SYNTHESIS OF 1,2,3,4-TETRAHYDRO-3-OXO-1,2,4-TRIAZINOIMIDAZOLE SYSTEMS

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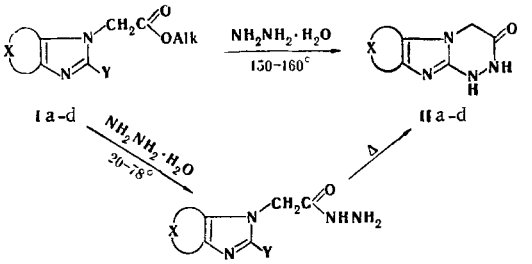
UDC 547.785.5'873.07

We have established that the final products in the reaction of condensed N-carboalkoxymethyl-2(8)-haloimidazoles (benzimidazoles, naphtho[1,2-d]imidazoles, and theophyllines) (Ia-d) with hydrazine hydrate in an organic solvent at 150-160°C in the presence of triethylamine or pyridine are the corresponding 1,2,3,4-tetrahydro-3-oxo-1,2,4-triazinoimidazoles (IIa-d). The initially formed corresponding hydrazides IIIa-d evidently split out a molecule of hydrogen halide under the reaction conditions to give derivatives IIa-d. In fact, we were able to isolate hydrazides such as III in the reaction of Ia-d with hydrazine hydrate in alcohol at 20-78°. Hydrazides III were converted to IIa-d in 30-90% yields when they were heated in dimethylformamide (DMF) containing triethylamine (or pyridine).



IIIa-d

Alk = CH_3; C_2H_5. Y = Cl; Br

I—III a X = 4,5-diphenyl; b X = condensed benzene; c X = naphthalene; d X = 1,3-dimethyl-2,6-dioxotetrahydropyrimidine ring

The individuality of IIa-d and IIIa-d was confirmed by thin-layer chromatography on Silufol UV-254, by the IR spectral data, and identification of IIb and IIc by means of samples obtained by alternative synthesis from the corresponding hydrazinoimidazoles and haloacetic acid esters. The results of elementary analysis of the synthesized compounds for C, H, and N were in agreement with the calculated values.

TABLE 1. Characteristics of the Compounds Obtained

Compound	Y	mp, °C (dec.)	Crystallization solvent	Yield, %
IIa	—	289—290	DMF—water	37
IIb	—	287—288	DMF	84
IIc	—	350	DMF	27—30
IId	—	273—275	Methanol—acetic acid	45
IIIa	Br	210—211	Methanol	57
IIIb	Cl	244—245	Water	83—85
IIIc	Cl	255—256	Ethanol	85—88
IIId	Br	235—236	Ethanol—water	95

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