ISSN 1070-3632, Russian Journal of General Chemistry, 2014, Vol. 84, No. 8, pp. 1519–1523. © Pleiades Publishing, Ltd., 2014.
Original Russian Text © L.V. Lapshina, A.V. Serebryannikova, I.E. Efremova, A.D. Perkhunova, S.V. Bortnikov, V.M. Berestovitskaya, 2014, published in Zhurnal Obshchei Khimii, 2014, Vol. 84, No. 8, pp. 1293–1298.

Reactions of Aroylhydrazines with Nitrosulfodienes of the 2-Benzylidene-3-methyl-4-nitro-3-thiolene-1,1-dioxide Series

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Received March 17, 2014

Abstract—Reactions of *s-trans*-nitrosulfodienes of the 2-benzylidene-3-methyl-4-nitro-3-thiolene-1,1-dioxide series with aroylhydrazines occur as nucleophilic 1,4-addition to give the aza-Michael adducts. Activity of the studied substrates depends on the nature of the substituents in the benzene ring of benzylidene fragment. Previously unexplored representatives of the 2-benzylidene-3-methyl-4-nitro-3-thiolene-1,1-dioxides have been synthesized.

Keywords: nitrosulfolene, nitrosulfodiene, aroylhydrazine, aza-Michael reaction

DOI: 10.1134/S1070363214080131

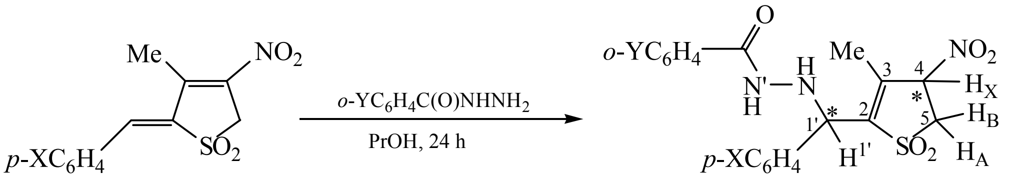
Heterocyclic sulfonitroalkenes of the nitrothiolene-1,1-dioxide series are reactive synthons for preparation of heterocyclic and diene derivatives [1–4], they can serve as models for certain theoretical studies, for example, those of oxime-nitrone tautomerism [2, 3], prototropic and halotropic rearrangement [5–7], and heteroring opening reactions [8].

Combination of the nitrosulfolene and conjugated exocyclic benzylidene moieties opens ways to preparation of peculiar *s-trans*-nitrosulfodienes such as the 2-benzylidene-4-nitro-3-thiolene-1,1-dioxides [9]. Conjugated system activated by two electron-withdrawing substituents (SO_2 and NO_2) enhances their reactivity in the reaction with *C*-nucleophiles resulting in 1,4-adducts [10]. The literature data concerning the formation of aza-Michael adducts in the reactions of the benzylidenenitrothiolene-1,1-dioxides with *N*-nucleophiles are scarce [11–13]. Moreover, the literature lacks data on reactions of the benzylidene-nitrothiolene-1,1-dioxides with hydrazine and its derivatives (aroylhydrazines, phenylhydrazine, and semicarbazide). However, it has been shown that the compounds resulting from aroylhydrazines addition at the double bond of 3-methyl-4-nitro-3-thiolene-1,1-dioxide possess the unusual ability to rupture the C^3–C^4 bond of the sulfolane ring leading to the previously unknown linear (2-nitroethylsulfonyl)propanone hydrazones [8]. In this regard, examination of the chemical behavior of 2-benzylidene-4-nitro-3-thiolene-1,1-dioxides **I**–**VI** (differing in by the nature of the substituents at the aromatic ring of benzylidene moiety) in the reactions with aroylhydrazines is a topical task.

Compounds **III** and **VI** were obtained for the first time taking advantage of the procedure to prepare nitrosulfodienes **I** and **II** [9], via condensation of 3-methyl-4-nitro-3-thiolene-1,1-dioxide with *p*-bromo- and *p*-hydroxybenzaldehyde, respectively. The synthesis was performed by refluxing the reactants mixture in glacial acetic acid in the presence of ammonium chloride. Spectral features of **III** and **VI** were similar to those previously described for 2-benzylidene-4-nitro-3-thiolene-1,1-dioxides **I** and **V** [9].

