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ARTICLE

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Synthesis of methyl 1,5-diaryl-1*H*-pyrrole-2-carboxylates via acid-catalyzed ring-opening/ring-closure sequence

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Abstract

A facile synthesis of methyl 1,5-diaryl-1*H*-pyrrole-2-carboxylates was reported in this article. Acid-catalyzed reaction of methyl 2-aroyl-1-phenoxycyclopropanecarboxylates with aromatic amines underwent smoothly to give methyl 1,5-diaryl-1*H*-pyrrole-2-carboxylates in high to excellent yields under mild conditions.

1 | INTRODUCTION

Multi-substituted pyrroles are one of the most important structural motifs in bioactive molecules and functional materials [1], and have received much attention from synthetic chemists. A variety of synthetic methods of multi-substituted pyrroles have established, involving the classical Knorr reaction, Hantzsch reaction, and Paal–Knorr condensation reaction [2]. The construction patterns for pyrrole core mainly include [3+2] annulation, [4+1] annulation, and three-component reactions with or without transition metal catalysis. Dimethyl acetylenedicarboxylate (DMAD) as a valuable electrophile in [3+2] annulation, is commonly used to react with α-amino ketones, or α-amino ketones generated in situ from the reaction of phenacyl bromides with aliphatic/aromatic amines, or α-nitroepoxides and primary amines [3–7]. The reaction of DMAD with β-enamino ketones or esters, or C-acylimines formed in situ from anilines and arylglyoxals, or treatment of α-silylaryl triflates, Schiff bases, and alkynes with CsF also afforded the muti-substituted pyrroles [2,8–11]. An alternative example is $Mn(OAc)_3$-promoted oxidative cyclization reaction of DMAD with anilines and styrene [12]. Besides DMAD, another alkyne reagent, the propargylic amine is also useful for the construction of a pyrrole ring. One-pot preparation of pyrroles was achieved via copper (II)-catalyzed [4+1] annulation of propargylic amines with *N*,*O*-acetals, or with alkyl/aryl glyoxylates [13,14]. In recent years, transition metal-catalyzed reactions of 2-diazoesters are successfully established to prepare pyrroles, such as the dual copper and dirhodium-catalyzed reaction of *tert*-butyldimethylsilyl-substituted vinyldiazoacetate with nitrones, copper-catalyzed condensation of imines with α-diazo-β-dicarbonyl compounds, copper (II)-catalyzed reaction of enaminones with 2-diazo-2-arylacetate, AgOTf-catalyzed reaction of enaminones with aryl diazoesters, and rhodium(II)-catalyzed intramolecular N—H insertion reactions of δ-amino-γ,γ-difluoro-α-diazo-β-ketoesters [15–19]. In addition,

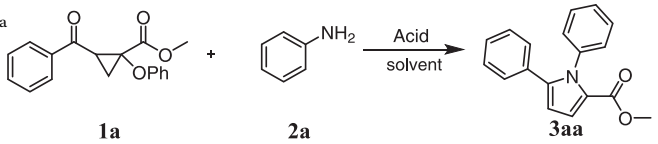
one-pot acid-catalyzed reaction between 2,3-diketoester, imine, and aldehyde produces fully substituted 3-aminopyrroles [20].

On the other hand, the chemistry of reactive cyclopropene species generated from 1,2-elimination of alkyl 1-aroyl-1-halocyclopropanecarboxylates has been carefully studied in our group [21]. The cascade reaction of this reactive cyclopropene species with nucleophilic reagent like acylhydrazones afforded functionalized pyrroles [22–24]. The reaction of alkyl 1-aroyl-1-halocyclopropanecarboxylates with amines was also assessed in the presence of Cs_2CO_3 [25]. As an extension of this investigation, the acid-catalyzed reaction of the substrates with amines has also been performed. As a consequence, the acid-promoted reaction of alkyl 1-aroyl-1-chlorocyclopropanecarboxylates with amines gave complicated products, whereas a satisfying result was observed when alkyl 1-aroyl-1-phenoxycyclopropanecarboxylates was used as the substrate. The experimental details about the reaction are described as follows.

2 | RESULTS AND DISCUSSION

In the beginning, the reaction between methyl 2-benzoyl-1-phenoxycyclopropanecarboxylate (**1a**) and aniline (**2a**) was carried out as a model in a sealed tube. Firstly, the catalytic activity of several common Lewis acids and protic acids was estimated in the solvent 1,2-dichloroethane (DCE), and the observed results are listed in Table 1. In the presence of strong Lewis acids $AlCl_3$ and $FeCl_3$, the conversion of substrate **1a** was completed after 24 h, and the yields of pyrrole **3aa** reached 68% and 55%, respectively (Table 1, entries 1 and 2). In contrast, using $BF_3{\cdot}OEt_2$ as the catalyst, a large amount of **1a** was not consumed during the reaction period, and the yield of **3aa** declined to 24% (Table 1, entry 3). Only 5% yield of **3aa** was obtained in the case of $ZnCl_2$, a relatively weak Lewis acid, indicative of a much lower catalytic activity (Table 1, entry 4). The effect of the strength of protic acids on the above reaction was also assessed. Although the superacid CF_3SO_3H can promote the reaction, a lot of **1a** was still maintained after 24 h under the reaction conditions, and the yield of **3aa** is only 38% (Table 1, entry 5). Relatively weaker acid CF_3COOH furnished **3aa** in 52% yield (Table 1, entry 6), whereas the much weaker acid CH_3COOH almost cannot promote the formation of **3aa** under the reaction conditions (Table 1, entry 7). Obviously, the proton transfer equilibrium between protic acid and aniline has a marked influence on the reaction. Based on the above

TABLE 1 Survey of the reaction conditions between **1a** and **2a**[a]



