Synthesis of 10-Amino-9-aryl-2,3,4,5,6,7,9,10-octahydroacridine-1,8-dione Derivatives

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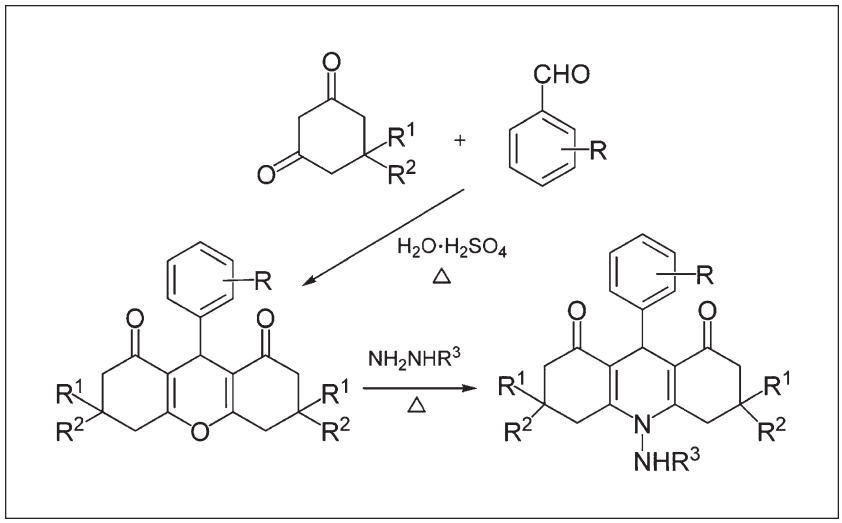
Received September 8, 2010
DOI 10.1002/jhet.764
Published online 24 August 2011 in Wiley Online Library (wileyonlinelibrary.com).



A series of novel 10-amino-9-aryl-2,3,4,5,6,7,9,10-octahydroacridine-1,8-dione derivatives **4** were synthesized by hydrazine or phenylhydrazine and 9-aryl-1,8-dioxo-2,3,4,5,6,7,9-heptahydroxanthene derivatives **3**, which were prepared by 5-substituted-1,3-cyclohexanedione **1** and aromatic aldehydes **2** in the presence of concentrated H_2SO_4 as a catalyst in water. The structures of all compounds were characterized by IR, MS, ^{1}H-NMR, and elemental analysis, and the title compounds possess good fluorescence properties.

J. Heterocyclic Chem., **49**, 195 (2012).

INTRODUCTION

The family of acridine derivatives is a class of the earliest discovered bioactive compounds, which were widely used as antibacterial, anti-inflammatory, antimalarial, deworming, antitumor, anti-blood-sucking insects, and memory enhancement agents [1–3]. The emergence of penicillin eclipsed acridines in antisepsis because of the greater therapeutic efficiency compared with the former. However, with current rapid increasement in drug-resistant bacterial infection, novel acridine derivatives may be of new use. A great many researches and developments have been carried out on acridine derivatives [4,5]. In these areas, recent study has been focused mainly on their functions as anticancer drugs, because of their ability of the acridine chromophore to intercalate DNA and inhibit topoisomerase enzyme, which further block the action of DNA-metabolizing proteins pharmacologically. At the same time, as acridine has large ring-conjugated system and a rigid planar structure, it is a good fluorescent reagent, which can be used as chemiluminescent DNA probes, DNA, and other macromolecules embedded in body [6–8]. This class of compounds belongs to several agents of clinical importance such as actinomycin-D, daunomycin, adriamycin, tacine, and amsacrine [9,10].

In terms of considerable synthetic attention being paid to the substituted aminoacridines, the mode of binding of acridine molecules involves intercalation of the acridine ring between adjacent base pairs in the DNA duplex and acridines moieties are held in place by van der Waals force supplemented by stronger ionic bonds to the phosphate ions of the DNA backbone [11–13]. Here, we reported the synthesis of the title compounds in this study. We hope that these novel acridine derivatives will be of anticancer activities and fluorescence probes.

