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Received March 24, 2005

3,4-Bis(3-nitrofurazan-4-yl)furoxan has been synthesized by nitration of *C,N*-bis(trimethylsilyl) deriva-
tive of 3-amino-4-methylfurazan with nitrogen oxides.

The reactions of 3-amino-4-methylfyrazan (**1**) with nitrating/nitrosating reagents have recently been shown to provide a very useful way for the amino group transformations in the preparation of nitramino-, nitro-, nitroso-, azo-, and azoxy-methylfurazans [1-3]. No reaction involving the methyl group of compound **1** has been described [4].

In connection with our project dealing with the chemistry of 3-lithiomethyl-4-alkyl-furazans, we have been exploring the chemical reactivity of the intermediates as a versatile building block in organic synthesis [5-8]. It is the most promising technique for the functionalization of the usually resistant methyl group bridged to the furazan ring. As an extension of this study, an investigation was undertaken with the aim of realizing the activation of the methyl group in compound **1**.

In this paper, we describe a combination of the activation approach with the nitration/nitrosation process. Our goal is to transform both the amino and methyl group into the nitramino/nitro group and furoxan moiety, respectively.

We expected that the requisite transformations of both amino and methyl groups could be accomplished *via* a three-step protocol involving first the lithiation of compound **1** at nitrogen and carbon atoms of these substituents with *n*-BuLi, followed by *N,C*-bis-silylation of the resulting di-lithium compound **2** with Me$_3$SiCl, and finally the nitration/nitrosation of the silylated derivative **3a** (Scheme 1) [9]. The sequence was expected to result in 3,4-bis(furazan-4-yl)furoxan with terminal nitro or nitramino groups, to compounds **4** or **5**, respectively.

Scheme 1
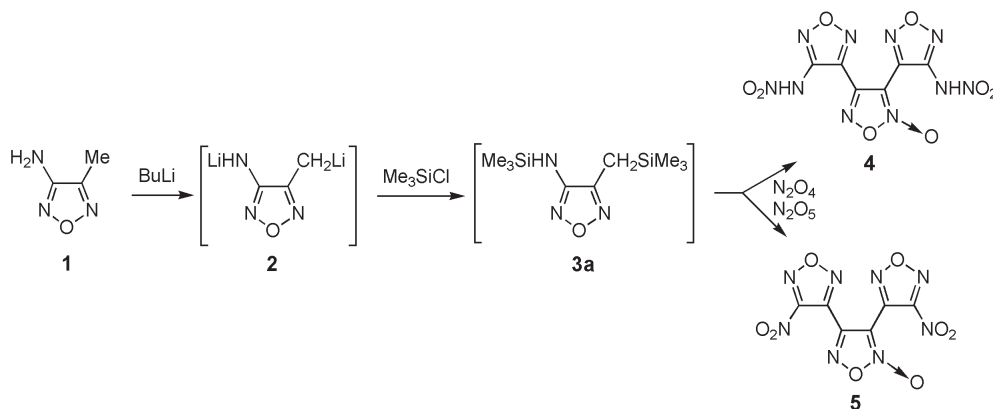
When a solution of compound **1** in glyme was allowed to react at -30 °C with *n*-BuLi (2.2 equiv) and then quenched with Me₃SiCl (2.2 equiv), the product was a mixture of disilylated regioisomers **3a** and **3b**, the ratio of which was determined from the ^{1}H NMR spectrum to be 1:3, respectively (Scheme 1 and 3). Without separation, the mixture was reacted with an excess of dinitrogen tetroxide (15 equiv, a solution in CH_2Cl_2) in the presence of sodium fluoride as a catalyst for 1 h at -30 °C, 2 h at 0 °C and 2 h at 25 °C to give, after careful chromatography, a mixture of 3-nitro-4-methylfurazan **6** (29%), 3-cyano-4-nitrofurazan **7** (7%), α,α-dinitropropionitrile **8** (6%), 4,4'-dimethylazofurazan **9** (35%), the desired product **5** (12%), and no nitramine **4**.

We have recently reported that N_2O_4 brings about desilylative nitration and nitrosation of (trimethylsilyl)methyl derivatives of furazans, providing access to 3,4-*bis*(3-methylfurazan-4-yl)furoxan *via* intermediated nitrolic acid [7]. The reaction to form the furoxan **5** involves loss of nitrous acid from the labile acid **10** and dimerization of nitrile oxide **11** (Scheme 2). The formation of nitrile **7** was attributed to the reduction of the nitrile oxide **11** by N_2O_3; the desoxygenation with N_2O_3 is a well documented side-reaction for nitrile oxides [10].

