Russian Journal of Organic Chemistry, Vol. 38, No. 12, 2002, pp. 1758–1763. Translated from Zhurnal Organicheskoi Khimii, Vol. 38, No. 12, 2002, pp. 1819–1824.
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Dedicated to Full Member of the Russian Academy of Sciences
V.A. Tartakovskii on the 70th Anniversary of His Birth

Synthesis of Aryl 3,5-Dinitrophenyl Sulfones and Sulfoxides. Transformations of 3,5-Dinitrodiphenyl Sulfone in Reactions with O- and S-Nucleophiles*

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Received April 25, 2002

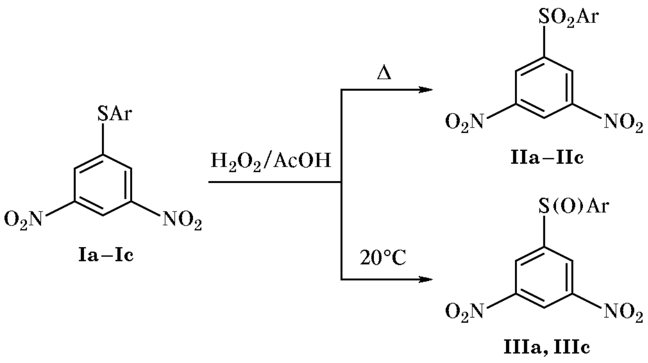
Abstract—1-Arylthio-3,5-dinitrobenzenes are selectively oxidized to the corresponding sulfones and sulfoxides by the action of hydrogen peroxide. Reactions of 3,5-dinitrodiphenyl sulfone with O- and S-centered nucleophiles (RXH, X = O, S) in dipolar aprotic solvents in the presence of K_2CO_3 result in replacement of the nitro group by the RX fragment; the reaction with methanol occurs in aqueous medium in the presence of $NaHCO_3$. Substitution of the nitro group in 3,5-dinitrodiphenyl sulfone by phenylthio group, followed by oxidation of the sulfur atom to SO_2 and further replacement of the remaining nitro group yields 1,3,5-tris(phenylsulfonyl)benzene. The phenylsulfonyl group in the latter is replaced by phenylthio group by reaction with PhSH in the presence of K_2CO_3. Mononitrosulfones obtained by nucleophilic substitution in the title compound can be reduced to the corresponding anilines.

1,3,5-Trinitrobenzene usually reacts with various nucleophiles to give stable anionic σ-adducts [1–3]. However, we previously found conditions under which arenethiols and their heteroaromatic analogs are capable of replacing one nitro group in 1,3,5-trinitrobenzene to give the corresponding 1-aryl(hetaryl)thio-3,5-dinitrobenzenes **I**. These conditions include the use of dipolar aprotic solvents [such as *N*-methylpyrrolidone (NMP), DMF, etc.] and alkalies or alkali metal carbonates (K_2CO_3 is the most convenient) at 50–80°C. Under more severe conditions (150°C), two nitro groups can be replaced [4–6].

We have found that sulfides **I** undergo selective oxidation by the action of H_2O_2 in AcOH, affording the corresponding sulfones or sulfoxides. Sulfones **IIa**–**IIc** are formed in the presence of a larger excess of H_2O_2 on heating under reflux. When the amount of hydrogen peroxide is close to equimolar and the reaction is carried out at room temperature (~20°C), sulfoxides **IIIa** and **IIIb** are obtained (Scheme 1).

We have studied transformations of 3,5-dinitrodiphenyl sulfone **IIa** in reactions with O- and S-nucleophiles (RXH, X = O, S) in the presence of bases with the goal of synthesizing new polyfunctional *meta*-substituted benzene derivatives. Sulfone **IIa** can be regarded as an analog of 1,3,5-trinitrobenzene; therefore, the reactions of **IIa** with nucleophiles were performed under conditions ensuring nitro group

Scheme 1.



Ar = Ph (**a**), 4-ClC_6H_4 (**b**), 2-benzothiazolyl (**c**).

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[*] This study was financially supported by the Russian Foundation for Basic Research (project no. 01-03-32261).

