"The Use of Logarithms and Antilogarithms in Pharmaceutical Assaying," by H. L. Thompson.

"The N. F. Petroxolins and Paralle Preparations," by H. A. Langenhan and G. J. Noll.

"Sterilized Distilled Water," by E. F. Cook and L. Gershenfeld.

"An Experiment on Compound Tincture of Benzoin," by J. C. and B. L. DeG. Peacock.

"Absence of Inspection under the Harrison Act," by J. C. Peacock.

This concluded the papers, all of which were referred for publication. Further nominations were called for, and after balloting on the names of those presented as candidates as associate members on the Committee, the officers for the ensuing year were reported to be as follows:

*Chairman*, R. W. Terry, Groveport, Ohio.

*Secretary*, Edward Davy, Columbus, Ohio.

*Associate Members*, William Gray and Irwin A. Becker, both of Chicago, Ill.

The new officers were installed. The retiring Chairman thanked his colleagues for their help, and the third session was adjourned by the newly-elected Chairman, Robert W. Terry.

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## CONTINUOUS PERCOLATION UNDER REDUCED PRESSURE—REPORT No. 1.*

BY J. G. BEARD.

This will be a preliminary report on the progress I have made to date on a new (or at least modified) process for percolating fluidextracts.

This process consists essentially of extracting drugs in the preparation of fluidextracts through the use of a specially designed percolater that keeps the drug constantly in contact with fresh menstruum but not new menstruum. It may be thought of as a modified form of Soxhlet extraction.

The apparatus involved consists of four parts: A generator which is a glass distilling flask having an upright side-arm tube to permit introduction of a thermometer and a mercury gauge for measuring pressure; a percolator shaped like the Oldberg form but having a lateral tube opening into the percolator above the top of the drug and also entering below the lower portion of the drug, the object being to allow vapors from the generator to pass around the powder to be extracted and reach the condenser; a double Soxhlet condenser to be used as in reflux operations; and a tube from the top of the condenser leading to a vacuum pump. All of these parts are connected together perpendicularly by means of tightly fitting rubber stoppers.

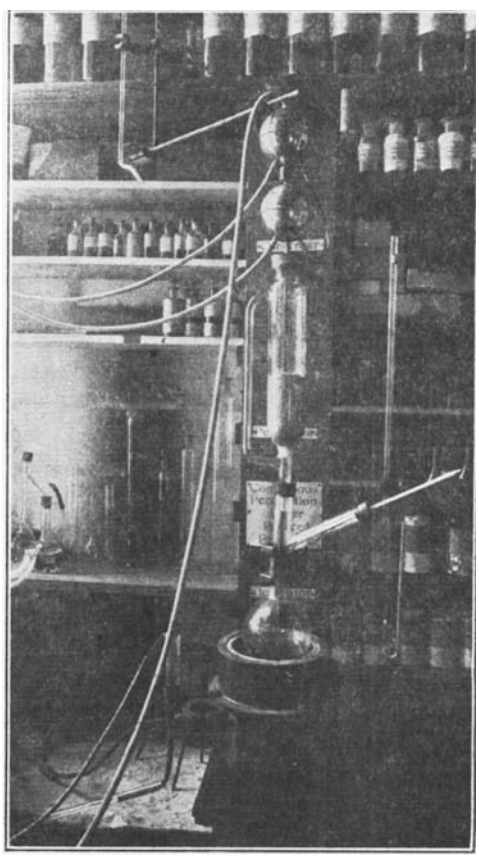
The process is conducted as follows:

The drug from which the fluidextract is to be made is macerated for sixty hours with enough menstruum to render it distinctly and uniformly damp. At the expiration of this time the drug is placed in the percolator, which has been provided with a pledget of cotton, in a succession of layers, moderately packing the drug after the addition of each layer. An amount of menstruum exactly equalling the volume of fluidextract to be made is placed in the generating flask. The parts of the apparatus are then tightly connected together by means of rubber stoppers. Low heat is applied to the generating flask from a constant level water bath, and when the thermometer registers a temperature of approximately 30° C. the vacuum pump is started. The heat and suction are carefully continued until such a pressure and temperature are obtained

* Read before Section on Practical Pharmacy and Dispensing, A. Ph. A., Chicago meeting, 1918.

