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Synthesis of Benzo-*ortho*-thiazines *S*-Oxides by Diels–Alder Reaction of *N*-Sulfinylanilines with Norbornadiene

Ya. V. Veremeichik[a], P. V. Merabov[a], A. V. Chuiko[a], O. A. Lodochnikova[b], and V. V. Plemenkov[a]

[a]*Kant Baltic Federal University, Kaliningrad, 236040 Russia*
e-mail: plem-kant@yandex.ru
[b]*Arbuzov Institute of Organic and Physical Chemistry, Kazan Scientific Center, Russian Academy of Sciences, Kazan, Russia*

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Abstract—From substituted *N*-sulfinylanilines acting as dienes in the Diels–Alder reaction with norbornadiene *S*-oxides of benzo-*ortho*-thiazines were obtained, which were oxidized into the corresponding *S,S*-dioxides belonging to the class of hybrid thiazinesulfonamide compounds.

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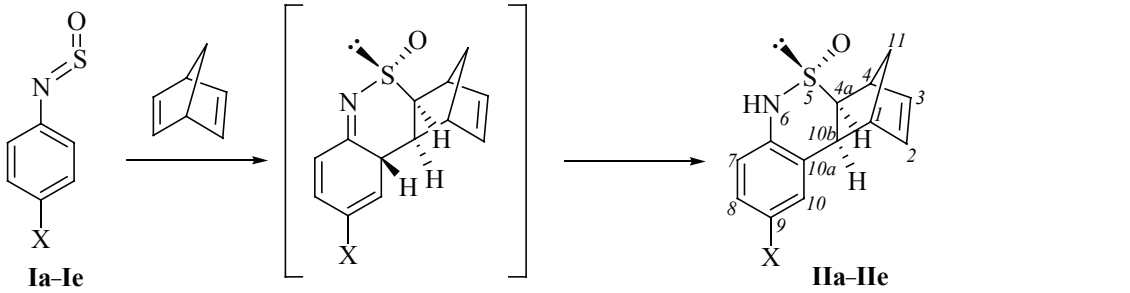
Aromatic *N*-sulfinylamines readily obtainable from the corresponding anilines and thionyl chloride [1] exhibit versatile ability in cycloaddition reactions playing the role of dienophiles [2] and dienes [3] in Diels–Alder reaction. The adducts of these reactions are compounds of hybrid structure with benzo-*ortho*-thiazine and sulfonamide fragments. Aromatic *N*-sulfinylamine react as dienes with strained cycloolefins used as dienophiles similar to styrenes [4, 5].

We formerly studied the reactions of substituted *N*-sulfinylaniline with norbornene, and these adducts were oxidized to the corresponding benzothiazinesulfonamides [6].

In this research we investigated reactions of a series of *N*-sulfinylanilines **Ia–Ie** with norbornadiene aiming at the preparation of benzothiazinesulfonamides containing a C=C bond in the bicyclic fragment of the molecule, which opened an opportunity of further functionalization of adducts to increase the range of potential sulfonamide substances of a new generation.

The reactions were carried out at the ratio thionylaniline–norbornadiene 1 : 1.5 without solvent or in benzene under an argon atmosphere in sealed ampules. At the room temperature the precipitation of the crystals of the reaction product started after several days whereas at 80–90°C the reaction completed in several hours.

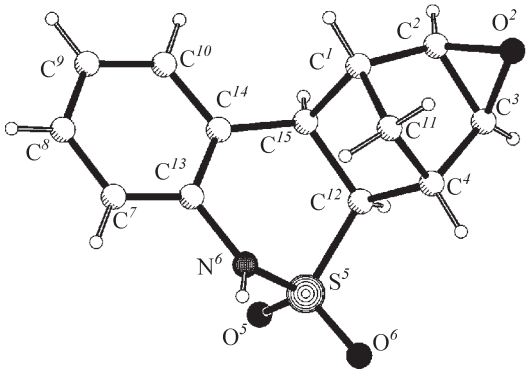
From *para*-substituted *N*-sulfinylanilines and norbornadiene we obtained adducts of the benzo-*ortho*-thiazine structure **IIa–IIe**, whose IR spectra contained characteristic absorption bands of the N–H (3193–3233 cm^{-1}) and S=O (1055–1063 cm^{-1}) bonds. In the ^1H NMR spectra the singlet signal of the proton of the N–H group appears in



