Polycyclic heterocycles with acidic N-H groups IX[1]. The unexpected decomposition route of 2-(3-oxo-3,4-dihydroquinoxalin-2-yl)benzenediazosulfonates

Iveta Fryšová,[a] Zdeněk Trávníček,[b] Jan Slouka,[a] and Petr Cankař[a]*

[a]Department of Organic Chemistry, Faculty of Science, Palacký University, tř. 17. listopadu 1192/12, 771 46 Olomouc, Czech Republic
[b]Department of Inorganic Chemistry, Faculty of Science, Palacký University, tř. 17. listopadu 1192/12, 771 46 Olomouc, Czech Republic
E-mail: cankar@orgchem.upol.cz

DOI: http://dx.doi.org/10.3998/ark.5550190.0012.210

Abstract

The diazosulfonates obtained by diazocoupling of 2-(3-oxo-3,4-dihydroquinoxalin-2-yl)benzene-diazonium chloride and its 6,7-dimethyl-derivative **2a,b** with sodium sulfite decompose by an unique route in aqueous solution to afford 1-(1*H*-indazol-3-yl)-1*H*-benzimidazol-2(3*H*)-one **4a**, and its 5,6-dimethyl-derivative **4b**, respectively. The structure of **4a** was confirmed by single crystal X-ray analysis.

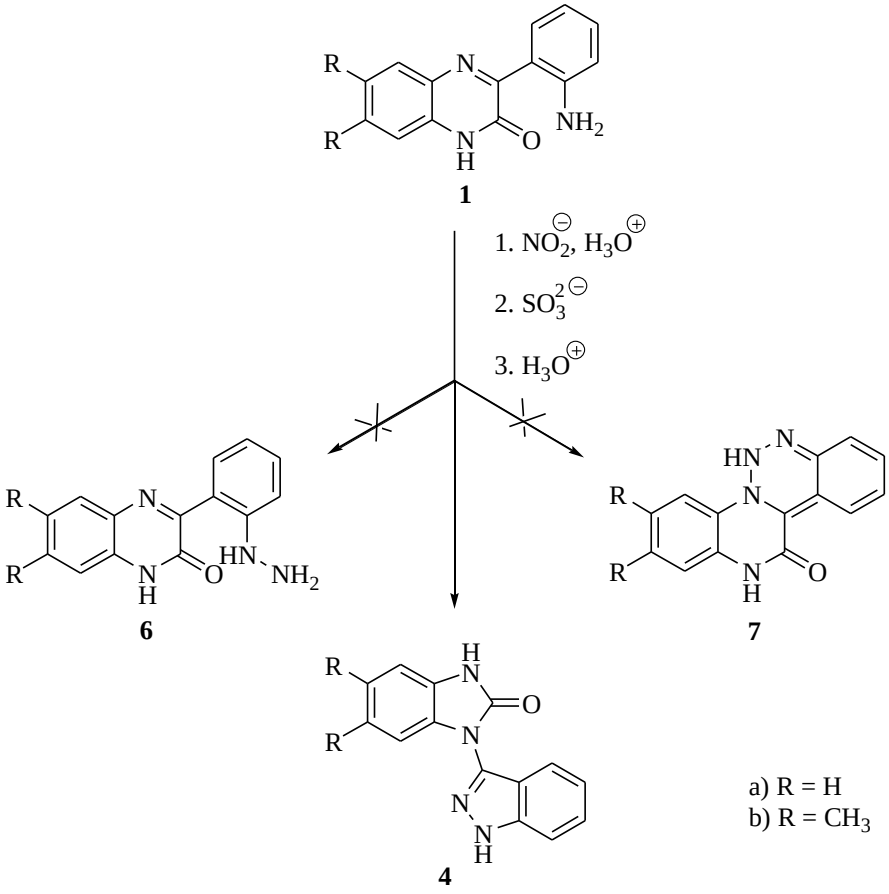
Keywords: Diazosulfonate rearrangement, 2-(3-oxo-3,4-dihydroquinoxalin-2-yl)-benzene-diazosulfonate, 1-(1*H*-indazol-3-yl)-1*H*-benzimidazol-2(3*H*)-ones