1a **2a** **3aa**

Entry	Acid (equiv)	Solvent	T/°C	t/h	Yield of 3aa/%
1	$AlCl_3$ (1)	DCE	100	24	68
2	$FeCl_3$ (1)	DCE	100	24	55
3	$BF_3{\cdot}OEt_2$ (1)	DCE	100	24	24
4	$ZnCl_2$ (1)	DCE	100	24	5
5	CF_3SO_3H (1)	DCE	100	24	38
6	CF_3COOH (1)	DCE	100	24	52
7	CH_3COOH (1)	DCE	100	24	0
8	$AlCl_3$ (1)	Toluene	100	24	59
9	$AlCl_3$ (1)	CH_3CN	100	24	43
10	$AlCl_3$ (1)	EtOH	100	24	90[b]
11	$AlCl_3$ (1)	MeOH	100	18	98
12	$AlCl_3$ (0.5)	MeOH	80	24	98
13	$AlCl_3$ (0.25)	MeOH	80	96	90
14	$AlCl_3$ (0.25)	MeOH	100	40	95
15	$AlCl_3$ (0.1)	MeOH	120	48	95
16	$AlCl_3$ (0)	MeOH	120	24	0

[a]Unless otherwise indicated, all the reactions of **1a** (0.25 mmol) with **2a** (0.3 mmol) were carried out in solvent (2.0 mL) in a sealed tube.
[b]A mixture of methyl ester and ethyl ester was obtained at the ratio of 2:1.

observations, $AlCl_3$ was chosen as the most suitable catalyst for the following investigation.

The solvent was then screened under the reaction conditions using $AlCl_3$ as the catalyst (Table 1, entries 8–11). The experimental results clearly showed that the solvent property has a great influence on the reaction yield. In nonpolar solvent toluene, a similar conversion to DCE was observed (Table 1, entry 8). Aprotic polar solvent CH_3CN disfavored the conversion of **1a**, affording **3aa** only in a 43% yield (Table 1, entry 9). Unexpectedly, the protic polar solvents EtOH or MeOH made the reaction undergo smoothly, providing the target pyrroles in up to 90% yields, despite the transesterification during the reaction (Table 1, entries 10 and 11). To avoid the undesired transesterification reaction in ethanol, methanol was chosen as the suitable solvent.

The effect of the amount of $AlCl_3$ on the reaction was next studied. As listed in Table 1, reducing the amount of $AlCl_3$ from 1.0 to 0.10 equiv, the above reaction still underwent readily and over 90% yield of **3aa** can be generated by extending the reaction time or raising the reaction temperature (Table 1, entries 11–15). In the case of 0.1 equiv $AlCl_3$, the reaction yield reached 95% (entry 15) when the reaction was conducted at 120°C for 48 h. A controlled experiment clearly demonstrated that the reaction did not proceed without $AlCl_3$ (Table 1, entry 16). Considering the affecting factors on the reaction yields, we chose the optimal reaction conditions as follows: the reaction of **1a** and **2a** was carried out in a sealed tube with $AlCl_3$ (0.5 equiv) in methanol at 80°C for 24 h.

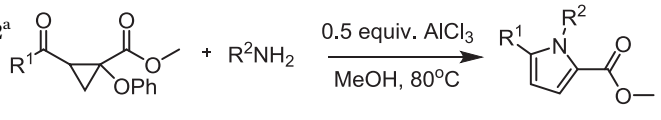
The substrate scope suitable for the reaction was explored under the above optimal reaction conditions. As shown in Table 2, the electronic property of R^1 groups for **1a–g** has little influence on the reaction yields. No matter electron-donating group (such as 4-MeO and 4-Me) or electron-withdrawing group (4-Cl) on the aromatic ring of R^1, the above reactions could proceed smoothly and the corresponding products **3ba–3da** were produced in excellent yields. However, it should be noticed that the transformation of **1d**,**e** with electron-withdrawing R^1 group needs a longer reaction time. In the case of **1e** with 4-Br group, the product **3ea** gradually precipitated during the reaction and can be isolated in 63% yield by filtration. In addition, the corresponding product **3fa** was obtained in an excellent yield, despite the bulky R^1 group of **1f**. In the case of **1g** with a heteroaryl group such as thiophene, the desired product **3ga** was given in a yield of 86% in the presence of 1 equiv of $AlCl_3$ after 48 h.

On the other hand, the influence of substituents of aromatic amines (R^2) on the reaction was also investigated. As depicted in Table 2, the electronic property of substituents of the amines has no obvious influence on the reaction yields. The desired pyrroles **3ab–3af** was obtained in 93%–98% yields, despite whether the substituents at the *para* position of anilines are electron-donating 4-MeO and 4-Me groups or electron-withdrawing 4-Cl, 4-Br, and 4-NO_2 groups. However, it is clear that electron-rich anilines needed a much longer time to complete the reaction than electron-deficient anilines, indicating that the relatively weak basicity of aniline favored the reaction to a certain extent. Besides the electronic property, the steric hindrance of substituents on the reaction was also assessed. Anilines **2g-j** with Me, F, Cl, or Br groups at the ortho site were tested. In the case of o-toluidine, the reaction was almost completed after 48 h, giving the product **3ga** in 96% yield. In contrast, the reaction of anilines **2h-j** was almost finished within 18 h under the same conditions and produced the corresponding products **3ha-ja** in 95%–98% yields. In addition, the reaction of m-chloroaniline (**2k**) proceeded much faster than that of o-chloroaniline (**2i**). This observation really shows a considerable influence of steric hindrance on the reaction rate. This opinion was further supported by the fact that the bulky 2,4,6-trimethylaniline (**2n**) gave the corresponding product **3an** only in 72% within 24 h. In addition, either electron-deficient m-nitroaniline (**2l**) or 1-naphthylamine (**2m**) with the fused ring was suitable for the reaction. Therefore, the above reaction is tolerant of a range of substituent groups on the aromatic ring of amines.

