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# Synthesis of 3,5-Disubstituted 1,2,4-Oxadiazoles from Amidoximes and Aldehydes in the Superbasic System NaOH/DMSO

**A. A. Shetnev[a,*], V. E. Pankratieva[b], A. S. Kunichkina[b], A. S. Vlasov[a], I. K. Proskurina[b], A. D. Kotov[b], and M. K. Korsakov[a]**

[a] *Dorogov Pharmaceutical Technology Transfer Center, Ushinsky Yaroslavl State Pedagogical University, Yaroslavl, 150000 Russia*

[b] *Ushinsky Yaroslavl State Pedagogical University, Yaroslavl, 150000 Russia*

**e-mail: zlodeus@gmail.com*

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**Abstract**—A new procedure has been proposed for the synthesis of 3,5-disubstituted 1,2,4-oxadiazoles by reaction of amidoximes with aldehydes in the superbasic system NaOH/DMSO at room temperature. The scope of the proposed procedure has been demonstrated by 15 syntheses from various amidoximes and aromatic aldehydes with 27–76% yields. The procedure is inapplicable to aliphatic aldehydes.

**Keywords:** aldehyde, amidoxime, oxadiazole, superbasic medium, condensation, dimethyl sulfoxide, sodium hydroxide

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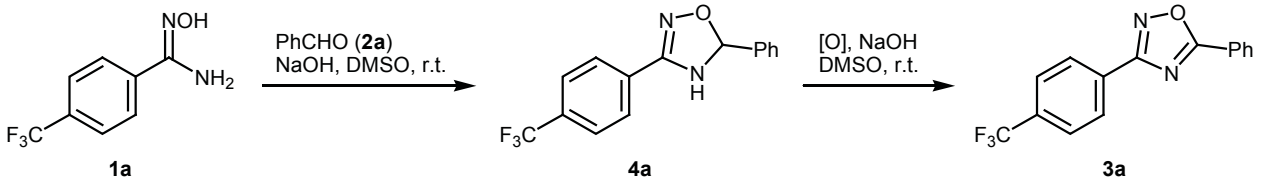
1,2,4-Oxadiazoles possess various useful properties, and they occupy an important place in applied chemistry. Derivatives of 1,2,4-oxadiazoles are components of many drugs [1–4] and are used as base structures of liquid crystals, energetic compounds [5], and luminophores [6]. Several methods of synthesis of 1,2,4-oxadiazoles have been reported [7–12], the most practical of which are those based on the condensation of amidoximes with carboxylic acids and their derivatives. Superbase-catalyzed reactions of amidoximes with electrophiles, in particular with acetylene [13, 14], were studied in [15–20], where 1,2,4-oxadiazoles were synthesized from esters, imidazolides, carboxylic acid anhydrides, and other carbonyl compounds in the system NaOH/DMSO. The products of these reactions were often found to possess valuable biological properties [21–25]. However, until recently, reactions of amidoximes with aldehydes have been reported in a few publications; these reactions were catalyzed by 4-toluenesulfonic [26] or acetic acid or carried out under microwave irradiation [27], and the yields were generally moderate to low. In our opinion, the lack of reliable methods of synthesis of needed heterocyclic systems of the 1,2,4-oxadiazole series using aldehydes as initial compounds is a serious gap in the set of tools of modern organic chemistry since aromatic and aliphatic aldehydes are synthetic precursors of carboxylic acids and their derivatives. In some cases, the use of aldehydes for the synthesis of practically important 1,2,4-oxadiazoles could be preferable from the economic viewpoint. An example is the large-scale production of such aldehydes as 4-hydroxymethylfurfural, furfural, fructose, formaldehyde, propanal, acetaldehyde, vanillin, and many others from natural raw materials.

Cesium carbonate-catalyzed thermal oxidative condensation of aldehydes and amidoximes [28] has become an important step in filling the above gap in the synthesis of 1,2,4-oxadiazoles. In this reaction, the aldehyde molecule acts as not only electrophile but also oxidant for initially formed dihydrooxadiazole ring. Further to this study, herein we report the reaction of amidoximes with aromatic and aliphatic aldehydes in the system NaOH/DMSO as a part of our research on superbase-catalyzed reactions of amidoxime. We found that amidoximes reacted with aromatic aldehydes in DMSO to give 1,2,4-oxadiazoles even at room temperature and that the target products can be isolated

**Scheme 1.**




**1a** **4a** **3a**

in acceptable yields despite the possibility of concurrent base-catalyzed Cannizzaro disproportionation of the aldehyde.

