tion of their melting points and refraction indices with those published earlier.[2–4]

Experimental

^{1}H NMR spectra were registered on a «Bruker AM-300» instrument for solutions in D_2O and acetone-d_6 with TMS as the internal standard.

Potassium 2,3-dihydroxypropylsulfamate (2). KOH (0.7 g, 10.8 mmol, 85 %) was added to solution of **1a** (2.28 g, 10 mmol) in H_2O (10.8 mL, 0.6 mol) and the mixture was stirred for 3—4 h at 100 °C. The solution was evaporated and the residue was recrystallized from MeOH to give potassium 2,3-dihydroxypropylsulfamate (1.92 g, ~91%), m.p. 116—118 °C (*cf.* Ref. 2). Compounds **2c—d** were prepared in a similar manner.

3-Methylaminopropane-1,2-diol (3b). Compound **2a** (4.46 g, 20 mmol) was added to a 30% solution of H_2SO_4 (10 mL) and the mixture was stirred for 8 h at 100 °C and neutralized by a solution of KOH to pH ~12.0. The reaction mixture was evaporated and the product was extacted with EtOH—Et_2O. This led to product **3b** (1.99 g, ~95%) with b.p. 98—102 °C (3—4 Torr) (Ref. 3: b.p. 239—241 °C (748 Torr)).

In a similar manner compounds **3a,c,d** were prepared: **3a**, b.p. 127—130 °C (2—3 Topp) (Ref. 4: b.p. 125—128 °C (2 Torr)); **3c**, b.p. 246—248 °C (758 Torr) (Ref. 3: b.p. 244—246 °C (750 Torr)). Compound **3d**, b.p. 106—109 °C (2—3 Torr). ^{1}H NMR (acetone-d_6, δ, ppm): 0.8—1.5 (m, 7 H, Pr), 2.40—2.65 (m, 4 H, CH_2NCH_2), 3.68 (m, 2 H, CH_2OH), 3.98 (m, 1 H, CHOH). Found (%): N, 10.02. $C_7H_{17}NO_2$. Calculated (%): N, 9.51.

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Interaction of diphenyl sulfide with sulfuryl chloride

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The reaction of diphenyl sulfide with sulfuryl chloride was studied. Depending on the reaction conditions, it occurs either as chlorination of the aromatic ring or as oxidation to give sulfoxide.

Key words: diphenyl sulfide, sulfuryl chloride, oxidation, chlorination.

Reactions of aromatic thioethers with sulfuryl chloride have not been studied much,[1] although reactions involving other electrophilic reagents, including halogens, have been covered adequately.[2] We studied the effect of the reaction conditions on the nature of the products of the interaction between diphenyl sulfide **1** and SO_2Cl_2.

When this reaction is carried out in inert organic solvents (dichloromethane, tetrachloromethane), it appears that a hydrolytically unstable intermediate of the sulfurane type is formed initially; the latter readily decomposes to give diphenyl sulfoxide (**2**). Thus, this process can serve as a convenient method for sulfoxidation of aromatic sulfides that does not involve undesirable further oxidation to sulfones, typical of methods based on the use of traditional oxidizing reagents.

When aluminum chloride is added to the reaction system, both the sulfur atom and the aromatic ring of sulfide **1** are chlorinated to give 4,4′-chlorodiphenyl sulfoxide (**3**), whose mass spectrum has an intense $[M-SO]^+$ peak and no molecular ion peak.

The reaction carried out without a solvent at a **1** : SO_2Cl_2 molar ratio of 1 : 2 gave 4,4′-dichlorodiphenyl sulfide (**4**) in a yield of 92%. Increasing the

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amount of SO_2Cl_2 and the reaction duration results in the formation of a low-melting mixture of polychlorinated compounds that was difficult to separate and which contained, according to mass spectrometry, tri-, tetra-, and pentachlorodiphenyl sulfides, in addition to sulfide **4**. A similar mass-spectrometric pattern was obtained for the mixture of products obtained in the reaction of compound **4** and SO_2Cl_2.

The use of the reagent BMS (a mixture of SO_2Cl_2, sulfur monochloride, and aluminum chloride) results in the exhaustive chlorination of sulfide **1** to give perchlorodiphenyl sulfide (**5**). According to previously reported data,[3] in this case, the SCl_3^+ or SO_2Cl^+ cation serves as the attacking electrophilic species. We did not observe cleavage of the sulfide bond to afford triphenylsulfonium salts under the action of the Friedel—Crafts catalyst, which is typical of chlorination of compound **1** with molecular chlorine.[4]

It is of interest that chlorination of sulfide **1** with thionyl chloride involves exclusively the aromatic ring irrespective of the reaction conditions, which is explained by the deoxygenating action of thionyl chloride on sulfoxides.

Experimental

Mass spectra were recorded on a Kratos MS-908 instrument (70 eV). The course of the reactions was monitored and the purity of the compounds synthesized was checked by TLC on Silufol UV-254 plates using a 9 : 1 chloroform—acetone mixture as eluent. Sulfide **1**, SO_2Cl_2, sulfur monochloride, and thionyl chloride were distilled twice prior to use. Compounds **3** and **4** were identified by comparison with authentic samples obtained by a known procedure.[5,6]

Diphenyl sulfoxide (2). At 0—5 °C, SO_2Cl_2 (2 mL, 25 mmol) was added dropwise to a solution of sulfide **1** (2.7 g, 10 mmol) in 50 mL of CCl_4 with intense stirring. 2 h later the solvent was evaporated on a rotary evaporator, and the residue was dried and recrystallized from $CHCl_3$ to give sulfoxide **2** in 74% yield; m.p. 99—100 °C (published data:[5] 98—99 °C). MS, m/z (I_{rel} (%)): 254 $[M-O]^+$ (100). Found (%): S, 11.93; Cl, 26.13. $C_{12}H_8Cl_2OS$. Calculated (%): S, 11.85; Cl, 26.29.

Perchlorodiphenyl sulfide (5). At -20 °C, a mixture of SO_2Cl_2 (7 mL, 87.5 mmol) and sulfur monochloride (5 mL, 62.5 mmol) was added dropwise to a suspension of anhydrous aluminum chloride (1.0 g, 7.5 mmol) in a solution of sulfide **1** (2.7 g, 10 mmol) in 50 mL of CCl_4. The reaction mixture was allowed to stand for 24 h, and the precipitate that formed was filtered off, dried, and extracted with acetone in Sohxlet apparatus. Sulfide **5** was obtained in 67% yield, m.p. 306—308 °C (from acetone). MS, m/z (I_{rel} (%)): 526 $[M]^+$ (100), 491 $[M-Cl]^+$ (86). Found (%): S, 5.98; Cl, 66.44. $C_{12}Cl_{10}S$. Calculated (%): S, 6.03; Cl, 66.85.

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