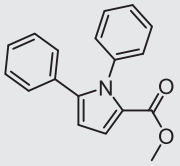
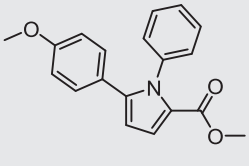
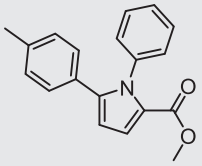
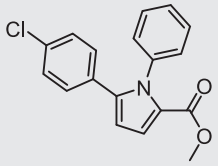
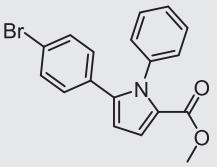
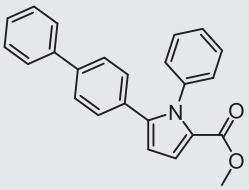
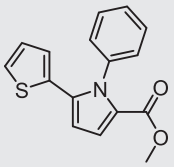
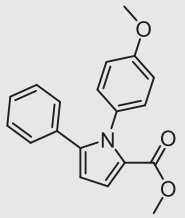
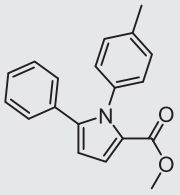
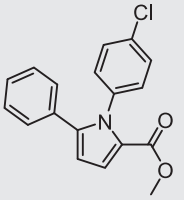
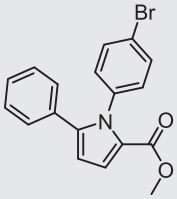
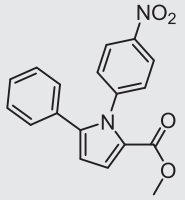
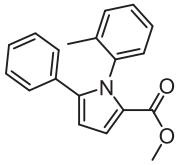
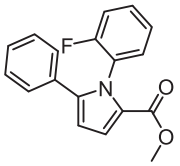
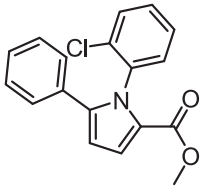
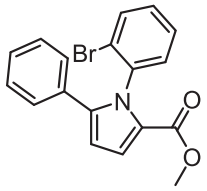
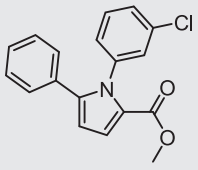
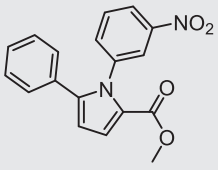
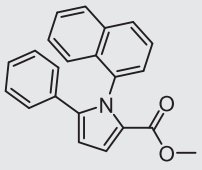
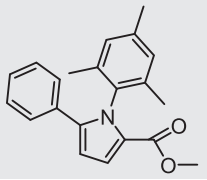
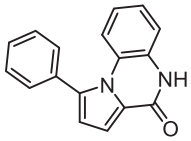
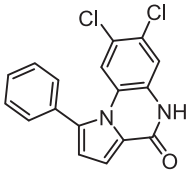
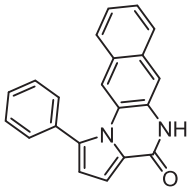
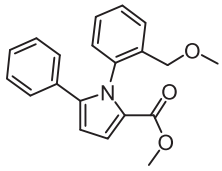
Since the pyrrole derivative has an ester group at its ortho position, we expected that some heterocyclic compounds with a big fused ring system would be constructed by introducing a functional group like amino at the ortho position of aromatic amine. Thus, the individual reaction of o-phenylenediamine, 4,5-dichloro-*o*-phenylenediamine, and naphthalene-2,3-diamine with **1a** was performed. The reaction proceeded smoothly as we expected, and the products **3ao-aq** containing the quinoxalinone structure were obtained in the yield of 98%, 87%, and 92%, respectively. Starting from o-aminobenzyl alcohol, the product **3ar** was isolated in a yield of 65%.

Based on the observations mentioned above, a plausible mechanistic pathway depicted in Scheme 1 was proposed for rationalizing the formation of the pyrrole ring of **3**. First, initial coordination of carbonyl group of the substrate **1** with Lewis acid $AlCl_3$ led to ring-opening of the strained cyclopropane slowly, giving a reactive carbocation intermediate **A**. Then, **A** was quickly attacked by the nucleophilic aromatic amine to generate the ammonium **B**, and **B** was next converted into 4-aminoketone **C** through subsequent intramolecular proton migration

TABLE 2 The scope for the acid-catalyzed reaction between **1** and **2**[a]



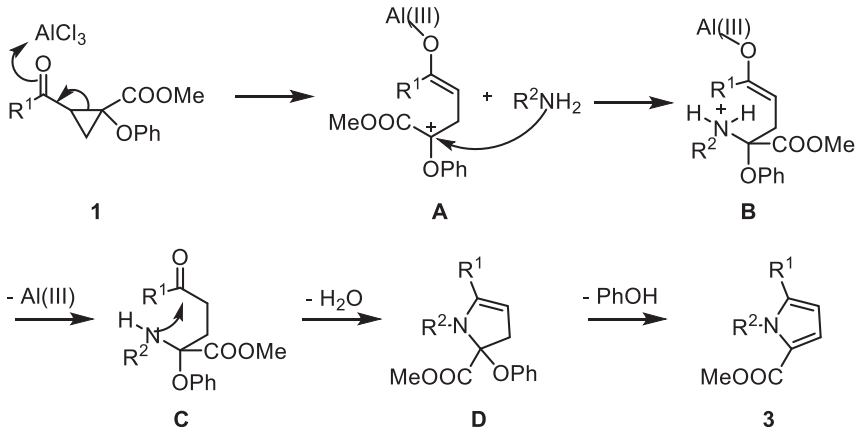
1 **2** **3**

			
3aa, 24 h, 98%	**3ba**, 24 h, 91%	**3ca**, 24 h, 95%	**3da**, 36 h, 96%
			
3ea, 24 h, 63%[b]	**3fa**, 20 h, 94%	**3ga**, 48 h, 86%[c]	**3ab**, 24 h, 94%
			
3ac, 20 h, 95%	**3ad**, 12 h, 96%	**3ae**, 15 h, 98%	**3af**, 10 h, 93%
			
3ag, 48 h, 96%	**3ah**, 12 h, 98%	**3ai**, 18 h, 95%	**3aj**, 18 h, 96%
			
3ak, 10 h, 95%	**3al**, 18 h, 98%	**3am**, 12 h, 96%	**3an**, 24 h, 72%
			
3ao, 8 h, 98%	**3ap**, 12 h, 87%	**3aq**, 12 h, 92%	**3ar**, 24 h, 65%

[a]Unless otherwise indicated the reactions of **1** (0.25 mmol) with **2** (0.3 mmol) were carried out with $AlCl_3$ (0.125 mmol) and methanol (2.0 mL) in a sealed tube.
[b]Isolated yield by filtration of the precipitate.
[c]The reaction was carried out with 1 equiv $AlCl_3$ (0.25 mmol).

from ammonium to C=C bond, releasing one molecule of $AlCl_3$. Intramolecular nucleophilic addition of nitrogen to carbonyl and elimination of a molecule of water resulted in the formation of dihydropyrrole **D**. Finally, the pyrrole product **3** was generated by 1,2-elimination of one molecule of phenol from **D**.

