One-Pot Synthesis of Novel Pyrrolo[1,2-*a*]quinoxaline-4(5*H*)-ones Using Benzene-1,2-diamine, Acetylenedicarboxylates, and *β*-Nitrostyrene Derivatives

by **Loghman Moradi***[a]), **Mohammad Piltan**[b]), and **Golaleh Abasi**[a])

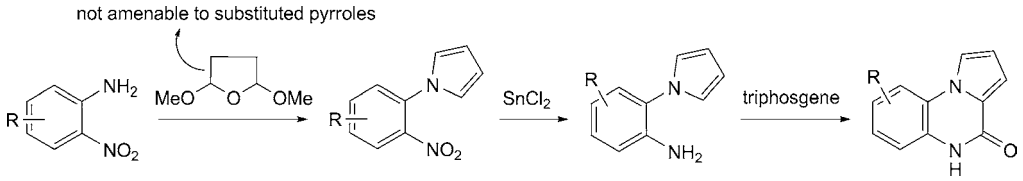
[a]) Chemistry Department, Kurdistan University, P.O. Box 416, Sanandaj, Iran (phone: +98-871-6662740; fax: +98-871-6624133; e-mail: l.moradi@uok.ac.ir, loghman352@yahoo.com)

[b]) Young Researchers and Elites Club, Sanandaj Branch, Islamic Azad University, Sanandaj, Iran

The reaction between a variety of *o*-phenylenediamines (= benzene-1,2-diamines), dialkyl acetylenedicarboxylates, and derivatives of nitrostyrene (= (*E*)-(2-nitroethenyl)benzene) in the presence of sulfamic acid (SA; H_3NSO_3) as catalyst led to the corresponding pyrrolo[1,2-*a*]quinoxaline-4(5*H*)-one derivatives in high yields.

Introduction. – The common method for the construction of pyrrolo[1,2-*a*]quinoxalines starts from 2-nitroanilines and proceeds in three steps (pyrrole ring formation, NO_2 group reduction, and cyclization with triphosgene (= bis(trichloromethyl) carbonate) as outlined in *Scheme 1*.

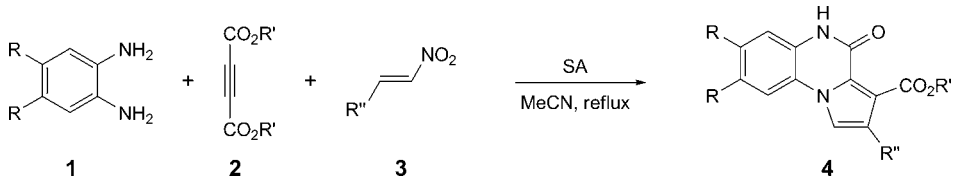
Scheme 1



This three-step procedure has limitations regarding substitution patterns on the pyrrole core [1]. Over the past decade, combinatorial methods using multicomponent reactions (MCRs) have become a powerful, fast, and convenient protocol for the construction of complex molecules. MCRs, by virtue of their convergence, productivity, facile execution, and generally high yields of products, have attracted much attention with respect to combinatorial chemistry [2–4]. Recently, utilization of a solid organic acid, sulfamic acid (H_2NSO_3H; SA), in catalytic and organic reactions as an alternative for conventional acidic materials has received much attention due to its unique features (nonvolatile, nonhygroscopic, odorless, and white crystalline solid with outstanding stability) [5]. As part of our study on the development of new routes to heterocyclic and carbocyclic systems [6–8], we here report a simple one-pot synthesis of functionalized pyrrolo[1,2-*a*]quinoxalines **4**. The reaction of *o*-phenylenediamines (= benzene-1,2-diamines) **1**, acetylenedicarboxylates **2**, and *β*-nitrostyrene (= (*E*)-(2-

nitroethenyl)benzene) derivatives **3** in the presence of SA leads to the corresponding functionalized pyrrolo[1,2-*a*]quinoxalines **4a**–**4l** in good yields (*Scheme 2*).

