

ethers containing iodine. The hydrochlorides were recrystallized from methanol, ethanol, methanol-ether, acetone, or ethyl acetate-ether.

The yields in the Williamson reactions averaged 25-35%. Low yields, sometimes below 5%, were obtained in several preparations involving dimethylaminoethyl chloride. Dialkylaminopropyl chlorides usually rendered the corresponding aryl ethers in yields of 50-90%. However, the optimum conditions have not been determined in all cases.

1-(2,4,5-Trichlorophenoxy)-2-*n*-butylaminoethane was prepared from *n*-butylaminoethyl chloride in an analogous manner. The yield was 18%.

2,4,5-Trichlorophenoxy Diethylaminomethane.—Diethylaminomethyl chloride hydrochloride was obtained in 81% yield by the direction of Prévost and de Mauny¹ and condensed with sodium 2,4,5-trichlorophenolate according to the procedure outlined above. The yield was 5%.

Chemical Analysis.—Most of the hydrochlorides listed in Table I were titrated in water or dilute alcohol solution with 0.05 *N* potassium hydroxide solution to a phenolphthalein end-point.

For several hydrochlorides, and water-insoluble deriva-

tives, the total halogen content was determined by the method of Schwenk, Papa and Ginsberg,⁷ using samples of 30-80 mg. and correspondingly small amounts of the required reagents. The silver nitrate and potassium thiocyanate solutions were 0.05 *N*. Reliable results were obtained on this semi-micro scale with most of the compounds; those derivatives analyzed for carbon, hydrogen or nitrogen did not give consistent halogen values by reduction with Raney nickel alloy.

Summary

A number of alkyl and dialkylaminoalkyl ethers of phenol and halogenated phenols with various substituents has been described. It has been found that nuclear iodine is not a prerequisite for the antitubercular action of such compounds.

(7) Schwenk, Papa and Ginsberg, *Ind. Eng. Chem., Anal. Ed.*, **15**, 576 (1943).

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NOTES

Isolation of Rutin from *Hydrangea Paniculata*, Var. *Grandiflora* Sieb.

BY JAMES F. COUCH AND JOSEPH NAGHSKI

Rutin, 3,5,7,3',4'-pentahydroxyflavone-3-rutinoside, has recently assumed some prominence in the treatment of increased capillary fragility associated with hypertension^{1,2} and is promising as a remedy for certain other diseases resulting from capillary breakdown. Rutin has been found in thirty-three species of plants and is, thus, one of the most widely distributed of the glucosides. This paper reports the isolation and identification of rutin in the flowers of a common garden species of *Hydrangea*. Previous chemical examinations of the roots of white-flowered species of *Hydrangea* have been reported.^{3,4,5} Hashimoto and Kawana⁶ extracted the dried flowers of *H. paniculata* with benzene and obtained a phenolic substance, C₉H₆O₃, but they do not mention rutin. The presence of rutin in relatively large quantities in the flowers has not previously been reported.

Experimental.—Fresh blossoms (67.5 g., moisture, 83.6%) were digested with alcohol (300 ml.) for several hours. The solvent was removed from the filtered extract. The residue was freed from fats and resins with benzene

and the insoluble matters were extracted with boiling water. On cooling and standing, 0.4 g. of rutin crystallized, m. p. 183-185°; raised by recrystallization from boiling water to 190-192°. A further crop, 0.05 g., was obtained by re-extracting the insoluble matters with boiling water; yield, 0.45 g. or 4.06% of the moisture-free plant.

*Anal.*⁷ Calcd. for C₂₇H₃₀O₁₆: C, 53.10; H, 4.95. Found: C, 53.34; H, 5.09.

The substance gave the usual tests for the identification of rutin. These data were confirmed on a larger sample (1.6 kg.) of fresh flowers.

(7) C and H determinations by C. L. Ogg.

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sym-Tetraphenylethane from DDT and Related Compounds¹

BY ELMER E. FLECK, ROBERT K. PRESTON AND H. L. HALLER

During an investigation of the effect of various solvents on the dehydrochlorination of 1-trichloro-2,2-bis-(*p*-chlorophenyl)-ethane (known as DDT),² it was noted that an abnormal reaction took place in the presence of anhydrous aluminum chloride and benzene. When one mole of anhydrous aluminum chloride was used with a large

(1) Some of the work reported was done under a transfer of funds, recommended by the Committee on Medical Research, from the office of Scientific Research and Development to the Bureau of Entomology and Plant Quarantine. Article not copyrighted.

(2) Fleck and Haller, *This Journal*, **66**, 2095 (1944).

(1) J. Q. Griffith, J. F. Couch and M. A. Lindauer, *Proc. Soc. Exp. Med. Biol.*, **55**, 228-229 (1944).

(2) J. F. Couch and C. F. Krewson, United States Department of Agriculture, Mimeograph Circular AIC-52, July, 1944.

(3) C. S. Bondurant, *Am. J. Pharm.*, **59**, 122-124 (1887).

(4) A. G. Leubert, *ibid.*, **70**, 550-552 (1898).

(5) H. J. M. Schroeter, *ibid.*, **61**, 117-118 (1889).

(6) A. Hashimoto and T. Kawana, *J. Pharm. Soc. Japan*, **55**, 183-186 (1935); *C. A.*, **29**, 5112 (1935).