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ORIGINAL PAPER

Novel one-pot access to 2-formyl/acetyl-1-substituted pyrrolo[2,3-*b*]quinoxalines under Sonogashira reaction conditions

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Abstract A one-pot reaction of *N*-alkyl-3-chloroquinoxaline-2-amines with acetylenic alcohols in the presence of a Pd–Cu catalyst leads to the formation of 2-formyl/acetyl-*N*-substituted pyrrolo[2,3-*b*]quinoxalines in good to high yields. A possible mechanism for the conversion has also been proposed.

Keywords Acetylenic alcohol · Pyrroloquinoxaline · Cross-coupling · Pd–Cu catalyst

Introduction

Quinoxalines and their derivatives are a very important class of nitrogen-containing heterocycles showing various biological activities, and can act as antiviral, antibacterial, antibiotic, anti-inflammatory, and kinase inhibitors [1–7]. They are potential building blocks for the synthesis of organic semiconductors [8], electroluminescent materials [9], cavitands [10, 11], and dehydroannulenes [12]. Quinoxalines also serve as useful rigid subunits in macrocyclic receptors [13], for molecular recognition, and chemically controllable switches [14].

The Pd-catalyzed cross-coupling reactions of aryl halides or triflates with terminal alkynes, commonly referred to as Sonogashira reactions, are powerful, versatile, and popular tools for selective construction of new C–C bonds [15, 16]. This strategy with subsequent cyclization has been successfully utilized for the synthesis of carbocylic [17] and heterocyclic compounds alike [18–20].

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We were intrigued by the prospect of applying this methodology to the synthesis of new heterocyclic compounds.

Although a few methods have been reported [21, 22] for the synthesis of pyrrolo[2,3-*b*]quinoxalines, a survey of the literature showed that there are no references concerning the use of acetylenic alcohols as a starting material. In continuation of our recent studies [23–26] on the Pd-catalyzed reaction of acetylenes leading to new heterocyclic compounds, and in particular pyrrolo[2,3-*b*]quinoxalines [27], we decided to investigate the reaction of *N*-alkyl-3-chloroquinoxaline-2-amines **1a**–**1h** with acetylenic alcohols **2a**, **2b** in organic solvents.

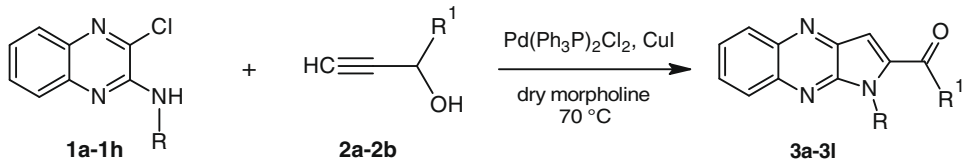
Results and discussion

When compounds **1a**–**1h** were reacted with acetylenic alcohols **2a**, **2b** in dry morpholine in the presence of $Pd(PPh_3)_2Cl_2$ and CuI at 70 °C, 2-formyl/acetyl-*N*-substituted pyrrolo[2,3-*b*]quinoxalines **3a**–**3l** were obtained in moderate to good yields (Scheme 1). The reactions were carried out under an argon atmosphere, and solvents were degassed prior to use.

We recently reported the synthesis of pyrrolo[2,3-*b*]quinoxaline-2-carbaldehydes from the reaction of *N*-alkyl-3-chloroquinoxaline-2-amines with propargyl bromide [28]. In this article we report the use of acetylenic alcohols instead; this has many advantages over propargyl bromide, which is not only an expensive chemical but also a very toxic, odorous, and unstable starting material with high vapor pressure.

For optimization of the reaction conditions, the reaction of **1b** with propargyl alcohol **2a** was chosen as a model reaction. The effect of solvents and bases was also examined. Several solvents and bases which were screened for

Scheme 1



R= Bn, Me, Et, *n*-Pr, *i*-Pr, *n*-Bu, *i*-Bu, $MeOCH_2CH_2$

R^1 = H, Me

the reaction in the presence of a catalytic amount of $Pd(PPh_3)_2Cl_2$ and CuI are shown in Table 1.

Amongst the various solvents and bases which were tested, cyclic secondary amines such as morpholine and pyrrolidine were the most suitable solvents and bases (Table 1, entries 15 and 16).

