Acknowledgment.—The authors wish to acknowledge the assistance of Pamela Strong and Robert Balchunis in conducting a portion of the experimental procedure.

A Precautionary Note on the Synthesis of Thiete Sulfone[1]

DONALD C. DITTMER,* M. E. CHRISTY, N. TAKASHINA, R. S. HENION, AND J. M. BALQUIST

Department of Chemistry, Syracuse University, Syracuse, New York 13210

Received November 4, 1970

In the original description[2] of the synthesis of thiete sulfone (thiete 1,1-dioxide), the procedure for the preparation of 3-thietanol 1,1-dioxide called for evaporation to dryness of the solution remaining after oxidation of the sulfide to the sulfone.[3] Since several researchers have reported to us that explosions had occurred during this step, we wish to emphasize that the evaporation must be done in an evaporating dish open to the atmosphere and that under no circumstances must the peroxide-containing solution be concentrated in a closed system. Therefore, the following procedure should be followed for the synthesis of 3-hydroxythietane 1,1-dioxide.

Experimental Section

3-Hydroxythietane 1,1-Dioxide.—3-Hydroxythietane (45 g, 0.50 mol) is mixed with 105 ml of glacial acetic acid in a 500-ml three-necked flask fitted with an addition funnel, thermometer, condenser, and a magnetic stirring bar. The flask is cooled in an ice bath, and, with stirring, hydrogen peroxide (116 g, 30%) is added dropwise, the reaction temperature not being allowed to rise above 20°.[4] After the addition of hydrogen peroxide the reaction mixture is kept in the ice bath for 1 hr, the stirring is stopped, and the mixture is allowed to stand at room temperature overnight.[5] It is diluted with 800 ml of distilled water in a 9- to 10-in. evaporating dish, and water and acetic acid are evaporated on a steam bath.[6] The colorless oil is cooled to a white, crystalline mass which is crushed in the evaporating dish and air-dried. The nearly dry solid is recrystallized from 100 ml of ethyl acetate.[7] After two recrystallizations from ethyl acetate, 38–40 g (62.3–65.6%) of 3-hydroxythietane 1,1-dioxide, mp 100°, is obtained.

Registry No.—3-Hydroxythietane 1,1-dioxide, 22524-35-2.

---

[1] The work on small ring sulfur chemistry has been supported by the National Science Foundation, and the synthesis reported in this note was developed with aid from Grant GP-726.

[2] D. C. Dittmer and M. E. Christy, *J. Org. Chem.*, **26**, 1324 (1961).

[3] A synthesis of thiete sulfone which does not involve an oxidation step has been reported recently: P. Chang and D. C. Dittmer, *ibid.*, **34**, 2791 (1969).

[4] Considerable heat is evolved during the first half of the addition of hydrogen peroxide and the addition must be slow. The reaction becomes more moderate and the last half of the peroxide may be added more rapidly.

[5] The flask is kept in a bath of tap water. A precipitate may appear at this point.

[6] This evaporation is crucial. Evaporation must be stopped at the first indication of a yellow color seen around the edge of the liquid in the dish. Yields are much lower if the heating is prolonged beyond this stage. *Do not evaporate in a closed system. Explosions have occurred when this was done.* The final volume of oily product and the slight amount of trapped solvent is usually about 100 ml.

[7] A fluffy, insoluble material is removed by filtration.