Fe^{3+}-montmorillonite: An Efficient Solid Catalyst for One-Pot Synthesis of Decahydroacridine Derivatives

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Fe^{3+}-montmorillonite was used as a solid acidic catalyst for the synthesis of decahydroacridine derivatives through the condensation of aromatic aldehydes, dimedone and aniline in ethanol. The influence of reaction time, reaction temperature, and the amount of catalyst were studied. All the compounds were characterized by IR, Mp, ^{1}H NMR and ^{13}C NMR analysis.

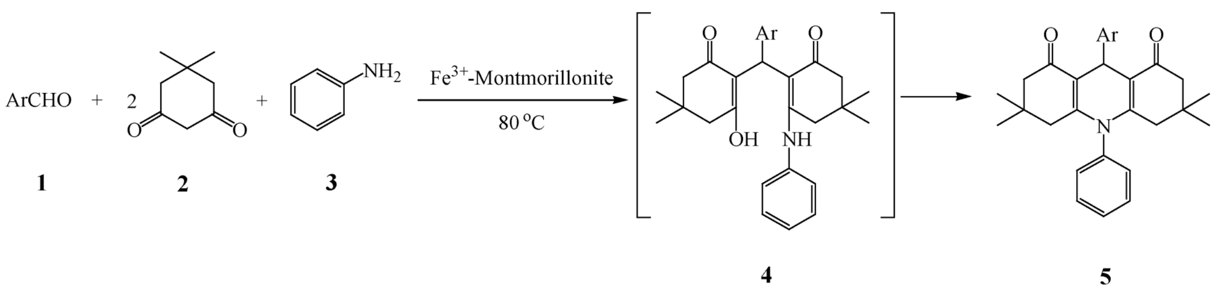
Keywords: Fe^{3+}-montmorillonite; Decahydroacridine derivatives; Solid acid catalysts; Aromatic aldehyde; Dimedone.

INTRODUCTION

In recent years, the use of solid acidic catalysts, such as montmorillonite clays, have received considerable attention in different areas of organic synthesis, because of their environmental compatibility, reusability, operational simplicity, non-toxicity, non-corrosiveness, low cost and the ease of isolation.[1] As solid acidic catalysts, montmorillonite clays act as both Brønsted and Lewis acids in their natural forms,[2] which enable them to function as acidic catalysts. Cation treated montmorillonite with transition metal ions can result in the change in acidities, which has given them an acidity-tunable property. This type of montmorillonite has been used for many typical acid-catalyzed reactions such as Friedel-Crafts reaction,[3] Beckmann rearrangement,[4] Paal-Knorr condensation,[5] esterification,[6] synthesis of phenylacetates,[7] the protection of aromatic aldehydes with acetic anhydride,[8] etc.

1,4-Dihydropyridine (1,4-DHPs) and their derivatives have attracted strong interest due to their pharmacological activity as the most important class of the calcium channel modulators. Different catalysis systems, such as 3,4,5-trifluorobenzeneboronic acid in ionic liquid,[9] tetrabutylammonium hydrogen sulfate,[10] iodotrimethylsilane,[11] iodine,[12] and silica gel/$NaHSO_4$,[13] have been used for their synthesis. Recently, chemists have paid more attention to 1,4-DHPs derivatives with different substituents,[14] in order to explore the structure-activity relationship of these compounds. Herein we report the synthesis of some decahydroacridine derivatives bearing DHPs (**5**) in the presence of the catalyst of Fe^{3+}-montmorillonite, as shown in Scheme I. To our knowledge, some of these compounds (**5**) are reported for the first time in the literature.

