REACTIONS OF N-CHLOROAMINES WITH OLEFINS IN THE PRESENCE OF SULFUR TRIOXIDE

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With halogenamines as examples, we developed a new approach to the activation of electrophilic reagents $X^{\delta+}-Y^{\delta-}$ in reactions with olefins by introducing SO_3 to form a more reactive reagent of type $X^{\delta+}-\delta^{-}OSO_2Y$, containing the nucleophilic anion YSO_2O^- (cf. [1]). Thus, dichloroamine, treated with equimolar amounts of SO_3 (-50°C, CH₂Cl₂) and then cyclohexene (-60°C gradual increase in temperature to 20°C) gives trans-l-chlorocyclohexan-2-ol diethylamidosulfate (I) in an 80% yield, according to the following scheme:



The individual chloride can be additionally purified by distillation (bp 101-102°C (1 mm), n_D^{21} 1.4774, R_f 0.76, silica gel Silpearl in the hexane:ethyl acetate = 6:1 system), or by chromatography on silica gel. The structure of the chloride (I) was confirmed by the data of ¹H NMR spectra (100 MHz, CCl₄, δ , ppm): 4.48 d.t (1H, H-C-O, $J_1 = J_2 = 7.5$, $J_3 = 4.0$ Hz) and 4.04 d.t. (1H, H-C-Cl, $J_1 = J_2 = 7.5$, $J_3 = 4$ Hz) and ¹³C NMR spectra (δ 81.79 (C-O) and 59.24 (C-Cl)) and by elemental analysis for C, H, N, S, and Cl. Similar dimethylamidosulfates of chlorohydrins were obtained from cyclopentene and 1-hexene. The limits of applicability of this new reaction of conjugated 1,2-addition and its synthetic possibilities (cf. [2]) are being studied.

LITERATURE CITED

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