REACTION OF BENZIMIDAZOLE-2-THIONE WITH 1,2-DIBROMOETHANE. CORONAND SYNTHESIS.

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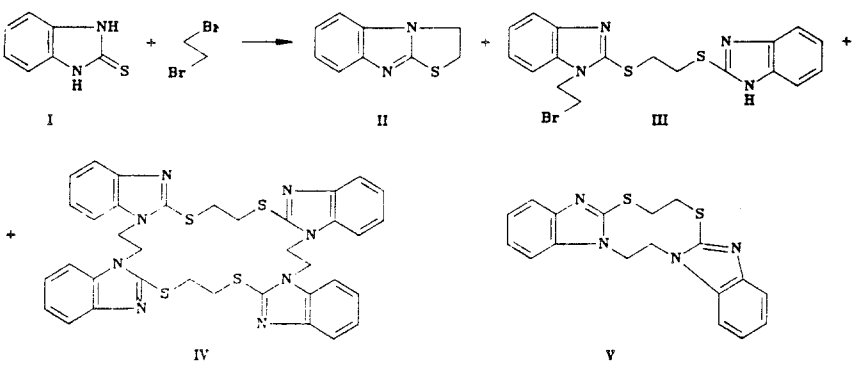
Benzimidazole-2-thione (I) reacts with 1,2-dibromoethane at room temperature in the presence of base to give compounds II and III, as well as coronand-20 (IV).



Compound II, which is soluble in cold dilute HCl, can be separated from the mixture of compounds III and IV, and can then be purified by column chromatography (Al_2O_3, ether) and precipitated with water from alcohol (5% yield, mp 90°C). The mixture of compounds III and IV can be dissolved in hot dilute HCl, cooled, and the precipitate of the hydrochloride of III removed [yield 54%, mp 215°C (from ethanol)]; the mother liquor contains only compound IV [yield 20%, mp 108°C (from CCl_4)]. Attempts to synthesize coronand IV via cyclodimerization of compound III in basic media were unsuccessful: treatment of compound III with KOH in DMSO solution with heating resulted in intramolecular cyclization to give coronand-10 [V, yield 76%, mp 110°C (from ethyl acetate)]. The results of elemental analysis, and the IR, PMR and mass spectral data, confirmed the structures of the newly synthesized compounds.

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