Synthesis of Furyl-Substituted Isoxazoles, Isoxazolines, and 1,2,4-Oxadiazoles

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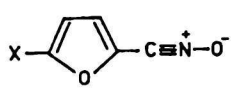
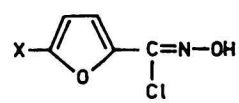
The synthesis of 3,5-disubstituted isoxazoles, isoxazolines, and 1,2,4-oxadiazoles by 1,3-dipolar cycloaddition of 5-X-2-furonitrile oxides (X = 4-, 3-, 2-nitrophenyl or nitro group) with phenylacetylene, propargyl bromide, styrene, allyl alcohol, allyl bromide, and *N*-cyanoguanidine is described.

1,3-Dipolar cycloaddition reactions of nitrile oxides have over the years preserved their synthetic potential and still represent the best way to synthesize isoxazoles and isoxazolines [1]. Isoxazolines in turn serve as viable precursors to many acyclic compounds, they have been increasingly involved in the synthesis of natural compounds [2, 3]. In the chemistry of furan the nitrile oxides have received little attention, some of them were used to synthesize cycloadducts with bacteriostatic and chemotherapeutic activity [4—6].



Ia—Id



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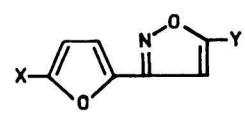
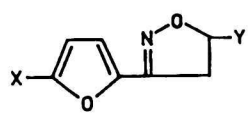
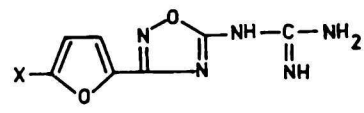


IIIa—IIIg



IVa—IVf



Va—Vc

	X
Ia, *IIa*	4-NO_2Ph
Ib, *IIb*	3-NO_2Ph
Ic, *IIc*	2-NO_2Ph
Id, *IId*	NO_2

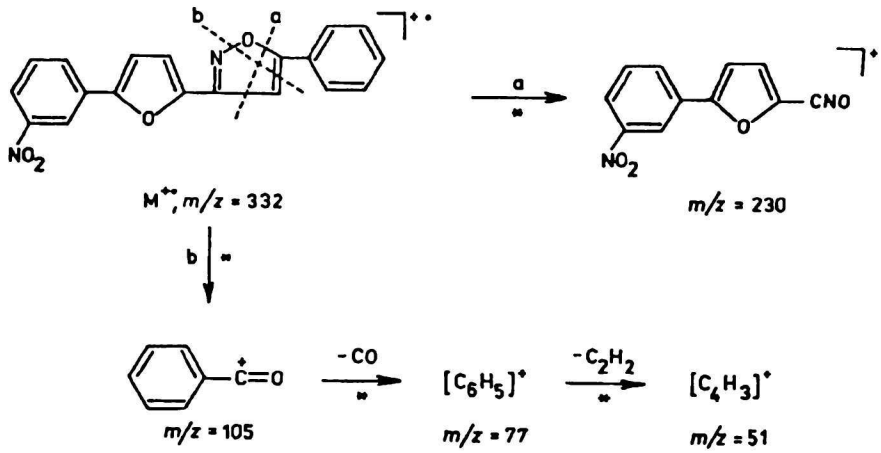
On the background of our earlier studies [7—9] of 1,3-dipolar cycloaddition reactions of substituted 2- and 3-furonitrile oxides with cyclic alkenes and heterocyclic dipolarophiles, this report deals with 1,3-dipolar cycloadditions of 5-substituted 2-furonitrile oxides *Ia—Id*, where the substituent was 4-, 3-, 2-nitrophenyl and nitro group, respectively, with dipolarophiles such as phenylacetylene, propargyl bromide, allyl alcohol, allyl bromide, styrene, and *N*-cyanoguanidine. The series was prepared in order to assess the influence of arylfuryl and phenyl group on the biological activity of isoxazolines and isoxazoles.

The studied cycloaddition reactions proceeded regioselectively (see ^{1}H NMR spectra in Experimental) to give solely 5-substituted isoxazoles and isoxazolines, exactly as predicted by MNDO calculations for used nitrile oxides [8] and in accord with the known experimental data. Reactions of nitrile oxides *I* with styrene, allyl bromide, and allyl alcohol proceeded with high yields (80—96 %), as could be expected from the generally higher reactivity of alkenes *vs.* alkynes in cycloaddition reactions [10].