Scheme I Synthesis of 1,8-dioxo-decahydroacridine derivatives catalyzed by Fe^{3+}-montmorillonite



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RESULTS AND DISCUSSION

Initially, the condensation of benzaldehyde, dimedone and aniline was selected as the model reaction to be studied in ethanol at 80 °C for 6 h, by using different metal-exchanged montmorillonites and K10 as catalysts, separately. The results are listed in Table 1. It was obvious that Fe^{3+}-montmorillonite appeared more active compared with other clay catalysts. The catalytic activities of the examined montmorillonite clays were gradually decreased in the following order: $Fe^{3+} > Co^{2+} > Zn^{2+} > Al^{3+} > Cu^{2+}$-montmorillonite > montmorillonite K10. However, almost no prospective product **5a** could be obtained after 6 h under catalyst-free conditions, and only intermediate **4a** (Mp 198-200 °C) was obtained in 38% yield (Table 1, entry 7). Consequently, the mechanism of the reaction could be described as follows:[15] intermediate **4a** was formed firstly by Knoevenagel addition and then attacked by the NH_2 group of aniline. Subsequently, **4a** was cyclized by elimination of water and product **5a** was obtained. In these processes, Fe^{3+}-montmorillonite played a crucial role in accelerating the reaction, especially for water elimination of intermediate. It could be verified by the fact that reaction under catalyst-free conditions gave a product of intermediate **4a**.

On the basis of the above exploration, the effects of catalyst amount, temperature and reaction time were also studied on the model reaction, and the results are listed in Table 2. Obviously, all the factors had much effect on the condensation. With the increase in catalyst amount, reaction temperature and reaction time, the yield also increased. The optimized condition was 15 wt% (based on aldehyde) of Fe^{3+}-montmorillonite, 80 °C and 6 h, under which product **5a** could be obtained in 92%.

Furthermore, other aldehydes and aromatic amines have been subjected to the abovementioned optimized conditions, and the results are listed in Table 3. The aromatic aldehydes reacted well with aniline to give the corresponding decahydroacridine in the yields ranging from 80% to 92% over the Fe^{3+}-montmorillonite catalyst. The substituents on the aromatic ring seemed to have some effects on the yield but no obvious regulation was shown for their electronic nature. Importantly, in this system, such acid-labile substrates as 4-methoxybenzaldehyde, vanillin and piperonal all worked well. When the aliphatic aldehyde was performed, the yield was low. (Table 3, entry 12). However, cinnamaldehyde had the electronic effect analogous to –CHO grafted on the benzene ring and gave a good yield. It is worth pointing out that these decahydroacridines (except **5j**[15]) have not been reported in the literature.

Because Fe^{3+}-montmorillonite was a solid material, it could be easily recycled after reaction by simple filtration. After washing and activating at 120 °C for 5 h, the catalyst had been further examined in next run; the result indicated that the catalyst could be reused four times and without significant loss of its activity. (Table 3, entry 1).

Table 1. Condensation of benzaldehyde, dimedone and aniline catalyzed by different catalysts[a]

Entry	Catalyst[a]	Time (h)	Yield (%)[b]
1	Fe^{3+}-mont	6	92
2	Co^{2+}-mont	6	84
3	Zn^{2+}-mont	6	70
4	Cu^{2+}-mont	6	66
5	Al^{3+}-mont	6	60
6	K10	6	56
7	None	6 (12)	38 (46)[c]

[a] Reaction conditions: the amount of catalyst was 15 wt% (based on benzaldehyde), reaction temperature: 80 °C, solvent: EtOH.
[b] Isolated yield of **5a**.
[c] Isolated yield of intermediate **4a**.

Table 2. Conditions optimization for the condensation among benzaldehyde, dimedone and aniline over Fe^{3+}-montmorillonite catalyst[a]

