more may be obtained readily by reducing the volume of the mother liquor to about one-fifth. Slightly yellow colored crystals of 90 + % purity have been obtained from several lots of fullers' earth in yields of about 28 to 50%. After recrystallization from 97–98% ethanol, pure white crystals melting at 243–244° (uncorr.) are obtained. When tested by a modified Smith rat curative method, such crystals contained an average of 325,000 International units of vitamin $B_1$ per gram, which is fully as active as the synthetic vitamin. Although most of the work has been done with rice polish, crystals have also been obtained from yeast, but the yields have been lower. Table III contains the results of a number of typical lots, as well as the results of two batches in which phenol and alkali were used in place of pyridine hydrochloride.

## Summary

Pure crystalline vitamin $B_1$ has been prepared from rice polish or yeast by a process which involves two adsorptions on fullers' earth, extraction with acid salts of nitrogen bases such as pyridine, and the use of organic solvents.

A study of the distribution of vitamin $B_1$ between water and certain immiscible organic solvents has shown that a number, especially phenol, have high solubility for $B_1$. The presence of sodium chloride in the water in many cases has a marked effect upon the distribution.

Combinations of phenol and butyl alcohol are very useful for the removal of inert material from $B_1$ concentrates and afford a good medium for its crystallization.

Charcoal has been used to remove substances which interfere with the adsorptions of $B_1$ on fullers' earth, both before the original adsorption and before the subsequent readsorption.

NEW BRUNSWICK, N. J. RECEIVED MAY 20, 1937

# NOTES

## The Preparation of Isopropyl *m*-Tolyl Ether from *m*-Cresol and Isopropyl Chloride[1]

BY THOMAS BOYD WITH ED. F. DEGERING

Synthetic thymol[2] is prepared by the rearrangement of isopropyl *m*-tolyl ether which, heretofore, has been synthesized from *m*-cresol and isopropyl alcohol[3] or bromide.[4] Better yields are obtained, however, from *m*-cresol and isopropyl chloride by use of the following procedure:

Place 25 g. of *m*-cresol, 30 ml. of isopropyl chloride, 9.2 g. of sodium hydroxide pellets, and 75 ml. of isopropyl alcohol in a 500-ml., electrically heated and thermostatically controlled autoclave. Slowly during one hour bring the temperature to about 150° and maintain a temperature range of 150–160° for three hours more.

Allow the bomb to cool, then empty and rinse in turn with water and with two 15-ml. portions of benzene. Extract the mixture and washings with three 25-ml. portions of benzene,[5] then wash the benzene extract with three 20-ml. portions of 15% sodium hydroxide solution.[6] Wash the benzene layer with water, dry with anhydrous potassium carbonate, and recover the solvent by distillation. Rectify the residue through a modified Podbielniak column, and collect a fraction at 193–197°; yield, about 23–27 g.

Various factors such as temperature, period of heating, the nature and quantity of the solvent, and the method of preparing the reaction mixture have been quite thoroughly studied. The best results were obtained by the procedure outlined above.

THE CHEMISTRY DEPARTMENT
PURDUE UNIVERSITY
LAFAYETTE, INDIANA

RECEIVED APRIL 26, 1937

(1) Presented before the Organic Division of the Fourteenth Midwest Regional meeting of the American Chemical Society, Omaha, Nebraska, May 1, 1937.

(2) J. B. Niederl and Samuel Natelson, THIS JOURNAL, **53**, 1928–34 (1931); F. J. Sowa, H. D. Hinton and J. A. Nieuwland, *ibid.*, **54**, 2019 (1932), and **55**, 3402 (1933); Richard A. Smith, *ibid.*, **56**, 717 (1934), and **55**, 849 (1933).

(3) Hashichi Ono and Minoni Imoto, *J. Soc. Chem. Ind.*, Japan, Suppl. Binding, **39**, 170 (1936); M. D. Curwen, *Ind. Eng. Chem., News Ed.*, **14**, 413 (1936).

(4) J. B. Niederl and Samuel Natelson, THIS JOURNAL, **53**, 1928–1934 (1931); **54**, 1063 (1932); F. J. Sowa, H. D. Hinton and J. A. Nieuwland, *ibid.*, **54**, 2019 (1932); Richard A. Smith, *ibid.*, **56**, 717 (1934).

(5) As a check, acidify the water layer with concd. hydrochloric acid. If an oily layer forms, extract with benzene and add to the previous extract.

(6) Acidify the sodium hydroxide extract and washings. Extract with benzene, and distil the solvent. The residue, 3–6 g., is recovered *m*-cresol.