Scheme 2



Results and Discussion. – First, we studied the reaction of *o*-phenylenediamine, diethyl acetylenedicarboxylate, and *β*-nitrostyrene to optimize the reaction conditions with respect to temperature, time, and the molar ratio SA substrate. We found that 20 mol-% of SA was sufficient to obtain the corresponding pyrrolo[1,2-*a*]quinoxaline **4a** in 88% yield within 24 h in MeCN at reflux. The reaction was conducted without SA as catalyst, and no product was obtained. We also examined other solvents such as MeOH, 1,2-dichloroethane, THF, and toluene. When MeOH, THF, or 1,2-dichloroethane were employed as solvents, approximately the same result as in MeCN, was obtained. However, the desired product was not obtained in toluene. Thus, the choice of an appropriate reaction medium is of crucial importance for successful synthesis in our work. After finding suitable conditions, the three-component reaction of *o*-phenylenediamines, dialkyl acetylenedicarboxylates, and also *β*-nitrostyrenes for the synthesis of pyrrolo[1,2-*a*]quinoxalines was studied. The results are compiled in the *Table*. The reactions allowed high functional group tolerance and afforded the corresponding quinoxalines with high efficiency (*Table*).

The ^{1}H- and ^{13}C-NMR spectra of the crude reaction mixtures clearly indicated formations of pyrrolo[1,2-*a*]quinoxalines **4a**–**4l** (*Table*). Products other than **4** could not be detected by NMR spectroscopy. The structures of compounds **4a**–**4l** were deduced from their elemental analyses, their IR, and ^{1}H- and ^{13}C-NMR data.

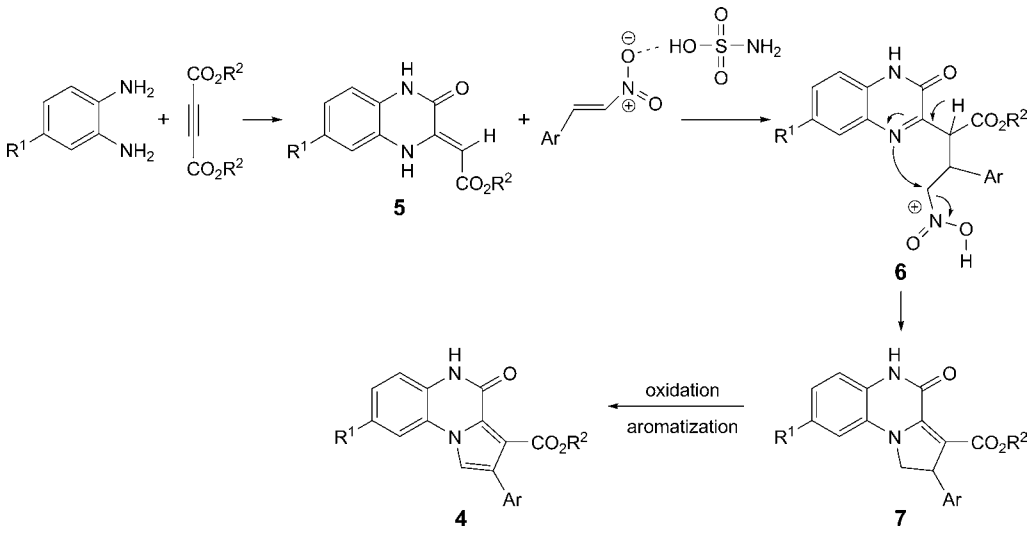
Table. *Formation of Alkyl 4,5-Dihydro-4-oxo-pyrrolo[1,2-*a*]quinoxaline-3-carboxylates* **4**