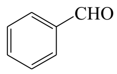
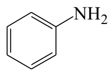
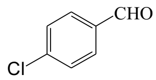
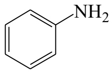
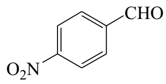
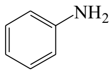
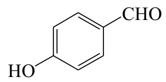
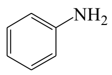
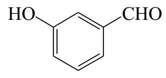
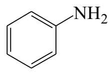
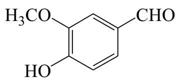
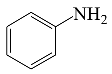
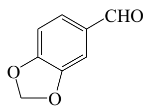
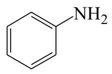
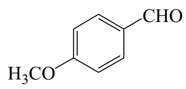
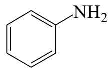
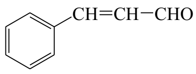
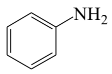
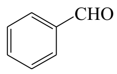
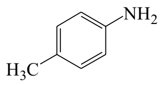
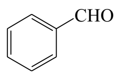
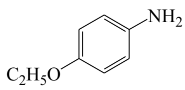
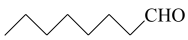
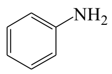
Entry	Catalyst amount (wt%)	Temperature (°C)	Time (h)	Yield (%)[b]
1	20	80	6	94
2	15	80	6	92
3	10	80	6	83
4	5	80	6	68
5	15	100	6	95
6	15	60	6	71
8	15	80	2	68
9	15	80	4	80

[a] Reaction conditions: benzaldehyde (1 mmol), dimedone (2 mmol) and aniline (1 mmol) Fe^{3+}-montmorillonite catalyst (15 wt% based on benzaldehyde) and EtOH as a solvent.
[b] Isolated yield of **5a**.

CONCLUSIONS

Using Fe^{3+}-montmorillonite as a catalyst, 1,8-dioxo-9-aryl-10-(4-phenyl)-decahydroacridine and their derivatives were synthesized successfully from aromatic aldehydes, dimedone and aniline with high yields. To the best of our knowledge, the synthesis of most of these compounds are reported in the literature for the first time. The experimental procedure offered the advantages of opera-

Table 3. The synthesis of various 1,8-dioxo-decahydroacridines and their derivatives catalyzed by Fe^{3+}-montmorillonite[a]

Entry	Aldehyde	Amine	Products	Yield (%)[b]	Mp (°C)
1			**5a**	92 (89[c])	200-205
2			**5b**	82	200-205
3			**5c**	86	216-218
4			**5d**	77	234-235
5			**5e**	79	242-245
6			**5f**	87	292-295
7			**5g**	78	252-255
8			**5h**	93	276-279
9			**5i**	81	271-273
10			**5j**	95	265-267
11			**5k**	72	219-221
12			**5l**	32	192-194

[a] All reactions were carried out at 80 °C for 6 hours.
[b] Isolated yields.
[c] The catalyst was reused for the fourth time.

tional simplicity, the ease of catalyst separation, and good catalyst recycling.

EXPERIMENTAL

Representative procedure for the synthesis of acridine and their derivatives

Benzaldehyde (1 mmol), dimedone (2 mmol), aniline (1 mmol) and Fe^{3+}-montmorillonite catalyst (15 wt% based on benzaldehyde) were added in ethanol (5 mL) in a round bottom flask, and stirred at 80 °C for 6 hours. After reaction, the solid catalyst was filtered and washed with ethanol. This ethanol solution was then put into a 100 mL beaker containing approx. 50 mL ice water, and the crude product was obtained. The pure product could be obtained by recrystallization in ethanol. Data of compounds is shown below.

Analytical data for compounds

3,3,6,6-Tetramethyl-1,8-dioxo-9-benzene-10-(4-phenyl)-decahydroacridine (5a)[16]

White solid, mp: 200-205 °C. IR (KBr): 3104, 2962, 2872, 1643, 1595, 1492, 1447, 1375, 1301, 1251, 1167, 1045, 870, 777, 722, 696 cm^{-1}. ^{1}H NMR (300 MHz,

$CDCl_3$): 0.88 (s, 6H, $2CH_3$), 0.98 (s, 6H, $2CH_3$), 2.19 (d, 2H, J = 16.0 Hz, H_{AB}, H-4a, H-5a), 2.28 (d, 2H, J = 16.0 Hz, H_{AB}, H-4e, H-5e), 2.44 (q, 4H, J = 9.8 Hz, $2CH_2$, H-2, H-7), 5.541 (s, 1H, H-9), 7.01 (s, 1H, ArH), 7.08 (d, J = 6.4 Hz, 2H, ArH), 7.14-7.34 (m, 5H, ArH), 7.48 (d, J = 6.4 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 212.2, 150.8, 145.1, 136.8, 131.5, 130.1, 128.6, 126.6, 126.1, 125.8, 124.2, 51.2, 42.9, 35.6, 23.1, 22.6. Anal Calcd for $C_{29}H_{31}NO_2$: C 81.88, H 7.29. Found: C 81.72, H 7.39.

