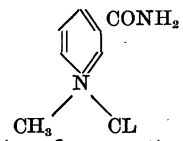
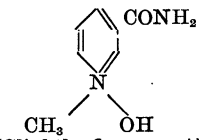
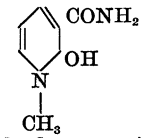
ment, is in large part converted into a carbinol. Such carbinols combine readily with alcohols to form carbinol-ethers. In the case of N-methyl chloro-nicotinamide, when treated with alkali and isobutanol, the reactions would be as follows:



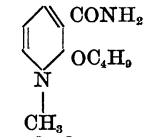
(non-fluorescent)
Quaternary salt



(Slightly fluorescent)
Quaternary base



(Slightly fluorescent)
Carbinol



(Very strongly fluorescent)
Carbinol-ether = F_2

Upon treatment of purified urinary eluate and of N-methyl chloro-nicotinamide with alkali, fluorescence develops at once, though consistently more rapidly with the former. The absorption spectra of both products are nearly identical (max. 264 μ, min. 250 μ). We were led to the conclusion that the resulting carbinol must form a condensation product with the alcohol from the observation that minute additions of isobutanol to the alkaline carbinol solution, insufficient to cause separation of an alcohol layer, caused nevertheless a very striking increase in fluorescence. The formation of a carbinol-ether is not an instantaneous reaction, but continues to progress even after an isobutanol extract has been made,[2] which explains the hitherto puzzling increase in fluorescence on standing. When the isobutanol extract is evaporated to dryness the carbinol-ether is broken down, leaving the carbinol itself.

We have prepared highly concentrated F_2 solutions which were evaporated to dryness yielding a waxy yellowish solid which could be crystallized from methanol. The elementary analysis of these crystals was found to correspond with the formula of the carbinol with the exception that the nitrogen content was only half as great. The latter finding was anticipated in view of the prolonged alkali treatment which was noted to liberate ammonia, presumably from hydrolysis of the amide. The elementary analyses of our product obtained from urine and the theoretical values of the carbinol of N-methyl nicotinic acid, are as follows:

	N	*C*	*H*
Product from urine	9.7	58.1	7.2
N-methyl nicotinic acid carbinol	9.1	54.0	6.0

In conclusion we feel that the complete structure of the F_2 precursor is not yet established, although it appears certain that it is a derivative of N-methyl nicotinamide. The highly fluorescent compound F_2 formed from this precursor on treatment with alkali and butanol appears to be a butyl ether of N-methyl nicotinamide α-carbinol.

VICTOR A. NAJJAR
VIRGINIA WHITE

DEPARTMENT OF PEDIATRICS,
JOHNS HOPKINS UNIVERSITY

SCIENTIFIC APPARATUS AND LABORATORY METHODS

A SIMPLIFIED LYOPHIL APPARATUS

THE lyophil apparatus described here is a compact and efficient piece of equipment which has proved very satisfactory for small-scale laboratory work. As shown in Fig. 1, the diffusion path of water vapor is short and the cross sectional area is large. The outer jacket has one small opening (A) at the top for evacuating the inner chamber and four openings (B) in the lower portion to which drying flasks are attached. These openings are made with standard taper 34/45 short female ground glass joints. The apparatus in Fig. 1 shows three joints on the side and one at the bottom. The dimensions given here are large enough to accommodate four flasks instead of three on the side if desired. There may also be some advantage in having the joints come off at a downward angle.

The flasks used are pear-shaped in order to facilitate removal of dried material. They are fitted with 34/45 male short joints. The joints are sealed with a film of stopcock grease. The condensing surface (C) is tapered in order to permit accumulation of a greater volume of ice. The apparatus as described in Fig. 1 will hold about 400 ml of ice on the condensing surface.

For operation, the condenser cone is filled with a freezing mixture of dry ice and ethyl cellosolve. The drying flasks are then filled to about 25 per cent. of their capacity with the solution to be dried and are placed in a dry ice freezing mixture. In order to obtain an even layer of material on the walls, the flask is held at an angle and rotated until all the material has frozen solid. When all the flasks are prepared, they are connected to the condenser and the assembly is evacuated. It is convenient to stopper unused openings of the condenser with sealed-off standard tapers which may also be used for drying small samples of material. The temperature inside the flasks depends upon the rate of evaporation, and under the

described conditions is well below freezing until all the water has been removed. The rate of evaporation may be increased by blowing a current of air over the flasks or immersing them in cold water. Materials being dried from very dilute solutions have a tendency to be carried out of the flask with the current of water vapor; this may be prevented without causing any appreciable decrease in evaporation by placing a gauze screen over the opening of the flask.

