ISSN 1070-4280, Russian Journal of Organic Chemistry, 2019, Vol. 55, No. 7, pp. 978–982. © Pleiades Publishing, Ltd., 2019.
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Heterocyclization of 5-(Arylmethylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-triones with Arenecarbaldehyde Oximes in the Presence of *N*-Bromosuccinimide and Triethylamine

A. G. Tyrkov[a]* and E. A. Yurtaeva[b]**

[a] *Astrakhan State University, Astrakhan, Russia*
**e-mail: tyrkov@rambler.ru*

[b] *Pyatigorsk Medical and Pharmaceutical Institute (Branch), Volgograd State Medical University, Pyatigorsk, Russia*
***e-mail: huitre_88@mail.ru*

Received January 9, 2019; revised March 10, 2019; accepted March 15, 2019

Abstract—5-(Arylmethylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-triones reacted with substituted benzaldehyde oximes in the presence of *N*-bromosuccinimide and triethylamine to give 1,4-diaryl-2-oxa-3,7,9-triazaspiro-[4.5]dec-3-ene-6,8,10-triones and 3,4-diaryl-1,2,5-oxadiazole *N*-oxides.

Keywords: 5-(arylmethylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-triones, arenecarbaldehyde oximes, 1,3-dipolar cycloaddition, *N*-bromosuccinimide, triethylamine, 1,4-diaryl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-triones, 3,4-diaryl-1,2,5-oxadiazole *N*-oxides.

DOI: 10.1134/S1070428019070108

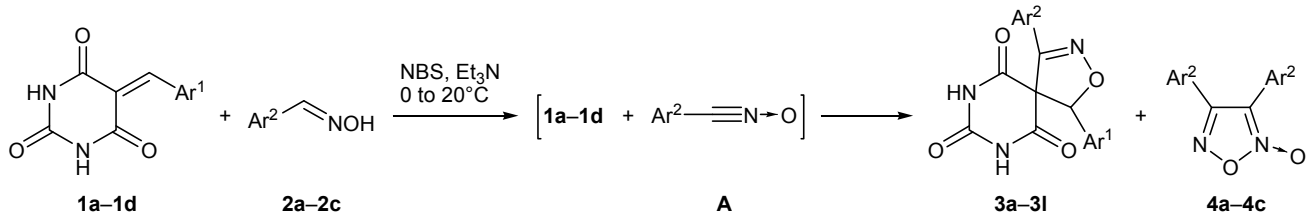
Published data on chemical transformations of benzonitrile oxides generated by dehydrohalogenation of benzohydroximoyl chlorides with triethylamine are limited to 1,3-dipolar cycloadditions to conjugated or unconjugated nitroethenes, which lead to the formation of 3,5-substituted dihydroisoxazoles [1, 2]. Reactions of nitrile oxides with trinitroacetonitrile and ethyl chloro(cyano)nitroacetate afforded 5-nitromethyl-1,2,4-oxadiazole derivatives [3–5]. Heterocyclizations of 5-(arylmethylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-triones with nitrile oxides generated *in situ* from aromatic aldehyde oximes by the action of *N*-bromosuccinimide (NBS) and triethylamine were not studied previously.

In continuation of our studies of the synthetic potential of reactions of nitrile oxides with various dipolarophiles, in this work we examined reactions of substituted benzonitrile oxides with 5-(arylmethylidene)barbituric acids **1a**–**1d**. The reactions of **1a**–**1d** with oximes **2a**–**2c** were carried out in the presence of NBS and triethylamine in DMF at 0–5°C. As a result, we isolated a series of previously unknown 1,4-diaryl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-triones **3a**–**3l** in 35–38% yield. In addition, from the reaction mixtures we isolated products of spontaneous dimerization of intermediate nitrile oxides, 3,4-diaryl-1,2,5-oxadiazole *N*-oxides **4a**–**4c** in 52–55% yield (Scheme 1).