Introduction

Some time ago, we attempted to prepare 3-(2-hydrazinylphenyl)quinoxalin-2(1*H*)-one **6** by reduction of the corresponding diazonium salt **2a** with sodium sulfite. Instead of the intended product **6** we isolated a compound of molecular formula $C_{14}H_{10}N_4O$, which we considered to be 6*H*-quinoxalino[1,6-*c*][1,2,3]benzotriazin-13(12*H*)-one **7**.[2] However, Lyčka *et al.*[3] proved that our proposed structure was incorrect. By NMR spectroscopy and independent synthesis they showed that the correct structure is 1-(1*H*-indazol-3-yl)-1*H*-benzimidazol-2(3*H*)-one **4a** (Scheme 1).

The aim of the present work was to arrive at a spatial depiction of this molecule by single crystal X-ray analysis and to propose a probable mechanism for this anomalous reaction. Furthermore, in accordance with the latest knowledge, we also revise the structure of the product

formed from 3-(2-aminophenyl)-6,7-dimethylquinoxalin-2(1*H*)-one **1b** by the reaction described in our previous communication[2] on the basis of analogy.



Scheme 1

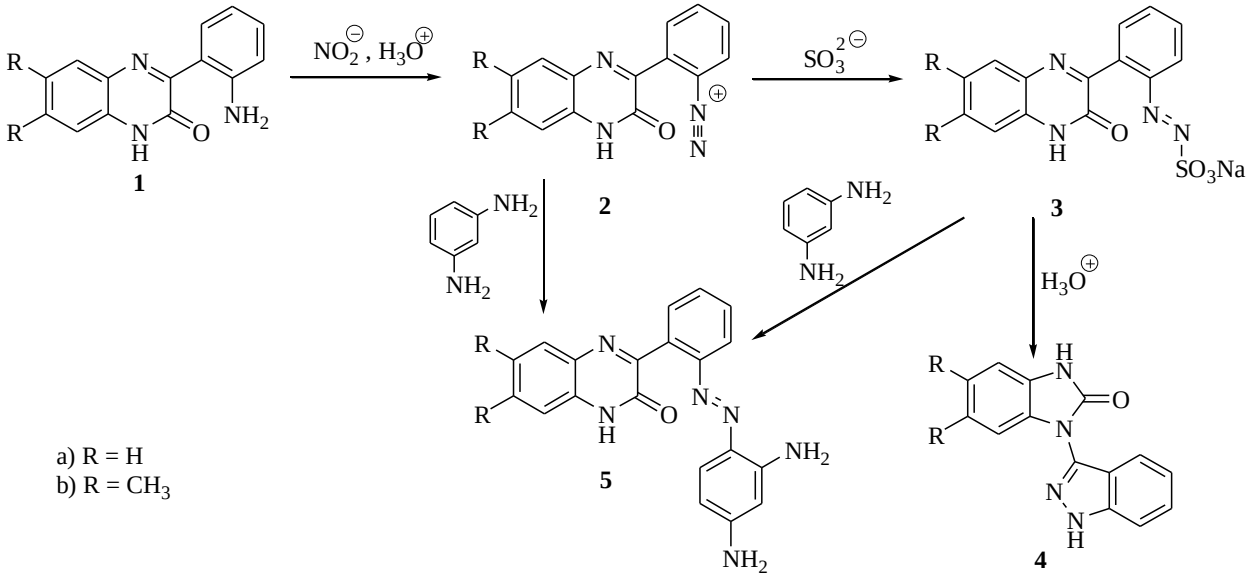
Results and Discussion

Primarily we were interested in whether the unusual transformation of diazonium salts **2a** and **2b**, as described in reference[2], occurs after the preceding coupling reaction with sodium sulfite forming the corresponding diazosulfonates **3a**, **3b**, or whether other factors are involved in the transformation. Therefore, the reaction of diazonium salts **2a** and **2b** with sodium sulfite was not carried out according to the previously published standard method for preparation of arylhydrazines, which we used in the previous paper[2]. As expected, we were successful in isolation of the diazosulfonates **3a** and **3b** by the reaction of diazonium salts with sodium sulfite without acidification of the reaction mixture.

