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Reaction of Vinyl Sulfoxides with Sodium Sulfide and Polysulfides

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Abstract—Sodium sulfide and polysulfides readily (50–55°C, 3 h, aqueous medium) react with alkyl vinyl sulfoxides to afford bis(alkylsulfinylethyl)sulfides and -polysulfides in up to 75% yield. Under comparable conditions the reaction of divinyl sulfoxide with sodium sulfide proceeds by the mechanism of addition-cyclization and results in 1,4-dithiane-1-oxide and 1,4-oxathiane-4-oxide. Microwave activation of the studied reactions allows to increase their rate and efficiency.

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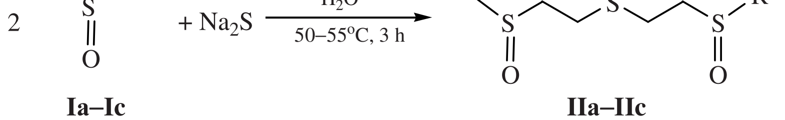
Organic sulfides and sulfoxides are intensely studied as synthons for organic synthesis [1], promising reagents for preparation of biologically active compounds [2] and materials for modern engineering processes [3, 4]. Combination of sulfide and sulfoxide functions in one molecule extends the fields of theoretical and practical use of these organosulfur compounds which already have turned out to be promising synthons and pharmacophore intermediates for preparation of functional nucleosides [5], as well as complexing solvents [6], effective extragents for noble metals [7], anti-scratch additives to oils [8, 9], flotoreagents [10], etc.

One of most convenient and atom-sparing methods of synthesis of these sulfidosulfoxides may be the reaction of available vinyl sulfoxides [11] with alkali metal sulfides and polysulfides. Vinyl sulfoxides, including divinyl sulfoxide easily prepared from acetylene, elemental sulfur and hydrogen peroxide in two steps [11, 12], under mild conditions add various *C*- [13], *N*- [4, 11, 12], *P*- [14], *O*- [11, 12, 15] and *S*-nucleophiles [11, 12, 16] to form functional, including cyclic sulfoxides [11, 17].

The goal of the present study was to obtain by an example of reaction of alkyl vinyl sulfoxides and divinyl sulfoxide with sodium sulfide and polysulfides new fundamental information on regularities of nucleophilic addition to vinyl sulfoxide group and development of a convenient method for the synthesis of new representatives of functional sulfoxides containing sulfide and polysulfide fragments. For activation of the process along with conventional heating we have used the method of microwave irradiation, which has already established itself in organic synthesis [18].

It was shown for the first time that alkyl vinyl sulfoxides **Ia–Ic** reacted with sodium sulfide under moderate heating (50–55°C, 3 h) in aqueous medium to afford the corresponding bis(alkylsulfinylethyl)sulfides **IIa–IIc** in good yield (55–75%).

Under the conditions of reaction (1) alkyl vinyl sulfoxides **Ia–Ic** react with sodium polysulfides prepared by heating sodium sulfide with two equivalents of elemental sulfur to afford the mixtures of the corresponding bis(alkylsulfinyl)sulfides **IIa–IIc**, -disulfides **IIIa–IIIc** and -trisulfides **IVa–IVc** in 51–70% total yield.

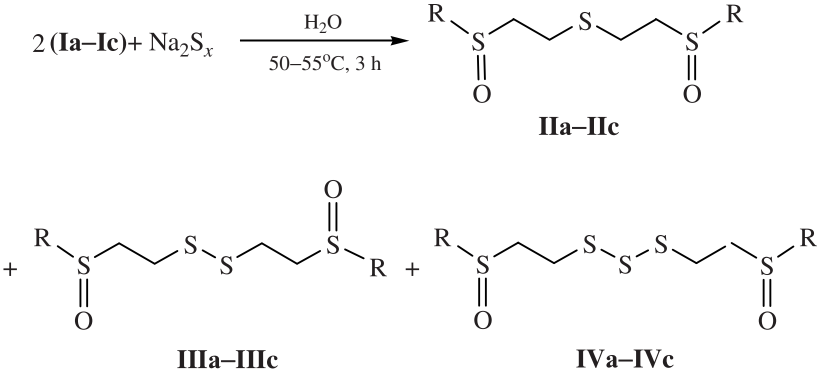


(1)

Ia–Ic **IIa–IIc**

R = Et (**Ia, IIa**); Pr (**Ib, IIb**); Bu (**Ic, IIc**).