Several methods were tried for the nitration/nitrosation of the mixture of disilylated regioisomers **3a** and **3b**. The use of dinitrogen pentoxide or a mixture of nitric acid and trifluoroacetic anhydride produced the desired compound **5**, but in low yield (9-11%). An attempted nitration of the **3a/3b** mixture with a mixture of fuming nitric and sulfuric acids was unsuccessful. Formation of the same byproducts was observed in these cases [11].

Evidently formation of the intermediate **3b** during the silylation is an undesirable side route that is responsible for the consumption of compound **1** solely in by-processes.

In light of these results, we needed a method providing a reliable C-silylation procedure. A step by step silylation was developed which is delineated in Scheme 4.

The triply-silylated derivative **13** was prepared *via* a two-step synthesis. The reaction of amine **1** with an excess of hexamethyldisilazane in PhMe [14] afforded compound **12**. This intermediate was reacted with *n*-BuLi (2.2 equiv) at -20 °C and then quenched with Me₃SiCl (2.2 equiv) in glym under dry reaction conditions to give compound **13**.

When a solution of the *C,N,N-tris*(trimethylsilyl) compound **13** in CH_2Cl_2 was treated with an excess of dinitrogen pentoxide (15 equiv) in the presence of solid sodium
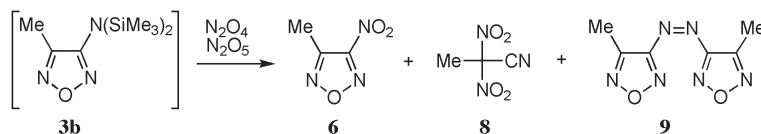
Scheme 2
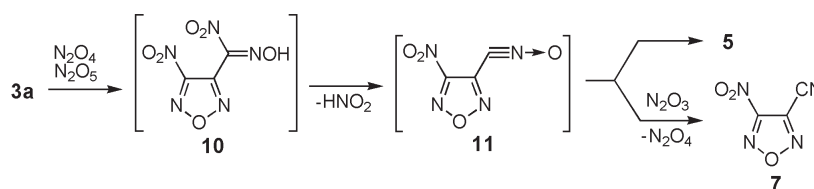
The formation of byproducts **6**, **8** and **9** could be attributed to nitration/nitrosation of compound **3b** in the reaction mixture (Scheme 3), the latter reacting with N_2O_4/N_2O_5 in the usual manner [3].

fluoride at -50°C and then allowed to warm to room temperature, the desired compound **5** was obtained in moderate yield (23%). Furoxan **5** was separated and purified by silica gel chromatography. Byproducts in the reaction were

Scheme 3

Scheme 4

compounds **6**, **8** and **9** alongside with some unidentified byproduct.

As an extension of this study an investigation was undertaken with the aim of realizing an alternative synthesis of compound **5**. The use of oxidation reactions is a time-tested strategy for preparation of nitrofurazans from aminofurazans, and the methodology has been reviewed [15,16]. Thus, as the next approach to compound **5** an oxidation of 3,4-bis(3-aminofurazan-4-yl)furoxan **14** [17] was investigated (Scheme 5). Treatment of the amine **14** with trifluoroperacetic acid in organic solvents according to Emmons's procedure [18] afforded the desired nitro compound **5** in moderate yield (50%).

Scheme 5
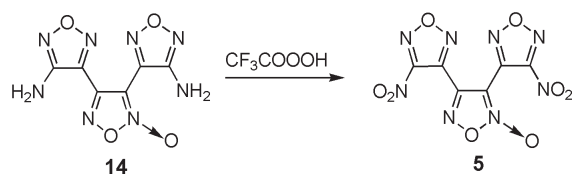
Compound **5** was characterized by its spectral properties which clearly showed the presence of nitro and the absence of nitramino groups that were present in compound **4**. Microanalysis and MS gave the molecular formula $C_6N_8O_8$. The electron impact mass spectrum of compound **5** showed a molecular ion at m/z 312 (M$^+$) and significant peaks due to loss of an oxygen atom (m/z 296 [M$^+$-O], 33%) and stepwise NO_2 and NO loss (m/z 250 [M$^+$- O - NO_2], 204 [M$^+$- O - $2NO_2$], 174 [M$^+$- O - $2NO_2$ - 2NO], and so on) from the molecular ion as might be expected from furoxan and furazan structures bearing nitro substituents. The ^{13}C NMR spectrum showed six separated resonances indicating an unsymmetrical molecule, and the

signal at *ca* 104 ppm supported the presence of a carbon atom of C=N$^+$-O$^-$ fragment from the furoxan ring. The presence of nitro groups was indicated by IR stretching bands at *ca* 1350 and *ca* 1560 cm^{-1}; no NH stretching was observed. The spectral properties agree with the literature data for furazans and furoxans [19-21].

