Synthesis and X-Ray Crystal Structure of Bis-3,3′-(nitro-*NNO*-azoxy)-difurazanyl Ether

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Abstract: Two approaches to the synthesis of bis-3,3′-(nitro-*NNO*-azoxy)difurazanyl ether **2** have been tested, that is, varying the sequence of the introduction of the nitro-*NNO*-azoxy group and ether bridge. Difurazanyl ether formation through base-promoted transformation of nitrofurazans has been studied in which the nitro groups were activated by *ortho* electron-withdrawing substituents such as nitro-*NNO*-azoxy and *tert*-butyl-*NNO*-azoxy groups. The nitro-*NNO*-azoxy group was formed by destructive *N*-nitration of the *tert*-butyl-*NNO*-azoxy group. The X-ray crystal structure of **2** is reported.

Keywords: azo compounds · furazans · heterocycles · nitro groups · structure elucidation

Introduction

The definition of the explosophore for the construction of highly energetic molecules has been the subject of the considerable scientific interest in recent years.[1] A number of nitrofurazans have been synthesized as potential materials for military and space applications and evaluated for their performance.[2] In our studies to characterize the optimum combinations for more powerful and stable furazan molecules,[3] we found that 3,3-dinitrodifurazanyl ether (**1**)[4] had hich-energy potency. Since the difurazanyl ether moiety in **1** is stable to thermal degradation, this compound is an excellent stable and more dense analogue for hexanitrodiphenyl ether.[5] In our studies to further define the requirements of these attractive furazan explosives, we examined analogues of **1** that possess different terminal groups.

A recent publication by Churakov et al.[6] described the first synthesis of new explosophoric groups, such as nitro-*NNO*-azoxy, through a destructive *N*-nitration of an X-*NNO*-azoxy compound (X = Ac, *t*Bu) with nitronium salts. In the present study, we report on the synthesis and characterization of a nitro-*NNO*-azoxy-containing difurazanyl ether, namely bis-3,3′-(nitro-*NNO*-azoxy)difurazanyl ether (**2**).

Results and Discussion

Synthesis: Up till now, the only method for the preparation of difurazanyl ethers has been through ether bond formation by base-promoted transformation of the corresponding 3-nitro-4-R-furazans.[4, 7] As a logical extension of this methodology, we investigated the behavior of 3-nitro-4-(nitro-*NNO*-azoxy)-furazan (**3**). Compound **3** was synthesized by a literature procedure.[6] The presence of the nitro-*NNO*-azoxy group met the requirement that the substituent R be electron-withdrawing.[4] Treatment of **3** with base, such as Na_2CO_3 in acetonitrile at 50 °C, followed by flash chromatography led to the isolation the desired product **2**, but only in low yield (8 %). Some by-products, the ether **1** and the hydroxyfurazans **4** and **5**, were isolated (Scheme 1) after column chromatography in yields of 6, 14, and 6 %, respectively. Despite a number of attempts, the unsymmetrical ether **6**[8] was not isolated. When the reaction was carried out at a higher temperature (approx. 80 °C) or with K_2CO_3 instead of Na_2CO_3 the starting material **3** decomposed, presumably to uncharacterized small fragments such as N_2, N_xO_y, CO_2, and so forth. Unfortunately, all attempts to form compound **2** in high yield from substrate **3** under a variety of conditions failed.

In light of these results, a stepwise approach to **2** was developed that is outlined in Scheme 2. The approach involved initial ether-bond formation followed by introduction of the nitro group to the *NNO*-azoxy moiety. The starting compound **7** was prepared by previously reported procedures.[9] The *tert*-butyl-*NNO*-azoxy group of compound **7** is more thermally stable and less electron-withdrawing than the nitro-*NNO*-azoxy group in compound **3**. Therefore, in contrast to **3**, compound **7** was relatively unreactive towards