3,3,6,6-Tetramethyl-1,8-dioxo-9-(4-Chlorophenyl)-10-(4-phenyl)-decahydroacridine (5b)

White solid, mp: 202-204 °C. IR (KBr): 3101, 2958, 2869, 1639, 1594, 1489, 1469, 1373, 1300, 1251, 1153, 1093, 829, 777, 586 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 0.88 (s, 6H, $2CH_3$), 0.99 (s, 6H, $2CH_3$), 1.54 (d, 2H, J = 16.8 Hz, H_{AB}, H-4a, H-5a), 2.28 (d, 2H, J = 16.8 Hz, H_{AB}, H-4e, H-5e), 2.40 (q, 4H, J = 14.6, $2CH_2$, H-2, H-7), 5.47 (s, 1H, H-9), 7.00 (s, 1H, ArH), 7.01 (d, J = 7.6 Hz, 2H, ArH), 7.10-7.34 (m, 4H, ArH), 7.48 (d, J = 6.4 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 213.6, 153.7, 151.4, 146.1, 137.3, 135.7, 132.3, 129.9, 128.1, 126.2, 125.6, 51.4, 42.7, 35.5, 23.1, 22.3. Anal Calcd for $C_{29}H_{30}NO_2Cl$: C 75.73, H 6.53. Found: C 75.65, H 6.79.

3,3,6,6-Tetramethyl-1,8-dioxo-9-(4-nitrophenyl)-10-(4-phenyl)-decahydroacridine (5c)[16]

White solid, mp: 216-218 °C. IR (KBr): 3075, 2962, 2928, 2870, 1638, 1595, 1492, 1447, 1375, 1300, 1250, 1167, 1045, 870, 777, 722, 695 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 0.85 (s, 6H, $2CH_3$), 1.10 (s, 6H, $2CH_3$), 1.54 (d, J = 16.2 Hz, 2H, H_{AB}, H-4a, H-5a), 2.28 (d, J = 16.2, 2H, H_{AB}, H-4e, H-5e), 2.40 (q, J = 14.6 Hz, 4H, H_{AB}, $2CH_2$, H-2, H-7), 5.54 (s, 1H, H-9), 7.03 (s, 1H, ArH), 7.10 (d, J = 6.4 Hz, 2H, ArH), 7.11-7.34 (m, 4H, ArH), 7.48 (d, J = 6.4 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 213.1, 153.7, 150.9, 146.4, 137.8, 135.8, 132.5, 129.9, 128.2, 126.3, 125.8, 51.9, 42.4, 35.8, 23.3, 22.7. Anal Calcd for $C_{29}H_{30}N_2O_4$: C 74.04, H 6.38. Found: C 74.22, H 6.13.

3,3,6,6-Tetramethyl-1,8-dioxo-9-(4-hydroxyphenyl)-10-(4-phenyl)-decahydroacridine (5d)

White solid, mp: 234-235 °C. IR (KBr): 3439, 2958, 2926, 2868, 1665, 1593, 1511, 1468, 1403, 1389, 1265, 1155, 1043, 833, 755, 736, 697 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 0.87 (s, 6H, $2CH_3$), 0.98 (s, 6H, $2CH_3$), 1.55 (d, J = 17.6 Hz, 2H, H_{AB}, H-4a, H-5a), 2.21 (d, J = 17.6, 2H, H_{AB}, H-4e, H-5e), 2.39 (q, J = 16.4 Hz, 4H, H_{AB}, $2CH_2$, H-2, H-7), 5.54 (s, 1H, H-9), 5.99 (s, 1H, OH), 7.10 (d, J = 6.4 Hz, 2H, ArH), 7.24-7.48 (m, 4H, ArH), 7.48 (d, J = 6.0 Hz, 2H, ArH), 9.11 (s, 1H, OH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 212.5, 161.4, 153.7, 151.1, 146.1, 137.9, 135.8, 132.5, 129.9, 128.3, 125.7, 51.8, 42.6, 35.8, 23.1, 22.5. Anal Calcd for $C_{29}H_{31}NO_3$: C 75.91, H 7.03. Found: C 75.82, H 7.13.

