Improved and Efficient Synthesis of Pyrrolo[2,3,4-*kl*]acridin-1-one Derivatives under Ultrasound Irradiation

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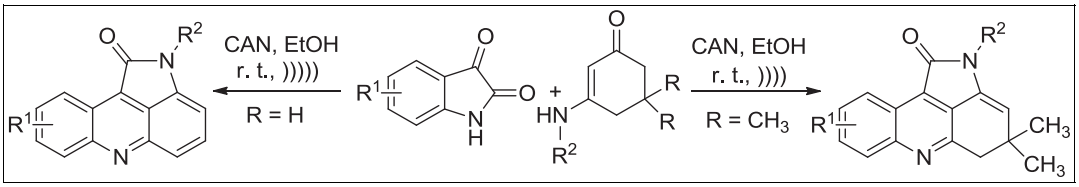
Received September 9, 2012

DOI 10.1002/jhet.1882

Published online 00 Month 2014 in Wiley Online Library (wileyonlinelibrary.com).



A improved and efficient procedure for the synthesis of pyrrolo[2,3,4-*kl*]acridin-1-one derivatives via the reaction of isatin and enaminone catalyzed by ceric ammonium nitrate under ultrasonic condition has been developed. Compared with the conventional methods, the remarkable advantages of this method are mild reaction conditions, operational simplicity, higher yield, and shorter reaction times.

J Hetercyclic Chem., **00,** 00 (2014).

INTRODUCTION

Nitrogen-containing heterocyclic compounds are widespread in natural products and medicinal agents [1], and their applications to biologically active pharmaceuticals, agrochemicals, and functional materials are becoming more and more important [2]. Among them, acridine derivatives are very interesting compounds and have received considerable attention as a result of their biological activity and an interesting template for medicinal chemistry. Many of those compounds are known as antiparasitic [3], antibacterial [4], and antitumor activity [5]. Pyrrolo[2,3,4-*kl*] acridin-1-ones are pyrrole-fused acridine derivatives, which are the skeleton of some alkaloids [6]. Recently, there have been studies on the synthesis of these molecules [7]. However, these methods require multistep synthesis. Thus, there is a need for the development of concise and efficient methods for the constructing of this heterocyclic skeleton library.

Ceric ammonium nitrate (CAN) is an orange solid and commercially available. It has been widely used in industry and academia during the past seven decades. In academia, CAN is explored extensively in organic reactions. Representative examples include oxidation [8], oxidative addition [9], photooxidation [10], nitration [11], deprotection [12], graft polymerization [13], and so forth. Intermediates formed in these reactions may undergo oxidative fragmentation [14], rearrangement [15], or cleavages of C—H and C—C bonds [16]. Recently, CAN have been used in catalyzing multicomponent reactions [17].

Ultrasonic irradiation [18], as a powerful tool in modern chemistry for the organic reactions, has attracted more attention of chemists in the recent two decades. The ultrasonic irradiation with its advantages of convenient operation, milder reaction conditions, short reaction time, and high efficiency has become particularly popular in recent years, and numerous examples under this condition for constructing heterocycles with interesting properties have been reported in the literature [19]. As a part of our interest in the synthesis of heterocyclic compounds under ultrasound irradiation [20], herein, we report the facile synthesis of pyrrolo[2,3,4-*kl*]acridin-1-one derivatives via the reaction of isatin and enaminone catalyzed by CAN under ultrasonic condition.

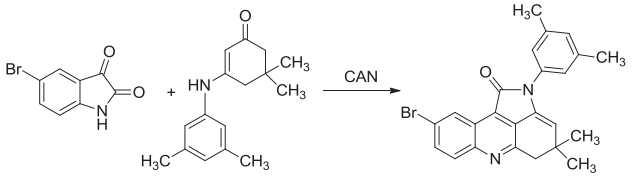
RESULTS AND DISCUSSION

To achieve suitable conditions for the synthesis of pyrrolo[2,3,4-*kl*]acridin-1-ones, various reaction conditions have been investigated in the reaction of 5-bromoisatin (**1i**) and 3-(3,5-dimethylphenylamino)-5,5-dimethylcyclohex-2-enone (**2i**) as a model reaction (Scheme 1).

Initially, we examined the effects of the solvents through some experiments. To search for the optimal solvent, the ultrasonic-assisted reaction was examined using ethanol, methanol, THF, acetonitrile, dioxane, toluene, and water as solvent. The results were summarized in Table 1. The observation revealed that the reaction using ethanol as solvent gave the best result, which afforded the product not only in good yield but also with higher reaction rates (94% yield in 1.5 h) (Table 1, entry 1). Methanol, THF, acetonitrile, dioxane, and toluene afforded moderate yields of desired products but took comparatively longer reaction time (Table 1, entries 2–6). When the reaction was performed in water, unfortunately, the desired product was not detected (Table 1, entry 7). Thus, ethanol was chosen as the solvent for all further reactions.

To optimize the catalyst loading, 5, 10, 15, and 20 mol% of CAN was tested, respectively. The results are summarized in Table 2. A 10 mol% loading of CAN was sufficient

Scheme 1. Modle reaction. CAN, ceric ammonium nitrate.



1i 2i 3i

Table 1

Optimization the solvents for the synthesis of **3i**.

