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Ionic liquid-mediated synthesis of (1*H*-1,2,3-triazol-1-yl)furoxans by [3 + 2] cycloaddition of azidofuroxans to acetylenes

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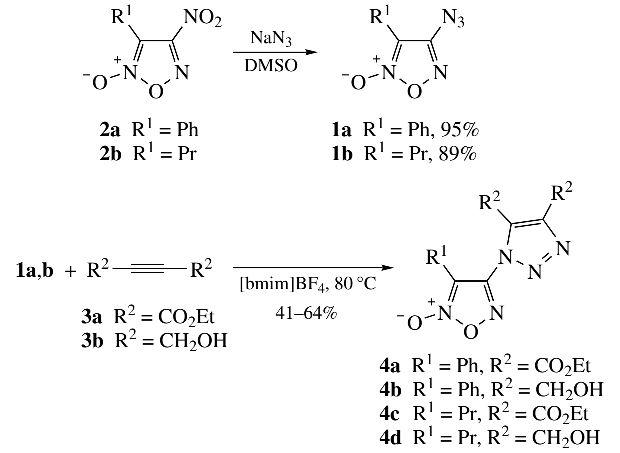
The [3 + 2] cycloaddition of azidofuroxans to internal and terminal acetylenes was found to occur only in ionic liquids on heating and afford (1*H*-1,2,3-triazol-1-yl)furoxans in moderate to good yields. The reaction with terminal acetylenes proceeds with high regioselectivity.

Construction of novel hybrid structures incorporating two different pharmacophoric groups in one molecule is an efficient approach to the design of new drug candidates with improved pharmacokinetic profiles.[1] Previously, we reported a synthesis of triazolylfurazans with various substituents in both rings by the [3 + 2] cycloaddition of azidofurazans to acetylenes,[2,3] morpholinonitroethylene,[3,4] 1,3-dicarbonyl compounds,[5–7] and methylene-active nitriles.[8] Studies on biological activity of triazolylfurazans have revealed that they exert a selective potentiating effect on the NO-dependent activation of sGC (a soluble form of guanylate cyclase),[9,10] can act as inhibitors of glycogen-synthase kinase-3[11] and possess antineoplastic properties.[12]

In this work we present a synthesis of close analogues, triazolylfuroxans, by the [3 + 2] cycloaddition of azidofuroxans to terminal and internal acetylenes. Since furoxan derivatives are NO donors and are able to activate NO-sensitive sGC,[13] we expected that the replacement of the furazan ring with the furoxan one would essentially affect the pharmacological activity of the synthesized compounds.

4-Azidofuroxans **1a**,**b** were chosen as initial substrates. Synthesis of known 4-azido-3-phenylfuroxan **1a**[14] involves nucleophilic substitution of the nitro group in 4-nitro-3-phenylfuroxan **2a** under the action of NaN_3 in DMSO at room temperature. 4-Azido-3-propylfuroxan **1b** was prepared in a similar manner from 4-nitrofuroxan **2b** (Scheme 1).† Recently, we have developed



Scheme 1

Table 1 Screening of the reaction conditions for the synthesis of (1*H*-1,2,3-triazol-1-yl)furoxan **4a** from **1a** and **3a**.

Entry	**3a** : **1a**	Solvent	Temperature/°C	Time/h	Yield of **4a** (%)
1	4 : 1	Benzene	80	240	–
2	1 : 1	EtOH	78	240	–
3	2 : 1	EtOH	78	240	–
4	3 : 1	EtOH	78	240	Trace
5	2 : 1	DMF	100	144	–
6	5 : 1	DMF	100	60	Trace
7	5 : 1	[bmim]BF_4	20	300	–
8	1 : 1	[bmim]BF_4	80	240	Trace
9	2 : 1	[bmim]BF_4	80	120	21
10	3 : 1	[bmim]BF_4	80	20	57
11	4 : 1	[bmim]BF_4	80	20	56
12	3 : 1	[bmim]PF_6	80	96	49

