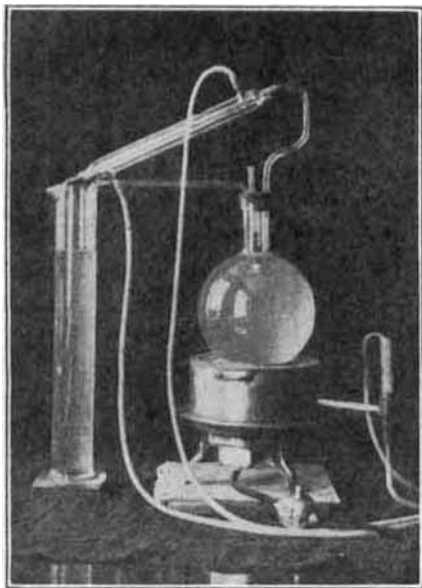


APPARATUS FOR CONTINUOUS PRECIPITATION BY COOLING.*†

BY M. R. MILLER.

Received for Publication, August 29, 1922.

A number of operations, particularly in the preparation of organic compounds, call for solution of a crude material in hot solvent and the recovery of a more pure substance by cooling the hot solution. The process depends obviously on the difference in solubility of the substance sought at different temperatures. An arrangement of apparatus according to the following description and illustration makes this process a continuous one.



Apparatus for Continuous Precipitation.

The material to be extracted, usually a mass of crude product, is placed in the large flask with sufficient solvent to fill the flask. This is set on a hot plate or water-bath whereby the temperature of the solvent is kept just below the boiling point.

Through the stopper there pass three tubes, one, which is the leg of a siphon, extending nearly to the bottom of the flask: this provides for the return of the solvent to the flask after depositing its dissolved material in the cooling vessel. The second tube, which is the leg of a second siphon, extends to just below the surface of the liquid: this provides for the outflow of hot concentrated solution to the cooling vessel. The third tube is a short one by means of which the siphons are filled by pressure or suction when the apparatus is ready to start working. To this tube may be attached, if necessary, a reflux condenser to prevent the loss of solvent through the escape of vapor.

The cooling vessel in the illustration consists of a liter graduate but a liter flask or other deep vessel may be used. It is so set that when filled the surface of the liquid in it is level with that in the flask and is best placed in a cooling bath of water from the tap or a cooling mixture. Such a bath is not shown in the illustration.

The opposite leg of the first siphon is only long enough to extend barely below the surface of the liquid in the cooling vessel. By means of this siphon the cooled liquid returns from the cooling vessel into the flask or extracting vessel. The opposite leg of the second siphon extends two or three inches below that of the first and through this siphon comes the hot concentrated solution from the flask. This second siphon also forms the tube of a condenser, which is inclined somewhat

*From the Department of Research Chemistry, Nevada Agricultural Experiment Station, Reno.

† Published with the permission of the Director of the Nevada Agricultural Experiment Station.

and whose short jacket is supplied with water from the tap. The function of the condenser or cooler is to promote circulation by increasing the specific gravity of the solution.

When properly set up the hot concentrated solution flows through the siphon with the condenser, from the flask to the cooling vessel where precipitation takes place through cooling. The precipitated material falls to the bottom of the cooling vessel where it collects until removed. It was found that the gentle current set up through the action of the two siphons would carry but very little of the solid material back to the flask. By means of the apparatus the solvent dissolves the crude material, or the desired substance from it, deposits it on being cooled and returns to the flask for a further quantity—the advantages being continuous operation, requiring no attention, and economy of solvent.

This apparatus has served well in the writer's laboratory in the purification of saponin from the dried crude plant extract by precipitation from hot alcohol by the Schrader method.

ASSAY OF DIGITALIS BY INTRAMUSCULAR INJECTION IN THE FROG.

BY M. S. DOOLEY AND C. D. HIGLEY.*

There is no satisfactory method for the standardization of the digitalis bodies in spite of the extensive efforts that have been made to find one. The present paper describes a method which presents advantages over the procedures now followed.

Observers have criticized the present official method mainly on account of lack of uniform absorption from the lymph sac. It is toward the obviation of this particular difficulty that our experiments have been directed. This lack of uniform absorption is not merely inconvenient, but it interferes with the reliability of the method. This is indicated by the fact that more than twenty different methods have been proposed since Famulener and Lyon published the one-hour method in 1902.¹ In the search for a better method investigators have covered a wide field—chemical as well as biological—but, with the exception of the cat method, none has attracted much more than passing notice.

In common with other observers we have long recognized great variability in the results obtained in routine assays by the lymph sac method, especially when the attempt was made to utilize it in research. This may be illustrated by an experience of one of us (D.) while working in Dr. Hatcher's laboratory in the summer of 1920 (the season is here referred to because the variabilities appear to be greater at this time). The problem under consideration was the question of the rate of elimination of digitalis substances—a problem in which it was desirable to know the potency of a given specimen in advance of attempting the study of its rate of elimination. Poor absorption from the lymph sac rendered the one-hour method useless for this purpose and the twenty-four-hour toxic method was finally substituted.

I. ORIGIN OF THE PROBLEM AND DESCRIPTION OF THE METHOD.

During the course of the above experiments upon the elimination of digitalis substances mention was made of the feasibility of making injections into the

* Department Pharmacology, Syracuse University College of Medicine, Syracuse, N. Y.