The requisite isoxazoles *IIIa*—*IIIg* were prepared by two independent procedures. The first one involved the generation of nitrile oxides *Ia*—*Id in situ* from the corresponding carbohydroximoyl chlorides *IIa*—*IId* at low temperatures [11] by the action of triethylamine. In an alternative method one refluxes the starting furancarbohydroximoyl chloride (*IIa*—*IId*) and the dipolarophile in a toluene solution as long as the hydrogen chloride is released. Thus the reaction of 5-(nitrophenyl)-2-furancarbohydroximoyl chlorides *IIa*—*IIc* with phenylacetylene furnished in about equal yields (39—41 %) the 3,5-disubstituted isoxazoles *IIIa*—*IIIc* (besides dimers of nitrile oxides) irrespective of which procedure was used. The structure of isolated cycloadducts was inferred from spectral and MS data. The first procedure afforded 5-bromomethyl-3-(X-furyl)-substituted isoxazoles *IIId*—*IIIf* in 42—58 % yields, whereas the thermally induced cycloadditions (the second procedure) gave a mixture of products. In case when propargyl bromide was the dipolarophile, mass spectra indicate the presence of two compounds, namely the expected 3-[5-(3-nitrophenyl)-2-furyl]-5-bromomethylisoxazole (*IIIe*), and an analogous 5-chloromethylisoxazole derivative. The latter compound could not be isolated in pure state, its presence is corroborated by the corresponding peaks in its mass spectrum: $M^{+\bullet}$ 348, $M^{+\bullet}$ 304, *m/z* (I_r/%): 269, 255, 227 (100), 195, 181, 160, 153.

1,3-Dipolar cycloadditions of 5-nitro-2-furonitrile oxide (*Id*) were studied by *Sasaki* [4], who prepared derivative *IIIg* by the second procedure, *i.e.* by the reaction of corresponding hydroximoyl chloride with propargyl bromide in refluxing toluene. We were successful also with classical Huisgen method [10], based on *in situ* generation of nitrile oxide.

1,2,4-Oxadiazoles *Va*—*Vc* were prepared by reaction of *N*-cyanoguanidine with hydroximoyl chlorides *IIa*—*IIc* carried out in boiling ethanol. Compounds *Va*—*Vc* are hard to crystallize; they were purified by crystallization from acetic acid, followed by alkaline hydrolysis to liberate free base. In the infrared spectra of the latter compounds there are stretching vibration bands of the C=N group at $\tilde{\nu} = 1628$—$1640\ cm^{-1}$ and 1662—1682 cm^{-1}, the higher wavenumbers being those of the C=N group in the guanidine moiety in position 5 of 1,2,4-oxadiazole. Ultraviolet spectra of compounds *III*—*V* resemble UV spectra of



	X	Y
IIIa	4-NO_2Ph	Ph
IIIb	3-NO_2Ph	Ph
IIIc	2-NO_2Ph	Ph
IIId	4-NO_2Ph	CH_2Br
IIIe	3-NO_2Ph	CH_2Br
IIIf	2-NO_2Ph	CH_2Br
IIIg	NO_2	CH_2Br
IVa	4-NO_2Ph	CH_2Br
IVb	3-NO_2Ph	CH_2Br
IVc	2-NO_2Ph	CH_2Br
IVd	3-NO_2Ph	Ph
IVe	2-NO_2Ph	Ph
IVf	3-NO_2Ph	CH_2OH
Va	4-NO_2Ph	
Vb	3-NO_2Ph	
Vc	2-NO_2Ph	

Scheme 1

5-X-2-furaldehyde oximes, or their functional derivatives [11], indicating that a similar type of chromophore is responsible for the maxima. In all compounds a conjugation between the nitrophenylfuryl group and the C=N bond can be expected [1].

^{1}H NMR spectra of selected compounds *III* support the proposed structure. In the spectrum of *IIIc* the signal of H-4 of isoxazole ring has been significantly upfield shifted ($\delta = 6.65$), when compared with compounds *IIId*, *IIIe*, and *IIIg* ($\delta = 7.05$), due to the shielding effect of the phenyl in position 5. The mass spectrum and the tentative fragmentation scheme of *IIIb* corroborate its proposed structure. The most intensive fragment ion peak *m/z* = 105 arises from the splitting of the isoxazole ring by the route *b* (Scheme 1). 1,3-Dipolar cycloreversion along route *a* is responsible for fragment *m/z* = 230. Fragment ions *m/z*: 39, 51, 63, 77 testify to the presence of aromatic ring in the molecule.

