On the reactivity of benzo[*a*]phenazine-5,6-dione 7-oxides with methanolic alkali and pyrrolidine

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When treated with methanolic alkali, benzo[a]phenazine-5,6-dione 7-oxides convert into 11H-indeno[1,2-b]quinoxalin-11-ones, whereas 11-hydroxy-11-(pyrrolidine-1-carbonyl)-11H-indeno[1,2-b]quinoxaline 10-oxides form upon treatment with pyrrolidine. Keywords: benzo[a]phenazine-5,6-dione 7-oxides, 11H-indeno[1,2-b]quinoxalin-11-ones, benzilic acid rearrangement.

Phenazine N-oxides and dioxides attract the attention of researchers as substances exhibiting a range of biological activities. It is known that phenazine N-oxide possesses antibacterial activity¹, and its hydroxyl and amide derivatives exhibit antitumor activity^{2,3}. Also, phenazine 5,10-dioxides were recently identified as prodrugs for antitumor therapy due to cytotoxic properties.^{4,5} Study of the chemical properties of phenazine N-oxides, therefore, is of interest.

We have found that benzo[a] phenazine-5,6-dione 7-oxides 1a-e are converted into 11H-indeno[1,2-b]quinoxalin-11-ones 2a-e when treated with methanolic potassium hydroxide solution at 20-25°C. The reaction is accompanied by the release of CO₂, contraction of the ortho-quinoid ring, and deoxygenation of the N-oxide fragment (Scheme 1).

The structure of 11*H*-indeno[1,2-*b*]quinoxalin-11-ones 2a-e, currently studied in view of their antimicrobial,





1, 2 a–d R² = H, a,e R¹ = H, b R¹ = Me, c R¹ = Cl, d R¹ = F, e R² = Me

antihypertensive, antituberculosis, antimalaria, anti-inflammatory, anticonvulsant, antiHIV, and anticancer activity,⁶⁻⁸ was verified by counter synthesis of compound **2a** from ninhydrin and 1,2-diaminobenzene^{9,10} (Scheme 2).

Characteristically, the signals of the quinonoid carbon atoms in the ¹³C NMR spectra of benzo[*a*]phenazine-5,6-dione 7-oxides 1a-e are in the 171-178-ppm range, while the

Scheme 2



signal of the single carbonyl carbon atom in ¹³C NMR spectra of 11*H*-indeno[1,2-*b*]quinoxalin-11-ones **2a–e** is at ~190 ppm, which correlates with literature data.¹¹ Our attempts to isolate any of the intermediates in the reaction $1\rightarrow 2$ were unsuccessful. When phenazine *N*-oxides **1a–e** were treated with methanolic KOH at low temperature (0–5°C), as well as treating them with potassium phenoxide in dioxane (at 15–20°C), only the corresponding quinoxalines **2a–e** are formed.

The reaction $1\rightarrow 2$ differs from classical cases of benzilic acid rearrangement of *ortho*-quinonoid polycyclic compounds such as phenanthrenequinone 3^{12} reaction with alkalis where the main products are derivatives of 9-hydroxyfluorene-9-carboxylic acid 4 (Scheme 3).

Notably, the rearrangement of phenanthrenequinones into 9-hydroxyfluorene-9-carboxylic acids is greatly facilitated by electron-withdrawing substituents in the benzenoid fragment of phenanthrenequinone. Thus, unsubstituted phenanthrenequinone isomerizes to the corresponding hydroxyfluorenecarboxylic acid under prolonged heating at 80°C, 2- and 4-mononitro-Scheme 3



phenanthrenequinones at 65°C, whereas 2,7- and 4,5-dinitrophenanthrenequinones at 15°C.

Dibenzophenazinedione **5**, a closer related analog of benzo[*a*]phenazine-5,6-dione 7-oxides, transforms in low yield into 2-quinoxalylbenzoic acid **6** only when boiled with concentrated aqueous NaOH¹³ (Scheme 4).