RESULTS AND DISCUSSION

The synthetic route is shown in Scheme 1; melting point and yields of the title compounds are given in

Scheme 1

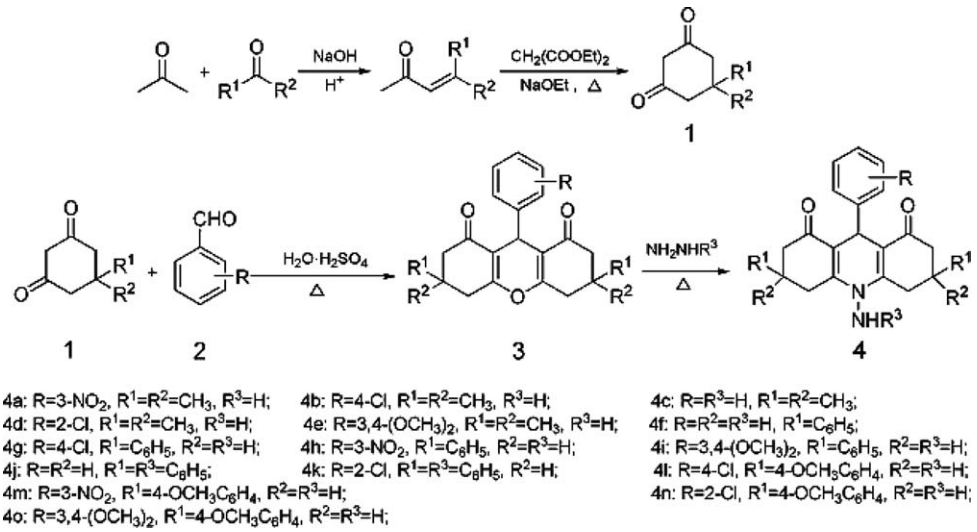


Table 1. Most importantly, aromatic aldehydes with either electron-donating or electron-withdrawing substituents reacted very well to afford the corresponding heptahydroxanthene derivatives **3**, and the title compounds were obtained with good yields and high purity.

A plausible formation mechanism of **4** from **3** is shown in Scheme 2. The oxygen atom in pyran ring exhibits stronger electron-withdrawing effect due to its attaching to a cyclic ketene moiety and promotes the Michael addition reaction of hydrazine with another cyclohexenone structure in the asymmetrical position. The electron transfer of negative ion of enol **3a**, as an initial intermediate in the nucleophilic addition reaction, results in ring opening to form an open-chain molecule **3b**, which is anew cyclized into a tricyclic intermediate **3c** by a conjugated addition reaction started from the nitrogen atom of enamine attacks toward the C=C of ketene-conjugated system in another six-membered ring. The further dehydration reaction constructs a wide-area conjugated system through the three cycles of the generated inner-salt-like intermediate **3d**, which finally rearranges to the title compound **4**.

The structures of the prepared 10-amino-9-aryl-2,3,4,5,6,7,9,10-octahydroacridine-1,8-dione **4a–o** were fully characterized by ^{1}H-NMR, MS, IR, and elemental analysis. For example, in the ^{1}H-NMR of **4a**, the two single proton peaks at δ 0.96 and 1.12 ppm were attributed to the protons of four gem-methyl groups at 3- and 6-position. The two groups of deformation doublets at 2.13–2.26 ppm, showed a large mutual coupling