Diazosulfonates **3a** and **3b** are bright yellow crystalline substances which are quite stable in the solid state at room temperature. In aqueous solution they transform slowly into 1-(1*H*-

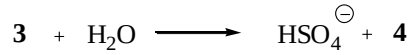
indazol-3-yl)-1*H*-benzo[*d*]imidazol-2(3*H*)-one **4a** resp. its 5,6-dimethylderivative **4b** even at low temperature. We were concerned with whether the tendency of these compounds **3a** and **3b** to undergo the described reaction would restrain their coupling ability, or not.

As reaction component we chose 1,3-diaminobenzene, which is a well-tested component for azocoupling reactions. We found that, in this regard, diazosulfonates **3a** and **3b** behaved identically to all known arenediazosulfonates[4] and it was possible to prepare the corresponding substituted chrysoidines **5a** or **5b**. The reaction rate of the described coupling reactions was substantially lower than that of the coupling reactions of diazonium salts **2a** or **2b** with the same compound. This corresponds to the known reactivity of diazosulfonates[4]. Interestingly, high antiprione activity[13] was discovered at the unsubstituted chrysoidine. From this point of view, substituted chrysoidines **5a** and **5b** may also possibly be of interest.



Scheme 2

As mentioned earlier, in aqueous solution the diazosulfonates **3a** and **3b** undergo a remarkable transformation yielding 1-(1*H*-indazol-3-yl)-1*H*-benzo[*d*]imidazol-2(3*H*)-one **4a** or its 5,6-dimethyl-derivative **4b**. The transformation proceeds rather rapidly and quantitatively as the diazosulfonates **3a** or **3b** are warmed in a highly acidic aqueous medium. This implies that diazosulfonates **3a** or **3b** are the key intermediates in the described[2,3] transformation of 3-(2-aminophenyl)quinoxalin-2(1*H*)-one **1a** and its 6,7-dimethyl derivative **1b** into compounds **4a** or **4b**. It is noteworthy that the described transformation of diazosulfonates **3** takes place even when mineral acids are absent. In this case the reaction is slower, but follows the same stoichiometry.



Also the quantity of hydrogen sulfate anion eliminated during the reaction precisely corresponds to the equations shown above. This was confirmed by adding $BaCl_2$ solution to the filtrate after the isolation of the compound **4.** The amount of precipitated $BaSO_4$ was precisely the stoichiometric amount. The product formed by the mentioned cleavage reaction of diazosulfonate **3a**, 1-(1*H*-indazol-3-yl)-1*H*-benzo[*d*]imidazol-2(3*H*)-one **4a**, has already been characterized in paper[2], however with an incorrect structure. It forms bright yellow crystals almost insoluble in water, and only slightly soluble in nonpolar solutions. During its formation from diazosulfonate **3a** and also during its crystallization from solvents containing water it crystallizes as the monohydrate **4a**×H_2O, which has been used for single crystal X-ray analysis.
The molecular structure of **4a**×H_2O is shown in Figure 1 while selected bond lengths and angles are given in Table 1. The molecule of **4a** contains two different N-containing heterocyclic rings: indazole (**A**) and benzimidazole (**B**). Each of the rings is essentially planar with the maximum deviation from the least-squares planes being 0.0114(33) Å for C11 (ring **A**), and 0.0120(29) Å for C6 (ring **B**). The dihedral angle between rings (**A**) and (**B**) is 47.663(54)°.

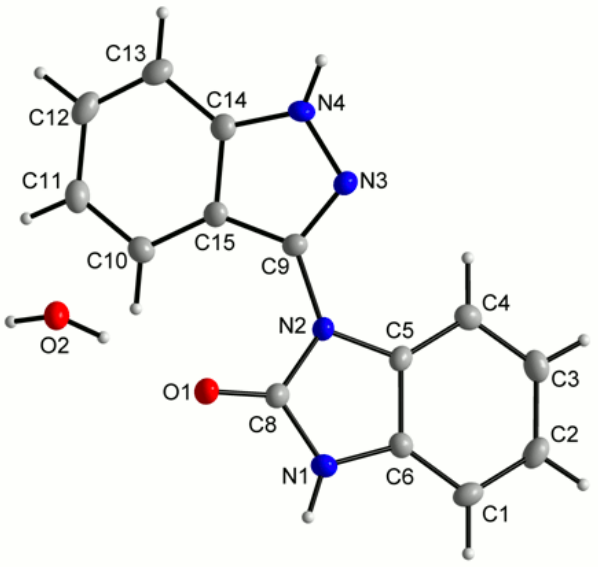


Figure 1. The molecular structure of compound **4a**×H_2O, showing the atom numbering scheme. Non-hydrogen atoms are drawn as thermal ellipsoids at the 50% probability level.