X = H (**a**), Me (**b**), MeO (**c**), Cl (**d**), Br (**e**).

the region 8.5–9.2 ppm; the signal system of the aromatic protons of the adduct of unsubstituted *N*-sulfinylaniline **IIa** is observed as two doublets (1H, 6.90 ppm, *J* 7.5 Hz; 1H, 6.50 ppm, *J* 7.4 Hz) and two triplets (1H, 6.78 ppm, *J* 7.5 Hz; 1H, 6.68 ppm, *J* 7.5 Hz), in the spectra of adducts of the *para*-substituted *N*-sulfinylanilines **IIb–IIe** this system gives rise to one singlet (1H, 6.88–7.48 ppm) and two doublets of the *AB* system (1H, 6.72–7.43 ppm, *J* 8.6–8.8 Hz; 1H, 6.32–6.78 ppm, *J* 8.3–8.4 Hz). Among the signals of the bicyclic fragments the characteristic protons are those at the C=C bond (m, 6.02–6.34 ppm) and the protons of the *endo*-methylene bridge (*AB* system, ^{2}J 8.6–9.0 Hz) in the region 0.83–1.91 ppm. The signals of the node protons H^{4a} and H^{10b} appear as two doublets of the *AB* system with ^{3}J 7.6–9.1 Hz (2.75–3.25 ppm), the signals of the node protons H^{1} and H^{4}, by two broadened singlets at 2.40–2.97 ppm. The spatial structure of adducts we assigned based on the data which had been obtained in the study of reactions between *N*-sulfinylanilines with norbornene [6].

Adducts **IIa–IIe** were oxidized with hydrogen peroxide in acetic acid at room temperature to the corresponding sulfonamides **IIIa–IIIe** whose structure was confirmed by the data of IR and NMR spectra and of XRD analysis. Their spectra contained the characteristic absorption bands of SO_2 group vibrations (1135–1140, 1309–1318 cm^{-1}) and the vibration bands of the N–H bond (3253–3263 cm^{-1}), the absorption band of the S=O bond characteristic of initial sulfinamides was absent. It should be noted that the absorption band of NH bond in sulfonamides suffered a considerable shift to higher frequencies as compared to the corresponding sulfinamides.



General view of the molecule of (6a*S*,7*S*,7a*R*,8a*S*,9*S*,9a*S*)-6a,7,7a,8a,9,9a-hexahydro-5*H*-7,9-methanobenzo[*c*]oxireno[2',3',4,5]benzo[1,2-*e*][1,2]-thiazine 6,6-dioxide (**IIIa**) according XRD data.

In ^{1}H NMR spectra of the oxidation products the signal of the proton of the N–H group is present at 10.2–10.3 ppm; the system of the signals of aromatic protons of compound **IIIa** appears as two doublets (1H, 7.40 ppm, *J* 7.7 Hz; 1H, 6.94 ppm, *J* 7.7 Hz) and two triplets (1H, 7.25 ppm, *J* 7.4 Hz; 1H, 7.16 ppm, *J* 7.4 Hz), the similar system of substituted oxides **IIIb–IIIe** gives rise to one singlet (1H, 7.42–7.68 ppm) and two doublets of the *AB* system (1H, 7.22–7.48 ppm, *J* 8.3–8.5 Hz; 1H, 6.63–6.83 ppm, *J* 8.3–8.5 Hz). Characteristically in the ^{1}H NMR spectra of all oxidation products the signals of the protons of the olefin fragment are absent and in the resonance region of the protons of the bicyclic scaffold a weakly resolved *AB* system of two protons at δ 3.42–3.54 ppm (^{3}J 3.5–3.7 Hz) is observed that may be ascribed to an epoxy fragment. The proton signals of the *endo*-methylene bridge (*AB* system, ^{2}J 10.6–10.9 Hz) are present in the region 1.08–1.54 ppm. The signals of node protons H^{4a} and H^{10b} are observed as two doublet *AB* systems, ^{3}J 9.0–9.2 Hz (3.50–3.63 ppm), the signals of node protons H^{1} and H^{4}, as two broadened singlets in the region 2.20–3.07 ppm

The XRD analysis of a single crystal of compound **IIIa** showed that the addition of the diene system of *N*-sulfinylaniline to the olefin bond of the norbornadiene actually occurs from the side of the *endo*-methylene bridge, and the epoxidation of the olefin bond also proceeds from the side of this bridge of the norbornadiene fragment (see the figure).