		With US[a]		Without US[b]	
Entry	Solvent	Time (h)	Yield[c] (%)	Time (h)	Yield[c] (%)
1	Ethanol	1.5	94	4	80
2	Methanol	2	82	4.5	62
3	THF	3	70	6	68
4	Acetonitrile	3.5	72	7	70
5	Dioxane	4	75	10	60
6	Toluene	2.5	80	5	76
7	Water	11	NR	24	NR

US, ultrasound
NR, no reaction.
[a]Reaction under ultrasonic waves at RT.
[b]Reaction under refluxing temperature.
[c]Yields of isolated products.

Table 2

Effect of the amount of catalyst for the synthesis of **3i**.

Entry	Amounts of CAN (mol%)	Time (h)	Isolated yield (%)
1	5	3	85
2	10	1.5	94
3	15	2	90
4	20	2	87

CAN, ceric ammonium nitrate.

to push the reaction forward, and 5 mol% of CAN was not enough. Higher amounts of CAN did not lead to significant change in the reaction yields.

After optimizing the reaction conditions, the generality of this method was examined by the reaction of several substituted isatins and enaminone in ethanol under ultrasound irradiation; the results are shown in Table 3. To our delight, under the aforementioned optimized conditions, the reactions proceeded smoothly, and a variety of the desired pyrrolo [2,3,4-*kl*]acridin-1-one derivatives **3** were obtained in good yields whether the isatin (**1**) or enaminone (**2**) are the bearing electron-withdrawing substituents or electron-donating substituents (Table 3, entries 1–11). To the best of our knowledge, this new procedure provides the first example of an efficient and ultrasound-promoted one-pot approach for synthesis of pyrrolo[2,3,4-*kl*]acridin-1-one derivatives.

To find the specific effect of ultrasound on this reaction, all previously mentioned were carried out under the same conditions in the absence of ultrasound irradiation (Table 3). When the reaction was carried out under conventional method, it gave comparatively low yields of products and took longer reaction time, whereas the same reaction carried in the influence of ultrasonic irradiation gave excellent yields of product in short reaction time. Thus, ultrasonic irradiation was found to have beneficial effect on the synthesis of pyrrolo[2,3,4-*kl*]acridin-1-one derivatives that was superior to the traditional method with respect to yield, reaction time, particularly when considering the basic green chemistry concept. It is apparent that ultrasound irradiation can accelerate the reaction significantly to give higher yield. The reason may be the phenomenon of cavitation produced by ultrasound. The effects of ultrasound observed during organic reaction are due to cavitation. It has been experimentally shown that, the cavitational collapse creates drastic conditions inside the medium for an extremely short time and temperatures of 2000–5000 K, pressures up to

Table 3

Synthesis of **3** under sonication and conventional conditions.

				With US		Without US	
Entry	Product	R^1	R^2	Time (h)	Yield[a](%)	Time (h)	Yield[a](%)
1	**3a**	H	4-$CH_3C_6H_4$	0.8	91	3	82
2	**3b**	H	2-ClC_6H_4	1.5	90	2.5	76
3	**3c**	5-F	4-$CH_3OC_6H_4$	0.8	91	3	80
4	**3d**	5-Cl	$CH_3(CH_2)_3$	1.5	89	3.5	80
5	**3e**	5-Cl	4-$NO_2C_6H_4$	1.5	84	4	65
6	**3f**	5-Cl	4-$(CH_3)_3CC_6H_4$	0.7	92	2	76
7	**3g**	6-Cl	2-ClC_6H_4	0.8	92	2.5	74
8	**3h**	5-Br	4-$CH_3OC_6H_4$	1	90	3	82
9	**3i**	5-Br	3,5-$(CH_3)_2C_6H_3$	1.5	94	2.5	79
10	**3j**	5-CH_3	C_6H_5	1	90	3	78
11	**3k**	7-CF_3	Naphthalene-1-yl	1	86	3.5	69

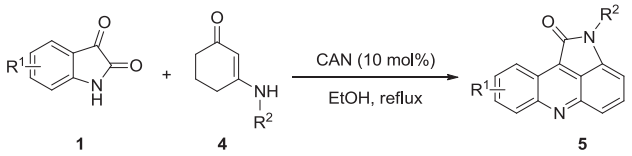
[a]Yields of isolated products.
[b]US, ultrasound.

1800 atm inside the collapsing cavity have been produced under sonic condition [21]. In addition, ultrasound is known to generate extremely fine emulsions from mixtures of immiscible phases. On closer inspection, this disturbance can be seen as a large number of tiny "explosions" at the interface, effectively sending small jets of liquid from one phase into the other. One of the main consequences of these emulsions is the dramatic increase in the interfacial contact area between the immiscible phases, that is, an increase in the region over which any reaction between species dissolved in the different phases can take place [22].

To expand the scope of the current method, *N*-substituted 3-aminocyclohex-2-enone (**4**) was examined as a replacement for the *N*-substituted 3-amino-5,5-dimethylcyclohex-2-enone (**2**). To our surprise, under the aforementioned optimized conditions, the desired products 4,5-dihydropyrrolo-[2,3,4-*kl*]acridin-1-one derivatives **3** were not obtained. Its oxidation products pyrrolo[2,3,4-*kl*]-acridin-1-one derivatives **5** were obtained instead in good yields (Scheme 2 and Table 4). So, the reaction pathways could therefore be controlled by varying the enaminones with a different substituted pattern to give a series of novel 4,5-dihydropyrrolo[2,3,4-*kl*]acridin-1-ones and pyrrolo [2,3,4-*kl*]-acridin-1-ones selectively.

The structures of the products **3** and **5** were identified from their IR, ^{1}H-NMR, ^{13}C-NMR, and HRMS spectra. The structure of compound **3a** was further confirmed by X-ray analysis (Figure 1).