3,3,6,6-Tetramethyl-1,8-dioxo-9-(3-hydroxyphenyl)-10-(4-phenyl)-decahydroacridine (5e)

White solid, mp: 240-246 °C. IR (KBr): 3425, 2959, 2926, 2869, 1669, 1595, 1512, 1445, 1403, 1371, 1254, 1168, 1041, 1014, 833, 755, 736, 589 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 0.81 (s, 6H, $2CH_3$), 0.99 (s, 6H, $2CH_3$), 1.55 (d, J = 17.6 Hz, 2H, H_{AB}, H-4a, H-5a), 2.27 (d, J = 17.6, 2H, H_{AB}, H-4e, H-5e), 2.43 (q, J = 16.4 Hz, 4H, H_{AB}, $2CH_2$, H-2, H-7), 5.47 (s, 1H, H-9), 5.97 (s, 1H, OH), 6.74 (d, J = 6.4 Hz, 2H, ArH), 7.04-7.34 (m, 4H, ArH), 7.51 (d, J = 6.0 Hz, 2H, ArH), 9.11 (s, 1H, OH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 213.1, 212.4, 160.7, 151.7, 150.9, 147.2, 146.4, 137.8, 136.5, 135.8, 134.7, 132.5, 132.1, 130.6, 129.9, 125.8, 125.2, 122.3, 121.8, 52.9, 51.9, 50.8, 43.1, 42.6, 35.8, 34.4, 23.3, 23.2, 22.3. Anal Calcd for $C_{29}H_{31}NO_3$: C 78.91, H 7.03. Found: C 78.78, H 7.13.

3,3,6,6-Tetramethyl-1,8-dioxo-9-(4-hydroxy-3-methoxy-phenyl)-10-(4-phenyl)-decahydroacridine (5f)[16]

White solid, mp: 292-295 °C. IR (KBr): 3442, 2956, 2868, 1629, 1581, 1510, 1485, 1440, 1378, 1273, 1233, 1196, 1140, 1008, 927, 768, 649 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 0.88 (s, 6H, $2CH_3$), 0.99 (s, 6H, $2CH_3$), 1.55 (d, J = 17.2 Hz, 2H, H_{AB}, H-4a, H-5a), 1.97 (d, J = 17.2, 2H, H_{AB}, H-4e, H-5e), 2.45 (q, J = 16.4 Hz, 4H, H_{AB}, $2CH_2$, H-2, H-7), 3.77 (s, 3H, OCH_3), 5.45 (s, 1H, H-9), 6.12 (s, 1H, OH), 6.75-6.91 (m, 2H, ArH), 7.12 (d, J = 10.4 Hz, 2H, ArH), 7.26 (s, 1H, ArH), 7.48 (d, J = 10.4 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 212.6, 212.3, 164.7, 157.3, 151.2, 147.3, 146.6, 137.9, 137.3, 135.8, 134.9, 133.5, 132.6, 130.6, 126.7, 125.8, 125.3, 122.9, 121.8, 53.2, 52.4, 50.8, 43.3, 42.8, 35.8, 34.9, 23.6, 23.3, 22.6, 22.2. Anal Calcd for $C_{30}H_{33}NO_4$: C 76.43, H 7.01. Found: C 76.52, H 6.93.