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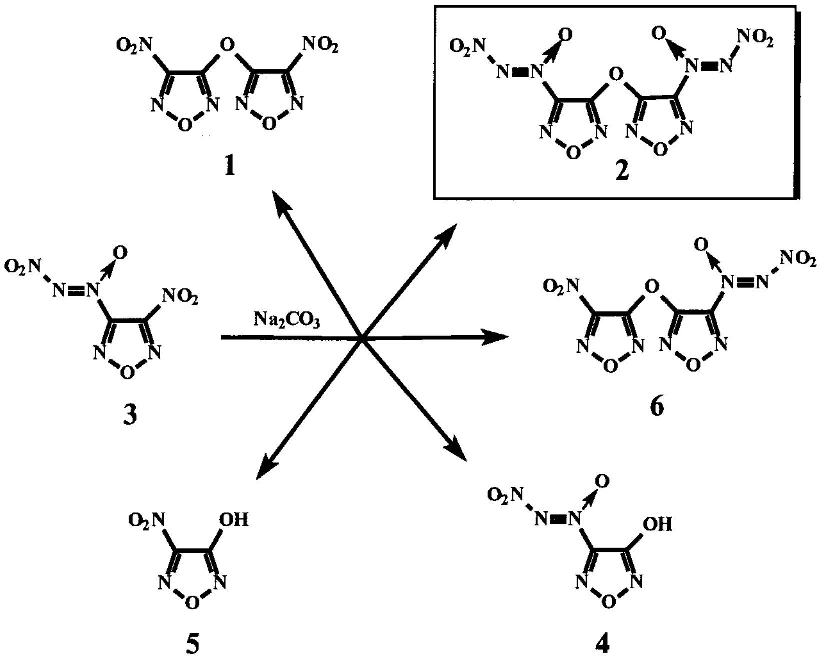
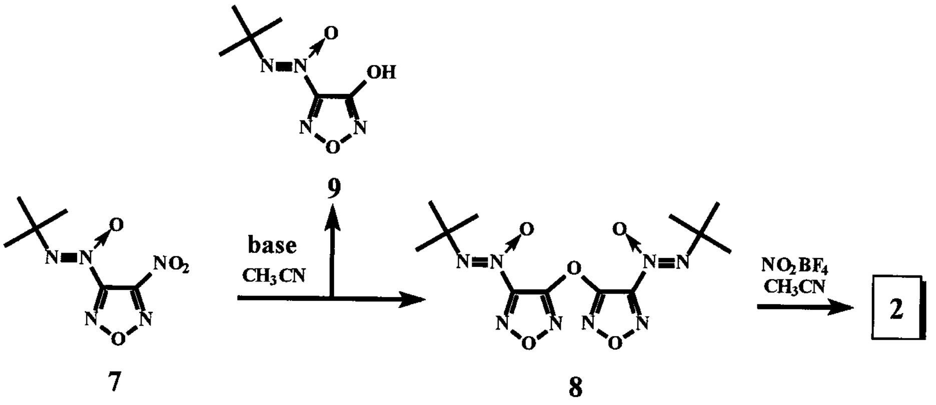


Scheme 1. Reaction scheme for the treatment of **3** with base.



Scheme 2. A stepwise approach to the formation of **2**.

Na$_2$CO$_3$ at 50 °C and was recovered quantitatively after 0.5 h. However, treatment of compound **7** with K$_2$CO$_3$ in CH$_3$CN under dry conditions at 80 °C afforded the ether **8** in 73 % yield and the hydroxyfurazan **9** in 20 % yield. No products arising from displacement of the *tert*-butyl-*NNO*-azoxy group were detected. Thus, compounds **3** and **7** have different chemical reactivities, indicating that the nucleofugacity of the *tert*-butyl-*NNO*-azoxy group is lower than that of a nitro group.[10]

Interestingly, the inclusion of 2–5 % water in the acetonitrile reaction medium gave a mixture of products that not only contained the ether **8** (23 %) and hydroxyfurazan **9** (56 %), but also compound **5** (12 %) and 3,4-dihydroxyfurazan **10** (9 %). Compound **1** was not detected. Thus, the displacement of both nitro and *tert*-butyl-*NNO*-azoxy groups occurs under these conditions. Compound **8** was readily converted to compound **2** by a modified literature procedure.[6] Thus, treatment of a solution of compound **8** in acetonitrile with excess NO$_2$BF$_4$ followed by chromatography on silica gel gave compound **2** as the only stable product in 29 % yield.

The structure of **2** was based on ^{13}C, ^{14}N, ^{15}N, and ^{17}O chemical shifts and heteronuclear-coupling information. The NMR data was consistent with that reported for furazans[3h, 11] and nitroazoxy compounds.[6] Thus, the ^{13}C chemical shift of the ether carbon atom (C–O) of **2** ($\delta = 154.3$) is approximately 2 ppm upfield compared with that of **1** ($\delta = 156.3$). Also, the nitrogen-substituted carbon atom (C–NONNO$_2$) of **2** ($\delta = 147.2$) is shifted upfield by approximately 6 ppm relative to the nitro carbon atom (C–NO$_2$) of **1** ($\delta = 153.9$). The azoxy nitrogen (N→O) signal in the ^{14}N NMR spectrum of **2** appears at $\delta = -71.5$. Other noteworthy chemical shifts are given in the Experimental Section.