The observed route of dienes addition and of epoxidation of compounds with bicyclo[2.2.1]heptene skeleton from the side of *endo*-methylene bridge has a fairly general character (cf.,e.g., [5, 7]).

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Bruker Vertex 70 from pellets with KBr. ^{1}H NMR spectra in DMSO-d_6 were registered on a spectrometer Bruker Avance IITM-500. Elemental analysis was carried out on a CHNS-analyzer EuroEA 3000. Melting points were measured using a digital analyzer of melting points Digital Mel-Temp 3.0.

XRD analysis of crystal **IIIa** was performed on a diffractometer Bruker SMART Apex II (graphite monochromator, λMoK_α 0.71073 Å). The correction for extinction was carried out semiempirically along SADABS program [8]. The structure was solved by the direct method ap-

plying SHELXS software [9]. Nonhydrogen atoms were refined first in the isotropic, then in the anisotropic approximation by SHELXL-97 software [10]. The hydrogen atoms were placed in the calculated positions and refined in the rider model. All calculations were carried out using programs WinGX [11] and APEX2 [12]. The figures were obtained with the help of PLATON program [13].

Crystals of compound **IIIa** are monoclinic, $C_{13}H_{13}NO_3S$. At 20°C *a* 12.866(4), *b* 10.469(3), *c* 8.853(3) Å, β 105.572(4)°, *V* 1148.7(6) E^3, *Z* 4, d_{calc} 1.522 gr/cm^3, space group *P* 2_1/c, μMo 2.81 cm^{-1}. The intensity of 8170 reflections were mesured, 1778 among them had $I \geq 2\sigma$. The final values of the divergence factors *R* 0.0512, R_w 0.1449. XRD results are deposited in the Cambridge Crystallographic Data Center (CCDC no. 917575).

N-Sulfinylanilines were synthesized from the corresponding anilines and $SOCl_2$ in benzene by the procedure [1].

The reactions of sulfinylanilines with norbornadiene were carried out by mixing the reagents (thionylaniline–norbornadiene, 1 : 1.5) in sealed ampules at room temperature. The formed precipitate was separated by decanting, washed with petroleum ether, and recrystallized from ethanol.

The oxidation of adducts was performed at heating in a minimal amount of glacial acetic acids using a large excess of H_2O_2. After cooling the solution the product precipitated as crystals which were recrystallized from alcohol.

(1*R*,4*S*,4a*R*,10b*S*)-1,4,4a,10b-Tetrahydro-6*H*-1,4-methanodibenzo[*c,e*][1,2]thiazine 5-oxide (IIa). A mixture of 2.9 g (0.02 mol) of *N*-sulfinylaniline **Ia** and 2.8 g (0.03 mol) of norbornadiene was charged into a glass ampule that was sealed. If after mixing the reagents the complete dissolution did not occur the ampule was slightly heated till total homogenization. The reaction completion was visually seen by the growing amount of precipitated crystals and the reduction in the volume of the liquid phase in the ampule. On opening the ampule the precipitate was separated by decanting, washed with petroleum ether, and recrystallized from warm ethanol. Yield 4.2 g (90%). White crystals, mp 187–188°C. IR spectrum, ν, cm^{-1}: 3165 (NH), 1054 (*S*=O). ^{1}H NMR spectrum, δ, ppm: 0.80 d (1H, H^{11a}, *J* 8.8 Hz), 1.45 d (1H, H^{11b}, *J* 9.2 Hz), 2.40–2.75 m (4H, H^{1,4,4a,10b}), 6.25 m (2H, H^{2,3}), 6.50 d (1H, H^{10}, *J* 7.9 Hz), 6.65 t (1H, H^9, *J* 7.5 Hz), 6.79 t (1H, H^8, *J* 7.2 Hz), 6.91 d (1H, H^7, *J* 7.2 Hz), 8.70 s (1H, H^6). Found, %: C 67.69; H 5.55; N 6.13; S 13.71. $C_{13}H_{13}NOS$. Calculated, %: C 67.50; H 5.66; N 6.06; S 13.86.

Compounds **IIb–IIe** were similarly obtained.