The present assignments were unequivocally verified by the X-ray analysis. The molecular structure of the compound **5** is depicted in Figure 1, while selected geometrical characteristics are presented in Table 1 in comparison with those calculated in terms of B3LYP/6-311G* level of theory.

The molecular structure in the crystal is not planar. Both nitro groups are twisted relative to their adjacent furazan rings and both furazan rings are twisted relative to the central furoxan fragment (Table 1). Bond lengths in both furazan rings are close to the average X-ray values (1.385, 1.298 and 1.428 Å for N-O, N-C and C-C bonds respectively) [22,23]. Geometry of the central furoxan moiety resembles that of unsubstituted furoxan [24]: the N(3)-C(3) bond is slightly longer than the N(4)-C(4) bond while N(3)-O(2) bond length is significantly elongated relative to N(4)-O(2). There are shortened nonbonded interatomic contacts in the molecule: between the O(8) atom and O(4)-N(7)-O(5) nitro group and between the N(1) atom and the other nitro group. In order to figure out whether the geometry is caused by the crystal field or by intramolecular forces, we carried out B3LYP/6-311G* calculation using the Gaussian program [25]. It can be seen from Table 1 that there is a qualitative agreement between the calculated structure of the isolated molecule and the X-ray one. Topological analysis of the calculated electron density of the isolated molecule in terms of the theory of atoms in molecules has revealed that intramolecular close contacts correspond to chemical interactions; the (3,-1) critical points (CPs) were found between O(4)...O(8) and N(1)...O(6) atomic pairs (see Figure 1). It allows us to suggest that such a molecular structure is stabilized by those interactions.

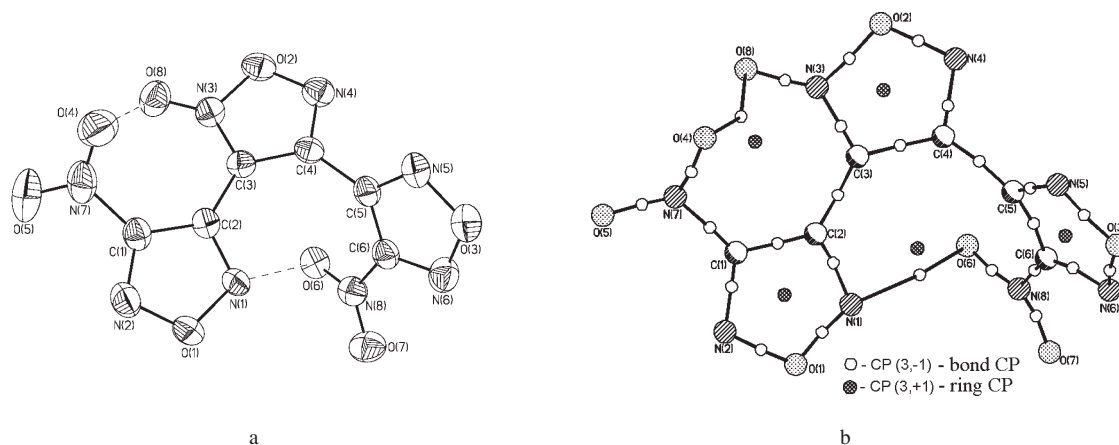a   b

Figure 1. General view of molecule **5**; a) ORTEP view of molecule; b) Quantum chemically obtained structure of **5** with bond (3,-1) and ring (3,+1) critical points.

Table 1

Selected geometrical parameters of compound **5** obtained by the X-ray and calculated by B3LYP/6-311G* method.