Both the oxygen atoms O1 and O2 lie nearly in the least-square plane B, with the deviations being 0.0329(20) Å (for O1) and 0.0225(20) Å (for O2). Surprisingly, no X-ray structure of the compounds involving the skeleton forming indazole and benzimidazole rings, and connected by a mode as in **4a**×H_2O, has been deposited in the Cambridge Structural Database (CSD, Version 5.29).[12] However, the bond lengths and angles are quite normal, thus supporting the presence of a high degree of aromaticity in **4a**. The secondary structure is further stabilized by intermolecular hydrogen bonds of the O-H…O, N-H…O and O-H…N type (Table 2, Figure 2).

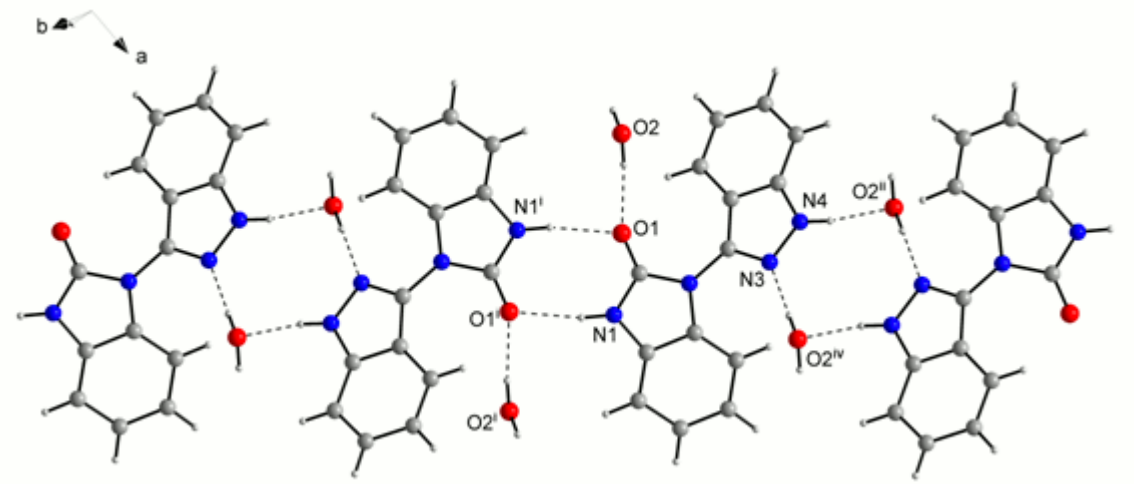
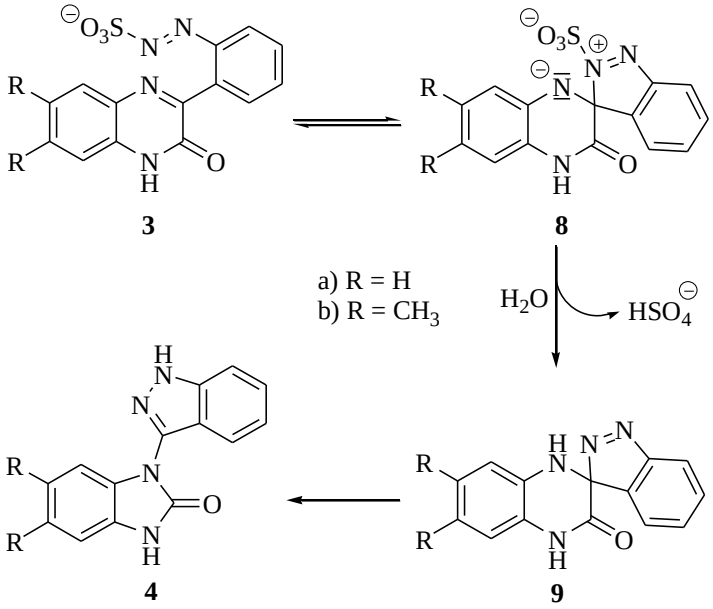


Figure 2. Part of the crystal structure of **4a**×H_2O, showing the formation of O—H…O, N—H...O and O—H…N hydrogen bonds (dashed lines). [Symmetry codes: (i) –x+1, -y+1, -z+1; (ii) –x+1, -y, -z, (iv) x+1, y, z].

