Δ^2-PYRAZOLINES FROM ARYLIDENEINDANEDIONES

V. D. Orlov, A. I. Roberman,
and V. F. Lavrushin

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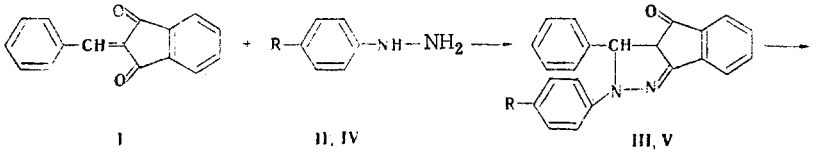
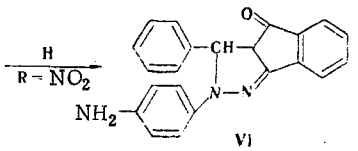
We have shown that Δ^2-pyrazoline III [mp 200°C, λ_{max} 233 nm, 1706 cm^{-1} (C=O)] can be obtained in 50% yield from 2-benzylidene-1,3-indanedione (I) and phenylhydrazine (II) in dry pyridine containing 5% piperidine at room temperature after 3 days. Δ^2-Pyrazoline V [mp 138°C, λ_{max} 390 nm, 1710 cm^{-1} (C=O)] is similarly formed with p-nitrophenylhydrazine (IV) after 10-12 h. Heating intensifies resinification. The nitro group of pyrazoline V was reduced by the method in [1] to amine VI [mp 300°C, 45% yield, λ_{max} 382 nm, 1700 (C=O) and 3426 cm^{-1} (NH_2)].



I II, IV III, V



VI

Under the usual conditions for the preparation of pyrazolines (weakly acidic or weakly alkaline media) dione I reacts with hydrazine IV to give exclusively hydrazone VII [mp 270°C, 43% yield, λ_{max} 400 nm, 1706 (C=O) and 3280 cm^{-1} (NH)], and a mixture of VII with pyrazoline V is obtained only after prolonged refluxing. Under the same conditions, I and II form only an unusually high-melting compounds with mp > 300°C, in the IR spectrum of which, however, characteristic bands of a carbonyl group and a pyrazoline ring are observed. The results of analysis of the substance for its nitrogen content* were in agreement with the calculated value, and the substance has weak luminescence; one may therefore assume that the molecules underwent condensation, probably in the direction described in [2]. Hydrazone VIII [mp 245°C, 52% yield, λ_{max} 406 nm, 1716 (C=O) and 3306 cm^{-1} (NH)] is formed when I and II are heated with KOH in a mixture of ethylene glycol and dioxane.

The difficulties that arise in the synthesis of pyrazolines of this series are undoubtedly due to the fact that the cyclization involves the strained five-membered ring of 2-arylideneindane dione.

LITERATURE CITED

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2. F. Pragst and B. Siefke, J. Prakt. Chem., 316, 267 (1974).

*Similar analyses for I-VIII gave results that were in good agreement with the calculated values.

A. M. Gor'kii Kharkov State University, Kharkov 310077. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 1, pp. 130-131, January, 1979. Original article submitted July 13, 1978.

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