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3-(Nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-ones: synthesis and structure

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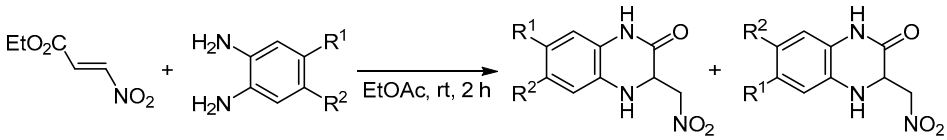


$R^1 = R^2 = H$, Me, Cl; R^1 = Me, Cl; $R^2 = H$

Reactions of ethyl 3-nitroacrylate with *o*-phenylenediamine and its substituted derivatives were used for the synthesis of 3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-ones, followed by structural characterization. These compounds were found to undergo elimination of nitromethane, leading to quinoxalin-2(1*H*)-ones.

Keywords: 1,2-diaminobenzene, ethyl 3-nitroacrylate, 3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one, quinoxalin-2(1*H*)-one, aza-Michael reaction.

Quinoxaline (1,4-benzopyrazine) is an important example of benzocondensed nitrogen-containing heterocycles.[1] This heterocycle is a part of the molecular structure of the natural antibiotic echinomycin,[2–4] as well as synthetic antibacterial drugs: quinoxidine and dioxidine.[5,6] The practical applications of quinoxaline derivatives, substituted quinoxalin-2(1*H*)-ones, show even greater promise.[7] Certain substituted quinoxalin-2(1*H*)-ones have exhibited antibacterial[8,9] and anti-inflammatory,[8] as well as antidiabetic[10] and antiviral activity, including effects against HIV.[11] Such compounds are being considered as effective inhibitors of aldose reductase[12] and partial antagonists of γ-aminobutyric acid.[13]

Compounds of quinoxalin-2(1*H*)-one series have been synthesized by various methods,[7] typically based on *o*-phenylenediamine reactions with bifunctional carbonyl substrates, such as acetylene dicarboxylates,[14] α-bromoesters,[15] glyoxylic acid and its esters,[16,17] as well as 2,3-epoxyaldehydes.[18] Representatives of nitroalkenes, ethyl 3-alkyl-3-nitroacrylates, have also been used successfully as substrates in reactions with *o*-phenylenediamine for the preparation of 3-(α-nitroalkyl)-3,4-dihydroquinoxalin-2(1*H*)-ones, although these products required additional chromatographic purification.[19]

In this work, we used a reaction of the simplest unsubstituted ethyl 3-nitroacrylate with a series of *o*-phenylenediamines to synthesize previously unknown derivatives of 3,4-dihydroquinoxalin-2(1*H*)-one that contained 3-nitromethyl groups.

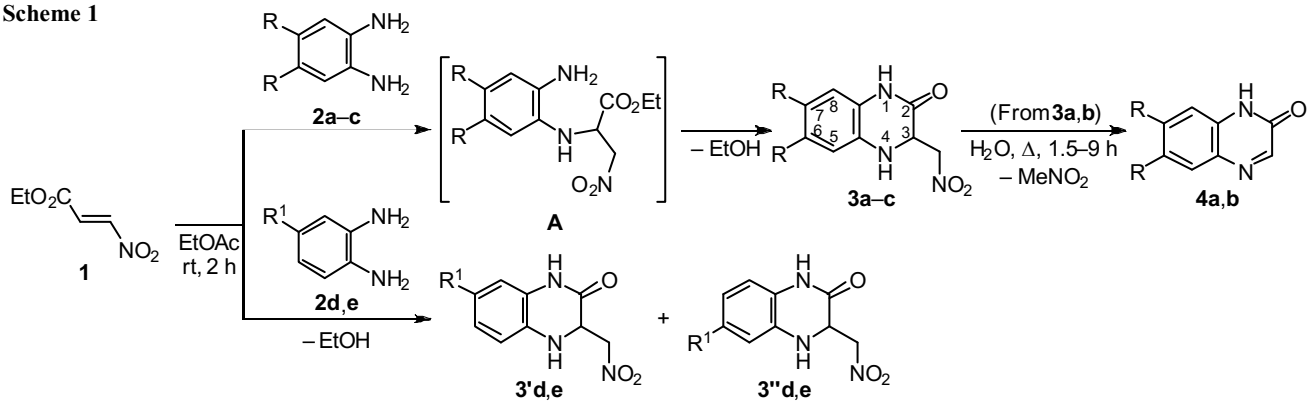
The reaction of ethyl 3-nitroacrylate (**1**) with *o*-phenylenediamines **2a**–**c** (in 1:1.25 ratio) in anhydrous ethyl acetate solution at room temperature was complete in 2 h and led to the formation of the respective 3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-ones **3a**–**c** in 51–81% yields (Scheme 1).