The reaction of *N*′-hydroxy-4-(trifluoromethyl)-benzimidamide (**1a**) with benzaldehyde (**2a**) was used as a model to optimize the conditions (Scheme 1); the results are summarized in Table 1.

We believe that the reaction proceeds in two steps. In the first step, amidoxime **1** reacts with aldehyde **2** in DMSO to give intermediate **4**. Under superbasic conditions, this reaction is complete in 1 h. The second step is oxidation of dihydrooxadiazole **4** to oxadiazole **3**, which takes 18–24 h. In order to improve the yield of target 1,2,4-oxadiazoles **3**, such conditions as reaction time, amount of the base, and reactant ratio were varied. In most experiments (Table 1, entry nos. 1–9), the dihydrooxadiazole ring in **4** was oxidized with excess aldehyde. In order to obtain satisfactory to good yield, more than 2 equiv of aldehyde should be used. However, in this case, the product isolation procedure was complicated due to the necessity of resorting to chromatographic purification from products of aldehyde disproportionation in alkaline medium. Apart from excess aldehyde, Oxone (entry no. 10), *N*-chlorosuccinimide (NCS, entry no. 11), atmospheric oxygen (entry no. 12), and dichlorodicyanoquinone (DCQ, entry no. 13) were tried as oxidants. Replacement of the solvent (DMSO) by *N*,*N*-dimethylformamide (entry no. 8) or *N*-methylpyrollidin-2-one (entry no. 9) did not affect the product yield to an appreciable extent. In all cases, the yields of **3a** were lower than in the model reaction with 2.2 equiv of benzaldehyde (entry no. 7), and we failed to reduce the consumption of aldehyde in the system NaOH/DMSO.

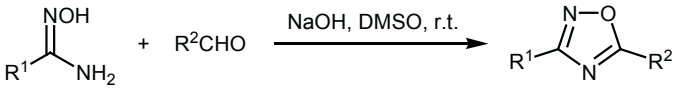
Thus, the optimal conditions included DMSO as solvent, temperature 20–25°C, 1.8 equiv of NaOH, amidoxime-to-aldehyde molar ratio 1:2.2, and reaction time no less than 22 h. These conditions were applied to reactions with other amidoximes **1b**–**1l** and aldehydes **2a**–**2e** to synthesize the corresponding 3,5-disubstituted 1,2,4-oxadiazoles **3b**–**3n** in up to 69% yield (Scheme 2). The substituent in the amidoxime fragment

**Table 1.** Condensation of *N*′-hydroxy-4-(trifluoromethyl)benzimidamide (**1a**) with benzaldehyde (**2a**) in superbasic systems at room temperature

| Entry no. | Solvent | Base (equiv) | Aldehyde (equiv) | Oxidant | Time, h | Yield of **3a**, % |
|---|---|---|---|---|---|---|
| 1 | DMSO | 1.5 | 1.1 | – | 2–48 | 21–24 |
| 2 | DMSO | 1.5 | 1.2 | – | 22 | 23 |
| 3 | DMSO | 1.5 | 1.5 | – | 22 | 28 |
| 4 | DMSO | 1.8 | 1.2 | – | 22 | 45 |
| 5 | DMSO | 1.8 | 1.5 | – | 2–22 | 52 |
| 6 | DMSO[a] | 1.8 | 1.5 | – | 2–22 | 50 |
| **7** | **DMSO** | **1.8** | **2.2** | – | **22** | **76** |
| 8 | DMF | 1.8 | 1.5 | – | 22 | 6 |
| 9 | DMA | 1.8 | 1.5 | – | 22 | 16 |
| 10 | DMSO | 1.8 | 1.1 | $KHSO_5$ (1 equiv) | 22 | 49 |
| 11 | DMSO | 1.8 | 1.1 | NCS[b] (1 equiv) | 22 | 31 |
| 12 | DMSO | 1.8 | 1.1 | $O_2$ | 22 | 52 |
| 13 | DMSO | 1.8 | 1.1 | DCQ[c] (1 equiv) | 22 | 61 |

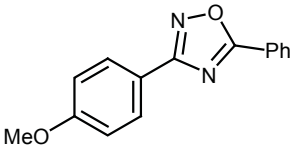
[a] At 100°C.
[b] *N*-Chlorosuccinimide.
[c] Dichlorodicyanoquinone.

