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SHORT COMMUNICATIONS

Iodoalkoxylation of Cyclohexene with Allyl and Propargyl Alcohols

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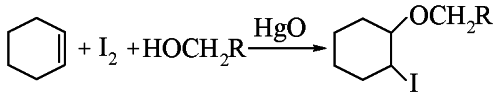
Iodoalkoxylation of olefins with unsaturated alcohols was not investigated [1]. Yet in can be a convenient preparation method for unsaturated ethers of β-iodoalcohols. Structural analogs of the latter are known to show biological activity, and they can be used in the organic synthesis [3–5].

We found that in the presence of mercury oxide the treatment with free iodine of a mixture of cyclohexene and propargyl or allyl alcohol resulted in formation of unsaturated ethers of 2-iodocyclo0 hexanol.



R = C≡CH(**I**), CH=CH$_2$ (**II**)

2-Iodocyclohexyl propargyl ether (I). Into a cooled (–5÷0°C) and vigorously stirred mixture of 14 g (0.25 mol) of propargyl alcohol and 20.5 g (0.25 mol) of cyclohexene was added 26 g (0.12 mol) of HgO, and then by portions (1 g each) 63 g (0.25 mol) of fine iodine powder. The stirring at room temperature was carried on for 2–2.5 h, then the mixture was filtered, the filtrate was diluted with water, washed with water solution of sodium carbonate, and the product was extracted into ether. The extract was dried on $CaCl_2$, and the residue after evaporation of the solvent was distilled in a vacuum. We obtained 40.9 (62%) of compound I, bp 115°C (I mm Hg). d_4^{20} 1.5215, n_D^{20} 1.5500; MR_D 55.03, calc. 54.20. IR spectrum (ν, cm^{-1}): 675 (C–I), 1100 (C–O–C), 2120 and 3310 (C≡CH). ^{1}H NMR spectrum (CCl_4, δ, ppm): 1.2–2.2 m [8H, $(CH_2)_4$, ring protons], 2.5 t (1H, ≡CH), 3.60 m (1H, CHO), 4.20 m (1H, ICH), 4.25 d (2H, OCH_2C≡). Found, %: C 40.42; H 4.41; I 48.31. $C_9H_{13}IO$. Calculated, %: C 40.91; H 4.96; I 48.07.

2-Iodocyclohexyl allyl ether (**II**) was obtained in similar way from allyl alcohol, cyclohexene, and iodine in 58% yield, bp 92–94°C (1 mm Hg), d_4^{20} 1.4939, n_D^{20} 1.5438; MR_D 56.24, calc. 55.86. IR spectrum (ν, cm^{-1}): 670 (C-I), 1100 (C–O–C), 1620 (CH=CH$_2$). ^{1}H NMR spectrum (CCl_4, δ, ppm): 1.0–2.15 m [8H, $(CH_2)_4$, ring protons], 3.6 m (1H, CHO), 4.01–4.21 m (3H, CHI, CH_2O), 5.1–5.8 m (3H, CH=CH$_2$). Found, %: C 41.13; H 5.74; I 47.32. $C_9H_{15}IO$. Calculated, %: C 40.61; H 5.68; I 47.71.

IR spectra were recorded on spectrometer UR-20. ^{1}H NMR spectra were registered on Tesla BS 487B, internal standard HMDS.

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