This tandem reaction involved initial formation of aza-Michael adduct **A**, which then underwent intramolecular heterocyclization *via* an attack by the second nucleophilic center at the ester group.

In contrast to the reactions of *o*-phenylenediamine (**2a**) and its symmetrically substituted analogs **2b**,**c**, the reactions of 4-methyl- and 4-chloro-1,2-diaminobenzenes (**2d**,**e**) with ethyl 3-nitroacrylate (**1**) under analogous conditions led to the formation of two regioisomers in the ratios of 17:10 (**3'd**:**3''d**) and 25:10 (**3'e**:**3''e**) according to ^{1}H NMR spectral data. Besides that, it was found that the synthesized dihydroquinoxalin-2(1*H*)-ones **3a**–**e** underwent elimination of nitromethane upon refluxing in aqueous solution, as demonstrated in the case of compounds **3a**,**b**, which were transformed in quantitative yields to the known quinoxalin-2(1*H*)-ones **4a**,**b**[16,20] under these conditions.

The structure of 3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-ones **3a**–**e** was characterized by IR, UV, and one-dimensional ^{1}H and ^{13}C NMR spectra, two-dimensional ^{1}H–^{1}H COSY, ^{1}H–^{1}H NOESY, ^{1}H–^{13}C HMQC, and ^{1}H–^{13}C HMBC experiments, while the physicochemical characteristics of quinoxalin-2(1*H*)-ones **4a**,**b** matched those described in

Scheme 1



2–4 a R = H, **b** R = Cl; **2**, **3 c** R = Me; **2d**, **3'd, 3''d** R^1 = Me; **2e**, **3'e**, **3''e** R^1 = Cl

the literature.[16, 20] IR spectra of compounds **3a**–**e** contained absorption bands due to a covalent nitro group at 1542–1548 (ν_{as}) and 1377–1380 cm^{-1} (ν_s), characteristic for aliphatic nitro compounds,[21,22] absorption bands of amide carbonyl group at 1674–1679 cm^{-1}, as well as a set of absorption bands characteristic for amide and amine NH groups (3183–3188, 3366–3417 cm^{-1}). UV absorption spectra of compounds **3a**–**e** contained long-wavelength absorption maxima at 267–277 (ε 3000–4100 $l \cdot mol^{-1} \cdot cm^{-1}$) and 307–325 nm (ε 4300–5100 $l \cdot mol^{-1} \cdot cm^{-1}$), and were in agreement with the data from structurally related dihydroquinoxalinones.[23–25]

An interesting structural feature of the synthesized dihydroquinoxalinones **3a**–**e** containing nitromethyl groups was the presence of a chiral C-3 atom in their molecules, creating a diastereotopic difference between the CH_2NO_2 group protons and forming an ABC spin system with the 3-CH methine proton. The signal of the latter (H_C) was observed as a multiplet at 4.38–4.66 ppm, while the protons of 3-CH_2 methylene group (H_A and H_B) gave double doublets at 4.72–4.76 and 4.82–4.97 ppm ($^2J_{AB}$ = 13.6–14.1, $^3J_{AC}$ = 4.2–4.7, $^3J_{BC}$ = 5.0–5.5 Hz). This coupling pattern was manifested in ^1H–^1H COSY spectra by the respective cross peaks, while the presence of cross peaks between the H_C methine proton signal and the broadened split signal at 6.06–6.74 ppm allowed to assign the latter to the 4-NH amine proton. Such interpretation was also supported by ^1H–^1H NOESY spectra, which contained additional cross peaks between the broadened downfield singlet of 1-NH group and the signal of H-8 proton in the aromatic ring.

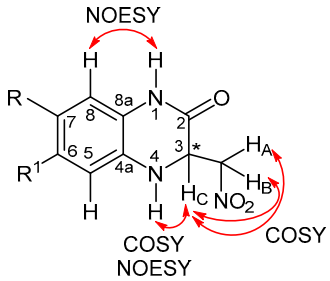


Figure 1. The main correlations in ^1H–^1H COSY and ^1H–^1H NOESY NMR spectra of compounds **3a**–**e**.

