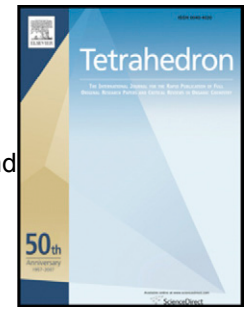
Accepted Manuscript



3-Phenacylideneoxindoles with Tosylmethyl Isocyanide and MeOH through C–C Bond Cleavage: Facile Synthesis of Pyrrole and 2*H*-pyrrolo[3,4-*c*]quinoline Derivatives†

Rong Wang, Xiao-Ping Xu, Hua Meng, Shun-Yi Wang, Shun-Jun Ji

PII: S0040-4020(12)01806-6

DOI: 10.1016/j.tet.2012.11.088

Reference: TET 23799

To appear in: *Tetrahedron*

Received Date: 15 October 2012

Revised Date: 21 November 2012

Accepted Date: 27 November 2012

Please cite this article as: Wang R, Xu X-P, Meng H, Wang S-Y, Ji S-J, 3-Phenacylideneoxindoles with Tosylmethyl Isocyanide and MeOH through C–C Bond Cleavage: Facile Synthesis of Pyrrole and 2*H*-pyrrolo[3,4-*c*]quinoline Derivatives†, *Tetrahedron* (2013), doi: 10.1016/j.tet.2012.11.088.

Graphical Abstract

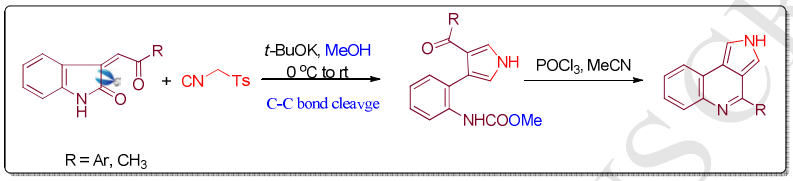
To create your abstract, type over the instructions in the template box below.
Fonts or abstract dimensions should not be changed or altered.

3-Phenacylideneoxindoles with Tosylmethyl Isocyanide via C-C Bond Cleavage: Facile Synthesis of Pyrrole and 2H-pyrrolo[3,4-c]quinoline Derivatives

Leave this area blank for abstract info.

Rong Wang, Xiao-Ping Xu, Hua Meng, Shun-Yi Wang*and Shun-Jun Ji*

College of Chemistry, Chemical Engineering and Materials Science, Soochow University, Suzhou 215123, P.R.China



Tetrahedron
journal homepage: www.elsevier.com

3-Phenacylideneoxindoles with Tosylmethyl Isocyanide and MeOH through C–C Bond Cleavage: Facile Synthesis of Pyrrole and 2*H*-pyrrolo[3,4-*c*]quinoline Derivatives†

Rong Wang, Xiao-Ping Xu, Hua Meng, Shun-Yi Wang*and Shun-Jun Ji*

Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry , Chemical Engineering and Materials Science, Soochow University, Suzhou 215123, China

ARTICLE INFO

Article history:
Received
Received in revised form
Accepted
Available online

Keywords:
C-C bonds cleavage
3-phenacylideneoxindoles
Tosylmethyl isocyanide
Pyrrole
2*H*-pyrrolo[3,4-*c*]quinolines

ABSTRACT

A novel reaction of 3-phenacylideneoxindoles (**1**) with tosylmethyl isocyanide (**2a**) and MeOH through C–C bond cleavage has been developed. The reaction proceeded under mild conditions, providing a powerful synthetic tool for the construction of pyrrole derivatives (**3**) from easily accessible starting materials and synthesis of 2*H*-pyrrolo[3,4-*c*]quinolines (**4**) by further dehydration of **3**.

1. Introduction

Nitrogen-containing heterocycles are prevalent in numerous natural products, and are extremely important in materials chemistry and medicinal chemistry.[1,2] Pyrroles and pyrroloquinolines, in particular, have attracted much attention because of their biological activities and many applications;[3] examples are the ATP-ase inhibitor (**I**),[4] and caspase inhibitor (**II**),[5] and the compounds with serotonergic activity (**III**)[6] (Figure 1)

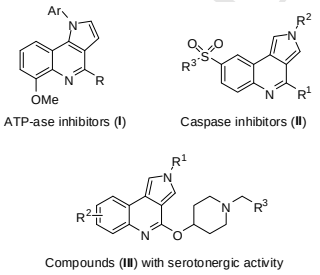
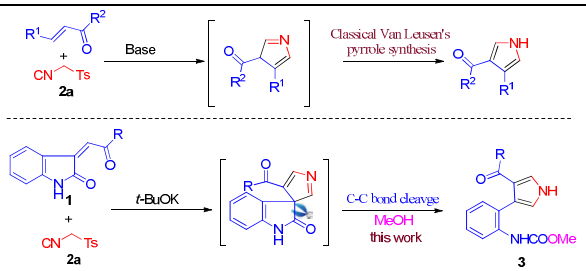


Figure. 1 Examples of biologically active pyrroloquinoline derivatives.

The efficient synthesis of pyrroles and pyrroloquinolines have become important challenges for both synthetic and pharmaceutical chemists. Although much attention has been paid to the synthesis of pyrroloquinolines using different methods,[7] there are only few reports in the literature on the synthesis of 2*H*-pyrrolo[3,4-*c*]quinoline derivatives (Scheme 1).[5,6,8] Although these methods are very useful, it has some drawbacks such as lengthy steps and poor overall yields. As a result of our continued synthetic interest in the nitrogen-containing heterocycles,[9] we report a simple and convenient synthetic approach toward pyrrole and 2*H*-pyrrolo[3,4-*c*]quinoline derivatives.

Van Leusen's pyrrole synthesis[10] through the reaction of tosylmethyl isocyanide (TosMIC) with Michael acceptors is one of the most convenient methods for obtaining 3,4-disubstituted 1*H*-pyrroles. The mechanism for Van Leusen's reaction involved the elimination of toluenesulfinate anion, followed by a final aromatization (Scheme 1). When 3-phenacylideneoxindole (**1a**)[11] and TosMIC (**2a**) were treated with 1.0 equiv of K_2CO_3 in MeOH at room temperature under the similar Van Leusen pyrrole conditions, the expected intermediate could not follow the classical mechanism of Van Leusen's reaction. Surprisingly, methyl [2-(4-benzoyl-1*H*-pyrrol-3-yl)phenyl]carbamate (**3a**) was obtained in 11% yield (Scheme 1) through C–C bond cleavage (the C_2–C_3 bond of **1a**) during final aromatization step, followed by the reaction with solvent (Table 1, entry 1). The structure of product **3a** was confirmed by NMR spectroscopy and unambiguously demonstrated by X-ray diffraction analysis (Figure 2).



