REACTION OF 1-METHYL-2(1H)-PYRIDONE WITH FUMARIC ACID AND ITS ESTER

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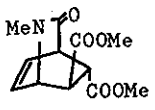
The further studies on the Diels-Alder reaction of 1-methyl-2(1H)-pyridone (I) with fumaric acid and its ester were carried out under the various conditions.

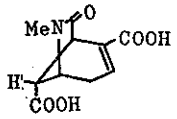
A) Reaction of I with dimethyl fumarate in toluene gave colorless needles (II) of mp 67-68° in about 4% yield, which was identified as dimethyl 2-methyl-3-oxo-2-azabicyclo[2,2,2]oct-7-ene-5-endo-6-exo-dicarboxylate by the comparison of its IR spectrum with that of the authentic sample.

B) Boiling of I and fumaric acid in water afforded a colorless crystalline powder (III) of mp 186-188° in 16.3% yield (the corrected yield 51.7%), which was an equimolar complex of I and the adduct (IV), besides the recovery of I in 68.4%. Treatment of III with 10% hydrochloric acid gave a colorless crystalline powder (IV) of mp 268-270°(decomp.) in a quantitative yield. The structure of IV was confirmed by its chemical and spectral property as 6-methyl-7-oxo-6-azabicyclo[3,2,1]oct-2-ene-2,8-endo-dicarboxylic acid.

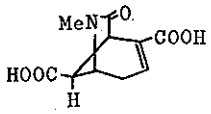
C) Heating of I and fumaric acid at 170° gave IV in 7.7% yield and colorless prisms (V) of mp 305-306°(decomp.) in about 10% yield. The structure of V was chemically determined as 6-methyl-7-oxo-6-azabicyclo[3,2,1]oct-2-ene-2,8-exo-dicarboxylic acid.



II



IV



V