Entry	R	R′	R″	Product	Yield [%]
1	H	Et	Ph	**4a**	88
2	H	Et	4-Me–C_6H_4	**4b**	94
3	Me	Et	4-Me–C_6H_4	**4c**	91
4	H	Et	4-Cl–C_6H_4	**4d**	92
5	Me	Et	4-Cl–C_6H_4	**4e**	90
6	H	Me	4-Me–C_6H_4	**4f**	92
7	Me	Me	Ph	**4g**	91
8	H	Me	Ph	**4h**	88
9	H	Me	4-Cl–C_6H_4	**4i**	89
10	Me	Me	4-Me–C_6H_4	**4j**	91
11	H	ᵗBu	Ph	**4k**	77
12	H	ᵗBu	4-Cl–C_6H_4	**4l**	76

For example, the ^{1}H-NMR spectrum of **4a** exhibited two single sharp lines readily recognized as arising from NH (δ(H) 11.48) and H–C(1) of the pyrrole ring (δ(H) 8.52). The signals at δ(H) 1.23 (t, $^3J = 7.1$, Me) and 4.28 (q, $^3J = 7.1$, CH_2) were attributed to the $MeCH_2OCO$ group at C(3). The Ph moiety gave rise to characteristic signals in the aromatic region of the spectrum. The IR spectrum of **4a** displayed characteristic amide C=O, ester C=O, and N–H vibrations at 1662, 1721, and 3314 cm^{-1}, respectively. The mass spectrum of **4a** displayed a molecular-ion peak at m/z 332. The ^{1}H-decoupled ^{13}C-NMR spectrum of **4a** showed 17 distinct resonances, which confirmed the proposed structure.

Although the mechanistic details of the reaction are not known, a plausible rationalization is presented in *Scheme 3*. On the basis of well-established chemistry of amines and DMAD [9], reaction between *o*-phenylenediamine and dialkyl acetylenedicarboxylate affords dihydroquinoxaline **5**. Compound **5** possesses enamine character [10] and thus readily reacts with β-nitrostyrene which is activated by SA to generate the intermediate **6**. The subsequent cyclization of intermediate **6**, followed by the elimination of the NO_2 group, leads to the pyrrole precursor **7**. The possibly oxidative dehydrogenation of **7** results in pyrrolo[1,2-*a*]quinoxalin-4(5*H*)-one **4**.

Scheme 3



In conclusion, the reaction between *o*-phenylenediamine, dialkyl acetylenedicarboxylates, and β-nitrostyrene derivatives in the presence of SA offers a simple one-pot access to pyrrolo[1,2-*a*]quinoxaline-4(5*H*)-one derivatives of potential synthetic and pharmacologically interest. A wide range of these fused heterocycles bearing different functional groups such as Me, ester, and Cl could be prepared from suitable substrates, thereby providing a versatile and reliable method for the synthesis of these pharmaceutically interesting compounds. The present method has the advantage of being performed under one-pot multicomponent conditions, and requiring no modification of

the reactants. The simplicity of the procedure renders it an interesting alternative to other known approaches.

Experimental Part

General. Compounds **1** and **2** were obtained from *Merck* and were used without further purification. β-Nitrostyrene was synthesized as described in [11]. M.p.: *Electrothermal 9100* apparatus. IR Spectra: *Shimadzu IR-460* spectrometer; in KBr; $\tilde{\nu}$ in cm^{-1}. ^{1}H- and ^{13}C-NMR spectra: *Bruker DRX-250.1 Avance* instrument; in (D_6)DMSO, at 250.1 and 62.9 MHz, resp.; δ in ppm rel. to Me_4Si, *J* in Hz. MS: *Finnigan-MAT-8430* mass spectrometer, 70 eV; *m/z* (rel. %). Elemental analyses for C, H, and N: *Heraeus CHN-O-Rapid* analyzer.

General Procedure for the Preparation of Compounds **4**. In a round-bottom flask equipped with a magnetic stirrer, a mixture of diethyl acetylenedicarboxylate (1.0 mmol), and *o*-phenylenediamine (1.2 mmol) in MeCN (3 ml) was stirred vigorously at r.t. Then, β-nitrostyrene (1.0 mmol) and SA (20 mol-%) were added. Then, the mixture was heated at reflux for 24 h. Upon completion, the mixture was cooled to r.t. and then poured into H_2O (3 ml). The solid product was removed by filtration and purified by recrystallization from 95% EtOH to afford the pure compounds **4**.

