2,2-Bis(methoxy-*NNO*-azoxy)ethyl Derivatives of 4,8-Dihydro-bis-furazano[3,4-*b*:3′4′-*e*]pyrazine: The Synthesis and X-ray Investigation

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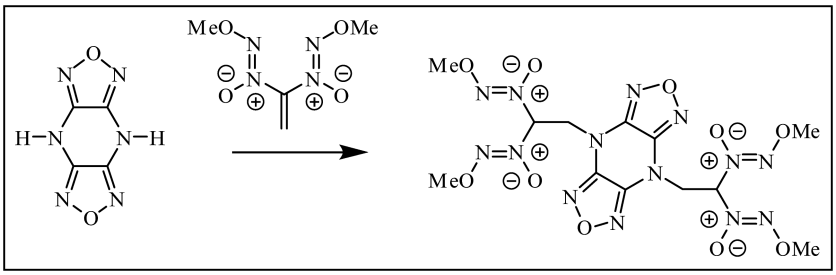

The first synthesis of 4,8-dihydro-bis-furazano[3,4-*b*:3′4′-*e*]pyrazine bearing 2,2-bis(methoxy-*NNO*-azoxy)ethyl groups has been developed. These compounds are obtained by aza-Michael reaction of 1,1-bis(methoxy-*NNO*-azoxy)ethene or its equivalents, such as 2,2-bis(methoxy-*NNO*-azoxy)ethanol derivatives, with 4,8-dihydro-bis-furazano[3,4-*b*:3′4′-*e*]pyrazine.

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INTRODUCTION

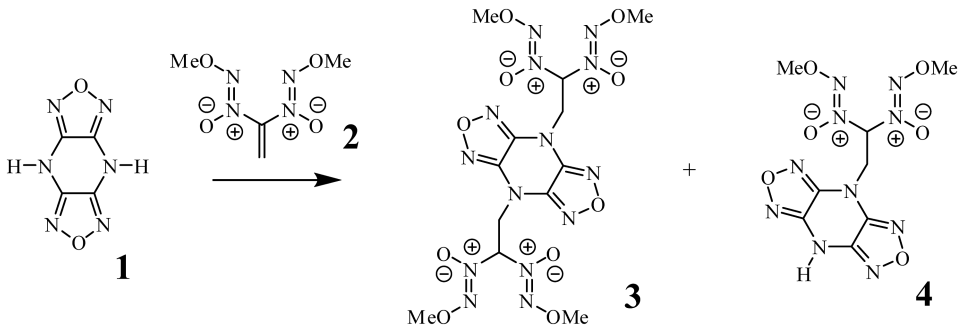
Since the discovery of alkoxydiazene N-oxides (ADO, O^2-substituted diazeniumdiolates), R—N(O)=N—OAlk, in 1891 [1], the research of suitable procedures for their preparations has become one of the main challenges in organic chemistry [2]. However, the chemistry of ADO has not been explored extensively. Compounds incorporating the alkoxydiazene N-oxide group have indeed found numerous potential applications ranging from chemical to biological (NO-releasing compounds for the treatment of hypertension and other cardiovascular disorders, etc. [3]) and technological field (high energetic materials [4]), and hence, new properties and new synthetic routes continue to appear.

The methoxy-*NNO*-azoxy group is isoster to the nitro group [5]. However, this group is weaker electron-withdrawing substituent (the Taft constant estimated by the NMR method is $\sigma^* = 0.35$ [6]) compared with the NO_2 group (the Taft constant $\sigma^* = 0.75$ [7]). On the other hand, the methoxy-*NNO*-azoxy group have the same elemental composition as the *N*-methyl-*N*-nitroamine group, R—N(NO_2)Me. The replacement of nitramino moiety by isomeric oxy-*NNO*-azoxy moiety tends to increase enthalpy of formation by 6–15 kcal/mol per one N_2O_2 moiety [8], and in so doing contributes markedly to the overall energetic performance. ADO, as a rule, has higher melting points than isomeric nitramines but usually reveal lower densities. All alkoxydiazene N-oxides are thermally stable and chemically inert [1b,9]. This indicates the manifold usability of the methoxy-*NNO*-azoxy moiety as attractive building block.

Furazan-based insensitive thermally stable explosive, such as 4,8-dihydro-bis-furazano[3,4-*b*:3′4′-*e*]pyrazine (**1**) has excellent performance, in part, because of the high positive heat of formation, high density, and moderate oxygen balance [10]. Recently [11], derivatives of compound **1** have been prepared in an effort to increase performance by improving the oxygen balance and heat of formation. There is no literature precedence for high-nitrogen energetic materials containing alkoxydiazene N-oxide, furazan, and pyrazine backbones.

