We conclude that, within our experimental error, phosphorus pentoxide is as efficient a desiccant at 90° as it is at room temperature. Alternatively, if we suppose that a gain in weight of as much as half a milligram escaped our notice, we can state that the aqueous pressure in the air passing the phosphorus pentoxide at 90° under our experimental conditions did not exceed by over 0.0009 mm. the aqueous pressure which passed both magnesium perchlorate and phosphorus pentoxide at room temperature.

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The Preparation of 1-Naphthonitrile

BY MELVIN S. NEWMAN

The preparation of 1-naphthonitrile from 1-naphthylamine by the Sandmeyer reaction is tedious and the yields often leave much to be desired. Recently, Rupe and Brentano[1] have reported an improved method by which yields of 75–78% may be obtained, but the process requires much in the way of time and attention.

The replacement of aromatically bound halogen by the cyano group has been carried out in several instances by heating the organic halogen compound with anhydrous cuprous cyanide, with or without added organic base, such as pyridine or quinoline. Thus, 1-naphthonitrile was prepared from 1-chloronaphthalene[2] and other halogen containing compounds[3] have been converted into the corresponding nitriles in excellent yields. The present report deals with the preparation of 1-naphthonitrile from 1-bromo- and 1-chloronaphthalene in yields of over 90% by heating with cuprous cyanide and pyridine.

Experimental

From 1-Bromonaphthalene.—In a dry flask fitted with a ground-in condenser and protected from moisture were placed 33.0 g. (0.16 mole) of bromonaphthalene, b. p. 153–154° at 11 mm., 17.3 g. (0.19 mole) of cuprous cyanide, and 20 cc. of pyridine (dried over barium oxide). The mixture was heated at 220° for fifteen hours, and the resulting brown solution poured into dilute ammonia in a separatory funnel. After vigorous shaking for several minutes, the nitrile was extracted with ether and the extract washed successively with dilute ammonia, water, dilute hydrochloric acid, water, and saturated sodium chloride solution. After the first treatment with ammonia it was usually necessary to remove some dark insoluble matter by filtration as this seriously interfered with separation of the layers. The ether was removed and the residue vacuum distilled. The temperature rose abruptly to the boiling point and the yield of colorless nitrile, b. p. 173–174° at 27 mm., was 22.8 g., or 93%. This material had a melting point of 34–35° and on hydrolysis with sulfuric acid–acetic acid–water yielded pure 1-naphthoic acid, m. p. and mixed m. p. 162–163°.

From 1-Chloronaphthalene.—In an experiment similar to the above, 18.1 g. of 1-chloronaphthalene, b. p. 144–146.5° at 29 mm., 12.0 g. of cuprous cyanide and 10 cc. of pyridine were heated at 245–250° (temperature of bath) for twenty-four hours. The yield of 1-naphthonitrile was 15.6 g., or 92%. In an experiment in which the heating period was only six hours, the yield was 70%, a fore run of unreacted 1-chloronaphthalene being obtained.

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[1] Rupe and Brentano, *Helv. Chim. Acta*, **19**, 581 (1936); see also McRae, THIS JOURNAL, **52**, 4550 (1930); and Clarke and Read, *ibid.*, **46**, 1001 (1924).

[2] German Patent, 293,094 (1916).

[3] Von Braun and Manz, *Ann.*, **488**, 116 (1931); Koelsch, THIS JOURNAL, **58**, 1329 (1936); Fieser and Seligman, *ibid.*, **58**, 2482 (1936); Fieser and Hershberg, *ibid.*, **59**, 394 (1937); Lock, *Ber.*, **70**, 926 (1937).

Ethoxyethylphenylurea

BY E. WERTHEIM

Continuing a study of compounds related to Dulcin, as suggested in an earlier paper,[1] the compound ethoxyethylphenylurea has been made. In this compound the aromatic ring and aromatic attachment of the carbamido group previously found to be essential to sweet taste have been retained, but the ethoxy group is given an aliphatic attachment. The sequence of four carbons between the taste groups, as in Dulcin, is retained. The compound was found to have only a very faint sweet taste.

Experimental

Ethylphenethyl Ether, $C_6H_5CH_2CH_2OC_2H_5$.—One hundred twenty grams of phenylethyl alcohol was treated with 5 g. of sodium, and subsequently with 30 g. of ethyl bromide. The precipitated salt was removed by filtration and washed with dry ether. The filtrate was heated and aerated with dry air to remove ether and excess ethyl bromide. The material was again treated with 5 g. of sodium, and the above cycle of operations was repeated until about 20 g. of sodium and some excess over the equivalent amount of ethyl bromide had been used. The material was then washed with water, dried, and fractionally distilled; b. p. 198–199°; $n^{32}\text{D}$ 1.4870.

Anal. Calcd. for $C_{10}H_{14}O$: C, 80.00; H, 9.33. Found: C, 79.81; H, 9.54.

Ethyl-*o*-nitrophenethyl Ether, $C_2H_5OCH_2CH_2C_6H_4NO_2$.—In a typical experiment 59 g. of acetyl nitrate was

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[1] Wertheim, THIS JOURNAL, **57**, 545 (1935).