2H, Ar), 7.33 (d, *J* 8.3 Hz, 2H, Ar), 2.79 (t, *J* 7.2 Hz, 2H, $CH_2$), 2.48-2.28 (m, 4H, 2 × $CH_2$), 2.22 (t, *J* 7.5 Hz, 2H, $CH_2$), 2.10-1.78 (m, 4H, 2 × $CH_2$). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta_C$ 192.6, 144.3, 132.8, 130.5, 128.6, 128.2, 126.7, 120.2, 119.3, 111.9, 111.3, 23.9, 22.9, 20.9. FT-IR (KBr): 3401, 3134, 2937, 1618, 1468, 1375, 1222, 1187, 1135, 1073, 1012, 984, 834, 627, 599 cm$^{-1}$. Anal. Calcd for $C_{20}H_{18}BrNO_3$: C, 60.01; H, 4.53; N, 3.50. Found: C, 60.27; H, 4.32; N, 3.54%.

**9-(4-Chlorobenzoyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione** (**4c**). White prisms; yield 90%; mp 242 °C. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta_H$ 11.50 (s, 1H, exchanged by $D_2O$ addition, OH), 9.87 (bs, 1H, exchanged by $D_2O$ addition, NH), 7.35 (d, *J* 8.4 Hz, 2H, Ar), 7.40 (d, *J* 8.4 Hz, 2H, Ar), 2.80 (bt, *J* 7.2 Hz, 2H, $CH_2$), 2.42-2.23 (m, 4H, 2 × $CH_2$), 2.22 (bt, *J* 7.2 Hz, 2H, $CH_2$), 2.09-1.79 (m, 4H, 2 × $CH_2$). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta_C$ 192.3, 144.3, 132.4, 130.8, 130.0, 128.2, 127.8, 126.4, 120.2, 111.8, 111.3, 23.9, 22.9, 20.9. FT-IR (KBr): 3237, 3137, 2941, 1608, 1469, 1374, 1138, 1074, 983, 829, 734, 634 cm$^{-1}$. Anal. Calcd for $C_{20}H_{18}ClNO_3$: C, 67.51; H, 5.10; N, 3.94. Found: C, 67.41; H, 5.29; N, 4.05%.

**9-(4-Fluorobenzoyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione** (**4d**). White needles; yield 70%; mp 230 °C. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta_H$ 11.45 (s, 1H, exchanged by $D_2O$ addition, OH), 9.85 (bs, 1H, exchanged by $D_2O$ addition, NH), 7.40 (dd, $J_1$ 8.7 Hz, $J_2$ 5.7 Hz, 2H, Ar), 7.14 (t, *J* 8.7 Hz, 2H, Ar), 2.79 (bt, *J* 6.6 Hz, 2H, $CH_2$), 2.43-2.35 (m, 4H, 2 × $CH_2$), 2.20 (bt, *J* 5.7 Hz, 2H, $CH_2$), 2.08-1.84 (m, 4H, 2 × $CH_2$). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta_C$ 192.6, 169.1, 160.2, 143.9, 139.6, 130.1, 128.6, 126.8, 126.7, 120.1, 116.6, 114.8, 111.4, 110.9, 23.9, 22.9, 20.9. FT-IR (KBr): 3431, 3225, 3183, 2942, 1608, 1466, 1375, 1505, 1227, 1142 cm$^{-1}$. Anal. Calcd for $C_{20}H_{18}FNO_3$: C, 70.78; H, 5.35; N, 4.13. Found: C, 70.89; H, 5.22; N, 4.21 %.

**9-(4-Methylbenzoyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione** (**4e**). White needles; yield 98%; mp 249 °C. $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta_H$ 11.39 (s, 1H, exchanged by $D_2O$ addition, OH), 9.75 (bs, 1H, exchanged by $D_2O$ addition, NH), 7.28 (d, $J_1$ 7.8 Hz, $J_2$ 5.7 Hz, 2H, Ar), 7.09 (d, *J* 7.8 Hz, 2H, Ar), 2.79 (bt, *J* 5.1 Hz, 2H, $CH_2$), 2.39-2.28 (m, 4H, 2 × $CH_2$), 2.25 (s, 3H, $CH_3$), 2.20 (bt, *J* 5.4 Hz, 2H, $CH_2$), 2.04-1.78 (m, 4H, 2 × $CH_2$). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta_C$ 192.6, 143.7, 135.5, 130.7, 130.2, 129.4, 126.7, 124.8, 120.1, 111.6, 110.5, 24.0,

22.9, 21.2, 20.9. FT-IR (KBr): 3253, 3149, 2944, 1611, 1505, 1467, 1429, 1377, 1072, 743 $cm^{-1}$. Anal. Calcd for $C_{21}H_{21}NO_3$: C, 75.20; H, 6.31; N, 4.18. Found: C, 75.33; H, 6.18; N, 4.09 %.