The conjugate addition of nucleophiles to electron-deficient α,β-unsaturated systems is a fundamental concept in organic chemistry and is widely used as one of versatile and powerful methods in target-oriented synthesis [12]. The versatility of the methodology is mainly due to the large variety of nucleophilic species (carbanions, heteroatom Michael donors) and acceptors (α,β-unsaturated carbonyl compounds, nitriles, esters, phosphates, sulfones, nitroalkenes, and alkynoates among others) that provide

Scheme 1



the desired combination of functional groups in the product. When using the methodology for energetic materials constructions, as Michael donors and acceptors were nitro compounds or derivatives of nitrogen heterocycles [13]. In the process, unstable 2,2-dinitroethene can be generated *in situ* from 2,2-dinitroethanol derivatives.

In connection with our program dealing with the chemistry of unsaturated oxydiazene N-oxide derivatives [14], we have been exploring the chemical reactivity of 1,1-bis (methoxy-*NNO*-azoxy)ethene (**2**) [5] as a versatile synthon in organic synthesis for installation 2,2-bis(methoxy-*NNO*-azoxy)ethyl group. In an earlier report, we described the Michael reaction of ethene **2** with trinitromethane to produce 1,1-bis(methoxy-*NNO*-azoxy)-3,3,3-trinitropropane [15]. We now wish to report our results on the synthesis of 4,8-dihydro-bis-furazano[3,4-*b*:3′4′-*e*] pyrazine bearing 2,2-bis(methoxy-*NNO*-azoxy)ethyl groups.

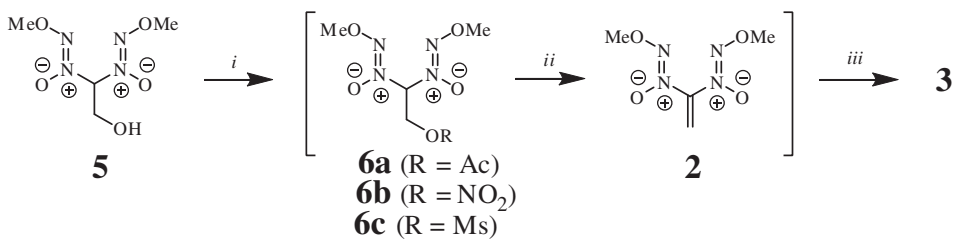
For our purpose, we planned to exploit the nucleophilicity of the nitrogen atoms in the 4,8-dihydropyrazine ring of compound **1**. The conjugate addition of the nitrogen nucleophile to activated α,β-unsaturated compounds (aza-Michael reaction) is the most obvious methods for preparing target compounds. The joint effect of two geminal MeON=(O)N groups on C=C bond is weaker than the effect of one NO_2 group in nitroalkenes. However, the more lower level of electrophilicity displayed by 1,1-bis(methoxy-*NNO*-azoxy)ethane **2** is sufficient to allow participation in reaction with nucleophile **1**, even at room temperature without a catalyst. As depicted in Scheme 1, a double Michael addition occurred when compound **1** was reacted with two equivalents of ethene **2** giving the bis-addition product **3** in 43% yield; a significant side product produced was the mono-adduct **4** (11%) together with some recovered starting material **1** and an oligomer from **2** [16]. The two compounds, **3** and **4**, could be readily separated; the solubility of compound **3** in water and organic solvents is rather low, whereas compound **4** is soluble in MeOH and basic aqueous media (pH > 7.4).

General application of this chemistry is, however, limited both by the tendency of the ethene **2** to hydrolysis, solvolysis, and polymerization and the forcing conditions required to generate and storage the highly reactive synthon **2**. One potential solution to this problem is the use of its equivalents. The most simple and efficient method of generating ethene **2** *in situ* involves the dehydration of easily evaluable 2,2-bis(methoxy-*NNO*-azoxy)ethanol **5** (through derivatives **6a-c**) [17], which are prepared from the bis(methoxy-*NNO*-azoxy)methane, and this was the approach adopted for the current work.

We investigated the base-promoted treatment of 2,2-bis (methoxy-*NNO*-azoxy)ethanol **5** with methanesulfonic anhydride (Ms_2O), as this route should afford ethene **2**, provided a nucleophilic addition of the piperazine **1** on the resulting double bond occurs.

The best results were obtained when compound **1** (1 equiv) was added to a solution of ethanol **5** (2 equiv) and Ms_2O in THF containing such base as di(isopropyl) ethylamine (Scheme 2). Target bis-adduct **3** was prepared from the reaction in near quantitative yield (96%). Other bases such as Na_2CO_3, KF, Na_2CO_3, or NEt_3 appeared

Scheme 2



Reagents and conditions: ***i***, Ac_2O, Ac_2O/HNO_3 or Ms_2O/base, 0-20°C; ***ii***, base, 20-50°C; ***iii***, compound **1**, 50-60°C.