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† *Synthesis of azidofuroxans* **1a**,**b** *(general procedure).* Sodium azide (25 mmol, 1.63 g) was added in one portion to a magnetically stirred solution of 4-nitrofuroxan **2a** or **2b** (10 mmol) in DMSO (15 ml) at room temperature. The mixture was stirred for 0.1–3 h until initial compound **2a** or **2b** disappeared [TLC monitoring; eluent, $CHCl_3$–CCl_4 (1:1)]. Then the reaction mixture was diluted with H_2O (30 ml). 4-Azido-3-phenylfuroxan **1a** was filtered, washed with H_2O and dried in air to afford compound **1a** in 95% yield. 4-Azido-3-propylfuroxan **1b** was extracted with CH_2Cl_2 (3×15 ml), the combined organic layers were washed twice with H_2O and dried over $MgSO_4$. Evaporation of the solvent gave compound **1b** in 89% yield.

*Synthesis of (1*H*-1,2,3-triazol-1-yl)furoxans* **4a–l** *and* **4′e–l** *(general procedure).* Appropriate acetylene (3 mmol) was added to the solution of azidofuroxan **1a** or **1b** (1 mmol) in [bmim]BF_4 (0.8 g). The reaction mixture was stirred at 80 °C until initial compound **1a** or **1b** disappeared [TLC monitoring; eluent, $CHCl_3$–CCl_4 (1:1)]. The reaction mixture was extracted with Et_2O (3×3 ml), the combined extracts were washed with H_2O and dried over $MgSO_4$. Then the solvent was evaporated and the residue was purified by column chromatography on SiO_2 (eluent, $CHCl_3$). Regioisomers **4e** and **4′e** were separated and other regioisomers were obtained as mixtures.

*4-[4,5-Bis(ethoxycarbonyl)-1*H*-1,2,3-triazol-1-yl]-3-phenylfuroxan* **4a**. Yield 0.212 g (57%) after first run of [bmim]BF_4, 0.209 g (56%) after second run of [bmim]BF_4, 0.216 g (58%) after third run of [bmim]BF_4, mp 124–126 °C. ^1H NMR ($CDCl_3$) δ: 1.25 (t, 3H, OCH_2Me, ^3J 7.7 Hz), 1.43 (t, 3H, OCH_2Me, ^3J 7.5 Hz), 4.30 (q, 2H, OCH_2Me, ^3J 7.7 Hz), 4.49 (q, 2H, OCH_2Me, ^3J 7.5 Hz), 7.37–7.49 (m, 5H, Ph). ^{13}C NMR ($CDCl_3$) δ: 13.50 and 13.94 (2 OCH_2Me), 62.38 and 63.62 (2 OCH_2Me), 111.24 (C-3 furoxan), 119.88, 126.74, 129.33, 131.38 (C_{Ph}), 130.74 (C-4 triazole), 139.34 (C-5 triazole), 151.14 (C-4 furoxan), 160.65 and 160.94 (2 C=O). HRMS (ESI), *m/z*: 374.1083 $[M + H]^+$ (calc. for $C_{16}H_{15}N_5O_6$, *m/z*: 374.1096). Found (%): C, 51.44; H, 4.02; N, 18.79. Calc. for $C_{16}H_{15}N_5O_6$ (%): C, 51.48; H, 4.05; N, 18.76.

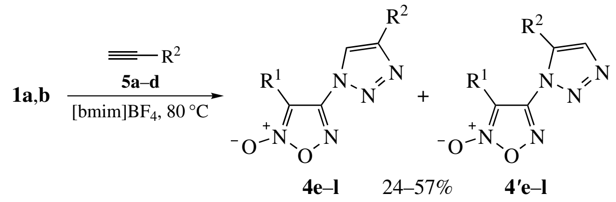
a convenient method for the synthesis of various 4-nitrofuroxans.[15]