In the IR spectra of diadducts **IIa–IVc** the absorption bands are observed at 643–647 cm^{-1} (stretching vibrations of C–S bond), 500–504 and 472–476 cm^{-1} that according to [19] can be assigned to the stretching vibrations of S–S bonds in disulfides **III** and trisulfides **IV**, respectively. The ^{1}H NMR data also confirm the formation of mono- (**II**), di- (**III**) and trisulfides **IV** in reaction (2).

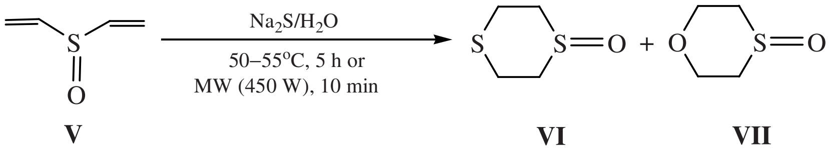


(2)

R = Et (**a**); Pr (**b**); Bu (**c**); x = 1–3.

Microwave activation (100 and 450 W) of the addition reaction of sodium sulfide and polysulfides to alkyl vinyl sulfoxides allows to reduce the time of contact of the reagents to 2–10 min and increase the yield of the corresponding diadducts, alkylsulfidosulfoxides, to 75–92%.

The reaction of divinylsulfoxide **V** with sodium sulfide (50–55°C, 5 h, water) proceeds by the scheme of addition-cyclization and leads to 1,4-dithiane-1-oxide **VI** and 1,4-oxathiane-4-oxide **VII**) in 22% total yield. The obtained heterocycles **VI, VII** are easily separated by sublimation. Microwave irradiation (450 W) of the reaction mixture allows to shorten the reaction time to 10 min and increase the yield of products **VI, VII** to 56%.

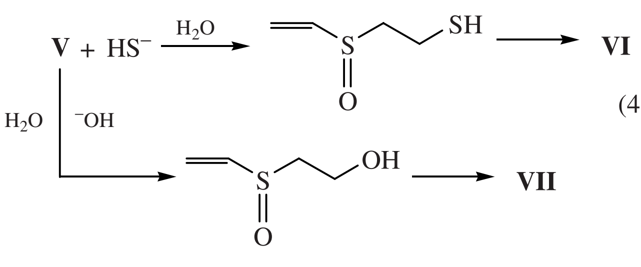


(3)

Since reaction (2) proceeds in aqueous medium where after hydrolysis of sodium sulfide two active nucleophiles (HS$^-$ and $^-$OH) are formed, the following scheme can be suggested for the formation of compounds **VI** and **VII** including the stage of addition of sulfide or hydroxide anions to the vinylsulfoxide group with subsequent cyclization of the monoadducts formed.

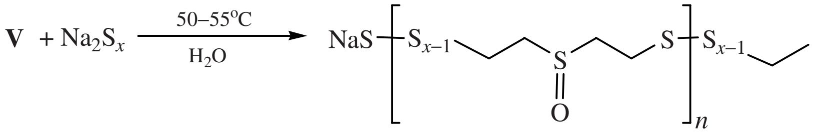
With sodium di-, tri- and tetrasulfidies divinylsulfoxide forms oligopolysulfidosulfoxides **VIIIa–VIIIc**, thiocols of a new type. The reaction proceeds under moderate heating (50–55°C) in aqueous medium within 3–3.5 h, the yield of oligomers **VIIIa–VIIIc** is 64–72%.

$$S^{2-} + H_2O \longrightarrow HS^- + {}^-OH$$



(4)

Polysulfidosulfoxides **VIIIa–VIIIc** are viscous compounds of dark-brown color, poorly soluble in water and insoluble in organic solvents. Their polymeric