Therefore, the presence of an *N*-oxide fragment in phenazinedione derivatives $1\mathbf{a}-\mathbf{e}$ enhances their reactivity towards and selectivity in the electrophilic reaction with alkalis. Increased electrophilic reactivity of phenazinedione *N*-oxides is also confirmed by the reaction of compounds $1\mathbf{a},\mathbf{b},\mathbf{e}$ with pyrrolidine, resulting in 11-hydroxy-11-(pyrrolidin-1-ylcarbonyl)-11*H*-indeno[1,2-*b*]quinoxaline 10-oxides $7\mathbf{a}-\mathbf{c}$ with preservation of the *N*-oxide moiety (Scheme 5). The structure and composition of compounds $7\mathbf{a}-\mathbf{c}$ were confirmed by physicochemical analysis. It should be noted that data on amine-induced cleavage of

Scheme 4



phenanthrenequinones and their diaza-analogs is absent in the literature.

To conclude, we have found unique reactions of benzo[*a*]phenazine-5,6-dione 7-oxides, with their direction explained by structural features of the starting materials, namely, the presence of *N*-oxide moiety in their molecules.

Scheme 5



Experimental

IR spectra were registered on a Shimadzu IRAffinity-1 spectrometer in KBr pellets. UV spectra were recorded on an Evolution 300 (10-mm cuvettes) spectrophotometer in acetonitrile. ¹H and ¹³C NMR spectra were acquired on a Bruker DRX spectrometer (500 and 125MHz, respectively) in DMSO- d_6 (compounds 1e, 2a-e) or CDCl₃ (compounds 7a-c), with TMS as internal standard. Chemical shifts of ¹³C nuclei were assigned using published data.¹¹ Mass spectra were recorded on a Finnigan MAT 8200 (EI ionization, 70 eV) mass spectrometer. High-resolution mass spectra for compounds 1e and 2e were recorded on a Thermo Scientific DFS (direct injection, EI ionization, 70 eV), whereas for compounds 7b,c they were acquired on a Bruker microOTOF II mass spectrometer with electrospray ionization in positive-ion mode (voltage applied to capillary 4500 V).¹⁴ Scanned mass range m/z 50– 3000. Direct infusion of acetonitrile solution of samples by syringe pump was used, flow rate 3 ml/min. Nitrogen nebulizing gas (4 l/min), interface temperature 180°C. Elemental analysis was performed on a EURO EA 3000 Elemental Analyzer. Melting points were determined on a Boetius heating bench. Monitoring of the reaction progress and assessment of the purity of synthesized compounds was done by TLC on Silufol UV-254 plates (eluent: toluene-acetone, 10:1).

The starting benzo[*a*]phenazine-5,6-dione 7-oxides 1a-d were prepared according to published procedures,¹⁵ while oxide 1e was prepared analogously. 4-(*o*-Tolylamino)-1,2-naphthoquinone was synthesized following a published method.¹⁶

11-Methyl-5,6-dioxobenzo[*a*]phenazine 7-oxide (1e). Nitrosylsulfuric acid, prepared from sodium nitrite (2.50 g, 35 mmol) and concentrated sulfuric acid (15 ml) was added over 5 min to a solution of 4-(o-tolylamino)-1,2-naphthoquinone (2.63 g, 10 mmol) in glacial acetic acid (60 ml). The reaction mixture was stirred for 1 h at 15-20°C, then poured with stirring into ice/water mixture (200 ml). The formed precipitate was filtered off, washed with water until neutral reaction, and dried. Yield 2.60 g (90%), red crystals, mp 248°C (DMF). IR spectrum, v, cm⁻¹: 1356 (N–O), 1672 (C=O). ¹H NMR spectrum, δ, ppm (J, Hz): 2.85 (3H, s, CH₃); 7.50– 7.63 (2H, m, H-3,9); 7.90 (1H, d, J = 7.2, H-10); 7.96 (1H, t. J = 7.8, H-2); 8.11 (1H, d, J = 7.8, H-1); 8.31 (1H, d, J = 8.6. H-4); 8.85 (1H, d, J = 7.9, H-8). ¹³C NMR spectrum, δ , ppm: 17.3 (CH₃); 117.4 (C-10); 126.7 (C-8); 128.4 (C-3); 131.1 (C-2); 132.2 (C-9); 132.3 (C-11a); 133.6 (C-12b); 135.0 (C-4a); 135.6 (C-1); 138.3 (C-7a); 139.1 (C-11); 142.5 (C-12a); 149.4 (C-6a); 171.5 (C-5); 177.7 (C-6). Found, *m/z*: 290.0684 [M]⁺. C₁₇H₁₀N₂O₃. Calculated, *m/z*: 290.0686. Found, %: C 69.86; H 3.28; N 9.66. C₁₇H₁₀N₂O₃. Calculated, %: C 70.34; H 3.47; N 9.65.