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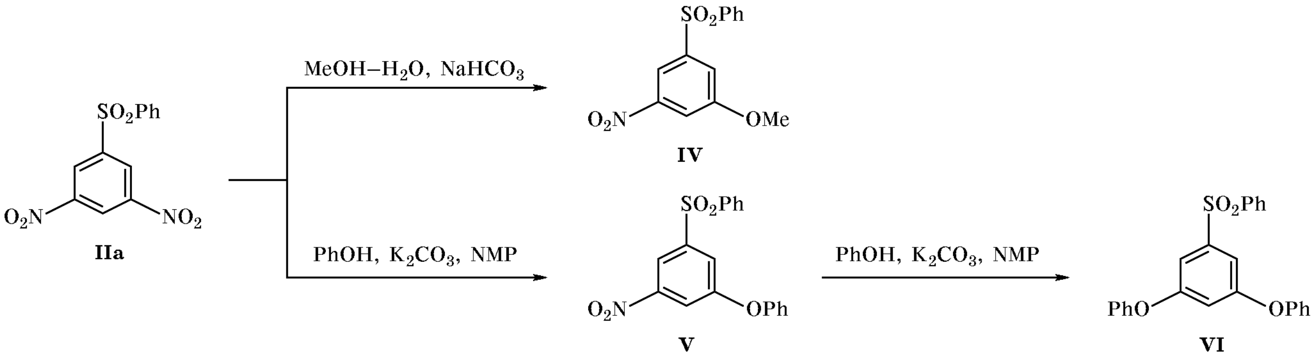
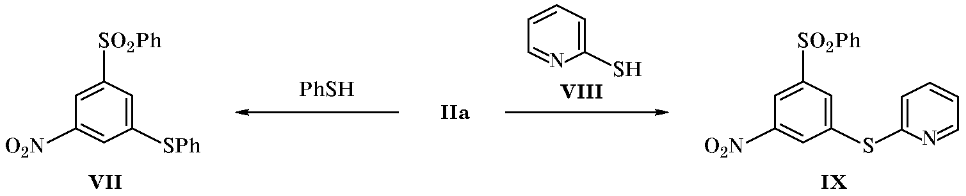
Scheme 2.



NMP is *N*-methylpyrrolidone.

Scheme 3.



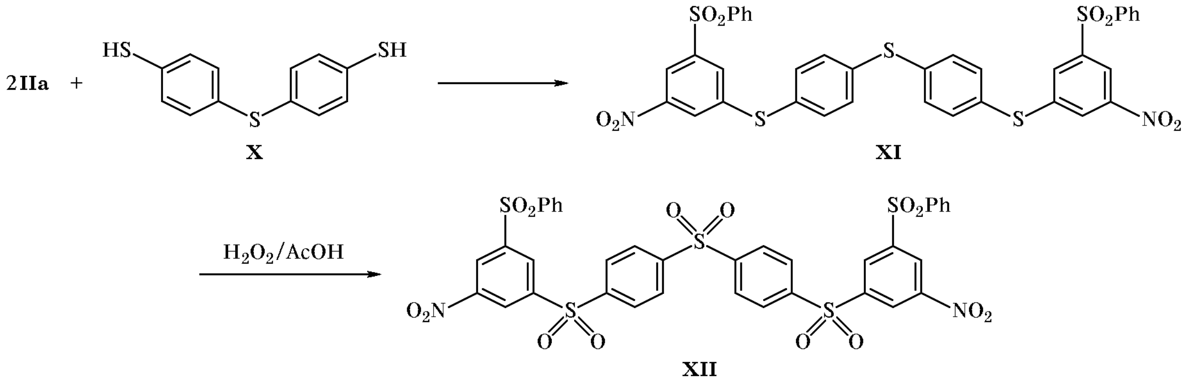
replacement in 1,3,5-trinitrobenzene. Both nitro and phenylsulfonyl groups are good nucleofuges in the aromatic nucleophilic substitution reactions [7], and replacement of the $PhSO_2$ group might also be expected. Although the nitro group is generally believed [8] to be considerably more labile than $PhSO_2$, it is known that selective replacement of either NO_2 or $PhSO_2$ group (depending on the substrate structure and nucleophile nature) can be effected in benzene derivatives where these group occupy *ortho* or *para* positions with respect to each other [9–11]. As far as we know, the order of replacement of *meta*-arranged nitro and phenylsulfonyl groups was not studied. Running ahead, it should be noted that in all the examined reactions only the nitro group was replaced, while no products of substitution of the $PhSO_2$ group were detected.

