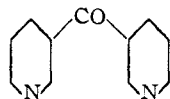


what modified process, whereby the vapors of nicotinic acid were passed through a heated column of thorium dioxide, gave a comparable yield of the ketone. This was identified by analysis of the dipicrate. The same result was obtained when aluminum oxide was used in place of thorium dioxide.



Experimental

β,β' -Dipyridyl Ketone.—A Pyrex tube, 25 inches long, closed at one end, was charged with 20 g. of nicotinic acid, followed by 100 g. of thorium dioxide. The outlet was connected with a descending condenser and the receiving flask was immersed in an ice-bath. Nicotinic acid was slowly distilled through the thorium oxide layer which was kept at 300° during the process. The distillate was collected, and pyridine and water were boiled off at atmospheric pressure, leaving a dark residue which was extracted with ether. After the ether was evaporated, this extract yielded 0.4 g. of a viscous oil. The dipicrate, formed in alcoholic solution and recrystallized from alcohol, was in dark green plates; m. p. 135°.

Anal. Calcd. for $C_{11}H_8N_2O \cdot 2C_6H_5N_3O_7$: C, 42.99; H, 2.17. Found: C, 43.00; H, 2.02.

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The Relative Efficiency of Some Polymerization Inhibitors¹

BY ROBERT L. FRANK AND CLARK E. ADAMS

Considerable losses often occur in the preparation of vinyl monomers due to their ease of polymerization, especially during distillation. The present comparison of inhibitors was undertaken to prevent such losses.

Equal weights (0.20 g.) of a number of compounds were added to 2.0-ml. samples of three monomers, styrene, 3,4-dichlorostyrene, and 5-ethyl-2-vinylpyridine, each freshly distilled. These monomers were chosen for their tendency toward ready polymerization. Each sample was sealed in a 10 × 110-mm. test-tube and allowed to stand in a refluxing water-bath. No effort was made to exclude oxygen from the tubes, but all were of the same size and had approximately the same air space above the monomer-inhibitor mixtures. The fluidity of the samples was periodically compared with the initial fluidity by means of the flow-times when the tubes were inverted. The heating time required to show a difference in flow-time is given in Table I as the "inhibition period." At the "total time of polymerization" the samples were too viscous to give a measurable flow time.

There appears to be no great variation in the order of inhibitory strength from one monomer to

(1) This investigation was carried out under the sponsorship of the Office of Rubber Reserve, Reconstruction Finance Corporation, in connection with the Government Synthetic Rubber Program,

another. A good inhibitor for one monomer is likely to be good for another.

TABLE I
EFFECT OF INHIBITORS ON POLYMERIZATION

Inhibitor	Inhibition period, hr.			Total time of polymerization, hr.		
	S ^a	DCS ^a	VEP ^a	S ^a	DCS ^a	VEP ^a
Picric acid	299	39	<120	>490	130	>120
Trinitrobenzene	299	39		>490	82	
2,5-Dihydroxy-1,4-benzoquinone	154	32		442	82	
1,4-Naphthoquinone	81	22	120	231	66	>120
1,4-Benzoquinone	81	22	<120	130	66	>120
Chloranil	81	9		>490	34	
9,10-Phenanthra-quinone	57	17		130	66	
<i>t</i> -Butylcatechol	34	9	12	154	17	72
4-Amino-1-naphthol	9	9	<120	130	17	>120
Hydroquinone	9	9	24	22	17	>120
Phenyl- β -naphthyl-amine	9	<9	12	17	9	72
Triphenyl phosphite	<9	<9		9	9	
Control	<9	<9	12	9	9	24

^a S stands for styrene; DCS for 3,4-dichlorostyrene; VEP for 5-ethyl-2-vinylpyridine.

One fact that stands out in the present study is that phenyl- β -naphthylamine and *t*-butylcatechol, two widely used inhibitors, are among the poorest in inhibitory action at least for the monomers tried.

Picric acid and trinitrobenzene are now being used in this Laboratory with great success during distillation of a wide variety of monomers. Some question has arisen concerning the possible hazard from explosion of polynitro compounds, but the use of traces should involve no danger.² Picric acid should probably not be used, however, in metal containers.

(2) Cf. Belyaev and Yuzefovich, *Compt. rend. acad. sci. (URSS)*, **27**, 133 (1940); *C. A.*, **34**, 7607 (1940).

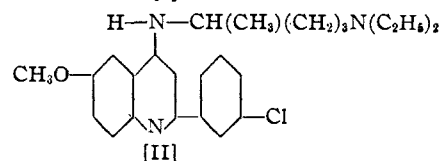
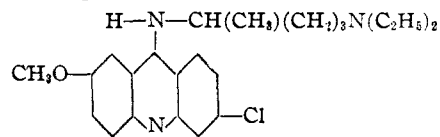
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Anils as "Open Models" of a Modified Atebrin

BY HENRY GILMAN AND SAMUEL P. MASSIE, JR.

In a recent study¹ concerned with some quinolines patterned as so-called open models of atebrin, [I], it was shown that a compound like 6-methoxy-2-(3'-chlorophenyl)-4-[(α -methyl-diethylaminobutyl)-amino]-quinoline, [II], was active in experimental avian malaria infections.



(1) Gilman and Spatz, *This Journal*, **66**, 621 (1944).