SCHEME 1 Plausible mechanistic pathways for the formation of **3**



3 | CONCLUSIONS

In summary, a novel and practical [4+1] annulation reaction have been developed for the synthesis of various multi-substituted pyrroles from methyl 2-acyl-1-phenoxycyclopropanecarboxylates **1** and aromatic amine in the presence of $AlCl_3$ as the catalyst. The reaction has the merits such as transition metal-free, easily available substrate, excellent yields, and mild conditions. The application study of the pyrroles as chiral ligand is ongoing in our group.

4 | EXPERIMENTAL

All reagents and solvents are of commercial grade and purified prior to use when necessary. Reactions are followed by thin-layer chromatography (TLC) analysis using silica gel 60 Å F-254 thin layer plates. Flash column chromatography was performed on silica gel 60 Å, 10–40 μm. All ^{1}H NMR and ^{13}C NMR spectra are recorded on a 400 or 600 MHz spectrometer with solvent resonances as the internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm). The following abbreviations are used to describe peak patterns where appropriate: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br s = broad signal. All coupling constants (*J*) are given in hertz. IR spectra were recorded on an infrared spectrometer. Melting points were recorded on a melting point detector. High-resolution mass spectrometry (HRMS) was measured on a quadrupole time-of-flight mass spectrometer equipped with an electrospray ionization (ESI) technique.

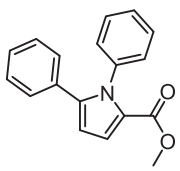
The substrate **1** was prepared according to the experimental procedures reported in our previous work [21].

4.1 | The typical procedures for the reaction of 1a with aniline

A sealed tube was charged with **1a** (74 mg, 0.25 mmol), aniline **2a** (27 μL, 0.3 mmol), MeOH (2.0 mL), and $AlCl_3$ (17 mg, 0.125 mmol). Then, the mixture was heated and stirred at 100°C for a period. Progress for the reaction was monitored by TLC until all the **1a** disappeared completely. The reaction mixture was cooled, quenched with 10 mL of saturated NaCl aqueous solution, and extracted with CH_2Cl_2 three times. Combined organic layers were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was isolated by silica gel column chromatography using petroleum ether and ethyl acetate as the eluent (v/v 40:1) to afford colorless solid **3aa** in 98% yield.

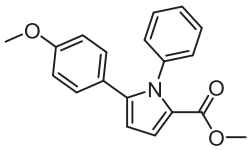
Unless otherwise specified, all other products **3** were synthesized according to the typical procedure, and the spectroscopic data are given as follows:

4.1.1 | Methyl 1,5-diphenyl-1*H*-pyrrole-2-carboxylate (**3aa**)



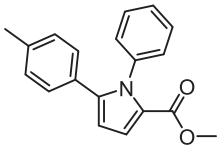
Colorless solid (total 68 mg, 98%); m.p. 136–137°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27–7.23 (m, 3H), 7.13–7.10 (m, 2H), 7.09–7.05 (m, 4H), 7.03–6.98 (m, 2H), 6.34 (d, J = 4.0 Hz, 2H), 3.61 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 141.4, 139.1, 132.0, 128.9, 128.7, 128.4, 128.1, 128.0, 127.4, 124.7, 118.5, 110.0, 51.1. IR (film) ν 3059, 2948, 1716, 1598, 1534, 1497, 1457, 1406, 1352, 1282, 1233, 1193, 1150, 1094, 1010, 752, 697 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{15}NO_2Na$ $[M + Na]^+$: 300.0995, found 300.0988.

4.1.2 | Methyl 5-(4-methoxyphenyl)-1-phenyl-1*H*-pyrrole-2-carboxylate (**3ba**)



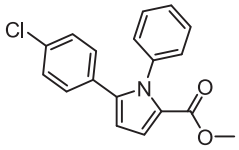
Colorless solid (total 70 mg, 91%); m.p. 127–129°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27–7.20 (m, 3H), 7.14–7.08 (m, 2H), 7.05 (d, J = 3.9 Hz, 1H), 6.92 (d, J = 8.7 Hz, 2H), 6.60 (d, J = 8.7 Hz, 2H), 6.26 (d, J = 3.9 Hz, 1H), 3.62 (s, 3H), 3.59 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 159.0, 141.4, 139.2, 130.1, 128.8, 128.5, 128.1, 124.5, 124.2, 118.5, 113.6, 109.4, 55.2, 51.1. IR (film) ν 3056, 2989, 2950, 2837, 1701, 1607, 1573, 1536, 1497, 1461, 1399, 1350, 1308, 1249, 1184, 1152, 1092, 1033, 1010, 913, 840, 793, 757, 696 cm^{-1}. HRMS (ESI) m/z calcd for $C_{19}H_{17}NO_3Na$ $[M + Na]^+$: 330.1101, found 330.1095.

4.1.3 | Methyl 1-phenyl-5-(*p*-tolyl)-1*H*-pyrrole-2-carboxylate (**3ca**)



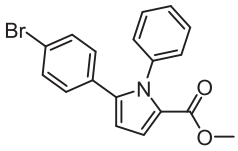
Colorless solid (total 69 mg, 95%); m.p. 147–149°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.30 (m, 3H), 7.22–7.17 (m, 2H), 7.14 (d, J = 3.9 Hz, 1H), 7.00–6.93 (m,4H), 6.38 (d, J = 3.9 Hz, 1H), 3.67 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 141.6, 139.2, 137.3, 129.1, 128.8, 128.8, 128.5, 128.1, 124.5, 118.5, 51.1, 21.2. IR (film) ν 3052, 2986, 2947, 1712, 1592, 1538, 1495, 1460, 1396, 1348, 1311, 1281, 1237, 1189, 1150, 1089, 1010, 950, 915, 824, 774, 751, 697 cm^{-1}. HRMS (ESI) m/z calcd for $C_{19}H_{17}NO_2Na$ $[M + Na]^+$: 314.1151, found 314.1147.