It is known that heating of 1,3,5-trinitrobenzene in aqueous methanol in the presence of a large excess of $NaHCO_3$ leads to replacement of one nitro group by methoxy [12]. Under similar contitions, one nitro group in sulfone **IIa** is also replaced by methoxy group to give 3-methoxy-5-nitrodiphenyl sulfone (**IV**) (Scheme 2). The reaction of sulfone **IIa** with phenol in the presence of K_2CO_3 in NMP at 80°C gives nitro(phenoxy)phenyl sulfone **V**. Treatment of the latter with 1 equiv of phenol under more severe conditions (at 150°C) results in substitution of the remaining nitro group with formation of diphenoxyphenyl sulfone **VI**. Under the same conditons, one or two nitro groups in 1,3,5-trinitrobenzene are replaced by the action of phenol [13, 14].

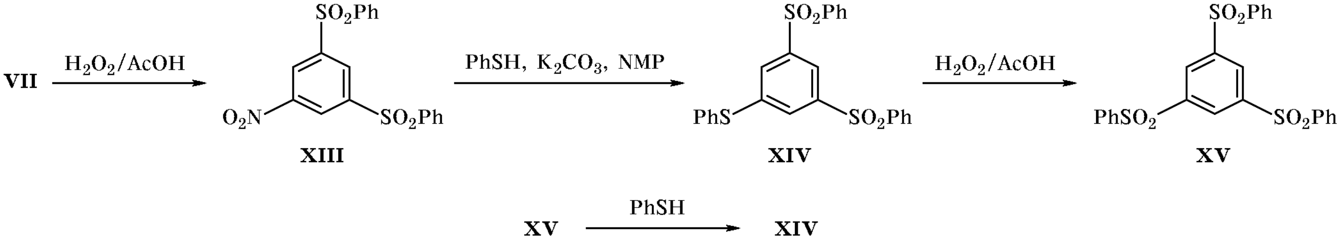
Sulfone **IIa** reacts with arenethiols in NMP in the presence of K_2CO_3 at 50°C, yielding products of replacement of one nitro group (Scheme 3). Likewise, one nitro group in 1,3,5-trinitrobenzene is replaced by the action of arenethiols under similar conditions [4–6]. The reaction of **IIa** with benzenethiol gave sulfone **VII**, and with a heterocyclic analog, pyridine-2-thiol (**VIII**), sulfone **IX** was formed. The best results were obtained in HMPA at ~20°C.

By reaction of bis(4-mercaptophenyl) sulfide (**X**) with 2 equiv of sulfone **IIa** we obtained product **XI**. All sulfide bridges in the latter were oxidized to SO_2 fragments with 35% hydrogen peroxide in acetic acid. As a result, polysulfone **XII** was obtained (Scheme 4). Analogous oxidation of sulfone **VII** gave bis-sulfone **XIII** (Scheme 5). Despite the presence of two $PhSO_2$ groups in **XIII**, only the nitro group was replaced in the reaction with benzenethiol (NMP, K_2CO_3, 50°C). The substitution product, bis-sulfone **XIV** was oxidized with H_2O_2/AcOH to tris-sulfone **XV**. On the other hand, the reaction of **XV** with PhSH under the conditions ensuring nitro group replacement in compound **XIII** (as well as in 1,3,5-trinitrobenzene [4–6]) gives bis-sulfone **XIV** as a result of substitution of the $PhSO_2$ group. Thus, the nitro group is always replaced first in aromatic substrates having

Scheme 4.



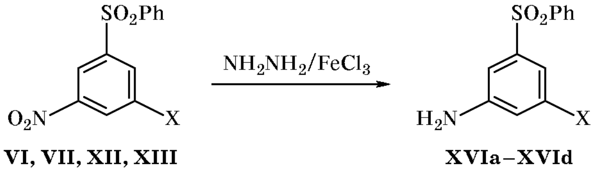
Scheme 5.



$PhSO_2$ and NO_2 groups in the *meta* position with respect to each other. If no nitro group is present, $PhSO_2$ group is replaced with the same ease. Almost complete analogy between the transformations of sulfone **IIa** and 1,3,5-trinitrobenzene in reactions with O- and S-nucleophiles should be noted, though compound **IIa** gives rise to a greater diversity of products.

Some of the prepared mononitrosulfones was reduced to the corresponding anilines **XVIa**–**XVId** with hydrazine hydrate in the presence of $FeCl_3$ and charcoal (Scheme 6; cf. [15]).