Structure assignment of compounds *IV*, prepared from the corresponding carbohydroximoyl chlorides *IIa—IIc* and styrene, allyl alcohol, and allyl bromide, respectively by Huisgen method, was done by ^{1}H NMR spectroscopy. Protons of isoxazoline ring in compounds *IV* form an ABX system. The H-5 proton signal in *IVd* and *IVe* appears as doublet of doublets at $\delta = 5.7$ and coupling constants $J = 8.5$ Hz and 9.4 Hz for *IVd* and $J = 8.9$ Hz and 9.4 Hz for *IVe*. If there is a bromomethyl or a hydroxymethyl group attached in position 5, the above signal appears as a somewhat downfield shifted multiplet ($\delta \approx 5.0$) in *IVa—IVc* and at $\delta = 4.8$ in *IVf*, as could be expected from the shielding effects of the respective substituents.

EXPERIMENTAL

Melting points were determined with a Kofler hot-stage. ^{1}H NMR spectra of hexadeuteroacetone solutions of the prepared compounds, with tetramethylsilane as internal standard, were taken with an 80 MHz spectrometer BS 487C (Tesla). Infrared spectra of KBr discs were determined with Specord 71 R (Zeiss, Jena). UV spectra of thermostated methanolic solutions (λ/nm, ε/(m^2 mol^{-1})) were measured with a spectrophotometer M-20 (Zeiss, Jena). Mass spectra were recorded on an AEI spectrometer MS 902S with direct inlet and an ionizing energy of 70 eV, capture current 100 μA and the temperature of ionizing chamber 80—215 °C.

The progress of reaction was monitored by TLC on silica gel (Silufol, Kavalier; eluant chloroform and hexane—ethyl acetate (φ_r = 4 : 1), respectively, detection by UV 254 nm). Purification of samples was carried out, unless otherwise specified, on silica gel columns, eluted by chloroform.

The requisite 5-substituted 2-furancarbohydroximoyl chlorides were prepared according to Refs. [11, 12].

3-(5-X-2-Furyl)-5-Y-isoxazoles *III*

Method A. Triethylamine (3 cm^3) is added to the stirred ethereal solution of 5-X-2-furancarbohydroximoyl chloride (0.01 mol; 40 cm^3), kept at − 35 °C [11]. The separated triethylammonium chloride was removed from the cool solution and the solution of nitrile oxide was left to react with 0.02 mol of dipolarophile. The reaction mixture was first stirred for 30 min at − 30 °C, then for 20 h at room temperature, thereupon the solvent was removed *in vacuo* and the solid residue was purified on a silica gel column and by subsequent crystallization.

Method B. The reaction mixture, consisting of 5-X-2-furancarbohydroximoyl chloride (0.01 mol), dipolarophile (0.015 mol), and toluene (40—50 cm^3) was refluxed until the evolution of hydrogen chloride ceased (12—28 h). The solvent was then removed and the product purified in the manner described under the method *A*.

3-[5-(4-Nitrophenyl)-2-furyl]-5-phenylisoxazole (*IIIa*), yield = 39 %, m.p. = 183—184 °C. For $C_{19}H_{12}N_2O_4$ (M_r = 322.2) w_i(calc.): 68.66 % C, 3.63 % H, 8.43 % N; w_i(found): 68.61 % C, 3.49 % H, 8.59 % N. UV spectrum, λ_{max}/nm (log {ε}): 207 (4.40), 267 (4.33), 363 (4.38). IR spectrum, $\tilde{\nu}$ / cm^{-1}: 1535 $\nu_{as}(NO_2)$, 1342 $\nu_s(NO_2)$, 1622 ν(C=N). Mass spectrum, *m/z*: $M^{+\bullet}$ 332, base peak 105.

3-[5-(3-Nitrophenyl)-2-furyl]-5-phenylisoxazole (*IIIb*), yield = 40 %, m.p. = 201—202 °C. For $C_{19}H_{12}N_2O_4$ (M_r = 322.2) w_i(calc.): 68.66 % C, 3.63 % H, 8.43 % N; w_i(found): 68.58 % C, 3.68 % H, 8.67 % N. UV spectrum, λ_{max}/nm (log {ε}): 210 (4.37), 274 (4.33), 311 (4.44). IR spectrum, $\tilde{\nu}$ / cm^{-1}: 1540 $\nu_{as}(NO_2)$, 1358 $\nu_s(NO_2)$, 1629 ν(C=N). Mass spectrum, *m/z*: $M^{+\bullet}$ 332, base peak 105.

