SYNTHESIS OF A NEW HETEROCYCLIC SYSTEM — SELENATHIETANE

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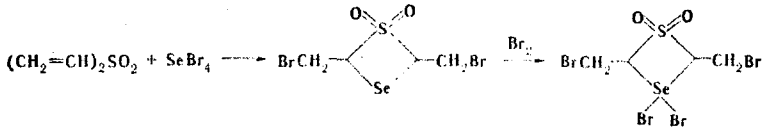
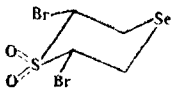
We have established that the reaction of selenium tetrabromide with divinyl sulfide leads to the formation of a crystalline substance, with mp 112-113°C (from $CHCl_3$) and R_f 0.56 [Al_2O_3 and ether–hexane (2:1)], in 81% yield. The IR spectrum of this compound contains absorption bands at 1100 (SO_2 group) and 730 cm^{-1} (C–Se stretching vibrations). On the basis of the PMR spectral data, the 3,3-dihydroxy-2,4-bis(bromomethyl)-1,3-selenathietane structure (I) was proposed for the isolated substance.



II

The PMR spectrum of I contains two groups of signals of a typical ABX system, analysis of which gives the following spin–spin coupling constants (SSCC) and chemical shifts of the protons: J_{AX} = 7.9 Hz, J_{BX} = 7.6 Hz, J_{AB} = 10.9 Hz, δ_{A-H} 3.72, δ_{B-H} 4.10, and δ_{X-H} 5.55 ppm. The close J_{AX} and J_{BX} values provide unambiguous evidence in favor of structure I (in which A–H and B–H are diastereotopic protons) and make it possible to exclude alternative structure III from examination. In saturated six-membered cyclic systems one of the J_{AX} constants is usually averaged because of ring inversion, whereas J_{BX} remains approximately constant at, as a rule, 2-4 Hz. The SSCC found are in good agreement with substituted selenathietane structure I.

Bromination of I gave II, with mp 122-123°C ($CHCl_3$), in quantitative yield. PMR spectrum (80 MHz, $CDCl_3$, d_6-DMSO): 5.63 (dd, X part of an ABX system, 2H, J_{AX} = 6.9 Hz, J_{BX} = 8.9 Hz); the AB part was observed as two doublets of doublets (dd) of identical intensity at 4.07 and 3.82 ppm with J_{AB} = 10.5 Hz. The individuality of the compounds was confirmed by thin-layer chromatography. The results of elementary analysis were in agreement with the calculated values.



III

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