

2(5H)-FURANONE IN THE WILSMEIER–HAACK REACTION

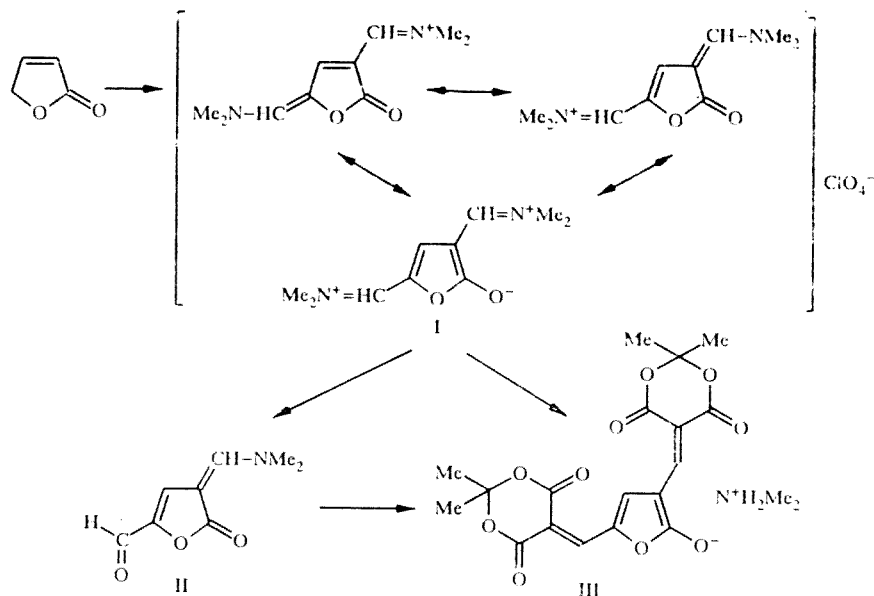
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The resonance stabilized salt (I) was obtained by treatment of 2(5H)-furanone with a DMF–POCl₃ mixture under Wilsmeier–Haack conditions with subsequent treatment of the reaction mixture with HClO₄. It was converted into 5-formylfuranone (II) under basic hydrolysis conditions (pH 8). On reaction with a strong CH acid (Meldrum's acid — 2,2-dimethyl-1,3-dioxane-4,6-dione), the salt I and the enaminoaldehyde II form a derivative of 5-hydroxy-2,4-furandicarbaldehyde (III).

The structures of compounds II and III were demonstrated by x-ray crystallography.

3-(N,N-Dimethylaminomethylidene)-5-(N,N-dimethylaminomethylidene)-2(3H)furanone Perchlorate (I, C₁₀H₁₅ClN₂O₆). Yield 75%. m.p. 245°C (dec.). IR Spectrum (Nujol): 1740 and 1725 cm⁻¹. ¹H NMR Spectrum (CF₃COOD): 3.43 (3H, s, CH₃–N), 3.47 (3H, s, CH₃–N), 3.53 (3H, s, CH₃–N), 3.57 (3H, s, CH₃–N), 7.50 (1H, s, CH), 7.85 (1H, s, CH), 8.02 ppm (1H, s, CH).

3-(N,N-Dimethylaminomethylidene)-5-formyl-2(3H)furanone (II, C₈H₉NO₃). Yield 70%. m.p. 194°C (dec.). UV Spectrum (water), λ_{max} (lg ε): 290 (3.84), 384 nm (4.53). IR Spectrum (Nujol): 1650 and 1720 cm⁻¹. ¹H NMR Spectrum (CDCl₃): 3.26 (3H, s, CH₃–N), 3.46 (3H, s, CH₃–N), 6.46 (1H, s, CH), 7.66 (1H, s, CH), 9.56 ppm (1H, s, CHO).



Dimethylammonium 3,5-[(2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)methyl]-2-oxofuran (III, C₂₀H₂₃NO₁₀). Yield 68%. m.p. 182°C (dec.). UV Spectrum (water), λ_{max} (log ε): 558 nm (4.64). IR Spectrum (Nujol): 1680 and 1720 cm⁻¹. ¹H NMR Spectrum (acetone-D₆): 1.69 (12H, s, 4 CH₃), 3.00 (6H, s (CH₃)₂N), 7.60 (1H, s, CH), 8.18 (1H, s, CH), 9.56 ppm (1H, br. s, CH).

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Translated from Khimiya Geterotsiklicheskh Soedinenii, No. 1, pp. 135-136, January, 1995. Original article submitted December 7, 1994.