Russian Journal of General Chemistry, Vol. 73, No. 4, 2003, pp. 646–651. Translated from Zhurnal Obshchei Khimii, Vol. 73, No. 4, 2003, pp. 685–690.
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Synthesis and Structure of Bromonitrothiolene 1,1-Dioxides

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Received May 7, 2002

Abstract — A procedure was developed for preparing mono- and dibromo derivatives of 4-nitro-2- and 3-thiolene 1,1-dioxides, and their structures were studied. The molecular geometry and structural parameters of 4-bromo-3-methyl-4-nitro-2-thiolene 1,1-dioxide were determined by single crystal X-ray diffraction.

Due to the high and diverse reactivity, thiolene 1,1-dioxides are promising precursors for constructing substances with valuable properties [1–3]; therefore, they attract steady researchers' attention. Introduction of a nitro group into the heterocycle not only activates transformations typical of thiolene 1,1-dioxides such as allyl–vinyl isomerization and cycloelimination, but also makes possible new transformations, considerably expanding the synthetic potential of these compounds [4, 5]. A combination of a nitro group with halogen atoms in the thiolene 1,1-dioxide molecule opens further synthetic prospects [6–8].

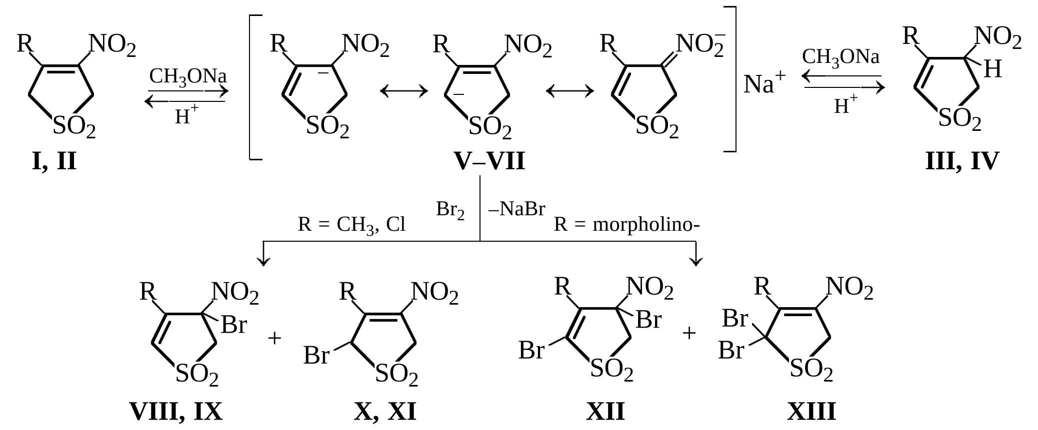
We have recently suggested a procedure for preparing bromo derivatives of nitrothiolene 1,1-dioxides by bromination of nitroalkane salts. The procedure involves synthesis of nitrothiolene 1,1-dioxide salts and their subsequent reaction with bromine [9].

By the reactions of substituted 2- and 3-nitrothiolene 1,1-dioxides **I**–**IV** with sodium methylate under the conditions similar to those of the synthesis of alkylnitronates [10, 11] (absolute ether, room temperature), we readily prepared in high (75–95%) yields a series of sodium 1,1-dioxo-1λ^6-thiolenyl-4-nitronates **V**–**VII**.

Thiolenylnitronates **V**–**VII** are high-melting colorless crystalline substances readily transforming into the starting thiolene 1,1-dioxides **I**–**IV** upon acidification. The spectral characteristics of **V**–**VII** (Table 1) are similar to those of nitroallyl and 3-nitro-1-cyclohexene anions [12, 13] and confirm their salt structure. For example, the IR spectra contain strong bands at 1340, 1280, 1240, 1180, 1160, and 1090 cm^{-1} assignable to the sulfonyl and ionized nitro groups [14, 15], and also the bands of the C=C and C=N bonds at 1650–1600 and 1570–1530 cm^{-1}. The UV spectra contain two absorption bands: at 235 (ε 4000–6000) and 345 nm (ε 9000–16 000 l mol^{-1} cm^{-1}). The steric effect of the bulky substituent in the anion of **VII** results in a hypsochromic shift of the long-wave absorption band (λ_{max} 315 nm). As compared to the model compounds (nitroallyl and nitrocyclohexene anions), thiolenylnitronates **V**–**VII** show a bathochromic shift of the absorption bands, which is apparently due to the contribution of the 3*d*-orbital reson-