*Ethyl 4,5-Dihydro-4-oxo-2-phenylpyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4a**). Yield: 0.29 g (88%). White crystals. M.p. 240–242°. IR: 3419 (NH, br.), 1721 (C=O), 1662 (C=O), 1615, 1533, 1403. ^{1}H-NMR: 1.23 (*t*, ^3J = 7.1, Me); 4.28 (*q*, ^3J = 7.1, CH_2O); 7.22–7.54 (*m*, 8 CH); 8.13 (*d*, ^3J = 7.7, CH); 8.53 (*s*, CH–N); 11.44 (*s*, NH). ^{13}C-NMR: 14.3 (Me); 61.4 (CH_2O); 115.8 (N–CH); 115.9 (C(3) or C(3a)); 117.0 (CH); 117.6 (CH); 121.5 (C); 122.2 (CH); 123.2 (CH); 126.4 (C); 126.8 (C); 127.4 (2 CH); 127.7 (CH); 129.0 (N–CH); 129.2 (2 CH); 133.2 (C(3) or C(3a)); 154.3 (C=O); 166.1 (C=O). MS: 332 (M^+, 100), 303 (7), 287 (95), 260 (75), 241 (5), 229 (17), 216 (8), 203 (10). Anal. calc. for $C_{20}H_{16}N_2O_3$ (332.12): C 72.28, H 4.85, N 8.43; found: C 72.34, H 4.80, N 8.45.

*Ethyl 4,5-Dihydro-2-(4-methylphenyl)-4-oxopyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4b**). Yield: 0.32 g (94%). Grey crystals. M.p. 290–292°. IR: 3424 (NH, br.), 1723 (C=O), 1663 (C=O), 1615, 1514, 1386. ^{1}H-NMR: 1.22 (*t*, ^3J = 6.7, Me); 2.30 (*s*, Me); 4.26 (*q*, ^3J = 6.7, CH_2O); 7.21–7.42 (*m*, 7 CH); 8.13 (*d*, ^3J = 7.7, CH); 8.51 (*s*, CH–N); 11.46 (*s*, NH). ^{13}C-NMR: 14.3 (Me); 21.1 (Me); 61.4 (CH_2O); 115.7 (N–CH); 115.8 (C(3) or C(3a)); 117.0 (C); 117.5 (CH); 121.4 (C); 122.2 (CH); 123.2 (CH); 126.3 (CH); 126.7 (C); 127.2 (2 CH); 129.0 (C); 129.8 (2 CH); 130.3 (N–CH); 137.0 (C(3) or C(3a)); 154.3 (C=O); 166.2 (C=O). Anal. calc. for $C_{21}H_{18}N_2O_3$ (346.38): C 72.82, H 5.24, N 8.09; found: C 72.52, H 5.30, N 8.15.

*Ethyl 4,5-Dihydro-7,8-dimethyl-2-(4-methylphenyl)-4-oxopyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4c**). Yield: 0.32 g (91%). Grey crystals. M.p. 275–277°. IR: 3425 (NH, br.), 1726 (C=O), 1659 (C=O), 1616, 1540, 1392. ^{1}H-NMR: 1.21 (*t*, ^3J = 5.7, Me); 2.47 (*s*, Me); 2.49 (*s*, Me); 3.34 (*s*, Me); 4.25 (*q*, ^3J = 5.7, CH_2O); 7.06–7.40 (*m*, 5 CH); 7.98 (*d*, ^3J = 5.5, CH); 8.43 (*s*, N–CH); 11.38 (*s*, NH). ^{13}C-NMR: 14.3 (Me); 21.1 (2 Me); 21.4 (Me); 61.3 (CH_2O); 115.4 (N–CH); 115.7 (C(3) or C(3a)); 116.9 (C); 117.2 (CH); 120.1 (C); 121.2 (C); 124.0 (CH); 126.1 (CH); 127.2 (2 CH); 128.8 (C); 129.8); 130.3 (C); 136.3 (N–CH); 136.9 (C(3) or C(3a)); 154.4 (C=O); 166.3 (C=O). Anal. calc. for $C_{23}H_{22}N_2O_3$ (374.44): C 73.78, H 5.92, N 7.48; found: C 73.68, H 5.50, N 7.54.