Scheme 2. The synthesis of compounds **5** under sonication and conventional conditions. CAN, ceric ammonium nitrate.



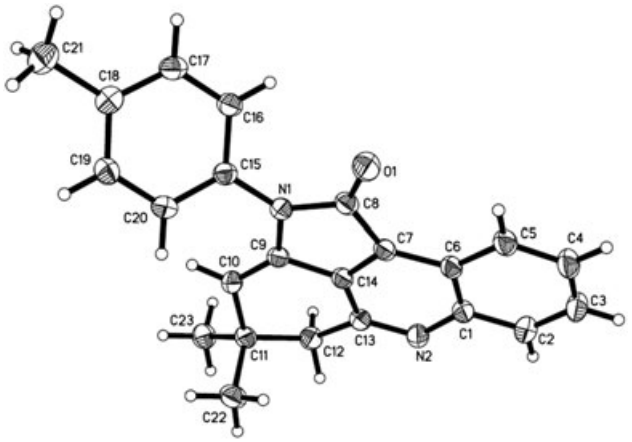


Figure 1. The structure of compound **3a**.

According to the literature [23], we proposed the plausible mechanism for the formation of pyrrolo[2,3,4-*kl*]acridine derivatives **3** (Scheme 3). The first step involves the formation of intermediate A by the nucleophilic addition of enaminone (**2**) to isatin (**1**) catalyzed by CAN. The intermediate A was followed by imine–enamine tautomerization to produce B. Intermediate B, through intramolecular cyclization and ring-opening reactions formed D. Subsequently, the NH_2 attacked the intramolecular carbonyl group catalyzed by CAN to form intermediate E, which, through losing water, produced F. Finally, F lost a molecule water to result in the product **3**.

In conclusion, we have described an approach to explore the use of ultrasound irradiation for the synthesis of pyrrolo [2,3,4-*kl*]acridin-1-one derivatives via domino reaction of isatin and enaminone catalyzed by CAN. Compared with traditional methods, the procedure offers several advantages including milder reaction conditions, excellent yields, and shorter reaction time.

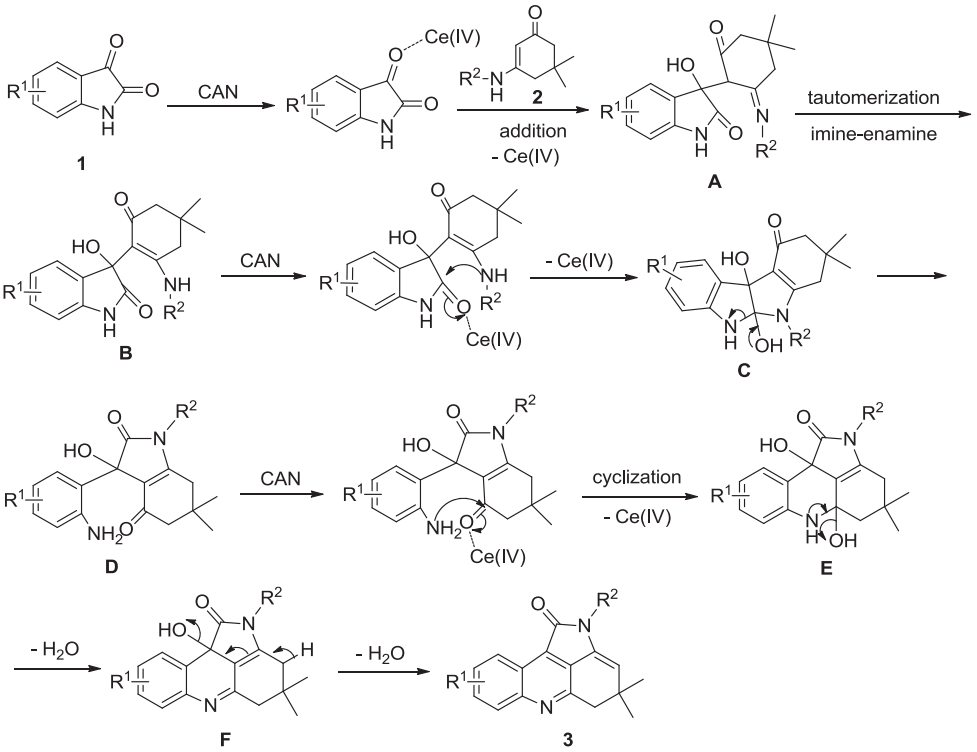
Table 4

Synthesis of **5** under sonication and conventional conditions.

				With US		Without US	
Entry	Product	R^1	R^2	Time (h)	Yield[a](%)	Time (h)	Yield[a](%)
1	**5a**	H	*n*-C_4H_9	1.5	70	3.5	56
2	**5b**	H	4-$NO_2C_6H_4$	2	56	4.5	35
3	**5c**	5-Cl	C_6H_5	1	77	4	66
4	**5d**	5-Cl	4-ClC_6H_4	2	72	4	58
5	**5e**	5-Cl	4-$CH_3C_6H_4$	1.5	74	4	65
6	**5f**	5-Cl	Naphthalene-1-yl	1	70	3	52
7	**5g**	5-Br	4-*t*-BuC_6H_4	1	81	3	65
8	**5h**	5-Br	3,5-$(CH_3)_2C_6H_3$	1.5	75	4	61
9	**5i**	5-CH_3	3,5-$(CH_3)_2C_6H_3$	1	78	3	64
10	**5j**	5-F	4-$CH_3OC_6H_4$	1.5	80	4	62
11	**5k**	7-CF_3	2,6-$Cl_2C_6H_3$	1	72	3.5	62

[a]Yields of isolated products.