Presumably, oximes **2a**–**2c** react with NBS and triethylamine to produce nitrile oxides **A**, and 1,3-dipolar cycloaddition of the latter to dipolarophiles **1a**–**1d** gives 1,4-diaryl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-triones **3a**–**3l**. The possibility of generation of nitrile oxides by the action of NBS on aromatic aldehyde oximes in the presence of triethylamine was demonstrated previously [6]. The low yields of **3a**–**3l** and the formation of nitrile oxide dimerization products **4a**–**4c** may be rationalized by the low reactivity of barbituric acids **1a**–**1d** in comparison to trinitroacetonitrile or ethyl chloro(cyano)nitroacetate. Presumably, nitrile oxides undergo dimerization independently of the presence of **1a**–**1d** in the reaction mixture.

Compounds **3a**–**3l** are stable colorless or colored high-melting solids soluble in ethanol. The structure of **3a**–**3l** was determined on the basis of their IR, ^{1}H and ^{13}C NMR, and mass spectra and elemental analyses. The IR spectra of **3a**–**3l** showed no C=C stretching band at 1625 cm^{-1}, which was typical of initial pyrimidine-2,4,6-triones **1a**–**1d** [7]. The ^{1}H and ^{13}C NMR spectral parameters of **3a**–**3l** were consistent with the

Scheme 1.



1, Ar^1 = Ph (**a**), 4-$MeOC_6H_4$ (**b**), 4-$Me_2NC_6H_4$ (**c**), 4-MeC_6H_4 (**d**); **2**, Ar^2 = Ph (**a**), 4-$MeOC_6H_4$ (**b**), 4-$Me_2NC_6H_4$ (**c**); **3**, $Ar^1 = Ar^2$ = Ph (**a**), Ar^1 = Ph, Ar^2 = 4-$MeOC_6H_4$ (**b**), 4-$Me_2NC_6H_4$ (**c**); Ar^1 = 4-$MeOC_6H_4$, Ar^2 = Ph (**d**), 4-$MeOC_6H_4$ (**e**), 4-$Me_2NC_6H_4$ (**f**); Ar^1 = 4-$Me_2NC_6H_4$, Ar^2 = Ph (**g**), 4-$MeOC_6H_4$ (**h**), 4-$Me_2NC_6H_4$ (**i**); Ar^1 = 4-MeC_6H_4, Ar^2 = Ph (**j**), 4-$MeOC_6H_4$ (**k**), 4-$Me_2NC_6H_4$ (**l**); **4**, Ar^2 = Ph (**a**), 4-$MeOC_6H_4$ (**b**), 4-$Me_2NC_6H_4$ (**c**).

proposed structure and with the spectra of model compounds of the dihydroisoxazole series [8]. For instance, in the ^{1}H NMR spectra of **3a**–**3l** we observed a signal at δ 5.30–5.38 ppm due to proton in the isoxazole ring, which was absent in the spectra of initial compounds. The ^{13}C NMR spectra displayed a new signal at δ_C 164.2–164.8 ppm due to the C=N carbon atom in the same ring. The molecular ion and $[M - H]^+$ ion peaks in the mass spectra of **3a**–**3l** had low intensity, but ion peaks corresponding to dissociative ionization via retro-1,3-dipolar cycloaddition at the C–O and C–C bonds of the isoxazole ring were distinguished. Apart from the above noted ions, there was a set of ion peaks to which several formulas could be assigned; therefore, more detailed interpretation of the mass spectra was difficult.

Thus, the reaction of 5-(arylmethylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-triones **1a**–**1d** with substituted benzaldehyde oximes **2a**–**2c** in the presence of NBS and triethylamine provides the possibility of incorporating a dihydroisoxazole ring into the base part of molecule **1a**–**1d** in a one-pot fashion. The obtained compounds are interesting as potential biologically active substances, in particular as antibacterial agents or imidazoline receptor agonists [9, 10].

EXPERIMENTAL

The IR spectra in the range 4000–400 cm^{-1} were recorded on an InfraLUM FT-02 spectrometer from samples pelletized with KBr. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avence II 300 SF instrument at 500 and 125 MHz, respectively, using DMSO-d_6 as solvent and hexamethyldisiloxane as internal standard. The mass spectra (electron impact, 70 eV) were obtained on a Finnigan SSQ-7000 mass spectrometer with direct sample admission into the ion source; vaporization temperature 500–550°C. The progress of reactions and the purity of the isolated compounds were monitored by ascending thin-layer chromatography on Silufol UV-254 plates using acetone–hexane (2:3) as eluent; spots were visualized by treatment with iodine vapor [11]. The elemental compositions were determined using a Euro Vector EA-3000 automated CHNS analyzer. The melting (decomposition) points were measured with an OptiMelt melting point apparatus.

