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LETTERS TO THE EDITOR

Reaction of 2,3-Dichloroprop-1-ene with Sulfur in Hydrazine Hydrate–Monoethanolamine System

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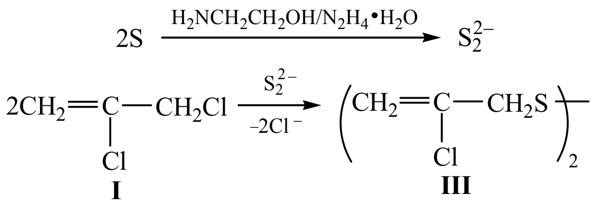
We have previously described the reaction of 2,3-dichloroprop-1-ene **I** with sulfur in hydrazine hydrate–KOH system yielding bis(2-chloroprop-1-en-3-yl) sulfide **II** [1]. In this system at the molar ratio KOH:S = 1:1 sulfur is reduced with the preferential formation of disulfide anions [2], but the subsequent reaction with dichloropropene **I** gives exclusively monosulfide derivative **II** in 78% yield. It has been suggested that the latter is formed due to the instability of the corresponding disulfide that contains the chlorine atoms and the double bond in β-position to the disulfide bridge [1].

In this study we unexpectedly found that performing the reaction of 2,3-dichloroprop-1-ene **I** with sulfur in the system hydrazine hydrate–monoethanolamine, where preferential reduction of sulfur to the disulfide-anions was also observed [3], led to the formation of bis(2-chloroprop-1-en-3-yl) disulfide **III** in 70% yield.



Disulfide **III** proved to be a stable compound and was isolated in individual state by a vacuum distillation. Reasons for different directions of this reaction depending on the type of the base-reduction system are currently investigated.

To a solution containing 1.8 ml of monoethanolamine and 15 ml of hydrazine hydrate was added 2.9 g of powdered sulfur at 60–65°C. The mixture was stirred for 2 h, after which 7.13 g of 2,3-dichloroprop-1-ene **I** was added at 25°C, and the stirring was continued for 3 h. The resulting organic layer was separated. Disulfide **III** was isolated by the vacuum distillation, bp 108–111°C (2 mm Hg). IR spectrum (thin layer), ν, cm^{-1}: 3109, 3021, 2968, 2916, 1628, 1605, 1405, 1373, 1265, 1204, 891, 799, 776, 759, 710, 682, 670, 629, 515, 480. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 3.52 s (2H, CH_2S), 5.32 d, 5.43 br. s (2H, $CH_2=$, ^2J 1.4 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 47.13 (CH_2S), 116.34 ($CH_2=$), 137.24 (=CCl). Mass spectrum, *m/z* (for the Cl-containing ions the data are given for ^{35}Cl) (*I*, %): 179 (0.5) $[M - Cl]^+$, 154 (1.1), 149 (14.2), 137 (1.9) $[C_3H_2S_2Cl]^+$, 115 (7.9) $[CH_4S_2Cl]^+$, 103 (6.5), 75 (26.2) $[CH_2CClCH_2]^+$, 64 (0.5) $[S_2]^+$, 45 (15.1) $[CHS]^+$, 39 (26.0) $[C_3H_3]^+$. Found, %: C 33.54; H 4.02; S 28.93; Cl 32.24. $C_6H_8Cl_2S_2$. Calculated, %: C 33.49; H 3.72; S 29.77; Cl 33.02.

The IR spectra were recorded on a Bruker IFS-25 spectrometer. The ^1H, ^{13}C NMR spectra were registered on a Bruker DPX-400 spectrometer operating at 400.13 and 100.62 MHz, respectively, in $CDCl_3$ with internal reference HMDS. The mass spectra were taken on a chromato-mass-spectrometer Shimadzu GCMS–QP5050A (column SPB-5, 60000× 0.25 mm), quadrupole mass analyzer, electron ionization (70 eV, ion source temperature 190°C, the range of detected mass 34-650 Da).

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