Scheme 6.



X = PhO (**a**), PhS (**b**), 2-C_5H_4NS (**c**), PhO_2 (**d**).

Thus, our study of transformations of sulfone **IIa** in reactions with O- and S-nucleophiles and subsequent oxidation and reduction of nucleophilic substitution products resulted in development of procedures for preparation of new polyfunctional benzene derivatives substituted in the *1,3,5*-positions. Among the obtained compounds, only tris-sulfone **XV** was reported previously [16].

The structure of the products was proved by elemental analyses and ^1H NMR, IR [$\nu_{as}(NO_2)$ 1540, $\nu_s(NO_2)$ 1260, $\nu(SO)$ 1340, $\nu_{as}(SO_2)$ 1350, $\nu_s(SO_2)$ 1150, $\nu(NH_2)$ 3480, $\delta(NH_2)$ 1620 cm^{-1}], and electron impact mass spectra (in all cases, the molecular ion peak was observed).

EXPERIMENTAL

The melting points were determined on a Boetius device (samples were heated at a rate of 4 deg/min). The IR spectra were recorded on a Specord M-80 spectrometer from samples pelleted with KBr. The mass spectra (70 eV) were measured on a Kratos MS-30 instrument. The ^1H NMR spectra were recorded on a Bruker AC-200 spectrometer. The progress of reactions was monitored by HPLC using a Liquochrom Model 2010 chromatograph (UV detector, λ 240 nm; Silasorb C-18 reversed phase; eluent acetonitrile–water, 3 : 1).

Aryl 3,5-dinitrophenyl sulfones IIa–IIc. A mixture of 0.03 mol of 1-arylthio-3,5-dinitrobenzene **Ia**–**Ic**, 10.2 ml of 35% H_2O_2, and 70 ml of glacial acetic acid was heated under reflux. The mixture was cooled, and the precipitate was filtered off, dried, and (if necessary) recrystallized.

3,5-Dinitrodiphenyl sulfone (IIa). Reaction time 2 h. Yield 93%. mp 169–170°C; ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.71 m (3H), 8.28 d (2H, ^3J = 8.1 Hz), 8.95 d (2H, ^4J = 2.3 Hz), 9.01 d (1H, ^4J = 2.3 Hz). Found, %: C 47.16; H 2.95; N 9.35; S 10.08. $C_{12}H_8N_2O_6S$. Calculated, %: C 46.75; H 2.62; N 9.09; S 10.04.

4-Chloro-3′,5′-dinitrodiphenyl sulfone (IIb). Reaction time 1.5 h. Yield 89%. mp 140–141°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.76 d (2H, ^3J = 8.2 Hz), 8.22 d (2H, ^3J = 8.1 Hz), 8.99 d (2H, ^4J = 2.1 Hz), 9.02 t (1H, ^4J = 2.1 Hz). Found, %: C 42.51; H 2.73; Cl 10.58; N 8.61; S 9.06. $C_{12}H_7ClN_2O_6S$. Calculated, %: C 42.06; H 2.06; Cl 10.34; N 8.17; S 9.36.

2-(3,5-Dinitrophenylsulfonyl)-1,3-benzothiazole (IIc). Reaction time 6 min. Yield 35%. mp 158–159°C (from MeCN–H_2O, 3 : 1). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.72 m (2H), 8.24 m (1H), 8.37 m (1H), 9.04 d (2H, ^4J = 2.3 Hz), 9.10 d (1H, ^4J = 2.3 Hz). Found, %: C 43.22; H 2.31; N 11.77; S 17.05. $C_{13}H_7N_3O_6S_2$. Calculated, %: C 42.74; H 1.93; N 11.50; S 17.55.

Aryl 3,5-dinitrodiphenyl sulfoxides IIIa and IIIc. A mixture of 0.025 mol of 1-arylthio-3,5-dinitrobenzene **IIa** or **IIc**, 2.25 ml of 35% hydrogen peroxide, and 45 ml of glacial acetic acid was stirred for 7 days at room temperature. It was then poured into 225 ml of cold water, and the precipitate was filtered off, dried, and recrystallized.