Scheme 2. Classical Van Leusen's pyrrole synthesis and this work

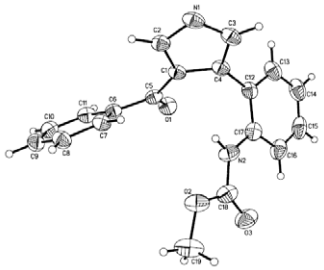
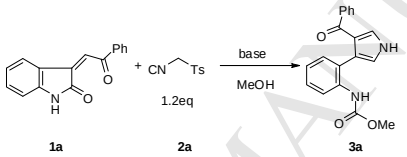


Figure 2. ORTEP diagram of **3a**

2. Results and discussion

When 3.0 equiv of K_2CO_3 were used, the yield of **3a** increased to 43% (Table 1, entry 3). In our efforts to increase the yield of **3a**, we used different bases, i.e., Cs_2CO_3, DBU, NEt_3, NaOH, KOH and *t*-BuOK, for this reaction. It was found that only trace amounts of product **3a** were formed when DBU or NEt_3 was used as the additive (Table 1, entries 5 and 6). However, it was found that 3.0 equiv of *t*-BuOK were sufficient to promote this reaction, and the yield of the desired product was increased to 65% (Table 1, entry 9). Increasing the amount of *t*-BuOK did not increase the yield of the desired product (Table 1, entry 10). After carefully checking the effects of temperature on the reaction, the optimal reaction conditions were obtained. The reaction of **1a** (0.5 mmol) and **2a** (0.6 mmol) in 2.0 mL of MeOH, using *t*-BuOK (3.0 equiv) as the additive, was initiated at 0 °C, and then the mixture was warmed to room temperature.

Table 1. Optimization of Reaction Conditions[a]

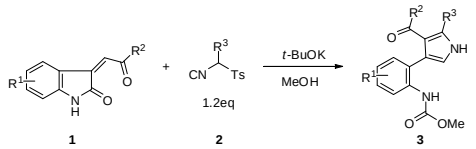


entry	Base (equiv)	time (h)	Temp (°C)	Yield(%)[b]
1	K_2CO_3 (1.0 eq.)	12	25	11
2	K_2CO_3 (2.0 eq.)	3	25	26
3	K_2CO_3 (3.0 eq.)	3	25	43
4	Cs_2CO_3 (3.0 eq.)	3	25	37
5	DBU (3.0 eq.)	12	25	trace
6	NEt_3 (3.0 eq.)	12	25	trace
7	NaOH (3.0 eq.)	3	25	58
8	KOH (3.0 eq.)	3	25	56
9	*t*-BuOK (3.0 eq.)	3	25	65
10	*t*-BuOK (4.0 eq.)	3	25	64
11	*t*-BuOK (3.0 eq.)	3	40	50
12	*t*-BuOK (3.0 eq.)	3	50	27
13	*t*-BuOK (3.0 eq.)	12	0	71
14	*t*-BuOK (3.0 eq.)	3	0°C to rt	69
15	*t*-BuOK (3.0 eq.)	3	-10°C to rt	65

[a] The reactions were performed with **1a** (0.5 mmol, 1.0 eq.) and TosMIC (0.6 mmol, 1.2 eq.) in the presence of base in 2 mL MeOH.
[b] Isolated yield.

With the reaction conditions optimized, we analyzed the scope of the reaction. The results are summarized in Table 2. These reactions tolerated a wide variety of functional groups. 3-Phenacylideneoxindoles bearing not only an electron-donating group but also an electron-withdrawing group worked well, affording the desired 3,4-disubstituted 1*H*-pyrrole derivatives in moderate to good yields (Table 2, entries 1–14). A yield of 83% was obtained in the case of 4-Br-substituted **1** as the starting material (Table 2, entry 12). When 3-acetonylideneoxindole was used in this reaction, the desired product **3s** was obtained in 51% yield (Table 2, entry 18). However, when the reaction was extended to other substituted TosMIC derivatives under identical conditions, no desired products were isolated (Table 2, entries 15–17); this was caused by steric hindrance of the TosMIC derivatives. However, poor yield(19%) was obtained when the reaction was applied ethanol as the solvent (Table 2, entry 19). When other alcohols were used as solvents, such as isopropanol, no desired products were isolated.

Table 2. Substrate Scope for Synthesis of 3,4-Disubstituted 1H-Pyrroles[a]



entry	R^1	R^2	R^3	time(h)	product	yield(%)[b]
1	5-CH_3	C_6H_5	H	5	**3b**	62
2	5-Cl	C_6H_5	H	4	**3c**	68
3	5-Br	C_6H_5	H	4	**3d**	67
4	6-Br	C_6H_5	H	4	**3e**	79
5	4-Cl	C_6H_5	H	5	**3f**	59
6	H	4-$CH_3C_6H_4$	H	4	**3g**	58
7	H	2-$CH_3C_6H_4$	H	4	**3h**	56
8	H	4-$OCH_3C_6H_4$	H	12	**3i**	66
9	H	4-$NH_2C_6H_4$	H	5	**3j**	69
10	H	4-FC_6H_4	H	9	**3k**	62
11	H	4-ClC_6H_4	H	4	**3l**	71
12	H	4-Br C_6H_4	H	4	**3m**	83
13	H	3-Cl C_6H_4	H	4	**3n**	76
14	H	4-NO_2 C_6H_4	H	3	**3o**	67
15	H	C_6H_5	CH_3	24	**3p**	trace
16	H	C_6H_5	Bn	24	**3q**	trace
17	H	C_6H_5	Allyl	24	**3r**	trace
18	H	C_6H_5	H	2	**3s**	51
19[c]	H	C_6H_5	H	6	**3t**	19

[a] The reactions were performed with **1** (0.5 mmol, 1.0 eq.) and **2** (0.6 mmol, 1.2 eq.) in the presence of *t*-BuOK in 2 mL MeOH.
[b] Isolated yield.
[c] The reactions were performed with **1** (0.5 mmol, 1.0 eq.) and **2** (0.6 mmol, 1.2 eq.) in the presence of *t*-BuOK in 2 mL EtOH.