5-(Arylmethylidene)pyrimidine-2,4,6-(1*H*,3*H*,5*H*)-triones **1a**–**1d** [12], aromatic aldehyde oximes **2a**–**2c** [13], and NBS [14] were synthesized according to previously described procedures; triethylamine of chemically pure grade was commercial product (Aldrich).

1,4-Diaryl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-triones 3a–3l (*general procedure*). A mixture of 6 mmol of *N*-bromosuccinimide and 6 mmol of aldehyde oxime **2a**–**2c** in 30 mL of anhydrous DMF was vigorously stirred for 30 min at 0°C, a solution of 6.5 mmol of triethylamine in 5 mL of DMF was added, and 5 mmol of compound **1a**–**1d** was then added under vigorous stirring. The mixture was stirred for 30 min at 0–5°C and kept for 24 h at 20–25°C, and the precipitate was filtered off and washed with 10 mL of DMF. The solvent was evaporated under reduced pressure, and the residue was subjected to chromatography in a glass column charged with activated silica (100–400 μm). Compounds **3a**–**3l** were eluted with ethanol, compounds **4a** and **4b**, with benzene, and compound **4c**, with chloroform.

1,4-Diphenyl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3a). Yield 0.586 g (35%), colorless crystals, mp 235–239°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 5.38 s (1H, CH), 7.35–7.58 m (10H, H_{arom}), 11.42 br.s (1H, NH), 11.47 br.s (1H, NH).

^{13}C NMR spectrum, δ_C, ppm: 62.3 (C^5), 69.5 (C^1), 128.1–142.5 (C_{arom}), 152.2 (C^6), 161.3 (C^{10}), 162.5 (C^8), 164.7 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 335 (10) $[M]^{+\cdot}$, 334 (4) $[M - 1]^+$, 216 (100), 119 (20.4). Found, %: C 64.32; H 3.76; N 12.36. $C_{18}H_{13}N_3O_4$. Calculated, %: C 64.47; H 3.91; N 12.53. *M* 335.31.

4-(4-Methoxyphenyl)-1-phenyl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3b). Yield 0.676 g (37%), pale yellow crystals, mp 245–248°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.82 s (3H, CH_3O), 5.37 s (1H, CH), 6.90–7.52 m (9H, H_{arom}), 11.43 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 55.6 (CH_3O), 62.4 (C^5), 68.4 (C^1), 114.4–158.5 (C_{arom}), 161.5 (C^6), 161.9 (C^{10}), 162.7 (C^8), 164.2 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 365 (8) $[M]^{+\cdot}$, 364 (5) $[M - 1]^+$, 216 (100), 149 (25.2). Found, %: C 62.31; H 3.97; N 11.32. $C_{19}H_{15}N_3O_5$. Calculated, %: C 62.46; H 4.14; N 11.50. *M* 365.34.

4-(4-Dimethylaminophenyl)-1-phenyl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3c). Yield 0.719 g (38%), red crystals, mp 310–314°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 2.43 s (6H, CH_3N), 5.37 s (1H, CH), 6.95–7.55 m (9H, H_{arom}), 11.43 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 40.7 (CH_3N), 62.5 (C^5), 68.3 (C^1), 112.6–154.4 (C_{arom}), 161.4 (C^6), 161.6 (C^{10}), 162.5 (C^8), 164.6 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 378 (9) $[M]^{+\cdot}$, 377 (6) $[M - 1]^+$, 216 (100), 162 (26.5). Found, %: C 63.32; H 4.67; N 14.62. $C_{20}H_{18}N_4O_4$. Calculated, %: C 63.48; H 4.79; N 14.81. *M* 378.38.

1-(4-Methoxyphenyl)-4-phenyl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3d). Yield 0.657 g (36%), pale yellow crystals, mp 215–219°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.81 s (3H, CH_3O), 5.36s (1H, CH), 6.90–7.55 m (9H, H_{arom}), 11.43 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 55.8 (CH_3O), 61.7 (C^5), 68.2 (C^1), 114.5–160.2 (C_{arom}), 161.3 (C^6), 161.7 (C^{10}), 162.2 (C^8), 164.5 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 365 (11) $[M]^{+\cdot}$, 364 (6) $[M - 1]^+$, 246 (100), 119 (20.6). Found, %: C 62.28; H 3.96; N 11.33. $C_{19}H_{15}N_3O_5$. Calculated, %: C 62.46; H 4.14; N 11.50. *M* 365.34.