3,5-Dinitrodiphenyl sulfoxide (IIIa). Yield 80%. mp 119–120°C (from CCl_4). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.59 m (3H), 7.73 m (2H), 8.85 t (1H, ^4J = 2.1 Hz), 8.94 d (2H, ^4J = 2.3 Hz). Found, %: C 49.93; H 3.09; N 10.01; S 10.21. $C_{12}H_8N_2O_5S$. Calculated, %: C 49.31; H 2.76; N 9.58; S 10.97.

2-(3,5-Dinitrophenylsulfinyl)-1,3-benzothiazole (IIIc). Yield 58%, mp 111–112°C (from CCl_4–$CHCl_3$, 2 : 1). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.57 m (2H), 8.10 m (1H), 8.21 m (1H), 8.91 t (1H, ^3J = 8.1 Hz), 8.97 d (2H, ^4J = 2.3 Hz). Found, %: C 45.05; H 2.51; N 12.35; S 18.02. $C_{13}H_7N_3O_5S_2$. Calculated, %: C 44.69; H 2.02; N 12.03; S 18.36.

3-Methoxy-5-nitrodiphenyl sulfone (IV). A mixture of 6.16 g (0.02 mol) of sulfone **IIa**, 120 ml of methanol, 7.72 g (0.09 mol) of $NaHCO_3$, and 30 ml of water was refluxed for 6 h. It was then cooled, and the precipitate was filtered off, washed with water, dried, and recrystallized. Yield 72%. mp 127–128°C (from MeOH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.94 s (3H), 7.60 m (3H), 7.77 t (1H, ^3J = 8.1 Hz), 7.87 t (1H, ^4J = 2.1 Hz), 7.98 d (2H, ^3J = 8.1 Hz), 8.30 t (1H, ^4J = 2.1 Hz). Found, %: C 53.55; H 4.02; N 4.99; S 10.51. $C_{13}H_{11}NO_5S$. Calculated, %: C 53.24; H 3.78; N 4.78; S 10.93.

3-Nitro-5-phenoxydiphenyl sulfone (V). A solution of 7.7 g (0.025 mol) of sulfone **IIa** in 10 ml of NMP was added to a mixture of 2.35 g (0.025 mol) of phenol, 3.45 g (0.025 mol) of K_2CO_3, and 15 ml of NMP. The mixture was stirred for 6 h at 80°C and poured into 120 ml of cold water, and the precipitate was filtered off, dried, and recrystallized. Yield 74%. mp 119–120°C (from MeOH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.20 d (2H, ^3J = 8.1 Hz), 7.31 m (1H), 7.50 m (2H), 7.68 m (3H), 7.86 d (1H, ^4J = 2.3 Hz), 7.98 d (1H, ^4J = 2.3 Hz), 8.09 d (2H, ^3J = 7.9 Hz), 8.20 d (1H, ^4J = 2.2 Hz). Found, %: C 61.20; H 4.02; N 4.24; S 8.80. $C_{18}H_{13}NO_5S$. Calculated, %: C 60.84; H 3.69; N 3.39; S 9.02.

3,5-Diphenoxydiphenyl sulfone (VI) was synthesized as described above for compound **V**, but the mixture was stirred for 4 h at 150°C. Yield 63%. mp 87–88°C (from 2-propanol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.78 d (1H, ^4J = 2.0 Hz), 7.14 m (8H), 7.42 m (4H), 7.64 m (3H), 7.91 d (2H, ^3J = 8.1 Hz). Found, %: C 71.98; H 4.90; S 7.37. $C_{24}H_{18}O_4S$. Calculated, %: C 71.62; H 4.51; S 7.97.

3-Nitro-5-phenylthiodiphenyl sulfone (VII). A solution of 7.7 g (0.025 mol) of sulfone **IIa** in 10 ml of NMP was added to a mixture of 2.55 ml (0.025 mol) of benzenethiol, 3.45 g (0.025 mol) of K_2CO_3, and 15 ml of NMP. The mixture was stirred for 3 h at 50°C and poured into 120 ml of cold water, and the precipitate was filtered off, dried, and recrystallized. Yield 75%. mp 89–90°C (from MeOH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.53 m (4H), 7.68 m (4H), 8.00 m (4H), 8.33 t (1H, ^4J = 2.1 Hz). Found, %: C 58.68; H 3.91; N 4.07; S 16.90. $C_{18}H_{13}NO_4S_2$. Calculated, %: C 58.21; H 3.53; N 3.77; S 17.27.