3,3,6,6-Tetramethyl-1,8-dioxo-9-(3,4-methylendioxy-phenyl)-10-(4-phenyl)-decahydroacridine (5g)

White solid, mp: 250-255 °C. IR (KBr): 3440, 2959, 2926, 2868, 1653, 1595, 1503, 1488, 1446, 1376, 1308, 1232, 1153, 1039, 934, 811, 600 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 0.85 (s, 6H, $2CH_3$), 1.10 (s, 6H, $2CH_3$), 1.54 (d, 2H, J = 16.8 Hz, H_{AB}, H-4a, H-5a), 2.10 (d, 2H, J = 16.8 Hz, H_{AB}, H-4e, H-5e), 2.38 (q, J = 14.6, 4H, $2CH_2$, H_{AB}, H-2, H-7), 5.46 (s, 1H, H-9), 5.91 (s, 2H, OCH_2O), 6.57-

6.69 (m, 2H, ArH), 7.26 (d, J = 9.6 Hz, 2H, ArH), 7.34 (s, 1H, ArH), 7.48 (d, J = 9.6 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 212.2, 147.4, 145.1, 136.8, 131.5, 130.1, 128.6, 126.6, 125.7, 125.1, 101.7, 53.2, 51.2, 42.1, 35.3, 23.2, 22.9. Anal Calcd for $C_{30}H_{31}NO_4$: C 76.76, H 6.61. Found: C 76.65, H 6.89.

3,3,6,6-Tetramethyl-1,8-dioxo-9-(4-methoxyphenyl)-10-(4-phenyl)-decahydroacridine (5h)[16]

White solid, mp: 282-284 °C. IR (KBr): 3101, 2958, 2922, 2869, 1665, 1626, 1601, 1586, 1511, 1464, 1360, 1284, 1262, 1197, 1035, 841, 745, 684 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): 0.82 (s, 6H, $2CH_3$), 1.01 (s, 6H, $2CH_3$), 1.53 (d, 2H, J = 16.8 Hz, H_{AB}, H-4a, H-5a), 2.22 (d, 2H, J = 16.8 Hz, H_{AB}, H-4e, H-5e), 2.47 (q, 4H, J = 14.6, $2CH_2$, H-2, H-7), 3.78 (s, 3H, OCH_3), 5.49 (s, 1H, H-9), 6.80 (s, 1H, ArH), 7.01 (d, J = 7.6 Hz, 2H, ArH), 7.26-7.34 (m, 3H, ArH), 7.48 (d, J = 6.4 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 212.8, 165.8, 149.7, 148.5, 147.2, 137.9, 135.6, 132.5, 129.8, 128.5, 126.3, 125.7, 52.7, 51.9, 42.4, 35.8, 23.3, 22.1. Anal Calcd for $C_{30}H_{33}NO_3$: C 79.12, H 7.25. Found: C 79.45, H 7.09.

3,3,6,6-Tetramethyl-1,8-dioxo-9-(4-propenylphenyl)-10-(4-phenyl)-decahydroacridine (5i)

White solid, mp: 270-275 °C. IR (KBr): 3442, 2957, 2868, 1670, 1623, 1596, 1514, 1495, 14689, 1378, 1269, 1250, 1220, 1152, 1030, 886, 781, 699 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): 0.85 (s, 6H, $2CH_3$), 0.92 (s, 6H, $2CH_3$), 1.55 (d, 2H, J = 16.8 Hz, H_{AB}, H-4a, H-5a), 2.09 (d, 2H, J = 16.8 Hz, H_{AB}, H-4e, H-5e), 2.47 (q, 4H, J = 14.6, $2CH_2$, H-2, H-7), 4.54 (s, 2H, CH=CH), 5.54 (s, 1H, H-9), 7.08 (s, 1H, ArH), 7.11 (d, J = 9.6 Hz, 2H, ArH), 7.26-7.34 (m, 5H, ArH), 7.48 (d, J = 9.6 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 212.7, 150.7, 149.9, 146.4, 142.1, 137.8, 136.9, 135.8, 132.5, 129.9, 128.2, 126.3, 125.8, 52.4, 42.5, 35.8, 23.2. Anal Calcd for $C_{31}H_{33}NO_2$: C 77.18, H 6.85. Found: C 77.21, H 7.01.