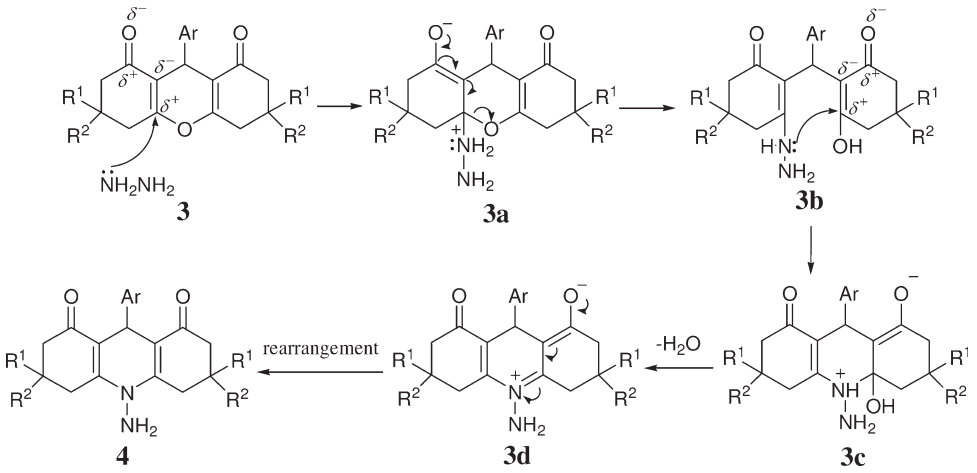
Table 1

Synthesis of 10-amino-9-aryl-2,3,4,5,6,7,9,10-octahydroacridine-1,8-dione derivatives.

Entry	R	R^1	R^2	R^3	m.p.[a] (°C)	Yield[b] (%)
4a	3-NO_2	CH_3	CH_3	H	224–226	69
4b	4-Cl	CH_3	CH_3	H	274–276	68
4c	H	CH_3	CH_3	H	260–262	71
4d	2-Cl	CH_3	CH_3	H	204–206	63
4e	3,4-$(OCH_3)_2$	CH_3	CH_3	H	212–214	65
4f	H	C_6H_5	H	H	244–246	58
4g	4-Cl	C_6H_5	H	H	188–190	65
4h	3-NO_2	C_6H_5	H	H	212–214	61
4i	3,4-$(OCH_3)_2$	C_6H_5	H	H	220–222	71
4j	H	C_6H_5	H	C_6H_5	236–238	82
4k	2-Cl	C_6H_5	H	C_6H_5	176–178	80
4l	4-Cl	4-$OCH_3C_6H_4$	H	H	>260	76
4m	3-NO_2	4-$OCH_3C_6H_4$	H	H	262–264	83
4n	2-Cl	4-$OCH_3C_6H_4$	H	H	242–244	74
4o	3,4-$(OCH_3)_2$	4-$OCH_3C_6H_4$	H	H	222–224	50

[a] Melting points were determined on an electrothermal apparatus, and the temperature was not calibrated.
[b] Yield refers to pure products after chromatography.

Scheme 2



constant (J = 16.20 Hz, $\Delta\nu$ = 22.02 Hz, $\Delta\nu/J$ = 1.36), were ascribable to the proton of two methylene at 4- and 5-position. The chemical shifts of four gem-protons with gem-coupling constant (J = 17.82 Hz, $\Delta\nu$ = 91.83 Hz, $\Delta\nu/J$ = 5.15), linked in 2- and 7-position, were found at 2.51–2.88 ppm in the form of deformation double peaks. With the decrease in the ratio of $\Delta\nu/J$, the outside proton peak gradually became smaller and the inside proton peak gradually became larger and closer [14]. The similar peaks pattern of the methylene at 4- and 5-position, and 2- and 7-position of compounds **4a–e** was also observed in the ^{1}H-NMR. The broad single proton peaks at δ 4.13 ppm disappeared after D_2O exchanging and therefore were attributed to the characteristic absorption proton peak of the two N—H of the amino group. The multiple proton peaks at 7.35–7.96 ppm were attributed to the characteristic absorption proton peak of the four H of the benzene ring. In the IR spectra of **4a**, several typical absorption bands at 1625 cm^{-1} for (C=O) and 3350 cm^{-1} for (N—H) were observed. In ESI mass spectra, the molecular ion peak m/z = 401.2.

Each of compounds **4f–o** should be as a diastereomeric mixture, so that the proton peak for the one H of 9-position displays two single peaks. It was speculated from the ^{1}H-NMR spectrum that the yields of the major product were above 90%. The specific stereostructures of the main product need to be further studied.

