LETTERS TO THE EDITOR

NEW METHOD OF SYNTHESIZING ALKYL ESTERS OF FUROXANDICARBOXYLIC ACID

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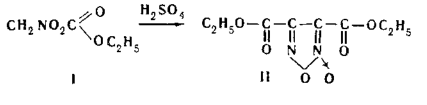
By the action of sulfuric acid on ethyl nitroacetate (I) we have isolated diethyl furoxandicarboxylate (II):



The structure of II was confirmed by the analogy of the IR spectrum with that of an authentic sample [1] and by its conversion into furoxandicarboxyamide by treatment with an aqueous solution of ammonia.

Diethyl furoxandicarboxylate (II). 13.3 g (0.1 mole) of I was added to 49 g (0.5 mole) of sulfuric acid (d_4^{20} 1.84) in such a way that the temperature did not exceed −5° C. After the reaction mixture had been kept at the same temperature for 12 hours, it was poured into water, and the oil that separated was extracted with ether. The ethereal extract was washed with water and dried with sodium sulfate. Distillation yielded II.

Esters of furoxandicarboxylic acid have been obtained from other alkyl nitroacetates under similar conditions.

The substances synthesized and their yields and constants are given in the table.

REFERENCE

1. H. R. Snyder and N. E. Boyer, J. Am. Chem. Soc., 77, 4238, 1955.

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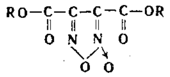

R	Bp, °C (pressure, mm)	d_4^{20}	n_d^{20}	MR_D		Empirical formula	Found, %			Calculated, %			Yield, %
				found	Calculated		C	H	N	C	H	N	
CH_3	112/2	1.410	1.486	41.14	40.90	$C_6H_6O_6N_2$	35.63	3.05	13.72	35.15	2.97	13.86	67
C_2H_5	113/2	1.2783	1.475	50.67	50.20	$C_8H_{10}O_6N_2$	42.03	4.02	12.33	41.78	4.35	12.17	65
C_3H_7	130/2	1.2051	1.472	59.95	59.50	$C_{10}H_{14}O_6N_2$	46.17	5.42	10.68	46.51	5.42	10.85	61
C_4H_9	143/2	1.1542	1.470	69.14	68.79	$C_{12}H_{18}O_6N_2$	49.73	6.23	10.06	50.00	6.25	9.73	73

REACTION OF sym-OCTAHYDROACRIDINE N-OXIDE WITH ACETIC ANHYDRIDE IN THE PRESENCE OF AN AROMATIC ALDEHYDE

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It is known that when sym-octahydroacridine N-oxide (I) is boiled with acetic anhydride, the acetate of sym-octahydroacridin-4-ol is formed [1]. We have found that if equimolecular amounts of I and p-nitrobenzaldehyde (PNBA) are boiled in acetic anhydride, C-acetoxylation takes place at one methylene group and condensation at another. No such reaction has previously been performed in the cycloalkylpyridine N-oxide series. The structure of the acetate of 4-(p-nitrobenzylidene)-sym-octahydroacridin-5-ol (II) formed was confirmed by independent synthesis. When equimolecular amounts of the previously-described [1] sym-octahydroacridin-4-ol (III) and PNBA are boiled in acetic anhydride, II is formed. On being heated with 20% HCl, the latter is hydrolyzed to 4-(p-nitrobenzylidene)-sym-octahydroacridin-5-ol (IV). The dehydration of IV with polyphosphoric acid leads to 4-(p-nitrobenzylidene)-1,2,3,4,7,8-hexahydroacridine (V).