4.1.4 | Methyl 5-(4-chlorophenyl)-1-phenyl-1*H*-pyrrole-2-carboxylate (**3da**)



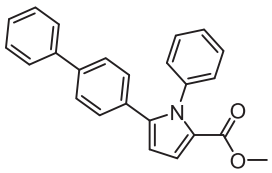
Colorless solid (total 75 mg, 96%); m.p. 173–175°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.23 (m, 3H), 7.12–7.07 (m, 2H), 7.07–7.01 (m, 3H), 6.92 (d, J = 8.6 Hz, 2H), 6.32 (d, J = 4.0 Hz, 1H), 3.61 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.9, 140.1, 138.8, 133.5, 130.5, 130.0, 128.6, 128.6, 128.3, 128.3, 125.0, 118.4, 110.1, 51.2. IR (film) ν 3054, 2992, 2949, 2844, 1714, 1590, 1529, 1494, 1456, 1390, 1347, 1271, 1237, 1192, 1152, 1108, 1083, 1050, 1010, 950, 915, 833, 778, 753, 696 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}ClNO_2Na$ $[M + Na]^+$: 334.0605, found 334.0598.

4.1.5 | Methyl 5-(4-bromophenyl)-1-phenyl-1*H*-pyrrole-2-carboxylate (**3ea**)



Colorless solid (total 56 mg, %); m.p. 191–193°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.33 (m, 3H), 7.29 (d, J = 8.5 Hz, 2H), 7.21–7.16 (m, 2H), 7.14 (d, J = 4.0 Hz, 1H), 6.94 (d, J = 8.5 Hz, 2H), 6.41 (d, J = 4.0 Hz, 1H), 3.70 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 140.0, 138.8, 131.3, 130.9, 130.3, 128.6, 128.3, 125.0, 121.7, 118.4, 110.1, 51.2. IR (film) ν 3052, 2992, 2948, 2842, 1710, 1590, 1526, 1495, 1454, 1386, 1346, 1271, 1236, 1191, 1151, 1094, 1070, 1008, 949, 914, 829, 776, 753, 720, 695 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}BrNO_2Na$ $[M + Na]^+$: 378.0100, found 378.0087.

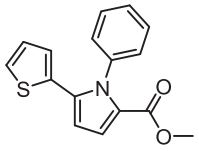
4.1.6 | Methyl 5-[(1,1′-biphenyl)-4-yl]-1-phenyl-1*H*-pyrrole-2-carboxylate(**3fa**)



Colorless solid (total 83 mg, 94%); m.p. 171–173°C. 1H NMR (400 MHz, CDCl3) δ 7.51 (d, J = 7.3 Hz, 2H), 7.42–7.34 (m, 7H), 7.30 (t, J = 7.3 Hz, 1H), 7.26–7.21 (m, 2H), 7.16 (dd, J = 10.8, 6.2 Hz, 3H), 6.47 (d, J = 4.0 Hz,

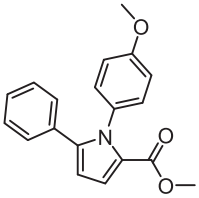
1H), 3.70 (s, 3H). 13C NMR (101 MHz, CDCl3) δ 160.9, 141.0, 140.3, 140.0, 139.1, 130.9, 129.1, 128.8, 128.8, 128.6, 128.2, 127.5, 126.9, 126.7, 124.9, 118.6, 110.1, 51.1. IR (film) ν 3059, 2949, 1720, 1596, 1497, 1460, 1424, 1347, 1307, 1275, 1232, 1189, 1144, 1091, 1042, 1007, 947, 910, 841, 793, 765, 734, 698 cm-1. HRMS (ESI) m/z calcd for $C_{24}H_{19}NO_2Na$ [M + Na]+: 376.1308, found 376.1301.

4.1.7 | Methyl 1-phenyl-5-(thiophen-2-yl)-1*H*-pyrrole-2-carboxylate (3ga)



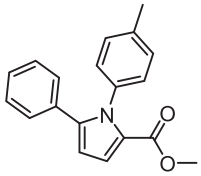
Faint yellow solid (total 61 mg, 86%); m.p. 94–96°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.31 (m, 3H), 7.24–7.18 (m, 2H), 7.02 (d, J = 4.1 Hz, 1H), 7.00 (dd, J = 5.1, 0.9 Hz, 1H), 6.72 (dd, J = 5.0, 3.7 Hz, 1H), 6.53 (dd, J = 3.6, 0.9 Hz, 1H), 6.43 (d, J = 4.1 Hz, 1H), 3.59 (s, 3H).. ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 139.0, 135.1, 133.6, 129.0, 128.9, 127.0, 125.9, 125.6, 124.9, 118.3, 109.6, 51.2. IR (film) ν 3101, 3070, 2990, 2946, 1712, 1540, 1497, 1468, 1431, 1410, 1346, 1265, 1234, 1203, 1141, 1067, 1042, 1005, 930, 894, 842, 770, 751, 697 cm^{-1}. HRMS (ESI) m/z calcd for $C_{16}H_{13}NO_2SNa$ [M + Na]$^+$: 306.0559, found 306.0553.

4.1.8 | Methyl 1-(4-methoxyphenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3ab**)



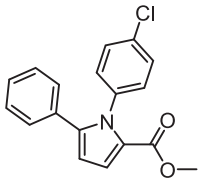
Colorless solid (total 72 mg, 94%); m.p. 154–156°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.06 (m, 8H), 6.84 (d, J = 8.8 Hz, 2H), 6.40 (d, J = 4.0 Hz, 1H), 3.78 (s, 3H), 3.69 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 159.1, 141.6, 132.1, 131.8, 129.6, 128.9, 128.1, 127.4, 124.7, 118.3, 113.6, 109.8, 55.3, 51.1. IR (film) ν 3061, 3019, 2942, 2840, 1711, 1608, 1513, 1458, 1432, 1353, 1300, 1280, 1242, 1184, 1154, 1092, 1020, 1002, 952, 914, 829, 793, 760, 696 cm^{-1}. HRMS (ESI) m/z calcd for $C_{19}H_{17}NO_3Na$ [M + Na]$^+$: 330.1101, found 330.1093.