In order to explore the scope and generality of this protocol, several *N*-alkyl-3-chloroquinoxalin-2-amines **1a**–**1h** were prepared [28], and then reacted with acetylenic alcohols **2a**, **2b** to afford 2-formyl/acetyl-*N*-substituted pyrrolo[2,3-*b*]quinoxalines **3a**–**3l** in moderate to good yields (Table 2).

The structural assignments of compounds **3a**–**3l** were based on the elemental analyses and the following spectral data. In ^{1}H NMR spectra of compounds **3a**–**3l**, a singlet aromatic proton appeared at 7.55–7.92 ppm, which was characteristic of a fused pyrrole ring. For 2-formyl compounds an unexchangeable peak at 10.21–10.25 ppm was assigned to the aldehyde proton, and for 2-acetyl compounds a singlet for methyl protons was observed at 2.76–2.78 ppm. Quinoxaline ring protons in all compounds appeared at 7.55–8.23 ppm. The IR spectra of all compounds **3a**–**3l** showed a strong absorption band at 1,668–1,685 cm^{-1} which was assigned to conjugated carbonyl groups. Therefore spectral and elemental analyses data strongly supported the synthesis of 2-formyl/acetyl-*N*-substituted pyrrolo[2,3-*b*]quinoxalines **3a**–**3l**.

Table 1 Effect of solvent and base on the heterocyclization via cross-coupling of compound **1b** with propargyl alcohol (**2a**)

Entry	Solvent	Base	Yield/%
1	DMF	Et_3N	22
2	DMF	DIEA	33
3	DMF	Morpholine	45
4	DMF	Et_2NH	25
5	DMF	Pyrrolidine	28
6	CH_3CN	Et_3N	50
7	CH_3CN	Morpholine	72
8	CH_3CN	Pyrrolidine	68
9	CH_3CN	Et_2NH	42
10	Dioxane	Et_3N	65
11	Dioxane	Morpholine	58
12	Dioxane	Pyrrolidine	70
13	Et_3N	–	63
14	Et_2NH	–	67
15	Morpholine	–	88
16	Pyrrolidine	–	78

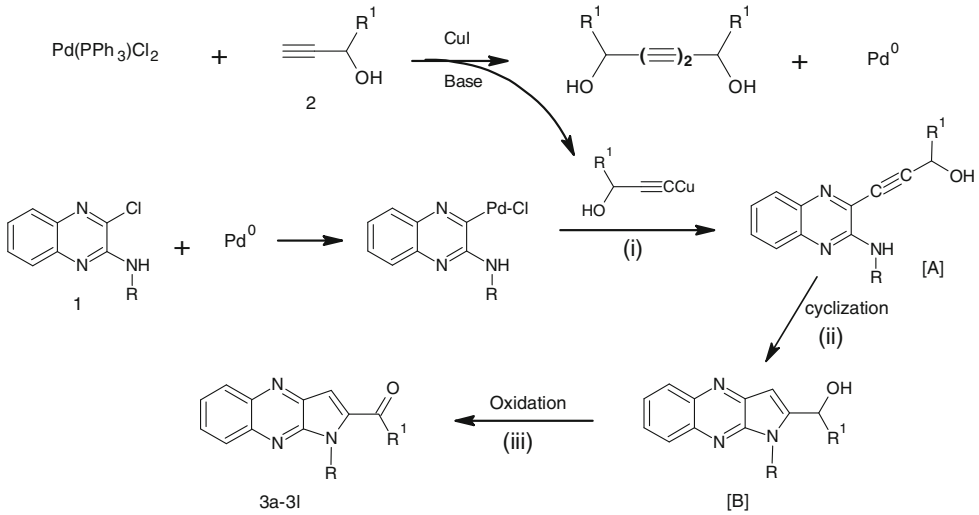
Table 2 Synthesis of 2-formyl/acetyl-*N*-substituted pyrrolo[2,3-*b*]quinoxalines

Entry	R	R^1	Product	Yield/%
1	Bn	H	**3a**	93
2	Me	H	**3b**	88
3	Me	Me	**3c**	80
4	Et	H	**3d**	85
5	*n*-Pr	Me	**3e**	68
6	*n*-Pr	H	**3f**	76
7	*i*-Pr	H	**3g**	70
8	*i*-Pr	Me	**3h**	65
9	*n*-Bu	H	**3i**	75
10	*i*-Bu	H	**3j**	80
11	Et	Me	**3k**	74
12	$MeOCH_2CH_2$	H	**3l**	83

