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A new and efficient synthesis of sulfonylurea

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술포닐 우레아유도체의 용이하고 새로운 합성방법

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식물 중 단자엽을 제외한 쌍자엽 식물은 선택적으로 제거할 수 있는 헤테로 고리 화합물을 기존에 알려진 방법보다 용이하게 합성할 수 있는 방법을 연구하였으며, 과정에 일부 헤테로 고리 치환기의 4위치에 염소기가 치환된 화합물 5a를 70%수율로 5b는 74%수율로 각각 수득하였다.

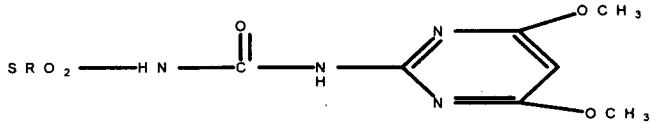
술폰 산화제로 potassium percarbonate를 사용하였을때 1)의 화합물을 90%수율로 고순도 화합물을 수득하였으며 2)의 경우 85%의 고수율, 고순도 화합물을 수득하였다. Sodium hypochlorite의 경우 피리미딘 헤테로 고리에 염소가 치환되는 불순물 생성으로 적합지 않음이 판명되었다.

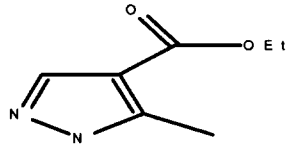
Abstract

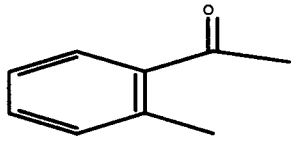
We developed new process for the large scale preparation of pyrazole sulfonyl urea and pyrazole sulfonyl urea carboalkoxyl benzene sulfonyl urea 1) in high yields and convenience. As compound with know procedure this procedure have found to be far large scale preparation. During the preparation we obtains two new sulfonyl urea 5a and 5b in 70% and 74% (purified by crystallization).

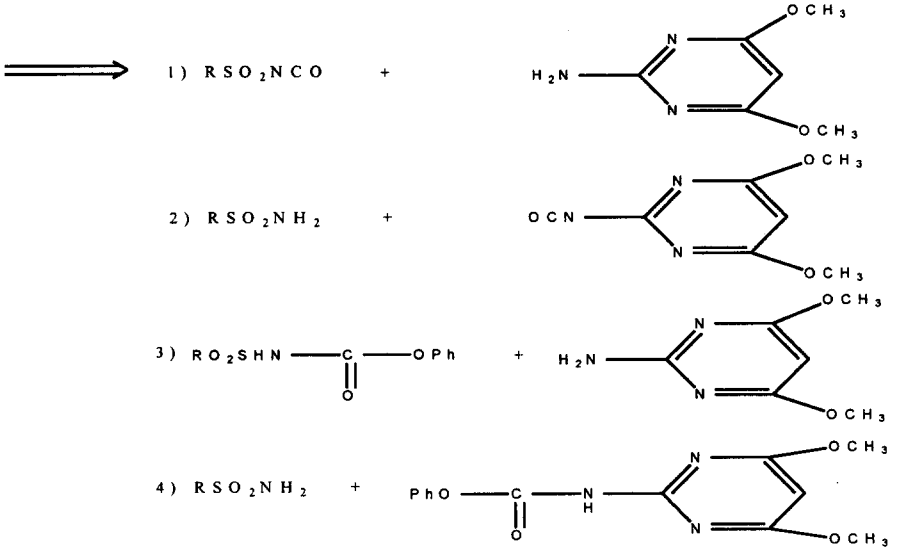
Key Word; Sulfonyl Urea, Potassium Percarbonate, Sodium Hypochlorite, benzene sulfonamide, Pyrazole Sulfonamide

Since the discovery of sulfonyl urea as a new herbicides which exhibit both preemergence and postemegency activity at extra-ordinary low rate of application there are numerous analogs many synthetic methods is developed. Synthesis was usually acomplished via urea formation, the key step using various sulfonyl isosyanate and carbamate as a intermediate(Fig1). However, the intermediate heterocyclic isocyanate

