into about 1 ml. of the alcohol.[3,4,5] After washing the allophanate with ether, it was recrystallized to constant melting point. Myristyl alcohol was first dissolved in a few milliliters of benzene.

Purification of the Alcohols.—The alcohols were dried over solid potassium hydroxide, filtered and distilled through a Pyrex column (2.2 × 75 cm.), packed with $^1/_8$-in. single turn Pyrex helices, and equipped with a total reflux partial take-off distilling head. A constant boiling middle cut was taken for conversion to the allophanate. Myristyl alcohol was recrystallized twice from benzene and twice from methanol to constant melting point.

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(3) J. Liebig and F. Wöhler, *Ann. Physik*, **20**, 369 (1830); *Ann.*, **59**, 291 (1846).

(4) A. Béhal, *Bull. soc. chim.*, [4] **25**, 373 (1919).

(5) M. A. Spielman, J. D. Barnes and W. J. Close, THIS JOURNAL, **72**, 2520 (1950).

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RECEIVED JUNE 29, 1950

Bis-(β-chloroethyl)-cyclohexylamine Hydrochloride[1]

Bis-(β-hydroxyethyl)-cyclohexylamine[2,3] (5.14 g., 0.027 mole) was converted to the hydrochloride by addition of concentrated hydrochloric acid (10 ml.). The excess acid and water were removed under reduced pressure. The residual oil was covered with benzene (10 ml.) and 4.9 ml. (0.068 mole) of thionyl chloride was added dropwise. After refluxing for ten minutes and cooling, a crystalline product separated. This crystalline material was removed by filtration and washed with acetone to give a crude yield of 5.75 g. (80.5%). One crystallization from acetone (61 ml./g.) failed to raise the melting point above that of the original of 176–178° (dec.).

Anal. Calcd. for $C_{10}H_{20}Cl_3N$: C, 46.08; H, 7.73; Cl, 40.81; N, 5.38. Found: C, 46.57; H, 7.62; Cl, 40.84; N, 5.54.

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(1) Bis-(β-chloroethyl)-cyclohexylamine and its hydrochloride have been mentioned in a table of vapor pressures by C. E. Redemann, *et al.*, THIS JOURNAL, **70**, 1648 (1948), but physical constants and analyses have not been published.

(2) J. P. Bain and C. B. Pollard, *ibid.*, **61**, 2704 (1939).

(3) F. F. Blicke and C. E. Maxwell, *ibid.*, **64**, 428 (1942).

CONTRIBUTION FROM THE
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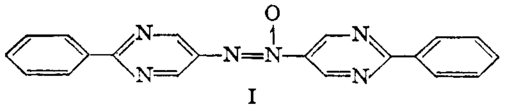
RECEIVED MAY 15, 1950

2,2′-Diphenyl-5,5′-azoxypyrimidine

A solution of 1.00 g. of 5-nitro-2-phenylpyrimidine[1] in 25 ml. of dioxane was shaken for 25 minutes with 2 g. of one-year-old Raney nickel under a hydrogen pressure of 2 atmospheres. Concentration of the solution gave 0.34 g. (40% yield) of 2,2′-diphenyl-5,5′-azoxypyrimidine (I). Upon recrystallization from dioxane it was obtained in the form of fine, yellow needles, m. p. 303–305°.

Anal.[2] Calcd. for $C_{20}H_{14}N_6O$: C, 67.78; H, 3.98; N, 23.72. Found: C, 67.78; H, 3.81; N, 23.90.



I

The analytical values agree very closely with the empirical formula corresponding to structure I and are significantly different from the calculated values for other possible reduction products, such as the amine, the nitroso compound or the corresponding azo-compound. Further evidence for structure I is provided by a comparison of the ultraviolet absorption spectrum with that of azobenzene and azoxybenzene, Fig. 1. The spectrum of I has no maximum in the visible region, and in this respect resembles the spectrum of azoxybenzene rather than that of azobenzene.

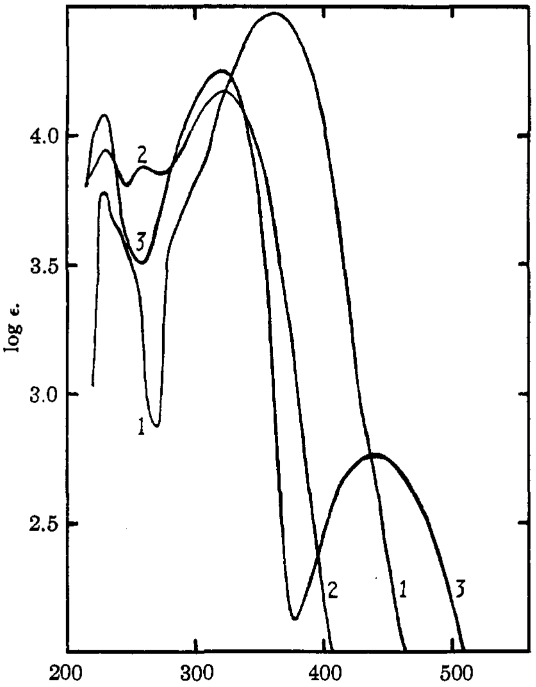


Fig. 1.—Molar extinction curves in 95% ethanol: (1) 2,2′-diphenyl-5,5′-azoxypyrimidine (I); (2) azoxybenzene; (3) azobenzene.

The only precedent found for this unexpected reduction product is a report[3] that nitrobenzene and several of its derivatives were deoxygenated by mixing with Raney nickel in an open beaker to give 35 to 50% yields of azo- and azoxybenzenes.

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(1) Hale and Brill, THIS JOURNAL, **34**, 91 (1912).

(2) Analysis by Micro-Tech Laboratories, Skokie, Illinois.

(3) Covert and Adkins, THIS JOURNAL, **54**, 4116 (1932).

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RECEIVED JUNE 29, 1950

Some Derivatives of Nitroguanidine[1]

The compounds listed in the Table I were prepared by the methods of McKay and Wright,[2] and McKay,[3] from 1-methyl-1-nitroso-3-nitroguanidine and the appropriate primary amine or hydrazine; they were recrystallized from aqueous ethyl alcohol.

1-Acetamido-3-nitroguanidine. Method A.—A solution of 11.9 g. of nitroaminoguanidine (0.1 mole), 30 ml. of glacial acetic acid and 10 ml. of acetic anhydride was

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(1) All melting points are corrected. The analyses were performed by Mr. Cliff Holford, Analytical Section, Chemistry Division, U. S. Naval Ordnance Test Station, and by Dr. Adalbert Elek, Los Angeles, California.

(2) McKay and Wright, THIS JOURNAL, **69**, 3028 (1947).

(3) McKay, *ibid.*, **71**, 1968 (1949).