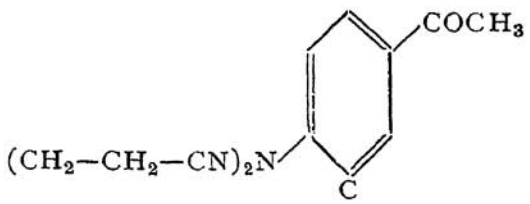
STUDIES IN CYANOETHYLATION

DURING the course of studies in cyanoethylation we investigated the reaction of *p*-aminoacetophenone (I) with acrylonitrile (II).

One mole of (I) and four moles of (II) were heated together in dioxan solution in the presence of triton-B at 30 to 40° C. for about two hours. After diluting the mixture with ice and acetic acid, it was kept overnight in a refrigerator when a colourless solid separated out. It could be crystallised from benzene or ethylacetate in colourless needles, m.p. 135° C. (Found : C, 69·6 ; H, 6·4 ; N, 17·0%. $C_{14}H_{15}N_3O$ requires C, 69·7 ; H, 6·3 ; N, 17·4%).

The results of analysis indicate that the condensation product (III) could have one of the following structures :



Hydrolysis of III with potassium hydroxide yielded colourless needles of an acid crystallised from water, m.p. 142° C. (Found : C, 59·8 ; H, 6·1 ; N, 5·2%. $C_{14}H_{17}NO_5$ requires C, 60·22 ; H, 6·09 ; N, 5·02%).

The molecular weight of the acid by the silver-salt method was found to be 251 which agrees with the calculated value at 279 for a dibasic acid.

The compound III fails to give an acetyl derivative nor does it form a quaternary salt on treatment with methyl iodide. These observations rule out the possibility of structures B and C for III. The latter on treatment with nitrous acid gives a yellow crystalline derivative crystallisable from alcohol having m.p. 125° which gives the Liebermann's test for N-nitrosoamines. (Found : N, 21 ; $C_{14}H_{14}N_4O_2$ requires N, 20·6%).

These data support the structure A for III which is also obtained by reaction of I and two moles of II at 30° C. or at higher temperatures.

Further work on structure III is in progress and will be reported elsewhere.

Institute of Science, J. R. MERCHANT.
Bombay, *April* 10, 1962. H. C. KAUSHIK.