Reactions of nitrosulfodienes **I**–**VI** with aroylhydrazines proceeded in propanol at room temperature within 24 h to afford 2-(1'-aroylhydrazino-1'-aryl)-methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxides **VII**–**XXI** with yields of 55–95%. The reaction efficiency depended on the nature of the substituent in the benzene ring of the parent benzylidenenitrothiolene-1,1-dioxide (Scheme 1).

Scheme 1.



I–VI **VII–XXI**

X = H (**I**), Cl (**II**), Br (**III**), NO_2 (**IV**), Me (**V**), OH (**VI**); X = H: Y = H (**VII**), Cl (**VIIIa**, **VIIIb**), OH (**IX**); X = Cl: Y = H (**X**), Cl (**XI**), OH (**XII**); X = Br: Y = H (**XIII**), Cl (**XIVa**, **XIVb**), OH (**XV**); X = NO_2: Y = H (**XVIa**, **XVIb**), Cl (**XVIIa**, **XVIIb**), OH (**XVIII**); X = Me: Y = H (**XIXa**, **XIXb**), Cl (**XX**), OH (**XXIa**, **XXIb**).

The highest yield (95%) was achieved in the case of the most electron-deficient substrate **IV**, with the nitrodiene system being additionally activated by nitrophenyl substituent. 1.5-fold excess of the reagent was required to prepare adducts **XIX–XXI** derived from *p*-tolyl-substituted diene **V**. Compound **VI** containing the electron-donor substituent (OH) in the aromatic ring did not react with the hydrazides irrespectively of the widely varied reaction conditions (2-fold excess of the reagent, solvent change, heating).

The aza-adducts obtained (**VII–XXI**) were colorless crystalline substances. They were stable at room temperature, but underwent the retro-reaction to form the parent nitrosulfodienes when crystallized from ethanol. Structures of compounds **VII–XXI** were confirmed by spectral methods; their composition was confirmed by elemental analysis data.

IR spectra of the adducts **VII–XXI** (Table 1) contained absorption bands of non-conjugated nitro (1558–1574 and 1350–1360 cm^{-1}) and sulfonyl groups (1293–1308 and 1133–1144 cm^{-1}) vibrations. Absorbance band at 3247–3423 cm^{-1} was assigned to the NH groups stretching. The amide groups vibrations gave rise to absorption bands in the ranges of 1639–1672 and 1510–1594 cm^{-1}. Absorption bands at 1589–1604 cm^{-1} were due to stretching of the C=C bonds of the aromatic and sulfolene rings [14].

Table 1. Melting point, yield and IR spectral data of 2-(1'-aroylhydrazino-1'-aryl)methyl-3-methyl-4-nitro-3-thiolene-1,1-dioxides **VII–XXI**

Comp. no.	Yield, %	mp, °C (**a** : **b**)	IR spectrum (KBr), ν, cm^{-1}				
			NO_2	SO_2	NH, OH	C(O)NH	C=C, Ar
VII	82	122–124	1564, 1356	1302, 1139	3397, 3264	1656, 1518	1601
VIIIa, VIIIb	81	126–128 (1 : 2)	1560, 1360	1303, 1142	3376, 3402	1658, 1539	1593
IX	85	137–140	1565, 1355	1308, 1139	3400, 3281	1639, 1520	1600
X	77	162–164	1563, 1356	1304, 1139	3413, 3256	1652, 1525	1600
XI	70	138–140	1563, 1357	1308, 1144	3247, 3277	1668, 1594	1594
XII	69	158–160	1564, 1358	1304, 1135	3419, 3309	1641, 1510	1600
XIII	83	158–161	1563, 1356	1305, 1139	3402, 3256	1652, 1523	1600
XIVa, XIVb	57	151–154 (1 : 2)	1574, 1359	1306, 1142	3410, 3351	1672, 1589	1589
XV	70	160–162	1564, 1358	1303, 1144	3422, 3313	1641, 1600	1600
XVIa, XVIb	95	177–179 (1 : 1)	1562, 1352	1308, 1133	3428, 3413	1660, 1525	1600
XVIIa, XVIIb	90	161–163 (2 : 3)	1566, 1350	1306, 1141	3403, 3269	1672, 1523	1593
XVIII	50	175–178	1564, 1353	1300, 1133	3423, 3307	1644, 1525	1600
XIXa, XIXb	80	143–146 (2 : 1)	1562, 1356	1293, 1139	3403, 3260	1655, 1516	1600
XX	95	121–123	1560, 1358	1307, 1144	3409, 3255	1670, 1512	1594
XXIa, XXIb	66	155–157 (2 : 1)	1558, 1354	1312, 1143	3391, 3324	1619, 1512	1604