**Scheme 2.**

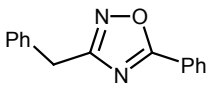



**1b–1l** **2a–2e** **3b–3n**

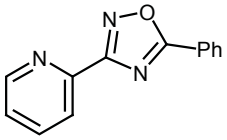
**1**, $R^1$ = 4-$MeOC_6H_4$ (**b**), $PhCH_2$ (**c**), pyridin-2-yl (**d**), 4-$MeC_6H_4$ (**e**), 2-$ClC_6H_4$ (**f**), 4-$FC_6H_4$ (**g**), Ph (**h**), 4-$O_2NC_6H_4$ (**i**), cyclopropyl (**j**), thophen-3-yl (**k**), 5-methylthiophen-2-yl (**l**); **2**, $R^2$ = Ph (**a**), thiophen-3-yl (**b**), furan-2-yl (**c**), 1*H*-pyrrol-2-yl (**d**), 5-bromofuran-2-yl (**e**).



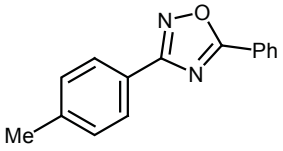

**3b**, 33%



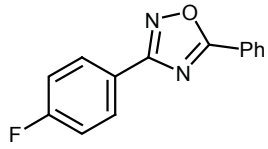

**3c**, 35%



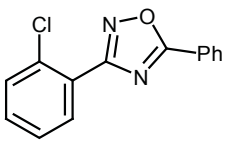

**3d**, 42%



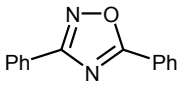

**3e**, 56%



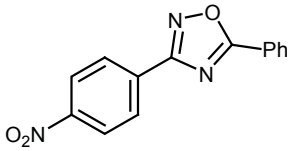

**3f**, 36%



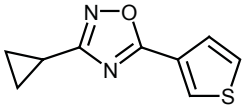

**3g**, 36%



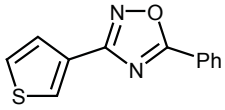

**3h**, 64%



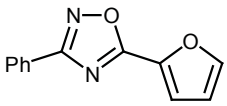

**3i**, 41%



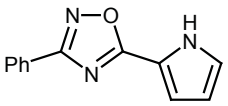

**3j**, 39%



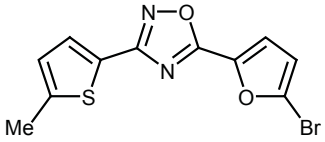

**3k**, 69%




**3l**, 59%




**3m**, 27%




**3n**, 69%

weakly affected the yield of oxadiazoles **3**. Nevertheless, somewhat lower yields were obtained in reactions with amidoximes having an electron-withdrawing substituent in the aromatic ring. Despite generally modest yields, the yields of compounds **3l** and **3m** in the reactions of *N*′-hydroxybenzimidamide (**1h**) with furfural (**2c**) and 1*H*-pyrrole-2-carbaldehyde (**2d**) were higher than those reported in [29, 18].

Attempts to obtain 3,5-disubstituted 1,2,4-oxadiazoles from amidoximes and aliphatic aldehydes (formaldehyde, acetaldehyde, propanal) were unsuccessful, and no intermediate dihydrooxazole was detected in the reaction mixtures by chromatography. Neither the use of dehydrating agents ($Na_2SO_4$, 4-Å molecular sieves) nor elevated temperature gave positive result. It should be noted than in the reaction of *N*′-hydroxy-4-methylbenzimidamide with formaldehyde under the proposed conditions, we isolated 54% of 4-methylbenzonitrile instead of desired oxadiazole **3**.

## EXPERIMENTAL

Unless otherwise stated, organic and inorganic reagents and solvents were commercial products (Aldrich, *Vekton*, *Ekros*) and were used without further purification. The progress of reactions was monitored by TLC on Silufol UV 254 plates using acetone–toluene–*n*-hexane (5:3:5) as eluent.