Scheme 3. Proposed mechanism for the formation of **3**. CAN, ceric ammonium nitrate.



EXPERIMENTAL

All reagents were purchased from commerical sources and used without further purification. Ultrasonication was performed in a KQ-250E medical ultrasound cleaner (Kunshan Ultrasonic Instruments Co., Ltd, Kunshan, China) with a frequency of 40 kHz and an output power of 250 W (Built-in heating, 30–110°C thermostatically adjustable). Melting points are uncorrected. IR spectra were recorded on Varian F-1000 spectrometer (Varian Inc., Palo Alto, CA, USA) in KBr with absorptions in reciprocal centimeters. ^{1}H-NMR and ^{13}C-NMR were determined on Varian Inova-300 or Inova-400 MHz spectrometers (Varian Inc., Palo Alto, CA, USA) in $CDCl_3$ solution. *J* values are in hertz. Chemical shifts are expressed in parts per million downfield from internal standard TMS. HRMS data were obtained using Bruker micrOTOF-Q instrument (Bruker Daltonics Inc., Billerica, MA, USA). X-Ray diffraction was recorded on a Rigaku Mercury CCD/AFC diffractometer (Rigaku Corporation, Tokyo, Japan). The reaction flask was located at the maximum energy area in the cleaner; the surface of reactants is slightly lower than the leave of the water.

General procedure for the synthesis of pyrrolo[2,3,4-*kl*] acridin-1-ones (3 and 5) under conventional conditions (method A). A 100-mL flask was charged with isatin (**1**) (1 mmol), enaminone **2** or **4** (1 mmol), and CAN (0.1 mmol) in ethanol (10 mL). The mixture was stirred at refluxing temperature. After the completion of the reaction (monitored by TLC), the solvent was evaporated under reduced pressure, and the residue was purified by column chromatography using petrolum ether–acetone (6:1) as eluent to produce the desired corresponding products **3** or **5**.

General procedure for the synthesis of pyrrolo[2,3,4-*kl*] acridin-1-ones (3 and 5) under sonochemical conditions (method B). A 100-mL flask was charged with isatin (**1**) (1 mmol), enaminone **2** or **4** (1 mmol), and CAN (0.1 mmol) in ethanol (10 mL). The mixture was sonicated in the water bath of an ultrasonic cleaner under air condition at RT. After the completion of the reaction (monitored by TLC), the solvent was evaporated under reduced pressure, and the residue was purified by column chromatography using petrolum ether–acetone (6:1) as eluent to produce the desired corresponding products **3** or **5**.

4,4-Dimethyl-2-(4-methylphenyl)-4,5-dihydropyrrolo[2,3,4-kl] acridin-1(2H)-one (3a). yellow solid; mp 176–178°C; IR (KBr, cm^{-1}): 3031, 2941, 1705, 1491, 1341, 1107, 817; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.32 (s, 6H, 2 × CH_3), 2.44 (s, 3H, CH_3), 3.21 (s, 2H, CH_2), 5.59 (s, 1H, CH), 7.37 (s, 4H, ArH), 7.63–7.78 (m, 2H, ArH), 8.17 (d, *J* = 8.4 Hz, 1H, ArH), 8.73 (d, *J* = 7.8 Hz, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 21.2, 30.9, 37.0, 44.2, 118.2, 122.6, 124.2, 125.0, 126.3, 126.4, 127.7, 129.4, 129.5, 130.5, 132.0, 133.5, 137.5, 149.7, 154.6, 166.8; HRMS Calcd for $C_{23}H_{20}N_2ONa$ [M + Na]: 363.1473, found: 363.1479.

2-(2-Chlorophenyl)-4,4-dimethyl-4,5-dihydropyrrolo[2,3,4-kl] acridin-1(2H)-one (3b). gray solid; mp 136–138°C; IR (KBr, cm^{-1}): 2966, 2866, 1715, 1655, 1587, 1484, 1376, 1349, 1246, 1138, 1090, 1059, 962, 859, 836, 771, 739, 674; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.31 (d, *J* = 7.2 Hz, 6H, 2 × CH_3), 3.22 (d, *J* = 7.8 Hz, 2H, CH_2), 5.29 (t, *J* = 10.8 Hz, 1H, CH), 7.45–7.47 (m, 3H, ArH), 7.62–7.76 (m, 3H, ArH), 8.20 (d, *J* = 9.3 Hz, 1H, ArH), 8.73 (d, *J* = 7.5 Hz, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 30.8, 37.1, 44.2, 118.1, 122.6, 124.3, 124.9, 126.9, 127.8, 129.4, 129.5, 130.2, 130.7, 132.3, 132.9, 133.3, 149.6, 154.7, 166.6; HRMS Calcd for $C_{22}H_{18}ClN_2O$ [M + H]: 361.1108, found: 361.1103.