Table 2. ^{1}H NMR spectral data of 2-(1'-aroylhydrazino-1'-aryl)methyl-3-methyl-4-nitro-3-thiolene-1,1-dioxides **VII**–**XXI**

Comp. no.	δ, ppm (*J*, Hz)						
	CH_3	C^5H_2	C^4H	$C^{1'}H$	NH	N'H	Ar
VII	1.94 s	3.72 d.d, 3.91 d.d	5.62 d.d	5.40 s	5.23 d	8.50 d	7.40–7.45 m, 7.59–7.71 m
		$^2J_{AB}$ 14.95, $^3J_{AX}$ 8.54, $^3J_{BX}$ 2.14			$^3J_{HNN'H}$ 6.10		
VIIIa	1.98 s	3.68–3.75 m, 3.87–3.92 m	5.61 m	5.47 s	5.24 m	8.27 m	7.32–7.48 m, 7.58–7.63 m
VIIIb	2.00 s		5.69 m	5.44 s			
IX	1.93 s	3.73 d.d, 3.92 d.d	5.62 d.d	5.38 s	5.28 d	8.85 d	6.86–6.93 m, 7.34–7.60 m, 11.65 (OH)
		$^2J_{AB}$ 14.95, $^3J_{AX}$ 8.54, $^3J_{BX}$ 2.14			$^3J_{HNN'H}$ 6.10		
X	1.94 s	3.72 d.d, 3.91 d.d	5.61 d.d	5.41 s	5.26 d	8.49 d	7.40–7.71 m
		$^2J_{AB}$ 14.95, $^3J_{AX}$ 8.54, $^3J_{BX}$ 1.83			$^3J_{HNN'H}$ 6.10		
XI	1.98 s	3.72 d.d, 3.90 d.d	5.61 d.d	5.48 s	5.27 d	8.24 d	7.32–7.62 m
		$^2J_{AB}$ 15.26, $^3J_{AX}$ 8.24, $^3J_{BX}$ 2.14			$^3J_{HNN'H}$ 6.10		
XII	1.93 s	3.74 d.d, 3.90 d.d	5.65 d.d	5.36 s	5.32 d	8.85 d	6.85–6.93 m, 7.38–7.58 m, 11.60 (OH)
		$^2J_{AB}$ 14.95, $^3J_{AX}$ 8.24, $^3J_{BX}$ 2.14					
XIII	1.91 s	3.72 d.d, 3.91 d.d	5.61 d.d	5.39 s	5.30 d	8.50 d	7.42–7.70 m
		$^2J_{AB}$ 14.95, $^3J_{AX}$ 8.54, $^3J_{BX}$ 1.83					
XIVa	1.98 s	3.66–3.74 m, 3.86–3.91 m	5.61 m	5.46 s	5.28 m	8.26 m	7.32–7.57 m
XIVb	2.00 s		5.67 m	5.42 s			
XV	1.91 s	3.74 d.d, 3.90 d.d	5.65 d.d	5.31–5.35 m		8.84 d	6.85–6.93 m, 7.38–7.58 m, 11.60 (OH)
		$^2J_{AB}$ 15.26, $^3J_{AX}$ 8.24, $^3J_{BX}$ 2.44					
XVIa	1.93 s	3.63–3.71 m, 3.83–3.89 m	5.60 m	5.53 s	5.35 m	8.45 d	7.36–7.80 m, 8.16–8.18 m
XVIb	1.94 s			5.48 s		8.49 d	
XVIIa	2.03 s	3.69–3.75 m, 3.88–3.92 m	5.70 m	5.62 s	5.40 d	8.30 m	7.41–7.43 m
XVIIb	2.05 s			5.64 s	5.42 d		
XVIII	1.96 s	3.73 d.d, 3.90 d.d	5.67 d.d	5.52 m		8.86 d	6.84–6.93 m, 7.38–7.47 m, 7.83 d, 8.23 d, 11.55 (OH)
		$^2J_{AB}$ 15.26, $^3J_{AX}$ 8.24, $^3J_{BX}$ 2.44					
XIXa	1.93 s	3.86–3.75 m, 3.88–3.93 m	5.61 m	5.36 s	5.15–5.19 m	8.48 d	7.20–7.71 m, 2.32 (CH_3)
XIXb	1.95 s	3.70–3.76 m, 3.86–3.91 m	5.66 m	5.32 s		8.51 d	
XX	1.91 s	3.72 d.d, 3.88 d.d	5.61 d.d	5.42 s	5.21 s	8.23 s	7.21 d, 7.50 d, 7.32–7.46 m, 2.32 (CH_3)
XXIa	1.91 s	3.69–3.76 m, 3.86–3.93 m	5.61 m	5.34 s	5.22 m	8.83 m	6.85–6.93 m, 7.23–7.48 m, 2.32 (CH_3), 11.65(OH)
XXIb			5.65 m	5.32 s			