(5)

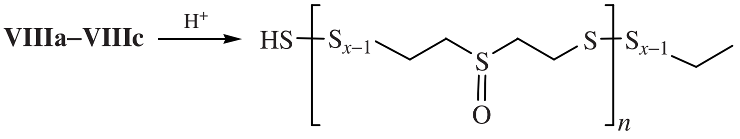
VIIIa–VIIIc

$x = 2$ (**VIIIa**), 3 (**VIIIb**), 4 (**VIIIc**).

structure is confirmed by the data of IR spectroscopy: high background and wide blurred absorption bands. Besides, in the IR spectra of these compounds the absorption band is present at 1020–1040 cm^{-1} corresponding to the stretching vibrations of the S=O group.

Upon long storage (several years) in closed vessels sulfidosulfoxides **VIIIa–VIIIc** practically do not change their consistence.

Treatment of polysulfidosulfoxides **VIIIa–VIIIc** with hydrochloride acid (to pH = 1) results in oligothiosulfoxides **IXa–IXc**, insoluble in water and organic solvents rubber-like dark substances, apparently having the following structure:



(6)

IXa–IXc

$x = 2$ (**IXa**), 3 (**IXb**), 4 (**IXc**).

Similar oligoethylthiosulfoxides were obtained earlier from divinylsulfoxide and dithiols [20]. In the IR spectra of these compounds the absorption band at 1020–1040 cm^{-1} corresponding to S=O group is observed. The data of elemental analysis are in agreement with the suggested structure of these oligomers. Upon storage in closed vessels they are rapidly converted into solid bituminous products. However, in the presence of hydroquinone, oligomers **IXa–IXc** retain their original elastic structure during one year. This apparently is indicative of the fact that mercapto groups of oligoethylthiosulfoxides **IXa–IXc** are readily oxidized to give disulfide fragments.

Sulfidosulfoxides **IIb–IVc** were tested as sulfur-containing additives in grease compositions on the bases of low-molecular ethylene, waste products from high-pressure polyethylene, and used diesel oil of locomotives. The tests were carried out on a frictional testing machine MI–1M. The results are indicative of high tribotechnical characteristics of the synthesized organosulfur compounds **IIb–IVc** which can be recommended as effective anti-scratch and antiwear additives, improving the operating characteristics of lubricants [9].

Therefore, on the basis of the reaction of sodium sulfides and polysulfides with alkyl vinyl sulfoxides and divinylsulfoxide a convenient and effective method was developed for the synthesis of organosulfur compounds combining the sulfide and sulfoxide functions in one molecule, promising anti-scratch additives to oils, complexing solvents, extragents, flotoreagents, as well as intermediates and synthons for synthesis of new medical and agricultural drugs.

EXPERIMENTAL

^1H NMR spectra were registered on a Bruker DPX 400 spectrometer (400 MHz) from solutions in $CDCl_3$, internal reference HMDS. IR spectra were obtained on a Bruker-IFS 25 instrument in KBr pellets. Electron impact mass spectra were recorded on a GCMS-QP5050A SHIMADZU instrument with the energy of ionizing electrons 70 eV. Microwave assisted reactions were carried out in a modified domestic microwave oven Samsung 181 DNR.

Reactions of alkyl vinyl sulfoxides with sodium sulfide and polysulfides *(general procedure)*. To the solution of 0.005 mol of sodium sulfide or polysulfides (prepared by heating of sodium sulfide with two equivalents of elemental sulfur) in 5 ml H_2O heated at 45°C 0.01 mol of sulfoxide **Ia–Ic** was added, the mixture was stirred at 50–55°C during 3 h. The reaction mixture was cooled, diluted with ~10 ml of water, extracted with chloroform (5 × 10 ml), dried over potassium carbonate. Chloroform was removed under reduced pressure, the residue dried in a vacuum to give the corresponding sulfidosulfoxides **IIa–IIc–IVa–IVc**.