2-Butyl-9-chloro-4,4-dimethyl-4,5-dihydropyrrolo[2,3,4-kl] acridin-1(2H)-one (3c). yellow solid; mp 144–146°C; IR (KBr, cm^{-1}): 2935, 1701, 1655, 1510, 1446, 1363, 1335,

1288, 1091, 964, 832, 795, 739, 719; ^{1}H-NMR (300 MHz, $CDCl_3$) δ (ppm) 0.98 (d, J = 6.9 Hz, 3H, CH_3), 1.25–1.50 (m, 8H, CH_2 and 2 × CH_3), 1.60–1.76 (m, 2H, CH_2), 3.14 (s, 2H, CH_2), 3.79 (s, 2H, CH_2), 5.55 (s, 1H, CH), 7.63 (d, J = 6.6 Hz, 1H, ArH), 8.03 (d, J = 8.7 Hz, 1H, ArH), 8.64 (s, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 9.2, 15.6, 26.4, 26.5, 32.5, 35.4, 39.5, 112.9, 118.6, 118.7, 120.3, 122.4, 125.5, 126.1, 128.4, 129.0, 143.3, 150.0, 162.4; HRMS Calcd for $C_{20}H_{22}ClN_2O$ [M + H]: 341.1421, found: 341.1426.

2-(4-tert-Butylphenyl)-9-chloro-4,4-dimethyl-4,5-dihydropyrrolo [2,3,4-kl]acridin-1(2H)-one (3d). yellow solid; mp 180–182°C; IR (KBr, cm^{-1}): 2952, 2865, 1703, 1652, 1602, 1460, 1417, 1340, 1292, 1205, 1151, 1119, 1093, 963, 841, 750, 656; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.33 (s, 6H, 2 × CH_3), 1.39 (s, 9H, C$(CH_3)_3$), 3.20 (s, 2H, CH_2), 5.69 (s, 1H, CH), 7.42 (d, J = 6.6 Hz, 2H, ArH), 7.56 (d, J = 7.8 Hz, 2H, ArH), 7.69 (d, J = 7.2 Hz, 1H, ArH), 8.09 (d, J = 9.0 Hz, 1H, ArH), 8.71 (s, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 31.4, 31.9, 35.3, 37.7, 44.6, 119.8, 123.7, 123.8, 124.8, 126.3, 126.9, 127.5, 130.8, 131.3, 132.3, 133.7, 134.3, 148.5, 151.1, 155.4, 166.8; HRMS Calcd for $C_{26}H_{26}ClN_2O$ [M + H]: 417.1734, found: 417.1726.

9-Chloro-4,4-dimethyl-2-(4-nitrophenyl)-4,5-dihydropyrrolo [2,3,4-kl]acridin-1(2H)-one (3e). red solid; mp 178–180°C; IR (KBr, cm^{-1}): 2962, 2867, 1735, 1633, 1594, 1521, 1499, 1401, 1302, 1241, 1181, 1145, 1113, 1065, 1018, 860, 832, 784, 767, 733, 691, 663; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.37 (s, 6H, 2 × CH_3), 3.31 (s, 2H, CH_2), 5.82 (s, 1H, CH), 7.77 (d, J = 8.4 Hz, 3H, ArH), 8.12 (d, J = 7.2 Hz, 1H, ArH), 8.43 (d, J = 8.7 Hz, 2H, ArH), 8.91 (d, J = 7.5 Hz, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 30.8, 37.3, 44.1, 120.4, 122.8, 124.0, 124.9, 126.1, 126.7, 127.8, 127.9, 128.2, 131.8, 140.5, 146.0, 146.1, 155.5, 165.9; HRMS Calcd for $C_{22}H_{17}ClN_3O_3$ [M + H]: 406.0958, found: 406.0955.

9-Fluoro-2-(4-methoxyphenyl)-4,4-dimethyl-4,5-dihydropyrrolo [2,3,4-kl]acridin-1(2H)-one (3f). yellow solid; mp 164–166°C; IR (KBr, cm^{-1}): 2956, 1709, 1651, 1620, 1453, 1419, 1346, 1252, 1215, 1119, 1035, 879, 794, 701; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.32 (s, 6H, 2 × CH_3), 3.19 (s, 2H, CH_2), 3.88 (s, 3H, CH_3O), 5.58 (s, 1H, CH), 7.06 (d, J = 6.6 Hz, 2H, ArH), 7.35–7.67 (m, 3H, ArH), 8.17 (d, J = 5.4 Hz, 1H, ArH), 8.33 (d, J = 8.7 Hz, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 26.3, 32.6, 39.4, 51.0, 103.6, 103.8, 110.1, 114.2, 114.5, 114.8, 118.8, 120.2, 122.3, 122.6, 123.2, 127.0, 127.1, 129.1, 142.1, 149.2, 154.3, 162.0; HRMS Calcd for $C_{23}H_{20}FN_2O_2$ [M + H]: 375.1509, found: 375.1505.

9-Bromo-2-(4-methoxyphenyl)-4,4-dimethyl-4,5-dihydropyrrolo [2,3,4-kl]acridin-1(2H)-one (3g). yellow solid; mp 140–142°C; IR (KBr, cm^{-1}): 2961, 2806, 1705, 1650, 1602, 1508, 1464, 1376, 1249, 1153, 1136, 1062, 1028, 834, 797, 775; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.33 (s, 6H, 2 × CH_3), 3.19 (s, 2H, CH_2), 3.89 (s, 3H, CH_3O), 5.60 (s, 1H, CH), 7.07 (d, J = 4.8 Hz, 2H, ArH), 7.40 (d, J = 4.8 Hz, 2H, ArH), 7.83 (d, J = 6.9 Hz, 1H, ArH), 8.03 (d, J = 8.4 Hz, 1H, ArH), 8.88 (s, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 27.7, 30.9, 37.1, 44.0, 55.5, 114.6, 119.0, 122.0, 123.5, 124.0, 126.4, 126.8, 127.1, 127.7, 127.8, 130.8, 132.9, 133.5, 148.1, 155.0, 158.8, 166.3; HRMS Calcd for $C_{23}H_{20}BrN_2O_2$ [M + H]: 435.0708, found: 435.0702.

