

This article was downloaded by:

On: 27 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

A CONVENIENT PROCEDURE FOR THE PREPARATION OF TRIETHYLAMINE-SULFUR TRIOXIDE

Vijay Nair^a; Seymour Bernstein^a

^a Medical Research Division, American Cyanamid Company, Pearl River, New York

To cite this Article Nair, Vijay and Bernstein, Seymour(1987) 'A CONVENIENT PROCEDURE FOR THE PREPARATION OF TRIETHYLAMINE-SULFUR TRIOXIDE', *Organic Preparations and Procedures International*, 19: 6, 466 – 467

To link to this Article: DOI: 10.1080/00304948709356213

URL: <http://dx.doi.org/10.1080/00304948709356213>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

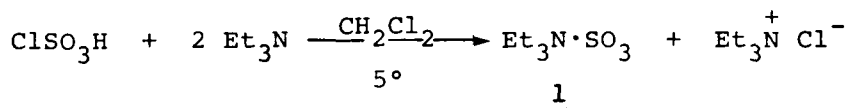
The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

A CONVENIENT PROCEDURE FOR THE PREPARATION OF
 TRIETHYLAMINE-SULFUR TRIOXIDE

Submitted by Vijay Nair* and Seymour Bernstein
 (10/21/86)

Medical Research Division
 American Cyanamid Company
 Pearl River, New York 10965

Sulfur trioxide complexes with amines such as pyridine and trimethylamine have been used extensively¹ 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



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°.

REFERENCES

1. E. E. Gilbert, Chem. Rev., 62, 549 (1962); G. A. Olah, Y. D. Vankar and A. P. Fung, Synthesis, 59 (1979).
2. J. P. Dusza, J. P. Joseph and S. Bernstein, Steroids, 9, 49 (1968).
3. K. B. Guiseley, "Some Novel Methods and Results in the Sulfation of Polysaccharides" in "Carbohydrate Sulfates", R. G. Schweiger, Ed., ACS Symposium Series, Vol. 77, p. 152, ACS, Washington, D. C., 1978.
4. V. Nair, J. P. Joseph, J. F. Poletto and S. Bernstein, Unpublished results.
5. Messrs E. R. Ruso and H. W. Marson of our Process Research and Development Section have been able to scale up the preparation to obtain several kilograms of 1.
6. F. Beilstein and E. Wiegand, Ber., 16, 1264 (1883).