3-Nitro-5-(2-pyridylthio)diphenyl sulfone (IX). A solution of 7.7 g of sulfone **IIa** in 10 ml of HMPA was added to a mixture of 2.275 g (0.025 mol) of pyridine-2-thiol (**VIII**), 3.45 g (0.025 mol) of K_2CO_3, and 15 ml of HMPA. The mixture was stirred for 48 h at 20°C and poured into 225 ml of cold water, and the precipitate was filtered off, dried, and recrystallized. Yield 57%. mp 106–107°C (from MeCN–H_2O, 3 : 1). ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 7.29 m (1H), 7.41 d (1H, ^3J = 8.3 Hz), 7.72 m (4H), 8.09 m (2H), 8.41 m (1H), 8.46 t (1H, ^4J = 2.1 Hz), 8.57 t (1H, ^4J = 2.1 Hz), 8.64 t (1H, ^4J = 2.1 Hz). Found, %: C 55.25; H 3.77; N 7.94; S 16.98. $C_{17}H_{12}N_2O_4S_2$. Calculated, %: C 54.83; H 3.25; N 7.52; S 17.22.

4,4′-Bis(3-nitro-5-phenylsulfonylphenylthio)diphenyl sulfide (XI). A solution of 3.08 g (0.01 mol) of sulfone **IIa** in 4 ml of NMP was added to a mixture of 1.25 g (0.005 mol) of 4,4′-dimercaptodiphenyl sulfide (**X**) and 1.38 g (0.01 mol) of K_2CO_3 in 6 ml of NMP. The mixture was stirred for 4 h at 50°C and poured into 50 ml of cold water, and the precipitate was filtered off, dried, and dissolved in 1,2-dichloroethane. The solution was filtered through a layer of silica gel (~10 g), the solvent was removed, and the residue was recrystallized. Yield 32%. mp 138–140°C (from MeOH); ^1H NMR spectrum (DMSO-d_6), δ, ppm: 7.58 m (14H), 8.07 m (8H), 8.38 m (2H). Found, %: C 55.32; H 3.91; N 3.04; S 20.06. $C_{36}H_{24}N_2O_8S_5$. Calculated, %: C 55.94; H 3.13; N 3.62; S 20.74.

4,4′-Bis(3-nitro-5-phenylsulfonylphenylsulfonyl)diphenyl sulfone (XII). A mixture of 1.544 g (0.002 mol) of tris-sulfide **XI**, 2 ml (0.024 mol) of 35% hydrogen peroxide, and 40 ml of acetic acid was heated for 2 h under reflux. The mixture was cooled, and the precipitate was filtered off and dried. Yield 37%. mp 288–290°C. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 7.65 m (12H), 8.12 m (4H), 8.22 m (4H), 8.40 m (4H), 8.82 m (6H). Found, %: C 49.16; H 3.17; N 2.60; S 17.88. $C_{36}H_{24}N_2O_{14}S_5$. Calculated, %: C 49.76; H 2.78; N 3.22; S 18.45.

1-Nitro-3,5-bis(phenylsulfonyl)benzene (XIII). A mixture of 11.13 g (0.03 mol) of sulfone **VII**, 10.2 ml of 35% hydrogen peroxide, and 70 ml of acetic acid was heated for 1 h under reflux. The mixture was cooled, and the precipitate was filtered off and dried. Yield 95%. mp 177–178°C. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 7.70 m (6H), 8.14 d (4H, ^3J = 8.1 Hz), 8.76 t (1H, ^4J = 2.1 Hz), 8.70 d (2H, ^4J = 2.3 Hz). Found, %: C 53.99; H 3.79; N 4.01; S 15.21. $C_{18}H_{13}NO_6S_2$. Calculated, %: C 53.59; H 3.25; N 3.47; S 15.90.

1-Phenylthio-3,5-bis(phenylsulfonyl)benzene (XIV). A solution of 4.03 g (0.01 mol) of compound **XIII** in 4 ml of NMP was added to a mixture of 1.02 ml (0.01 mol) of benzenethiol, 1.38 g (0.01 mol) of K_2CO_3, and 6 ml of NMP. The mixture was heated for 30 min at 50°C and poured into 50 ml of cold water, and the precipitate was filtered off, dried, and recrystallized. Yield 73%. mp 137–138°C (from MeOH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 7.51 m (5H), 7.62 m (4H), 7.72 m (2H), 7.81 d (2H, ^4J = 2.3 Hz), 7.92 d (4H, ^3J = 8.1 Hz), 8.09 t (1H, ^4J = 2.1 Hz). Found, %: C 62.16; H 4.12; S 20.04. $C_{24}H_{18}O_4S_3$. Calculated, %: C 61.78; H 3.89; S 20.62.