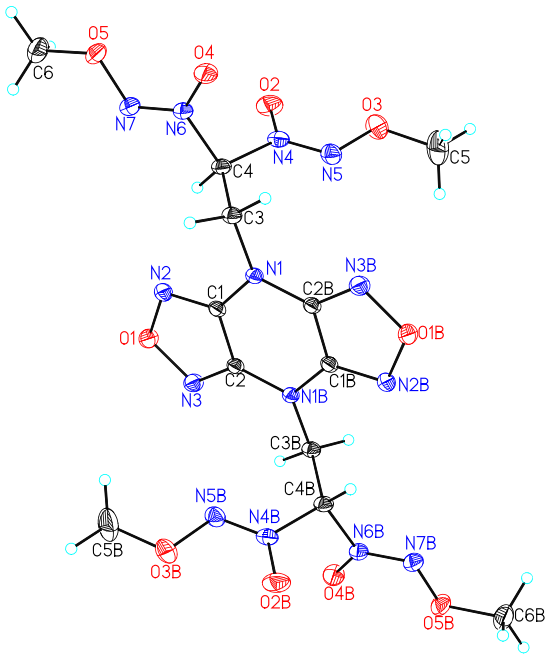


Figure 1. ORTEP view (drawn at 50% probability of thermal displacement ellipsoids) of compound **3** (Only the first independent molecule is shown.). Atoms labeled with letter "B" are generated by the symmetry center. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

to be ineffective. Alternatively, reaction of the compound **1** (1 equiv) with methanesulfonate **6c** (1 equiv) in the presence of di(isopropyl)ethylamine (DIPEA) afforded a mixture of mono-**4** (separated yield is ~30%) and bis-adduct **3** (~16%).

The structures of products were assigned on the basis of a detailed NMR analysis (and on comparison with the literature data [6,16,18]) and firmly established by single crystal X-ray crystallographic study of bis-adduct **3**. A perspective view of the molecular structure of bis-adduct **3** is depicted in Figure 1. An independent unit cell contains two halves of compound **3**. Molecules are located in special positions at the center of symmetry. Both independent molecules adopt nearly the same structure. Central tricyclic skeleton is planar as was earlier observed for other its structurally characterized derivatives [11a,19]. The methoxy-*NNO*-azoxy fragments adopt nearly planar structure and oriented nearly perpendicularly to each other in both $CH_2CH(N(O)=NOCH_3)_2$ moieties. Similar conformation was observed for $CR'R''(N(O)=NOR)_2$ compounds and their salts [18] and can be explained (at least in part) by sterical repulsion of oxygen atoms (O2 and O4 in Fig. 1).

In conclusion, a protocol for the β-addition to Michael acceptor activated methoxy-*NNO*-azoxy groups has been developed. The 2,2-bis(methoxy-*NNO*-azoxy)ethylation of 4,8-dihydro-bis-furazano[3,4-*b*:3′4′-*e*]pyrazine provided stable products in good yield. Access to the synthesis of high nitrogen compounds incorporating 2,2-bis(methoxy-*NNO*-azoxy)ethyl building block using the Michael addition approach is currently in progress.

EXPERIMENTAL

Melting points were determined on Gallenkamp melting point apparatus and they are not corrected. Infrared spectra were determined in KBr pellets on a Perkin-Elmer Model 577 spectrometer. Mass-spectra were recorded on a Varian MAT-311A instrument. ^{1}H-, ^{13}C-, and ^{14}N-NMR spectra were recorded on a Bruker AM-300 instrument at 300.13 MHz, 75.47 MHz, and 21.68 MHz, respectively. The chemical shift values (δ) are expressed relative to the chemical shift of the solvent-*d* or to external standard without correction nitromethane (^{14}N). Analytical TLC was conducted on precoated silica gel plates (Silufol UV_{254}). The plates were visualized under UV. The compounds **5** and **6c** were prepared as previously reported [5].

