LETTERS TO THE EDITOR

SYNTHESIS OF INDOLE-3-THIOCARBOXALDEHYDE

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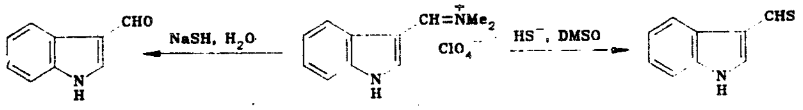
Using the conditions for the synthesis of 3-thioformyl indoles and indolizines we have shown that work up of the corresponding methineimmonium salts with aqueous sodium hydrosulfide solution [1] caused the N-[(3-indolyl)methylene]-N,N-dimethylimmonium perchlorate I to be hydrolyzed to indole-3-carboxaldehyde II. Hydrothiolysis of I was readily brought about by the action of anhydrous NaSH or H_2S in DMSO at 20°C and led to the formation of indole-3-thiocarboxaldehyde III. PMR Spectrum (DMSO-d_6): 7.44-7.64 (m, 5-, 6-, and 7-H), 8.44 (1H, s, 2-H), 8.80 (1H, m, 4-H), 9.26 (1H, br s, NH), 11.41 ppm (1H, s, CH=S). UV Spectrum (λ_{max}, nm, log ε): 275 (4), 374 (4.5), 512 (1.6); $E_{1/2} = -0.83$ V.



II I III

Dilution of the red reaction mixture with water gave a pale colored thial III solvate with DMSO (3:1) in 90% yield with mp 114-116°C (with decomp.). Elemental analytical data and molecular weight (determined mass spectrometrically) were in agreement with those calculated.

Solution in bipolar aprotic solvents (DMSO, DMF, pyridine) caused the solvate to dissociate to the thioaldehyde III and DMSO which was confirmed by PMR spectroscopy in DMF-d_6.

When treated with 2,4-dinitrophenylhydrazine in DMF the solvate formed a red hydrazone with mp 310°C (from methanol).

LITERATURE CITED

1. S. McKenzie and D. H. Reid, J. Chem. Soc., Chem. Commun., No. 13, 401 (1966).

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