Nitrosylsulfuric acid as a tandem reagent in the synthesis of 3,5-diarylisoxazoles from 1,2-diarylcyclopropanes

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It was demonstrated that nitrosylsulfuric acid can be successfully used as a tandem nitrosating and oxidizing agent in the synthesis of 3,5-diarylisoxazoles from 1,2-diarylcyclopropanes. The reaction proceeds highly regioselectively in the case of symmetric diarylcyclopropanes. The mixtures of regioisomeric 3,5-diarylisoxazoles were obtained from non-symmetric 1,2-diarylcyclopropanes.

Key words: 3,5-diarylisoxazoles, 1,2-diarylcyclopropanes, nitrosylsulfuric acid, nitrosation, oxidation.

Representatives of the isoxazole and isoxazoline classes, connected by a single step of oxidative transformation, are known for their anti-inflammatory, antifungal, and analgesic properties. They are also effective against HIV and Alzheimer's disease.[1,2] According to recent studies, the 3,5-diarylisoxazole fragment is a convenient structural unit in the synthesis of compounds with potential biological activity. This fragment is found in synthetic analogs of combretastatin A4,[3,4] and is used as bioisosteres in drugs applied to treat and prevent leishmaniasis and Chagas disease.[5] Due to their rigid structure, derivatives of 3,5-diphenylisoxazole are used in the design of liquid crystal materials[6] and molecular hubs capable of accumulating light energy.[7]

Among many ways to construct the isoxazole/isoxazoline cycle, the main ones are [3+2]-dipolar cycloaddition of nitrile oxides to multiple bonds[8] and the reaction of hydroxylamine with β-diketones or their synthetic equivalents;[9,10] the nitrosation reaction of three-membered carbocycles has been further developed in recent years.[11] It should be noted that the cyclopropane fragment imparts conformational rigidity to the molecules of physiologically active compounds and, at the same time, it can be easily modified using cycloaddition reactions, the ring-expansion and ring-opening reactions.[12] The possibility of further transformation of three-membered carbocycles in such structures opens up new prospects. It is directly related to the search for new reagents, including nitrosating agents. Earlier, we carried out the nitrosation-heterocyclization of readily accessible, but hardly reactive, *gem*-dihalocyclopropanes using the activation of nitrosyl chloride with sulfur trioxide. This provided direct access to 5-haloisoxazoles of various structures.[13—15] Continuing research in this direction, we used nitrosylsulfuric acid (NSA) as a nitrosating reagent. Note that nitrosylsulfuric acid is a commercially available nitrosating reagent that is widely used in technological schemes.[16] In the present work, 1,2-diarylcyclopropanes, available by various methods, were used as the objects for nitrosation.[17,18]

It is known that nitrosation of 1,2-diarylcyclopropanes regardless of the nature of the nitrosating agent ($NaNO_2$/CF_3CO_2H,[19] $NOBF_4$,[20] $NOCl \cdot 2SO_3$[21]) results in 3,5-diaryl-4,5-dihydroisoxazoles (isoxazolines). At the same time, an excess of nitrosating reagent (2 equiv. of $NaNO_2$/CF_3CO_2H) afforded 3,5-diarylisoxazoles, the dehydrogenation (oxidation) products of the corresponding isoxazolines.[22]

In this work, we have showed that in the presence of an excess of nitrosylsulfuric acid, which is able to exhibit both nitrosating and oxidative properties,[23] 1,2-diarylcyclopropanes transform readily and smoothly into the corresponding 3,5-diarylisoxazoles *via* successive steps of nitrosation-heterocyclization and oxidation. Earlier, we reported that NSA is a convenient reagent for the oxidation of 3,5-diaryl-4,5-dihydroisoxazoles to the corresponding 3,5-diarylisoxazoles.[24] Indeed, isoxazoline **2a** and the corresponding isoxazole **3a** were obtained by the reaction of 1,2-diphenylcyclopropane **1a** with 1 equiv. of NSA in dichloromethane at room temperature. The reaction mixture contained up to 30% of the starting cyclopropane **1a** (Scheme 1; Table 1, run *1*). Increasing amount of NSA to 2.0—2.5 equiv. resulted in quantitative conversion of cyclopropane **1a** to isoxazole **2a** (see Table 1, run *4*). The search for optimal reaction conditions showed that the most suitable solvent for the reaction is nitromethane. In this solvent, the reactions proceeded cleaner and faster (see Table 1, runs *3* and *4*).