According to ^{1}H NMR data, 2-(1'-aroylhydrazino-1'-aryl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxides **VII**, **IX**–**XIII**, **XV**, **XVIII**, **XX** were stereochemically pure; the products **VIII**, **XIV**, **XVI**, **XVII**, **XIX**, **XXI** were mixtures of diastereomers. The stereoisomers with the upfield-shifted signal of the methyl protons and the downfield-shifted signal of the methine proton ($C^{1'}H$) were indicated with "**a**," other isomers being marked with "**b**." ^{1}H NMR spectra of **VII**–**XXI** were similar (Table 2). In particular, the spectrum of **VII** contained the following signals of the heterocycle fragment: singlet of the methyl protons (1.94 ppm), doublets of doublets of the ABX system of the nitromethine proton (5.62 ppm, $^3J_{AX}$ 8.54, $^3J_{BX}$ 2.14 Hz), and methylene protons (3.72, 3.91 ppm, $^2J_{AB}$ 14.95 Hz). Exocyclic methine proton manifested as singlet at 5.40 ppm. The doublets at 5.23 and 8.50 ppm ($^3J_{NHN'H}$ 6.10 Hz) were assigned to hydrazine moiety. The aromatic protons resonated as multiplets in the ranges of 7.40–7.45 and 7.59–7.71 ppm.

In summary, 2-benzylidene-4-nitro-3-thiolene-1,1-dioxides were found to be reactive Michael acceptors in the reaction with aroylhydrazines; the ability of studied substrates to undergo nucleophilic addition and the required reaction conditions depend substantially on the nature of the substituent at benzene ring of the benzylidene group.

EXPERIMENTAL

^{1}H NMR spectra of the solutions in CD_3CN were recorded with the Jeol ECX400A spectrometer operating at 399.78 MHz relative to the signal of residual solvent protons. IR spectra were registered with the Shimadzu IRPrestige-21 Fourier-spectrometer (KBr). Electronic absorption spectra were recorded with the Shimadzu UV-2401 PC spectrophotometer in acetic acid using quartz cell (l = 0.1 cm, $c \approx 1.0 \times 10^{-3}$ mol L^{-1}). Elemental analysis was performed using the EuroVector EA 3000 analyzer (CHN Dual mode).

2-Benzylidene-3-methyl-4-nitro-3-thiolene-1,1-dioxides I, **IV** and **V** were prepared as described elsewhere [9]; **2-*p*-chlorobenzylidene-3-methyl-4-nitro-3-thiolene-1,1-dioxide II** was obtained according to [15]. Their analogs **III** and **VI** were obtained and characterized for the first time in the present study.