LUMINESCENT PROPERTIES

The xanthene derivatives **3** are converted to acridine derivatives **4** by replacing the oxygen atoms with the nitrogen atoms bearing amino. As the electronegativity of nitrogen atoms is lower than that of oxygen atoms, the nonconjugated bond of the lone pair electrons is relatively easy to delocalize, so that the absorption wavelength of acridine derivatives **4** occurs red shift, and the photoluminescent spectra of the compounds in dichloromethane solution are of particular interest. Fluorescence efficiency can be correlated with many structural features of chemicals including π–π* and n–π* transitions, structural rigidity, noncovalent interactions (*e.g.*, hydrogen bonds, π–π interactions, and hydrophilic and hydrophobic interactions), interior intermolecular energy transfers, and photoinduced electron transfers [15,16]. Only the fluorescence of **4e** and **4g** is detected. The dichloromethane solution fluorescent spectra for compound **4e** at room temperature reveal an emission peak at 521/496 nm (the exciting radiation set at 367/398 nm) as shown in Figure 1. The dichloromethane solution fluorescent spectra for compound **4g** at room temperature reveal an emission peak at 522/498 nm (the exciting radiation set at 357/410 nm) as shown in Figure 2. The emission band may be attributed to the π–π* transition of the pyridine ring [17,18].

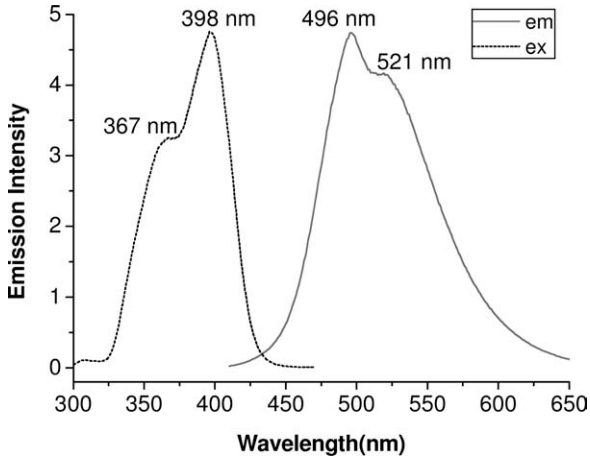


Figure 1. Fluorescent excitement (left) and emission (right) spectra of the compound **4e**.

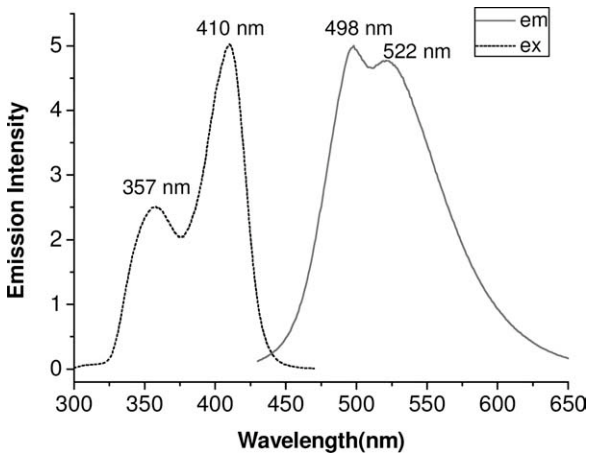


Figure 2. Fluorescent excitement (left) and emission (right) spectra of the compound **4g**.

CONCLUSION

In summary, during the synthesis of 10-amino-9-aryl-2,3,4,5,6,7,9,10-octahydroacridine-1,8-dione derivatives **4**, we first prepared 9-aryl-1,8-dioxo-2,3,4,5,6,7,9-heptahydroxanthene derivatives **3** with H_2SO_4/H_2O as a catalyst. Then, the title compounds **4** were obtained by the reaction of the compounds **3** with hydrazine or phenylhydrazine under solvent-free condition. Both the method and the 10-amino-9-aryl-2,3,4,5,6,7,9,10-octahydro-acridine-1,8-dione derivatives have not been reported. The xanthene derivatives **3** are converted to acridine derivatives **4** by replacing the oxygen atoms with the nitrogen atoms bearing amino; the absorption wavelength of acridine derivatives **4** occurs red shift, and the photoluminescent spectra of the title compounds in dichloromethane solution are of particular interest. Only the fluorescence of **4e** and **4g** is detected, and both of them show good fluorescence properties.

EXPERIMENTAL

Melting points were determined on an electrothermal apparatus, and the temperature was not calibrated. Microanalysis was performed by the Perkin-Elmer 2400 Microanalytical Service. Infrared spectra were recorded as thin films on KBr using a Perking-Elmer 1700 spectrophotometer. The ^{1}H-NMR spectra were recorded on a Bruker ARX-300 spectrometer. Sample solutions were prepared in $CDCl_3$ or DMSO-d_6 containing TMS as an internal standard. Mass spectra were recorded by JMS-DX300 at 70 eV.

