

tion on alumina. *Anal. Calcd.* for $C_{12}H_8N_2O$: C, 73.4; H, 4.11. Found: C, 73.8; H, 4.25.

The 2-chlorophenazine was made by the method of Waterman and Vivian,⁵ starting from 4-chloro-2-nitrodiphenylamine.

The antitubercular activity of the phenazine dioxides⁶ makes both of the new dioxides reported above of interest, especially the alkali-soluble hydroxy compound; and by this synthesis the 2-phenazinol itself is rendered more easily accessible.

(5) Waterman and Vivian, *J. Org. Chem.*, March, 1949; U. S. Patent 2,292,808 (Aug. 11, 1942).

(6) Ihland, *Nature*, **161**, 1010 (June 26, 1948). Antitubercular activity of certain other phenazines: Barry, Belton, Conalty and Twomey, *ibid.*, **162**, 622 (Oct. 16, 1948).

CHEMOTHERAPY SECTION

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PREPARATION OF HIGH-PURITY HYDROGEN DEUTERIDE FROM LITHIUM ALUMINUM HYDRIDE

Sir:

Hydrogen deuteride of very high purity has been prepared previously by the fractional distillation of a mixture of H_2 , D_2 , and HD at liquid hydrogen temperatures.¹ We have now found that very pure HD can be prepared more simply by the action of certain metallic hydrides on heavy water. Thus, sodium hydride with heavy water gives 87% HD with H_2 and D_2 as impurities. Lithium aluminum hydride with heavy water gives 93% HD in some cases and in others 97%, with 2.5% H_2 + 0.5% D_2 .² Determinations were made on a consolidated mass spectrometer for which instrumental condition were: magnet current of 0.15 ampere and voltages of 3800 for mass 1 (H^+), 1900 for mass 2 (H_2^+ and D^+), 1267 for mass 3 (HD^+), and 950 for mass 4 (D_2^+).

It was suspected that the heat evolved in the reaction promoted the formation of D_2 and H_2 . Accordingly the preparation with lithium aluminum hydride was carried out in a bath at 0°, with the resulting formation of 99% pure HD. The exact nature of the temperature dependence is not known, but the variation in composition of the gas product may be due to a shifting of the $H_2 + D_2 \rightleftharpoons HD$ equilibrium, as well as to the presence of impurities in the lithium aluminum hydride.

Our preferred procedure for this preparation, therefore, was as follows:

Pure D_2O (99.8%) contained in a hypodermic syringe was injected through a neoprene serum stopper, into a stirred ice-cold slurry of lithium aluminum hydride in *n*-butyl ether contained in the reaction flask of a modified Zerewitinoff apparatus.³ After the vigorous reaction subsided,

(1) Scott and Brickwedde, *Phys. Rev.*, **48**, 483 (1935).

(2) Beutler, Brauer and Junger, *Naturwissenschaften*, **24**, 347 (1936) reported the preparation of a mixture rich in HD by the action of lithium hydride on heavy water.

(3) Orchin and Wender. *Anal. Chem.*, in press.

the evolved gas was collected in an evacuated sample bottle.

This very simple preparation of HD from lithium aluminum hydride and knowledge of its fragmentation pattern should improve the mass spectrometric analyses of H_2 , D_2 and HD mixtures, especially those low in H_2 .

We wish to acknowledge the helpful assistance of A. G. Sharkey, Jr.

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BOOKS RECEIVED

January 10, 1949—February 10, 1949

EDWARD S. AMIS. "Kinetics of Chemical Change in Solution." The Macmillan Company, 60 Fifth Avenue, New York, N. Y. 1949. 332 pp. \$5.00.

MELVIN CALVIN, CHARLES HEIDELBERGER, JAMES C. REID, BERT M. TOLBERT AND PETER F. YANKWICH. "Isotopic Carbon." John Wiley and Sons, Inc., 440 4th Avenue, New York 16, N. Y. 1949. 376 pp. \$5.50.

HANS T. CLARKE, JOHN R. JOHNSON AND SIR ROBERT ROBINSON, EDITORS. "The Chemistry of Penicillin." Princeton University Press, Princeton, N. J. 1949. 1094 pp. \$36.00.

SAUL DUSHMAN. "Scientific Foundation of Vacuum Technique." John Wiley and Sons, Inc., 440 Fourth Avenue, New York 16, N. Y. 1949. 882 pp. \$15.00.

JOSEPH E. FLYNN, EDITOR. "Blood Clotting and Allied Problems." Josiah Macy, Jr. Foundation, 565 Park Avenue, New York, N. Y. 1948. 179 pp. \$3.25.

DAVID GLICK. "Techniques of Histo- and Cytochemistry." Interscience Publishers, Inc., 215 Fourth Avenue, New York 3, N. Y. 1949. 531 pp. \$8.00.

JOHN HONEYMAN. "An Introduction to the Chemistry of Carbohydrates." Oxford University Press, 114 Fifth Avenue, New York 11, N. Y. 1949. 143 pp. \$4.50.

ELVIN A. KABAT AND MANFRED MAYER. "Experimental Immunochemistry." 2nd Edition. Charles C. Thomas, Publisher, 301-327 East Lawrence Avenue, Springfield, Ill. 1949. 567 pp. \$8.75.

HOWARD W. POST. "Silicones and Other Organic Silicon Compounds." Reinhold Publishing Corporation, 330 West 42nd Street, New York 18, N. Y. 1949. 230 pp. \$5.00.

FRANCIS OWEN RICE AND EDWARD TELLER. "The Structure of Matter." John Wiley and Sons, Inc., 440 Fourth Avenue, New York 16, N. Y. 1949. \$5.00.

L. ROSENFELD. "Nuclear Forces." Part II. Interscience Publishers, Inc., 215 Fourth Avenue, New York 3, N. Y. 1949. 543 pp. \$7.50.

HENRY TAUBER. "The Chemistry and Technology of Enzymes." John Wiley and Sons, Inc., 440 Fourth Avenue, New York 16, N. Y. 1949. 550 pp. \$7.50.

N. TROENSEGAARD. "On the Structure of the Protein Molecule." 2nd Edition. Einar Munksgaard, Publisher, Copenhagen, Denmark. 1944. 126 pp. Dan. cr. 14.00.