X-ray crystal structure analysis: Although crystallographic data of substituted difurazanyl ethers[4] and nitro-*NNO*-azoxy compounds[6a] are available, structural analyses of nitro-*NNO*-azoxyfurazans have not been reported. We obtained crystals of **2** suitable for a crystallographic structure determination by recrystallization from CHCl$_3$. Figure 1 shows the molecular structure in the crystal. The illustration clearly indicates a nonplanar conformation. Actually, the nitro-*NNO*-azoxy group involves two planar moieties, namely O(4)O(5)N(31)-N(21) and O(3)-

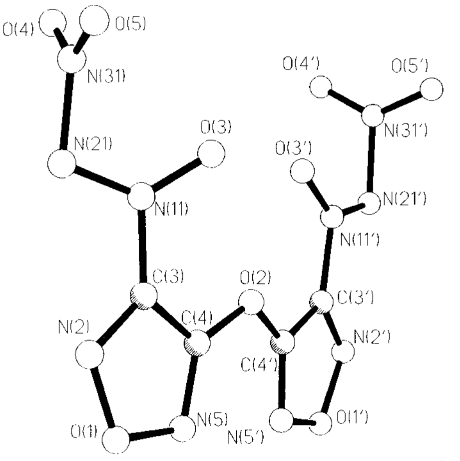


Figure 1. View of the molecular structure of **2**. Selected bond lengths [Å] and angles [°]: O(2)–C(4) 1.347, C(3)–N(11) 1.463, N(11)–N(21) 1.267, N(21)–N(31) 1.503, N(11)–O(3) 1.235; O(31)-N(11)-N(21) 130, N(11)-N(21)-N(31) 105, O(4)-N(31)-O(5) 130.

N(11)-N(21)-N(31), that are oriented nearly perpendicular to each other (84.5°). The metrical parameters for the difurazanyl ether moiety in **2** are quite similar to those reported for **1**.[4] Thus, the torsion angles C-O-CC in **2** are 11.8 and 12.7° and are closely related to the angles in one of the independent molecules of **1**.

The N(21)–N(31) bond length (1.503 Å) within the nitro-*NNO*-azoxy group is significantly longer than the standard N–N single bond length (1.47 Å)[12] and the metrical parameters for the nitro group (N–O 1.195 Å, O-N-O 130°) are quite similar to those for a free NO_2 molecule (1.193 Å and 134°).[13] These parameters provide the reason for believing that the initial step leading to molecular decomposition may be the breaking of the N(21)–N(31) bond.

In the structure of **2**, all intramolecular nonbonded contacts are van der Waals interactions. Comparison of such parameters as the coefficient of molecular packing (K_p) and packing density ($D_{mol} = M_r/V_{mol}$) for molecules **1** and **2** indicates that replacement of the nitro group by a nitro-*NNO*-azoxy group gives rise to a slight increase of D_{mol} (2.90 $g\,cm^{-3}$ for **1** and 2.93 $g\,cm^{-3}$ for **2**). A decrease of magnitude of K_p (0.653 for **1** and 0.628 for **2**) thereby causes the decrease of crystal density (1.898 $g\,cm^{-3}$ for **1** and 1.845 $g\,cm^{-3}$ for **2**). These results are in the excellent agreement with the earlier data on the tendency to increase the looseness of the molecular packing with the increase of molecule density.[14]

Thus, our initial goal of preparing **2** was successfully accomplished. Despite compound **2** having high density and excellent detonation properties, the compound was more sensitive and less thermally stable than compound **1**. Nonetheless nitro-*NNO*-azoxy compounds may be important candidates for further study.

In conclusion, the synthetic procedure by which the nitro-*NNO*-azoxy group is formed in the last step has shown promise in the preparation of other high-energy materials. Current efforts in this laboratory are focused on further applications of this methodology for the formation of related compounds with high nitrogen content.

Experimental Section

CAUTION! Nitroazoxy-containing compounds are highly explosive and may be sensitive to shock or heating and must be handled with appropriate precautions.