(1*R*,4*S*,4a*R*,10b*S*)-9-Methyl-1,4,4a,10b-tetrahydro-6*H*-1,4-methanodibenzo[*c,e*][1,2]thiazine 5-oxide (IIb) was obtained from 3.1 g (0.02 mol) of reagent **Ib**. Yield 4.4 g (88%). White crystals, mp 185–186°C. IR spectrum, ν, cm^{-1}: 3233 (NH), 1056 (S=O). ^{1}H NMR spectrum, δ, ppm: 1.14 d (1H, H^{11a}, *J* 8.7 Hz), 1,76 d (1H, H^{11b}, *J* 8.7 Hz), 2.22 s (3H, CH_3), 2.75–3.00 m (4H, H^{1,4,4a,10b}), 6.35 m (2H, H^{2,3}), 6.71 d (1H, H^8, *J* 8.6 Hz), 6.92 d (1H, H^7, *J* 8.6 Hz), 7.05 s (1H, H^{10}), 8.90 s (1H, H^6). Found, %: C 68.59; H 6.15; N 5.73; S 13.11. $C_{14}H_{15}NOS$. Calculated, %: C 68.53; H 6.17; N 5.71; S 13.07.

(1*R*,4*S*,4a*R*,10b*S*)-9-Methoxy-1,4,4a,10b-tetrahydro-6*H*-1,4-methanodibenzo[*c,e*][1,2]thiazine 5-oxide (IIc) was obtained from 3.4 g (0.02 mol) of reagent **Ic**. Yield 4.8 g (91%). White crystals with a reddish tint, mp 195–196°C. IR spectrum, ν, cm^{-1}: 3222 (NH), 1063 (S=O). ^{1}H NMR spectrum, δ, ppm: 1.15 d (1H, H^{11a}, *J* 8.9 Hz), 1,80 d (1H, H^{11b}, *J* 8.8 Hz), 2.97–3.25 m (4H, H^{1,4,4a,10b}), 3.72 s (3H, Me), 6.30–6.38 m (2H, H^{2,3}), 6.73 d (1H, H^8, *J* 8.8 Hz), 6.78 d (1H, H^7, *J* 8.5 Hz), 6.92 s (1H, H^{10}), 9.60 s (1H, H^6). Found, %: C 64.20; H 5.60; N 6.53; S 12.11. $C_{14}H_{15}NOS$. Calculated, %: C 64.34; H 5.79; N 5.36; S 12.27.

(1*R*,4*S*,4a*R*,10b*S*)-9-Chloro-1,4,4a,10b-tetrahydro-6*H*-dibenzo[*c,e*][1,2]thiazine 5-oxide (IId) was obtained from 3.5 g (0.02 mol) of reagent **Id** and 2.8 g (0.03 mol) of norbornadiene. Yield 4.7 g (89%). Light yellow crystals, mp 250°C (decomp.). IR spectrum, ν, cm^{-1}: 3228 (NH), 1056 (S=O). ^{1}H NMR spectrum, δ, ppm: 1.15 d (1H, H^{11a}, *J* 8.8 Hz), 1,73 d (1H, H^{11b}, *J* 8.8 Hz), 2.77–3.05 m (4H, H^{1,4,4a,10b}), 6.30 m (2H, H^{2,3}), 6.85 d (1H, H^8, *J* 8.4 Hz), 7.17 d (1H, H^7, *J* 8.4 Hz), 7.34 s (1H, H^{10}), 9.40 s (1H, H^6). Found, %: C 58.69; H 4.55; N 5.23; S 12.11. $C_{13}H_{12}ClNOS$. Calculated, %: C 58.75; H 4.56; N 5.27; S 12.06.

(1*R*,4*S*,4a*R*,10b*S*)-9-Bromo-1,4,4a,10b-tetrahydro-6*H*-1,4-methanodibenzo[*c,e*][1,2]thiazine-5-oxide (IIe) was obtained from 4.4 g (0.02 mol) of reagent **Ie** and 2.8 g (0.03 mol) of norbornadiene. Yield 5.2 g (84%). Light brown crystals, mp 250°C (decomp.). IR spectrum, ν, cm^{-1}: 3229 (N–H), 1055 (S=O). ^{1}H NMR spectrum, δ, ppm: 1.15 d (1H, H^{11a}, *J* 8.8 Hz), 1.72 d (1H, H^{11b},

J 8.8 Hz), 2.77–3.05 m (4H, H^{1,4,4a,10b}), 6.33 m (2H, H^{2,3}), 6.80 d (1H, H^{8}, *J* 8.4 Hz), 7.28 d (1H, H^{7}, *J* 8.4 Hz), 7.48 s (1H, H^{10}), 9.20 s (1H, H^{6}). Found, %: C 50.39; H 3.85; N 4.53; S 10.41. $C_{14}H_{15}BrNOS$. Calculated, %: C 50.33; H 3.90; N 4.51; S 10.34.