With the 3,4-disubstituted 1*H*-pyrroles **3** in hand, we next focused on the construction of the 4-phenyl-2*H*-pyrrolo[3,4-*c*]quinoline unit through an intramolecular dehydration reaction using 10.0 equiv of $POCl_3$. The dehydrations of **3a**, **3c**, **3e**, **3l**, and **3m** were investigated in anhydrous MeCN under reflux conditions. The results are summarized in Scheme 3. All the desired 4-aryl-2*H*-pyrrolo[3,4-*c*]quinolines **4** were obtained in similar good yields. The structure of 4-phenyl-2*H*-pyrrolo[3,4-*c*]quinoline (**4a**) was also confirmed by single-crystal X-ray crystallographic analysis (Figure 3). The 4-aryl-2*H*-pyrrolo[3,4-*c*]quinolines **4** not only had the useful pyrrolo[3,4-*c*]quinoline core structure, but also contained useful functional groups such as Br and Cl, making it possible to modify them and synthesize more complex molecules by cross-coupling reactions.

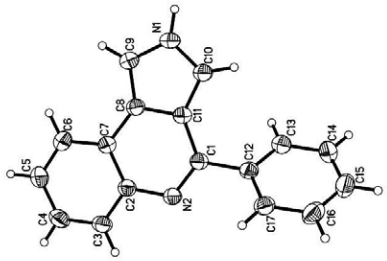
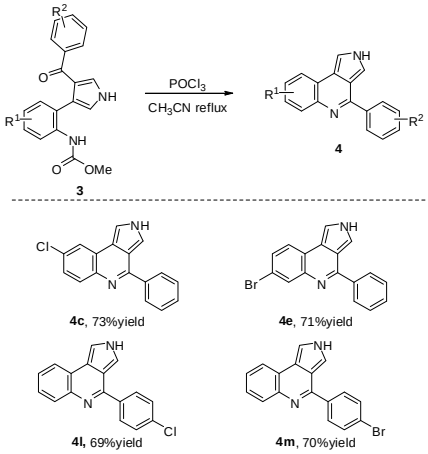


Figure 3.ORTEP diagram of **4a**



Scheme 3 Synthesis of 4-Phenyl-2*H*-pyrrolo[3,4-*c*]quinolines **4**[a]

[a] The reactions were performed with **3** (0.5 mmol, 1.0 eq.) and $POCl_3$ (5.0 mmol, 10.0 eq.) in CH_3CN.

3. Conclusion

In conclusion, we have demonstrated a simple and highly efficient synthetic strategy for preparing of pyrrole derivatives via C-C bond cleavage in good yields and obtaining 2*H*-pyrrolo[3,4-*c*]quinoline derivatives by further dehydration. This method involved a non-classical Van Leusen's pyrrole synthesis through the reaction of 3-phenacylideneoxindoles with TosMIC in MeOH under basic conditions, and intramolecular dehydration of 3,4-disubstituted 1*H*-pyrroles using $POCl_3$. It would be interesting to synthesize more functionalized 2*H*-pyrrolo[3,4-*c*]quinoline derivatives by cross-coupling reactions of Br- or Cl-substituted **4**, which have potential applications in pharmaceutical chemistry and materials chemistry. These results will be reported in due course.

4. Experimental Section

4.1. General

All reactions were carried out at ambient temperature in oven-dried glassware. All solvents were dried and distilled according to standard procedures. Column chromatography was generally performed on silica gel (300-400 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions. Melting points were recorded on an Electrothermal digital melting point apparatus and are uncorrected. IR spectra were recorded on an FT-IR spectrometer using KBr optics. NMR spectra were recorded at room temperature in d_6-DMSO at 300/400 Hz and 75/100 Hz with TMS as an internal reference. High resolution mass spectra (HRMS) were obtained on a TOF MS instrument with ESI source. X-ray diffraction data were recorded with graphite monochromatic Mo-Kα radiation.

General procedures for products 3a-3t:

To a suspension of *t*-BuOK (0.204 g, 1.5 mmol) in 2 mL of anhydrous MeOH was added a solution of 3-phenacylideneoxindoles **1** (0.5 mmol) and TosMIC **2** (0.6 mmol) in 2 mL of anhydrous MeOH at 0 °C. After stirring for 10 min at 0 °C, the reaction was stirred at room temperature for appropriate time. After reaction completion (monitored by TLC), the solvent was removed under reduced pressure. The residue was purified by preparative silica gel column chromatography to give the corresponding products **3** as white solid.

General procedures for products 4a, 4c, 4e, 4l, 4m:

To a solution of **3** (0.50 mmol) in 10 mL of dry CH_3CN was added freshly distilled $POCl_3$ (0.46 mL, 5.0 mmol) under N_2 at room temperature. The reaction mixture was warmed to reflux and stirred overnight. The reaction mixture was cooled to room temperature, diluted with water, treated with aqueous 10% NaOH solution to pH=8 and extracted with ethyl acetate (3×20 mL). The combined organic layer was washed with brine, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel to provide **4** as yellow solid.