Table 1. Spectral characteristics of sodium 1,1-dioxo-1λ^6-thiolenylnitronates **V**–**VII** and of the model compound $CH_2=CH–CH=NOO^-$

Comp. no., compound	IR spectrum, ν, cm^{-1}		UV spectrum, λ_{max}, nm (ε, l mol^{-1} cm^{-1})
	C=C, C=N	NOO^-, SO_2	
V	1650, 1550	1370, 1340, 1270, 1240, 1160, 1090	230 (4400), 330
VI	1640, 1530	1370, 1280, 1240, 1180, 1155, 1090	235 (4000), 345 (16 000)
VII	1600, 1570, 1560, 1520	1380, 1305, 1280, 1260, 1180, 1140	240 (6000), 315 (9000)
$CH_2=CH–CH=NOO^-$	1603, 1558	1340, 1190, 1165, 1030, 995	213 (7400), 277 (22 000)



R = CH_3 (**I, V, VIII, X**), Cl (**II, III, VI, IX, XI**), morpholino- (**IV, VII, XII, XIII**).

ance of the sulfonyl group [16–18] to the electronic structure of the molecules.

Thiolenylnitronates **V**–**VII** were brominated under the same conditions (room temperature, diethyl ether); we found, however, that the reaction result largely depends on the nature of substituent at C^3. Bromination of methyl- and chloro-substituted nitronates **V** and **VI** yields a mixture of isomeric 4-bromo-4-nitro-2- (**VIII, IX**) and 2-bromo-4-nitro-3-thiolene 1,1-dioxides (**X, XI**), originating from the electrophilic attack of bromine at the C^2 and C^4 centers of the resonance-stabilized nitrothiolene 1,1-dioxide anions. In the case of **VII**, the reaction sites are similar, but the electron-donor effect of the morpholine amino group causes additional activation of the heterocyclic nitroallyl anion, and electrophilic bromination proceeds further, yielding a mixture of 2,4-dibromo-3-morpholino-4-nitro-2-thiolene dioxide **XII** and 2,2-dibromo-3-morpholino-4-nitro-3-thiolene 1,1-dioxide **XIII**.

The resulting mixtures of isomeric bromo derivatives of nitrothiolene 1,1-dioxides **VIII** + **X**, **IX** + **XI**, and **XII** + **XIII** were separated by fractional crystallization. Compounds **VIII** (colorless), **X** (colorless), and **XII** (yellow) were isolated as pure crystalline substances. Bromonitrothiolene dioxides **IX**, **XI**, and **XIII** were only identified by ^1H NMR spectroscopy in mixtures with their structural isomers (Table 2).

The ^1H NMR spectra of bromo derivatives of 4-nitro-2-thiolene 1,1-dioxides **VIII**, **IX**, and **XII** show similar patterns of methylene proton signals (quartets at 4.63–4.14 ppm, $^2J_{AB}$ 15 Hz); the downfield shift of the signal in the spectrum of **XII** is apparently due to additional electron-acceptor effect of the second bromine atom at C^3. The spectra of **VIII** and **IX** also contain singlets of olefinic protons at 6.74 and 7.16 ppm, respectively (Table 2). The IR spectra of **VIII**, **IX**, and **XII** contain absorption bands at 1640–1600 cm^{-1} belonging to the C=C vibrations, and also vibration bands of the sulfonyl group (1360–1340, 1170–1160 cm^{-1}) and nonconjugated nitro group (1600–1580, 1360–1330 cm^{-1}). The observed difference between the $\nu_{as}(NO_2)$ and $\nu_s(NO_2)$ frequencies

Table 2. Spectral characteristics of 4-bromo-4-nitro-2-thiolene 1,1-dioxides **VIII, IX**, and **XII** and of 2-bromo-4-nitro-3-thiolene 1,1-dioxides **X, XI**, and **XIII**

Comp. no.	^1H NMR spectrum, δ, ppm			IR spectrum, ν, cm^{-1}		
	CH_2	CH	R	C=C, C=N	NO_2 ($C=NOO^-$)	SO_2
VIII	4.52, 4.14	6.74	2.23	1640	1580, 1340	1340, 1160
IX	4.63, 4.25	7.16	–	1620	1580, 1330	1360, 1170
XII	4.35, 4.55	–	3.47, 3.85	1600	1600, 1360	1360, 1170
X	4.39	5.48	2.49	1630	1540, 1350	1330, 1170
XI	4.56	5.64	–	1630	1535, 1360	1360, 1170
XIII	4.56	–	3.41, 3.81	1600	(1565, 1370, 1130)	1360, 1170