3-[5-(2-Nitrophenyl)-2-furyl]-5-phenylisoxazole (*IIIc*), yield = 41 %, m.p. = 98—99 °C. For $C_{19}H_{12}N_2O_4$ (M_r = 322.2) w_i(calc.): 68.66 % C, 3.63 % H, 8.43 % N; w_i(found): 68.79 % C, 3.58 % H, 8.48 % N. UV spectrum, λ_{max}/nm (log {ε}): 208 (4.46), 289 (4.45). IR spectrum, $\tilde{\nu}$ /cm^{-1}: 1548 $\nu_{as}(NO_2)$, 1361 $\nu_s(NO_2)$, 1631 ν(C=N). ^{1}H NMR spectrum (deuteroacetone), δ: 6.65 (s, 1H, H-4), 6.67 (d, 1H, H-3′), J = 3.5 Hz, 6.92 (d, 1H, H-4′), 7.28—7.71 (m, 4H, H_{arom}).

3-[5-(4-Nitrophenyl)-2-furyl]-5-bromomethylisoxazole (*IIId*), yield = 45 %, m.p. = 167 °C. For $C_{14}H_9BrN_2O_4$ (M_r = 349.1) w_i(calc.): 48.16 % C, 2.59 % H, 8.02 % N; w_i(found): 47.92 % C, 2.46 % H, 8.14 % N. UV spectrum, λ_{max}/nm (log {ε}): 208 (4.26), 236 (inflex), 359 (4.38). IR spectrum, $\tilde{\nu}$ /cm^{-1}: 1535 $\nu_{as}(NO_2)$, 1347 $\nu_s(NO_2)$, 1690 ν(C=N). ^{1}H NMR spectrum: 4.81 (s, 2H, CH_2), 7.05 (s, 1H, H-4), 7.21 (d, 1H, H-3′), J = 3.5 Hz, 7.38 (d, 1H, H-4′), 8.08 (d, 2H, $H_{arom\ 2,6}$), 8.34 (d, 2H, $H_{arom\ 3,5}$), J = 9.5 Hz.

3-[5-(3-Nitrophenyl)-2-furyl]-5-bromomethylisoxazole (*IIIe*), yield = 58 %, m.p. = 145 °C. For $C_{14}H_9BrN_2O_4$ (M_r = 349.1) w_i(calc.): 48.16 % C, 2.59 % H, 8.02 % N; w_i(found): 47.98 % C, 2.53 % H, 8.25 % N. UV spectrum, λ_{max}/nm (log {ε}): 212 (4.40), 268 (inflex), 310 (4.44). IR spectrum,

$\tilde{\nu}$/cm^{-1}: 1545 $\nu_{as}(NO_2)$, 1360 $\nu_s(NO_2)$, 1672 ν(C=N). ^{1}H NMR spectrum, δ: 4.81 (s, 2H, CH_2), 7.04 (s, 1H, H-4), 7.23 (d, 1H, H-3′), J = 3.5 Hz, 7.31 (d, 1H, H-4′), 7.65—8.62 (m, 4H, H_{arom}). Mass spectrum, *m/z*: $M^{+\bullet}$ 348, base peak 227.

3-[5-(2-Nitrophenyl)-2-furyl]-5-bromomethylisoxazole (*IIIf*), yield = 51 %, m.p. = 77 °C. For $C_{14}H_9BrN_2O_4$ (M_r = 349.1) w_i(calc.): 48.16 % C, 2.59 % H, 8.02 % N; w_i(found): 48.03 % C, 2.48 % H, 8.29 % N. UV spectrum, λ_{max}/nm (log {ε}): 213 (4.30), 295 (4.31). IR spectrum, $\tilde{\nu}$/cm^{-1}: 1550 $\nu_{as}(NO_2)$, 1365 $\nu_s(NO_2)$, 1688 ν(C=N).

3-(5-Nitro-2-furyl)-5-bromomethylisoxazole (*IIIg*), yield = 42 %, m.p. = 109—110 °C. For $C_8H_5BrN_2O_4$ (M_r = 273.0) w_i(calc.): 35.19 % C, 1.84 % H, 10.26 % N; w_i(found): 35.03 % C, 2.02 % H, 10.14 % N. ^{1}H NMR spectrum, δ: 4.83 (s, 2H, CH_2), 7.09 (s, 1H, H-4), 7.38 (d, 1H, H-3′), J = 3.5 Hz, 7.67 (d, 1H, H-4′).