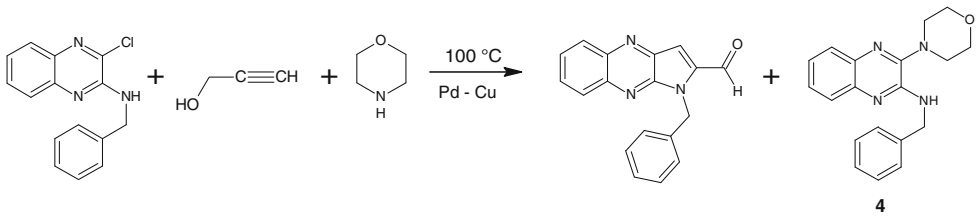
The following steps could be postulated for the mechanism of the formation of 2-formyl/acetyl-*N*-substituted pyrrolo[2,3-*b*]quinoxalines **3a**–**3l** (Scheme 2) [29]: (1) standard Sonogashira cross-coupling reaction between *N*-alkyl-3-chloroquinoxalin-2-amine **1** and acetylenic alcohols **2** gives rise to [A], (2) ring closure by hydroamination/cyclization leads to compound [B], (3) aerial oxidation gives the aromatic products **3a**–**3l**. It seems that aerial oxidation took place during aqueous work-up and column chromatography.

Control of the reaction temperature was critical. Because when the temperature rose to 100 °C, nucleophilic displacement of the chlorine atom on the aromatic ring was observed, and *N*-benzyl-3-(morpholin-4-yl)quinoxalin-2-amine (**4**) was formed as the major product (Scheme 3). But on the basis of ^{1}H NMR data, 15 % of 1-benzyl-1*H*-pyrrolo[2,3-*b*]quinoxaline-2-carbaldehyde (**3a**) was still produced under the reaction conditions used.

Scheme 2



Scheme 3



Conclusion

We have developed a one-pot reaction for the synthesis of 2-formyl/acetyl-*N*-substituted pyrrolo[2,3-*b*]quinoxalines via Pd-catalyzed reaction of readily available *N*-alkyl-3-chloroquinoxaline-2-amines with acetylenic alcohols in dry morpholine.

Experimental

All the reagents used were of general reagent grade. IR spectra were obtained from potassium bromide pellets in the range of 400–4,000 cm^{-1} on a Shimadzu model 460 spectrometer. ^1H NMR spectra were recorded on a Bruker BRX 500 AVANCE spectrometer. Elemental analyses were performed on a Thermo Finnigan Flash EA micro-analyzer.

General procedure for the preparation of 2-formyl/acetyl-N-substituted pyrrolo[2,3-b]quinoxalines ***3a–3l***

A mixture of *N*-alkyl-3-chloroquinoxaline-2-amines **1** (0.56 mmol), 19.6 mg $(PPh_3)_2PdCl_2$ (0.028 mmol), and 10.7 mg CuI (0.056 mmol) was stirred in 2 cm^3 dry morpholine under an argon atmosphere. Acetylenic alcohol **2** (1.12 mmol) was then added slowly, and the mixture was heated at 70 °C with stirring for 20 h. After completion of the reaction, the resulting solution was concentrated in vacuum, then 5 cm^3 hydrochloric acid (10 %) was added, and the mixture was extracted with $CHCl_3$. The chloroform layer was separated, washed with water, and dried over anhydrous magnesium sulfate. Concentration of the solution gave the crude product which was subjected to column chromatography using $CHCl_3/CH_3OH$ (98:2) as eluent to afford an analytically pure product.

1-Benzyl-1H-pyrrolo[2,3-b]quinoxaline-2-carbaldehyde (**3a**, $C_{18}H_{13}N_3O$)
M.p.: 180–182 °C; $R_f = 0.55$; ^1H NMR (500 MHz, DMSO-d_6): $\delta = 5.91$ (s, 2H, CH_2), 7.16 (d, $J = 7.3$ Hz, 2H, 2CH), 7.23 (t, $J = 6.9$ Hz, 1H, CH), 7.29 (t, $J = 7.2$ Hz, 2H, 2CH), 7.81 (t, $J = 7.7$ Hz, 1H, CH), 7.87 (t, $J = 7.7$ Hz, 1H, CH), 7.92 (s, 1H, CH pyrrole), 8.16 (d, $J = 8.5$ Hz, 1H, CH), 8.25 (d, $J = 8.4$ Hz, 1H, CH), 10.21 (s, 1H, CHO) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 185.4$, 143.3, 142.0, 141.5, 140.7, 140.1, 137.8, 130.0, 129.5, 128.5, 128.2, 127.7, 127.3, 126.6, 113.6, 45.8 ppm; IR (KBr): $\bar{\nu} = 1{,}675$ (C=O), 1,525, 1,433, 1,090, 750 cm^{-1}.