9-Bromo-2-(3,5-dimethylphenyl)-4,4-dimethyl-4,5-dihydropyrrolo [2,3,4-kl]acridin-1(2H)-one (3h). yellow solid; mp 160–162°C; IR (KBr, cm^{-1}): 2957, 1700, 1650, 1596, 1439, 1415, 1366, 1314, 1211, 1150, 1098, 967, 891, 847, 825, 792, 748, 689; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.33 (s, 6H, 2 × CH_3), 2.41 (s, 6H, 2 × CH_3), 3.18 (s, 2H, CH_2), 5.64 (s, 1H, CH), 7.08 (d, J = 4.8 Hz, 3H, ArH), 7.82 (d, J = 9.0 Hz, 1H, ArH), 8.02 (d, J = 9.0 Hz, 1H, ArH), 8.88 (s, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 16.8, 26.3, 32.6, 39.6, 114.6, 117.5, 119.1, 119.6, 120.0, 122.0, 122.3, 125.0, 126.3, 128.3, 128.8, 129.7, 134.7, 143.6, 150.5, 161.7; HRMS Calcd for $C_{24}H_{22}BrN_2O$ [M + H]: 433.0915, found: 433.0906.

4,4,9-Trimethyl-2-phenyl-4,5-dihydropyrrolo[2,3,4-kl]acridin-1(2H)-one (3i). yellow solid; mp 168–170°C; IR (KBr, cm^{-1}): 2958, 1712, 1651, 1594, 1511, 1500, 1418, 1401, 1365, 1344, 1249, 1153, 1120, 1095, 1065, 887, 824, 759; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.32 (s, 6H, 2 × CH_3), 2.59 (s, 3H, CH_3), 3.20 (s, 2H, CH_2), 5.61 (s, 1H, CH), 7.41–7.56 (m, 6H, ArH), 8.06 (d, J = 8.4 Hz, 1H, ArH), 8.51 (s, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 22.3, 31.4, 37.6, 44.6, 118.7, 123.1, 123.8, 126.9, 127.0, 127.9, 129.6, 129.9, 132.2, 133.9, 135.4, 138.6, 148.8, 153.9, 167.4; HRMS Calcd for $C_{23}H_{21}N_2O$ [M + H]: 341.1654, found: 341.1677.

8-Chloro-2-(2-chlorophenyl)-4,4-dimethyl-4,5-dihydropyrrolo [2,3,4-kl]acridin-1(2H)-one (3j). yellow solid; mp 162–164 C; IR (KBr, cm^{-1}): 2962, 1715, 1654, 1508, 1484, 1380, 1294, 1162, 1119, 1093, 1065, 901, 883, 848, 767, 653; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.32 (s, 6H, 2 × CH_3), 3.21 (s, 2H, CH_2), 5.31 (s, 1H, CH), 7.45 (s, 3H, ArH), 7.62 (d, J = 6.3 Hz, 2H, ArH), 8.18 (s, 1H, ArH), 8.63 (d, J = 8.4 Hz, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 30.7, 30.8, 37.2, 44.1, 118.7, 121.0, 124.9, 125.3, 126.9, 127.9, 128.7, 130.3, 130.6, 130.7, 132.1, 132.7, 133.2, 135.4, 150.0, 156.0, 166.2; HRMS Calcd for $C_{22}H_{17}Cl_2N_2O$ [M + H]: 395.0718, found: 395.0717.

2-Butylpyrrolo[2,3,4-kl]acridin-1(2H)-one (5a). read solid; mp 110–112°C; IR (KBr, cm^{-1}): 2962, 2872, 1700, 1634, 1513, 1462, 1401, 1362, 1315, 1244, 1145, 1078, 1005, 962, 922, 858, 785, 721; ^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) 0.98 (t, J = 7.2 Hz, 3H, CH_3), 1.46 (dd, J = 15.2, 7.6 Hz, 2H, CH_2), 1.81–1.86 (m, 2H, CH_2), 3.96 (t, J = 7.2 Hz, 2H, CH_2), 6.86 (d, J = 6.8 Hz, 1H, ArH), 7.65 (dd, J_1 = 6.8 Hz, J_2 = 8.8 Hz, 1H, ArH), 7.75–7.79 (m, 2H, ArH), 7.85–7.89 (m, 1H, ArH), 8.38 (d, J = 8.8 Hz, 1H, ArH), 8.84 (d, J = 8.4 Hz, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 14.0, 20.4, 31.1, 40.5, 104.8, 119.9, 122.2, 123.1, 124.3, 128.5, 129.1, 130.6, 130.8, 133.0, 140.5, 146.5, 151.9, 168.2; HRMS Calcd for $C_{18}H_{17}N_2O$ [M + H]: 277.1341, found: 277.1355.

2-(4-Nitrophenyl)pyrrolo[2,3,4-kl]acridin-1(2H)-one (5b). yellow solid; mp >300°C; IR (KBr, cm^{-1}): 2925, 1751, 1687, 1595, 1349, 1116, 852, 772, 720, 657; ^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) 7.18 (d, J = 7.6 Hz, 1H, ArH), 7.75–7.96 (m, 6H, ArH), 8.48 (d, J = 8.0 Hz, 3H, ArH), 8.93 (d, J = 8.8 Hz, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 106.8, 120.2, 123.2, 123.9, 124.2, 125.3, 125.6, 126.9, 130.0, 131.1, 131.2, 132.7, 132.9, 138.6, 141.0, 146.7, 152.2, 166.9; HRMS Calcd for $C_{20}H_{12}N_3O_3$ [M + H]: 342.0879, found: 342.0863.