Table 3. Bond lengths *d* in the molecule of **VIII**

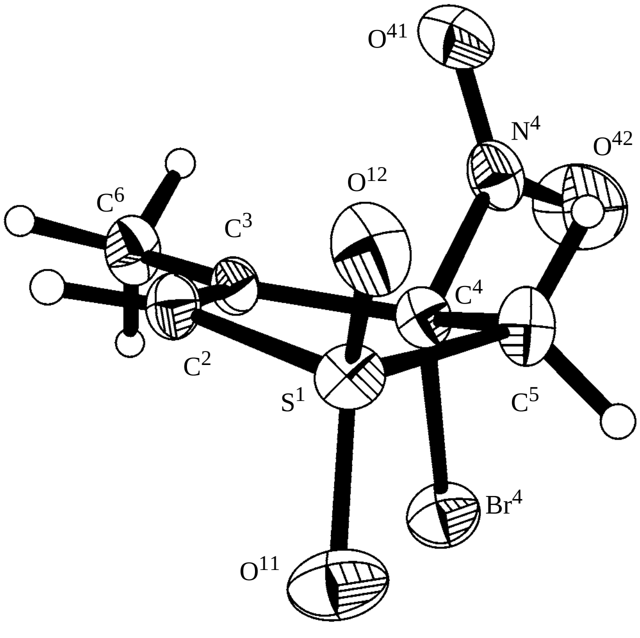
Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
$S^{1}{-}O^{11}$	1.431(2)	$C^{5}{-}H^{52}$	0.9700	$C^{2}{-}C^{3}$	1.323(3)
$S^{1}{-}O^{12}$	1.4329(17)	$C^{3}{-}C^{4}$	1.513(3)	$C^{3}{-}C^{6}$	1.493(3)
$S^{1}{-}C^{2}$	1.742(2)	$C^{4}{-}N^{4}$	1.549(3)	$C^{2}{-}H^{2}$	0.9300
$S^{1}{-}C^{5}$	1.795(2)	$C^{4}{-}Br^{4}$	1.930(2)	$C^{6}{-}H^{61}$	0.9600
$C^{4}{-}C^{5}$	1.526(3)	$N^{4}{-}O^{42}$	1.200(3)	$C^{6}{-}H^{62}$	0.9600
$C^{5}{-}H^{51}$	0.9700	$N^{4}{-}O^{41}$	1.217(3)	$C^{6}{-}H^{63}$	0.9600

Table 4. Bond angles ω in the molecule of **VIII**

Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
$O^{11}S^{1}O^{12}$	117.14(12)	$H^{51}C^{5}H^{52}$	108.9	$C^{2}C^{3}C^{4}$	113.9(2)
$O^{11}S^{1}C^{2}$	110.45(12)	$C^{3}C^{4}C^{5}$	109.58(18)	$C^{6}C^{3}C^{4}$	120.1(2)
$O^{12}S^{1}C^{2}$	112.08(12)	$C^{3}C^{4}N^{4}$	109.37(18)	$C^{3}C^{2}S^{1}$	113.37(17)
$O^{11}S^{1}C^{5}$	110.32(12)	$C^{5}C^{4}N^{4}$	107.58(19)	$C^{3}C^{2}H^{2}$	123.3
$O^{12}S^{1}C^{5}$	110.39(12)	$C^{3}C^{4}Br^{4}$	108.86(15)	$S^{1}C^{2}H^{2}$	123.3
$C^{2}S^{1}C^{5}$	94.10(11)	$C^{5}C^{4}Br^{4}$	112.07(16)	$C^{3}C^{6}H^{61}$	109.5
$C^{4}C^{5}S^{1}$	104.75(16)	$N^{4}C^{4}Br^{4}$	109.34(15)	$C^{3}C^{6}H^{62}$	109.5
$C^{4}C^{5}H^{51}$	110.8	$O^{42}N^{4}O^{41}$	126.0(3)	$H^{61}C^{6}H^{62}$	109.5
$S^{1}C^{5}H^{51}$	110.8	$O^{42}N^{4}C^{4}$	120.1(2)	$C^{3}C^{6}H^{63}$	109.5
$C^{4}C^{5}H^{52}$	110.8	$O^{41}N^{4}C^{4}$	113.8(2)	$H^{61}C^{6}H^{63}$	109.5
$S^{1}C^{5}H^{52}$	110.8	$C^{2}C^{3}C^{6}$	125.9(2)	$H^{62}C^{6}H^{63}$	109.5

($\Delta\nu$ ~240 cm^{-1}) is typical of α-halo nitro compounds [19, 20].