In the case of symmetric 1,2-diarylcyclopropanes **1a—c**, the reaction proceeded highly regioselectively with

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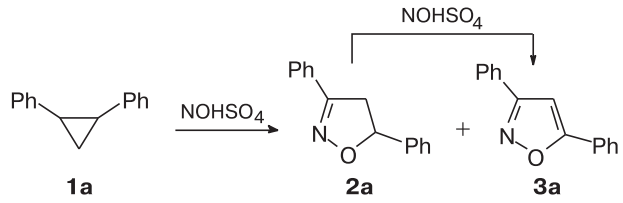
Table 1. Optimization of the reaction conditions of nitrosation-oxidation of cyclopropane **1a** in the presence of nitrosylsulfuric acid[a]

Run	Solvent	$NOHSO_4$ (equiv.)	*t*/h	Conversion of **1a** (%)[b]	Yield (%)	
					2a	**3a**
1	CH_2Cl_2	1.0	20	70	36	10
2	CH_2Cl_2	1.8	20	100	63	16
3	$MeNO_2$	1.3	1	100	69	23
4	$MeNO_2$	2.0	20	100	—	90

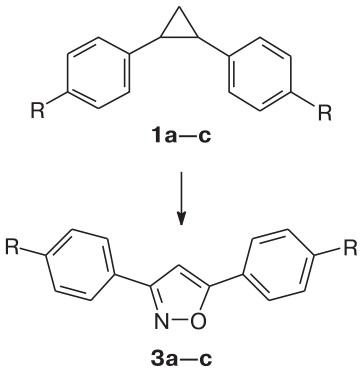
[a] Conditions: $[C_0]_{\mathbf{1a}} = 0.1$ mol L^{-1}, 20 °C.
[b] Conversion of cyclopropane **1a** and the yield of compounds **2a** and **3a** were evaluated by ^1H NMR spectroscopy.

Scheme 1



the cleavage of the C(1)—C(2) bond of the cyclopropane ring to give good yields of the corresponding 3,5-diarylisoxazoles **3a**—**c** (Scheme 2).

Scheme 2

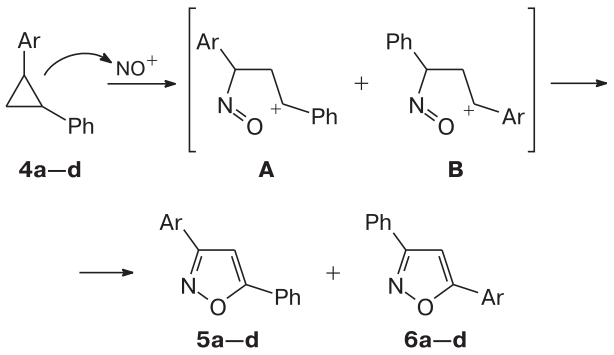


Compounds **1** and **3**	R	Yield of **3** (%)
a	H	65
b	F	78
c	Br	84

Reagents and conditions: $NOHSO_4$ (2.5 equiv.), $MeNO_2$, 20 °C.

In the case of asymmetric 1,2-diarylcyclopropanes **4a**—**d**, selective cleavage of the C(1)—C(2) bond of the cyclopropane occurred. However, the reaction produced the mixtures of regioisomeric 3,5-diarylisoxazoles **5** and **6** (Scheme 3). The location of aryl substituents in the isoxazole cycle was determined by the mass spectrometry using an ion with *m/z* 105 corresponding to the $[PhC{=}O]^+$ as a characteristic one for isoxazoles **5a**—**d**.[25] Afterwards, the isomers were assigned using the NMR spectra. The predominant formation of one or another regioisomer during the reaction is obviously related to the stability of the intermediate carbocation (**A** or **B**) and is determined by the nature and position of the substituents in the aromatic rings.

Scheme 3



Compounds **4—6**	Ar	Ratio **5** : **6**	Yield of **5** + **6** (%)
a	3,4-$Me_2C_6H_3$	1 : 4.5	45
b	2,5-$Me_2C_6H_3$	1 : 1.8	64
c	4-BrC_6H_4	1 : 1.5	98
d	2-FC_6H_4	1.4 : 1	73

Reagents and conditions: $NOHSO_4$ (2.5 equiv.), $MeNO_2$, 20 °C.