To optimize the reaction conditions we used 4-azido-3-phenylfuroxan **1a** and diethyl acetylenedicarboxylate (DEAD) **3a** as model compounds. The reactions were carried out at different molar ratios **3a** : **1a** and in different solvents at various temperatures (Scheme 1, Table 1). Unfortunately, in contrast to reactions of azidofurazans with acetylene derivatives, the expected cycloaddition product (**4a**) was not obtained in none of the organic solvents even at prolonged heating (entries 1–6). Previously,[3] we have found that the replacement of common organic solvents with ionic liquids (ILs) (see ref. 16) accelerated the cycloaddition of 4-amino-3-azidofurazan to acetylene derivatives. Therefore, we investigated the interaction **1a** + **3a** in [bmim]BF_4 and [bmim]PF_6 (butylmethylimidazolium tetrafluoroborate and hexafluorophosphate) which are readily available and stable to air and moisture ILs.

At room temperature, azidofuroxan **1a** did not react with DEAD in [bmim]BF_4 even with a 5-molar excess of dipolarophile **3a** (entry 7). Target 3-phenyl-4-(1*H*-1,2,3-triazol-1-yl)furoxan **4a** was formed only on heating (entries 9–11). Hence, the optimal conditions were molar ratio **3a** : **1a** = 3 : 1, 80 °C, 20 h (TLC monitoring) (entry 10). We also succeeded in preparing compound **4a** in another IL, [bmim]PF_6, though 96 h were required for this reaction to complete (entry 12). Therefore, all subsequent reactions were carried out in [bmim]BF_4.

The processing of azidofuroxan **1b** with DEAD **3a** and the cycloaddition of both substrates **1a**,**b** to another internal acetylene, but-2-yne-1,4-diol **3b**, afforded (1*H*-1,2,3-triazol-1-yl)furoxans **4b–d** in good and moderate yields. However, the reactions with azidofuroxan **1b** were slower (~30 h) than those with azidofuroxan **1a** (~20 h) (TLC monitoring) (see Scheme 1).

Cycloaddition of azidofuroxans **1a**,**b** to terminal acetylenes **5a–d** proceeded analogously, while the reactions of azidofuroxan **1b** with acetylenes **5a–d** lasted longer (Scheme 2).[†] All the reactions occurred regioselectively and afforded a mixture of two regioisomers **4e–l** and **4′e–l** in total yields 24–57% with the predominance of regioisomers **4e–l**. The **4e–l** : **4′e–l** ratio (3 : 1–6 : 1) was determined from the integral intensity ratio of CH groups of the triazole rings in the ^{1}H NMR spectra.



5a	$R^2 = CO_2Me$	**4e**	$R^1 = Ph, R^2 = CO_2Me$	**4i**	$R^1 = Pr, R^2 = CO_2Me$
5b	$R^2 = CH_2OH$	**4f**	$R^1 = Ph, R^2 = CH_2OH$	**4j**	$R^1 = Pr, R^2 = CH_2OH$
5c	$R^2 = C(O)Me$	**4g**	$R^1 = Ph, R^2 = C(O)Me$	**4k**	$R^1 = Pr, R^2 = C(O)Me$
5d	$R^2 = Ph$	**4h**	$R^1 = R^2 = Ph$	**4l**	$R^1 = Pr, R^2 = Ph$

Scheme 2

Synthesized triazolylfuroxans **4** were isolated from IL by extraction with diethyl ether. Therefore, ILs can be regenerated and reused no less than three times without any loss of efficiency (as exemplified by the reaction of azidofuroxan **1a** with DEAD **3a**). Structures of all the synthesized compounds were established by elemental analysis and spectral data (^{1}H, ^{13}C NMR and mass spectra). To correctly attribute the structure of the synthesized compounds to regioisomers **4** or **4′**, regioisomers **4e** and **4′e** were separated by column chromatography on SiO_2 (eluent $CHCl_3$) and, in addition, predominant regioisomer **4i** was isolated under the same conditions. The isolated regioisomers were analyzed using {^{1}H-^{1}H}gNOESY, {^{1}H-^{13}C}HSQC, {^{1}H-^{13}C}HMBC and {^{1}H-^{15}N}HMBC methods. The most important NOE interactions of protons in regioisomers **4e**, **4′e** and **4i** were revealed in the NOESY experiments (Figure 1). The chemical shifts of protons and carbon atoms in NMR spectra of predominant regioisomers are more upfield than those of minor regioisomers.