In the ^{1}H NMR spectra of **X**, **XI**, and **XIII**, in contrast to those of the corresponding Δ^{2} isomers, the methylene protoin signals (4.56–4.39 ppm) are singlets; the spectra of monobromides **X** and **XI** also contain singlets of the bromomethine protons at 5.48–5.64 ppm (Table 2). The IR spectra of **X**, **XI**, and **XIII** contain absorption bands of the multiple bonds (1630–1600 cm^{-1}), conjugated nitro group (1540–1535, 1360–1350 cm^{-1}), and sulfonyl group (1360–1330, 1170 cm^{-1}). The presence of the nitro enamine fragment in the structure of **XIII** is confirmed by the vibration bands of the ionized nitro group (1565, 1370, 1130 cm^{-1}) [14, 15].



Molecular geometry of **VIII** in the crystal.

Exhaustive information on the molecular structure of 4-bromo-3-methyl-4-nitro-2-thiolene 1,1-dioxide **VIII** was obtained by single crystal X-ray diffraction. Compound **VIII** exists in the crystal as an enantiomeric pair. The main geometric parameters of the enantiomer with the *R* configuration of C^{4} (see figure) are listed in Tables 3–5. The five-membered heteroring has the *envelope* conformation: the $S^{1}C^{2}C^{3}C^{4}$ fragment is planar within 0.0103 Å, with the C^{5} atom deviating from this plane by 0.357(2) Å. The chiral center has a regular tetrahedral structure ($\angle N^{4}C^{4}Br^{4}$ 109.34°, $\angle C^{3}C^{4}N^{4}$ 109.37°, $\angle C^{3}C^{4}Br^{4}$ 108.86°, $\angle C^{5}C^{4}N^{4}$ 107.58°, $\angle C^{5}C^{4}Br^{4}$ 112.07°, $\angle C^{3}C^{4}C^{5}$ 109.58°) (Table 4). The plane of the sulfonyl group

Table 5. Torsion angles τ in the molecule of **VIII**

Angle	τ, deg	Angle	τ, deg	Angle	τ, deg
$O^{11}S^1C^5C^4$	95.08(0.18)	$Br^4C^4N^4O^{42}$	–18.04(0.30)	$N^4C^4C^3C^6$	52.93(0.27)
$O^{12}S^1C^5C^4$	–133.89(0.16)	$C^3C^4N^4O^{41}$	44.82(0.26)	$Br^4C^4C^3C^6$	–66.48(0.24)
$C^2S^1C^5C^4$	–18.50(0.18)	$C^5C^4N^4O^{41}$	–74.14(0.25)	$C^6C^3C^2S^1$	174.55(0.19)
$S^1C^5C^4C^3$	20.52(0.22)	$Br^4C^4N^4O^{41}$	163.93(0.18)	$C^4C^3C^2S^1$	–1.09(0.26)
$S^1C^5C^4N^4$	139.34(0.16)	$C^5C^4C^3C^2$	–13.45(0.28)	$O^{11}S^1C^2C^3$	–101.20(0.20)
$S^1C^5C^4Br^4$	–100.44(0.15)	$N^4C^4C^3C^2$	–131.16(0.21)	$O^{12}S^1C^2C^3$	126.22(0.19)
$C^3C^4N^4O^{42}$	–137.16(0.26)	$Br^4C^4C^3C^2$	109.43(0.20)	$C^5S^1C^2C^3$	12.26(0.20)
$C^5C^4N^4O^{42}$	103.89(0.28)	$C^5C^4C^3C^6$	170.64(0.21)		

forms with the $C^2C^3C^4C^5$ plane an angle of 101.20° ($O^{11}S^1C^2C^3$) (Table 5). The bond lengths and bond angles in the C–NO_2 group (N^4–O^{41} 1.217, N^4–O^{42} 1.200, C^4–N^4 1.549 Å; $\angle O^{41}N^4O^{42}$ 126°) are close to those in structurally related 4-nitro-3-chloro-2-thiolene 1,1-dioxide [7] and are in good agreement with the average parameters of this group in aliphatic and alicyclic nitro compounds [21]. The C–S (S^1–C^2 1.742, S^1–C^5 1.795 Å) and S–O (S^1–O^{11} 1.431, S^1–O^{12} 1.4329 Å) bond lengths are close to those in the chloronitro-2-thiolene dioxide molecule [7], but the C^2=C^3 double bond (1.323 Å) is shorter by 0.015 Å. The C^4–Br^4 bond length is 1.930 Å, in agreement with published data for aliphatic bromo derivatives [22].