**9-(3-Methoxybenzoyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione** (**4f**). White needles; yield 92%; mp 230 °C. $^1H$ NMR (300 MHz, DMSO-$d_6$): $\delta_H$ 11.45 (s, 1H, exchanged by $D_2O$ addition, OH), 9.86 (bs, 1H, exchanged by $D_2O$ addition, NH), 7.21 (t, *J* 7.8 Hz, 1H, Ar), 7.04-6.98 (m, 2H, Ar), 6.72 (d, *J* 8.1 Hz, 1H, Ar), 3.71 (s, 3H, $OCH_3$), 2.80 (bt, *J* 5.4 Hz, 2H, $CH_2$), 2.36-2.33 (m, 4H, 2 × $CH_2$), 2.21 (bt, *J* 5.4 Hz, 2H, $CH_2$), 2.03-1.81 (m, 4H, 2 × $CH_2$). $^{13}C$ NMR (75.5 MHz, DMSO-$d_6$): $\delta_C$ 192.6, 159.6, 144.0, 134.8, 130.7, 129.1, 128.8, 120.2, 113.0, 111.7, 111.4, 110.2, 109.8, 54.4, 23.9, 22.9, 21.0. FT-IR (KBr): 3233, 2937, 1606, 1467, 1424, 1380, 1335, 1234, 1131, 1069, 1038, 985, 749, 696, 603, 573 $cm^{-1}$. Anal. Calcd for $C_{21}H_{21}NO_4$: C, 71.78; H, 6.02; N, 3.99. Found: C, 71.69; H, 6.27; N, 4.11 %. UV (Solvent: MeOH): $\lambda_{max}$ (nm) 207, 249, 294 ($\pi\rightarrow\pi^*$, $n\rightarrow\sigma^*$, $n\rightarrow\pi^*$). In addition, the $^1H$ NMR spectrum of **4f** in $CDCl_3$ showed CH keto form: $^1H$ NMR (300 MHz, $CDCl_3$): δ 8.81 (bs, 1H, exchanged by $D_2O$ addition, NH), 6.95-6.86 (m, 3H, Ar), 6.80 (d, *J* 7.5 Hz, 1H, Ar), 3.81 (s, 1H, CH), 3.76 (s, 3H, $OCH_3$), 2.86 (t, *J* 5.7 Hz, 2H, $CH_2$), 2.64-2.50 (m, 6H, 3 × $CH_2$), 2.46-2.30 (m, 4H, 2 × $CH_2$).

**9-(4-Methoxybenzoyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione** (**4g**). Cream fine crystals; yield 96%; mp 243 °C. $^1H$ NMR (300 MHz, DMSO-$d_6$): $\delta_H$ 11.32 (s, 1H, exchanged by $D_2O$ addition, OH), 9.87 (bs, 1H, exchanged by $D_2O$ addition, NH), 7.31 (d, *J* 9 Hz, 2H, Ar), 6.87 (d, *J* 9 Hz, 2H, Ar), 3.73 (s, 3H, $OCH_3$), 2.78 (bt, *J* 5.4 Hz, 2H, $CH_2$), 2.40-2.23 (m, 4H, 2 × $CH_2$), 2.19 (bt, *J* 7.2 Hz, 2H, $CH_2$), 2.03-1.29 (m, 4H, 2 × $CH_2$). $^{13}C$ NMR (75.5 MHz, DMSO-$d_6$): $\delta_C$ 192.5, 158.1, 143.5, 129.4, 128.0, 126.2, 120.0, 114.9, 114.1, 111.7, 109.7, 56.4, 24.0, 22.9, 20.93. FT-IR (KBr): 3426, 3239, 3107, 2941, 1608, 1507, 1464, 1372, 1253, 1035, 749 $cm^{-1}$. Anal. Calcd for $C_{21}H_{21}NO_4$: C, 71.78; H, 6.02; N, 3.99. Found: C, 71.63; H, 6.11; N, 4.09 %.

**9-(4-Nitrobenzoyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione** (**4h**). Yellow needles; yield 79%; mp 295 °C. $^1H$ NMR (300 MHz, DMSO-$d_6$): $\delta_H$ 11.83 (s, 1H, exchanged by $D_2O$ addition, OH), 10.08 (bs, 1H, exchanged by $D_2O$ addition, NH), 8.18 (d, *J* 8.7 Hz, 2H, Ar), 7.64 (d, *J* 8.7 Hz, 2H, Ar), 2.83 (bt, *J* 6.9 Hz, 4H, 2 × $CH_2$), 2.50-2.30 (m, 2H, $CH_2$), 2.24 (t, *J* 7.2 Hz, 2H, $CH_2$), 2.05-1.85 (m, 4H, 2 × $CH_2$). $^{13}C$ NMR (75.5 MHz, DMSO-$d_6$): $\delta_C$ 192.7, 145.9, 145.0, 140.1, 127.2, 126.3, 125.2, 123.4, 120.6, 115.6, 111.0, 23.7, 22.9, 20.8. FT-IR (KBr): 3421, 3228, 2948, 1608, 1514, 1344, 1188, 1130 $cm^{-1}$. Anal. Calcd for $C_{20}H_{18}N_2O_5$: C, 65.57; H, 4.95; N, 7.65. Found: C, 65.66; H, 4.88; N, 7.52%.

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## Supplementary Information

$^{1}$H-NMR, $^{13}$C-NMR and FT-IR spectral data for compounds **4a-h** are available as supplementary information.

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