All chemical reagents were commercially available and purified with standard methods before use. Solvents were dried in routine ways and redistilled. The 5-substituted-1,3-cyclohexanedione **1** was prepared as building block from aromatic aldehyde, acetone, and diethyl malonate [19a]. A mixture of 5-substituted-1,3-cyclohexanedione (**1**, 10mmol), aromatic aldehyde (**2**, 5mmol), and H_2SO_4 (0.1 mL) in water (40 mL) was stirred at 70–80°C for 2 h [19b] and then the mixture was cooled to rt. The solid (the crude product **3**) was filtered off and washed with water. The mixture of crude product **3** and hydrazine (5 mL) or phenylhydrazine (5 mL) was stirred at 80–90°C, and the reaction mixture was cooled to rt when the reaction was over by TLC analysis. The solid (the crude title compounds **4**) was filtered off, washed with water, and purified with silica gel flash chromatography using ethyl acetate/hexane mixture (1:2) as an eluent to give pure compounds.

Data of compounds are shown as follows: **4a**: Yield: 69%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 0.96 (s, 6H, 2×CH_3), 1.12 (s, 6H, 2×CH_3), 2.13–2.26 (m, 4H, C^4+C^5-H), 2.51–2.88 (m, 4H, C^2+C^7-H), 4.13 (br s, 2H, N—H), 5.27 (s, 1H, C^9-H), 7.35–7.96 (m, 4H, Ph-H); IR (KBr) ν: 3350 (NH), 1625 (C=O); MS (70 eV) *m/z* (%): 410.2 (M+1, 100); Anal. calcd for $C_{23}H_{27}N_3O_4$: C, 67.46; H, 6.65; N, 10.26. Found: C, 67.50; H, 6.71; N, 10.35.

4b: Yield: 68%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 0.96 (s, 6H, 2×CH_3), 1.12 (s, 6H, 2×CH_3), 2.14–2.25 (m, 4H, C^4+C^5-H), 2.47–2.83 (m, 4H, C^2+C^7-H), 4.02 (br s, 2H, N—H), 5.15 (s, 1H, C^9-H), 7.13–7.23 (m, 4H, Ph-H); IR (KBr) ν: 3210 (NH), 1580 (C=O); MS (70 eV) *m/z* (%): 399.2 (M+1, 100); Anal. calcd for $C_{23}H_{27}ClN_2O_2$: C, 69.25; H, 6.82; N, 7.02. Found: C, 69.16; H, 6.75; N, 7.10.

4c: Yield: 71%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 0.97 (s, 6H, 2×CH_3), 1.11 (s, 6H, 2×CH_3), 2.13–2.26 (m, 4H, C^4+C^5-H), 2.47–2.83 (m, 4H, C^2+C^7-H), 4.01 (br s, 2H, N—H), 5.20 (s, 1H, C^9-H), 7.06–7.28 (m, 5H, Ph-H); IR (KBr) ν: 3331 (NH), 1629 (C=O); MS (70 eV) *m/z* (%): 365.2 (M+1, 100); Anal. calcd for $C_{23}H_{28}N_2O_2$: C, 75.79; H, 7.74; N, 7.69. Found: C, 77.70; H, 7.67; N, 7.73.

4d: Yield: 63%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 0.97 (s, 6H, 2×CH_3), 1.11 (s, 6H, 2×CH_3), 2.14–2.21 (m, 4H, C^4+C^5-H), 2.47–2.81 (m, 4H, C^2+C^7-H), 4.05 (br s, 2H, N—H), 5.34 (s, 1H, C^9-H), 7.02–7.21 (m, 4H, Ph-H); IR (KBr) ν: 3213 (NH), 1565 (C=O); MS (70 eV) *m/z* (%): 399.2 (M+1, 100); Anal. calcd for $C_{23}H_{27}ClN_2O_2$: C, 69.25; H, 6.82; N, 7.02. Found: C, 69.35; H, 6.90; N, 7.11.