1-Methyl-1H-pyrrolo[2,3-b]quinoxaline-2-carbaldehyde (**3b**, $C_{12}H_9N_3O$)
M.p.: 176–177 °C; $R_f = 0.52$; ^1H NMR (500 MHz, DMSO-d_6): $\delta = 4.15$ (s, 3H, CH_3), 7.76 (s, 1H, CH

pyrrole), 7.77 (t, $J = 7.6$ Hz, 1H, CH), 7.87 (t, $J = 7.7$ Hz, 1H, CH), 8.16 (d, $J = 8.5$ Hz, 1H, CH), 8.22 (d, $J = 8.5$ Hz, 1H, CH), 10.22 (s, 1H, CHO) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 185.1$, 148.6, 147.6, 143.7, 142.6, 141.4, 130.3, 128.9, 125.8, 125.1, 112.7, 30.6 ppm; IR (KBr): $\bar{\nu} = 1{,}680$ (C=O), 1,454, 1,095, 777 cm^{-1}.

2-Acetyl-1-methyl-1H-pyrrolo[2,3-b]quinoxaline (**3c**, $C_{13}H_{11}N_3O$)
M.p.: 169–171 °C; $R_f = 0.53$; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 2.77$ (s, 3H, CH_3), 4.23 (s, 3H, CH_3), 7.71 (s, 1H, CH pyrrole), 7.68–7.80 (m, 2H, 2CH), 8.16 (d, $J = 8.0$ Hz, 1H, CH), 8.22 (d, $J = 8.1$ Hz, 1H, CH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 188.7$, 146.8, 144.5, 143.7, 143.2, 141.5, 130.3, 128.9, 125.7, 125.1, 112.2, 30.4, 27.3 ppm; IR (KBr): $\bar{\nu} = 1{,}668$ (C=O), 1,442, 1,210, 749 cm^{-1}.

1-Ethyl-1H-pyrrolo[2,3-b]quinoxaline-2-carbaldehyde (**3d**, $C_{13}H_{11}N_3O$)
M.p.: 130–132 °C; $R_f = 0.54$; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.48$ (t, 3H, $J = 7.2$ Hz, CH_3), 4.86 (q, $J = 7.2$ Hz, 2H, CH_2), 7.55 (s, 1H, CH pyrrole), 7.73 (t, $J = 7.7$ Hz, 1H, CH), 7.81 (t, $J = 7.6$ Hz, 1H, CH), 8.17 (d, $J = 8.3$ Hz, 1H, CH), 8.25 (d, $J = 8.4$ Hz, 1H, CH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 185.2$, 149.2, 144.7, 144.2, 142.5, 138.8, 130.3, 128.6, 125.7, 125.2, 115.8, 39.6, 13.6 ppm; IR (KBr): $\bar{\nu} = 1{,}680$ (C=O), 1,460, 1,095, 780 cm^{-1}.

2-Acetyl-1-propyl-1H-pyrrolo[2,3-b]quinoxaline (**3e**, $C_{15}H_{15}N_3O$)
M.p.: 129–130 °C; $R_f = 0.55$; ^{1}H NMR (500 MHz, DMSO-d_6): $\delta = 0.85$ (t, $J = 7.6$ Hz, 3H, CH_3), 1.77 (m, $J = 7.5$ Hz, 2H, CH_2), 2.76 (s, 3H, CH_3), 4.63 (t, $J = 7.4$ Hz, 2H, CH_2), 7.73 (m, 3H, CH and CH pyrrole), 8.14 (d, $J = 8.4$ Hz, 1H, CH), 8.20 (d, $J = 8.3$ Hz, 1H, CH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 189.5$, 147.3, 146.6, 146.1, 145.7, 139.3, 130.5, 128.9, 125.7, 125.2, 113.2, 49.2, 27.3, 21.5, 12.2 ppm; IR (KBr): $\bar{\nu} = 1{,}675$ (C=O), 1,440, 1,418, 1,350, 1,290, 1,150, 745 cm^{-1}.