9-Chloro-2-phenylpyrrolo[2,3,4-kl]acridin-1(2H)-one (5c). yellow solid; mp 206–208 C; IR (KBr, cm^{-1}): 3035, 1700, 1632, 1503, 1382, 1123, 1087, 829, 721; ^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) 7.03 (d, J = 7.2 Hz, 1H, ArH), 7.47 (t, J = 6.8 Hz, 1H, ArH), 7.58–7.72 (m, 5H, ArH), 7.85 (dd, J_1 = 8.8 Hz, J_2 = 7.2 Hz, 2H, ArH), 8.36 (d, J = 9.6 Hz, 1H, ArH), 8.90 (d, J = 2.4 Hz, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 106.5, 120.2, 123.1, 123.5, 124.6, 126.3, 128.1, 129.7, 130.1, 131.2, 131.3, 133.3, 135.3, 140.5, 147.0, 152.4, 167.6; HRMS Calcd for $C_{20}H_{12}ClN_2O$ [M + H]: 331.0638, found: 331.0619.

9-Chloro-2-(4-chlorophenyl)pyrrolo[2,3,4-kl]acridin-1(2H)-one (5d). gray solid; mp 220–222°C; IR (KBr, cm^{-1}): 3096, 1710, 1634, 1499, 1464, 1418, 1380, 1325, 1250, 1156, 1070, 952, 884, 830, 774, 732, 662; ^{1}H-NMR (400 MHz, $CDCl_3$): δ

(ppm) 7.03 (d, $J = 7.2$ Hz, 1H, ArH), 7.58 (s, 4H, ArH), 7.72 (t, $J = 7.6$ Hz, 1H, ArH), 7.85–7.91 (m, 2H, ArH), 8.38 (d, $J = 9.2$ Hz, 1H, ArH), 8.90 (s, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 106.6, 122.7, 123.4, 124.5, 125.8, 126.3, 126.5, 127.8, 129.3, 129.6, 132.1, 133.2, 134.5, 134.6, 140.0, 146.6, 150.2, 166.6; HRMS Calcd for $C_{20}H_{11}Cl_2N_2O$ [M + H]: 365.0248, found: 365.0236.

9-Chloro-2-(4-methylphenyl)pyrrolo[2,3,4-kl]acridin-1(2H)-one (5e). yellow solid; mp 230–232°C; IR (KBr, cm^{-1}): 2993, 1746, 1700, 1632, 1514, 1384, 1126, 938, 827, 777; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 2.47 (s, 3H, CH_3), 6.97 (s, 1H, ArH), 7.27–7.51 (m, 4H, ArH), 7.68 (s, 1H, ArH), 7.83 (s, 2H, ArH), 8.34 (s, 1H, ArH), 8.87 (s, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 106.4, 119.9, 122.5, 122.8, 123.0, 125.7, 126.8, 130.2, 131.9, 132.0, 132.1, 133.1, 135.8, 137.8, 140.2, 146.5, 150.0, 166.7; HRMS Calcd for $C_{21}H_{13}ClN_2ONa$ [M + Na]: 367.0614, found: 367.0602.

9-Chloro-2-(naphthalen-1-yl)pyrrolo[2,3,4-kl]acridin-1(2H)-one (5f). red solid; mp 208–210°C; IR (KBr, cm^{-1}): 2924, 1710, 1631, 1577, 1483, 1403, 1318, 1261, 1154, 1120, 1083, 874, 721; ^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) 6.60 (d, $J = 6.8$ Hz, 1H, ArH), 7.50–7.73 (m, 6H, ArH), 7.89 (d, $J = 8.8$ Hz, 2H, ArH), 7.88–7.90 (m, 2H, ArH), 8.42 (d, $J = 9.6$ Hz, 1H, ArH), 8.94 (d, $J = 2.0$ Hz, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 106.9, 116.6, 118.1, 122.7, 123.2, 125.9, 126.8, 126.9, 127.4, 128.9, 129.9, 130.5, 131.0, 132.3, 132.5, 133.5, 135.0, 136.1, 141.4, 144.8, 146.8, 150.4, 151.0; HRMS Calcd for $C_{24}H_{14}ClN_2O$ [M + H]: 381.0795, found: 381.0790.

9-Bromo-2-(4-tert-butylphenyl)pyrrolo[2,3,4-kl]acridin-1(2H)-one (5g). red solid; mp 208–210°C; IR (KBr, cm^{-1}): 2958, 1711, 1631, 1503, 1460, 1381, 1252, 1154, 1126, 1055, 1007, 888, 823, 776, 720; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 1.40 (s, 9H, $C(CH_3)_3$), 7.01 (d, $J = 4.8$ Hz, 1H, ArH), 7.50–7.68 (m, 5H, ArH), 7.83 (d, $J = 8.7$ Hz, 1H, ArH), 7.95 (d, $J = 8.1$ Hz, 1H, ArH), 8.26 (d, $J = 9.0$ Hz, 1H, ArH), 9.06 (s, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 37.0, 40.5, 112.3, 128.2, 128.9, 130.0, 130.9, 131.9, 132.2, 132.6, 137.5, 137.8, 138.8, 140.0, 145.7, 152.2, 155.7, 156.5, 172.3; HRMS Calcd for $C_{24}H_{20}BrN_2O$ [M + H]: 431.0759, found: 431.0737.