4.2. Methyl (2-(4-benzoyl-1H-pyrrol-3-yl)phenyl)carbamate (3a). Yield: 69%; White solid; melting point: 215.5-216.4 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.76 (s, 1H), 8.01 (s, 1H), 7.71 (d, *J* = 7.6 Hz, 2H), 7.56 (dd, *J* = 15.2, 7.6 Hz, 2H), 7.45 (t, *J* = 7.4 Hz, 2H), 7.32 (s, 1H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.4 Hz, 1H), 7.04 (t, *J* = 7.4 Hz, 1H), 6.96 (s, 1H), 3.48 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 191.0, 154.4, 140.0, 136.5, 131.9, 131.5, 129.3, 129.0, 128.5, 128.1, 127.3, 123.9, 122.7, 121.9, 121.4, 121.1, 51.9; MS: Anal. Calcd. For C19H16N2O3: 320.1161 Found: 319.1089 (M-H$^+$); IR (KBr, cm^{-1}): ν 3338, 3127, 2949, 1726, 1618, 1518, 1444, 1297, 1206, 1055. Crystal data for **3a**, which has been deposited at the Cambridge Crystallographic Data Centre as supplementary publication with No. CCDC 902575. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (E-mail: linstead@ccdc.cam.ac.uk or deposit@ccdc.cam.ac.uk; fax: +44 01223 336033): Structural parameters for product **3a**: data collection: Rigaku Mercury CCD area detector; crystal size: 0.80×0.25×0.19 mm^3; $C_{19}H_{15}N_2O_3$, Mr= 319.33, monoclinic, space group P 21/n, a = 13.611(4), b= 7.2333(16), c = 17.110(5) Å, a = 90.00, b=110.804 (6), g = 90.00, V = 1574.7(7) Å^3, Z = 4, Dc= 1.347 g cm^{-3}, R[I > 2σ(I)] = 0.0775, wR[I > 2 s (I)] =0.1828.

4.3. Methyl (2-(4-benzoyl-1H-pyrrol-3-yl)-4-methylphenyl)carbamate (3b). Yield: 62%; White solid; melting point: 208.5-209.6 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.73 (s, 1H), 7.95 (s, 1H), 7.70 (d, *J* = 7.1 Hz, 2H), 7.54 (t, *J* = 6.8 Hz, 1H), 7.46-7.43 (m, 3H), 7.30 (s, 1H), 7.03 (d, *J* = 7.8 Hz, 1H), 6.95 (d, *J* = 8.7 Hz, 2H), 3.47 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 191.1, 154.6, 140.1, 134.0, 133.0, 132.0, 131.9, 129.3, 128.5, 128.1, 127.8, 123.1, 122.0, 121.6, 121.0, 51.8, 20.7; MS: Anal. Calcd. For $C_{20}H_{18}N_2O_3$: 334.1317

Found: 333.1223 (M-H^+); IR (KBr, cm^{-1}): ν 3181, 2943, 2687, 1737, 1597, 1390, 1302, 1218, 1074.

4.4. Methyl (2-(4-benzoyl-1H-pyrrol-3-yl)-4-chlorophenyl)carbamate (3c). Yield: 68%; White solid; melting point: 211.5-213.4 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.81 (s, 1H), 8.11 (s, 1H), 7.71 (d, *J* = 7.1 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 2H), 7.46 (t, *J* = 7.4 Hz, 2H), 7.33-7.28 (m, 2H), 7.19 (d, *J* = 2.5 Hz, 1H), 7.04 (s, 1H), 3.46 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ190.7, 154.4, 139.9, 135.5, 131.9, 131.1, 130.7, 129.3, 128.5, 128.2, 127.8, 127.0, 124.4, 121.8, 121.5, 120.0, 52.0; MS: Anal. Calcd. For $C_{19}H_{15}N_2O_3Cl$: 354.0771 Found: 353.0710 (M-H^+); IR (KBr, cm^{-1}): ν 3125, 2945, 1720, 1625, 1371, 1220, 1074.

4.5. Methyl (2-(4-benzoyl-1H-pyrrol-3-yl)-4-bromophenyl)carbamate (3d). Yield: 67%; White solid; melting point: 191.4-193.1 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.81 (s, 1H), 8.09 (s, 1H), 7.72 (dd, *J* = 7.2, 1.0 Hz, 2H), 7.58-7.54 (m, 2H), 7.48-7.41 (m, 3H), 7.33 (d, *J* = 2.3 Hz, 2H), 7.04 (d, *J* = 1.8 Hz, 1H), 3.46 (s, 3H). ^{13}C NMR (d_6-DMSO, 100 MHz): δ 195.7, 159.3, 144.9, 141.00, 138.5, 136.9, 136.3, 134.9, 134.3, 133.4, 133.2, 129.6, 126.8, 126.5, 124.9, 120.8, 57.0; MS: Anal. Calcd. For $C_{19}H_{15}N_2O_3Br$: 398.0266 Found: 397.0196 (M-H^+); IR (KBr, cm^{-1}): ν 3132, 2955, 1723, 1620, 1301, 1217, 1071.

4.6. Methyl (2-(4-benzoyl-1H-pyrrol-3-yl)-5-bromophenyl)carbamate (3e). Yield: 79%; White solid; melting point: 219.0-220.2 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.79 (s, 1H), 8.11 (s, 1H), 7.82 (s, 1H), 7.71 (d, *J* = 7.1 Hz, 2H), 7.58-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.33 (s, 1H), 7.23 (d, *J* = 7.8 Hz, 1H), 7.11 (d, *J* = 8.1 Hz, 1H), 6.99 (s, 1H), 3.48 (s, 3H). ^{13}C NMR (d_6-DMSO, 100 MHz): δ 195.7, 159.2, 144.9, 143.0, 138.0, 136.9, 134.3, 133.5, 133.3, 132.9, 131.4, 129.5, 126.8, 126.2, 125.1, 124.7, 57.1; MS: Anal. Calcd. For $C_{19}H_{15}N_2O_3Br$: 398.0266 Found: 397.0193 (M- H^+); IR (KBr, cm^{-1}): ν 3402, 3191, 2948, 1738, 1513, 1215, 1067.

4.7. Methyl (2-(4-benzoyl-1H-pyrrol-3-yl)-3-chlorophenyl)carbamate (3f). Yield: 59%; White solid; melting point: 235.3-237.0 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.80 (s, 1H), 7.71-7.66 (m, 4H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.38 (s, 1H), 7.24 (t, *J* = 8.1 Hz, 1H), 7.14 (d, *J* = 8.0 Hz, 1H), 6.95 (s, 1H), 3.54 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 190.1, 154.1, 139.8, 138.7, 134.5, 131.8, 129.1, 128.53, 127.6, 127.3, 124.4, 122.5, 121.4, 119.8, 116.7, 52.3. MS: Anal. Calcd. For $C_{19}H_{15}N_2O_3Cl$: 354.0771 Found: 353.0694 (M-H^+); IR (KBr, cm^{-1}): ν 3337, 3127, 2949, 1726, 1618, 1444, 1297, 1055.