2-*p*-Bromobenzilidene-3-methyl-4-nitro-3-thiolene (III). A mixture of 4 g (28 mmol) of *p*-bromobenzaldehyde and 0.05 g (1 mmol) of ammonium chloride was added to a suspension of 4 g (23 mmol) of 3-methyl-4-nitro-3-thiolene-1,1-dioxide in 5 mL of acetic acid. The reaction mixture was refluxed during 3 h. After cooling, the precipitate was filtered off and washed with diethyl ether. Yield 1.00 g (13%), mp 195–197°C (ethanol). IR spectrum, ν, cm^{-1} (KBr): 1308 br, 1121 (SO_2, NO_2^-), 1591 (C=C). Electronic spectrum (AcOH), λ_{max}, nm (ε): 240 (7900), 359 (26500). ^{1}H NMR spectrum, δ, ppm (CD_3CN): 2.49 t (3H, CH_3), 4.38 q (2H, CH_2), 7.51 s (1H, =CH), 7.62 d and 7.79 d (4H, C_6H_4). Found, %: C 41.80; H 2.89; N 3.73. $C_{12}H_{10}BrNO_4S$. Calculated, %: C 41.88; H 2.93; N 4.07.

2-*p*-Hydroxybenzylidene-3-methyl-4-nitro-3-thiolene-1,1-dioxide (VI) was obtained similarly. Yield 23%, mp 227–230°C (ethanol). IR spectrum, ν, cm^{-1} (KBr): 1320, 1294, 1114 (SO_2, NO_2^-), 1595 (C=C). Electronic spectrum (AcOH), λ_{max}, nm (ε): 258 (7300), 402 (35100). ^{1}H NMR spectrum, δ, ppm (CD_3CN): 2.49 t (3H, CH_3), 4.38 q (2H, CH_2), 7.44 s (1H, =CH), 6.90 d and 7.85 d (4H, C_6H_4), 7.79 (1H, OH). Found N, %: 4.76. $C_{12}H_{11}NO_5S$. Calculated N, %: 4.98.

2-(1'-Benzoylhydrazino-1'-phenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (VII). 0.13 g (1 mmol) of benzoylhydrazine was added to a suspension of 0.26 g (1 mmol) of **I** in 7 mL of propanol. The reaction mixture was stirred at room temperature during 1 day. Colorless precipitate was filtered off, washed with propanol, and dried in air. Yield 0.33 g (82%), mp 122–124°C (ethanol). Found, %: C 56.38; H 4.52; N 10.50. $C_{19}H_{19}N_3O_5S$. Calculated, %: C 56.85; H 4.77; N 10.47.

2-(1'-*o*-Chlorobenzoylhydrazino-1'-phenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (VIIIa, VIIIb) was obtained similarly. Yield 81%, mixture of diastereomers (**a** : **b** = 1 : 2), mp 126–128°C (ethanol). Found, %: C 52.49; H 4.25; N 9.54. $C_{19}H_{18}ClN_3O_5S$. Calculated, %: C 52.35; H 4.16; N 9.64.

2-(1'-*o*-Hydroxybenzoylhydrazino-1'-phenyl)-methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (IX) was obtained similarly. Yield 85%, mp 137–140°C (ethanol). Found, %: C 54.58; H 4.58; N 9.98. $C_{19}H_{19}\cdot N_3O_6S$. Calculated, %: C 54.67; H 4.59; N 10.07.

2-(1'-Benzoylhydrazino-1'-*p*-chlorophenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (X) was obtained similarly. Yield 77%, mp 162–164°C (ethanol). Found N, %: 9.46. $C_{19}H_{18}ClN_3O_5S$. Calculated N, %: 9.64.

2-(1'-*o*-Chlorobenzoylhydrazino-1'-*p*-chlorophenyl)-methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XI) was obtained similarly. Yield 70%, mp 138–140°C (ethanol). Found N, %: 8.52. $C_{19}H_{17}Cl_2N_3O_5S$. Calculated N, %: 8.93.

