from petroleum ether (b. p. 60–110°) as white needles of m. p. 110–110.5°. A mixed melting point with the compound obtained by the oxidation of desoxyvasicine melted at 110–110.5°, thus indicating the identity of the two products.

Anal. Calcd. for $C_{11}H_{10}O_2N_2$: C, 71.51; H, 5.38; N, 15.05. Found: C, 71.51; H, 5.62; N, 14.83.

Oxidation of 2,3-Trimethylene-4-quinazolone to 2,3-(α-Hydroxytrimethylene) - 4 - quinazolone (XI).—To a solution of 0.200 g. of 2,3-trimethylene-4-quinazolone in 15 cc. of thiophene-free, dry benzene was added 0.600 g. of freshly prepared lead tetraacetate. The mixture was well stirred and heated at 50–60° for twenty hours. After filtering, the solution was evaporated and the residue extracted with chloroform. Upon evaporation of the chloroform a light yellow oil remained. It was shaken with a 5% sodium carbonate solution until the two layers had disappeared. The product was then extracted with chloroform and the chloroform evaporated. The residue was extracted three times with 5-cc. portions of hot petroleum ether (b. p. 60–110°) to remove the starting material, over half of which was recovered. The residue was crystallized from a mixture of benzene and petroleum ether (b. p. 60–110°) as white microscopic needles, m. p. 211–212° (dec.); yield 15 mg. A mixed melting point with the oxidation product of vasicine melted at 212° (dec.), indicating the identity of these two products.

Anal. Calcd. for $C_{11}H_{10}O_2N_2$: C, 65.34; H, 4.96. Found: C, 65.52; H, 4.86.

Summary

1. Vasicine and desoxyvasicine have been oxidized to the corresponding 4-keto derivatives.

2. The structure of the oxidized desoxyvasicine was proven by synthesis.

3. Oxidized desoxyvasicine reacts with lead tetraacetate to give a hydroxy derivative identical with oxidized vasicine.

4. These facts indicate that the hydroxyl in vasicine is on the methylene attached to the 2-carbon atom.

URBANA, ILL. RECEIVED APRIL 22, 1935

NOTES

Note on the Construction of Glass Helices for Fractionating Column Packing

BY EDWIN E. ROPER, GEORGE F. WRIGHT, JOHN R. RUHOFF AND WALTER R. SMITH

In the past year we have prepared a large quantity (1600 cc.) of broken glass helices for fractionating column packing of the type first described by Wilson, Parker and Laughlin.[1] From this work we feel we have gained experience that may prove valuable in the fabrication, breaking and sorting of the helices.

We have followed, in general, the method outlined by Wilson, Parker and Laughlin for winding the helices. We use 75 cm. lengths of one-eighth inch diameter steel rod for the winding forms and wind the spiral coils from a 3-mm. Pyrex rod. This requires an oxygen flame about 4 cm. high, and precludes the use of brass as a winding form. The steel rods are drilled at one end (1 mm. diameter hole) and then made slightly conical toward this end by the use of emery paper. This facilitates the removal of the wound coil. The winding form is given a very thin coat of natural graphite by rubbing it with a cloth upon which the graphite has been sprinkled, together with a drop of light lubricating oil if necessary; this makes the removal of the finished spiral much easier, and also helps in the rotation of the form while the spiral is being wound. It is relatively simple to prepare spiral coils 40 cm. in length, which can be removed in one piece if desirable.

We use a simple bearing for the left end of the winding form, and the rotation is performed with the left hand, while the feeding of the Pyrex rod and the regulating of its angle to the long axis of the winding form are done with the right hand.

The long spiral coils obtained from the winding operation are threaded on to a convenient length (*ca.* 60 cm.) of bare No. 18 copper wire, and the blade of a thick knife is pressed down between each turn in order to break the fiber. The more closely the coil is wound, the easier is this step. Small helices of any desired number of turns or fractions of turn can be formed from the long coils by taking care to break off a definite number of turns from the end of the large helix. For instance, we wished to make single turns from our long coils; however, if we attempted to break off exactly one turn, a large number of the coils were less than three-quarters of a turn, but by attempting to break off one and a quarter turns, we obtained coils which were largely one turn, and only relatively small numbers of larger and smaller coils.

The broken coils of less than one turn fall off from the wire. The breaking is done over a black cloth, so that the material which falls off the wire can be collected and sorted more easily. The material which remains on the wire is sorted as it is removed. The coils that consist of more than the desired number of turns are best broken by hand over the black cloth and then this material is added to that which fell off the wire during the breaking operation. We have tried screening this combined lot and have found that a small (20-mesh) screen will take out the finer particles (one-quarter turn or less) and that an ordinary kitchen colander (circular holes about 4 mm. in diameter, spaced about 7 mm. between centers) will allow the half-turn size to drop through and retain the majority of the three-quarter turn size. An eight mesh wire screen does not do this nearly as satisfactorily, since the larger size

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(1) (a) THIS JOURNAL, **55**, 2795 (1933); (b) **56**, 1396 (1934).

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).

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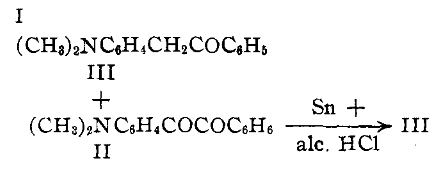
RECEIVED SEPTEMBER 7, 1934

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



$$\underset{\text{I}}{(CH_3)_2NC_6H_4COCH(OH)C_6H_5} \xrightarrow{\text{Alc. HCl}} \underset{\text{III}}{(CH_3)_2NC_6H_4CH_2COC_6H_5} + \underset{\text{II}}{(CH_3)_2NC_6H_4COCOC_6H_6} \xrightarrow[\text{alc. HCl}]{\text{Sn +}} \text{III}$$

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.

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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).