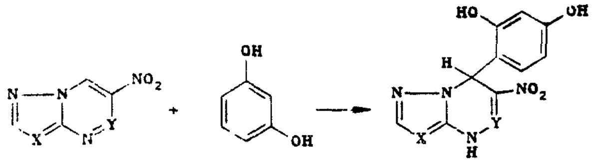
DIRECT INTRODUCTION OF AZOLOAZINE RESIDUES INTO RESORCINOL

V. L. Rusinov, T. L. Pilicheva,
A. V. Myasnikov, N. A. Klyuev,
and O. N. Chupakhin

UDC 547.565.2'792.9'859.3.04

The reaction of azoloazines with phenols has already been described. In [1], an azoloazine – 3-cyano-6,7-dicarbethoxypyrazolo[1,5-a]pyrimidine – is used in the form of a quaternary salt. As the result, benzofuran or cyclohexadienylidene derivatives are formed, and not addition products of phenols, as should have been expected.

We succeeded in carrying out a direct hetarylation of resorcinol by azoloazines Ia-d without converting them into the cationic form. When a mixture of azoloazines Ia-d and resorcinol is boiled in butanol, 6-nitro-4,7-dihydro-7-(2,4-dihydroxyphenyl)azolo[1,5-a] pyrimidines (IIa-c) and 6-nitro-4,7-dihydro-7-(2,4-dihydroxyphenyl)azolo[5,1-c] [1,2,4]-triazine (IId), retaining the resorcinol fragment in unchanged state, are formed in a 40-60% yield.



I a-d II a-d

I, IIa $X=N$, b $X=CNO_2$, c,d $X=CCOOC_2H_5$; a–c $Y=CH$, d $Y=N$

The molecular weight, determined mass spectrometrically, and the results of the elemental analysis of the synthesized compounds correspond to the calculated values. The IR and PMR spectra of adducts IIa-d confirm the proposed structure. Compound IIa, mp 260°C, yield 58%; compound IIb, mp 300°C, yield 60%; compound IIc, mp 246°C, yield 55%; compound IId, mp 200-202°C, yield 41%.

In the IR spectra of compounds obtained, the nitro group absorbs at 1550, 1340 cm^{-1}, the NH group of the azine fragment – at the 3300 cm^{-1} region; and OH – at 3450 cm^{-1}. In the mass spectra fragmentary ions with m/z 110 and $[M - 110]^+$ are recorded, corresponding to the resorcinol and hetaryl residues.

In a typical PMR spectrum (DMSO-D_6, TMS) there are characteristic one-proton singlets at 6.65 (7-H), 7.78 (2-H), 8.40 (5-H), 12.60 (4-H), 9.40, 9.60 ppm (OH) and doublets at 6.20 (2'-H, $J_{2',4'}$ = 3 Hz), 7.12 (5'-H, $J_{5',4'}$ = 9 Hz), as well as a doublet of doublets at 6.20 (4'-H, $J_{4',5'}$ = 9 Hz).

LITERATURE CITED

1. T. Kurihara and K. Nasu, Chem. Pharm. Bull., 30, 2723 (1982).

S. M. Kirov Ural Polytechnic Institute, Sverdlovsk 620002. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 8, pp. 1137-1138, August, 1986. Original article submitted March 14, 1986.