1,3,5-Tris(phenylsulfonyl)benzene (XV). A mixture of 4.66 g (0.01 mol) of compound **XIV**, 3.4 ml of 35% hydrogen peroxide, and 70 ml of acetic acid was heated for 30 min under reflux. The mixture was cooled, and the precipitate was filtered off and dried. Yield 68%. mp 222–224°C; published data [16]: mp 218°C. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 7.69 m (9H), 8.09 m (6H), 8.28 s (1H), 8.60 s (2H). Found, %: C 58.23; H 3.98; S 18.87. $C_{24}H_{18}O_6S_3$. Calculated, %: C 57.81; H 3.64; S 19.29.

Reaction of 1,3,5-tris(phenylsulfonyl)benzene (XV) with benzenethiol. A solution of 4.98 g (0.01 mol) of compound **XV** in 4 ml of NMP was added to a mixture of 1.02 ml (0.01 mol) of benzenethiol, 1.38 g (0.01 mol) of K_2CO_3, and 6 ml of NMP. The mixture was stirred for 30 min at 50°C and poured into 50 ml of cold water, and the precipitate of bis-sulfone **XIV** was filtered off, dried, and recrystallized.

3-Phenylsulfonylanilines XVIa–XVId. A mixture of 0.015 mol of mononitrosulfone **V**, **VII**, **XII**, or **XIII**, 1.2 ml of hydrazine hydrate, 0.04 g of $FeCl_3 \cdot 6H_2O$, 0.5 g of charcoal, and 55 ml of methanol was heated for 3 h under reflux. It was then filtered, the filtrate was cooled, and the precipitate was filtered off and dried.

3-Phenoxy-5-phenylsulfonylaniline (XVIa). mp 156–157°C. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 5.78 s (2H), 6.36 t (1H, ^4J = 2.1 Hz), 6.57 t (1H, ^4J = 2.1 Hz), 6.85 t (1H, ^4J = 2.1 Hz), 7.04 d (2H, ^3J = 7.8 Hz), 7.20 t (1H, ^3J = 7.8 Hz), 7.41 m (2H), 7.65 m (3H), 7.87 d (2H, ^3J = 8.6 Hz). Found, %: C 66.92; H 4.97; N 4.99; S 9.27. $C_{18}H_{15}NO_3S$. Calculated, %: C 66.44; H 4.65; N 4.30; S 9.85.

3-Phenylsulfonyl-5-phenylthioaniline (XVIb). mp 134–135°C. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 5.82 s (2H), 6.64 t (1H, ^4J = 1.9 Hz), 6.81 t (1H, ^4J = 1.9 Hz), 6.96 t (1H, ^4J = 1.9 Hz), 7.40 m (5H), 7.65 m (3H), 7.82 d (2H, ^3J = 8.1 Hz). Found, %: C 63.94; H 4.85; N 4.80; S 18.06. $C_{18}H_{15}NO_2S_2$. Calculated, %: C 63.31; H 4.43; N 4.10; S 18.78.

3-Phenylsulfonyl-5-(2-pyridylthio)aniline (XVIc). Reaction time 3.5 h. Yield 64%. mp 126–127°C. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 5.91 s (2H), 6.94 m (1H), 7.10 m (3H), 7.20 m (1H), 7.66 m (4H), 7.89 m (2H), 8.42 m (1H). Found, %: C 60.15; H 4.80; N 8.62; S 17.91. $C_{17}H_{14}N_2O_2S_2$. Calculated, %: C 59.63; H 4.12; N 8.18; S 18.78.

3,5-Bis(phenylsulfonyl)aniline (XVId). mp 225–226°C. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 6.29 s (2H), 7.30 d (2H, ^4J = 2.3 Hz), 7.41 t (1H, ^4J = 2.1 Hz), 7.68 m (6H), 7.91 d (4H, ^3J = 8.1 Hz). Found, %: C 58.27; H 4.51; N 4.05; S 16.91. $C_{18}H_{15}NO_4S_2$. Calculated, %: C 57.89; H 4.05; N 3.75; S 17.17.

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