Melting points were determined on a Kofler hot-stage microscope and are uncorrected. IR spectra were recorded using a Perkin–Elmer 577 spectrometer as thin films on KBr disks. Mass spectra were obtained on a Varian MAT-311A instrument. ^{1}H, ^{13}C, $^{14}N/^{15}N$, and ^{17}O NMR spectra were recorded on a Bruker AM300 instrument at 300.13, 75.47, 21.68/30.42, and 40.69 MHz respectively. The chemical shift values (δ) are expressed relative to the chemical shift of the deuterated solvent, to an external standard without correction nitromethane ($^{14}N/^{15}N$), or to water (^{17}O). Analytical thin-layer chromatography (TLC) was conducted on precoated silica gel plates (Silufol F_{254}). The plates were visualized under UV light after development followed by spraying with a DPA reagent (5% diphenylamine in hexane).

Reaction of 3-nitro-4-(nitro-*NNO*-azoxy)furazan (3) with base: Na_2CO_3 (0.1 g, 1 mmol) was added to a solution of **3** (0.41 g, 2 mmol) in CH_3CN (5 mL) under a dry atmosphere at 50 °C. The resulting suspension was stirred for 0.5–1 h at 50 °C. The progress of the reaction was monitored by TLC and by the consumption of starting material. The reaction was cooled. After addition of CH_2Cl_2 (50 mL), the resulting mixture was washed (H_2O), dried ($MgSO_4$), filtered, and evaporated. The residue was a mixture of **1** and **2**. The products were separated by silica gel flash chromatography with CH_2Cl_2/pentane (2:1) as eluent.

Fraction 1: 3,3-Dinitrodifurazanyl ether (**1**) was obtained as colorless crystals, m.p. 63–64 °C, (lit. m.p. 63–64 °C). On the basis of IR spectroscopy and TLC, the substance corresponded in all respects with the compound described earlier.[4]

Fraction 2: Bis-3,3′-(nitro-*NNO*-azoxy)difurazanyl ether (**2**) was afforded as colorless crystals. M.p. 66 °C; ^{13}C NMR ($CDCl_3$): $\delta = 147.2$ ($C-N_3O_3$), 154.3 (C–O); ^{14}N NMR ($CDCl_3$): $\delta = -46.8$ ($\Delta\nu_{1/2} = 50$ Hz, NO_2), -71.5 ($\Delta\nu_{1/2} = 60$ Hz, N→O); $C_4N_{10}O_9$ (332.11): calcd C 14.46, N 42.18; found C 14.42, N 42.05.

The aqueous phase was strongly acidified with H_2SO_4 and extracted with Et_2O (4 × 20 mL). The combined extracts were dried ($MgSO_4$) and filtered. The solvent was removed by rotary evaporation, and the residue was separated by silica gel column chromatography with $CH_2Cl_2 \rightarrow CH_3CN$ as eluent.

Fraction 1: 3-Hydroxy-4-nitrofurazan (**5**) was obtained as a yellow oil: on the basis of IR spectroscopy, MS, and TLC, the substance corresponded in all respects with the compound described earlier.[4]

Fraction 2: 3-(Nitro-*NNO*-azoxy)-4-hydroxyfurazan (**4**) was obtained as unstable colorless crystals: m.p. 28–30 °C; $C_2H_1N_5O_5$ (175.06): calcd C 13.72, H 0.58, N 40.01; found C 13.80, H 1.15, N 39.92. Compound **4** is strongly acidic and gives a stable ammonium salt. M.p. 123 °C; IR: $\tilde{\nu} =$ 3100–2800, 1620, 1545, 1510, 1470, 1350, 1310, 1235, 1180, 1005 cm^{-1}; ^{13}C NMR ([D_4]methanol): $\delta = 150.3$ ($C-N_3O_3$), 160.9 (C–O); ^{14}N NMR ([D_6]acetone): $\delta = -36.7$ ($\Delta\nu_{1/2} = 25$ Hz, NO_2), -59.5 ($\Delta\nu_{1/2} = 40$ Hz, N→O), -366.8 ($\Delta\nu_{1/2} = 25$ Hz, NH_4); $C_2H_4N_6O_5$ (192.09): calcd C 12.51, H 2.01, N 43.75; found C 12.40, H 2.15, N 43.84.

