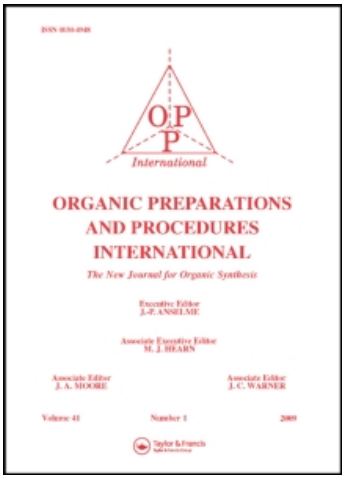

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A CONVENIENT PROCEDURE FOR THE PREPARATION OF TRIETHYLAMINE-SULFUR TRIOXIDE

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A CONVENIENT PROCEDURE FOR THE PREPARATION OF TRIETHYLAMINE-SULFUR TRIOXIDE

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Sulfur trioxide complexes with amines such as pyridine and trimethylamine have been used extensively[1] for the synthesis of sulfate esters of alcohols as well as for other organic transformations. Triethylamine-sulfur trioxide (1) is a superior reagent for the sulfation of steroidal alcohols.[2] Recently, it has also been reported that in a comparative study of the sulfation of polysaccharides[3,4] with a number of sulfur trioxide complexes, the use of 1 has led to cleaner reaction products with the highest degree of sulfation. Unfortunately, this reagent is not commercially available and no simple procedure has been reported for its preparation. We now describe a convenient procedure for the preparation of 1 that is adaptable to large scale operations.[5]

$$ClSO_3H + 2\ Et_3N \xrightarrow[5^\circ]{CH_2Cl_2} \underset{\underline{1}}{Et_3N\cdot SO_3} + Et_3\overset{+}{N}\ Cl^-$$

EXPERIMENTAL SECTION

Triethylamine-Sulfur trioxide (1).- To a solution of triethylamine (121.2 g, 1.2 mol) in dichloromethane (400 ml) in a 2L three-necked flask equipped with a mechanical stirrer and drying tubes, cooled in an ice-bath was added dropwise over a period of 1.5 hr a solution of chlorosulfonic acid (69.6 g, 0.6 mol) in dichloromethane (200 ml) with continuous stirring. A vigorous reaction occurred and the precipitation of triethyl-

amine hydrochloride was observed. When the reaction was completed, the mixture was poured into ice water (300 ml). The organic layer was separated and washed with water. It was then dried over anhydrous sodium sulfate, treated with a small amount of Darco^{R} and filtered through a bed of Magnesol^{R}. The colorless filtrate on evaporation in vacuo afforded 81.0 g (75%) of a crystalline solid, mp. 89-91°. Recrystallization from dichloromethane/heptane afforded colorless shiny prisms, mp. 92-93°, lit.[6] mp. 91.5°.

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