The NMR spectra were recorded on a Varian XL-400 spectrometer at 25°C from solutions in DMSO-$d_6$ or $CDCl_3$ using the residual proton and carbon signals of the solvent as reference (DMSO-$d_5$, δ 2.50 ppm; DMSO-$d_6$, $\delta_C$ 39.5 ppm). The high-resolution mass spectra (electron impact, 70 eV, ion source temperature 100–220°C) were obtained with a Kratos MS-30 instrument (UK) at the Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences (Moscow). Elemental analyses were carried out with a Perkin Elmer 2400 analyzer. The melting points were measured with a Büchi M-560 apparatus.

Initial amidoximes were synthesized according to the procedure described in [30].

**General procedure for the synthesis of 3,5-disubstituted 1,2,4-oxadiazoles from amidoximes and aldehydes in the superbasic system NaOH/DMSO.** A 10-mL flask was charged with 1 mmol of amidoxime which was dissolved in 1.5 mL of DMSO, and 2.2 mmol of aldehyde **2** and 0.05 mmol (2 mg) of powdered sodium hydroxide were added each in one portion. The mixture was stirred for 1 h, 1.75 mol (70 mg) of powdered sodium hydroxide was added, and the mixture was stirred for 21 h more. After completion of the reaction (TLC), the resulting suspension was diluted with 7 mL of distilled water, and the precipitate was filtered off and washed with 2 mL of water. If a tarry material was formed, the supernatant was separated by decanting, and the residue was washed with three 2–4-mL portions of water. The products were purified by chromatography using acetone–toluene–petroleum ether (5:3:5) as eluent.

**5-Phenyl-3-[4-(trifluoromethyl)phenyl]-1,2,4-oxadiazole (3a)** [31]**.** Yield 221 mg (76%), white powder, mp 93–95°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 7.53–7.77 m (3H), 7.91 d (2H, $J$ = 7.7 Hz), 8.19 m (4H).

**3-(4-Methoxyphenyl)-5-phenyl-1,2,4-oxadiazole (3b)** [32]**.** Yield 83 mg (33%), white powder, mp 92–94°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 3.85 s (3H), 7.14 d (2H, $J$ = 8.4 Hz), 7.60–7.78 m (3H), 8.04–8.06 m (2H), 8.22–8.17 m (2H).

**3-Benzyl-5-phenyl-1,2,4-oxadiazole (3c)** [33]**.** Yield 83 mg (35%), off-white powder, mp 89–91°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 4.11 s (2H), 7.13 d (2H, $J$ = 7.8 Hz), 7.23 d (2H, $J$ = 7.8 Hz), 7.64–7.91 m (5H), 8.06 d (2H, $J$ = 7.4 Hz).

**5-Phenyl-3-(pyridin-2-yl)-1,2,4-oxadiazol (3d)** [16]**.** Yield 94 mg (42%), white powder, mp 129–131°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 7.41–7.73 m (5H), 8.04 s (1H), 8.17 t (3H, $J$ = 8.2 Hz), 8.79 s (1H).

**3-(4-Methylphenyl)-5-phenyl-1,2,4-oxadiazole (3e)** [18]**.** Yield 116 mg (52%), white powder, mp 102–104°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 2.38 s (3H), 7.38 d (2H, $J$ = 8.1 Hz), 7.64 t (2H, $J$ = 7.4 Hz), 7.72 t (1H, $J$ = 7.4 Hz), 7.96 d (2H, $J$ = 8.1 Hz), 8.15 d (2H, $J$ = 7.1 Hz).

**3-(2-Chlorophenyl)-5-phenyl-1,2,4-oxadiazole (3f)** [18]. Yield 93 mg (36%), white powder, mp 87–88°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 7.59–7.62 m (4H), 7.67–7.74 m (2H), 8.08 d (1H, $J$ = 7.6 Hz), 8.08 d (1H, $J$ = 7.6 Hz).

**3-(4-Fluorophenyl)-5-phenyl-1,2,4-oxadiazole (3g)** [33]**.** Yield 86 mg (36%), pinkish white powder, mp 132–134°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 7.46 t (2H, $J$ = 11.4 Hz), 7.63 s (2H), 8.09 d (2H, $J$ = 8.6 Hz), 8.14–8.19 m (2H).

