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Synthesis of Certain Chalcones

by

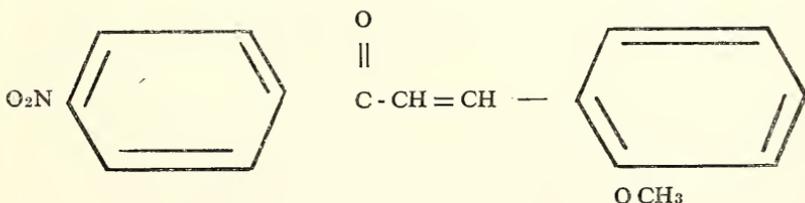
Kamalakar B. Raut*

Preparations of certain chalcones for biological studies^{1,2} using two different temperatures, 0°C and 60°C is reported. The yields in cold condensations are better than yields at 60°C. The reactions are described below.

Experimental.

Preparation of 1-(4-nitrophenyl)-3-(2-methoxyphenyl) propenone. (I)

In a 125 ml erlenmeyerflask 1.65 g. (0.01 mole) of-nitroacetophenone and 1.51 g. (0.01 mole) of o-methoxybenzaldehyde were dissolved in 50 ml of ethanol. Seven drops of 50% potassium hydroxide solution were added to the reacting mixture. The mixture was then kept at 0°C for 14 hours. After that period the solution was neutralized with a 3% hydrochloric acid solution dropwise. The precipitate was washed with water and crystallized from hot ethanol. Similarly other compounds were prepared. These compounds are being tested for biological activity at the Medical College in Charleston, South Carolina. The results of these tests will be communicated later.



¹Clerk, Samuel F., U. S. Patent 3,805, 184, September 3, 1957, C. A. 62, 2325 (1958).

²Marrion, D. H., Russel, P. B. and Todd, A. R., J. Chem. Soc., 1934, 218.

*The investigator is indebted to Mr. Johnny Weatherspoon, Chemistry Student at Savannah State College for his cooperation and assistance.

Condensation products formed by reacting p-nitroacetophenone with aldehydes.

Aldehyde	M.P.	Reaction Time	Coloration with Con. H ₂ SO ₄
o-methoxybenzaldehyde	143-4	40 hrs.	From yellow to light red
Anisaldehyde	142-3	20 "	Ditto
1-naphthaldehyde	183-5	36 "	From yellow to green
p-dimethylaminobenzaldehyde	210-2	19 "	From red to yellow
Vanillin	74-6	16 "	None

Synthesis of some new azo dyes

By

Kamalakar B. Raut

Friz Ullman discovered two very useful copper-catalysed reactions of aryl halides, and both bear his name in common usage. In the second of his reactions he found that O-chlorobenzoic acid and copper powder in refluxing aniline gave N-phenylanthranilic acid. Subsequently study showed that pure O-chlorobenzoic acid does not react with aniline, but that minute quantities of copper salts are sufficient to catalyse the reaction, leading to a satisfactory yield. Salts of iron, nickel, platinum and zinc also had some catalytic activity, but manganese and tin salts were ineffective. Performing the reaction in the presence of potassium carbonate gave superior results, because alkali salts of N-phenylanthranilic acid are less prone to undergo decarboxylation and because acid catalysed resinification reactions are avoided. Additional papers from Ullmann's laboratory showed that a wide variety of anthranilic acids and other diarylamines can be prepared by condensing aniline with O-chlorobenzoic acids or other aryl halides, or by employing, for instance anthranilic acid and bromobenzene. These variations of the synthesis were usually run in refluxing amyl alcohol or nitrobenzene.

It was soon recognized that copper facilitates the condensation of unactivated or slightly activated aryl halides with all sorts of nucleophilic reagents. For instance, the reaction of bromobenzene with potassium phenoxide at 200°C for 12 hours gives only a trace of diphenyl ether, but the same ingredients with a little copper powder give an 87 per cent yield of diphenyl ether in only 2.5 hours. Copper catalysis of condensations of slightly activated aryl halides with amines, ammonia, phenoxides, alkoxides, and hydroxide has found wide application in synthetic work.

The Rosenmund-von Braun nitrile synthesis, which involves the condensation of aryl halides with cuprous cyanide, can be regarded as a special case of the Ullmann reaction. Good yields of aromatic nitriles are obtained by means of it, but in the absence of ions of copper (or neighboring metals) scarcely any nitrile is produced from aryl halides and alkali cyanides.

Another interesting variation is the copper-catalysed condensation of sodium O-bromobenzoate with the sodium derivatives of ethyl malonate and similar active methylene compounds. Both copper metal and copper acetate were effective; the latter also catalysed the hydrolyses of O-bromobenzoic acid to salicylic acid by only 30 minutes boiling in aqueous sodium acetate solution. Strangely, ethyl O-bromobenzoate would enter into none of these reactions, nor would p-bromobenzoic acid.

From the above review it will be seen that copper serves as an efficient catalyst in many organic reactions. All these reactions how-

ever consist in the replacement of the aromatic halogen by other groups. Ordinarily use of copper powder for activating halogen in monohalogen aliphatic compounds is not necessary. But with increasing number of halogen atoms the reactivity of the halogen atoms diminishes considerably. Thus chloroform and carbon tetrachloride are only feebly reactive and require high temperature with amino compounds.

We are interested in finding out whether copper powder will react similarly as a catalyst in the reactions of aromatic amines and polyhalogenated compounds like carbon tetrachloride and chloroform.

In our preliminary work we have isolated a number of new compounds (Table I). It was expected that these compounds will have a secondary or tertiary amino group but all these compounds gave a positive test for a primary amine. These compounds have been coupled with B-naphthol and a new series of azo dyes have been synthesized. The properties of these azo dyes are under investigation and will be reported separately.

Table I
Summary of Results

Condensations of	Reacting Period	M. P.	M.P. OF the dye
1. Aniline + Chloroform	9 hours 90°C	168-9°C	126°
2. Aniline + Carbon Tetrachloride	8 hours 90°C	146°	118-20°
3. Aniline + Bromoform	12 hours 90°	140°	110°
4. Aniline + 1, 1, 2, 2-tetrachloroethane	8 hours 196-8°	150-2	97°
5. p-phenetidine + Chloroform	3 days Room temp.	171°	98°
6. p-phenetidine + Carbontetrachloride	4½ days Room temp.	189-90°	157-8°
7. p-phenetidine + 1, 1, 2, 2, tetra chloroethane	8 hours 198°	178°	80-3°
8. p-Toluidine + Carbon tetrachloride	8 hours 90°	158°	113-4°
9. p-Anisidine + Chloroform	4 hours 90°	167°	105-8°
10. p-Anisidine + Carbon tetrachloride	21 hours	119°	119°