4.8. Methyl (2-(4-(4-methylbenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3g). Yield: 58%; White solid; melting point: 188.2-189.8 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ11.74 (s, 1H), 8.07 (s, 1H), 7.64 (d, *J* = 7.8 Hz, 2H), 7.58 (d, *J* = 7.5 Hz, 1H), 7.31 (s, 1H), 7.27-7.22 (m, 3H), 7.15 (d, *J* = 7.3 Hz, 1H), 7.04 (t, *J* = 7.4 Hz, 1H), 6.95 (s, 1H), 3.48 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 196.1, 159.7, 147.4, 142.5, 141.8, 136.8, 134.9, 134.3, 133.1, 132.6, 129.2, 126.6, 126.3, 57.2, 26.7; MS: Anal. Calcd. For $C_{20}H_{18}N_2O_3$: 334.1317 Found: 333.1249 (M-H^+); IR (KBr, cm^{-1}): ν 3190, 2944, 1731, 1589, 1389, 1296, 1219, 1064.

4.9. Methyl (2-(4-(2-methylbenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3h). Yield: 56%; White solid; melting point: 178.8-180.2 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ11.76 (s, 1H), 7.82 (s, 1H), 7.65 (d, *J* = 7.3 Hz, 1H), 7.35-7.29 (m, 2H), 7.26-7.19 (m, 4H), 7.09-7.04 (m, 2H), 6.93 (s, 1H), 3.55 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ193.1, 154.4, 141.2, 136.6, 135.3, 131.5, 131.0, 129.8, 129.6, 127.9, 127.5, 125.4, 123.9, 123.2, 121.8, 120.9, 52.0, 19.5; MS: Anal. Calcd. For $C_{20}H_{18}N_2O_3$: 334.1317 Found: 333.1247 (M-H^+); IR (KBr, cm^{-1}): ν 3412, 3198, 2951, 1733, 1609, 1517, 1307, 1217, 1071.

4.10. Methyl (2-(4-(4-methoxybenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3i). Yield: 66%; White solid; melting point: 179.5-180.8 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.71 (s, 1H), 8.16 (s, 1H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.58 (d, *J* = 6.9 Hz, 1H), 7.31 (s, 1H), 7.24 (t, *J* = 7.4 Hz, 1H), 7.14 (d, *J* = 7.2 Hz, 1H), 7.04 (t, *J* = 7.3 Hz, 1H), 6.99 (d, *J* = 8.4 Hz, 2H), 6.94 (s, 1H), 3.82 (s, 3H), 3.49 (s, 3H). ^{13}C NMR (d_6-DMSO, 100 MHz): δ 195.0, 167.5, 159.4, 141.5, 137.3, 136.8, 136.5, 132.3, 132.1, 128.9, 127.8, 127.0, 126.3, 125.8, 118.7, 60.8, 56.9; MS: Anal. Calcd. For $C_{20}H_{18}N_2O_4$: 350.1267 Found: 349.1186 (M-H^+); IR (KBr, cm^{-1}): ν 3418, 3154, 1738, 1594, 1378, 1306, 1237, 1156, 1074.

4.11. Methyl (2-(4-(4-aminobenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3j). Yield: 69%; White solid; melting point: 237.8-238.8 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.60 (s, 1H), 8.61 (s, 1H), 7.55 (d, *J* = 8.1 Hz, 3H), 7.24 (s, 2H), 7.11 (d, *J* = 7.2 Hz, 1H), 7.03 (d, *J* = 7.2 Hz, 1H), 6.88 (s, 1H), 6.53 (d, *J* = 8.1 Hz, 2H), 5.94 (s, 2H), 3.52 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 189.9, 154.5, 153.5, 136.9, 132.4, 131.8, 129.5, 127.4, 126.6, 125.7, 123.9, 122.8, 122.4, 121.3, 120.4, 112.8, 51.9; MS: Anal. Calcd. For $C_{20}H_{18}N_2O_4$: 335.1270 Found: 334.1191 (M-H^+); IR (KBr, cm^{-1}): ν 3345, 3147, 1718, 1591, 1533, 1448, 1377, 1305, 1226, 1158, 1063.

4.12. Methyl (2-(4-(4-fluorobenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3k). Yield: 62%; White solid; melting point: 197.5-198.8 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.77 (s, 1H), 8.04 (s, 1H), 7.79-7.76 (m, 2H), 7.55 (d, *J* = 7.5 Hz, 1H), 7.35 (s, 1H), 7.26-7.21 (m, 3H), 7.14 (d, *J* = 7.4 Hz, 1H), 7.03 (t, *J* = 7.4 Hz, 1H), 6.97 (s, 1H), 3.47 (s, 3H). ^{13}C NMR (d_6-DMSO, 100 MHz): δ 194.5, 170.6, 168.2, 159.5, 141.4, 141.3, 137.1, 137.0, 136.4, 132.8, 132.3, 129.0, 126.8, 126.3, 126.0, 120.4, 120.2, 56.9; MS: Anal. Calcd. For $C_{19}H_{15}N_2O_3F$: 338.1067 Found: 337.0895 (M-H^+); IR (KBr, cm^{-1}): ν 3356, 3127, 1722, 1614, 1517, 1441, 1298, 1220.

4.13. Methyl (2-(4-(4-chlorobenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3l). Yield: 71%; White solid; melting point: 156.7-158.1 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.79 (s, 1H), 8.03 (s, 1H), 7.71 (d, *J* = 8.5 Hz, 2H), 7.56-7.48 (m, 3H), 7.36 (s, 1H), 7.23 (t, *J* = 7.7 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 1H), 7.03 (t, *J* = 7.4 Hz, 1H), 6.97 (s, 1H), 3.47 (s, 3H). ^{13}C NMR (d_6-DMSO,100 MHz): δ 194.7, 159.5, 143.6, 141.7, 141.4, 136.4, 136.2, 133.9, 133.5, 133.1, 132.3, 129.0, 127.9, 126.7, 126.3, 126.1, 56.9; MS: Anal. Calcd. For $C_{19}H_{15}N_2O_3Cl$: 354.0771 Found: 353.0681 (M-H^+); IR (KBr, cm^{-1}): ν 3404, 3128, 2350, 1720, 1514, 1297, 1216, 1076.