11H-Indeno[1,2-b]quinoxalin-11-one (2a). Benzo[a]phenazine-5,6-dione 7-oxide (1a) (0.8 g, 2.9 mmol) was added to a solution of KOH (1.0 g, 0.018 mol) in MeOH (15 ml), and the resulting mixture heated for 60 min at 20-25°C. Then 5% aqueous hydrochloric acid was added to the mixture. The precipitate was filtered off, washed with water followed by ethanol. Yield 0.56 g (83%), yellow crystals, mp 227°C (toluene) (mp 195–200°C⁶, mp 222°C⁷). IR spectrum, v, cm⁻¹: 1730 (C=O). UV spectrum, λ_{max} , nm $(\log \epsilon)$: 286 (4.54). ¹H NMR spectrum, δ , ppm (*J*, Hz): 7.71 (1H, t, J = 7.2, H-3); 7.83-7.90 (2H, m, H-7.8); 7.88 (1H, m, H-7.8);d, J = 7.2, H-4), 7.92 (1H, t, J = 7.2, H-2); 8.08–8.13 (2H, m, H-6,9); 8.17 (1H, d, J = 7.2, H-1). ¹³C NMR spectrum, δ , ppm: 122.8 (C-4); 124.7 (C-2); 129.8 (C-7); 130.9 (C-8); 131.4 (C-3); 132.9 (C-6); 133.3 (C-9); 137.1 (C-11a); 137.4 (C-1); 141.5 (C-4a); 142.3 (C-5a); 142.6 (C-4b); 150.3 (C-9a); 156.9 (C-10a); 189.8 (C-11). Mass spectrum, m/z (I_{rel} ,%): 232 [M]⁺(100), 204 (59), 76 (51), 32 (48). Found, %: C 77.61; H 3.42; N 11.84. C₁₅H₈N₂O. Calculated, %: C 77.58; H 3.47; N 12.06.

8-Methyl-11*H*-indeno[1,2-*b*]quinoxalin-11-one (2b) was synthesized analogously to compound 2a. Yield 0.55 g (77%), yellow crystals, mp 230°C (benzene). IR spectrum, v, cm⁻¹: 1720 (C=O). UV spectrum, λ_{max} , nm (log ϵ): 295 (4.55). ¹H NMR spectrum, δ, ppm (*J*, Hz): 2.56 (3H, s, 8-CH₃); 7.68 (1H, t, J = 7.8, H-3); 7.74 (1H, d, J = 8.5, H-7); 7.85 (1H, t, J = 7.8, H-2); 7.86 (1H, d, J = 7.8, H-4); 7.94 (1H, s, H-9); 8.01 (1H, d, J = 8.5, H-6); 8.04 (1H, d, J = 7.8, H-1). ¹³C NMR spectrum, δ , ppm: 21.6 (CH₃); 122.6 (C-4); 124.7 (C-2); 129.4 (C-7); 130.4 (C-3); 133.0 (C-6); 134.9 (C-9); 136.9 (C-1); 137.4 (C-11a); 141.0 (C-8); 141.2 (C-4a); 141.6 (C-5a); 142.4 (C-4b), 150.1 (C-9a); 156.3 (C-10a); 190.0 (C-11). Mass spectrum, m/z (I_{rel} ,%): 246 [M]⁺(100), 218 (39), 89 (36), 32 (71). Found, %: C 77.61; H 4.03; N 11.12. C₁₆H₁₀N₂O. Calculated, %: C 78.04; H 4.09; N 11.38.

8-Chloro-11*H***-indeno[1,2-***b***]quinoxalin-11-one (2c) was synthesized analogously to compound 2a. Yield 0.45 g**