1-Propyl-1H-pyrrolo[2,3-b]quinoxaline-2-carbaldehyde (**3f**, $C_{14}H_{13}N_3O$)
M.p.: 137–138 °C; $R_f = 0.53$; ^{1}H NMR (500 MHz, DMSO-d_6): $\delta = 0.88$ (t, $J = 7.4$ Hz, 3H, CH_3), 1.81 (m, $J = 7.3$ Hz, 2H, CH_2), 4.67 (t, $J = 7.2$ Hz, 2H, CH_2), 7.80 (t, $J = 7.6$ Hz, 1H, CH), 7.84 (s, 1H, CH pyrrole), 7.87 (m, 1H, CH), 8.14 (d, $J = 8.5$ Hz, 1H, CH), 8.21 (d, $J = 8.5$ Hz, 1H, CH), 10.23 (s, 1H, CHO) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 184.9$, 147.0, 145.3, 143.2, 140.4, 131.3, 128.9, 125.7, 125.1, 115.8, 47.2, 21.3, 11.8 ppm; IR (KBr): $\bar{\nu} = 1{,}678$ (C=O), 1,447, 1,110, 770 cm^{-1}.

1-Isopropyl-1H-pyrrolo[2,3-b]quinoxaline-2-carbaldehyde (**3g**, $C_{14}H_{13}N_3O$)
M.p.: 134–135 °C; $R_f = 0.54$; ^{1}H NMR (500 MHz, DMSO-d_6): $\delta = 1.73$ (d, $J = 6.9$ Hz, 6H, 2CH_3), 5.73 (m, $J = 7.4$ Hz, 1H, CH), 7.81 (t, $J = 7.6$ Hz, 1H, CH), 7.85 (s, 1H, CH pyrrole), 7.87 (t, $J = 7.7$ Hz, 1H, CH), 8.15 (d, $J = 8.5$ Hz, 1H, CH), 8.22 (d, $J = 8.5$ Hz, 1H, CH), 10.23 (s, 1H, CHO) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 185.0$, 148.2, 145.4, 143.3, 141.3, 140.1, 130.8, 129.1, 126.0, 125.2, 116.2, 48.8, 20.6 ppm; IR (KBr): $\bar{\nu} = 1{,}670$ (C=O), 1,450, 1,010, 770 cm^{-1}.

2-Acetyl-1-isopropyl-1H-pyrrolo[2,3-b]quinoxaline (**3h**, $C_{15}H_{15}N_3O$)
M.p.: 124–125 °C; $R_f = 0.51$; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.59$ (d, $J = 7.3$ Hz, 6H, 2CH_3), 4.23 (m, $J = 7.1$ Hz, 1H, CH), 7.30 (s, 1H, CH pyrrole), 7.68–7.79 (m, 2H, CH), 8.16 (d, $J = 7.8$ Hz, 1H, CH), 8.28 (d, $J = 7.9$ Hz, 1H, CH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 189.1$, 149.5, 148.2, 145.1, 143.6, 140.5, 131.1, 128.8, 125.8, 125.2, 115.3, 50.5, 27.3, 20.6 ppm; IR (KBr): $\bar{\nu} = 2{,}900$, 1,677 (C=O), 1,400, 1,420, 1,350, 1,210, 1,190, 1,110, 760 cm^{-1}.

1-Butyl-1H-pyrrolo[2,3-b]quinoxaline-2-carbaldehyde (**3i**, $C_{15}H_{15}N_3O$)
M.p.: 146–147 °C; $R_f = 0.52$; ^{1}H NMR (500 MHz, DMSO-d_6): $\delta = 0.90$ (t, $J = 7.5$ Hz, 3H, CH_3), 1.27 (m, $J = 7.6$ Hz, 2H, CH_2), 1.79 (m, $J = 7.5$ Hz, 2H, CH_2), 4.71 (t, $J = 7.4$ Hz, 2H, CH_2), 7.82 (t, $J = 8.3$ Hz, 1H, CH), 7.86 (s, 1H, CH pyrrole), 7.89 (t, $J = 8.9$ Hz, 1H, CH), 8.16 (d, $J = 8.6$ Hz, 1H, CH), 8.23 (d, $J = 8.1$ Hz, 1H, CH), 10.23 (s, 1H, CHO) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 184.8$, 150.0, 145.8, 143.4, 141.2, 129.9, 128.9, 124.9, 124.2, 114.9, 44.0, 30.3, 20.7, 14.0 ppm; IR (KBr): $\bar{\nu} = 1{,}675$ (C=O), 1,450, 1,100, 770 cm^{-1}.