4.14. Methyl (2-(4-(4-bromobenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3m). Yield: 83%; White solid; melting point: 118.1-119.5 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.81 (s, 1H), 8.04 (s, 1H), 7.64 (s, 4H), 7.56 (d, *J* = 7.8 Hz, 1H), 7.37-7.36 (m, 1H), 7.23 (t, *J* = 7.7 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.98 (s, 1H), 3.48 (s, 3H). ^{13}C NMR (d_6-DMSO,75 MHz): δ 189.8, 154.5, 139.0, 136.4, 131.5, 131.4, 129.0, 128.2, 127.3, 125.7, 124.0, 123.0, 121.7, 121.4. 121.2, 51.9; MS: Anal. Calcd. For $C_{19}H_{15}N_2O_3Br$: 398.0266 Found: 397.0176 (M-H^+); IR (KBr, cm^{-1}): ν 3550, 3467, 1719, 1614, 1457, 1382, 1304, 1232, 1069.

4.15. Methyl (2-(4-(3-chlorobenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3n). Yield: 76%; White solid; melting point: 138.1-140.2 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.79 (s, 1H), 8.02 (s, 1H), 7.66-7.64 (m, 1H), 7.61 (s, 1H), 7.58-7.53 (m, 2H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.37 (s, 1H), 7.24-7.15 (m, 2H), 7.04 (t, *J* = 7.4 Hz, 1H), 6.97 (s, 1H), 3.46 (s, 3H). ^{13}C NMR (d_6-DMSO, 100 MHz): δ 194.2, 159.4, 146.9, 141.3,

138.2, 136.5, 136.3, 135.4, 133.8, 132.2, 132.8, 132.3, 129.0, 127.9, 126.6, 126.4, 126.2, 56.8; MS: Anal. Calcd. For $C_{19}H_{15}N_2O_3Cl$: 354.0771 Found: 353.0698 (M-H^+); IR (KBr, cm^{-1}): ν 3125, 2945, 1720, 1625, 1371, 1302, 1220, 1074.

4.16. Methyl (2-(4-(4-nitrobenzoyl)-1H-pyrrol-3-yl)phenyl)carbamate (3o). Yield: 67%; White solid; melting point: 184.6-186.5 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.88 (s, 1H), 8.25 (d, *J* = 8.5 Hz, 2H), 8.02 (s, 1H), 7.87 (d, *J* = 8.5 Hz, 2H), 7.53 (d, *J* = 7.7 Hz, 1H), 7.39 (s, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.3 Hz, 1H), 7.05-7.00 (m, 2H), 3.48 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 189.2, 154.6, 151.9, 149.1, 145.6, 136.3, 135.9, 131.4, 130.3, 129.0, 127.4, 124.1, 123.6, 121.6, 121.5, 121.4, 51.9; MS: Anal. Calcd. For $C_{19}H_{15}N_3O_5$: 365.1012 Found: 364.0942 (M-H^+); IR (KBr, cm^{-1}): ν 3408, 3274, 2940, 1732, 1607, 1518, 1488, 1351, 1216, 1058.

4.17. Methyl (2-(4-acetyl-1H-pyrrol-3-yl)phenyl)carbamate (3s). Yield: 51%; White solid; melting point: 142.7-144.3 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.71 (s, 1H), 7.77 (s, 1H), 7.70 (s, 1H), 7.62 (d, *J* = 7.4 Hz, 1H), 7.26 (t, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 7.4 Hz, 1H), 7.05 (t, *J* = 7.3 Hz, 1H), 6.82 (s, 1H), 3.53 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 194.3, 154.4, 136.7, 131.5, 129.1, 127.7, 127.6, 123.93, 123.4, 122.3, 121.3, 120.0, 52.1, 28.3; MS: Anal. Calcd. For $C_{14}H_{14}N_2O_3$: 258.1004 Found: 257.0925 (M-H^+); IR (KBr, cm^{-1}): ν 3420, 3126, 2954, 1733, 1632, 1511, 1300, 1229, 1144, 1070.

4.18. Ethyl (2-(4-benzoyl-1H-pyrrol-3-yl)phenyl)carbamate (3t). Yield: 19%; White solid; melting point: 147.9-149.4 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 11.76 (s, 1H), 7.87 (s, 1H), 7.72 (d, *J* = 7.4 Hz, 2H), 7.57 (dd, *J* = 18.3, 7.8 Hz, 2H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.32 (s, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.3 Hz, 1H), 7.04 (t, *J* = 7.4 Hz, 1H), 6.96 (s, 1H), 3.92 (q, *J* = 7.0 Hz, 2H), 1.04 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 196.1, 159.2, 145.3, 141.7, 137.2, 136.6, 134.6, 134.6, 133.7, 133.4, 132.6, 129.1, 127.8, 127.2, 126.6, 126.4, 65.7, 20.0; MS: Anal. Calcd. For $C_{20}H_{18}N_2O_3$: 334.1317 Found: 333.1237 (M-H^+); IR (KBr, cm^{-1}): ν 3417, 3301, 2923, 1730, 1619, 1588, 1527, 1443, 1376, 1211, 1066.

4.19. 4-Phenyl-2H-pyrrolo[3,4-c]quinoline (4a). Yield: 72%; Yellow solid; melting point: 217.6-219.5 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 12.65 (s, 1H), 8.22-8.20 (m, 1H), 8.10 (d, *J* = 7.1 Hz, 2H), 7.97 (s, 1H), 7.95-7.93 (m, 1H), 7.75 (s, 1H), 7.57 (dd, *J* = 15.7, 8.2 Hz, 3H), 7.48 (dd, *J* = 5.7, 3.4 Hz, 2H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 155.6, 142.4, 140.1, 129.7, 129.47, 128.9, 128.8, 126.3, 126.2, 123.1, 123.0, 121.9, 116.4, 114.9, 110.6; MS: Anal. Calcd. For $C_{14}H_{14}N_2O_3$: 244.2906 Found: 243.0932 (M-H^+); IR (KBr, cm^{-1}): ν 3138, 2910, 1726, 1586, 1456, 1395, 1215, 1075. Crystal data for **4a**, which has been deposited at the Cambridge Crystallographic Data Centre as supplementary publication with No. CCDC 894400. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (E-mail: linstead@ccdc.cam.ac.uk or deposit@ccdc.cam.ac.uk; fax: +44 01223 336033): Structural parameters for product **4a**: data collection: Rigaku Mercury CCD area detector; crystal size: 0.50×0.30×0.20 mm3; C17H12N2, Mr= 244.29, monoclinic, space group P 21/c, a = 11.1391(19), b= 15.066(3), c = 7.2723(14) Å, a = 90.00, b=99.299 (5), g = 90.00, V = 1204.4(4) Å3, Z = 4, Dc= 1.347 g cm-3, R[I > 2σ(I)] = 0.0526, wR[I > 2 s (I)] =0.1248.

