A NEW HETEROCYCLE SYSTEM — 2-OXO-5-ALKYL-1,3,2-DIOXASELENOLANE

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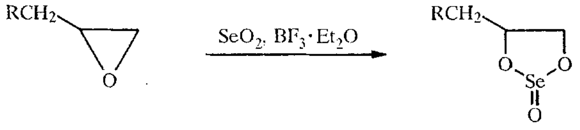
The synthesis of cyclic esters of seleninic acid is a little-studied region of organic chemistry [1, 2].

We have shown that the reaction of equimolar quantities of selenium dioxide and epichlorohydrin (Ia) or propylene oxide (Ib) in the presence of boron trifluoride etherate under mild conditions provides a new and convenient method for the synthesis of functionally substituted 1,3,2-dioxaselenolanes IIa, b.



Ia, b IIa,b

I,II a R = Cl. b R = H

Thus, a solution of 27 mmoles oxirane Ia or Ib in 20 ml dioxane was added dropwise to a solution of 27 mmoles SeO_2 in 25 ml dioxane plus 0.5 ml $BF_3 \cdot Et_2O$ at 20°C under argon. The mixture was stirred for 3 h at 40°C (for Ia) or for 2 h at 20°C (for Ib) and the reaction mixture was then evaporated under vacuum (60°C, 3 mm) and the residue chromatographed on a column of Al_2O_3 using 1:4 benzene–ethanol and collecting the fraction with R_f 0.45. **2-Oxo-5-chloromethyl-1,3,2-dioxaselenolane (IIa), $C_3H_5ClO_3Se$** was obtained in 85% yield, n_D^{20} 1.5310, d_4^{20} 1.7766. IR spectrum (cm^{-1}): 680 (C–Cl), 760-840 (Se–O), 1130, 1260, 1310 (Se=O). PMR spectrum (DMF-D_6, δ, ppm): 3.72 (2H, d, OCH_2), 4.39 (2H, t, CH_2Cl), 4.58 (1H, m, CHO). Mass spectrum (m/z, I_{rel}, %): 203 (40), 154 (80), 125 (42), 96 (30), 80 (15), 40 (17).

2-Oxo-5-methyl-1,3,2-dioxaselenolane (IIb, $C_3H_6O_3Se$), yield 90%, n_D^{20} 1.5510, d_4^{20} 1.6100. IR spectrum (cm^{-1}): 760-785 (Se–O), 1120, 1340 (Se=O). PMR spectrum (DMF-D_6, δ, ppm): 2.72 (3H, d, CH_3), 4.10 (2H, d, CH_2O), 5.05 (1H, m, CHO). Mass spectrum (m/z, I_{rel}, %): 169 (22), 125 (55), 111 (30), 95 (41), 80 (8), 79 (54).

The results of elemental analyses for compounds IIa, b were in agreement with those calculated.

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