

not having a large amount of calcium oxychloride is obvious. The navy blue tint cannot be mistaken for the purple tint of aniline.

Ethylaniline.—Monoethylaniline, according to Hofmann,¹ also gives no color reaction with bleaching powder solution. Kahlbaum's ethylaniline was purified by two fractionations, with the aid of a Glinsky apparatus and made up to a 1/100 M solution. It gave an analogous color reaction to that of methylaniline, except that the blue color goes through green and dark brownish hues, before finally leaving a yellow solution. Also the time of appearing and the period of retaining color is two to three times longer than with methylaniline. It is especially to be noted that an excess of the reagents should be avoided. The tests were also found to be very delicate in showing small quantities of the base. An interpretation, similar to that given above for the production of a dye in the case of methylaniline, would also be possible here. It is quite likely that all monoalkylanilines will be found to show a similar behavior; no further representatives have been examined by me.

THE PREPARATION OF BENZOYLCHLOROAMIDE.

BY RASIK LAL DATTA AND TARAPADA GHOSH.

Received June 24, 1913.

Benzoylchloroamide² is generally prepared by treating a cold aqueous solution of benzamide with acetic acid and then adding a concentrated solution of bleaching powder and finally extracting with ether. On evaporation of the ethereal solution and crystallization of the residue from water, the substance is obtained in fine needles. The addition of bleaching powder solution and extraction with ether have to be repeated to completely transform all the benzamide present in solution into benzoylchloroamide. Since benzamide is only sparingly soluble in water, the quantity turned out at each operation even with a large amount of water is necessarily small. Moreover, it necessitates the employment of a good quantity of ether, the free use of which is troublesome and expensive. Besides, the method is very clumsy and inconvenient.

Linebarger³ showed that benzoylbromamide, which is prepared by the action of bromine and caustic potash on benzamide, when treated with concentrated hydrochloric acid is transformed into benzoylchloroamide. But as is evident, this is not a very satisfactory method of preparing the compound.

Having had to prepare large quantities of the compound, we found both the above methods unsuited for our purpose. This led to a search for a better method of preparing the compound and it is found that chlorine is

¹ *Ann.*, 77, 130 (1851).

² *Bender, Ber.*, 15, 410 (1882).

³ *Am. Chem. J.*, 16, 218 (1894).

able to directly transform benzamide into its monochloro derivative, without the help of any other reagent.

Preparation.—Chlorine gas after being washed by passing through water, is led into a flask in which finely powdered benzamide is suspended in water. At the outset, the mass appears very light but as the current of chlorine is passed for some time, the mass gradually assumes a yellowish color and appears to become heavy and increase in bulk. After passing the current of chlorine for several hours, according to the quantity of the substance taken, the reaction becomes complete and the benzamide is quantitatively transformed into benzoylchloroamide. Though in this reaction much hydrochloric acid is formed, yet the transformation becomes complete since benzamide does not unite even loosely with hydrochloric acid. As there is no physical indication by which the transformation of benzamide into benzoylchloroamide can be judged complete, it is generally expedient to pass the current of chlorine for a good length of time and test the melting point of a portion of the resulting substance after recrystallizing from hot water. The pure substance gives a melting point, 116° , but if the substance gives a lower melting point, then the substance is contaminated with some unchanged benzamide, in which case the chlorination has to be continued for some time longer. Since the substance is not decomposed by water, it may be kept in water for a day or two during the process. When a portion of the substance gives the correct melting point, the product is filtered off from the chlorinated mother liquor, washed with water and finally recrystallized from boiling water, when it is obtained in fine crystallin needles, which have a sharp melting point, 116° , previous observers giving 116° (Bender), 113° (Linebarger).

The hypochlorite method used by Bender has been varied a little with the following results: Instead of taking an aqueous solution, finely powdered benzamide is suspended in water in a separating funnel and to it some acetic acid is added and then a concentrated solution of bleaching powder, when an immediate haziness is produced which on shaking soon collects to a yellow oil. The addition of bleaching powder solution is repeated with the production of a similar haziness collecting to an oil. When the further addition of bleaching powder does not produce any haziness, the oil is taken up in ether and the ethereal extract evaporated when a yellowish solid is obtained. The solid is not at all soluble even in boiling water and could not be recrystallized therefrom. It has not a sharp melting point. It seems to melt with decomposition between 153° – 163° .

We are at present engaged in studying the compound with a view to ascertain its nature and composition.