(6a*S*,7*S*,7a*R*,8a*S*,9*S*,9a*S*)-6a,7,7a,8a,9,9a-Hexahydro-5*H*-7,9-methanobenzo[*c*]oxireno-[2',3',4,5]benzo[1,2-*e*][1,2]thiazine 6,6-dioxide (IIIa). In glacial acetic acid was dissolved at heating 3.5 g (0.015 mol) of adduct **IIa**. After the homogenization of the solution the heating was stopped, and slowly 10 mL of H_2O_2 was added. On cooling the precipitated crystals were separated by decanting and recrystallized from ethanol. Yield 3.0 g (75%). White crystals, mp 129–131°C. IR spectrum, ν, cm^{-1}: 3257 (NH), 1310, 1137 (SO_2). ^{1}H NMR spectrum, δ, ppm: 1.20 d (1H, H^{10a}, *J* 10.7 Hz), 1.54 d (1H, H^{10b}, *J* 10.9 Hz), 2.65 s (1H, H^{7}), 3.07 s (1H, H^{9}) 3.52 d (1H, H^{7a}, *J* 3.5 Hz), 3.54 d (1H, H^{8a}, *J* 3.5 Hz), 3.58 d (1H, H^{9a}, *J* 9.1 Hz), 3.63 d (1H, H^{6a}, *J* 9.0 Hz), 6.94 d (1H, H^{1}, *J* 7.8 Hz), 7.15 t (1H, H^{2}, *J* 7.5 Hz), 7.25 t (1H, H^{3}, *J* 7.3 Hz), 7.40 d (1H, H^{4}, *J* 7.6 Hz), 10.20 s (1H, H^{5}). Found, %: C 59.29; H 4.95; N 5.34; S 12.21. $C_{13}H_{13}NO_3S$. Calculated, %: C 59.30; H 4.98; N 5.32; S 12.18.

Compounds **IIIb–IIIe** were similarly obtained.

(6a*S*,7*S*,7a*R*,8a*S*,9*S*,9a*S*)-2-Methyl-6a,7,7a,8a,9,9a-hexahydro-5*H*-7,9-methanobenzo[*c*]oxireno-[2',3',4,5]benzo[1,2-*e*][1,2]thiazine 6,6-dioxide (IIIb) was obtained from 4.2 g (0.015 mol) of adduct **IIb**. Yield 3.2 g (77%). Yellow crystals, mp 187–188°C. IR spectrum, ν, cm^{-1}: 3223 (N–H), 1320, 1138 (SO_2). ^{1}H NMR spectrum, δ, ppm: 1.06 d (1H, H^{10a}, *J* 10.7 Hz), 1.38 d (1H, H^{10b}, *J* 10.7 Hz), 2.22 s (3H, C^{11}H$_3$), 2.54 s (1H, H^{7}), 2.94 s (1H, H^{9}), 3.38 d (1H, H^{8a}, *J* 3.8 Hz), 3.42 d (1H, H^{7a}, *J* 3.9 Hz), 3.46 d (1H, H^{6a}, *J* 8.6 Hz), 3.48 d (1H, H^{9a}, *J* 8.7 Hz), 6.72 d (1H, H^{4}, *J* 8.0 Hz), 6.96 d (1H, H^{3}, *J* 7.9 Hz), 7.09 s (1H, H^{1}), 9.80 s (1H, H^{5}). Found, %: C 60.67; H 5.42; N 5.03; S 11.61. $C_{14}H_{15}NO_3S$. Calculated, %: C 60.63; H 5.46; N 5.05; S 11.56.