The key step of the mentioned unusual transformation of diazosulfonates **3a** and **3b** into indazolylbenzimidazoles **4a** resp. **4b** is the contraction of the quinoxaline cycle to the benzimidazole ring. A possible mechanism could be initiated by a nucleophilic addition of the N atom of the diazosulfonate group to a C=N quinoxaline cycle bond resulting in the spirocycle **8**. After hydrolytic cleavage of hydrogen sulfate the spirocycle **9** rearranges into benzimidazole derivative **4**. This step, in which C-C bond splitting of the quinoxaline cycle occurs, differs this transformation from similar previously known reactions[5,14] in which the quinoxaline ring also contracts.



3 **8** **9** **4**

Scheme 3

Table 1. Selected bond lengths [Å] and angles [°] for **4a**×H_2O

O(1)-C(8)	1.248(4)	C(8)-N(1)-C(6)	110.3(2)
N(1)-C(8)	1.356(4)	C(8)-N(2)-C(5)	109.3(2)
N(1)-C(6)	1.396(4)	C(8)-N(2)-C(9)	124.9(2)
N(2)-C(8)	1.381(4)	C(5)-N(2)-C(9)	125.8(2)
N(2)-C(5)	1.407(4)	C(9)-N(3)-N(4)	106.0(2)
N(2)-C(9)	1.408(4)	C(14)-N(4)-N(3)	111.4(2)
N(3)-C(9)	1.321(4)	C(6)-C(5)-N(2)	106.5(2)
N(3)-N(4)	1.355(3)	C(1)-C(6)-C(5)	121.4(3)
N(4)-C(14)	1.353(4)	O(1)-C(8)-N(1)	127.0(3)
C(1)-C(6)	1.385(4)	O(1)-C(8)-N(2)	126.1(3)
C(5)-C(6)	1.393(4)	N(1)-C(8)-N(2)	106.9(2)
C(9)-C(15)	1.423(4)	N(3)-C(9)-N(2)	120.2(3)
C(14)-C(15)	1.397(4)	N(3)-C(9)-C(15)	111.5(2)
		N(2)-C(9)-C(15)	128.2(3)
		N(4)-C(14)-C(15)	107.5(3)
		H(1W)-O(2)-H(1V)	106(3)

Table 2. Hydrogen bonding geometry for **4a**×H_2O

D-H...A	*d*(D-H)	*d*(D...A)	*d*(H-A)	<(DHA)
O(2)-H(1V)...O(1)	0.927(19)	1.91(2)	2.823(3)	166(4)
N(1)-H(1A)...O(1)$^{(i)}$	0.88	1.98	2.843(3)	166.3
N(4)-H(4A)...O(2)$^{(ii)}$	0.88	1.90	2.774(3)	170.7
O(2)-H(1W)...N(3)$^{(iii)}$	0.938(19)	1.94(2)	2.869(3)	172(3)

Symmetry transformations used to generate equivalent atoms: (i): -x+1, -y+1, -z+1, (ii): -x+1, -y, -z, (iii): x-1, y, z.

Experimental Section

Melting points were determined on a Boetius stage apparatus. NMR spectra of solutions in DMSO-d_6 were measured on a Bruker Avance 300 spectrometer (300 MHz). Elemental analyses were obtained with an EA 1108 Elemental Analyzer (Fison Instrument). Mass spectrometric experiments were performed using an LCQ ion trap mass spectrometer (Finnigan MAT, San Jose, CA, USA).