1,4-Bis(4-methoxyphenyl)-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3e). Yield 0.751 g (38%), pale yellow crystals, mp 282–286°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 3.80 s (3H, CH_3O), 3.81 s (3H, CH_3O), 5.37 s (1H, CH), 6.90–7.52 m (8H, H_{arom}), 11.43 br.s (1H, NH), 11.46 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 55.3 (CH_3O), 55.5 (CH_3O), 61.6 (C^5), 61.8 (C^1), 113.8–158.3 (C_{arom}), 161.4 (C^6), 161.8 (C^{10}), 162.3 (C^8), 164.1 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 395 (8) $[M]^{+\cdot}$, 394 (5) $[M - 1]^+$, 246 (100), 149 (26.5). Found, %: C 60.58; H 4.17; N 10.48. $C_{20}H_{17}N_3O_6$. Calculated, %: C 60.76; H 4.33; N 10.63. *M* 395.37.

4-(4-Dimethylaminophenyl)-1-(4-methoxyphenyl)-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3f). Yield 0.755 g (37%), pale yellow crystals, mp 305–309°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 2.42 s (6H, CH_3N), 3.80 s (3H, CH_3O), 5.34 s (1H, CH), 6.95–7.55 m (8H, H_{arom}), 11.42 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 40.5 (CH_3N), 55.4 (CH_3O), 62.6 (C^5), 68.1 (C^1), 112.8–155.2 (C_{arom}), 161.5 (C^6), 161.8 (C^{10}), 162.8 (C^8), 164.5 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 408 (10) $[M]^{+\cdot}$, 407 (5), $[M - 1]^+$, 246 (100), 162 (25.3). Found, %: C 61.60; H 4.77; N 13.54. $C_{21}H_{20}N_4O_5$. Calculated, %: C 61.76; H 4.94; N 13.72. *M* 408.41.

1-(4-Dimethylaminophenyl)-4-phenyl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3g). Yield 0.663 g (35%), pink crystals, mp 280–285°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 2.41 s (6H, CH_3N), 5.37 s (1H, CH), 6.95–7.58 m (9H, H_{arom}), 11.42 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 40.7 (CH_3N), 62.7 (C^5), 68.3 (C^1), 112.5–154.7 (C_{arom}), 161.5 (C^6), 161.7 (C^{10}), 162.6 (C^8), 164.6 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 378 (12) $[M]^{+\cdot}$, 377 (8), $[M - 1]^+$, 259 (100), 119 (20.5). Found, %: C 63.32; H 4.61; N 14.62. $C_{20}H_{18}N_4O_4$. Calculated, %: C 63.48; H 4.79; N 14.81. *M* 378.13.

1-(4-Dimethylaminophenyl)-4-(4-methoxyphenyl)-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3h). Yield 0.779 g (38%), pink crystals, mp 320–325°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 2.40 s (6H, CH_3N), 3.80 s (3H, CH_3O), 5.30 s (1H, CH), 6.90–7.54 m (8H, H_{arom}), 11.42 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 40.4 (CH_3N), 55.3 (CH_3O), 62.5 (C^5), 68.1 (C^1), 112.7–155.4 (C_{arom}), 161.4 (C^6), 161.7 (C^{10}), 162.8 (C^8), 164.5 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 408 (10) $[M]^{+\cdot}$, 407 (5), $[M - 1]^+$, 259 (100), 149 (25.2). Found, %: C 61.58; H 4.81; N 13.54. $C_{21}H_{20}N_4O_5$. Calculated, %: C 61.76; H 4.94; N 13.72. *M* 408.41.