Bis-3,3′-(*tert*-butyl-*NNO*-azoxy)difurazanyl ether (8): A suspension of **7** (2.15 g, 10 mmol) and K_2CO_3 (1.38 g, 10 mmol) in dry CH_3CN (15 mL) was stirred and refluxed. The progress of the reaction was monitored by TLC and by the consumption of starting material. After cooling and addition of water (100 mL), the resulting mixture was extracted with CH_2Cl_2 (4 × 50 mL). The combined extracts were washed (H_2O), dried ($MgSO_4$), filtered, and evaporated. Recrystallization of the residue from pentane afforded colorless crystals of **8** (1.3 g, 73%). M.p. 66-67.5 °C; MS: *m/z*: 354 [M^+], 339 [$M^+ - CH_3$], 337, 299, 281, 265, 235, 224, 210, 180; IR: $\tilde{\nu} = 3015$, 2985, 2945, 2880, 1590, 1510, 1500, 1460, 1370, 1315, 1245, 1220, 1190, 1175, 1035, 910, 870 cm^{-1}; ^{1}H NMR ([D_6]acetone): $\delta = 1.39$ (s); ^{13}C NMR ([D_6]acetone): $\delta = 25.3$ (Me), 61.7 (CMe_3), 152.7 (C–NNO), 158.4 (C–O); ^{14}N NMR ([D_6]acetone): $\delta = -73.2$ ($\Delta\nu_{1/2} = 30$ Hz, N→O); ^{17}O NMR ($CDCl_3$/[D_6]acetone): $\delta = 480$ (N→O), 410 (N-O-N), 50 (C-O-C); $C_{12}H_{18}N_8O_5$ (354.33): calcd C 40.68, H 5.12, N 31.62; found C 40.73 H 5.30, N 31.45.

The aqueous phase was acidified with hydrochloric acid and extracted with Et_2O (4 × 50 mL). The combined extracts were dried ($MgSO_4$) and filtered. The solvent was removed by rotary evaporation, and the residue was purified by chromatography on silica gel (CH_2Cl_2/pentane) to give 3-(*tert*-butyl-*NNO*-azoxy)-4-hydroxyfurazan (**9**) (0.37 g, 20%) as a colorless liquid. MS: *m/z*: 171 [$M^+ - CH_3$], 160, 141, 128, 113, 102, 84; IR: $\tilde{\nu} =$ 3160–2860, 1575, 1250, 920 cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 1.58$ (Me), 8.8 (OH); ^{13}C NMR ($CDCl_3$): $\delta = 25.3$ (Me), 61.2 (CMe_3), 148.8 (C–NNO), 158.9 (C–O); ^{14}N NMR ($CDCl_3$): $\delta = -67.2$ ($\Delta\nu_{1/2} = 30$ Hz, N→O); $C_6H_{10}N_4O_3$ (186.17): calcd C 38.71, H 5.41, N 30.09; found C 38.82, H 5.53, N 29.94.

Bis-3,3′-(nitro-*NNO*-azoxy)difurazanyl ether (2): NO_2BF_4 (2.5 g, 19 mmol) was added to a solution of **8** (2.0 g, 5.65 mmol) in dry CH_3CN (9 mL) under dry conditions at 10 °C. The mixture was stirred for 0.5 h at 10 °C and for 1 h at 25 °C. Then next portion of NO_2BF_4 (1.0 g, 7.5 mmol) was added. After stirring 1 h at 30–32 °C the resulting mixture was diluted with CH_2Cl_2 (35 mL). The resulting oil was separated and extracted with CH_2Cl_2 (2 × 10 mL). The combined extracts were washed (H_2O), dried ($MgSO_4$), filtered, and evaporated. The residue was purified by chromatography on silica gel (benzene) to give the product **2** (0.54 g, 29%) as colorless crystals: m.p. 66 °C (from $CHCl_3$).

X-ray analysis of 2: $C_4N_{10}O_9$, $M = 332.11$, monoclinic, space group $P2_1/c$, $a = 15.456(1)$ Å, $b = 7.956(1)$ Å, $c = 9.852(1)$ Å, $\beta = 103.18(4)°$, $V = 1195.8(2)$ Å^3, $\rho_{calcd} = 1.845\ g\,cm^{-3}$, $Z = 4$, $\lambda(Cu_{K\alpha}) = 1.54178$ Å. The intensities of 1846 reflections (1758 observed) were measured on a Syntex $P2_1$

diffractometer (graphite-monochromated $Cu_{K\alpha}$ radiation), by means of the $\theta-2\theta$ scan technique ($2\theta < 132°$). The structure was solved by direct methods, SHELX-86,[15] and refined by full matrix least squares with SHELX-93.[16] The final R_F was 0.073. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-100727. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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