Thus, in present work was demonstrated that nitrosylsulfuric acid can be successfully used for the synthesis of 3,5-diarylisoxazoles from 1,2-diarylcyclopropanes. The advantages of this method are the simplicity of the synthesis and the availability of the starting compounds, namely, 1,2-diarylcyclopropanes and nitrosylsulfuric acid.

Experimental

^1H, ^{13}C, and ^{19}F spectra of compounds were recorded in $CDCl_3$ using Bruker Avance-400 and Agilent 400-MR spectrometers with working frequencies of 400.13, 100.67, and 376.29

MHz, respectively (internal standards were HMDS for ^{1}H and ^{13}C, and $CFCl_3$ for ^{19}F). Chemical shifts were measured with an accuracy of 0.01 ppm, spin-spin coupling constants were measured with an accuracy of 0.1 Hz. Mass spectra were recorded using a Thermo Scientific TSQ 8000 gas chromatograph-mass spectrometer (capillary chromatographic column SPB^{TM}-5 (15 m×0.25 mm), carrier gas was helium, gas flow rate was 1 mL min^{-1}). The ionization method was electron impact (electron energy was 70 eV). Temperature conditions were 70 °C (2 min), heating at a rate of 20 °C min^{-1}, maintaining at 280 °C (5 min). The melting points of the obtained compounds were measured using a Mel-TempII instrument and were not corrected. The course of the reactions and the purity of the products were monitored by TLC on Silufol plates. The starting 1,2-diarylcyclopropanes **1a** and **4c** were obtained by a known method.[17] *trans*-1,2-Bis(4-bromophenyl)cyclopropane (**1c**) was obtained by bromination of *trans*-1,2-diphenylcyclopropane.[26] The starting cyclopropanes, with the exception of compound **1c**, were introduced into the reaction as a mixtures of *cis*/*trans* isomers in a 1 : 1 ratio. Nitrosylsulfuric acid was obtained according to a known method[27] and stored at −20 °C under a layer of dichloromethane in a tightly closed flask. Nitrosylsulfuric acid was transferred to a weighted round-bottom flask before the reaction and dichloromethane was evaporated using a rotary evaporator. Dry solid reagent was quickly weighed to carry out the reaction.

Nitrosation of 1,2-diarylcyclopropanes 1a—c and 4a—d (general procedure). Cyclopropane (1 mmol), nitromethane (10 mL), and NSA (0.176 g, 1.5 mmol) were loaded into a 50-mL round-bottom flask equipped with a magnetic stirrer. The reaction mixture was stirred at room temperature for 1.0—1.5 h, then additional amount of NSA (0.117 g, 1 mmol) was added and stirring was continued until completion of the reaction (TLC monitoring). Then the mixture was neutralized with a 0.1 *M* solution of $NaHCO_3$ and the organic compounds were extracted with chloroform (3×20 mL). The organic extracts were combined, dried over sodium sulfate, and the solvent was evaporated using a rotary evaporator. Isoxazoles **3a—c** were purified by recrystallization from ethanol. Isoxazoles **6a**,**b** were isolated in a pure form by fractional crystallization (diethyl ether—hexane), obtaining up to 35% of the pure isomer. The other compounds were isolated by preparative column chromatography (silica gel 40/100, eluent was benzene—petroleum ether, 3 : 2).

3,5-Diphenylisoxazole (3a). Yield 65%, R_f 0.7, m.p. 140 °C (*cf.* Ref. 11: m.p. 140—142 °C). ^{1}H NMR, δ: 6.86 (s, 1 H, C(4)H$_{is}$); 7.51—7.54 (m, 6 H, Ar); 7.89—7.91 (m, 4 H, Ar).

3,5-Bis(4-fluorophenyl)isoxazole (3b). Yield 78%, m.p. 188 °C (*cf.* Ref. 24: m.p. 190 °C). ^{1}H NMR, δ: 6.76 (s, 1 H, C(4)H$_{is}$); 7.20 (m, 4 H, Ar); 7.86 (m, 4 H, Ar).