1,4-Bis(4-dimethylaminophenyl)-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3i). Yield 0.782 g (37%), red crystals, mp 272–277°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^1H NMR spectrum, δ, ppm: 2.40 s (6H, CH_3N), 2.41 s (6H, CH_3N), 5.33 s (1H, CH), 6.95–7.62 m (8H, H_{arom}), 11.43 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 40.5 and 40.6 (CH_3N), 62.3 (C^5), 68.2 (C^1), 112.9–154.5 (C_{arom}), 161.3 (C^{10}), 161.4 (C^6), 162.5 (C^8), 164.5 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 421 (11) $[M]^{+\cdot}$, 420 (6), $[M - 1]^+$, 259 (100), 162 (24.5). Found, %: C 62.52; H 5.34; N 16.47. $C_{22}H_{23}N_5O_4$. Calculated, %: C 62.70; H 5.50; N 16.62. *M* 421.45.

1-(4-Methylphenyl)-4-phenyl-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3j). Yield 0.663 g (38%), colorless crystals, mp 255–260°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^1H NMR spectrum, δ, ppm: 2.32 s (3H, CH_3), 5.35 s (1H, CH), 7.05–7.55 m (9H, H_{arom}), 11.42 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 21.6 (CH_3), 62.4 (C^5), 68.6 (C^1), 125.6–137.3 (C_{arom}), 161.4 (C^6), 161.7 (C^{10}), 162.5 (C^8), 164.8 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 349 (10) $[M]^{+\cdot}$, 348 (5), $[M - 1]^+$, 230 (100), 119 (18.5). Found, %: C 65.14; H 4.17; N 11.85. $C_{19}H_{15}N_3O_4$. Calculated, %: C 65.32; H 4.33; N 12.03. *M* 349.34.

4-(4-Methoxyphenyl)-1-(4-methylphenyl)-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3k). Yield 0.663 g (35%), colorless crystals, mp 265–270°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^1H NMR spectrum, δ, ppm: 2.31 s (3H, CH_3), 3.80 s (3H, CH_3O), 5.31 s (1H, CH), 6.92–7.52 m (8H, H_{arom}), 11.42 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 21.5 (CH_3), 55.4 (CH_3O), 62.4 (C^5), 68.2 (C^1), 112.6–155.3 (C_{arom}), 161.2 (C^6), 161.5 (C^{10}), 162.6 (C^8), 164.6 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 379 (10) $[M]^{+\cdot}$, 378 (10), $[M - 1]^+$, 230 (100), 149 (25.5). Found, %: C 63.13; H 4.37; N 10.91. $C_{20}H_{17}N_3O_5$. Calculated, %: C 63.32; H 4.52; N 11.08. *M* 379.37.

4-(4-Dimethylaminophenyl)-1-(4-methylphenyl)-2-oxa-3,7,9-triazaspiro[4.5]dec-3-ene-6,8,10-trione (3l). Yield 0.725 g (37%), colorless crystals, mp 301–307°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 (NH), 1770, 1750 (C=O). ^1H NMR spectrum, δ, ppm: 2.32 s (3H, CH_3), 2.42 s (6H, CH_3N), 5.32 s (1H, CH), 6.90–7.55 m (8H, H_{arom}), 11.42 br.s (1H, NH), 11.48 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 21.4 (CH_3), 40.5 (CH_3N), 62.5 (C^5), 68.3 (C^1), 112.8–155.2 (C_{arom}), 161.4 (C^6), 161.6 (C^{10}), 162.7 (C^8), 164.6 (C^4). Mass spectrum, *m*/*z* (I_{rel}, %): 392 (12) $[M]^{+\cdot}$, 391 (10), $[M - 1]^+$, 230 (100), 162 (22.4). Found, %: C 64.09; H 4.95; N 14.11. $C_{21}H_{20}N_4O_4$. Calculated, %: C 64.28; H 5.14; N 14.28. *M* 392.41.

3,4-Diphenyl-1,2,5-oxadiazole *N*-oxide (4a). Yield 0.747 g (52%), colorless crystals, mp 114°C [15].

3,4-Bis(4-methoxyphenyl)-1,2,5-oxadiazole *N*-oxide (4b). Yield 0.969 g (54%), colorless crystals, mp 113°C [15].

3,4-Bis(4-dimethylaminophenyl)-1,2,5-oxadiazole *N*-oxide (4c). Yield 1.065 g (55%), colorless crystals, mp 123°C [15].

FUNDING

This study was performed under financial support by the Ministry of Science and Higher Education of the Russian Federation (project no. 4.9288.2017BCh).

CONFLICT OF INTERESTS

The authors declare the absence of conflict of interests.

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