9-Bromo-2-(3,5-dimethylphenyl)pyrrolo[2,3,4-kl]acridin-1(2H)-one (5h). red solid; mp 196–198°C; IR (KBr, cm^{-1}): 2957, 1722, 1598, 1554, 1468, 1404, 1384, 1200, 1076, 883, 779; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 2.43 (s, 6H, $2 \times CH_3$), 6.99 (d, $J = 6.3$ Hz, 1H, ArH), 7.09 (s, 1H, ArH), 7.21 (s, 2H, ArH), 7.70 (d, $J = 6.9$ Hz, 1H, ArH), 7.83 (d, $J = 9.0$ Hz, 1H, ArH), 7.96 (d, $J = 8.4$ Hz, 1H, ArH), 8.26 (d, $J = 9.0$ Hz, 1, ArH), 9.07 (s, 1H, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 21.4, 106.6, 119.7, 122.4, 123.2, 123.5, 124.3, 126.2, 126.4, 129.5, 132.1, 133.1, 134.3, 139.4, 140.1, 146.5, 150.0, 166.6; HRMS Calcd for $C_{22}H_{16}BrN_2O$ [M + H]: 403.0446, found: 403.0425.

2-(3,5-Dimethylphenyl)-9-methylpyrrolo[2,3,4-kl]acridin-1(2H)-one (5i). gray solid; mp 186–188°C; IR (KBr, cm^{-1}): 3012, 1702, 1634, 1595, 1476, 1381, 1337, 1206, 1155, 1079, 922, 825, 775, 687; ^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 2.43 (s, 6H, $2 \times CH_3$), 2.67 (s, 3H, CH_3), 6.98 (d, $J = 6.6$ Hz, 1H, ArH), 7.08 (s, 1H, ArH), 7.20–7.27 (m, 2H, ArH), 7.65 (t, $J = 6.9$ Hz, 1H, ArH), 7.75 (d, $J = 7.5$ Hz, 1H, ArH), 7.86 (d, $J = 9.0$ Hz, 1H, ArH), 8.31 (d, $J = 8.7$ Hz, 1H, ArH), 8.68 (s, 1, ArH); ^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) 21.4, 22.2, 106.0, 119.6, 122.4, 123.1, 123.5, 126.3, 129.3, 130.3, 132.1, 133.1, 134.6, 139.1, 139.8, 145.5, 150.5, 167.1; HRMS Calcd for $C_{23}H_{19}N_2O$ [M + H]: 339.1497, found: 339.1506.

9-Fluoro-2-(4-methoxyphenyl)pyrrolo[2,3,4-kl]acridin-1(2H)-one (5j). red solid; mp 240–242 C; IR (KBr, cm^{-1}): 2922, 1701, 1635, 1520, 1474, 1384, 1299, 1254, 1180, 1035, 968, 865, 826, 798, 771, 721, 660; ^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) 3.90 (s, 3H, CH_3O), 6.94 (d, $J = 6.8$ Hz, 2H, ArH), 7.10 (d, $J = 8.8$ Hz, 2H, ArH), 7.51 (d, $J = 8.8$ Hz, 2H, ArH), 7.65–7.72 (m, 2H, ArH), 7.85 (d, $J = 8.8$ Hz, 1H, ArH), 8.42–8.49 (m, 2H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 55.8, 106.5, 107.2, 107.4, 115.1, 120.0, 122.2, 122.5, 122.7, 127.4, 127.6, 132.9, 133.6, 133.7, 140.4, 146.0, 149.4, 159.2, 167.3; HRMS Calcd for $C_{21}H_{14}FN_2O_2$ [M + H]: 345.1039, found: 345.1040.

2-(2,6-Dichlorophenyl)-7-trifluoromethylpyrrolo[2,3,4-kl]acridin-1(2H)-one (5k). orange solid; mp 210–212 C; IR (KBr, cm^{-1}): 3009, 1713, 1638, 1492, 1460, 1440, 1415, 1343, 1307, 1255, 1142, 1103, 1059, 1015, 942, 867, 800, 687; ^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) 6.68 (d, $J = 6.8$ Hz, 1H, ArH), 7.46 (dd, $J_1 = 7.6$ Hz, $J_2 = 8.8$ Hz, 1H, ArH), 7.59 (d, $J = 8.4$ Hz, 2H, ArH), 7.74 (dd, $J_1 = 7.2$ Hz, $J_2 = 9.2$ Hz, 1H, ArH), 7.86 (t, $J = 8.0$ Hz, 1H, ArH), 8.02 (d, $J = 9.2$ Hz, 1H, ArH), 8.32 (d, $J = 7.2$ Hz, 1H, ArH), 9.13 (d, $J = 8.4$ Hz, 1H, ArH); ^{13}C-NMR (101 MHz, $CDCl_3$): δ (ppm) 106.7, 123.7, 127.6, 127.8, 128.8, 129.3, 129.6, 131.3, 133.8, 136.1, 138.5, 147.1, 147.8, 166.4; HRMS Calcd for $C_{21}H_{10}Cl_2F_3N_2O$ [M + H]: 433.0122, found: 433.0123.

Acknowledgment. This work were partially supported by the Major Basic Research Project of the Natural Science Foundation of the Jiangsu Higher Education Institutions (No. 10KJA150049), A Project Funded by the Priority Academic Project Development of Jiangsu Higher Education Institutions and the Foundation of Key Laboratory of Organic Synthesis of Jiangsu Province (No. JSK0812).

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