4.20. 8-Chloro-4-phenyl-2H-pyrrolo[3,4-c]quinoline (4c). Yield: 73%; Yellow solid; melting point: 265.3-266.2 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 12.74 (s, 1H), 8.33 (s, 1H), 8.09 (d, *J* = 7.6 Hz, 3H), 7.93 (d, *J* = 8.6 Hz, 1H), 7.78 (s, 1H), 7.57 (q, *J* = 6.3 Hz, 3H), 7.48 (d, *J* = 8.6 Hz, 1H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 156.1, 141.1, 139.9, 131.3, 130.5, 129.9, 128.9, 128.8, 126.2, 124.4, 122.4, 121.0, 116.3, 115.1, 111.6; MS: Anal. Calcd. For $C_{14}H_{14}N_2O_3$: 278.0611 Found: 277.0550 (M-H^+); IR (KBr, cm^{-1}): ν 3041, 2849, 1786, 1585, 1464, 1395, 1158, 1081.

4.21. 7-Bromo-4-phenyl-2H-pyrrolo[3,4-c]quinoline (4e). Yield: 71%; Yellow solid; melting point: 227.8-229.7 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 12.76 (s, 1H), 8.19 (d, *J* = 8.4 Hz, 1H), 8.10 (d, *J* = 6.0 Hz, 3H), 8.03 (s, 1H), 7.79 (s, 1H), 7.63 (dd, *J* = 8.5, 1.4 Hz, 1H), 7.58 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 157.0, 143.6, 139.7, 131.3, 130.0, 129.0, 128.8, 128.1, 125.1, 122.1, 121.2, 118.2, 116.3, 115.5, 111.2; MS: Anal. Calcd. For $C_{14}H_{14}N_2O_3$: 322.0106 Found: 321.0033 (M-H^+); IR (KBr, cm^{-1}): ν 3139, 2914, 1716, 1594, 1453, 1075.

4.22. 8-Bromo-4-phenyl-2H-pyrrolo[3,4-c]quinoline (4l). Yield: 69%; Yellow solid; melting point: 245.6-247.2 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 12.69 (s, 1H), 8.236-8.20 (m, 1H), 8.14 (d, *J* = 8.3 Hz, 2H), 7.99 (s, 1H), 7.95-7.93 (m, 1H), 7.78 (s, 1H), 7.63 (d, *J* = 8.3 Hz, 2H), 7.50-7.48 (m, 2H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 154.2, 153.5, 142.3, 138.9, 134.4, 130.5, 129.5, 129.0, 126.5, 126.2, 123.1, 121.9, 116.1, 114.7, 110.7; MS: Anal. Calcd. For $C_{14}H_{14}N_2O_3$: 278.0611 Found: 277.0545 (M-H^+); IR (KBr, cm^{-1}): ν 3060, 2859, 1797, 1588, 1462, 1399, 1263, 1090.

4.23. 4-(4-Bromophenyl)-2H-pyrrolo[3,4-c]quinoline (4m). Yield: 70%; Yellow solid; melting point: 229.5-231.6 ºC; ^{1}H NMR (400 MHz, d_6-DMSO): δ 12.69 (s, 1H), 8.22-8.20 (m, 1H), 8.07 (d, *J* = 8.3 Hz, 2H), 7.98 (s, 1H), 7.95-7.931 (m, 1H), 7.76 (d, *J* = 8.2 Hz, 3H), 7.50-7.48 (m, 2H). ^{13}C NMR (d_6-DMSO, 75 MHz): δ 154.3, 142.4, 139.3, 131.9, 130.8, 129.6, 126.5, 126.2, 123.1, 123.0, 121.9, 116.1, 114.6, 110.7; MS: Anal. Calcd. For $C_{14}H_{14}N_2O_3$: 322.0106 Found: 321.0035 (M-H^+); IR (KBr, cm^{-1}): ν 3061, 2860, 1732, 1586, 1461, 1398, 1265, 1160, 1082.

Acknowledgements

We gratefully acknowledge support from the National Natural Science Foundation of China (No. 21172162), the Young National Natural Science Foundation of China (No. 21202111), the Young Natural Science Foundation of Jiangsu Province (BK2012174), Natural Science Basic Research of Jiangsu Province for Higher Education (No. 10KJB150016), PAPD, and Soochow University for financial support.

References

1. (a) Young, I. S.; Thornton, P. D.; Thompson, A. *Nat. Prod. Rep.*, **2010**, *27*, 1801; (b) Estévez, V.; Villacampa, M.; Menéndez, J. C. *Chem. Soc. Rev.*, **2010**, ***39***, 4402; (c) Thirumalairajan, S.; Pearce, B. M.; Thompson, A. *Chem. Commun.*, **2010**, *46*, 1797; (d) Fan, H.; Peng, J.; Hamann, M. T.; Hu, J.-F. *Chem. Rev.*, **2008**, *108*, 264; (e) Loudet, A.; Burgess, K. *Chem. Rev.*, **2007**, *107*, 4891; (f) Wood, T. E.; Thompson, A. Chem. Rev., **2007**, *107*, 1831.
2. (a) Chen, Y.; Zeng, D.; Xie, N.; Dang, Y. *J. Org. Chem.*, **2005**, *70*, 5001; (b) Skotheim, T. A.; Elsenbaumer, R. L.; Reynolds, J. R., Eds. Handbook of Conducting Polymers, 2nd ed.; Marcel Dekker: New York, **1998**.
3. (a) Helissey, P.; Parrot-Lopez, H.; Renault, J.; Cros, S. *Eur. J. Med. Chem.*, **1987**, *22*, 366; (b) Gasparotto, V.; Castagliuolo, I.; Pezzi, G.; Chiarelotto, V.; Montanaro, G. Brun, ; P.; Palu, G.; Ferlin, M. G. *J. Med. Chem.*, **2006**, *49*, 1910.
4. (a) Brown, T. H.; Ife, R. J.; Keeling, D. J.; Laing, S. M.; Leach, C. A.; Parsons, M. E., Price, C. A.; Reavill, D. R.; Wiggall, K. J. *J. Med. Chem.*, **1990** , *33*, 527; (b) Leach, C. A.; Brown, T. H.; Ife, R. J.; Laing, D. J.; Keeling, S. M.; Parsons, M. E.; Price, C. A.; Wiggall, K. J. *J. Med. Chem.*, **1992**, *35*, 1845; (c) Escolano, C.; Jones, K. *Tetrahedron Lett.*, **2000**, *41*, 8951.
5. (a) Kravchenko, D. V.; Kysil, V. M.; Tkachenko, S. E.; Maliarchouk, S.; Okun, I. M.; Ivachtchenko, A. V. *Farmaco*,