Thus, we have developed a convenient procedure for preparing sodium 1,1-dioxo-1λ^6-2-thiolenyl-4-nitronates and mono- and dibromo derivatives of 4-nitro-2- and -3-thiolene 1,1-dioxides. The electrophilic attack of bromine occurs at the C^2 and C^4 centers of resonance-stabilized nitrothiolene 1,1-dioxide anions; the extent of bromination is largely determined by the electron-donor power of the substituent at the C^5 atom of the heterocyclic anion.

The structure of the bromonitrothiolene 1,1-dioxides prepared was studied by ^1H NMR and IR spectroscopy. The molecular geometry and structural parameters of 4-bromo-3-methyl-4-nitro-2-thiolene 1,1-dioxide **VIII** were determined by single crystal X-ray diffraction.

EXPERIMENTAL

The IR spectra were recorded on Specord IR-75 and UR-20 spectrophotometers (KBr pellets, working ranges of the LiF and NaCl prisms). The ^1H NMR spectra were taken on a Bruker AC-200 spectrometer (200 MHz, solvent CD_3CN). The chemical shifts were determined relative to internal or external HMDS with an accuracy of ±0.5 Hz; the shifts are given in the δ scale.

Compound **VIII** (colorless crystals) crystallizes in the triclinic system. Unit cell parameters at 20°C: a 6.2883(13), b 7.3810(15), c 9.919(2) Å; α 99.09(3)°, β 95.01(3)°, γ 111.63(3)°; V 417.24(15) $Å^3$, Z 2, d_{calc} 2.038 Mg m^{-3}. The experimental set of reflections was obtained with a Nonius KappaCCD diffractometer (MoK_α radiation, graphite monochromator) at 17–19°C. The corrections for the Lorentz and polarization factors were made. The correction for X-ray absorption was not made. A total of 3279 reflections were measured, among them 1913 reflections were unique [R_{int} 0.926, 1744$F > 4\sigma(F)$]. Space group $P\bar{1}$ was determined after preliminary examination of the crystal. The HKLint program package was used for data processing.

The structure of **VIII** was solved by the direct method and refined by the least-squares method in the full-matrix anisotropic approximation with respect to F^2. All nonhydrogen atoms were localized with anisotropic temperature factors. The hydrogen atoms were refined using the rider model. Calculations were performed using the SHELXTL program package (version 5.1) [23]. The atomic coordinates are listed in Table 6.

Sodium 1,1-dioxo-1λ^6-2-thiolenyl-4-nitronates V–VII. A solution of 0.1 g of sodium methylate in 3 ml of absolute methanol was added with stirring at room temperature to a suspension of 1 mmol of appropriate 4-nitrothiolene 1,1-dioxide in 7 ml of absolute diethyl ether. The starting nitrothiolene 1,1-dioxide dissolved. After keeping for 1 h (4 h in the case of **V**), a colorless precipitate of thiolenylnitronate formed (yield 75–95%), which was filtered off, washed with absolute ether, and dried in a vacuum desiccator over calcium chloride. Compounds **V**–**VII** are unstable on heating; therefore, they were brought into subsequent transformations without recrystallization.

4-Bromo-3-methyl-4-nitro-2-thiolene 1,1-dioxide VIII and 2-bromo-3-methyl-4-nitro-3-thio-

Table 6. Coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2 \times 10^3$) of nonhydrogen atoms, and coordinates ($\times 10^4$) and isotropic temperature factors ($Å^2 \times 10^3$) of hydrogen atoms in **VIII**