*Ethyl 2-(4-Chlorophenyl)-4,5-dihydro-4-oxopyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4d**). Yield: 0.33 g (92%). Grey crystals. M.p. 315–317°. IR: 3420 (NH broad), 1721 (C=O), 1667 (C=O), 1614, 1535, 1386. ^{1}H-NMR: 1.20 (*t*, ^3J = 6.7, Me); 4.25 (*q*, ^3J = 6.7, CH_2O); 7.25–7.48 (*m*, 7 CH); 8.09 (*d*, ^3J = 7.2, CH); 8.53 (*s*, CH); 11.47 (*s*, NH). ^{13}C-NMR: 14.2 (Me); 61.6 (CH_2O); 115.8 (N–CH=C); 116.1 (C); 117.0 (C); 117.4 (CH); 121.6 (C); 122.0 (CH); 123.4 (CH); 125.1 (CH); 127.0 (C); 128.9 (C); 129.1 (2 CH); 129.2 (2 CH); 132.0 (N–CH=C); 132.4 (C); 154.2 (C=O); 166.0 (C=O). Anal. calc. for $C_{20}H_{15}ClN_2O_3$ (366.8): C 65.49, H 4.12, N 7.64; found: C 65.52, H 4.06, N 7.65.

*Ethyl 2-(4-Chlorophenyl)-4,5-dihydro-7,8-dimethyl-4-oxopyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4e**). Yield: 0.34 g (90%). White crystals. M.p. 318–320°. IR: 3424 (NH broad), 1724 (C=O), 1658 (C=O), 1616, 1536, 1425. ^{1}H-NMR: 1.21 (*t*, ^3J = 6.5, Me); 2.31 (*s*, Me); 2.37 (*s*, Me); 4.26 (*q*, ^3J = 6.5, CH_2O); 7.02–7.49 (*m*, 5 CH); 7.96 (*d*, ^3J = 8.0, CH); 8.50 (*s*, N–CH); 11.42 (*s*, NH). ^{13}C-NMR: 14.3 (Me);

21.1 (Me); 21.3 (Me); 61.5 (CH_2O); 115.9 (N–CH); 116.9 (C(3) or C(3a)); 118.4 (CH); 120.0 (C); 121.7 (C); 124.1 (CH); 124.9 (C); 128.8 (CH); 129.0 (2 CH); 129.2 (2 CH); 132.0 (C); 132.3 (C); 134.3 (N–CH); 136.5 (C(3) or C(3a)); 154.3 (C=O); 166.0 (C=O). Anal. calc. for $C_{22}H_{19}ClN_2O_3$ (394.86): C 66.92, H 4.85, N 7.09; found: C 65.90, H 4.75, N 7.05.

*Methyl 4,5-Dihydro-2-(4-methylphenyl)-4-oxopyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4f**). Yield: 0.30 g (92%). Grey crystals. M.p. 296–298°. IR: 3433 (NH, br.), 1730 (C=O), 1660 (C=O), 1615, 1422, 1382. ^{1}H-NMR: 2.29 (*s*, Me); 3.77 (*s*, MeO); 7.23–7.37 (*m*, 7 CH); 8.13 (br. *s*, CH); 8.52 (*s*, N–CH); 11.52 (*s*, NH). ^{13}C-NMR: 21.1 (Me); 52.8 (MeO); 115.7 (N–CH); 115.8 (C(3) or C(3a)); 117.0 (CH); 121.4 (C); 122.1 (CH); 123.3 (CH); 126.4 (CH); 126.8 (C); 127.1 (2 CH); 128.8 (C); 129.9 (2 CH); 130.2 (C); 137.0 (N–CH); 154.2 (C(3) or C(3a)); 162.8 (C=O); 166.8 (C=O). Anal. calc. for $C_{20}H_{16}N_2O_3$ (332.35): C 72.28, H 4.85, N 8.43; found: C 72.34, H 4.72, N 8.22.