3-(5-X-2-Furyl)-5-Y-4,5-dihydroisoxazoles *IV*

Triethylamine (2 cm^3 in dry ether) was during 1 h added to the stirred, cooled (–15 °C) solution of 5-X-2-furancarbohydroximoyl chloride (0.01 mol) and dipolarophile (0.05 mol) in dry ether (30—40 cm^3). The reaction mixture was stirred for another 18 h at room temperature, the solvent removed and the solid residue thoroughly washed with water in order to remove the triethylammonium chloride. The raw reaction mixture was then separated on a silica gel column and the obtained products were purified by crystallization.

3-[5-(4-Nitrophenyl)-2-furyl]-5-bromomethyl-4,5-dihydroisoxazole (*IVa*), yield = 83 %, m.p. = 144—146 °C. For $C_{14}H_{11}BrN_2O_4$ (M_r = 351.2) w_i(calc.): 47.87 % C, 3.16 % H, 7.97 % N; w_i(found): 47.69 % C, 3.34 % H, 7.96 % N. UV spectrum, λ_{max}/nm (log {ε}): 282 (2.89), 364 (3.32). ^{1}H NMR spectrum, δ: 3.16—3.76 (m, 4H, H_2-4, CH_2Br), 4.96—5.14 (m, 1H, H-5), 6.87 (d, 1H, H-3′), J = 3.53 Hz, 6.97 (d, 1H, H-4′), 7.89 (d, 2H, $H_{arom\ 2,6}$), 8.21 (d, 2H, $H_{arom\ 3,5}$), J = 8.7 Hz.

3-[5-(3-Nitrophenyl)-2-furyl]-5-bromomethyl-4,5-dihydroisoxazole (*IVb*), yield = 79 %, m.p. = 124—126 °C. For $C_{14}H_{11}BrN_2O_4$ (M_r = 351.20) w_i(calc.): 47.87 % C, 3.16 % H, 7.97 % N; w_i(found): 47.79 % C, 3.42 % H, 8.00 % N. UV spectrum, λ_{max}/nm (log {ε}): 316 (3.44). ^{1}H NMR spectrum, δ: 3.26—3.77 (m, 4H, H_2-4, CH_2Br), 4.86—5.13 (m, 1H, H-5), 6.86 (d, 1H, H-3′), J = 3.5 Hz, 6.92 (d, 1H, H-4′), 7.48—8.53 (m, 4H, H_{arom}).

3-[5-(2-Nitrophenyl)-2-furyl]-5-bromomethyl-4,5-dihydroisoxazole (*IVc*), yield = 84 %, m.p. = 117—118 °C. For $C_{14}H_{11}BrN_2O_4$ (M_r = 351.20) w_i(calc.): 47.87 % C, 3.16 % H, 7.97 % N; w_i(found): 47.96 % C, 3.36 % H, 7.93 % N. UV spectrum, λ_{max}/nm (log {ε}): 299 (3.20). ^{1}H NMR spectrum, δ: 3.28—3.68 (m, 4H, H_2-4, CH_2Br), 4.78—5.17 (m, 1H, H-5), 6.73 (d, 1H, H-3′), J = 3.47 Hz, 6.84 (d, 1H, H-4′), 7.43—7.80 (m, 4H, H_{arom}).

3-[5-(3-Nitrophenyl)-2-furyl]-5-phenyl-4,5-dihydroisoxazole (*IVd*), yield = 84 %, m.p. = 142—144 °C. For $C_{19}H_{14}N_2O_4$ (M_r = 320.46) w_i(calc.): 71.21 % C, 4.63 % H, 8.74 % N; w_i(found): 71.32 % C, 4.51 % H, 8.67 % N. UV spectrum, λ_{max}/nm (log {ε}): 317 (3.41). ^{1}H NMR spectrum, δ: 3.32 (dd, 1H, H_B-4), 3.81 (dd, 1H, H_A-4), J_{AB} = 16.7 Hz, 5.77 (dd, 1H, H-5), J = 10.6 Hz, 6.87 (d, 1H, H-3′), J = 3.5 Hz, 6.91 (d, 1H, H-4′), 7.23—8.53 (m, 9H, H_{arom}).