X-ray diffraction data for the compound **4a**×H_2O were collected on a four-circle κ-axis Xcalibur2 diffractometer (Oxford Diffraction, Ltd.) equipped with a CCD detector using

monochromated (monochromator Enhance, Oxford Diffraction, Ltd.) Mo Kα radiation (λ = 0.71073 Å) at 110(2) K. Data collection, cell parameter refinements and data reduction were performed with the CrysAlis program package (Oxford Diffraction, Ltd.).[9] The structure was solved by direct methods using SHELXS-97 and all non-hydrogen atoms were refined anisotropically on all F^2 data using a full-matrix least-squares procedure (SHELXL-97) with weight: $w = 1/[\sigma^2(F_o)^2 + (0.07P)^2 + 0.80P]$, where $P = (F_o^2 + 2F_c^2)/3$.[10] All atoms were localized in differential maps of electron densities and their parameters were refined with a riding model, with C-H distances of 0.95 Å and N-H distances of 0.88 Å, and with U_{iso}(H) values of $1.2U_{eq}$(C,N), while parameters of the crystal water molecule were refined with the O-H distances restrained to 0.94(2) Å. Additional calculations were performed using the DIAMOND program.[11] The crystal data and the structure refinement of compound **4a**×H_2O are given in Table 3.

Table 3. Crystal data and structure refinement for **4a**×H_2O

Molecular formula	$C_{14}H_{12}N_4O_2$
Formula weight	268.28
Temperature, K	110(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 7.3378(3) Å, b = 9.3343(5) Å
	c = 9.6459(5) Å, α = 102.002(4) °
	β = 100.759(4) °, γ = 96.932(4) °
Volume	626.07(5) $Å^3$
Z, Calculated density	2, 1.423 g/cm^3
Absorption coefficient	0.100 mm^{-1}
F(000)	280
Crystal size, mm	0.35 x 0.30 x 0.30
Theta range for data collection	2.77 to 25.00 °
Limiting indices	$-8 \le h \le 8$, $-10 \le k \le 11$, $-10 \le l \le 11$
Reflections collected / unique	5238 / 2204, [R_{int} = 0.0233]
Completeness to θ = 25.0°	99.9 %
Data / restraints / parameters	2204 / 2 / 189
Goodness-of-fit on F^2	1.264
Final R indices [I>2σ(I)]	R_1 = 0.0519, wR_2 = 0.1580
R indices (all data)	R_1 = 0.0648, wR_2 = 0.1734
Largest diff. peak and hole	0.335 and -0.319 $e.A^{-3}$

Sodium 2-(3-oxo-3,4-dihydroquinoxalin-2-yl)benzenediazosulfonate (**3a**). To a mixture of 3-(2-aminophenyl)quinoxalin-2(1*H*)-one **1**[6,7] (474.5 mg, 2.0 mmol), water (60 ml) and 35% HCl (6 ml) cooled to 0-5 °C, a solution of $NaNO_2$ (142.0 mg, 2.06 mmol) in ice water (6 ml) was added

over 8 min and the resulting reaction mixture was stirred (ca. 1 hour) until a solution was obtained. During vigorous stirring a solution of Na_2SO_3 (12.0 g, 74.0 mmol) in ice water (55 ml) was quickly added to this solution. The bright yellow solution formed was stirred at the temperature between 0-5 °C until deposition of the crystalline precipitate started (after ca. 30-40 min). The solution was kept at of 0-5 °C for 48h. The bright yellow crystalline precipitate was later collected by suction, washed several times with a small amount of ice water and dried in the air at room temperature to constant weight. The yield of monohydrate was 586.0 mg (79%), mp 230 °C (decomp.) Collected filtrates were used for the preparation of compounds **4a**, see following. ^{1}H NMR (300 MHz, DMSO-d_6): δ 7.28 (m, 2H, ArH); 7.54 (m, 1H, ArH); 7.60 (m, 4H, ArH); 7.75 (dd, 1H, ArH, J_1=1.44 Hz, J_2= 9.72 Hz); 12.39 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ 115.2, 118.3, 123.0, 128.7, 130.0, 130.1, 130.3, 131.0, 131.9, 132.4, 134.1, 149.3, 154.2, 158.4. Anal. Calcd. for $C_{14}H_9N_4NaO_4S.H_2O$ = $C_{14}H_{11}N_4O_5SNa$ (370.32): C, 45.41; H, 2.91; N, 15.13; S, 8.66. Found: C, 45.32; H, 3.03; N, 14.96; S, 8.50.