Ethyl 2-[2-(ethylsulfinyl)ethyl]sulfanylethyl sulfoxide (IIa). Yield 55%, yellow powder, mp 59–62°C. IR spectrum (KBr, cm^{-1}): 2950, 2920, 2870 ν(CH, CH_2); 1450, 1410, 1370, 1280 δ(CH, CH_2); 1020 ν(S=O); 960 ν(C–C); 640, 530 ν(C–S). ^1H NMR, δ, ppm: 1.35 t (6H, Me, $^3J_{HH}$ 7.45 Hz), 2.75 q (4H, CH_2Me, $^3J_{HH}$ 7.45 Hz), 2.91 m (4H, SCH_2CH_2SO), 3.01 m (4H, SCH_2). Found, %: C 39.83; H 7.85; S 37.92. M 242.41. $C_8H_{18}O_2S_3$. Calculated, %: C 39.64; H 7.48; S 39.68.

Propyl 2-[2-(propylsulfinyl)ethyl]sulfanylethyl sulfoxide (IIb). Yield 74%, yellow powder, mp 59–60°C. IR spectrum (KBr, cm^{-1}): 2955, 2930 , 2870 ν (CH, CH_2); 1460, 1400, 1370, 1300 δ(CH, CH_2); 1015 ν(S=O); 955 ν(C–C); 669, 642 ν(C–S). ^1H NMR, δ, ppm: 1.09 t (6H, Me, $^3J_{HH}$ 7.39 Hz), 1.82 q (4H, CH_2Me, $^3J_{HH}$ 7.39 Hz), 2.63 and 2.76 m (4H, CH_2SO), 2.91 m (4H, SCH_2CH_2SO), 3.00 m (4H, SCH_2). Found, %: C 44.17; H 8.60; S 34.49. M 270.46. $C_{10}H_{22}O_2S_3$. Calculated, %: C 44.41; H 8.20; S 35.56.

Butyl 2-[2-(butylsulfinyl)ethyl]sulfanylethyl sulfoxide (IIc). Yield 75%, white powder, mp 96–98°C. IR spectrum (KBr, cm^{-1}): 2950, 2920, 2870 ν(CH, CH_2); 1470, 1400, 1370, 1300 δ(CH, CH_2); 1019 ν(S=O); 955 ν(C–C); 700, 669, 644 ν(C–S). ^1H NMR, δ, ppm: 1.01 t (6H, Me, $^3J_{HH}$ 7.25 Hz), 1.54 m (4H, CH_2Me), 1.80 m (4H, CH_2), 2.77 m (4H, CH_2SO), 2.97 m (4H, SCH_2CH_2SO), 3.05 m (4H, SCH_2). Found, %: C 47.84; H 8.99; S 32.57. M 298.52. $C_{12}H_{26}O_2S_3$. Calculated, %: C 48.28; H 8.78; S 32.22.

Ethylsulfidosulfoxide (IIa–IVa). Yield 51%, brown powder, mp 92–96°C. IR spectrum (KBr, cm^{-1}): 2950, 2920, 2900, 2870 ν(CH, CH_2); 1450, 1410, 1370, 1280 δ(CH, CH_2); 1010 ν(S=O); 960 ν(C–C); 790, 730, 650 ν(C–S). ^1H NMR, δ, ppm: 1.35 m (Me in **IIa**), 1.37 m (Me in **IIIa**), 1.40 m (Me in **IVa**), 2.71 m (CH_2Me), 2.88 m (SCH_2CH_2SO), 2.98 m (SCH_2). Found, %: C 36.74; H 6.79; S 43.26. M 258.44. $C_8H_{18}O_2S_{3.5}$. Calculated, %: C 37.18; H 7.02; S 43.42.

Propylsulfidosulfoxide (IIb–IVb). Yield 56%, brown powder, mp 62–66°C. IR spectrum (KBr, cm^{-1}): 2950, 2920, 2900, 2865 ν(CH, CH_2); 1470, 1410, 1375, 1280 δ(CH, CH_2); 1012 ν(S=O); 1000 ν(C–C); 780, 698, 681, 643 ν(C–S); 576, 504 ν(S–S in **IIIb**); 492, 472, 454 ν(S–S in **IVb**). ^1H NMR, δ, ppm: 1.09 m (Me in **IIb**), 1.10 m (Me in **IIIb**), 1.11 m (Me in **IVb**), 1.82 m (CH_2Me), 2.64 and 2.76 m (CH_2SO), 2.90 m (SCH_2CH_2SO), 3.00 m (SCH_2). Found, %: C 39.87; H 7.39; S 42.32. M 302.52. $C_{10}H_{22}O_2S_4$. Calculated, %: C 39.70; H 7.33; S 42.39.