X-ray study of compound 13. At 100 K, single crystals of **3** ($C_{12}H_{18}N_{14}O_{10}$) are monoclinic, space group $P2_1/c$: a = 15.9941(7) Å, b = 11.5640(5) Å, c = 12.5736(6) Å, β = 112.3960(10)°, V = 2150.2(2) Å^3, Z = 4, d_{calc} = 1.601 g cm^{-3}, μ = 0.139 mm^{-1}. The 30506 reflections were collected on SMART APEX2 CCD difractometer (λ(Mo-Kα) = 0.71073 Å, graphite monochromator, ω-scans, 2θ < 64°) at 100 K. An analysis of measured intensities was carried out with the SAINT and SADABS programs included in the APEX2 program package [20]. The structure was solved by the direct methods and refined by the full-matrix least-squares procedure against F^2 in anisotropic approximation. All the hydrogen atoms were placed in geometrically calculated positions and included in the refinement within riding model with $U_{iso}(H) = nU_{eq}(C)$, where n = 1.5 for methyl carbon atoms and 1.2 for the other carbons. The 7503 independent reflections (R(int) = 0.0298) were used in the refinement procedure (for 329 parameters) that was converged to wR_2 = 0.1168 calculated on F^2_{hkl} (GOF = 1.022, R_1 = 0.0413 calculated on F_{hkl} using 6037 reflections with $I > 2\sigma(I)$). The refinement was carried out with the SHELXTL program [21]. Atomic coordinates, bond lengths, bond angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre (CCDC). These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Any request to the CCDC should quote the full literature citation and the reference number 794558.

4,8-Bis[2,2-di(methoxy-*NNO*-azoxy)ethyl]-bis-furazano[3,4-*b*:3′4′-*e*]pyrazine (3). Methanesulfonic anhydride (1.32 g, 7.5 mmol) was added to a slurry of compound **5** (0.97 g, 5 mmol) in THF (6 mL), and the mixture was heated at 50°C until all the material was completely dissolved. DIPEA (2.8 mL, 2.08 g, 16 mmol) was then added dropwise during 10 min. To the solution, compound **1** (0.33 g, 2 mmol) was added in one portion. The reaction mixture was stirred for 2 h at 50–60°C; a precipitate of product **3** started to form in 10 min from the beginning. The mixture was then cooled to 20°C, diluted with methanol (10 mL), and stirred at 0°C for a further 24 h. The colorless precipitate was filtered off, washed with methanol (5 mL), Et_2O (5 mL), and dried. The crude compound (1 g, 96%) was recrystallized from acetone and dried at 80°C *in vacuo* to give bis-addition product **3** (0.83 g, 82%).

Mp 270°C (dec). ^{1}H-NMR (DMSO-d_6) δ 4.09 (c, 12H, MeO); 5.82 (d, 4H, CH_2, J = 6.1); 6.72 (t, 2H, CHN, J = 6.1). ^{13}C-NMR (DMSO-d_6) δ 45.7 (NCH_2), 62.2 (CH_3O), 87.5 (CHN_2), 147.4. ^{14}N-NMR (DMSO-d_6) δ −73 (broad, N → O). Anal. Calcd for $C_{12}H_{18}N_{14}O_{10}$ (518.36): C, 27.81; H, 3.50; N, 37.83. Found: C, 28.02; H, 3.31; N, 38.95.

4-[2,2-Di(methoxy-*NNO*-azoxy)ethyl]-bis-furazano[3,4-*b*:3′4′-*e*]pyrazine (4). A solution of compound **6c** (0.36 g, 2.1 mmol), DIPEA (0.6 g, 4.6 mmol) and compound **1** (0.33 g, 2 mmol) in anhydrous $CHCl_3$ (5 mL) refluxed for 1.5 h. Stirring was continued while the mixture cooled, after which a solid (compound **3**, 0.15 g, 14.3%) was filtered off. The filtrate was treatment with 1*M* hydrochloric acid (3 mL) and the second crop **3** was collected, washed with MeOH, dried, and crystallized to yield the bis-addition product **3** (total yield 0.171 g, 16.5%): mp 270°C (dec.). The chloroform layer was concentrated *in vacuo*, and the residue was crystallized from acetone. The purification afforded 0.203 g (29.6%) of the mono-adduct **4** as a colorless solid.

Mp 206–207°C (dec.). IR (KBr) ν 3146, 2956, 1652, 1600, 1596, 1500, 1444, 1424, 1373, 1304, 1280, 1264, 1228, 1200, 1124, 1076, 1060, 996, 948 cm^{-1}; ^{1}H-NMR (DMSO-d_6) δ 4.08 (c, 6H, MeO), 4.77 (d, 2H, CH_2, J = 6.0 Hz), 6.72 (t, 1H, CHN, J = 6.0 Hz). ^{13}C-NMR (DMSO-d_6) δ 45.9 (NCH_2), 62.5 (CH_3O), 88.0 (CHN_2), 146.9, 147.8. ^{14}N-NMR (DMSO-d_6) δ −75 (broad, N → O). Anal. Calcd for $C_8H_{10}N_{10}O_6$ (342.23): C, 28.08; H, 2.95; N, 40.93. Found: C, 27.85; H, 3.11; N, 40.66.

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