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%.

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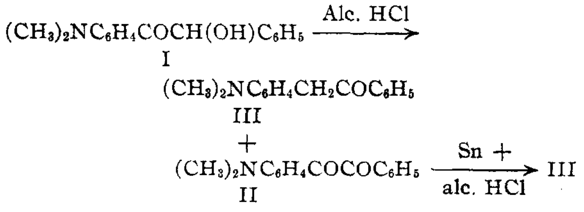
(2) This column was subsequently used for the separation of *cis* and *trans* butene-2, boiling less than 2° apart. See Kistiakowsky, Ruhoff, Smith and Vaughan, THIS JOURNAL, **57**, 876 (1935).

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



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

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
TOKYO, JAPAN
RECEIVED SEPTEMBER 17, 1934

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-

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

ride (1 g.), sodium acetate (1.5 g.) and some alcohol was refluxed for four hours. The product gave colorless prisms from alcohol, m. p. 184°; yield, 2 g.

Anal. Calcd. for $C_{16}H_{18}O_2N_2$: N, 10.37. Found: N, 10.21.

Beckmann Change with the Oxime.—Thionyl chloride (11 g.) was added dropwise into the ice cold suspension of the oxime (1 g.) in chloroform (50 cc.). The resulting solution, after standing, was shaken with ice water. Subsequent to removal of chloroform the residual yellow oil (1 g.) was dissolved in ether and treated with concd. sodium bisulfite. After being dried with potassium carbonate, and removal of ether, 0.5 g. of oil which soon turned to colorless prisms was obtained. It was pressed on a tone plate and crystallized from ether, m. p. 75–76°. Folin found the same m. p. for his specimen of *p*-dimethylaminobenzonitrile.[2]

Anal. Calcd. for $C_9H_{10}N_2$: C, 73.97; H, 6.85; N, 19.18 Found: C, 74.23; H, 6.78; N, 19.26.

On working up the bisulfite solution, 0.4 g. of benzaldehyde was obtained. It was identified by converting it into phenylhydrazone which alone or mixed with a known specimen melted at 155–156°.

Anal. Calcd. for $C_{13}H_{12}N_2$: N, 14.29. Found: N, 14.28.

***p*-Dimethylaminobenzoic Acid.**—A solution of the nitrile (0.2 g.) and potassium hydroxide (1 g.) in alcohol (9 cc.) and water (1 cc.) was refluxed for eight hours until liberation of ammonia had ceased. The product gave colorless prismatic needles from alcohol, m. p. 235° (dec.).

Anal. Calcd. for $C_9H_{11}O_2N$: N, 8.48. Found: N, 8.64.

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[2] Folin, *Am. Chem. J.*, **19**, 333 (1897).

COMMUNICATIONS TO THE EDITOR

THE ISOMERIZATION OF NORMAL HEPTANE

Sir:

C. D. Nenitzescu and A. Dragan have reported [*Ber.*, **66**, 1892 (1933)] that *n*-hexane and *n*-heptane heated on a water-bath in the presence of aluminum chloride yield a large amount of isohexane and isoheptane, respectively. The data presented by these authors do not substantiate these statements with great certainty: the starting materials were not very pure, the products obtained boiled over wide ranges, and the assertion regarding the compounds formed is based only on these boiling ranges, without the corroborating evidence of other physical properties. A. D. Petrow, A. P. Meschtscherjakow and D. N. Andrejew [*ibid.*, **68**, 1 (1935)] state that *n*-heptane is isomerized in 25% yield by heating for six hours at 300–400° in the presence of zinc chloride. In this case the density of the fractions obtained is obviously too high to correspond to any of the branched-chain heptanes. We have repeated the work of Nenitzescu and Dragan, using 2650 g. of pure *n*-heptane from Jeffrey pine. The product boiling from 50 to 98.4° was carefully fractionated, and the following properties determined for the fractions: $n^{20}\text{D}$, average molecular weight (by vapor density), and critical temperature of solution in aniline. A comparison of these data with the properties of *n*-hexane and all the heptanes indicates the presence of *n*-hexane and 2-methylhexane, and of no other isomeric heptane.

We estimate that the *n*-hexane found represents about 1% and the 2-methylhexane about 4% of the *n*-heptane consumed in the reaction.

A further investigation of this reaction is in progress.

RESEARCH LABORATORIES
ETHYL GASOLINE CORPORATION
DETROIT, MICHIGAN

GEORGE CALINGAERT
DONAL T. FLOOD

RECEIVED JANUARY 18, 1935

ERGOTOCIN: THE ACTIVE PRINCIPLE OF ERGOT RESPONSIBLE FOR THE ORAL EFFECTIVENESS OF SOME ERGOT PREPARATIONS ON HUMAN UTERI

Sir:

It has been found by the authors, working in conjunction with Drs. Davis, Adair and Rogers of the Department of Obstetrics and Gynecology of The University of Chicago, that the alkaloids ergotoxine, ergotamine and sensibamine are uniformly ineffective when administered orally to human mothers in doses of 2 mg. Larger doses (2–4 mg.) often induce unpleasant side reactions such as nausea, vomiting, increase in blood pressure, diarrhea, etc. However, even