Chemistry of Heterocyclic Compounds, Vol. 34, No. 3, 1998

SIMPLE METHOD FOR THE SYNTHESIS OF 3,5-DIMETHYLPYRAZOLYL-1-ACETIC ACID

N. L. Nam, I. I. Grandberg, and V. I. Sorokin

The method for the synthesis of 3,5-dimethylpyrazolyl-1-acetic acid (I) by the condensation of the hydrochloride of ethyl hydrazineacetate (II) with acetylacetone and subsequent hydrolysis is known. However, compound (II) is unstable, and the synthesis itself of this compound with the yield of 65% is not simple [1].

We propose the alkylation of 3,5-dimethylpyrazole with chloroacetic acid in water with the subsequent neutralization of the hydrochloric acid by sodium formate, which renders possible the ready isolation of the amphoteric compound (I) by extraction with isopropanol.

The equimolar mixture (0.1 mole) of 3,5-dimethylpyrazole and chloroacetic acid in 20 ml of water is boiled for 8 h prior to the addition of the equimolar amount of sodium formate, and the mixture is boiled for 15 min more. The reaction mass is concentrated to dryness on a rotary evaporator, and is maintained for 1 h at 100°C (5 mm of Hg). The dry mass is heated by boiling with 30 ml of dry isopropanol, and the isopropanol is decanted; this operation is repeated three more times. The isopropanol is distilled from the combined isopropanol extracts, and the remaining pyrazolylacetic acid is boiled with 80 ml of hexane for 1 h using a reflux condenser and cooled for 1 h at 0°C. Crystals of (I) are filtered off. The yield of pure 3,5-dimethylpyrazolyl-1-acetic acid is 11.3 g (84%). The mp is 179°C using a sealed capillary. Found, %: C 54.22 and H 6.6. $C_7H_{10}N_2O_2$. Calculated, %: C 54.5 and H 6.5. The UV spectrum (Specord M-40, alcohol) is characterized by the λ_{max} 221 nm, log ε 3.80. The IR spectrum (Perkin-Elmer, KBr) is characterized at 1730 cm^{-1}. The PMR spectrum (Bruker AM-300, DMSO-D_6) is as follows: 2.07 ppm (3H, s, 3-CH_3), 2.14 ppm (3H, s, 5-CH_3), 4.77 ppm (2H, s, CH_2), 5.81 ppm (1H, s, 4-H), and 12.9 ppm (1H, broad s, COOH).

REFERENCES

1. A. Carmi, G. Pollak, and H. Yillin, J. Org. Chem., **25**, 44 (1960).

A. K. Timiryazev Moscow Agricultural Academy, Moscow 127550, Russia. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 3, p. 416, March, 1998. Original article submitted January 20, 1998.

 0009-3122/98/3403-0382$20.00