2005, *60*, 804. (b) Kravchenko, D. V.; Kysil, V. M.; Tkachenko, S. E.; Maliarchouk, S.; Okun, I. M.; Ivachtchenko, A. V. *Eur. J. Med. Chem.*, **2005**, *40*, 1377. (c) Kravchenko, D. V.; Kysil, V. M.; Vladimir, V.; Ilyn, A. P.; Tkachenko, S. E.; Maliarchouk, S.; Okun, I. M.; Ivachtchenko, A. V. *Bioorg. Med. Chem. Lett.*, **2005**, *15*, 1841. (d) Kravchenko, D. V.; Kuzovkova, J. A.; Kysil, V. M.; Tkachenko, S. E.; Maliarchouk, S.; Okun, I.; Ivachtchenko, A. V. *Lett. Drug. Des. Discov.*, **2006**, *3*, 61. (e) Okun, I.; Maliarchouk, S.; Dubrovskaya, E.; Khvat, A.; Tkachenko, S.; Kysil, V.; Kravchenko, D.; Ivachtchenko, A. V. *J. Biomol. Screen.*, **2006**, *11*, 694. (f) Sharma, S.; Sahu, K.; Jain, P.; Mourya, V. K.; Agrawal, R. K. *Med. Chem. Res.*, **2008**, *17*, 399.

6. Alisi, M. A.; Cazzolla, N.; Costi, R.; Disanto, R.; Furlotti, G.; Guglielmotti, A.; Polenzani, L. WO2010012611A1, **2010**.
7. Selected examples of synthesis of the pyrroloquinoline core: (a) Gurjar, M. K.; Pal, S.; Rao, A. V. R. *Heterocycles*, **1997**, *45*, 231; (b) Ho, T. C. T.; Jones, K. *Tetrahedron*, **199**7, *53*, 8287; (c) Snider, B. B.; Ahn, Y.; Foxman, B. M. *Tetrahedron Lett.*, **1999**, *40*, 3339; (d) Nieman, J. A.; Ennis, M. D. *Org. Lett.*, **2000**, *2*, 1395; (e) Nyerges, M.; Fejes, I.; Toke, L. *Tetrahedron Lett.*, **2000**, *41*, 7951; (f) Hadden, M.; Nieuwenhuyzen, M.; Potts, D.; Stevenson, P. J.; Thompson, N. *Tetrahedron*, **2001**, *57*, 5615; (g) Batey, R. A.; Powell, D. A. *Chem. Commun*., **2001**, 2362; (h) Hara, O.; Sugimoto, K.; Makino, K.; Hamada, Y. *Synlett*, **2004**, 1625; (i) Yadav, J. S.; Subba, R. B. V.; Sunitha, V.; Srinivasa, R. K.; Ramakrishna, K. V. S. *Tetrahedron Lett.*, **2004**, *45*, 7947; (j) Shaw, J. T.; Masse, C. E.; Ng, P. Y. *Org. Lett.*, **2006**, *8*, 3999; (k) Testa, M. L.; Lamartina, L.; Mingoia, F. *Tetrahedron*, **2004**, *60*, 5873; (l) Nyerges, M. *Heterocycles*, **2004**, *63*, 1685; (m) Zhang, Z.; Zhang, Q.; Yan, Z.; Liu, Q. *J. Org. Chem.*, **2007**, *72*, 9808; (n) Zhou, F.; Liu, J.; Ding, K.; Liu, J.; Cai, Q. *J. Org. Chem.*, **2011**, *76*, 5346.
8. (a) Santo, R. D.; Costi, R.; Forte, M.; Galeffi, C. *Arkivoc*, **2004**, 181. (b) Ho, T. C. T.; Jones, K. *Tetrahedron*, **1997**, *53*, 8287. (c) Umio, S.; Kariyone, K.; Nakamura, H. Japan Pat., 71 11,021 (Cl. C 07d, A61k). (d) Fritz, H.; Siegfrid, S. *Justus Liebig Ann. Chem.*, **1975**, 255.
9. (a) Wu, X.-J.; Jiang, R.; Wu, B.; Su, X.-M.; Xu, X.-P.; Ji, S.-J. *Adv. Synth. Catal.*, **2009**, *351*, 3150. (b) Hao, Y.; Xu, X.-P.; Chen, T.; Zhao, L.-L.; Ji, S.-J. *Org. Biomol. Chem.*, **2012**, *10*, 724.
10. (a) van Leusen, A. M.; Siderius, H.; Hoogenboom, B. E.; van Leusen, D. *Tetrahedron Lett.*, **1972**, *52*, 5337; (b) Barton, D. H. R.; Kervagoret, J.; Zard, S. Z. *Tetrahedron*, **1990**, *46*, 7587; (c) ten Have, R.; Leusink, F. R.; van Leusen, A. M. *Synthesis*, **1996**, 871; (d) Pavri, N. P.; Trudell, M. L. *J. Org. Chem.*, **1997**, *62*, 2649; (e) Dijkstra, H. P.; ten Have, R.; van Leusen, A. M. *J. Org. Chem.*, **1998**, *63*, 5332; (f) Van Leusen, D.; Van Leusen, A. M. *Org. React.*, **2001**, *57*, 417; (g) Smith, N. D.; Huang, D.; Cosford, N. D. P. *Org. Lett.*, **2002**, *4*, 3537.
11. Lindwall, H.G.; Maclennan, J.S. *J. Am. Chem. Soc.* **1932**, *54*, 4739.