remove the fat and then extracting with acetone. After evaporation of the acetone the residue was extracted repeatedly with ether to remove traces of fat. The residue was then dissolved in a small quantity of water and treated with absolute alcohol; 1.4551 g. of a crystalline substance was obtained. From the specific rotation and reducing value following hydrolysis with invertase the substance was found to be sucrose.

The sample was dissolved in water and made to 25 ml. at 20°. The $[\alpha]^{20}\text{D}$ of the solution was found to be +66.45°. The theoretical value for sucrose is +66.48°. Five ml. of the solution was diluted to 500 ml. Twenty-five ml. aliquots containing 14.551 mg. of the substance were hydrolyzed with melibiase-free invertase. The reducing value was determined by a combination of the Quisumbing and Thomas and Bertrand methods. The average of five determinations was 14.72 mg. of sucrose; the theoretical amount was 14.55 mg.

Recently we have isolated sucrose from soybeans by two different methods.

In the first method soybean flakes were extracted with petroleum ether or with ether followed by extraction with 99% alcohol. The alcohol extract was concentrated on a steam-bath until sugar began to crystallize on the sides of the beaker and on a stirring rod standing in the solution. After standing for two days the crystals were filtered and washed with ether. A 5% solution of the first crop without recrystallization gave $[\alpha]^{20}\text{D}$ 65.81°. Refractive indices determined by means of the polarizing microscope gave $\alpha = 1.537$ $\beta = 1.565$, $\gamma = 1.57$. This is in agreement with the constants in Groth's "Chemische Krystallographie," Volume III, p. 448, namely, $\alpha = 1.5371$, $\beta = 1.5653$, $\gamma = 1.5705$. By this simple method sucrose can be obtained much easier and in larger yields than by the customary method in which lead acetate and barium hydroxide are used to clear the solution.

In the second method a 5-liter portion of an 80% alcohol extract of soybean flakes was concentrated before a fan to a thick sirup. About 2 liters of water was added and the solution was treated with lead acetate. The precipitate was filtered and the filtrate was treated with barium hydroxide. The lead and barium were removed with sulfuric acid. The solution was shaken with ether to remove acetic acid, and was then concentrated to a thick sirup. An equal volume of 99% alcohol was added and the sirup settled to the bottom. After stirring the sirup in the alcohol on the steam-bath the alcohol was decanted and let stand for several days. Crystals appeared in the alcohol extract. The first two fractions were combined and recrystallized. A 5% solution of these crystals gave a specific rotation of 66.57°. The refractive indices were the same as those of pure sucrose.

DEPARTMENT OF AGRICULTURAL CHEMISTRY
PURDUE UNIVERSITY AGRICULTURAL EXPERIMENT STATION
LAFAYETTE, INDIANA, AND
THE REGIONAL SOYBEAN INDUSTRIAL PRODUCTS LABORATORY
U. S. DEPARTMENT OF AGRICULTURE
URBANA, ILLINOIS
RECEIVED SEPTEMBER 7, 1937

Efficiency at 90° of Phosphorus Pentoxide as a Desiccant

BY DONALD A. LACOSS AND ALAN W. C. MENZIES

Information as to the efficiency of this desiccant at other than room temperatures appears to be absent from the literature. Accordingly, in order to avoid any mere assumptions about the matter, we made the experiment here described at a time when we contemplated desiccation experiments at temperatures near 100°.

A current of air was passed at the rate of about 650 cc. per hour through our apparatus train for thirty-five days, namely, about 558 liters in all. This entered successively (1) a 500-cc. calcium chloride tower, (2) a series of three helical type washing bottles containing sulfuric acid of such a concentration as to yield an aqueous pressure of about 1 mm.,[1] (3) a U-tube of about 80-cc. capacity charged with Baker "plugs" of purified phosphorus pentoxide in half-inch (1.25-cm.) layers separated by layers of ignited asbestos,[2] (4) an ordinary stoppered 30-cc. U-tube containing magnesium perchlorate, (5) a U-tube of purified phosphorus pentoxide charged like (3) and of size similar to (4), (6) a calcium chloride guard tube, (7) a calibrated gas meter and (8) an aspirating device. Connections for the weighing tubes (4) and (5) were lubricated ground joints. Tube (3) was maintained at 90° in a thermostat, while the rest of the train was at room temperature.

Tubes (4) and (5) were weighed from time to time against counterpoises which were handled in a like manner prior to weighing. Although over 0.5 g. of water had entered tube (3) as vapor, no change in weight was found in either (4) or (5).

---

(1) *Cf.* E. M. Collins, *J. Phys. Chem.*, **37**, 1191 (1933).

(2) *Cf.* H. B. Baker, *J. Chem. Soc.*, **65**, 611 (1894).

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.

FRICK CHEMICAL LABORATORY
PRINCETON UNIVERSITY
PRINCETON, N. J.
RECEIVED SEPTEMBER 21, 1937

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.

CHEMISTRY LABORATORY
OHIO STATE UNIVERSITY
COLUMBUS, OHIO
RECEIVED SEPTEMBER 27, 1937

---

[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

---

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