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SHORT COMMUNICATIONS

Electrophilic Addition of Butyl Chloride to 2-Allylphenol in the Presence of Sulfur Trioxide

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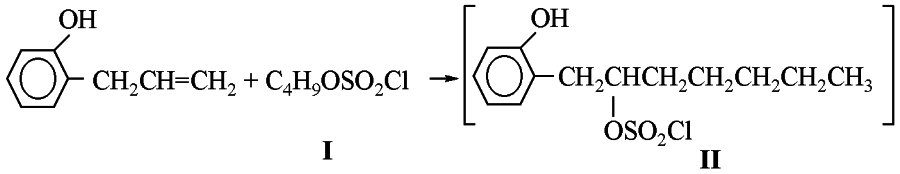
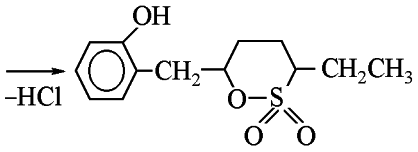
Evidence was reported [1–3] on insertion of sulfur trioxide into ordinary element–element bonds providing new reagents of high electrophilicity capable of addition to unsaturated systems [4]. The aim of this study was extension of sulfonate activation strategy to increasing electrophilic reactivity of chlorination and alkylation agents by an example of reaction between butyl chloride and 2-allylphenol in the presence of sulfur trioxide.

By the known procedure [5] we converted butyl chloride into reagent **I** with a sulfonate structure originating from insertion of sulfur trioxide into C–Cl bond. Then we carried out reaction of the agent with 2-allylphenol. By variation of reaction temperature and the order of reagents addition we found that the optimum procedure required addition of cooled solution of 2-allylphenol in carbon tetrachloride to the reagent **I** solution at –78°C. To avoid formation of the side products equimolar amounts of 2-allylphenol and reagent **I** were used. The reaction progress was monitored by TLC on Silufol UV-254 plates by following reagent **I** consumption since at heating of the plate the sulfonate appeared as a characteristic black spot (development in iodine vapor, eluent ethyl acetate–hexane, 1 : 5).

Apparently reagent **I** first added to the multiple bond of 2-allylphenol, and then intermediate **II** at room temperature underwent intramlecular cyclization forming 4-(*o*-hydroxybenzyl)-1-ethylbutane-1,4-sultone (**III**) in 61% yield.





4-(*o*-Hydroxybenzyl)-1-ethylbutane-1,4-sultone (III). To a mixture of 0.8 g (10 mmol) of SO_3 in 20 ml of CCl_4 at –78°C was added at stirring a cooled solution of 0.925 g of butyl chloride in 5 ml of CCl_4. The stirring was continued for another 30 min, and then a cooled solution of 1.34 g (10 mmol) of 2-allylphenol in 5 ml of CCl_4 was added. After 15 min the cooling was stopped, and the stirring was carried on at room temperature for 30 min with vigorous HCl liberation. After the end of HCl liberation the solvent was removed on a rotary evaporator. The reaction product was isolated by column chromatography on silica gel L 5/40 μ, eluent ethyl acetate–hexane, 1 : 5.

Compound **III** is a light-brown viscous substance soluble in water, alcohols, acetone, ethyl acetate, and

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DMSO. IR spectrum (KBr, ν, cm^{-1}) 578 (–C–S–); 883, 1022 (1,2-substituted benzene), 1465, 1620, 3030 (arom); 1172, 1228 (SO2); 2967, 2940, 2900 (CH_3, CH_2, CH); 3200 (OH). ^{1}H NMR spectrum (DMSO), δ, ppm: 1.0 t (3H, CH_3), 1.05 m (2H, $SO_2CH\underline{CH}_2$), 1.15 m (2H, $OCH\underline{CH}_2$), 1.37 m (2H, CH_2), 2.5 d (2H, $\underline{CH}_2C_6H_4$), 2.75 m (1H, SO_2CH), 3.45 m (1H, OCH), 5.75 (OH), 6.7 (2H, H arom), 7.35 (2H, H arom). ^{13}C NMR spectrum (DMSO), δ, ppm: 9.399, 18.661, 21.665, 36.151, 56.150, 79.961, 121.434, 122.973, 125.921, 127.328, 132.001.

IR spectrum was recorded on IKAR instrument. NMR spectra were registered on Varian X-400 spectrometer at operating frequencies 400 for ^{1}H and 100 MHz for ^{13}C nuclei, reference TMS.

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