**3,5-Diphenyl-1,2,4-oxadiazole (3h)** [34]**.** Yield 142 mg (64%), white powder, mp 91–93°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 7.64 s (5H), 8.08–8.13 m (4H). $^{13}$C NMR spectrum (101 MHz, DMSO-$d_6$), $\delta_C$, ppm: 126.0, 126.1, 126.8, 127.2, 128.8, 129.4, 131.9, 128.0, 168.5, 174.5.

**3-(4-Nitrophenyl)-5-phenyl-1,2,4-oxadiazole (3i)** [35]**.** Yield 109 mg (41%), yellow crystalline powder, mp 131–133°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 7.83 d (3H, $J$ = 8.0 Hz), 8.12 t (2H, $J$ = 8.4 Hz), 8.43–8.49 m (4H).

**5-Cyclopropyl-3-(thiophen-3-yl)-1,2,4-oxadiazole (3j)** [23]**.** Yield 75 mg (39%), off-white powder, mp 35–37°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 1.20–1.14 m (2H), 1.30–1.23 m (2H), 2.33–2.40 m (1H), 7.54 d.d (1H, $J$ = 5.1, 1.2 Hz), 7.77–7.71 m (1H), 8.24–8.18 m (1H). $^{13}$C NMR spectrum (101 MHz, DMSO-$d_6$), $\delta_C$, ppm: 7.6, 10.4, 126.1, 128.1, 128.8, 128.9, 164.6, 181.9.

**5-Phenyl-3-(thiophen-3-yl)-1,2,4-oxadiazole (3k)** [23]**.** Yield 75 mg (69%), white solid, mp 125–127°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 7.70–7.61 m (3H), 7.73 t (1H, $J$ = 7.4 Hz), 7.81 d.d (1H, $J$ = 5.0, 3.0 Hz), 8.22–8.12 m (2H), 8.37 d.d (1H, $J$ = 2.7, 0.9 Hz).

**5-(Furan-2-yl)-3-phenyl-1,2,4-oxadiazole (3l)** [29]**.** Yield 125 mg (59%), off-white solid, mp 101–102°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 6.88–6.90 m (1H), 7.57–7.65 m (4H), 8.07–8.09 m (2H), 8.18 s (1H).

**3-Phenyl-5-(1*H*-pyrrol-2-yl)-1,2,4-oxadiazole (3m)** [18]**.** Yield 214 mg (27%), off-white powder, mp 131–132°C. $^1$H NMR spectrum (400 MHz, DMSO-$d_6$), δ, ppm: 6.34 t (1H, $J$ = 4.4 Hz), 7.08 d (1H, $J$ = 3.6 Hz), 7.22 s (1H), 7.56–7.59 m (3H), 8.06 d.d (2H, $J$ = 8.0, 4.4 Hz), 12.44 s (1H).

**5-(5-Bromofuran-2-yl)-3-(5-methylthiophen-2-yl)-1,2,4-oxadiazole (3n).** Yield 214 mg (69%), off-white powder, mp 89–90°C. $^1$H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm: 7.65 d (1H, $J$ = 3.5 Hz), 7.28 d.d (1H, $J$ = 3.8, 1.6 Hz), 6.86–6.75 m (1H), 6.57 d.d (1H, $J$ = 3.6, 1.6 Hz). $^{13}$C NMR spectrum

(101 MHz, $CDCl_3$), $\delta_C$, ppm: 15.74, 76.91, 77.23, 77.54, 114.72, 118.96, 125.38, 126.60, 128.48, 130.61, 141.91, 145.13, 165.01, 166.39. Mass spectrum: *m*/*z* 310.9479 $[C_{11}H_7BrN_2O_2S + H]^+$. Calculated: *M* + H 310.9485.

**4-Methylbenzonitrile** [36]**.** Yield 63 mg (54%), light yellow crystals, mp 27–29°C. $^1$H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm: 2.42 s (3H), 7.27 d (2H, *J* = 1.0 Hz), 7.53 d (2H, *J* = 1.0 Hz).

## FUNDING

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## CONFLICT OF INTEREST

The authors declare no conflict of interest.

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