coils can pass through by twisting over the wires in the screen.

The rate at which the coils can be wound on the steel rod is about 150 cc. of unbroken spiral coils per hour. We broke our helices into one turn coils, with limits of from three-quarters to one and a half turns. The breaking and sorting of our helices required about one hour for 5 cc. of finished product. We have found that from one liter of the long unbroken spirals we obtain about 100 cc. of product and 20 cc. of discarded material—mainly half-turn size. Our coils were wound to have a fiber diameter of about 0.6 mm., and adjacent turns were practically touching; the outside diameter of the coils was 4.4 mm. Some measurements upon average pieces of the finished product are as follows: weight of 300 individual coils, 2.43 g., which occupy a volume of 5 cc. in a graduated cylinder of 17 mm. inside diameter, thus closely approximating the packing conditions in one of our columns.

From these data, and the dimensions of our column No. 3, we can calculate the following: Column No. 3 in which we placed 900 cc. of packing was 16 mm. internal diameter, and had a packed section of 4.6 meters.[2] The packing consisted of between 50,000 and 60,000 coils, with an actual volume of 194 cc. of glass, a surface of about 14,000 sq. cm., and a free space of about 80%.

(2) This column was subsequently used for the separation of *cis* and *trans* butene-2, boiling less than 2° apart. See Kistiakowsky, Ruhoff, Smith and Vaughan, THIS JOURNAL, **57**, 876 (1935).

MALLINCKRODT CHEMICAL LABORATORY
HARVARD UNIVERSITY
CAMBRIDGE, MASS. RECEIVED SEPTEMBER 7, 1934

The Behavior of β-*p*-Dimethylaminobenzoin with Alcoholic Hydrochloric Acid

BY KONOMU MATSUMURA

On refluxing a mixture of β-*p*-dimethylaminobenzoin (I) and alcoholic hydrochloric acid with or without the addition of copper sulfate, *p*-dimethylaminobenzil (II) and α-*p*-dimethylaminodesoxybenzoin (III) were formed and on reduction of *p*-dimethylaminobenzil with tin and hydrochloric acid, I could isolate α-*p*-dimethylaminodesoxybenzoin (III) in accordance with the scheme

$$(CH_3)_2NC_6H_4COCH(OH)C_6H_5 \xrightarrow{\text{Alc. HCl}} \underset{\text{I}}{(CH_3)_2NC_6H_4CH_2COC_6H_5} \underset{\text{III}}{} + \underset{\text{II}}{(CH_3)_2NC_6H_4COCOC_6H_5} \xrightarrow[\text{alc. HCl}]{\text{Sn +}} \text{III}$$

with the simple benzoin or benzanisoin similar reaction could not be observed to take place.

Experimental

Treatment of β-*p*-Dimethylaminobenzoin with Alcoholic Hydrochloric Acid.—A mixture of β-*p*-dimethylaminobenzoin (2 g.), alcohol (16 cc.), concd. hydrochloric acid (3 cc.) and copper sulfate (0.5 g.) was refluxed for five hours. On addition of water, a yellow solid of m. p. 110–115° separated. Crystallized from alcohol, it formed yellow prisms (1.2 g.) melting at 115–116° alone or mixed with an authentic specimen of *p*-dimethylaminobenzil (m. p. 115–116°).

Anal. Calcd. for $C_{16}H_{15}O_2N$: N, 5.53. Found: N, 5.45.

The filtrate on being made alkaline with ammonia gave a colorless solid which afforded colorless needles (0.45 g.) from alcohol melting at 127–128° alone or mixed with an authentic specimen of α-*p*-dimethylaminodesoxybenzoin (m. p. 127–128°).

Anal. Calcd. for $C_{16}H_{17}ON$: N, 5.86. Found: N, 5.74.

An experiment in a similar manner without the addition of copper sulfate gave also the benzil and α-desoxy compound both in poor yields

Reduction of *p*-Dimethylaminobenzil.—A mixture of *p*-dimethylaminobenzil (1.4 g.), tin foil (2 g.), concd. hydrochloric acid (2.5 cc.), copper sulfate (0.2 g.) and alcohol (8 cc.) was refluxed for four hours, then made alkaline with sodium carbonate. The resulting precipitate, after being dried, was extracted with hot alcohol. The alcoholic extract on concentration gave colorless prisms (0.8 g.) melting at 127–128° alone or mixed with an authentic specimen of α-*p*-dimethylaminodesoxybenzoin.

CHEMICAL LABORATORY OF
KITASATO INSTITUTE
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Oxime of β-*p*-Dimethylaminobenzoin

BY KONOMU MATSUMURA

It was found by Buck and Ide[1] that the interaction of β-*p*-dimethylaminobenzoin and hydroxylamine in alcoholic sodium hydroxide yields the oxime of m. p. 140° which on Beckmann transformation affords benzaldehyde and *p*-dimethylaminobenzoisonitrile, and they assigned the structure of *syn*-phenyl oxime for their product. On the study of this benzoin, the present author obtained by another method the oxime of m. p. 184° and by its Beckmann change, the formation of benzaldehyde and *p*-dimethylaminobenzonitrile could be ascertained, showing that the oxime now obtained would be the other stereoisomeride, and leading to the same structure for β-*p*-dimethylaminobenzoin as was deduced by Buck and Ide from a Beckmann reaction on the oxime of m. p. 140°.[1]

Experimental

β-*p*-Dimethylaminobenzoin Oxime.—A mixture of β-*p*-dimethylaminobenzoin (2 g.), hydroxylamine hydrochlo-

(1) Buck and Ide, THIS JOURNAL, **53**, 1912 (1931).