4.1.9 | Methyl 5-phenyl-1-(*p*-tolyl)-1*H*-pyrrole-2-carboxylate (**3ac**)



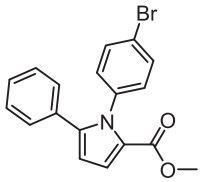
Colorless solid (total 69 mg, 95%); m.p. 121–123°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09–6.94 (m, 10H), 6.31 (dd, J = 3.9, 0.9 Hz, 1H), 3.60 (s, 3H), 2.26 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 141.5, 137.9, 136.5, 132.1, 129.2, 128.9, 128.4, 128.1, 127.4, 124.6, 118.4, 109.9, 51.1, 21.3. IR (film) ν 3060, 3033, 2948, 2922, 2851, 1713, 1598, 1514, 1458, 1435, 1411, 1351, 1311, 1283, 1231, 1911, 1149, 1111, 1092, 1048, 1008, 949, 914, 822, 797, 757, 699 cm^{-1}. HRMS (ESI) m/z calcd for $C_{19}H_{17}NO_2Na$ [M + Na]$^+$: 314.1151, found 314.1143.

4.1.10 | Methyl 1-(4-chlorophenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3ad**)



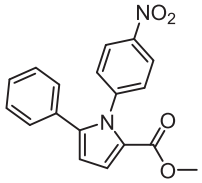
Colorless solid (total 75 mg, 96%); m.p. 141–143°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, J = 8.6 Hz, 2H), 7.20–7.17 (m, 3H), 7.15 (d, J = 4.0 Hz, 1H), 7.11 (d, J = 8.6 Hz, 2H), 7.10–7.04 (m, 2H), 6.40 (d, J = 4.0 Hz, 1H), 3.69 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.9, 141.5, 137.6, 134.0, 131.7, 130.0, 129.0, 128.7, 128.3, 127.7, 124.6, 118.8, 110.3, 51.2. IR (film) ν 3089, 3061, 2985, 2946, 2849, 1711, 1596, 1532, 1494, 1458, 1432, 1409, 1356, 1314, 1278, 1236, 1189, 1153, 1090, 1052, 1011, 950, 916, 841, 825, 794, 758, 697 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}ClNO_2Na$ [M + Na]$^+$: 334.0605, found 334.0591.

4.1.11 | Methyl 1-(4-bromophenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3ae**)



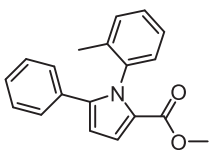
Yellow solid (total 87 mg, 98%); m.p. 144–146°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, J = 8.5 Hz, 2H), 7.22–7.13 (m, 4H), 7.11–6.98 (m, 4H), 6.40 (dd, J = 2.3, 1.6 Hz, 1H), 3.70 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.9, 141.5, 138.1, 131.7, 131.6, 130.4, 129.0, 128.3, 127.7, 124.6, 122.1, 118.8, 110.3, 51.2. IR (film) ν 3059, 2947, 1710, 1595, 1529, 1493, 1458, 1434, 1404, 1349, 1314, 1276, 1231, 1191, 1150, 1092, 1069, 1046, 1006, 947, 913, 829, 795, 756, 699 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}BrNO_2Na$ $[M + Na]^+$: 378.0100, found 378.0096.

4.1.12 | Methyl 1-(4-nitrophenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3af**)



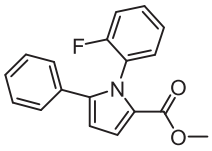
Faint yellow solid (total 75 mg, 93%); m.p. 123–125°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, J = 8.8 Hz, 2H), 7.26 (d, J = 8.8 Hz, 2H), 7.14–7.06 (m, 4H), 7.00–6.92 (m, 2H), 6.36 (d, J = 3.9 Hz, 1H), 3.62 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.9, 147.0, 144.7, 141.5, 131.2, 129.8, 129.0, 128.4, 128.0, 124.7, 123.8, 119.4, 111.0, 51.4. IR (film) ν 3057, 2958, 1715, 1599, 1527, 1499, 1460, 1434, 1408, 1356, 1287, 1235, 1189, 1153, 1092, 1044, 1011, 952, 916, 860, 796, 753, 699 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}N_2O_4Na$ $[M + Na]^+$: 345.0846, found 345.0839.

4.1.13 | Methyl 5-phenyl-1-(*o*-tolyl)-1*H*-pyrrole-2-carboxylate (**3ag**)



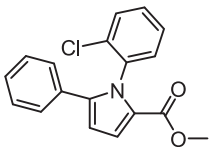
Faint yellow solid (total 70 mg, 96%); m.p. 86–88°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23–7.15 (m, 1H), 7.13–6.96 (m, 9H), 6.38 (d, J = 4.0 Hz, 1H), 3.60 (s, 3H), 1.81 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 141.0, 138.5, 136.3, 132.0, 130.4, 129.1, 128.6, 128.4, 128.1, 127.5, 126.1, 124.2, 118.3, 109.8, 51.1, 17.4. IR (film) ν 3058, 3022, 2983, 2945, 2919, 2848, 1718, 1602, 1535, 1494, 1459, 1406, 1352, 1314, 1284, 1265, 1234, 1186, 1147, 1091, 1045, 1009, 950, 914, 872, 795, 771, 754, 697 cm^{-1}. HRMS (ESI) m/z calcd for $C_{19}H_{17}NO_2Na$ $[M + Na]^+$: 314.1151, found 314.1147.

4.1.14 | Methyl 1-(2-fluorophenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3ah**)



Colorless solid (total 72 mg, 98%); m.p. 89–91°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28–7.19 (m, 1H), 7.13–6.92 (m, 9H), 6.36 (dd, J = 3.4, 1.3 Hz, 1H), 3.62 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.7, 158.8, 156.3, 140.6, 130.5, 129.4, 129.1, 129.0, 127.6, 127.1, 126.7, 126.3, 126.2, 123.7, 122.9, 122.8, 117.6, 114.9, 114.7, 109.2, 50.1. IR (film) ν 3064, 3024, 2951, 2851, 1713, 1592, 1544, 1503, 1460, 1434, 1411, 1352, 1286, 1261, 1237, 1187, 1146, 1111, 1088, 1039, 1007, 948, 916, 819, 794, 755, 699 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}FNO_2Na$ $[M + Na]^+$: 318.0901, found 318.0895.