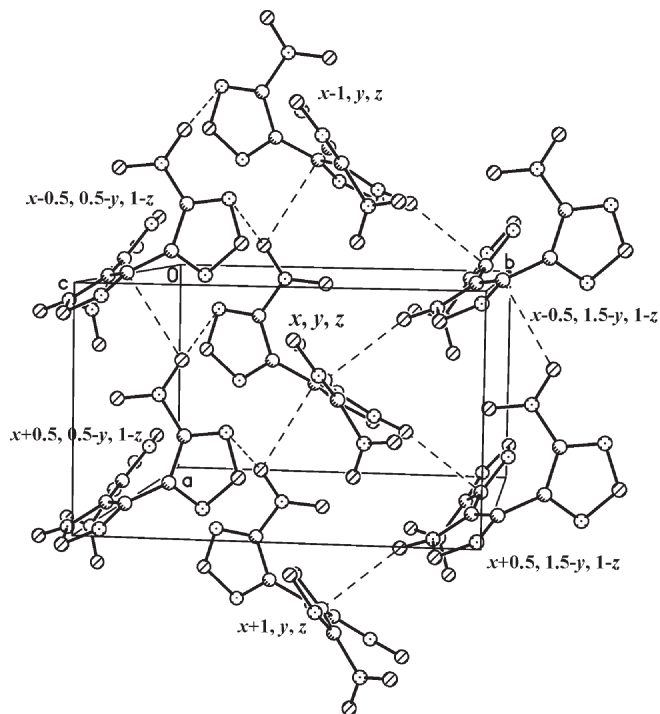| Geometrical parameter	X-ray	B3LYP/6-311G*
angle between O(4)-N(7)-O(5) and C(1)-C(2)-N(1)-O(1)-N(2)	46.9(4)	36.0
angle between O(6)-N(8)-O(7) and C(5)-C(6)-N(6)-O(3)-N(5)	19.1(3)	9.8
angle between furoxan ring and		
C(1)-C(2)-N(1)-O(1)-N(2)	23.5(2)	30.9
C(5)-C(6)-N(6)-O(3)-N(7)	59.1(2)	68.8
O(4)…O(8)	2.757(4)	2.811
O(6)…N(1)	2.971(4)	3.120
O(1)-N(1)	1.377(4)	1.367
O(1)-N(2)	1.372(4)	1.355
O(2)-N(3)	1.440(4)	1.465
O(2)-N(4)	1.372(4)	1.352
N(3)-C(3)	1.339(4)	1.338
N(4)-C(4)	1.306(4)	1.303
O(3)-N(5)	1.381(4)	1.371
O(3)-N(6)	1.370(5)	1.355

Figure 2. A fragment of crystal packing; the central molecule (x, y, z) with its nearest neighbours in the layer is shown.

In the crystal structure of compound **5** there are several slightly shortened intermolecular contacts: O(7)…N(6) (-0.5+x; 0.5-y; 1-z) (2.976(5)Å), O(8)…C(2) (0.5+x; 1.5-y; 1-z) (2.880(5)Å), O(7)…C(4) (-1+x, y, z) (3.070(5)Å) (Figure 2) (for nonbonded radii, see ref [26]). The molecules connected through the system of such contacts form layers parallel to *ab* crystallographic plane. So the interactions inside the layer should be somewhat stronger than

those between the layers. This is supported by the atom-atom potential calculations [27] (using Mirsky parameterization [28]). The strongest pair interactions of the molecule with its nearest neighbors in the layer are –4.8 kcal/mol for (-0.5+x; 0.5-y; 1-z) and (0.5+x; 0.5-y; 1-z), –4.6 kcal/mol for (-0.5+x; 1.5-y; 1-z) and (0.5+x; 1.5-y; 1-z) and –3.8 kcal/mol for (-1+x, y, z) and (1+x, y, z), while the strongest pair interaction between the layers is -2.9 kcal/mol for (0.5-x; 1-y; 0.5+z) and (0.5-x; 1-y; -0.5+z). The above-mentioned close contacts as well as absence of hydrogen atoms in the molecules of the compound **5** lead to a sufficiently dense structure (1.920 g/cm³).

Nitrofurazans are well known for their high energy content, and methods for the synthesis of these compounds have been reviewed [15,16]. In a recent communication [29], Lobbecke and co-workers reported on the identification of compound **5** as a possible energetic material. However, synthesis of the compound **5** has not previously been reported in the literature.

EXPERIMENTAL

Caution! Nitrofurazans are highly explosive and may be sensitive to shock or heating and must be handled with appropriate precautions.

Melting points were determined on Gallenkamp apparatus and are reported uncorrected. 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, 75.47, 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 F_{254}). The plates were visualized under UV after development followed by spraying with DPA reagent (5% diphenylamine in hexane). Gas-liquid chromatography was performed on a Biochrom-5 instrument with a SE-54 capillary column (0.2 mm × 20 m). 3-Amino-4-methylfurazan **1** [4] and 3,4-dis(3-aminofurazan-45-yi)furoxan [17] were prepared according to literature procedures.

X-Ray Crystallography.