1-(2-Methylpropyl)-1H-pyrrolo[2,3-b]quinoxaline-2-carbaldehyde (**3j**, $C_{15}H_{15}N_3O$)
M.p.: 142–143 °C; $R_f = 0.54$; ^{1}H NMR (500 MHz, DMSO-d_6): $\delta = 0.83$ (d, $J = 6.6$ Hz, 6H, 2CH_3), 2.30 (m, $J = 6.8$ Hz, 1H, CH), 4.51 (d, $J = 7.5$ Hz, 2H, CH_2), 7.77 (t, $J = 7.3$ Hz, 1H, CH), 7.81 (s, 1H, CH pyrrole), 7.86 (t, $J = 7.4$ Hz, 1H, CH), 8.12 (d, $J = 8.5$ Hz, 1H, CH), 8.21 (d, $J = 8.4$ Hz, 1H, CH), 10.24 (s, 1H, CHO) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 185.1$, 148.9, 146.8, 146.0, 143.5, 140.6, 130.5, 128.7, 125.8, 125.3, 114.9, 52.5, 26.1, 20.2 ppm; IR (KBr): $\bar{\nu} = 1{,}680$ (C=O), 1,445, 1,095, 775 cm^{-1}.

2-Acetyl-1-ethyl-1H-pyrrolo[2,3-b]quinoxaline (**3k**, $C_{14}H_{13}N_3O$)
M.p.: 150–151 °C; $R_f = 0.50$; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.44$ (t, 3H, $J = 7.5$ Hz, CH_3), 4.82 (q, $J = 7.3$ Hz, 2H, CH_2), 7.35 (s, 1H, CH pyrrole), 7.55–7.79

(m, 2H, 2CH), 8.18 (d, $J = 7.9$ Hz, 1H, CH), 8.21 (d, $J = 8.0$ Hz, 1H, CH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 190.1$, 147.6, 146.7, 146.5, 145.8, 139.5, 130.6, 129.3, 125.7, 125.1, 113.6, 41.1, 27.3, 13.6 ppm; IR (KBr): $\bar{\nu} = 2{,}900$, 1,676 (C=O), 1,448, 1,260, 1,100, 750 cm^{-1}.

1-(2-Methoxyethyl)-1H-pyrrolo[2,3-b]quinoxaline-2-carbaldehyde (**3l**, $C_{14}H_{13}N_3O_2$)
M.p.: 126–127 °C; $R_f = 0.53$; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.32$ (s, 3H, CH_3), 3.82 (t, $J = 5.6$ Hz, 2H, CH_2), 5.08 (t, $J = 5.6$ Hz, 2H, CH_2), 7.56 (s, 1H, CH pyrrole), 7.72 (t, $J = 6.4$ Hz, 1H, CH), 7.80 (t, $J = 6.8$ Hz, 1H, CH), 8.18 (d, $J = 8.8$ Hz, 1H, CH), 8.22 (d, $J = 8.0$ Hz, 1H, CH), 10.21 (s, 1H, CHO) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 184.9$, 150.0, 145.9, 143.4, 140.1, 131.5, 129.1, 125.9, 125.3, 114.9, 72.9, 57.8, 41.8 ppm; IR (KBr): $\bar{\nu} = 1{,}680$ (C=O), 1,465, 1,098, 780 cm^{-1}.

N-Benzyl-3-(morpholin-4-yl)quinoxalin-2-amine
(**4**, $C_{19}H_{20}N_4O$)
^{1}H NMR (500 MHz, DMSO-d_6): $\delta = 3.23$ (m, 4H, $2CH_2$), 3.85 (m, 4H, $2CH_2$), 4.70 (d, $J = 6.0$ Hz, 2H, CH_2), 7.18–7.39 (m, 8H, ArH and NH), 7.47 (d, $J = 8.5$ Hz, 1H, CH), 7.60 (d, $J = 8.0$ Hz, 1H, CH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 151.5$, 141.5, 139.9, 137.1, 136.8, 129.8, 128.3, 127.8, 127.3, 123.7, 123.6, 65.8, 45.8, 43.6 ppm; IR (KBr): $\bar{\nu} = 3{,}400$ (NH), 1,560, 1,500, 1,085 cm^{-1}.

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