The residue was cooled, extracted with four 100-ml. portions of chloroform, the organic layer dried with drierite and the solvent removed by distillation on the steam-bath. The solvent-free residue was distilled *in vacuo*, b. p. 144° (0.06 mm.) in an all-glass-interjoint apparatus to give a light yellow oil, which could not be induced to crystallize; yield, 60%.

The base was identified as the dipicrate, which was prepared in anhydrous diethyl ether and recrystallized several times from a large volume of methanol, m. p. 168–169° (cor.).

Anal. Calcd. for $C_{30}H_{33}N_9O_{15}$: C, 47.44; H, 4.38. Found: C, 47.54; H, 4.50.

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RECEIVED AUGUST 4, 1944

COMMUNICATIONS TO THE EDITOR

CINCHONA ALKALOIDS PREPARED BY ION EXCHANGE

Sir:

An economical process for the isolation of alkaloids from low-grade cinchona barks has been a prime necessity since the loss of the Far Eastern sources of quinine and its raw materials. During a study of the acid extraction of South American bark, it was decided to investigate the use of cation-exchange adsorbents as a means of increasing the efficiency of extraction.

Three possibilities were seen for the application of ion exchange to cinchona extraction, namely: to recover alkaloids from the mother liquors of acid extracts of the bark after the major portion had been removed by alkaline precipitation; to purify the crude totaquine obtained from alkaline precipitation; to use ion exchange directly in the acid extraction of the bark in a cyclic system so that the bark is constantly percolated or extracted by an acid medium free from alkaloids. These experiments provide specific information for the first two of the above-mentioned applications. Cyclic extraction has been studied and will be reported upon at a later date.

Basic facts as to the adsorption capacity of the cation-exchanger for cinchona alkaloids were obtained using quinine as a representative alkaloid. Capacity determinations were run on a two-hundred mm. "Zeo-Karb" column[1,2] using quinine concentrations of 0.033 and 0.0033M and flow rates of approximately 5 and 50 ml./min., respectively.

The capacity of a 200-ml. bed of "Zeo-Karb" for quinine from acid solution (1% H_2SO_4) was found to be between 7 and 8 g. before breakthrough (Mayers reagent). To liberate the alkaloids from the column, ammoniacal alcohol was used as a combined regenerant and elution solvent. After the exchanger was used once or twice, recoveries were almost quantitative.

The purification of totaquine prepared by alkaline precipitation of an acid extract of the bark was now attempted by ion exchange. From 20 g. of the crude totaquine precipitate, 2.5 g. of a white crystalline material was obtained. A comparison of the properties of alkaline precipitated totaquine and the alkaloid prepared from it by ion-exchange is given in the table.

	Totaquine	
	Alkaline precipitated	Ion exchange
Color	Dark reddish brown	White
Form	Amorphous powder	Crystalline
Total alkaloids, %	23.4	94
Sol. in acid, %	Approx. 45	100
Sol. in $CHCl_3$, %	Approx. 20	100

The results suggest that ion exchange could prove a valuable aid in the extraction of cinchona by enabling the recovery of alkaloids which would otherwise be lost in the mother liquors following alkaline precipitation. Ion exchange also represents an excellent technique for purifying crude totaquine preparations, improving solubility, appearance, and removing non-alkaloidal, non-ionic contaminants.

The writer also has used this technique successfully in the isolation of atropine, scopolamine and morphine and will report more fully upon these experiments.

This work was performed at the Rutgers University College of Pharmacy in conjunction with the research program of the Foreign Economic Administration supervised by Professor Martin S. Ulan. The helpful advice of Dr. F. C. Nachod of the Permutit Company is gratefully acknowledged.

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RECEIVED OCTOBER 11, 1944

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(1) The Permutit Co., N. Y.

(2) F. C. Nachod and S. Sussman, *J. Chem. Ed.*, **21**, 56 (1944).

AN UNIDENTIFIED GROWTH FACTOR FOR A GAS GANGRENE CLOSTRIDIUM[1]

Sir:

In an investigation of the nutritional requirements of *Clostridium perfringens* it was found that a complex synthetic medium, such as that supporting the growth of *Clostridium tetani,* was inadequate for the growth of *Clostridium perfringens.*

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(1) This work was supported by a grant from the Josiah Macy, Jr., Foundation.