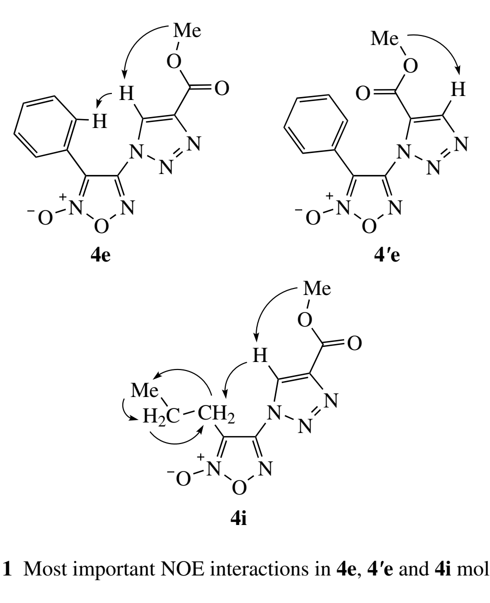


Figure 1 Most important NOE interactions in **4e**, **4′e** and **4i** molecules.

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*4-[4,5-Bis(ethoxycarbonyl)-1*H*-1,2,3-triazol-1-yl]-3-propylfuroxan* **4c**. Yield 0.163 g (48%), non-distillable oil. ^{1}H NMR ($CDCl_3$) δ: 1.05 (t, 3H, *Me*CH_2CH_2, ^3J 7.5 Hz), 1.21 (t, 3H, OCH_2*Me*, ^3J 7.4 Hz), 1.38 (t, 3H, OCH_2*Me*, ^3J 7.7 Hz), 1.66–1.75 (m, 2H, MeC*H*$_2$$CH_2$), 2.86 (t, 2H, $MeCH_2C$*H*$_2$, ^3J 7.5 Hz), 4.27 (q, 2H, OC*H*$_2$Me, ^3J 7.4 Hz), 4.43 (q, 2H, OC*H*$_2$Me, ^3J 7.7 Hz). ^{13}C NMR ($CDCl_3$) δ: 13.11 (*Me*CH_2CH_2), 13.44 and 13.73 (2OCH_2*Me*), 18.95 (Me*C*H_2CH_2), 24.79 ($MeCH_2$*C*H_2), 62.59 and 63.85 (2O*C*H_2Me), 111.72 (C-3 furoxan), 130.84 (C-4 triazole), 138.83 (C-5 triazole), 150.96 (C-4 furoxan), 160.83 and 161.13 (2C=O). Found (%): C, 45.99; H, 5.04; N, 20.67. Calc. for $C_{13}H_{17}N_5O_6$ (%): C, 46.02; H, 5.05; N, 20.64.

*4-[4-(Methoxycarbonyl)-1*H*-1,2,3-triazol-1-yl]-3-phenylfuroxan* **4e**. Yield 0.075 g (26%), mp 81–83 °C. **4e** : **4′e** ratio is 3 : 1. ^{1}H NMR ($CDCl_3$) δ: 4.03 (s, 3H, OMe), 7.47–7.55 (m, 5H, Ph), 8.67 (s, 1H, CH triazole). ^{13}C NMR ($CDCl_3$) δ: 52.73 (OMe), 110.97 (C-3 furoxan), 120.06, 128.09, 129.28, 131.64 (C_{Ph}), 129.43 (C-4 triazole), 132.28 (C-5 triazole), 148.79 (C-4 furoxan), 159.88 (C=O). Found (%): C, 50.16; H, 3.19; N, 24.36. Calc. for $C_{12}H_9N_5O_4$ (%): C, 50.18; H, 3.16; N, 24.38.