3,5-Bis(4-bromophenyl)isoxazole (3c). Yield 84%, m.p. 218 °C (*cf.* Ref. 28: m.p. 214 °C). ^{1}H NMR, δ: 6.83 (s, 1 H, C(4)H$_{is}$); 7.65 (m, 4 H, Ar); 7.74 (m, 4 H, Ar).

3-(3,4-Dimethylphenyl)-5-phenylisoxazole (5a) and **5-(3,4-dimethylphenyl)-3-phenylisoxazole (6a)** were synthesized from cyclopropane **4a** (0.96 g) as a mixture in 45% yield. Found (for mixture of **5a** and **6a**) (%): C, 81.97; H, 6.05; N, 5.57. $C_{17}H_{15}NO$. Calculated (%): C, 81.90; H, 6.06; N, 5.62.

3-(3,4-Dimethylphenyl)-5-phenylisoxazole (5a).* ^{1}H NMR, δ: 2.29 (s, 3 H, CH_3); 2.32 (s, 3 H, CH_3); 6.82 (s, 1 H, C(4)H$_{is}$); 7.25 (d, 1 H, Ar, ^3J = 7.2 Hz); 7.49—7.51 (m, 3 H, Ar); 7.60 (d, 1 H, Ar, ^3J = 7.2 Hz); 7.68 (s, 1 H, Ar); 7.84—7.86 (m, 2 H, Ar). ^{13}C NMR, δ: 19.73 and 19.76 (2 CH_3), 97.4 (C(4)$_{is}$), 124.2 (CH), 125.1 (CH), 125.8 (2 CH_{Ph}), 126.6 (C), 127.6 (C), 128.9 (2 CH_{Ph}), 130.08 (CH), 130.11 (CH), 137.2 (C—Me), 138.8 (C—Me), 163.0 (C=N or C—O), 170.1 (C—O or C=N). MS, *m*/*z* (I_{rel} (%)): 249 (15) $[M]^+$, 172 (10) $[M - Ph]^+$, 105 (100) $[PhCO]^+$, 77 (18) $[Ph]^+$, 51 (33), 39 (20).

5-(3,4-Dimethylphenyl)-3-phenylisoxazole (6a). M.p. 124—126 °C. ^{1}H NMR, δ: 2.33 (s, 3 H, CH_3); 2.35 (s, 3 H, CH_3); 6.78 (s, 1 H, C(4)H$_{is}$); 7.25 (d, 1 H, Ar, ^3J = 7.3 Hz); 7.47—7.49 (m, 3 H, Ar); 7.58 (d, 1 H, Ar, ^3J = 7.3 Hz); 7.63 (s, 1 H, Ar); 7.86—7.89 (m, 2 H, Ar). ^{13}C NMR, δ: 19.79 and 19.80 (CH_3), 96.8 (C(4)$_{is}$), 123.3 (CH), 126.8 (2 CH_{Ph}), 126.9 (CH), 127.9 (C), 128.9 (2 CH_{Ph}), 129.3 (C), 129.9 (CH), 130.2 (CH), 137.3 (C—Me), 139.2 (C—Me), 162.9 (C=N or C—O), 170.7 (C—O or C=N). MS, *m*/*z* (I_{rel} (%)): 249 (23) $[M]^+$, 133 (100) $[ArCO]^+$, 105 (41) $[Ar]^+$, 103 (18) $[C_6H_3CO]^+$, 77 (70) $[Ph]^+$, 51 (45), 39 (22).

3-(2,5-Dimethylphenyl)-5-phenylisoxazole (5b) and **5-(2,5-dimethylphenyl)-3-phenylisoxazole (6b)** were synthesized from cyclopropane **4b** (0.7 g) as a mixture in 64% yield. Found (for mixture of **5b** and **6b**) (%): C, 81.62; H, 6.00; N, 5.55. $C_{17}H_{15}NO$. Calculated (%): C, 81.90; H, 6.06; N, 5.62.