3,3,6,6-Tetramethyl-1,8-dioxo-9-benzene-10-(4-methyl-phenyl)-decahydroacridine (5j)

White solid, mp: 262-263 °C. IR (KBr): 3075, 2959, 2869, 1661, 1594, 1522, 1492, 1374, 1303, 1264, 1222, 1165, 1043, 818, 696 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): 0.80 (s, 6H, $2CH_3$), 0.94 (s, 6H, $2CH_3$), 2.34 (d, 2H, J = 16.0 Hz, 2H, H_{AB}, H-4a, H-5a), 2.39 (d, 2H, J = 16.0 Hz, H_{AB}, H-4e, H-5e), 2.44 (q, J = 9.8 Hz, 4H, H_{AB}, $2CH_2$, H-2, H-7), 5.54 (s, 1H, H-9), 7.08 (d, J = 6.4 Hz, 2H, ArH), 7.11-7.40 (m, 5H, ArH), 7.48 (d, J = 6.4 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 213.1, 151.2, 150.9, 146.4, 137.8, 135.8, 132.5, 129.9, 128.2, 126.3, 125.8, 51.9, 42.4, 35.8, 23.3, 21.5. Anal Calcd for $C_{30}H_{33}NO_2$: C 82.00, H 7.52. Found: C 82.06, H 7.46.

3,3,6,6-Tetramethyl-1,8-dioxo-9-benzene-10-(4-ethoxy-phenyl)-decahydroacridine (5k)

White solid, mp: 219-221 °C. IR (KBr): 3075, 2962, 2830, 2870, 1673, 1595, 1492, 1447, 1375, 1299, 1250, 1168, 871, 777, 721, 695 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): 0.88 (s, 6H, $2CH_3$), 0.91 (m, 3H, CH_3), 1.10 (s, 6H, $2CH_3$), 1.54 (d, 2H, J = 16.0 Hz, 2H, H_{AB}, H-4a, H-5a), 2.28 (d, 2H, J = 16.0 Hz, H_{AB}, H-4e, H-5e), 2.44 (q, J = 9.8 Hz, 4H, H_{AB}, $2CH_2$, H-2, H-7), 4.79 (m, 2H, CH_2), 5.54 (s, 1H, H-9), 7.08 (d, J = 6.4 Hz, 2H, ArH), 7.14-7.34 (m, 5H, ArH), 7.48 (d, J = 6.4 Hz, 2H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 213.1, 151.7, 150.9, 146.4, 137.8, 135.8, 132.5, 129.9, 128.2, 126.3, 125.8, 65.6, 51.9, 42.4, 35.8, 23.3, 15.2. Anal Calcd for $C_{31}H_{35}NO_3$: C 79.32, H 7.46. Found: C 79.43, H 7.40.

3,3,6,6-Tetramethyl-1,8-dioxo-9-heptyl-10-(4-phenyl)-decahydroacridine (5l)

White solid, IR (KBr): 3104, 2964, 2870, 1637, 1594, 1486, 1447, 1375, 1311, 1252, 1161, 1045, 872, 772, 726, 698 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): 0.87 (s, 12H, $4CH_3$), 0.89 (m, 3H, CH_3), 1.53 (m, 10H, $5CH_2$), 1.61 (m, 2H, CH_2), 1.83 (d, J = 16.0 Hz, 2H), 2.14 (d, J = 16.0 Hz, 2H), 2.44 (q, J = 9.8 Hz, 4H, $2CH_2$), 5.52 (s, 1H, H-9), 7.01 (m, 2H, ArH), 7.24-7.38 (m, 2H, ArH), 7.41 (m, 1H, ArH). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 213.1, 152.1, 151.4, 135.8, 129.9, 129.1, 126.3, 51.9, 38.2, 45.3, 42.4, 35.8, 31.8, 29.4, 29.1, 25.8, 23.3, 22.7, 22.2, 14.3. Anal Calcd for $C_{30}H_{41}NO_2$: C 80.54, H 9.17. Found: C 80.72, H 9.13.

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