in the apparatus as will enable the menstruum to boil between 35–40° C. With the source of heat remaining a constant, the tube leading to the vacuum pump is then tightly closed with a screw pinch-cock to seal the apparatus. The menstruum is rapidly vaporized on account of the reduced pressure and passes upward. When the vapors encounter the neck of the percolator they take the path of least resistance through the side-arm tube and thus pass around and out of contact with the drug. These vapors quickly reach the reflux condenser, are cooled, liquefied, and the condensed cold fluid drops straight down upon the drug where it acts precisely as in ordinary percolation, extracting the soluble principles in passing downward. When the percolate thus formed completes its passage through the drug it drops into the generating flask. The portion constituting the solvent is immediately vaporized again as fresh menstruum, and as such is capable of exercising the same solvent effects as on the first trip through the apparatus. The drug as a consequence is constantly subjected to the action of fresh solvent until finally all soluble matter is extracted and the process is completed.

When the operator believes that this point has been reached, he disconnects the generating flask and tests a small portion of the percolate as it leaves the neck of the percolator with an appropriate reagent, for example in the case of alkaloidal drugs, with Mayer's reagent. If the test is negative nothing further need be done beyond collecting the last drops of percolate and adding to the portion in the flask. If the test shows the presence of active principles the apparatus must be reconnected and the process continued for such time as in the judgment of the operator will have allowed the menstruum to complete the extraction. If the process has been carefully conducted the final volume of fluidextract will equal in mils the number of grammes of drug taken. Whether the finished product will be one hundred percent strong depends altogether upon the strength of the powdered drug taken. In the experiments so far conducted by the author the fluidextracts made by this process have represented in all cases a very close approximation in strength to that of the drug before extraction, and in some cases have tallied to two points to the right of the decimal.



Apparatus for Continuous Percolation under Reduced Pressure

In the case of alkaloidal drugs for which appropriate assays are prescribed by the Pharmacopoeia, the plan of the ninth revision can be followed and the fluidextract tested and brought to standard as well by this process as by the one official.

Up to the time this report was prepared the fluidextracts made were only

from drugs the official menstrua of which contained only alcohol, or alcohol and water. In the case of hydroalcoholic menstrua the water element has never been present in a greater ratio than one to three. There seems no apparent reason, however, why, for example, a glycerin, alcohol and water menstruum might not be used provided the first portion containing the non-volatile glycerin were added *to the drug* as in Type Process B.

A few fluidextracts like that of aconite are debarred from this process, since even the low heat of 35° C. would injure the active principles, but their number is very small. It should be borne in mind that in this process fluidextracts are made, as a result of reduced pressure, at a temperature no higher than normal blood heat.

Sufficient time has not elapsed to determine the keeping qualities of the fluidextracts made by Continuous Percolation under Reduced Pressure. The oldest preparation so made is from nux vomica—eight months old; however, in each case a fluidextract of the same drug was made by the U. S. P. method and reserved as a check. Both kinds of fluidextracts show the same degree of slight precipitation after standing several months.

The advantages which it is believed this process offers are threefold: (1) A saving of menstruum, the only loss being that amount retained by the drug after pressing. (2) Economy of time—after the process is started, the operator's time can be given to other work. (3) If the method be proved as practicable as incomplete experimentation leads one to believe, fluidextracts can be made at less cost than they can be purchased.

The fluidextracts which have been satisfactorily made by the above process are those of nux vomica, hyoscyamus, cannabis, guarana, pilocarpus, podophyllum, buchu, gentian, spigelia, staphisagria.

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## CARBON TETRACHLORIDE AS A SOLVENT FOR FATS.*

BY J. P. SNYDER.

The ninth edition of the pharmacopoeia has in the case of certain drugs which contain considerable quantities of fats and oils directed that these be removed by treating them with purified petroleum benzene. There is a decided improvement in the finished preparations over those of the U. S. P. VIII, which is particularly noticeable in Tincture of Strophanthus and Fluidextract of Colchicum Seed. Formerly, these preparations, when made according to the previous official formula presented a rather unsightly appearance and precipitated badly. The use of a solvent for the fats is evidently the logical method for the removal of these inert substances as our experience has been that it is preferable to attempting to freeze out the fats in the finished preparation.

Petroleum benzene, however, is open to serious objections: Firstly, as it is extremely difficult to drive off completely its peculiar odor which may be easily detected in the finished preparation.

* Read before Section on Practical Pharmacy and Dispensing, A. Ph. A., Chicago, meeting, 1918.