3-[5-(2-Nitrophenyl)-2-furyl]-5-phenyl-4,5-dihydroisoxazole (*IVe*), yield = 96 %, m.p. = 108—110 °C. For $C_{19}H_{14}N_2O_4$ (M_r = 320.46) w_i(calc.): 71.21 % C, 4.63 % H, 8.74 % N; w_i(found): 71.04 % C, 4.47 % H, 8.62 % N. UV spectrum, λ_{max}/nm (log {ε}): 302 (3.24). ^{1}H NMR spectrum, δ: 3.26 (dd, 1H, H_B-4), 3.75 (dd, 1H, H_A-4), J_{AB} = 16.9 Hz, 5.70 (dd, 1H, H-5), J = 8.8 Hz, 6.77 (d, 1H, H-3′), J = 3.5 Hz, 6.87 (d, 1H, H-4′), 7.26—7.81 (m, 9H, H_{arom}).

3-[5-(3-Nitrophenyl)-2-furyl]-5-hydroxymethyl-4,5-dihydroisoxazole (*IVf*), yield = 92 %, m.p. = 183—186 °C. For $C_{14}H_{12}N_2O_5$ (M_r = 288.27) w_i(calc.): 73.66 % C, 5.30 % H, 12.27 % N; w_i(found): 73.45 % C, 5.11 % H, 12.09 % N. UV spectrum, λ_{max}/nm (log {ε}): 317 (3.39). ^{1}H NMR spectrum, δ: 3.32—3.89 (m, 4H, H_2-4, CH_2—OH), 4.70—4.98 (m, 1H, H-5), 6.84 (d, 1H, H-3′), J = 3.5 Hz, 6.90 (d, 1H, H-4′), 7.43—8.21 (m, 4H, H_{arom}), 8.54 (s, 1H, OH).

3-(5-X-2-Furyl)-5-guanidino-1,2,4-oxadiazoles *V*

The mixture consisting of the respective hydroximoyl chloride *II* (0.01 mol) and *N*-cyanoguanidine (0.011 mol), dissolved in ethanol (30 cm^3), was refluxed for 3—5 h. After cooling to room temperature, the separated solid part was crystallized from acetic acid; the free base was then liberated by 10 % sodium hydroxide.

3-[5-(4-Nitrophenyl)-2-furyl]-5-guanidino-1,2,4-oxadiazole (*Va*), yield = 48 %, m.p. > 320 °C. For

$C_{13}H_{10}N_6O_4$ (M_r = 314.25) w_i(calc.): 49.68 % C, 3.20 % H, 26.74 % N; w_i(found): 49.37 % C, 3.28 % H, 26.64 % N. UV spectrum, λ_{max}/nm (log {ε}): 259 (4.38), 361 (4.31). IR spectrum, $\tilde{\nu}$ /cm^{-1}: 1532 $\nu_{as}(NO_2)$, 1338 $\nu_s(NO_2)$, 1640 ν(C=N), 1682 ν(C=N).

3-[5-(3-Nitrophenyl)-2-furyl]-5-guanidino-1,2,4-oxadiazole (*Vb*), yield = 62 %, m.p. > 320 °C. For $C_{13}H_{10}N_6O_4$ (M_r = 314.25) w_i(calc.): 49.68 % C, 3.20 % H, 26.74 % N; w_i(found): 49.90 % C, 3.28 % H, 26.68 % N. UV spectrum, λ_{max}/nm (log {ε}): 268 (4.44), 308 (4.46). IR spectrum, $\tilde{\nu}$ /cm^{-1}: 1534 $\nu_{as}(NO_2)$, 1345 $\nu_s(NO_2)$, 1628 ν(C=N), 1680 ν(C=N).

3-[5-(2-Nitrophenyl)-2-furyl]-5-guanidino-1,2,4-oxadiazole (*Vc*), yield = 65 %, m.p. = 275—276 °C. For $C_{13}H_{10}N_6O_4$ (M_r = 314.25) w_i(calc.): 49.68 % C, 3.20 % H, 26.74 % N; w_i(found): 49.69 % C, 3.23 % H, 26.79 % N. UV spectrum, λ_{max}/nm (log {ε}): 262 (4.38), 288 (inflex). IR spectrum, $\tilde{\nu}$ / cm^{-1}: 1540 $\nu_{as}(NO_2)$, 1348 $\nu_s(NO_2)$, 1638 ν(C=N), 1662 ν(C=N).

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