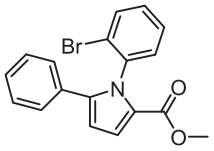
4.1.15 | Methyl 1-(2-chlorophenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3ai**)



Faint yellow solid (total 74 mg, 95%); m.p. 126–128°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 7.9 Hz, 1H), 7.32–7.26 (m, 1H), 7.23–7.09 (m, 8H), 6.45 (d, J = 3.9 Hz, 1H), 3.69 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 141.3, 137.3, 133.7, 131.6, 130.5, 129.7, 129.7, 128.7, 128.2,

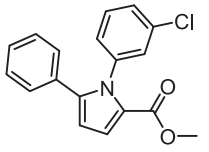
127.8, 127.0, 124.5, 118.4, 110.1, 51.2. IR (film) ν 3073, 3032, 3005, 2957, 2850, 2810, 1704, 1585, 1537, 1486, 1459, 1407, 1350, 1286, 1234, 1187, 1148, 1100, 1072, 1036, 1006, 947, 912, 796, 755, 687 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}ClNO_2Na$ $[M + Na]^+$: 334.0605, found 334.0600.

4.1.16 | Methyl 1-(2-bromophenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3aj**)



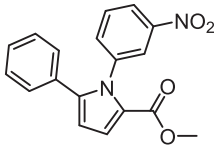
Colorless solid (total 85 mg, 96%); m.p. 158–160°C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.58 (dd, J = 7.8, 0.6 Hz, 1H), 7.33–7.09 (m, 9H), 6.45 (d, J = 4.0 Hz, 1H), 3.69 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 141.1, 138.9, 132.8, 131.6, 130.6, 129.9, 128.8, 128.2, 127.8, 127.7, 124.3, 124.1, 118.4, 110.2, 51.3. IR (film) ν 3071, 3032, 3003, 2956, 2850, 1704, 1580, 1537, 1500, 1482, 1459, 1405, 1349, 1285, 1234, 1187, 1148, 1096, 1065, 1031, 1005, 947, 912, 796, 755, 690 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}BrNO_2Na$ $[M + Na]^+$: 378.0100, found 378.0091.

4.1.17 | Methyl 1-(3-chlorophenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3ak**)



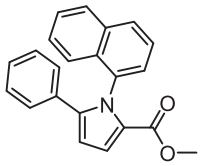
Faint yellow solid (total 74 mg, 95%); m.p. 130–131°C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.23 (d, J = 8.1 Hz, 1H), 7.17 (d, J = 7.9 Hz, 1H), 7.14–7.08 (m, 4H), 7.06 (d, J = 3.9 Hz, 1H), 7.00 (d, J = 6.7 Hz, 3H), 6.32 (d, J = 3.9 Hz, 1H), 3.61 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 141.5, 140.2, 133.9, 131.6, 129.3, 129.1, 128.9, 128.4, 128.2, 127.7, 127.2, 124.7, 118.8, 110.3, 51.2. IR (film) ν 3068, 2988, 2945, 2844, 1711, 1585, 1533, 1458, 1432, 1351, 1311, 1278, 1230, 1191, 1145, 1093, 1075, 1047, 1017, 953, 914, 875, 795, 752, 699 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}ClNO_2Na$ $[M + Na]^+$: 334.0605, found 334.0597.

4.1.18 | Methyl 1-(3-nitrophenyl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3al**)



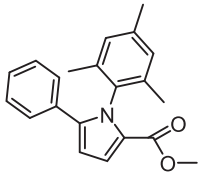
Faint yellow solid (total 79 mg, 98%); m.p. 159–161°C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.16–8.06 (m, 1H), 7.99 (t, J = 1.8 Hz, 1H), 7.48–7.35 (m, 2H), 7.14–7.07 (m, 4H), 7.01–6.93 (m, 2H), 6.36 (d, J = 4.0 Hz, 1H), 3.62 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.8, 146.9, 140.6, 139.0, 133.9, 130.0, 128.0, 128.0, 127.3, 126.9, 123.6, 123.1, 121.9, 118.1, 109.7, 50.2. IR (film) ν 3076, 2951, 2851, 1704, 1601, 1532, 1459, 1406, 1348, 1286, 1260, 1233, 1195, 1151, 1084, 1048, 1020, 956, 919, 869, 806, 759, 698 cm^{-1}. HRMS (ESI) m/z calcd for $C_{18}H_{14}N_2O_4Na$ $[M + Na]^+$: 345.0846, found 345.0839.

4.1.19 | Methyl 1-(naphthalen-1-yl)-5-phenyl-1*H*-pyrrole-2-carboxylate (**3am**)



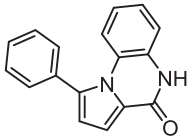
Gray solid (total 78 mg, 96%); m.p. 119–121°C. ^1H NMR (400 MHz, CDCl3) δ 7.74 (d, J = 8.2 Hz, 2H), 7.37–7.23 (m,4H), 7.22–7.13 (m, 2H), 7.01–6.81 (m, 5H), 6.45 (d, J = 4.0 Hz, 1H), 3.44 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6, 142.3, 136.2, 133.7, 132.2, 131.9, 128.9, 128.4, 128.3, 128.0, 127.6, 127.3, 126.5, 126.3, 125.8, 125.0, 122.7, 118.4, 110.1, 51.1. IR (film) ν 3057, 2949, 1717, 1596, 1540, 1505, 1461, 1415, 1351, 1279, 1235, 1185, 1145, 1081, 1026, 974, 936, 913, 866, 800, 777, 752, 696 cm^{-1}. HRMS (ESI) m/z calcd for $C_{22}H_{17}NO_2Na$ $[M + Na]^+$: 350.1151, found 350.1143.