(58%), yellow crystals, mp 282°C (benzene). IR spectrum, v, cm⁻¹: 1730 (C=O). UV spectrum, λ_{max} , nm (log ε): 291 (4.52). ¹H NMR spectrum, δ , ppm (*J*, Hz): 7.74 (1H, t, *J* = 7.5, H-3); 7.89 (1H, d, *J* = 7.5, H-4); 7.91 (1H, t, *J* = 7.5, H-2); 7.95 (1H, dd, *J* = 7.4, *J* = 2.4, H-7); 8.1 (1H, d, *J* = 7.4, H-6); 8.16 (1H, d, *J* = 7.5, H-1); 8.28 (1H, d, *J* = 2.4, H-9). ¹³C NMR spectrum, δ , ppm: 122.6 (C-4); 124.9 (C-2); 130.4 (C-3); 130.7 (C-7); 132.8 (C-6); 133.2 (C-9); 136.1 (C-8); 136.7 (C-1); 137.0 (C-11a); 141.4 (C-4a); 141.7 (C-5a); 143.0 (C-4b); 150.8 (C-9a); 156.7 (C-10a); 189.6 (C-11). Mass spectrum, *m/z* (*I*_{rel},%): 268 [M(³⁷Cl)]⁺ (35), 266 [M(³⁵Cl)]⁺ (100), 75 (45), 50 (35), 32 (24). Found, %: C 67.41; H 2.54; N 10.49; CI 13.30. C₁₅H₇ClN₂O. Calculated, %: C 67.56; H 2.65; N 10.50; Cl 13.29.

8-Fluoro-11*H***-indeno[1,2-***b***]quinoxalin-11-one (2d) was prepared analogously to compound 2a. Yield 0.59 g (81%), yellow crystals, mp 298°C (benzene). IR spectrum, v, cm⁻¹: 1724 (C=O). UV spectrum, \lambda_{max}, nm (log \varepsilon): 288 (4.52). ¹H NMR spectrum, \delta, ppm (***J***, Hz): 7.69–7.73 (2H, m, H-2,3); 7.81–7.86 (1H, m, H-7); 7.88 (1H, d,** *J* **= 7.5, H-4); 7.96 (1H, d,** *J* **= 10.0, H-6); 8.06 (1H, d,** *J* **= 7.5, H-4); 7.96 (1H, d,** *J* **= 10.0, H-6); 8.06 (1H, d,** *J* **= 7.5, H-1); 8.17–8.22 (1H, m, H-9). ¹³C NMR spectrum, \delta, ppm: 115.3 (C-7); 115.5 (C-9); 122.5 (C-4); 124.9 (C-2); 131.5 (C-3); 132.6 (C-6); 136.5 (C-1); 137.0 (C-11a); 140.3 (C-4a); 141.5 (C-5a); 143.6 (C-4b); 150.0 (C-9a); 156.2 (C-10a); 163.2 (C-8); 189.6 (C-11). Mass spectrum,** *m/z* **(***I***_{rel},%): 250 [M]⁺(100), 222 (24), 94 (18), 75 (20), 50 (20). Found, %: C 72.07; H 2.80; N 11.47. C₁₅H₇FN₂O. Calculated, %: C 72.00; H 2.82; N 11.19.**

6-Methyl-11*H*-indeno[1,2-*b*]quinoxalin-11-one (2e) was synthesized similar to compound 2a. Yield 0.59 g (83%), yellow crystals, mp 210°C (toluene). IR spectrum, v, cm⁻¹: 1716 (C=O). UV spectrum, λ_{max} , nm (log ϵ): 299 (4.55). ¹H NMR spectrum, δ , ppm (*J*, Hz): 2.78 (3H, s, 6-CH₃); 7.65 (1H, t, J = 7.5, H-3); 7.68 (1H, t, J = 7.5, H-1); 7.77 (1H, d, J = 7.2, H-7); 7.86 (1H, t, J = 7.2, H-8); 7.88 (1H, d, J = 7.5, H-4); 7.99 (1H, d, J = 8.0, H-9); 8.07 (1H, d, J = 7.5, H-1). ¹³C NMR spectrum, δ , ppm: 17.2 (CH₃); 122.4 (C-4); 124.5 (C-2); 129.1 (C-7); 130.2 (C-8,9); 130.3 (C-8,9); 132.9 (C-3); 136.8 (C-6); 137.2 (C-1); 137.7 (C 4); 141.9 (C-4a); 141.4 (C-4b); 142.2 (C-5a); 149.6 (C-9a); 155.7 (C-10a); 189.8 (C-11). Found, m/z: 246.0789 [M]⁺. C₁₆H₁₀N₂O. Calculated, *m/z*: 246.0788. Found, %: C 78.16; H 3.90; N 11.35. C₁₆H₁₀N₂O. Calculated, %: C 78.04; H 4.09; N 11.38.

