SIMPLE SYNTHESIS OF 2-METHYL-3,4-EPOXY-1-BUTENE

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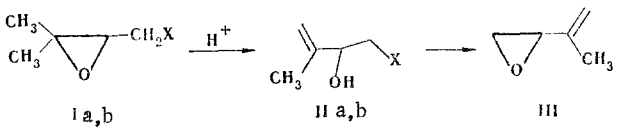
Because of the lack of a convenient preparative method for the synthesis of 2-methyl-3,4-epoxy-1-butene (III), up until now there have been no reliable data [1, 2] even regarding the physicochemical characteristics of this isoprene oxide.

We have shown that oxide III can be obtained by isomerization of readily accessible 1-halo-3-methyl-2,3-epoxybutanes (I) [3] under the influence of acidic catalysts to give unsaturated halohydrins II and by dehydrohalogenation of the latter:



I a,b II a,b III

I, II a X = Cl; b X = Br

A mixture of 0.1 mole of I and 0.33 g of p-toluenesulfonic acid in 20 ml of benzene was heated at 70-80°C for 45-60 min, after which the catalyst was neutralized with aqueous sodium carbonate solution, and alcohol II was isolated by distillation after drying. Compound IIa, with bp 152-154°C (653 mm), n_D^{20} 1.4650, and d_4^{20} 1.0809, was obtained in 75% yield. PMR spectrum (CCl_4): 4.92 and 5.01 (2H, m, $=CH_2$); 4.18 (1H, t, CH); 3.53 (1H, s, OH); 3.44 (2H, d, CH_2); 1.71 ppm (3H, d, CH_3). Compound IIb, with bp 71-72°C (14 mm), n_D^{20} 1.4990, and d_4^{20} 1.4539, was obtained in 79% yield. PMR spectrum: 4.93 and 5.06 (2H, m, J = 1.5 Hz, $=CH_2$); 4.22 (1H, t, CH); 3.46 (1H, s, OH); 3.37 (2H, d, CH_2); 1.72 ppm (3H, d, CH_3).

Oxide III, with bp 80-82°C (650 mm), n_D^{20} 1.4275, and d_4^{20} 0.8835, was obtained in 70 and 76% yields, respectively, from alcohols IIa and IIb. PMR spectrum: 4.92 and 5.19 (2H, m, J = 1.5 Hz, $=CH_2$); 3.29 (1H, m, CH); 2.42-2.78 (2H, m, CH_2); 1.5 ppm (3H, d, CH_3).

LITERATURE CITED

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