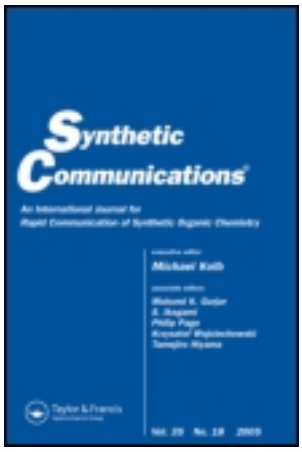

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A FACILE SYNTHESIS OF AMINO-IMINOMETHANESULFONIC ACID

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A FACILE SYNTHESIS OF AMINO-IMINOMETHANESULFONIC ACID

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ABSTRACT

Aminoiminomethanesulfonic acid was conveniently obtained by oxidation of aminoiminomethanesulfinic acid with H_2O_2 in a high yield and good purity.

Aminoiminomethanesulfonic acid (AIMSOA) is an important agent for synthesis of guanyl and guanido derivatives.[1–6] It is readily attacked by nucleophiles. Its preparation has been reported by Miller[1] and Kim.[2] Peracetic acid was employed as oxidant by both of them. When we repeated their procedures, unfortunately we failed to achieve the reported yield. We therefore attempted to find a cheap, simple and reliable procedure for preparation of AIMSOA. Under optimized conditions, we employed aminoiminomethanesulfinic acid (AIMSA) as the starting material as Keekyung Kim[2] did, with H_2O_2 as oxidant in aqueous medium.

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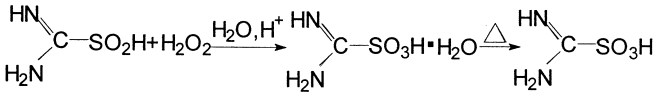
AIMSOA was obtained in 93% yield and 98.5% purity. AIMSOA·H_2O was first reported and studied by us. The reaction takes place as illustrated below.



The starting material (AIMSA) is a cheap and commercially available reagent. The method reported here has several advantageous over the reported procedures.[1,2] Further, AIMSOA·H_2O has not been reported previously. We have studied its properties and optimized the conditions for its preparation.

AIMSOA·H_2O was analysed by differential thermal analysis (DTA) and thermo-gravimetric analysis. Dehydration occurs at 42°C. The thermo-gravimetric analytical results determined the amount of water to be 12.3%, while the calculated value of AIMSOA·H_2O was 12.68%.

The reaction parameters were optimized as reported here for increasing the yield of AIMSOA. Above 70°C the reaction was fierce and out of control, while the reaction proceed smoothly at 50°C. Given the probability of side-reactions caused by concentrated H_2SO_4, we employed 0.5 N H_2SO_4 to catalyse the reaction. The theoretical amount of H_2O_2 was sufficient and the optical reaction time was 70 min.

The proposed processes are therefore expected to be advantageous procedures for the economical manufacture of AIMSOA. We have successfully prepared amino guanidine and guanyl glycine with the obtained AIMSOA.

EXPERIMENTAL

IR spectra were recorded on a Shimadzu IR-440 infrared spectrophotometer as KBr discs. Elemental analyses were performed on a Carlo-Erba 1106 elemental analyser. Differential thermal analyses were carried out with a CRT-1 differential thermal analyser made in Shanghai.

Preparation of AIMSOA

In a two-necked round bottom flask (100 mL) equipped with a magnetic stirrer, 21.6 g AIMSA was added H_2O_2 (0.2 mol) in 23 mL 0.5 N H_2SO_4

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aqueous solution by dropwise at 50°C. After stirring at the same temperature for 70 min. The mixture was cooled by ice–water and filtered to afford white glittering crystals. The crystals were washed twice by ice–water and crystallized from water and dried at less than 30°C. The obtained AIMSOA·H_2O was heated at 60°C for 5 h and AIMSOA was prepared in 93% yield and 98.5% purity.

AIMSOA · H_2O: IR (KBr) ν: 3420, 3245, 1700, 1220, 1050 cm^{-1}. Anal. calcd. for $CH_6O_4N_2S$: C, 8.45; H, 4.23; N, 19.7; S, 22.5; Found: C: 8.74; H: 4.17; N: 19.8; S: 22.7.

AIMSOA: IR (KBr) ν: 3430, 3240, 1710, 1220, 1050 cm^{-1} Anal. calcd. for $CH_4O_3N_2S$: C, 9.68; H, 3.22; N, 22.58; S, 25.81; Found: C: 9.7; H: 3.1; N: 22.3; S: 25.7.

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