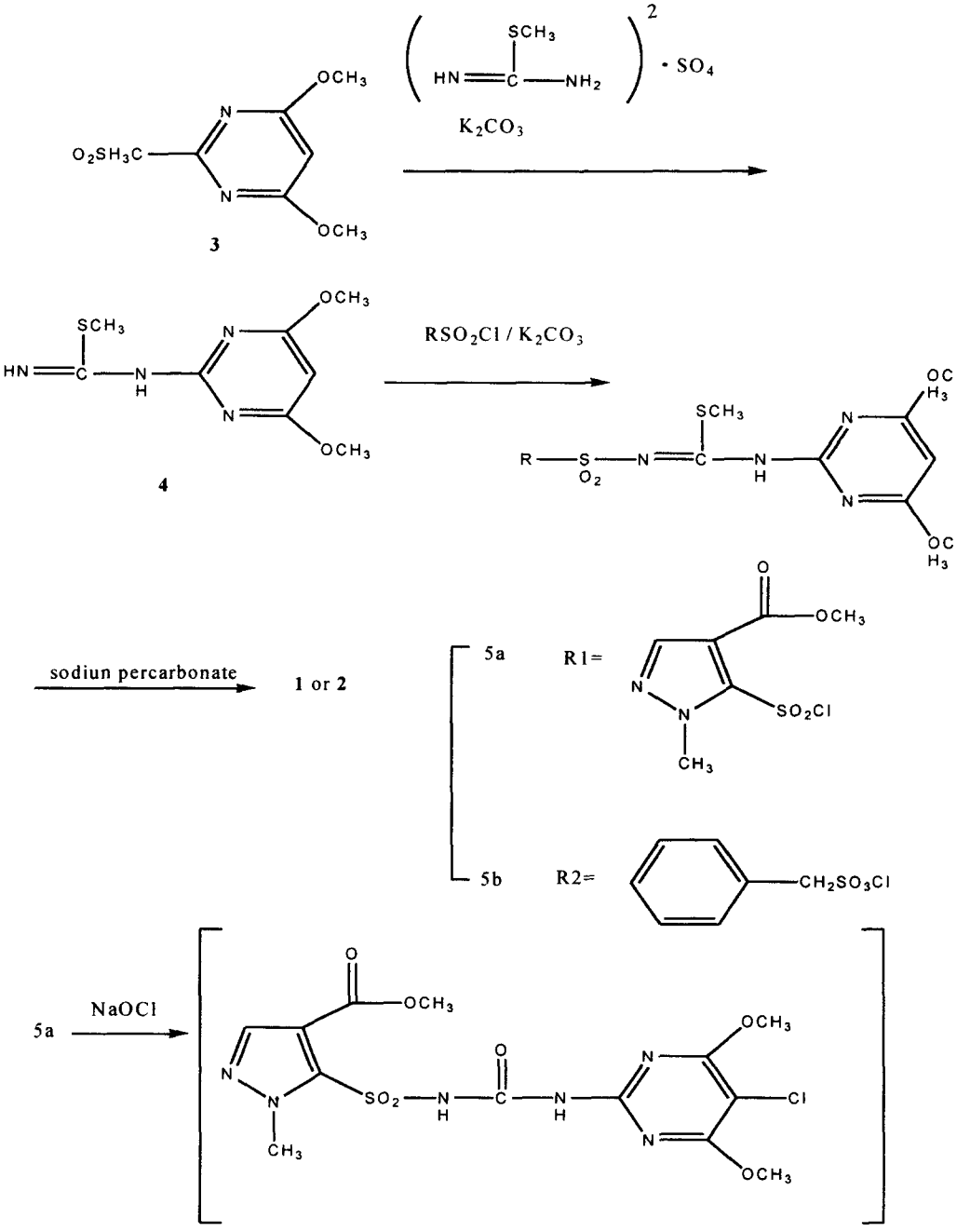


R =  NC-311 1

R =  Rondax 2



is usually hard to synthesize and NC-311 Fig1 requires highly unstable intermediates thus we need to develope new process which is mild, nontoxic and crystalline and we developed a new synthetic route as shown below, via direct substitution of 2-methane sulfonyl group in 4,6-dimethoxy pyrimidine with sulfonyl isothiourea.



in 4 step in 60% yield. The key intermediate 3 was prepared by the known procedure. The resulting sulfone was easily substituted with sulfonyl isothiourea in the presence of powdered Potassium carbonate to give compound 4 in 80% yield. The resulting pyrimidyl isothiourea 4 was treated with various sulfonyl chloride to give 5 in moderate yield to good yields. (5a: 70%, 5b: 74%, not optimized)

Finally, oxidation and insitu hydrolysis of 5 with sodium percarbonate gave the corresponding sulfonyl ureas in high yield(1. 90%, 2. 85% yield) Our initial trials using (NaOCl) as oxidizing agents gave the ring chlorinated sulfonyl urea in high yield.

This approach to the synthesis of sulfonyl urea proven to be efficient and general for dimethoxy pyrimidyl sulfonyl urea and applicable for the synthesis of a number of potential new sulfonyl urea derivatives.

감사의 글

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References

Morimoto, K, T.Sato, and S. Yamamoto,.and H.Takeuchi.,J.Hetcyclic Chem.,537(1997)

Robert Bellemin and Dider Festal J.Hetrocyclic Chem.,1017(1984)

Richardn F.Sauers, Hockessin Del United Stated Patent 4,420,325 (1985)

Suzuki, Fumio and Daiichi-nissanmachi, European Patent application 83101870.0(1983)

Schmidt, P, and K.Eichenberger,and M. Wilheim Helv. Chim.Acta349 (1959)