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RECYCLIZATION REACTIONS. SYNTHESIS OF 3-(2,3-EPITHIOPROPYL)-BENZOTHIAZOL-2-ONE FROM 2-(ALLYLTHIO)BENZOTHIAZOLE

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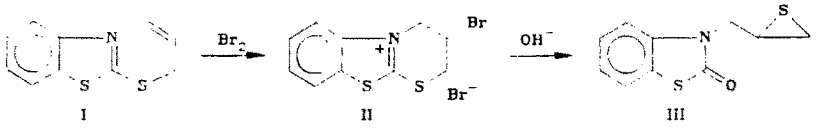
We have established that 3-(2,3-epithiopropyl)benzothiazol-2-one (III) is obtained in satisfactory yield by bromination of 2-(allylthio)benzothiazole (I) and subsequent alkaline treatment of reaction product II.



I II III

The formation of salt II, which ensures the success of this method for the synthesis of thiiranes, is not predetermined unambiguously by the known analogies [1, 2], according to which the product of the corresponding bromomethylthiazolidinium salt was most likely. However, the recyclization of salt II to thiirane III apparently takes place in the ion pair of the initially formed pseudobase.

The cyclization of I was accomplished by the action of bromine on a solution of allylthio derivative I in dioxane or in acetic acid at 20°C. To complete the process, the reaction mixture was then refluxed for 3-4 h. The recyclization of salt II was carried out by the action of sodium hydroxide on a solution of the salt in epichlorohydrin or in the two-phase ether–water system at room temperature.

2-(Allylthio)benzothiazole (I, $C_{10}H_{15}NS_2$). This compound was obtained by the action of allyl bromide on the triethylammonium salt of benzothiazole-2-thione in DMF at 20°C. The product was obtained in 96% yield and had R_f 0.97 [here and subsequently, on Silufol with elution with chloroform–methanol (10:1)]. IR spectrum (thin layer): 1640 (m, C=C), 2860 (w), 2980 (w, CH_2), 2922 (m, CH), 3015 (w), 3080 (m, $CH=CH_2$), 3030 (w), 3065 cm^{-1} (m, CH_{arom}). PMR spectrum ($CDCl_3$): 3.80 (2H, d, CH_2), 4.90-5.30 (2H, m, CH_2=C), 5.50-6.30 (m, 1H, CH=C), 7.13-7.80 ppm (4H, m, H_{arom}).

3-Bromo-3,4-dihydro-2H-benzothiazolo(2,3-b)(1,3)thiazinium Bromide (II, $C_{10}H_9Br_2NS_2$). This compound was obtained in 52% yield and had mp 221-222°C (from acetic acid) and R_f 0.03. IR spectrum (in Nujol): 770 (m, C–Br), 1500 cm^{-1} (sh, aromatic). PMR spectrum (d_6-DMSO): 3.96 (2H, m, CH_2–S), 5.06 (2H, m, CH_2–N), 5.36 (1H, m, CH–Br), 7.76 (2H, m, H_{arom}), 8.26 ppm (2H, m, H_{arom}).

3-(2,3-Epithiopropyl)benzothiazol-2-one (III). This compound was obtained in 92% yield and had mp 50-51°C (from hexane) and R_f 0.95. IR spectrum (thin layer): 3070 (w, CH_2–S), 1682 cm^{-1} (s, C=O). PMR spectrum ($CDCl_3$): 2.53 (2H, d, CH_2–S), 3.30 (1H, m, CH–S), 4.17 (2H, d, CH_2–N), 7.10 ppm (4H, m, H_{arom}). No melting-point depression was noted for a mixture of this product with III obtained in accordance with [3].

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