Thus, we have shown that the reaction of ethyl 3-nitroacrylate with 1,2-diaminobenzenes at room temperature gave good yields of heterocyclic compounds belonging to 3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one series. These compounds showed a tendency to eliminate nitromethane, resulting in conversion to quinoxalin-2(1*H*)-ones.

Experimental

IR spectra were recorded on a Shimadzu IR Prestige-21 FT-IR spectrometer in KBr pellets. Electronic absorption spectra were recorded on a Shimadzu UV–2401PC spectrophotometer in DMSO solution in quartz cuvettes (optical path length 1.01 mm). ^1H, ^{13}C, ^1H–^1H COSY, ^1H–^1H NOESY (mixing time 1 s), ^1H–^{13}C HMQC, and ^1H–^{13}C HMBC NMR spectra were acquired on a Jeol ECX–400A instrument at 400 (for ^1H nuclei) and 100 MHz (for ^{13}C nuclei) in DMSO-d_6. The internal standards were residual solvent protons for ^1H NMR spectra (δ 2.47 ppm) and solvent carbon atoms for ^{13}C NMR spectra (δ 40.0 ppm). Elemental analysis was performed on a EuroVector EA3000 CHN Dual analyzer.

Ethyl 3-nitroacrylate was synthesized according to a modified literature procedure.[26]

3-(Nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3a). A solution of ethyl 3-nitroacrylate (**1**) (160 mg, 1.103 mmol) in anhydrous ethyl acetate (10 ml) was added dropwise at room temperature to a solution of *o*-phenylenediamine (**2a**) (149 mg, 1.379 mmol) in anhydrous ethyl acetate (10 ml), and the reaction mixture was maintained for 2 h. After the removal of solvent, the resinified residue was crystallized from ethyl alcohol and the precipitate that formed was filtered off. Yield 169 mg (74%), yellow powder, mp 165–167°C (EtOH). IR spectrum, ν, cm^{-1}: 1380 (s), 1542 (s, NO_2), 1674 (vs, C=O), 3188 (w), 3382 (w), 3417 (m, NH). UV spectrum, λ_{max}, nm (ε, $l \times mol^{-1} \times cm^{-1}$): 268 (3000), 307 (4300). ^1H NMR spectrum, δ, ppm (*J*, Hz): 4.49–4.56 (1H, m, 3-CH); 4.76 (1H, dd, ^2J = 13.8, ^3J = 4.5) and 4.89 (1H, dd, ^2J = 13.8, ^3J = 5.2, CH_2NO_2); 6.32 (1H, br. d, ^3J = 1.9, 4-NH); 6.58 (1H, t, ^3J = 7.5, H-7); 6.64 (1H, d, ^3J = 7.3, H-5); 6.70 (1H, d, ^3J = 7.9, H-8); 6.74 (1H, t, ^3J = 7.5, H-6); 10.51 (1H, s, 1-NH). ^{13}C NMR spectrum, δ, ppm: 54.4 (C-3); 76.5 (CH_2NO_2); 114.1 (C-5); 115.5 (C-8); 118.7 (C-7); 123.6 (C-6); 125.6 (C-8a); 133.3 (C-4a); 164.5 (C=O). Found, %: C 52.23; H 4.43;

N 20.33. $C_9H_9N_3O_3$. Calculated, %: C 52.17; H 4.38; N 20.28.

6,7-Dichloro-3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3b) was obtained from 1,2-diamino-4,5-dichlorobenzene (**2b**) (244 mg, 1.379 mmol) and ethyl 3-nitroacrylate (**1**) (160 mg, 1.103 mmol) analogously to compound **3a**. Yield 154 mg (51%), yellow powder, mp 264–266°C (EtOH). IR spectrum, ν, cm^{-1}: 1379 (s), 1546 (s, NO_2), 1675 (vs, C=O), 3184 (w), 3363 (m), 3380 (m), 3397 (m, NH). UV spectrum, λ_{max}, nm (ε, $l \times mol^{-1} \times cm^{-1}$): 277 (3400), 325 (4900). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.60–4.66 (1H, m, 3-CH); 4.77 (1H, dd, ^2J = 14.1, ^3J = 4.2) and 4.97 (1H, dd, ^2J = 14.1, ^3J = 5.0, CH_2NO_2); 6.74 (1H, br. d, ^3J = 1.7, 4-NH); 6.80 (1H, s, H-5); 6.82 (1H, s, H-8); 10.74 (1H, s, 1-NH). ^{13}C NMR spectrum, δ, ppm: 53.8 (C-3); 76.7 (CH_2NO_2); 114.4 (C-5); 116.1 (C-8); 118.9 (C-7); 124.7 (C-6); 125.8 (C-8a); 133.7 (C-4a); 164.1 (C=O). Found, %: C 39.29; H 2.45; N 15.46. $C_9H_7Cl_2N_3O_3$. Calculated, %: C 39.15; H 2.56; N 15.22.

