



EXPERIMENTAL

Preparation of 2-amino-3-cyano-4-acetyl-5-methylfuran (I a). 3-Chloro-2,4-pentanedione (2.69 g) and malonitrile (1.32 g) were dissolved in ethanol (10 ml), and sodium hydroxide (8.26 ml of 2.42 N solution) was added. Colourless crystals of I a started separating immediately. The next day they were collected by filtration and washed with 50 % ethanol. Yield: 3.05 g, m. p. about 240° (decomp.). The product can be recrystallized from ethanol. (Found: C 58.5; H 4.85; N 17.1; O 19.5. Calc. for $\text{C}_8\text{H}_8\text{N}_2\text{O}_2$: C 58.5; H 4.9; N 17.1; O 19.5.)

2,4-Dinitrophenylhydrazone: M.p. 166–168° (decomp.) (Found: C 48.9; H 3.7; N 24.3; O 23.4. Calc. for $\text{C}_{14}\text{H}_{12}\text{N}_6\text{O}_6$: C 48.8; H 3.5; N 24.4; O 23.2.) Schiff's base with *o*-nitrobenzaldehyde (Found: C 60.6; H 4.0; N 14.0. Calc. for $\text{C}_{15}\text{H}_{11}\text{N}_3\text{O}_4$: C 60.6; H 3.7; N 14.1.)

Preparation of 2-amino-3-ethoxycarbonyl-4-acetyl-5-methylfuran (I b). 3-Chloro-2,4-pentanedione (2.69 g) and ethyl cyanoacetate (2.26 g) were dissolved in ethanol (7 ml). When sodium hydroxide (8.26 ml of 2.42 N solution) was added, colourless crystals of I b soon started separating. The following day the product was filtered and washed with 50 % ethanol. Yield: 3.30 g; m. p. after recrystallization from ethanol: 136°. (Found: C 57.0; H 6.2; N 6.7; O 30.1. Calc. for $\text{C}_{10}\text{H}_{13}\text{NO}_4$: C 56.9; H 6.2; N 6.6; O 30.3.)

Preparation of 2-amino-3-ethoxycarbonyl-4,4,5-triacetyl-6-methylpyran (II). Bromine (3.2 g) was added with stirring to a cooled (12°) solution of 2,4-pentanedione (2.0 g) in 8.26 ml of 2.42 N sodium hydroxide solution and 15 ml of ethanol. After cooling the solution to 8°, sodium hydroxide (4.1 ml of 2.42 N solution) was added and — one minute later — ethyl cyanoacetate (1.38 g) and another 5.0 ml of sodium hydroxide solution. After 5 h the precipitate formed was filtered and washed with 50 % ethanol. Yield: 0.56 g; m. p. 166° (decomp.). (Found: C 58.2; H 6.2; N 4.7; O 30.9; mol. wt 320. Calc. for $\text{C}_{16}\text{H}_{19}\text{NO}_6$: C 58.2; H 6.2; N 4.5; O 31.0; mol. wt 309.)

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