Sodium 2-(6,7-dimethyl-3-oxo-3,4-dihydroquinoxalin-2-yl)benzenediazosulfonate (**3b**). This compound was prepared similarly to the compound **3a** using 3-(2-aminophenyl)-6,7-dimethylquinoxalin-2(1*H*)-one[8] (265.3 mg, 1.0 mmol) suspended in the mixture of ice water (40 ml), 35% HCl (3.8 ml), a solution of $NaNO_2$ (75.0 mg, 1.09 mmol) in ice water (5 ml) and a solution of Na_2SO_3 (7.6 g, 46.9 mmol) in ice water (30 ml). The yield was 322.8 mg (81%), m.p. 200-202 °C (decomp.). ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.29 (s, 3H, CH_3); 2.32 (s, 3H, CH_3); 7.09 (s, 1H, ArH); 7.53 (s, 1H, ArH); 7.62 (m, 4H, ArH); 12.26 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ 18.9, 19.7, 115.2, 117.9, 128.5, 128.8, 129.7, 130.3, 130.4, 130.9, 131.6, 134.6, 139.6, 149.4, 154.3, 157.0. Anal. Calcd. for $C_{16}H_{13}N_4NaO_4S\times H_2O$ = $C_{16}H_{15}N_4O_5SNa$ (398.37): C, 48.24; H, 3.80; N, 14.07; S, 8.05. Found: C, 48.30; H, 3.93; N, 14.03; S, 8.02. Collected filtrates were used for preparation of compounds **4b**, see following.

1-(1*H*-Indazol-3-yl)-1*H*-benzo[*d*]imidazol-2(3*H*)-one (**4a**)

(a) Decomposition of diazosulfonate (3a) in neutral medium. A mixture of diazosulfonate monohydrate **3a** (37.0 mg, 0.10 mmol) and water (10 ml) was stirred at room temperature until a solution was formed. This was placed in a water bath and after ca. 15 min. the crystalline precipitate began to deposit. The reaction mixture was heated in the water bath for 90 min. with constant stirring and cooled afterwards. Next day, the bright yellow crystalline precipitate was collected by suction, washed several times with water and dried in air. The yield of monohydrate was 25.4 mg (95%). This substance is in every attribute (m.p., ms, IR, NMR spectra, anal. C,H,N) identical with the one prepared previously[2,3].

(b) Decomposition of diazosulfonate (3a) in acidic medium. To the solution of monohydrate diazosulfonate **3a** (37.0 mg, 0.10 mmol) in water (9.0 ml), 35% HCl (1.0 ml) was added and reactive mixture was put into the boiling water bath. Just after less than half a minute the precipitate began to separate. After 15 min. of heating and cooling of reaction mixture it was collected by suction, washed with water and dried. The yield was 24.1 mg (90%). This substance is identical with the substance prepared previously. A solution of $BaCl_2$ (5 ml, 0.1 M) was added

into the filtrate. The amount of separated $BaSO_4$ precipitate (23.2 mg), obtained by filtration, washing with water and drying, precisely corresponds with 0.1 mmol of sulfate anions.

(c) Decomposition of diazosulfonate in the mother liquor after isolation (3a). To the collected filtrates after the isolation of the diazosulfonate **3a** (see procedure for preparation of **3a**) 35% HCl (12.0 ml) was added and the reaction mixture was left for three days at room temperature. The separated crystalline precipitate was later collected by suction, washed with water and dried. The yield was 75.3 mg (14% - related to the initial amino derivative **1a**). After the recrystallization from the acetic acid–aqua mixture the substance was identical with the substance as prepared previously.

1-(1*H*-Indazol-3-yl)-5,6-dimethyl-1*H*-benzo[*d*]imidazol-2(3*H*)-one (**4b**). This substance was prepared from diazosulfonate **3b** by the same two methods as the compound **4a** from diazosulfonate **3a**. The preparation was identical with those methods. The compound **4b** was obtained in the same way (mentioned above) also from the filtrate after the isolation of diazosulfonate **3b**. It is in every way congruent with the compound prepared in the way described in the paper[2] which was incorrectly presented as 2,3-dimethyl-6*H*-benzo[4,5][1,2,3]triazino[1,6-*a*]quinoxalin-12(13*H*)-one, numbered in this work by coincidence also as the compound **4b**.

