A NEW PATHWAY FOR THE SYNTHESIS OF CYCLOBUTANE

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The cyclobutane yield does not exceed 11% in the reaction of 1,4-dihalobutanes with a suspension of sodium in toluene [1]. This yield may be raised to 70% by carrying out the cyclization of 1,4-dibromobutane with lithium amalgam in dioxane at reflux [2]. Since four-membered heterocyclic silicon compounds are formed in good yields in the gas-phase reaction of K/Na atoms with 1,4-dihalosilane derivatives [3, 4], we attempted to use this part of alkali metals for the synthesis of cyclobutane although Bawn and Milsted [5] have indicated that the dehalogenation of 1,4-dihalobutanes under these conditions leads to the formation of a mixture of ethylene and butenes. We are the first to establish that the major product of the reaction of K/Na atoms with 1,4-dichlorobutane is cyclobutane and not ethylene and butene as indicated previously [5].

$$ClCH_2CH_2CH_2CH_2Cl \xrightarrow[190-270^\circ,\ 10^{-1}\ torr]{K/Na\ vapor} \begin{matrix} H_2C-CH_2 \\ | \quad | \\ H_2C-CH_2 \end{matrix}$$

The cyclobutane content in the reaction mixture is enhanced and the content of ethylene and butenes is diminished with decreasing temperature. The cyclobutane yield is 35% (70% relative to converted to converted 1,4-dichlorobutane) and the conversion is 50% at 220°C. Ethylene traces were removed by evacuation at −100°C while the butenes were removed by passage through bromine water. After purification, the reaction product was identified by the low-temperature IR spectra method using an argon matrix at 10°K (ν, cm^{-1}): 626 s, 633 m, 902 s, 909 w, 1131 w, 1223 w, 1256 s, 1352 w, 1382 m, 1448 m, 2872 s, 2907 m, 2942 v.s., 2972 v.s. Mass spectrum at 70 eV, m/z (%): 56(62), 55(20), 41(89), 28(100), 27(42), 26(23).

LITERATURE CITED

1. W. B. Smith, J. Org. Chem., 23, 509 (1958).
2. D. S. Connar and E. R. Wilson, Tetrahedron Lett., 4925 (1967).
3. L. E. Gusel'nikov, Yu. P. Polyakov, E. A. Volnina, and N. S. Nametkin, J. Organomet. Chem., 292, 189 (1985).
4. L. E. Gusel'nikov, Yu. P. Polyakov, E. A. Volnina, and N. S. Nametkin, in: H. Sakurai (editor), Organosilicon and Bioorganosilicon Chemistry, Ellis Horwood, Chichester (1985), p. 115.
5. C. E. H. Bawn and J. Milsted, Trans Faraday Soc., 35, 885 (1939).

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