4e: Yield: 65%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 1.01 (s, 6H, 2×CH_3), 1.11 (s, 6H, 2×CH_3), 2.14–2.22 (m, 4H, C^4+C^5-H), 2.41–2.52 (m, 4H, C^2+C^7-H), 3.80 (s, 3H, OCH_3), 3.96 (s, 3H, OCH_3), 4.22 (br s, 2H, N—H), 4.70 (s, 1H, C^9-H), 6.70–6.94 (m, 3H, Ph-H); IR (KBr) ν: 3242 (NH), 1596 (C=O); MS (70 eV) *m/z* (%): 425.3 (M+1, 100); Anal. calcd for $C_{25}H_{32}N_2O_4$: C, 70.73; H, 7.60; N, 6.60. Found: C, 70.65; H, 7.69; N, 6.68.

4f: Yield: 58%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.52–2.71 (m, 4H, C^4+C^5-H), 3.25–3.42 (m, 4H, C^2+C^7-H), 3.47–3.82 (m, 2H, C^3+C^6-H), 4.09 (br s, 2H, N—H), 5.35 and 5.39 (each s, 1H, C^9-H), 7.15–7.43 (m, 15H, Ph-H); IR (KBr) ν: 3348 (NH), 1660 (C=O); MS (70 eV) *m/z* (%): 461.3 (M+1, 100); Anal. calcd for $C_{31}H_{28}N_2O_2$: C, 80.84; H, 6.13; N, 6.08. Found: C, 80.89; H, 6.07; N, 6.15.

4g: Yield: 65%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.51–2.70 (m, 4H, C^4+C^5-H), 2.76–2.96 (m, 4H, C^2+C^7-H), 3.27–3.52 (m, 2H, C^3+C^6-H), 4.14 (br s, 2H, N—H), 5.12 and 5.13 (each s, 1H, C^9-H), 7.06–7.49 (m, 14H, Ph-H); IR (KBr) ν: 3296 (NH), 1561 (C=O); MS (70 eV) *m/z* (%): 495.2 (M+1, 100); Anal. calcd for $C_{31}H_{27}ClN_2O_2$: C, 75.22; H, 5.50; N, 5.66. Found: C, 75.30; H, 5.60; N, 5.10.

4h: Yield: 61%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.56–2.80 (m, 4H, C^4+C^5-H), 2.86–3.00 (m, 4H, C^2+C^7-H), 3.30–3.53 (m, 2H, C^3+C^6-H), 4.06 (br s, 2H, N—H), 4.96 and 4.98 (each s, 1H, C^9-H), 7.17–7.98 (m, 14H, Ph-H); IR (KBr) ν: 3255 (NH), 1593 (C=O); MS (70 eV) *m/z* (%): 506.2 (M+1, 100); Anal. calcd for $C_{31}H_{27}N_3O_4$: C, 73.65; H, 5.38; N, 8.31. Found: C, 73.58; H, 5.45; N, 8.25.Y **4i**: Yield: 71%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.52–2.71 (m, 4H, C^4+C^5-H), 3.14–3.41 (m, 4H, C^2+C^7-H), 3.43–3.58 (m, 2H, C^3+C^6-H), 3.77 (s, 3H, OCH_3), 3.82 (s, 3H, OCH_3), 4.06 (br s, 2H, N—H), 5.30 and 5.34 (each s, 1H, C^9-H), 7.07–7.46 (m, 13H, Ph-H); IR (KBr) ν: 3325 (NH), 1653 (C=O); MS (70 eV) *m/z* (%): 521.3 (M+1, 100); Anal. calcd for $C_{33}H_{32}N_2O_4$: C, 76.13; H, 6.20; N, 5.38. Found: C, 76.23; H, 6.13; N, 5.42.

4j: Yield: 82%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.22–2.39 and 2.58–2.87 (m, 8H, C^4+C^5+C^2+C^7-H), 3.13–3.35 (m, 2H, C^3+C^6-H), 5.18 (br s, 1H, N—H), 5.55 and 5.60 (each s, 1H, C^9-H), 6.87–7.64 (m, 20H, Ph-H); IR (KBr) ν: 3212 (NH), 1650 (C=O); MS (70 eV) *m/z* (%): 537.3 (M+1, 100); Anal. calcd for $C_{37}H_{32}N_2O_2$: C, 82.81; H, 6.01; N, 5.22. Found: C, 82.75; H, 6.09; N, 5.30.