2-(1'-*o*-Hydroxybenzoylhydrazino-1'-*p*-chlorophenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XII) was obtained similarly. Yield 69%, mp 158–160°C (ethanol). Found N, %: 8.95. $C_{19}H_{18}ClN_3O_6S$. Calculated N, %: 9.30.

2-(1'-Benzoylhydrazino-1'-*p*-bromophenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XIII) was obtained similarly. Yield 83%, mp 158–161°C (ethanol). Found N, %: 8.75. $C_{19}H_{18}BrN_3O_5S$. Calculated N, %: 8.75.

2-(1'-*o*-Chlorobenzoylhydrazino-1'-*p*-bromophenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XIVa, XIVb) was obtained similarly. Yield 57%, mixture of diastereomers (**a** : **b** = 1 : 2), mp 151–154°C (ethanol). Found N, %: 8.01. $C_{19}H_{17}BrClN_3O_5S$. Calculated N, %: 8.16.

2-(1'-*o*-Hydroxybenzoylhydrazino-1'-*p*-bromophenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XV) was obtained similarly. Yield 70%, mp 160–162°C (ethanol). Found, %: C 46.46; H 3.84; N 8.92. $C_{19}H_{18}Br\cdot N_3O_6S$. Calculated, %: C 45.98; H 3.66; N 8.47.

2-(1'-Benzoylhydrazino-1'-*p*-nitrophenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XVIa, XVIb) was obtained similarly. Yield 95%, mixture of diastereomers (**a** : **b** = 1 : 1), mp 177–179°C (ethanol). Found N, %: 12.34. $C_{19}H_{18}N_4O_7S$. Calculated N, %: 12.55.

2-(1'-*o*-Chlorobenzoylhydrazino-1'-*p*-nitrophenyl)-methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XVIIa, XVIIb) was obtained similarly. Yield 90%, mixture of diastereomers (**a** : **b** = 2 : 3), mp 161–163°C (ethanol). Found N, %: 11.44. $C_{19}H_{17}ClN_4O_7S$. Calculated N, %: 11.65.

2-(1'-*o*-Hydroxybenzoylhydrazino-1'-*p*-nitrophenyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XVIII) was obtained similarly. Yield 50%, mp 175–178°C (ethanol). Found, %: C 49.85; H 4.00; N 12.18. $C_{19}H_{18}N_4O_8S$. Calculated, %: C 49.35; H 3.92; N 12.12.

2-(1'-Benzoylhydrazino-1'-*p*-tolyl)methyl-3-methyl-4-nitro-3-thiolene-1,1-dioxide (XIXa, XIXb). 0.10 g (0.75 mmol) of benzoylhydrazine was added to a suspension of 0.14 g (0.5 mmol) of **V** in 7 mL of propanol. The reaction mixture was stirred at room temperature during 1 day. Colorless precipitate was filtered off, washed with propanol, and dried in air. Yield 0.16 g (80%), mixture of diastereomers (**a** : **b** = 2 : 1), mp 143–146°C (ethanol). Found N, %: 9.72. $C_{20}H_{21}N_3O_5S$. Calculated N, %: 10.11.

2-(1'-*o*-Chlorobenzoylhydrazino-1'-*p*-tolyl)methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XX) was obtained similarly. Yield 95%, mp 121–123°C (ethanol). Found N, %: 9.54. $C_{20}H_{20}ClN_3O_5S$. Calculated N, %: 9.34.

2-(1'-*o*-Hydroxybenzoylhydrazino-1'-*p*-tolyl)-methyl-3-methyl-4-nitro-2-thiolene-1,1-dioxide (XXIa, XXIb) was obtained similarly. Yield 66%, mixture of diastereomers (**a** : **b** = 2 : 1), mp 155–157°C (ethanol). Found N, %: 10.03. $C_{20}H_{21}N_3O_6S$. Calculated N, %: 9.74.

Physico-chemical studies were performed at the Center for Collective Use in Herzen State Pedagogical University of Russia.

ACKNOWLEDGMENTS

This work was financially supported by the Ministry of Education of Russia in the frame of the basic part of the governmental task.

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