Butylsulfidosulfoxide (IIc–IVc). Yield 70%, brown powder, mp 76-78°C. IR spectrum (KBr, cm^{-1}): 2950, 2920, 2860 ν(CH, CH_2); 1470, 1450, 1410, 1310, 1300, 1280 δ(CH, CH_2); 1022 ν(S=O); 955 ν(C–C); 700, 647 ν(C–S); 573, 504 ν(S–S in **IIIc**); 476 ν(S–S in **IVc**). ^1H NMR, δ, ppm: 0.92–1.02 m (Me), 1.35–1.53 m (CH_2Me), 1.65–1.77 m (CH_2), 2.57–2.75 m ($CH_2\cdot CH_2SO$), 2.83–3.05 m [$S(O)CH_2CH_2SCH_2CH_2S(O)$]. Found, %: C 45.24; H 8.59; S 35.80. M 314.54. $C_{12}H_{26}O_2S_{3.5}$. Calculated, %: C 45.82; H 8.33; S 35.68.

Reaction of alkyl vinyl sulfoxides with sodium sulfide and polysulfides under microwave irradiation *(general procedure)*. The solution of 0.004 mol of sodium sulfide and polysulfides (prepared by heating of sodium sulfide with two equivalents of elemental sulfur) in 3 ml H_2O and 0.008 mol of alkyl vinyl sulfoxide **Ib** and **Ic** were placed into 100 ml round-bottom flask equipped with condenser; the reaction mixture was stirred and irradiated in a microwave oven (100 W, 10 min, or 450 W, 2 min). The reaction mixture was cooled, diluted with water (~10 ml), extracted with chloroform (5 × 10 ml) and dried over potassium carbonate. Chloroform was removed under reduced pressure, the residue dried in vacuum. The corresponding propylsulfidosulfoxide **IIb–IVb** and butylsulfidosulf-oxide **IIc–IVc** were obtained in 75% and up to 92% yield, respectively.

Reaction of divinylsulfoxide with sodium sulfide. *a*. The solution of 0.72 g of $Na_2S \cdot 9H_2O$ and 0.305 g of sulfoxide **V** in 10 ml H_2O was stirred at 50–55°C during 5 h. The reaction mixture was cooled, diluted with water (~10 ml), extracted with chloroform (5 × 10 ml) and dried over potassium carbonate. Chloroform was removed under reduced pressure, the residue was dried in a vacuum. We obtaind 0.09 g (22%) of the mixture of heterocycles **VI** and **VII** in the 12:1 ratio (according to GC–MS). Compounds **VI** and **VII** were separated by sublimation.

1,4-Dithiane-1-oxide (VI). Colorless crystals, mp 125–126°C (125–127°C [21]). IR spectrum (KBr, cm^{-1}): 2962, 2913, 2861 ν(CH_2); 1744, 1462, 1406, 1324 δ (CH_2); 1274, 1212, 1169, 1128; 1021 ν(S=O); 628, 510 ν(C–S). ^{1}H NMR, δ, ppm: 2.50–2.54 m (2H, CH_2S), 2.96–3.02 m (2H, CH_2SO), 3.09–3.14 m (2H, CH_2SO), 3.54–3.61 m (2H, CH_2S). Found, %: C 35.66; H 5.88; S 46.83. *M* 136.22. $C_4H_8OS_2$. Calculated, %: C 35.27; H 5.92; S 47.07. Mass spectrum (EI, 70 eV), *m/z* ($I_{rel.}$, %): 136 $[M]^+$ (53), 119 (4), 108 (3), 92 (8), 87 (43), 77(56), 73 (24), 64 (9), 63 (15), 61 (25), 60 (98), 59 (72), 58 (24), 53 (5), 50 (3), 48 (13), 47 (22), 46 (42), 45 (100), 41 (12), 35 (5).