4k: Yield: 80%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.25–2.40 and 2.62–2.92 (m, 8H, C^4+C^5+C^2+ C^7-H), 3.10–3.36 (m, 2H, C^3+C^6-H), 5.09 (br s, 1H, N—H), 5.69 and 5.70 (each s, 1H, C^9-H), 6.86–7.41 (m, 19H, Ph-H); IR (KBr) ν: 3306 (NH), 1642 (C=O); MS (70 eV) *m/z* (%): 571.3 (M+1, 100); Anal. calcd for $C_{37}H_{31}ClN_2O_2$: C, 77.81; H, 5.47; N, 4.91. Found: C, 77.75; H, 5.53; N, 4.98.

4l: Yield: 76%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.13–2.38 (m, 4H, C^4+C^5-H), 2.60–2.89 (m, 4H, C^2+C^7-H), 3.12–3.36 (m, 2H, C^3+C^6-H), 3.73 (s, 6H, 2×OCH_3), 4.99 (br s, 2H, N—H), 5.55 and 5.57 (each s, 1H, C^9-H), 6.69–7.31 (m, 12H, Ph-H); IR (KBr) ν: 3403 (NH), 1663 (C=O); MS (70 eV) *m/z* (%): 555.2 (M+1, 100); Anal. calcd for $C_{33}H_{31}ClN_2O_4$: C, 71.41; H, 5.63; N, 5.05. Found: C, 71.35; H, 5.66; N, 5.15.

4m: Yield: 83%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.20–2.38 (m, 4H, C^4+C^5-H), 2.58–2.88 (m, 4H, C^2+C^7-H), 3.12–3.30 (m, 2H, C^3+C^6-H), 3.75 (s, 3H, OCH_3), 3.77 (s, 3H, OCH_3), 5.01 (br s, 2H, N—H), 5.58 and 5.60 (each s, 1H, C^9-H), 6.69–7.50 (m, 12H, Ph-H); IR (KBr) ν: 3340 (NH), 1639 (C=O); MS (70 eV) *m/z* (%): 566.3 (M+1, 100); Anal. calcd for $C_{33}H_{31}N_3O_6$: C, 70.07; H, 5.52; N, 7.43. Found: C, 70.03; H, 5.58; N, 7.35.

4n: Yield: 74%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.25–2.42 (m, 4H, C^4+C^5-H), 2.65–2.93 (m, 4H, C^2+C^7-H), 3.11–3.35 (m, 2H, C^3+C^6-H), 3.81 (s, 3H, OCH_3), 3.82 (s, 3H, OCH_3), 4.55 (br s, 2H, N—H), 5.63 and 5.65 (each s, 1H, C^9-H), 6.78–7.43 (m, 12H, Ph-H); IR (KBr) ν: 3484 (NH), 1656 (C=O); MS (70 eV) *m/z* (%): 555.2 (M+1, 100); Anal. calcd for $C_{33}H_{31}ClN_2O_4$: C, 71.41; H, 5.63; N, 5.05. Found: C, 71.35; H, 5.69; N, 5.13.Y **4o**: Yield: 50%; ^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 2.25–2.40 (m, 4H, C^4+C^5-H), 2.62–2.90 (m, 4H, C^2+C^7-H), 3.14–3.38 (m, 2H, C^3+C^6-H), 3.75 (s, 3H, OCH_3), 3.76 (s, 3H, OCH_3), 3.79 (s, 3H, OCH_3), 3.81 (s, 3H, OCH_3), 4.55 (br s, 2H, N—H), 5.61 and 5.62 (each s, 1H, C^9-H), 6.78–7.43 (m, 12H, Ph-H); IR (KBr) ν: 3347 (NH), 1598 (C=O); MS (70 eV) *m/z* (%): 581.3 (M+1, 100); Anal. calcd for $C_{35}H_{36}N_2O_6$: C, 72.39; H, 6.25; N, 4.82. Found: C, 72.35; H, 6.33; N, 4.75.

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