6,7-Dimethyl-3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3c) was obtained from 1,2-diamino-4,5-dimethylbenzene (**2c**) (188 mg, 1.379 mmol) and ethyl 3-nitroacrylate (**1**) (160 mg, 1.103 mmol) analogously to compound **3a**. Yield 189 mg (73%), orange powder, decomp. temp. 295°C (CCl_4–MeOH, 3:2). IR spectrum, ν, cm^{-1}: 1377 (s), 1544 (s, NO_2), 1679 (vs, C=O), 3184 (m), 3191 (m), 3387 (w), 3416 (m, NH). UV spectrum, λ_{max}, nm (ε, $l \times mol^{-1} \times cm^{-1}$): 271 (3000), 313 (4600). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.01 (3H, s, CH_3); 2.02 (3H, s, CH_3); 4.38–4.47 (1H, m, 3-CH); 4.72 (1H, dd, ^2J = 13.6, ^3J = 4.7) and 4.82 (1H, dd, ^2J = 13.6, ^3J = 5.5, CH_2NO_2); 6.06 (1H, br. d, ^3J = 2.1 4-NH); 6.44 (1H, s, H-5); 6.48 (1H, s, H-8); 10.38 (1H, s, 1-NH). ^{13}C NMR spectrum, δ, ppm: 19.1 (CH_3); 19.5 (CH_3); 54.7 (C-3); 76.4 (CH_2NO_2); 115.7 (C-5); 116.7 (C-8); 123.5 (C-8a); 126.1 (C-7); 130.9 (C-4a); 131.0 (C-6); 164.5 (C=O). Found, %: C 56.00; H 5.63; N 17.81. $C_{11}H_{13}N_3O_3$. Calculated, %: C 56.16; H 5.57; N 17.86.

7-Methyl-3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3d') and **6-methyl-3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3d")** were obtained from 1,2-diamino-4-methylbenzene (**2d**) (316 mg, 2.586 mmol) and ethyl 3-nitroacrylate (**1**) (300 mg, 2.069 mmol) analogously to compound **3a**. Yield 372 mg (81%), yellow powder, a mixture of compounds **3d'** and **3d"** (17:10) with mp 154–155°C (CCl_4–MeOH, 3:1). IR spectrum, ν, cm^{-1}: 1377 (s), 1548 (s, NO_2), 1676 (vs, C=O), 3183 (m), 3366 (m), 3388 (s, NH). UV spectrum, λ_{max}, nm (ε, $l \times mol^{-1} \times cm^{-1}$): 267 (3500), 311 (4600). **Isomer 3d'**. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.10 (3H, s, CH_3); 4.43–4.51 (1H, m, 3-CH); 4.74–4.78 (1H, m) and 4.85 (1H, dd, ^2J = 13.7, ^3J = 5.4, CH_2NO_2); 6.15 (1H, br. d, ^3J = 2.2, 4-NH); 6.51–6.57 (3H, m, H-5,6,8); 10.46 (1H, s, 1-NH). ^{13}C NMR spectrum, δ, ppm: 20.8 (CH_3); 54.5 (C-3); 76.4 (CH_2NO_2); 114.2 (C-5); 116.0 (C-8); 124.0 (C-6); 125.7 (C-8a); 127.6 (C-7); 130.9 (C-4a); 164.8 (C=O). **Isomer 3d"**. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.10 (3H, s, CH_3); 4.43–4.51 (1H, m, 3-CH); 4.70–4.74 (1H, m) and 4.86 (1H, dd, ^2J = 13.7, ^3J = 5.2, CH_2NO_2); 6.24 (1H, br. d, ^3J = 2.0, 4-NH); 6.39 (1H, dd, ^3J = 7.8, ^4J = 1.2, H-7); 6.45 (1H, br. s, H-5); 6.58 (1H, d, ^3J = 7.8, H-8); 10.43 (1H, s, 1-NH). ^{13}C NMR spectrum, δ, ppm: 21.2 (CH_3), 54.4 (C-3); 76.5 (CH_2NO_2); 114.6 (C-5); 115.4 (C-8); 119.2 (C-7); 123.3 (C-8a); 132.6 (C-6); 133.1 (C-4a); 164.3 (C=O). Found, %: C 54.41; H 5.01; N 18.76. $C_{10}H_{11}N_3O_3$. Calculated, %: C 54.29; H 5.01; N 19.00.