1,4-Oxathiane-4-oxide (VII). Transparent needle crystals, mp 40–41°C (40–42°C [22]). IR spectrum (KBr, cm^{-1}): 2962, 2922, 2865 ν(CH_2); 1744, 1463, 1405, 1321 δ(CH_2); 1276, 1213, 1149, 1097 ν(COC); 1063, 1021 ν(S=O). ^{1}H NMR, δ, ppm: 2.77–2.72 m (2H, CH_2SO); 2.97–2.89 m (2H, CH_2SO); 3.86–3.80 m (2H, CH_2O); 4.42-4.36 m (2H, CH_2O). Mass spectrum, (EI, 70 eV), *m/z* ($I_{rel.}$, %): 120 $[M]^+$ (33), 104 (2), 92 (98), 77(31), 76 (25), 63 (24), 62 (8), 61 (8), 59 (43), 50 (14), 48 (42), 47 (56), 46 (35), 45 (35), 43 (100), 42 (10).

b. The solution of 0.72 g of $Na_2S{\cdot}9H_2O$ in 10 ml H_2O and 0.305 g of sulfoxide **V** were placed into 100 ml round-bottom flask equipped with condenser; the reaction mixture was stirred and irradiated in a microwave oven (450 W, 10 min). The reaction mixture was cooled, diluted with water (~10 ml), extracted with chloroform (5 × 10 ml) and dried over potassium carbonate. Chloroform was removed under reduced pressure, the residue dried in a vacuum. 0.224 g (56%) of the mixture of heterocycles **VI** and **VII** were obtained in the 5:1 ratio (according to GC–MS).

Oligopolysulfidosulfoxides VIIIa–VIIIc *(general procedure)*. To the solution of 0.15 mol of the corresponding sodium polysulfide in 25 ml of water at heating (50°C) 0.15 mol of sulfoxide **V** was added, the mixture was stirred for 3–3.5 h at 50–55°C, and poured into acetone. The precipitate formed was washed with acetone, ether, and dried in vacuum to afford the corresponding oligomer.

Oligodisulfidosulfoxide (VIIIa). Yield 72%. Found, %: C 26.92; H 4.90; S 55.73. $C_4H_8OS_3$. Calculated, %: C 28.55; H 4.79; S 57.15.

Oligotrisulfidosulfoxide (VIIIb). Yield 68%. Found, %: C 21.03; H 4.67; S 63.17. $C_4H_8OS_4$. Calculated, %: C 23.98; H 4.02; S 64.01.

Oligotetrasulfidosulfoxide (VIIIc). Yield 64%. Found, %: C 21.17; H 4.25; S 68.53. $C_4H_8OS_5$. Calculated, %: C 20.67; H 3.47; S 68.97.

Oligoethylthiosulfoxides IXa–IXc *(general procedure)*. To the solution of 0.15 mol of the corresponding sodium polysulfide in 25 ml of water at heating (50°C) 0.15 mol of sulfoxide **V** was added, the mixture was stirred for 3–3.5 h at 50–55°C and acidified with concn. hydrochloric acid (to pH = 1). The precipitate formed was washed with water to neutral reaction, with acetone, ether, dried in a vacuum to obtain the corresponding oligomer.

Oligoethylthiosulfoxide (IXa). Yield 64%. Found, %: C 28.59; H 4.98; S 56.47. $C_4H_8OS_3$. Calculated, %: C 28.55; H 4.79; S 57.15.

Oligoethylthiosulfoxide (IXb). Yield 55%. Found, %: C 23.57; H 4.41; S 63.36. $C_4H_8OS_4$. Calculated, %: C 23.98; H 4.02; S 64.01.

Oligoethylthiosulfoxide (IXc). Yield 60%. Found, %: C 20.13; H 3.86; S 68.24. $C_4H_8OS_5$. Calculated, %: C 20.67; H 3.47; S 68.97.

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