*Methyl 4,5-Dihydro-7,8-dimethyl-4-oxo-2-phenylpyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4g**). Yield: 0.30 g (91%). Grey crystals. M.p. 297–299°. IR: 3435 (NH, br.), 1728 (C=O), 1659 (C=O), 1617, 1533, 1395. ^{1}H-NMR: 2.31 (*s*, Me); 2.39 (*s*, Me); 3.77 (*s*, MeO); 7.05–7.50 (*m*, 6 CH); 7.99 (*d*, ^3J = 8.0, CH); 8.48 (*s*, N–CH); 11.40 (*s*, NH). ^{13}C-NMR: 21.0 (Me); 21.1 (Me); 52.8 (MeO); 115.8 (N–CH); 116.9 (C(3) or C(3a)); 120.0 (CH); 121.9 (C); 124.1 (C); 126.2 (CH); 126.6 (C); 127.2 (2 CH); 127.7 (CH); 128.9 (CH); 129.3 (2 CH); 132.9 (C); 133.2 (N–CH); 136.4 (C(3) or C(3a)); 154.4 (C=O); 166.8 (C=O). MS: 346 (M^+, 100), 301 (88), 284 (32), 272 (30), 258 (8), 243 (11), 229 (10). Anal. calc. for $C_{21}H_{18}N_2O_3$ (346.39): C 72.82, H 5.28, N 8.04; found: C 72.78, H 5.20, N, 8.09.

*Methyl 4,5-Dihydro-4-oxo-2-phenylpyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4h**). Yield: 0.27 g (88%). Grey crystals. M.p. 268–270°. IR: 3433 (NH, br.), 1729 (C=O), 1656 (C=O), 1610, 1525, 1402. ^{1}H-NMR: 3.79 (*s*, MeO); 7.21–7.51 (*m*, 8 CH); 8.12 (*d*, ^3J = 8.0, CH); 8.52 (*s*, N–CH); 11.48 (*s*, NH). ^{13}C-NMR: 52.7 (MeO); 115.8 (N–CH); 115.9 (C(3) or C(3a)); 117.0 (CH); 117.1 (C); 121.6 (C); 122.1 (CH); 123.3 (CH); 126.4 (CH); 126.8 (CH); 127.3 (2 CH); 127.7 (N–CH); 128.9 (C); 129.3 (2 CH); 133.1 (C(3) or C(3a)); 154.3 (C=O); 166.7 (C=O). Anal. calc. for $C_{19}H_{14}N_2O_3$ (318.33): C 71.69, H 4.43, N 8.88; found: C 71.82, H 4.40, N 8.85.

*Methyl 2-(4-Chlorophenyl)-4,5-dihydro-4-oxopyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4i**). Yield: 0.31 g (89%). Grey crystals. M.p. 300–302°. IR: 3422 (NH, br.), 1724 (C=O), 1661 (C=O), 1619, 1457. ^{1}H-NMR: 3.79 (*s*, MeO); 6.83–8.20 (*m*, 7 CH); 8.31 (*d*, ^3J = 10.0, CH); 8.57 (*s*, N–CH); 11.50 (*s*, NH). ^{13}C-NMR: 52.9 (MeO); 115.9 (N–CH); 116.2 (C(3) or C(3a)); 117.1 (CH); 121.8 (C); 122.0 (C); 123.4 (CH); 125.1 (CH); 127.0 (CH); 129.0 (2 CH); 129.2 (C); 129.3 (2 CH); 131.5 (C); 132.0 (N–CH); 132.4 (C(3) or C(3a)); 154.2 (C=O); 166.5 (C=O). Anal. calc. for $C_{19}H_{13}ClN_2O_3$ (352.77): C 64.69, H 3.71, N 7.94; found: C 64.72, H 3.74, N 7.95.