3-(2,5-Dimethylphenyl)-5-phenylisoxazole (5b).* ^{1}H NMR, δ: 2.39 (s, 3 H, CH_3); 2.49 (s, 3 H, CH_3); 6.70 (s, 1 H, C(4)H$_{is}$); 7.19 (m, 2 H, Ar); 7.40 (s, 1 H, Ar); 7.49—7.52 (m, 3 H, Ph); 7.85—7.87 (m, 2 H, Ph). ^{13}C NMR, δ: 20.6 (CH_3), 20.9 (CH_3), 100.2 (C(4)$_{is}$) 125.9 (2 CH_{Ph}), 127.6 (C), 128.6 (C), 129.0 (2 CH_{Ph}), 130.1 (CH), 130.2 (CH), 130.3 (CH), 131.1 (CH), 133.7 (C—Me), 135.5 (C—Me), 163.8 (C=N or C—O), 169.5 (C—O or C=N). MS, *m*/*z* (I_{rel} (%)): 249 (25) $[M]^+$, 248 (24) $[M - 1]^+$, 172 (23) $[M - Ph]^+$, 144 (25) $[M - PhCO]^+$, 105 (59) $[PhCO]^+$, 77 (100) $[Ph]^+$, 51 (50).

5-(2,5-Dimethylphenyl)-3-phenylisoxazole (6b). M.p. 63 °C. ^{1}H NMR, δ: 2.40 (s, 3 H, CH_3); 2.53 (s, 3 H, CH_3); 6.72 (s, 1 H, C(4)H$_{is}$); 7.18 (d, 1 H, Ar, ^3J = 7.8 Hz); 7.22 (d, 1 H, Ar, ^3J = 7.8 Hz); 7.48—7.52 (m, 3 H, Ph); 7.60 (s, 1 H, Ar); 7.90—7.91 (m, 2 H, Ph). ^{13}C NMR, δ: 20.5 (CH_3), 20.6 (CH_3), 100.1 (C(4)$_{is}$), 126.3 (C), 126.4 (2 CH_{Ph}), 128.5 (2 CH_{Ph}), 128.6 (CH), 128.9 (C), 129.5 (CH), 130.4 (CH), 130.9 (CH), 132.7 (C—Me), 135.4 (C—Me), 162.2 (C=N or C—O), 170.3 (C—O or C=N). MS, *m*/*z* (I_{rel} (%)): 249 (35) $[M]^+$, 146 (25), 133 (35) $[ArCO]^+$, 105 (35) $[Ar]^+$, 103 $[C_6H_3CO]^+$, 77 (100) $[Ph]^+$, 63 (25), 51 (65), 39 (30).

3-(4-Bromophenyl)-5-phenylisoxazole (5c) and **5-(4-bromophenyl)-3-phenylisoxazole (6c)** were obtained from cyclopropane **4c** (0.40 g) as a mixture in 98% yield.

3-(4-Bromophenyl)-5-phenylisoxazole (5c). M.p. 184 °C (*cf.* Ref. 11: m.p. 182—183 °C). ^{1}H NMR, δ: 6.83 (s, 1 H, C(4)H$_{is}$); 7.52 (m, 3 H, Ar); 7.65 (d, 2 H, Ar, ^3J = 8.4 Hz); 7.77 (d, 2 H, Ar, ^3J = 8.4 Hz); 7.86 (m, 2 H, Ar). ^{13}C, δ: 97.3 (C(4)$_{is}$), 124.3 (CBr), 125.9 (2 CH), 127.3 (C), 128.1 (C), 128.3 (2 CH), 129.1 (2 CH), 130.4 (CH), 132.2 (2 CH), 162.1 (C=N or C—O), 170.8 (C—O or C=N). MS, *m*/*z* (I_{rel} (%)): cluster 301 (10), 299 (9) $[M]^+$; cluster 157 (4), 155 (4) $[C_6H_4Br]^+$; 115 (6) $[M - Br - PhCO]^+$, 105 (100) $[PhCO]^+$, 77 (65) $[Ph]^+$, 51 (29).

5-(4-Bromophenyl)-3-phenylisoxazole (6c). M.p. 164 °C (*cf.* Ref. 29: m.p. 156—158 °C). ^{1}H NMR, δ: 6.86 (s, 1 H, C(4)H$_{is}$); 7.50 (m, 3 H, Ar); 7.66 (d, 2 H, Ar, ^3J = 8.7 Hz); 7.74 (d, 2 H, Ar, ^3J = 8.7 Hz); 7.88 (m, 2 H, Ar). ^{13}C NMR, δ: 97.9 (C(4)$_{is}$),

* Isoxazoles **5a** and **5b** are characterized from the mixtures **5a : 6a** and **5b** : **6b** = 2 : 1.