X-Ray quality crystals of compound **5** were grown by slow evaporation of a chloroform solution at room temperature. The single crystals of **5** are orthorhombic, space group P2$_1$2$_1$2$_1$: a = 6.662(3) Å, b = 10.740(4)Å, c = 15.093(5)Å, V = 1079.9(7) Å³, Z=4, M=312.1, d$_{calc}$= 1.920 g·cm⁻³, μ=0.180 mm⁻¹. Carefully chosen sample (0.4 × 0.35 × 0.25 mm) has been used for the X-ray study. 5618 reflections were collected at SMART 1000 CCD difractometer (λ(Mo-Kα)=0.71073 Å, graphite monochromator, ω-scans, 2θ<60°) at 120K. The structure was solved by the direct methods and refined by the full-matrix least-squares procedure in anisotropic approximation. 1654 independent reflections [R$_{int}$ = 0.0482] were used in the refinement procedure (for 199 parameters) that was converged to wR$_2$ = 0.1217 calculated on F²$_{hkl}$ (GOF = 0.928, R$_1$ = 0.0505 calculated on F$_{hkl}$ using 1255 reflections with I>2σ(I)). For the analysis of data collected and crystal

structures refinement we used SAINT Plus [30], SADABS [31] and SHELXTL-97 [32] program packages. Because of absence of heavy atoms in **5** the absolute structure was not determined. Atomic coordinates and thermal parameters, bond lengths and angles have been deposited at the Cambridge Crystallographic Data Centre as supplementary publication numbers 270417.

3,4-Bis(3-nitrofurazan-4-yl)furoxan (**5**).

Method a.

The reactions were carried out at dried conditions under an argon atmosphere. To a slurry of 3-amino-4-methylfurazan **1** (0.99 g, 10 mmol) in toluene (5 ml) excess of hexamethyldisilazane (2.42 g, 15 mmol) was added. The mixture was heated at reflux for 3 h and then volatiles were removed. The residue (crude compound **12**) was dissolved in glym (60 ml) and cooled to ca -20 °C. To the well-stirred solution was slowly added a solution of n-BuLi (22 mmol) in hexane (as a 1.6 M solution) and then a solution of Me$_3$SiCl (2.4 g, 22 mmol) in glym (15 ml). Addition was complete after 30 min. The mixture was stirred at -20 °C for 1 h and then warmed to -10 °C. A solid (LiCl) was filtered off and washed with ether/pentane (5 ml, 1/1). The filtrate was evaporated in vacuo at 20 °C to give crude compound **13**, as slightly yellow oil; **ms**: (m/z) 315 (M$^+$), 285 (M$^+$ - NO).

A solution of obtained compound **13** in dry CH$_2$Cl$_2$ (30 ml) was slowly added to a well-stirred mixture of N$_2$O$_5$ (16 g, 150 mmol) and a catalytic amount of solid sodium fluoride in CH$_2$Cl$_2$ (100 ml) maintained at -50 °C by a CHCl$_3$-dry ice bath. The cooling bath was replaced by an ice-water bath and the solution stirred for 1 h. The mixture was then allowed to warm to room temperature and the solvent was distilled off at atmospheric pressure. The residue was adsorbed onto silica gel and chromatography (CH$_2$Cl$_2$/pentane 3/1) gave product **5** (0.36 g, 23%) as a colorless crystals, mp 108-109°C (lit. mp 108°C [29]); ir: 1640, 1584, 1564, 1516, 1448, 1356, 1316, 1284, 1176, 1000, 964, 912 cm^{-1}; ^{13}C nmr: 104.3, 138.1, 140.4, 143.7, 160.7, 160.7. ^{14}N nmr: -20.2 (N→O, $\Delta\nu_{1/2} \approx 250$ Hz), -37.9 (NO$_2$, $\Delta\nu_{1/2} = 10$ Hz).

Anal. Calcd. for C$_6$N$_8$O$_8$ (312.11): C, 23.09; N, 35.90. Found: C, 23.13; N, 39.85.

Method b.

Trifluoroacetic anhydride (25 ml) was carefully added dropwise to a solution of 96% H$_2$O$_2$ (11.3 ml) in CH$_2$Cl$_2$ (90 ml) at 0-5 °C. To the resulting solution amine **14** (3.4 g, 13.5 mmol) was added portion-wise keeping the temperature below 20 °C. The mixture was left at 20 °C for 1 h and then heated to reflux for 2-3 h (until the green color had discharged). The reaction mixture was poured into water (200 ml). The organic layer was separated. Washing the layer with 3% aqueous NaHCO$_3$ (to the neutral pH value) and with water (50 ml), drying (MgSO$_4$), and removal of the solvent afforded the resulting product **5** (2.0 g, 50%), mp 109-110 °C (from 1,2-dichloroethane); on the basis of spectroscopy and TLC, the substance corresponded in all respects to the compound prepared by method a.

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