11-Hydroxy-11-(pyrrolidin-1-ylcarbonyl)-11*H***-indeno-[1,2-***b***]quinoxaline 10-oxide (7a). Pyrrolidine (0.2 g, 2.8 mmol) was added with stirring over 10 min at 20–25°C to a solution of compound 1a (0.56 g, 2.0 mmol) in tetrahydrofuran (10 ml). After 30 min, the formed precipitate was filtered off, washed on filter with water (50 ml), followed by ethanol (20 ml). Yield 0.53 g (76%), yellow crystals, mp 275°C (compounds 7a–c** begin to change visually at ~100°C). UV spectrum, λ_{max} , nm (log ϵ): 322 (4.47). ¹H NMR spectrum, δ , ppm (*J*, Hz): 1.63–1.73 (4H, m, 3,4-CH₂ pyrrolidine); 2.75–2.82 (2H, m) and 2.82–2.91 (2H, m, CH₂NCH₂); 7.68 (1H, t, *J* = 7.4, H-3); 7.82–7.89 (2H, m, H-7,8); 7.93 (1H, t, *J* = 7.4, H-2); 8.10 (1H, d, *J* = 7.4, H-1); 8.23 (1H, d, *J* = 8.4, H-6); 8.64 (1H, d, *J* = 8.4, H-9); 8.73 (1H, d, *J* = 7.8, H-4); 8.83 (1H, s, OH). Found, %: C 69.32; H 4.76; N 11.98. C₂₀H₁₇N₃O₃. Calculated, %: C 69.15; H 4.93; N 12.10.

11-Hydroxy-8-methyl-11-(pyrrolidin-1-ylcarbonyl)-11H-indeno[1,2-b]quinoxaline 10-oxide (7b) was prepared from compound **1b** (0.59 g, 2.0 mmol) and pyrrolidine (0.37 g, 5.2 mmol) similar to compound **7a**. Yield 0.53 g (73%), yellow crystals, mp 245°C. UV spectrum, λ_{max} , nm (log ε): 326 (4.42). ¹H NMR spectrum, δ , ppm (*J*, Hz): 1.63–1.73 (4H, m, 3,4-CH₂ pyrrolidine); 2.66 (3H, s, CH₃); 2.75–2.83 (2H, m) and 2.84–2.89 (2H, m, CH₂NCH₂); 7.68 (1H, t, *J* = 7.6, H-3); 7.73 (1H, d, *J* = 7.3, H-7); 7.83 (1H, t, *J* = 7.4, H-2); 8.08–8.13 (2H, m, H-1,6); 8.42 (1H, s, H-9); 8.69 (1H, d, *J* = 7.6, H-4); 8.93 (1H, s, OH). Found, *m/z*: 362.1496 [M+H]⁺. C₂₁H₂₀N₃O₃. Calculated, *m/z*: 362.1499. Found, %: C 69.82; H 5.09; N 11.30. C₂₁H₁₉N₃O₃. Calculated, %: C 69.79; H 5.30; N 11.63.

11-Hydroxy-6-methyl-11-(pyrrolidin-1-ylcarbonyl)-11*H*-indeno[1,2-*b*]quinoxaline 10-oxide (7c) was synthesized from compound 1e (0.59 g, 2.0 mmol) and pyrrolidine (0.37 g, 5.2 mmol) analogously to compound 7a. Yield 0.56 g (77%), yellow crystals, mp 250°C. UV spectrum, λ_{max} , nm (log ε): 327 (4.46). ¹H NMR spectrum, δ, ppm (J, Hz): 1.63-1.73 (4H, m, 3.4-CH₂ pyrrolidine); 2.86 (3H, s, CH₃); 2.75–2.83 (2H, m, CH₂NCH₂); 2.84– 2.89 (2H, m, CH₂NCH₂); 7.65–7.77 (3H, m, H-3,7,8); 7.83 (1H, t, J = 7.5, H-2); 8.10 (1H, d, J = 7.5, H-1); 8.47 (1H, d, J = 8.3, H-9; 8.75 (1H, d, J = 7.7, H-4); 8.91 (1H, s, OH). Found, m/z: 362.1496 $[M+H]^+$. C₂₁H₂₀N₃O₃. Calculated, m/z: 362.1499. Found, %: C 69.53; H 5.01; N 11.69. C₂₁H₁₉N₃O₃. Calculated, %: C 69.79; H 5.30; N 11.63.

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