124.6 (CBr), 126.3 (C), 126.8 (2 CH), 127.3 (2 CH), 128.9 (C), 129.0 (2 CH), 130.1 (CH), 132.3 (2 CH), 163.1 (C=N or C—O), 169.3 (CO or C=N). MS, *m/z* (I_{rel} (%)): cluster 301 (21), 299 (20) $[M]^+$; 220 (2) $[M - Br]^+$, cluster 185 (100), 183 (82) $[BrC_6H_4C{=}O]^+$; cluster 157 (41) 155 (40) $[C_6H_4Br]^+$, 144 (15) $[M - C_6H_4Br]^+$, 89 (25), 77 (42) $[Ph]^+$, 39 (14).

3-(2-Fluorophenyl)-5-phenylisoxazole (5d) and **5-(2-fluorophenyl)-3-phenylisoxazole (6d)** were synthesized from cyclopropane **4d** (0.20 g) as a mixture in 73% yield. Found (for mixture **5d** and **6d**) (%): C, 75.15; H, 4.42; N, 5.66. $C_{15}H_{10}FNO$. Calculated (%): C, 75.30; H, 4.21; N, 5.85.

3-(2-Fluorophenyl)-5-phenylisoxazole (5d). M.p. 84 °C. ^{1}H NMR, δ: 7.00 (d, 1 H, C(4)H_{is}, $^5J_{HF}$ = 3.5 Hz); 7.22 (ddd, 1 H, Ar, ^3J = 7.6 Hz, ^4J = 1.1 Hz, $^3J_{HF}$ = 11.1 Hz); 7.28 (td, 1 H, Ar, $^3J_{HH} = {}^4J_{HF}$ = 7.6 Hz, $^4J_{HH}$ = 1.1 Hz); 7.43—7.53 (m, 4 H, Ph + Ar); 7.87 (m, 2 H, Ph); 8.06 (td, 1 H, Ar, ^3J = 7.5 Hz, ^4J = 1.7 Hz). ^{13}C NMR, δ: 100.0 (d, C(4)H_{is}, $^4J_{CF}$ = 9.2 Hz); 116.4 (d, C(3)H_{Ar}, $^2J_{CF}$ = 21.7 Hz); 117.2 (d, $C(1)_{Ar}$, $^2J_{CF}$ = = 12.4 Hz); 124.6 (d, CH_{Ar}, J_{CF} = 3.5 Hz); 125.8 (2 CH_{Ph}); 127.4 $(C(1)_{Ph})$; 128.9 (2 CH_{Ph}); 129.1 (d, CH_{Ar}, J_{CF} = 3.1 Hz); 130.2 (CH_{Ph}); 131.6 (d, CH_{Ar}, J_{CF} = 8.5 Hz); 158.3 (C=N); 160.3 (d, C—F, $^1J_{CF}$ = 251.9 Hz); 170.3 (C—O). ^{19}F NMR, δ: −114.3 (m, 1 F). MS, *m/z* (I_{rel} (%)): 239 (16) $[M]^+$, 162 (6) $[M - Ph]^+$, 134 (5) $[M - PhCO]^+$, 105 (100) $[PhCO]^+$, 77 (63) $[Ph]^+$.

5-(2-Fluorophenyl)-3-phenylisoxazole (6d). M.p. 91—92 °C (*cf.* Ref. 24: m.p. 91—92 °C). ^{1}H NMR, δ: 7.05 (d, 1 H, C(4)H_{is}, $^5J_{HF}$ = 3.7 Hz); 7.23 (ddd, 1 H, Ar, $^3J_{HF}$ = 10.9 Hz, J = 7.8 Hz, J = 1.0 Hz); 7.31 (td, 1 H, Ar, J = 7.8 Hz, J = 1.0 Hz); 7.42—7.47 (m, 1 H, Ar); 7.48—7.50 (m, 3 H, Ar); 7.91 (m, 2 H, Ar); 8.03 (td, 1 H, Ar, J = 7.8 Hz, J = 1.7 Hz).

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