*4-[5-(Methoxycarbonyl)-1*H*-1,2,3-triazol-1-yl]-3-phenylfuroxan* **4′e**. Yield 0.026 g (9%), mp 73–75 °C. ^{1}H NMR ($CDCl_3$) δ: 3.82 (s, 3H, OMe), 7.30–7.33 (m, 2H, Ph), 7.39–7.48 (m, 3H, Ph), 8.32 (s, 1H, CH triazole). ^{13}C NMR ($CDCl_3$) δ: 53.24 (OMe), 111.74 (C-3 furoxan), 120.52, 126.49, 129.43, 131.35 (C_{Ph}), 131.01 (C-4 triazole), 137.46 (C-5 triazole), 148.96 (C-4 furoxan), 156.86 (C=O). Found (%): C, 50.15; H, 3.17; N, 24.41. Calc. for $C_{12}H_9N_5O_4$ (%): C, 50.18; H, 3.16; N, 24.38.

*4-[4-(Methoxycarbonyl)-1*H*-1,2,3-triazol-1-yl]-3-propylfuroxan* **4i** *and 4-[5-(methoxycarbonyl)-1*H*-1,2,3-triazol-1-yl]-3-propylfuroxan* **4′i**. Yield 0.094 g (37%), mixture of regioisomers, **4i** : **4′i** ratio is 4.5 : 1. For **4i**: ^{1}H NMR ($CDCl_3$) δ: 1.01 (t, 3H, *Me*CH_2CH_2, ^3J 7.4 Hz), 1.71–1.80 (m, 2H, MeC*H*$_2$$CH_2$), 2.97 (t, 2H, $MeCH_2C$*H*$_2$, ^3J 7.4 Hz), 4.03 (s, 3H, OMe), 8.86 (s, 1H, CH triazole). ^{13}C NMR ($CDCl_3$) δ: 13.49 (*Me*CH_2CH_2), 19.23 (Me*C*H_2CH_2), 25.13 ($MeCH_2$*C*H_2), 52.77 (OMe), 111.18 (C-3 furoxan), 126.13 (C-5 triazole), 140.61 (C-4 triazole), 150.05 (C-4 furoxan), 159.87 (C=O). For **4′i**: ^{1}H NMR ($CDCl_3$) δ: 1.09 (t, 3H, *Me*CH_2CH_2, ^3J 7.6 Hz), 1.86–1.97 (m, 2H, MeC*H*$_2$$CH_2$), 3.12 (t, 2H, $MeCH_2C$*H*$_2$, ^3J 7.6 Hz), 3.84 (s, 3H, OMe), 8.31 (s, 1H, CH triazole). ^{13}C NMR ($CDCl_3$) δ: 13.62 (*Me*CH_2CH_2), 19.84 (Me*C*H_2CH_2), 25.76 ($MeCH_2$*C*H_2), 53.24 (OMe), 111.92 (C-3 furoxan), 130.81 (C-4 triazole), 137.61 (C-5 triazole), 148.73 (C-4 furoxan), 159.96 (C=O). Found (%): C, 42.71; H, 4.35; N, 27.65. Calc. for $C_9H_{11}N_5O_4$ (%): C, 42.69; H, 4.38; N, 27.66.

For characteristics of compounds **1a**,**b**, **4b**,**d**,**f–h**,**j–l**, see Online Supplementary Materials.

In conclusion, we have developed a simple access to previously unknown hybrid molecules, 3-R-4-(1*H*-1,2,3-triazol-1-yl)furoxans, based on the [3+2] cycloaddition of azidofuroxans to internal and terminal acetylenes. The advantages of this method are operational simplicity and the use of environmentally friendly ionic liquids. The developed protocol provides a powerful tool for the synthesis of new types of potential NO donors.

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Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.mencom.2015.07.007.

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