*Methyl 4,5-Dihydro-7,8-dimethyl-2-(4-methylphenyl)-4-oxopyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4j**). Yield: 0.31 g (91%). Grey crystals. M.p. 288–290°. IR: 3425 (NH, br.), 1728 (C=O), 1661 (C=O), 1615, 1539, 1425. ^{1}H-NMR: 2.30 (*s*, 2 Me); 2.34 (*s*, Me); 3.77 (*s*, MeO); 7.09–7.36 (*m*, 5 CH); 7.98 (br. *s*, CH); 8.43 (*s*, N–CH); 11.39 (*s*, NH). ^{13}C-NMR: 21.1 (2 Me); 21.3 (Me); 52.8 (MeO); 115.4 (N–CH); 115.7 (C(3) or C(3a)); 116.9 (C); 120.0 (CH); 121.4 (C); 124.1 (C); 126.2 (CH); 126.5 (CH); 127.1 (2 CH); 128.8 (C); 129.9 (2 CH); 130.3 (C); 136.4 (N–CH); 137.0 (C(3) or C(3a)); 154.4 (C=O); 166.9 (C=O). Anal. calc. for $C_{22}H_{20}N_2O_3$ (360.42): C 73.32, H 5.59, N 7.77; found: C 73.44, H 5.46, N 7.67.

tert-*Butyl 4,5-Dihydro-4-oxo-2-phenylpyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4k**). Yield: 0.28 g (77%). Grey crystals. M.p. 280–282°. IR: 3431 (NH, br.), 1723 (C=O), 1654 (C=O), 1611, 1524, 1400. ^{1}H-NMR: 1.48 (*s*, ^{t}Bu); 7.21–7.51 (*m*, 8 CH); 8.21 (*s*, CH); 8.42 (*d*, ^3J = 8.0, N–CH); 11.48 (*s*, NH). ^{13}C-NMR: 29.9 (Me_3C); 80.2 (Me_3C); 115.8 (N–CH); 116.0 (C(3) or C(3a)); 116.1 (CH); 116.2 (C); 120.6 (C); 122.2 (CH); 123.4 (CH); 126.5 (CH); 126.9 (CH); 127.3 (2 CH); 127.8 (N–CH); 128.9 (C); 129.0 (2 CH); 133.1 (C(3) or C(3a)); 154.3 (C=O); 166.8 (C=O). Anal. calc. for $C_{22}H_{20}N_2O_3$ (360.42): C 73.22, H 5.59, N7.77; found: C 73.21, H 5.44, N 7.82.

tert-*Butyl 2-(4-Chlorophenyl)-4,5-dihydro-4-oxopyrrolo[1,2-*a*]quinoxaline-3-carboxylate* (**4l**). Yield: 0.30 g (76%).Grey crystals. M.p. 310–312°. IR: 3426 (NH, br.), 1723 (C=O), 1664 (C=O), 1612, 1450. ^{1}H-NMR: 1.57 (*s*, ^{t}Bu); 7.25–7.50 (*m*, 7 CH); 8.10 (*s*, CH); 8.57 (*s*, N–CH); 11.50 (*s*, NH). ^{13}C-NMR: 28.5 (Me_3C); 83.8 (Me_3C); 115.8 (N–CH); 116.2 (C(3) or C(3a)); 117.1 (CH); 122.1 (C); 124.44 (C); 125.2 (CH); 127.1 (CH); 129.0 (2 CH); 129.4 (2 CH); 132.0 (C); 133.5 (N–CH); 153.2

(C=O); 167.1 (C=O). Anal. calc. for $C_{22}H_{19}ClN_2O_3$ (394.86): C 66.92, H 4.85, N 7.09; found: C 66.72, H 4.84, N 7.12.

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