4.1.20 | Methyl 1-mesityl-5-phenyl-1*H*-pyrrole-2-carboxylate (**3an**)



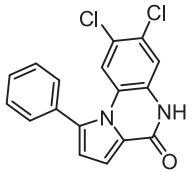
Colorless solid (total 57 mg, 72%); m.p. 107–109°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.14 (m, 4H), 7.13–7.06 (m, 2H), 6.85 (s, 2H), 6.51 (d, J = 4.0 Hz, 1H), 3.69 (s, 3H), 2.28 (s, 3H), 1.87 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6, 140.2, 137.9, 135.8, 135.2, 132.0, 128.7, 128.2, 127.7, 127.5, 123.2, 118.4, 109.8, 51.1, 21.2, 17.7. IR (film) ν 3064, 3027, 2996, 2949, 2918, 2852, 1701, 1600, 1533, 1490, 1457, 1434, 1403, 1352, 1309, 1278, 1259, 1233, 1181, 1144, 1090, 1038, 1008, 937, 912, 860, 793, 750, 696 cm^{-1}. HRMS (ESI) m/z calcd for $C_{21}H_{21}NO_2Na$ $[M + Na]^+$: 342.1465, found 342.1458.

4.1.21 | 1-Phenylpyrrolo[1,2-*a*]quinoxalin-4(5*H*)-one (**3ao**)



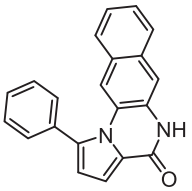
Faint yellow powder (total 64 mg, 98%); m.p. 244–246°C. ^{1}H NMR (400 MHz, DMSO) δ 11.35 (s, 1H), 7.50 (s, 5H), 7.31 (d, J = 7.9 Hz, 1H), 7.18–7.12 (m, 2H), 7.00 (d, J = 8.4 Hz, 1H), 6.78 (t, J = 7.3 Hz, 1H), 6.59 (d, J = 3.8 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 155.7, 134.5, 133.7, 129.9, 129.8, 129.3, 129.3, 125.8, 125.5, 123.7, 121.8, 117.4, 116.9, 115.9, 112.0. IR (film) ν 3320, 3175, 3123, 3034, 2981, 2916, 2855, 2775, 1666, 1611, 1512, 1494, 1462, 1439, 1380, 1262, 1170, 1153, 1070, 1026, 936, 867, 838, 791, 768, 753, 734, 700, 660 cm^{-1}. HRMS (ESI) m/z calcd for $C_{17}H_{12}N_2ONa$ $[M + Na]^+$: 283.0842, found 283.0837.

4.1.22 | 7,8-Dichloro-1-phenylpyrrolo[1,2-*a*]quinoxalin-4(5*H*)-one (**3ap**)



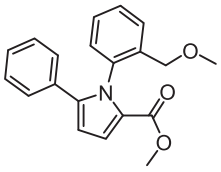
Gray powder (total 72 mg, 87%); m.p. 311–312°C. ^{1}H NMR (400 MHz, DMSO) δ 11.50 (s, 1H), 7.66–7.49 (m, 5H), 7.41 (s, 1H), 7.18 (d, J = 3.8 Hz, 1H), 7.00 (s, 1H), 6.67 (d, J = 3.8 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 155.3, 134.9, 132.8, 130.2, 130.0, 129.8, 129.6, 127.4, 125.0, 123.4, 123.2, 118.3, 117.8, 116.2, 112.8. IR (film) ν 3317, 3167, 3119, 3048, 3016, 2927, 2890, 2825, 2728, 1662, 1610, 1545, 1496, 1465, 1409, 1371, 1278, 1251, 1174, 1137, 1077, 1035, 1003, 966, 919, 873, 842, 793, 765, 738, 698, 675 cm^{-1}. HRMS (ESI) m/z calcd for $C_{17}H_{10}Cl_2N_2ONa$ $[M + Na]^+$: 351.0062, found 351.0055.

4.1.23 | 1-Phenylbenzo[*g*]pyrrolo[1,2-a]quinoxalin-4(5*H*)-one (**3aq**)



Faint red powder (total 71 mg, 92%); m.p. 292–294°C. ^{1}H NMR (400 MHz, DMSO) δ 11.52 (s, 1H), 7.77 (d, J = 8.2 Hz, 1H), 7.66 (s, 1H), 7.62–7.50 (m, 5H), 7.38 (t, J = 7.5 Hz, 1H), 7.32 (s, 1H), 7.26 (t, J = 7.5 Hz, 1H), 7.20 (d, J = 3.8 Hz, 1H), 7.12 (d, J = 8.2 Hz, 1H), 6.65 (d, J = 3.8 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 155.8, 135.5, 133.6, 130.7, 130.1, 129.6, 129.5, 129.4, 128.1, 127.6, 126.7, 125.5, 125.3, 124.1, 115.9, 114.6, 113.0, 112.5. IR (film) ν 3326, 3164, 3115, 3034, 2992, 2949, 2904, 2859, 2822, 2781, 1663, 1590, 1571, 1524, 1492, 1471, 1444, 1389, 1342, 1268, 1215, 1179, 1152, 1128, 1074, 1020, 945, 915, 876, 833, 796, 768, 737, 702, 668 cm^{-1}. HRMS (ESI) m/z calcd for $C_{21}H_{14}N_2ONa$ $[M + Na]^+$: 333.0998, found 333.0991.

4.1.24 | Methyl 1-[2-(methoxymethyl)phenyl]-5-phenyl-1*H*-pyrrole-2-carboxylate (**3ar**)



Colorless solid (total 52 mg, 65%); m.p. 92–94°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, J = 7.5 Hz, 1H), 7.40 (t, J = 7.5 Hz, 1H), 7.30 (dd, J = 10.8, 4.2 Hz, 1H), 7.21 (d, J = 7.7 Hz, 1H), 7.19–7.08 (m, 6H), 6.47 (d, J = 4.0 Hz, 1H), 4.03 (dd, J = 32.6, 12.8 Hz, 2H), 3.67 (s, 3H), 3.15 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 141.3, 137.4, 136.6, 131.7, 129.2, 128.8, 128.5, 128.1, 128.1, 127.6, 127.6, 124.8, 118.4, 109.9, 70.1, 58.4, 51.1. IR (film) ν 3139, 3066, 3033, 2994, 2951, 2926, 2889, 2849, 2825, 2733, 1711, 1601, 1536, 1495, 1458, 1409, 1381, 1351, 1310, 1283, 1265, 1232, 1188, 114, 1096, 1040, 1007, 969, 949, 913, 871, 795, 757, 725, 699 cm^{-1}. HRMS (ESI) m/z calcd for $C_{19}H_{17}NO_3Na$ $[M + Na]^+$: 330.1101, found 330.1094.

ACKNOWLEDGMENT

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available online in the supplementary material of this article.

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