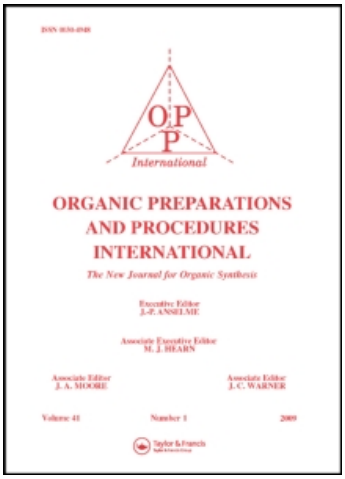

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A CONVENIENT SYNTHESIS OF 5-OXAZOLONES. 2-PHENYL-5-OXAZOLONE

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A CONVENIENT SYNTHESIS OF 5-OXAZOLONES. 2-PHENYL-5-OXAZOLONE

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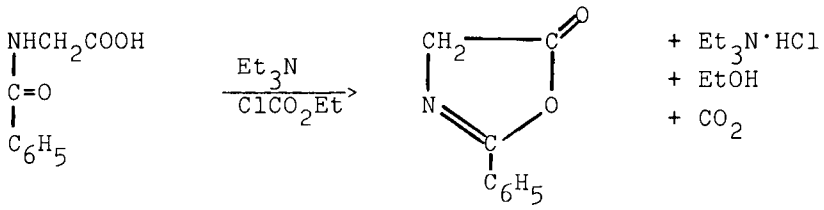

The usual method of preparing 5-oxazolones involves the classical cyclization of an N-acyl-α-amino acid with acetic anhydride. The laboratory preparation of 2-phenyl-5-oxazolone using this method has recently been reported.[1] This method has several disadvantages, especially with oxazolones with reactive hydrogen atoms in the 4-position. Distillations are usually required and, even in cases where side reactions are controlled, the lower boiling oxazolones are difficult to separate from acetic acid or acetic anhydride. We wish to report an alternative procedure for the easy preparation of oxazolones utilizing the mixed carbonic-carboxylic acid anhydride route. This method was previously reported but appears to have been overlooked.[2] A negative result is reported in a reference work of wide availability.[3]

We find that the mixed anhydride synthesis is an outstanding method for the preparation of oxazolones. If one does not wish to isolate the oxazolone but use it directly, again this method is the one of choice because the oxazolone may be generated in an inert solvent such as benzene, ether, methylene chloride, hexane or ethyl acetate and then allowed to react with an amine. We will illustrate a typical procedure with the synthesis of 2-phenyl-5-oxazolone.[6]

Experimental[4]

2-Phenyl-5-oxazolone. To a slurry of 36.1 g. (0.2 mole) of hippuric acid in 190 ml. of ethyl acetate is added 19.2 ml. (0.2 mole) of ethyl chloroformate. After this mixture is cooled to 0-5°, 28.3 ml. (0.2 mole) of triethylamine is added dropwise at a rate sufficiently slow to keep the temperature at about 5°. When the addition is complete, the mixture is stirred for 30 minutes. The reaction mixture is quickly warmed to 40° for several minutes to insure complete solution of the product. The reaction mixture is filtered to remove triethylamine hydrochloride and this salt is washed with 20 ml. of warm ethyl acetate. To the combined filtrates is added enough hexane to produce a cloud point. The solution is cooled in a dry ice-acetone bath and the product crystallizes in the form of small off-white crystals. The product is filtered and dried for several hours in a vacuum oven set at 30°.[5]

The yield of 2-phenyl-5-oxazolone is 26.1 g. (80%), m.p. 89-91°; i.r. (KBr) 1810 (C=O) and 1650 cm^{-1} (C=N); nmr ($CDCl_3$) δ 4.43 (s,2), 7.37-7.70 (m,3), 7.83-8.06 (m,2).

Anal. Calcd. for $C_9H_7NO_2$: C, 67.1; H, 4.3; N, 8.7.

Found: C, 67.0; H, 4.5; N, 8.7.

References

1. G. E. VandenBerg, J. B. Harrison, H. E. Carter and B. J. Magerlein, Organic Syntheses, 47, 101 (1967).

2. K. Nowak, I. Z. Siemon and H. Siemieniewski, Ann. Soc. Chim Polonorom, 36, 557 (1962).

3. N. F. Albertson, Organic Reactions, 12, 180 (1962).

4. All reactants and solvents were "Eastman Grade" from Distillation Products Industries.

5. This compound should not be subjected to elevated temperatures since some type of condensation occurs causing the material to turn bright yellow.

6. For further examples, see J. Polymer Sci., part B, In press.

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