Atom	*x*	*y*	*z*	U_{eq}
S^1	6716(1)	5003(1)	8463(1)	28(1)
O^{11}	4555(3)	3791(3)	7592(2)	47(1)
O^{12}	7219(3)	4392(3)	9713(2)	40(1)
C^5	9062(4)	5371(4)	7489(3)	33(1)
C^4	9481(4)	7354(3)	7057(2)	28(1)
Br^4	8098(1)	6995(1)	5169(1)	42(1)
N^4	12 135(4)	8505(3)	7214(2)	39(1)
O^{41}	13 084(4)	9198(3)	8408(2)	53(1)
O^{42}	13 076(4)	8601(5)	6210(3)	77(1)
C^3	8480(4)	8544(3)	7997(2)	28(1)
C^2	7080(4)	7497(3)	8763(2)	29(1)
C^6	8974(5)	10666(4)	7933(3)	38(1)
H^{51}	10 435	5430	8053	40
H^{52}	8643	4300	6684	40
H^2	6362	8039	9398	35
H^{61}	8269	10 728	7052	58
H^{62}	10 618	11 396	8049	58
H^{63}	8351	11 239	8656	58

lene 1,1-dioxide X. Bromine (0.83 g) was added dropwise with stirring at room temperature to a suspension of 0.50 g of sodium 3-methyl-1,1-dioxo-1λ^6-2-thiolenyl-4-nitronate **V** in 15 ml of absolute ether. Salt **V** dissolved, and a colorless crystalline precipitate formed. After keeping for 1.5 h, the sodium bromide crystals were filtered off, and the reaction solution was concentrated in a Petri dish in a vent hood. A colorless oily substance was obtained (0.63 g, 98%), containing, according to the ^{1}H NMR spectrum, a 1 : 3 mixture of **VIII** and **X**. By fractional crystallization from CCl_4, we isolated 0.21 g (33%) of 4-bromo-3-methyl-4-nitro-2-thiolene 1,1-dioxide **VIII**, mp 108–110°C, and 0.38 g (60%) of 2-bromo-3-methyl-4-nitro-3-thiolene 1,1-dioxide **X** as a viscous oil. Compound **VIII**. Found, %: C 23.68, 23.69; H 2.67, 2.66; N 5.54, 5.52. $C_5H_6BrNO_4S$. Calculated, %: C 23.40; H 2.34; N 5.47. Compound **X**. Found, %: C 23.35, 23.42; H 2.47, 2.48; N 5.39, 5.38. $C_5H_6BrNO_4S$. Calculated, %: C 23.40; H 2.34; N 5.47.

Bromo-3-chloro-4-nitro-2-thiolene 1,1-dioxide IX and 2-bromo-3-chloro-4-nitro-3-thiolene 1,1-dioxide XI were prepared similarly from sodium 1,1-dioxo-3-chloro-1λ^6-2-thiolenyl-4-nitronate **VI** (keeping time 45 min). A colorless oily substance was obtained (yield 83%), which contained, according to the ^{1}H NMR spectrum, a 3 : 1 mixture of **IX** and **XI**. We failed to separate this mixture by fractional crystallization. Found, %: C 17.31, 17.31; H 1.35, 1.36; N 5.01, 5.02. $C_4H_3BrClNO_4S$. Calculated, %: C 17.36; H 1.08; N 5.06.

2,4-Dibromo-3-morpholino-4-nitro-2-thiolene 1,1-dioxide XII and 2,2-dibromo-3-morpholino-4-nitro-3-thiolene 1,1-dioxide XIII. Bromine (0.32 g) was added dropwise with stirring at room temperature to a suspension of 0.25 g of sodium 3-morpholino-1,1-dioxo-1λ^6-2-thiolenyl-4-nitronate **VII** in 10 ml of absolute ether. Salt **VII** dissolved, and a precipitate formed. After keeping for 1 h, the product was filtered off and washed with water (to remove sodium bromide) and ether. A yellow crystalline precipitate was obtained (0.16 g, 50%), which contained, according to ^{1}H NMR data, a 3 : 1 mixture of 2,4-dibromo-3-morpholino-4-nitro-2-thiolene 1,1-dioxide **XII** and 2,2-dibromo-3-morpholino-4-nitro-3-thiolene 1,1-dioxide **XIII**; mp 123°C. By fractional crystallization from ethanol, we isolated 0.11 g (34%) of 2,4-dibromo-3-morpholino-4-nitro-2-thiolene 1,1-dioxide **XII**, mp 128°C. Mixture of **XII** and **XIII**. Found, %: C 23.30, 23.29; H 2.36, 2.36; N 6.64, 6.62. $C_8H_{10}Br_2N_2O_5S$. Calculated, %: C 23.65; H 2.46; N 6.90. Compound **XII**. Found, %: C 23.34, 23.28; H 2.35, 2.36; N 6.62, 6.63. $C_8H_{10}Br_2N_2O_5S$. Calculated, %: C 23.65; H 2.46; N 6.90.

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