(6a*S*,7*S*,7a*R*,8a*S*,9*S*,9a*S*)-2-Methoxy-6a,7,7a,8a,9,9a-hexahydro-5*H*-7,9-methanobenzo[*c*]oxireno-[2',3',4,5]benzo[1,2-*e*][1,2]thiazine 6,6-dioxide (IIIc) was obtained from 4.4 g (0.015 mol) of adduct **IIc**. Yield 3.3 g (75%). White crystals, mp 145–147°C. IR spectrum, ν, cm^{-1}: 3265 (N–H), 1318, 1135 (SO_2). ^{1}H NMR spectrum, δ, ppm: 1.06 d (1H, H^{10a}, *J* 10.7 Hz), 1.38 d (1H, H^{10b}, *J* 10.5 Hz), 2.60 s (1H, H^{7}), 2.93 s (1H, H^{9}), 3.12 d (1H, H^{9a}, *J* 9.1 Hz), 3.38–3.43 m (2H, H^{8a,7a}), 3.50 d (1H, H^{6a}, *J* 9.1 Hz), 3.72 s (3H, OCH_3), 6.72–6.78 (2H, H^{4,3}), 6.88 s (1H, H^{1}), 9.60 s (1H, H^{5}). Found, %: C 57.34; H 5.18; N 4.73; S 10.95. $C_{14}H_{15}NO_4S$. Calculated, %: C 57.32; H 5.16; N 4.77; S 10.93.

(6a*S*,7*S*,7a*R*,8a*S*,9*S*,9a*S*)-2-Chloro-6a,7,7a,8a,9,9a-hexahydro-5*H*-7,9-methanobenzo[*c*]oxireno-[2',3',4,5]benzo[1,2-*e*][1,2]thiazine 6,6-dioxide (IIId) was obtained from 4.5 g (0.015 mol) of adduct **IId**. Yield 3.4 g (75%). White crystals with a reddish tint, mp 257–258°C. IR spectrum, ν, cm^{-1}: 3223 (N–H), 1318, 1135 (SO_2). ^{1}H NMR spectrum, δ, ppm: 1.08 d (1H, H^{10a}, *J* 10.7 Hz), 1.36 d (1H, H^{10b}, *J* 10.7 Hz), 2.60 s (1H, H^{7}), 2.90 s (1H, H^{9}), 3.36 d (1H, H^{7a}, *J* 3.2 Hz), 3.42 d (1H, H^{8a}, *J* 2.8 Hz), 3.51 d (1H, H^{9a}, *J* 9.2 Hz), 3.56 d (1H, H^{6a}, *J* 9.2 Hz), 6.83 d (1H, H^{3}, *J* 8.5 Hz), 7.22 d (1H, H^{4}, *J* 8.5 Hz), 7.42 s (1H, H^{1}), 10.20 s (1H, H^{5}). Found, %: C 52.42; H 4.12; N 4.73; S 10.81. $C_{13}H_{12}ClNO_3S$. Calculated, %: C 52.44; H 4.07; N 4.70; S 10.77.

(6a*S*,7*S*,7a*R*,8a*S*,9*S*,9a*S*)-2-Bromo-6a,7,7a,8a,9,9a-hexahydro-5*H*-7,9-methanobenzo[*c*]oxireno-[2',3',4,5]benzo[1,2-*e*][1,2]thiazine 6,6-dioxide (IIIe) was obtained from 5.1 g (0.015 mol) of adduct **IIe**. Yield 3.8 g (74%), white crystals, mp 264–265°C. IR spectrum, ν, cm^{-1}: 1137, 1310 (SO_2), 3259 (N–H). ^{1}H NMR spectrum, δ, ppm: 1.08 d (1H, H^{10a}, *J* 10.6 Hz), 1.35 d (1H, H^{10b}, *J* 10.8 Hz), 2.48 s (1H, H^{7}), 2.57 s (1H, H^{9}), 3.34 d (1H, H^{7a}, *J* 3.6 Hz), 3.40 d (1H, H^{8a}, *J* 3.6 Hz), 3.50 d (1H, H^{9a}, *J* 9.2 Hz), 3.58 d (1H, H^{6a}, *J* 9.2 Hz), 6.77 d (1H, H^{3}, *J* 8.4 Hz), 7.35 d (1H, H^{4}, *J* 8.5 Hz), 7.55 s (1H, H^{1}), 10.30 s (1H, H^{5}). Found, %: C 45.62; H 3.52; N 4.07; S 9.41. $C_{13}H_{12}BrNO_3S$. Calculated, %: C 45.63; H 3.54; N 4.09; S 9.37.

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