7-Chloro-3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3e') and **6-chloro-3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3e")** were obtained from 1,2-diamino-4-chlorobenzene (**2e**) (197 mg, 1.379 mmol) and ethyl 3-nitroacrylate (**1**) (160 mg, 1.103 mmol) analogously to compound **3a**. Yield 162 mg (61%), brown powder, a mixture of compounds **3e'** and **3e"** (25:10) with mp 182–185°C (CCl_4–MeOH, 3:2). IR spectrum, ν, cm^{-1}: 1378 (s), 1544 (s, NO_2), 1675 (vs, C=O), 3184 (m), 3371 (m), 3392 (s, NH). UV spectrum, λ_{max}, nm (ε, $l \times mol^{-1} \times cm^{-1}$): 273 (4100), 317 (5100). **Isomer 3e'**. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.54–4.61 (1H, m, 3-CH); 4.757 (1H, dd, ^2J = 13.9, ^3J = 4.4) and 4.927 (1H, dd, ^2J = 13.9, ^3J = 5.0, CH_2NO_2); 6.51 (1H, d, ^3J = 1.9, 4-NH); 6.63 (1H, d, ^3J = 8.4, H-5); 6.71 (1H, d, ^4J = 2.3, H-8), 6.76 (1H, dd, ^3J = 8.4, ^4J = 2.3, H-6); 10.64 (1H, br. s, 1-NH). ^{13}C NMR spectrum, δ, ppm: 54.1 (C-3); 76.5 (CH_2NO_2); 114.8 (C-8); 115.1 (C-5); 121.6 (C-7); 123.0 (C-6); 126.8 (C-8a); 132.3 (C-4a); 164.5 (C=O). **Isomer 3e"**. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.54–4.61 (1H, m, 3-CH); 4.761 (1H, dd, ^2J = 13.9, ^3J = 4.3) and 4.933 (1H, dd, ^2J = 13.9, ^3J = 5.1, CH_2NO_2); 6.59 (1H, dd, ^3J = 8.3, ^4J = 2.2, H-7); 6.60 (1H, br. s, 4-NH); 6.65 (1H, d, ^4J = 2.2, H-5); 6.67 (1H, d, ^3J = 8.3, H-8); 10.64 (1H, br. s, 1-NH). ^{13}C NMR spectrum, δ, ppm: 53.9 (C-3); 76.6 (CH_2NO_2); 113.1 (C-5); 116.5 (C-8); 117.8 (C-7); 124.5 (C-8a); 127.2 (C-6); 134.7 (C-4a); 164.1 (C=O). Found, %: C 44.64; H 3.48; N 17.27. $C_9H_8ClN_3O_3$. Calculated, %: C 44.74; H 3.39; N 17.39.

Quinoxalin-2(1*H*)-one (4a). A suspension of 3-nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (**3a**) (100 mg, 0.483 mmol) in water (10 ml) was refluxed for 1.5 h by heating a flask equipped with reflux condenser on a sand bath. The mixture was cooled to room temperature and maintained for 18 h, the precipitate that formed was filtered off on a sintered glass filter. Yield 69 mg (98%), light-yellow crystals, mp 263–265°C (H_2O) (mp 268–269°C (EtOH)[16]). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.24–7.30 (2H, m), 7.48–7.55 (1H, m) and 7.71–7.77 (1H, m, H-5,6,7,8); 8.13 (1H, s, 3-CH); 12.40 (1H, br. s, 1-NH). ^{13}C NMR spectrum, δ, ppm: 116.2; 123.8; 129.3; 131.3; 132.3; 132.5 (C-5,6,7,8,4a,8a); 152.1 (C-3); 155.4 (C=O).

6,7-Dichloroquinoxalin-2(1*H*)-one (4b) was obtained analogously to compound **4a** from 6,7-dichloro-3-(nitromethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (**3b**) (100 mg, 0.362 mmol) by refluxing in water (10 ml) for 9 h. Yield 59 mg (75%), yellowish-white crystals, decomp. temp. 286°C (H_2O) (decomp. temp. 275°C (2-PrOH)[20]). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.41 (1H, s) and 8.02 (1H, s, H-5,8); 8.18 (1H, s, 3-CH); 12.51 (1H, br. s, 1-NH).

Supplementary information file containing spectral data for the synthesized compounds is available at http://link.springer.com/journal/10593.

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