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Simplified Preparation of 6-Hydroxytropinone

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6-Hydroxytropinone (IV) has been prepared by Sheehan and Bloom [1] from 2,5-dimethoxy-2,5-dihydrofuran(I)through a four-step reaction (yield 7 %) and by Stoll, Becker and Jucker [2] from the corresponding ethoxy compound through a three-step reaction (yield probably about 15 %).

If an acid solution of dimethoxydihydrofuran is left standing for some time at room temperature, neutralized and added to a buffered solution of methylamine and acetonedicarboxylic acid, 6-hydroxytropinone is formed in a 55 % yield. Apparently malealdehyde (II), which is formed very rapidly by hydrolysis of dimethoxydihydrofuran, adds one mole of water whereby malaldehyde (III) is formed, which then condenses with methylamine and acetonedicarboxylic acid to hydroxytropinone.

Experimental. (Microanalyses by E. Boss.) *6-Hydroxytropinone (IV).* I[3] (0.65 g, 0.005 mole) was dissolved in hydrochloric acid (3 *N*, 12.5 ml) and the mixture left standing for 18 hours. The yellowish-brown solution was neutralized with a solution of sodium hydroxide (6 *N*, 6.2 ml) and added to a solution of acetonedicarboxylic acid (1.47 g, 0.01 mole), methylamine hydrochloride (0.68 g, 0.01 mole) and sodium acetate (3.4 g) in water (80 ml). The mixture (pH 4.3) was left standing for 2 days, whereby the acidity decreased to pH 4.9. Potassium carbonate (25 g) and sodium chloride (25 g) were dissolved in the light-brown reaction mixture and the solution continuously extracted with ether. The etheral extract was evaporated in a vacuum, leaving white crystals of hydroxytropinone embedded in a brown oil. By addition of a hot solution of picric acid (1.0 g) in ethanol (9 ml) the picrate of hydroxytropinone was obtained. The yield was 1.05 g (55 %), m. p. 199—200° (dec.) (Hershberg app., corr.), previously found [2] 199° (dec.). (Found: C 43.9; H 4.1; N 14.6. Calc. for $C_{14}H_{16}O_9N_4$ (384.3): C 43.8; H 4.2; N 14.6).

The free base was prepared from the picrate (250 mg) in the usual way and purified by sublimation. The yield of sublimed hydroxytropinone was 82 mg (82 %), m. p. 121—122°, previously found [1] 122.5—123.5°. (Found: C 61.7; H 8.5; N 8.8. Calc. for $C_8H_{13}O_2N$ (155.2): C 61.9; H 8.4; N 9.0).

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