REACTION OF FERVENULIN-4-OXIDE WITH ACETOACETIC ESTER HYDRAZONES

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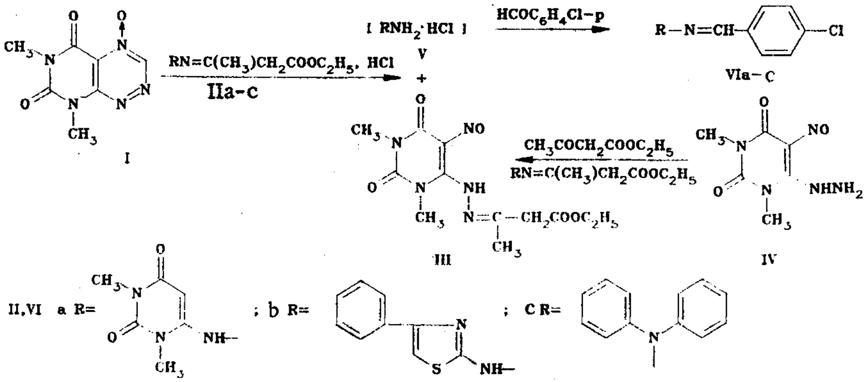
We have discovered that in the reaction of 5,6,7,8-tetrahydro-6,8-dimethylpyrimido[5,4-e][1,2,4]triazine-5,7-dione-4-oxide (fervenulin-4-oxide) (I) with an equimolar amount of acetoacetic ester hydrazones IIa-c in the presence of hydrochloric acid, 1,2,3,4-tetrahydro-1,3-dimethyl-5-nitroso-6-(1-carbethoxy-2-methylethylidene-2)hydrazinopyrimidine-2,4-dione (III) is formed in a 70-80% yield. Red crystals, mp 159-160°C (from ethanol). IR spectrum (in mineral oil): 1670, 1715, 1724 cm^{-1} (CO). The reaction proceeds in aqueous ethanol at room temperature for 12-15 h.



The structure of compound III was confirmed by its alternative synthesis from 1,2,3,4-tetrahydro-1,3-dimethyl-5-nitroso-6-hydrazinopyrimidine-2,4-dione (IV) and acetoacetic ester on heating in ethanol for 3-5 min.

After compound III had been removed from the reaction mixture, and the mother liquor had been treated with p-chlorobenzaldehyde, hydrazones VI were isolated in high yields (70-80%); these hydrazones were obtained from the corresponding known hydrazino derivatives V also.

It is possible that under the reaction conditions, the triazine ring of fervenulin-4-oxide is opened with the formation of nitrosohydrazine IV. The latter enters the transhydrazination reaction with V. In fact, it has been experimentally found that under similar conditions, nitrosohydrazine IV readily transhydrazinates hydrazones IIa-c to form compound III in nearly quantitative yield.

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