3-(2´,4´-Diaminoazobenzene-2-yl)quinoxalin-2(1*H*)-one (**5a**)

(a) From diazonium salt (2a). To the mixture of aminoderivate **1a** (119.0 mg, 0.50 mmol), water (20 ml) and 35% HCl (1.1 ml) cooled in an ice-bath to 0-5 °C, a solution of $NaNO_2$ (36.5 mg, 0.53 mmol) in ice water (6 ml) was added portionwise. The reaction mixture was stirred in the ice bath until the solution was formed (approximately for 1h). Diazonium salt solution was added over 8 min. under stirring to the solution of 1,3-diaminobenzene (200.0 mg, 1.85 mmol) and CH_3COONa (1.2 g, 14.6 mmol) dissolved in water (20 ml), which had been already cooled to 0-5 °C. The reaction mixture was stirred for 30 min mostly in the cooling bath and then was left in the fridge at 0-5 °C. The red precipitate was the next day collected by suction, thoroughly washed with water and dried at the room temperature. The yield of dihydrate was 184.5 mg (94 %), m.p. 201-203 °C (ethanol/water). ^{1}H NMR (300 MHz, DMSO-d_6): δ 7.28 (m, 2H, ArH); 7.54 (m, 1H, ArH); 7.60 (m, 4H, ArH); 7.75 (dd, 1H, ArH, J_1=1.44 Hz, J_2=9.72 Hz); 12.39 (s, 1H, NH). MS (+APCI): *m/z* 357 $[M+H]^+$. Anal. Calcd. for $C_{20}H_{16}N_6O\times2H_2O$ = $C_{20}H_{20}N_6O_3$ (392.41): C, 61.21; H, 5.14; N, 21.42. Found: C, 61.10; H, 5.26; N, 21.21.

(b) From diazosulfonate (3a). A solution of monohydrate **3a** (37.0 mg, 0.10 mmol) and 1,3-diaminobenzene (108.0 mg, 1.00 mmol) was left in a closed flask for 14 days at 5-10 °C with occasional stirring. The separated crystalline precipitate was then collected by suction, washed with water and dried. The yield was 12.1 mg. After the following 19 days another portion of the same compound (11.0 mg) was separated from the filtrate. Therefore, the total yield was 23.1 mg (60%). After recrystallization from ethanol-water mixture, the substance is identical with the azo dye, which was prepared by the preceding method.

3-(2´,4´-Diaminoazobenzene-2-yl)-6,7-dimethylquinoxalin-2(1*H*)-one (**5b**). Prepared in the same way as the compound **5a** while using diazonium salt solution **2b** obtained from amine **1b**[12] (133.0 mg, 0.50 mmol) suspended in ice water (30 ml) by diazotation of a solution of $NaNO_2$ (35.0 mg, 0.51 mmol) in ice water (5 ml) and by coupling with a solution of 1,3-diaminobenzene (200.00 mg, 1.85 mmol) and CH_3COONa (1.2 g) in ice water (20 ml) in yield 148.2 mg (77%), m.p. 204-206 °C (ethanol). ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.29 (s, 3H, CH_3); 2.33 (s, 3H, CH_3); 5.74 (s, 1H, ArH); 5.89 (m, 3H, ArH, NH_2); 7.08 (m, 4H, ArH); 7.33 (t, 1H, ArH, *J*=7.92 Hz); 7.50 (m, 3H, ArH); 7.73 (d, 1H, ArH, *J* =9.72 Hz); 12.24 (s, 1H, NH). MS (+APCI): *m/z* 385 $[M+H]^+$. Anal. Calcd. for $C_{22}H_{20}N_6O$ (384,43): C, 68.73; H, 5.24; N, 21.86. Found: C, 68.53; H, 5.31; N, 21.62.

Acknowledgements

Financial support of this work from the Ministry of Education, Youth and Sports of the Czech Republic (grant nos. MSM6198959218 and MSM 6198959216) is gratefully acknowledged.

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