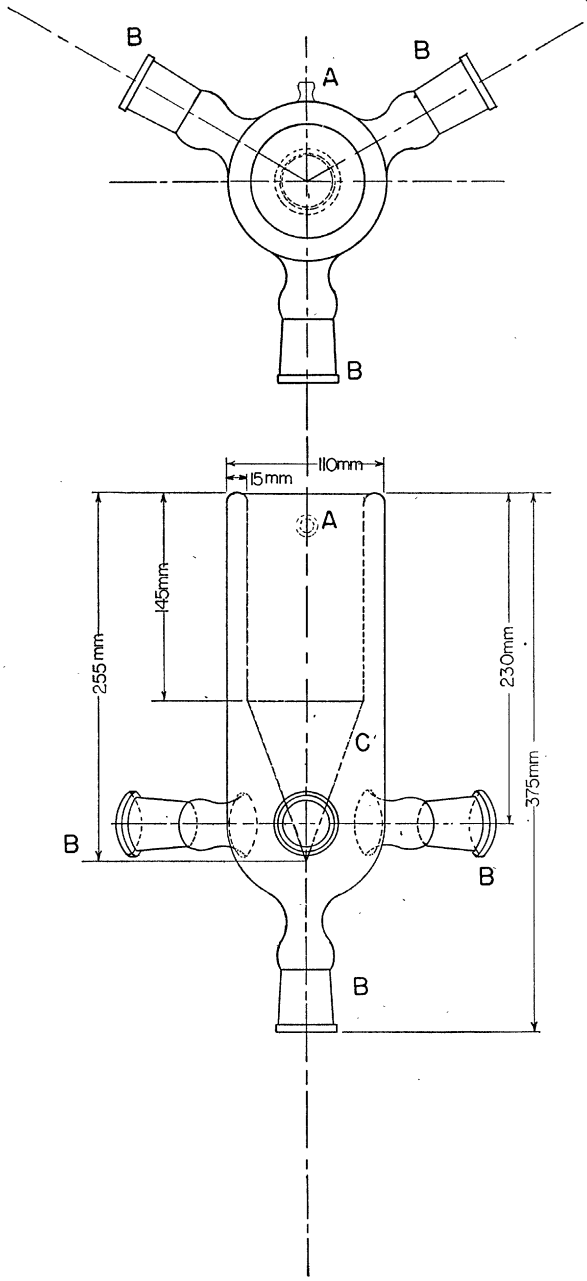


FIG. 1. Diagram of lyophil apparatus showing side and cross section views.

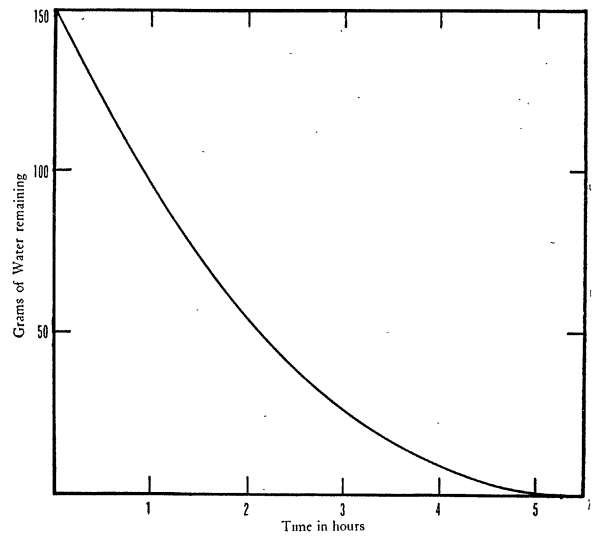


FIG. 2. Amount of water remaining in flasks, plotted against time.

Fig. 2 shows the amount of water remaining as a function of time for 150 gms of distilled water distributed equally among three 200 ml flasks. Complete dryness was achieved in about five hours. The decrease in rate at the end is mainly due to decrease in surface of the subliming ice. Water is evaporated from protein solutions at a comparable rate, depending to some extent on the hygroscopic nature of the material.

DAN H. CAMPBELL
DAVID PRESSMAN

GATES AND CRELLIN LABORATORIES
OF CHEMISTRY,
CALIFORNIA INSTITUTE OF TECHNOLOGY

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